

EFFECTS OF AMORPHOUS CONSTITUENTS ON SOME MINERALOGICAL AND CHEMICAL PROPERTIES OF A PANAMANIAN LATOSOL*

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Abstract—This study was an investigation of the effects of sesquioxide constituents on some mineralogical and physiochemical properties of a Panamanian latosol.

Latosols are soils characterized by high concentrations of iron and aluminum oxides and a general absence of free silica and alkaline earths.

X-ray diffraction studies revealed sesquioxide coatings existed on the surfaces of the clay minerals. Mineralogically, the soil was composed of kaolin, amorphous minerals, hydrated iron and aluminum oxides, free silica, quartz grains, and magnetite. The results of DTA data suggested the presence of amorphous colloids in the soil. This suggestion was subsequently confirmed by selective dissolution analysis which revealed the unexpected presence of 17% amorphous silica in the coarse clay size fraction. Grain size analysis and scanning electron microscopy studies showed that the clay minerals are probably agglomerated by the sesquioxides into silt size clusters. CEC values obtained were primarily attributed to the amorphous colloids rather than the crystalline clay minerals because the sesquioxides probably partially blocked the exchange sites of the clays.

Removal of the iron and aluminum oxides by sodium dithionite citrate-bicarbonate procedures (Mehra and Jackson, 1960), (a) sharpened and exposed previously "masked" X-ray diffraction peaks, (b) disaggregated the clay clusters producing greater amounts of clay size particles, and (c) altered the Cation Exchange Capacity (CEC) and water retention characteristics of the soil constituents.

This investigation demonstrated that amorphous silica and iron and aluminum oxides greatly influence the properties of this latosol by coating and aggregating the clay minerals. These sesquioxide coatings suppress the ordinary behavioral characteristics of the indigenous clay minerals and consequently the observed behavior of the soil is dominated by the amorphous constituents.

INTRODUCTION

"LATERITE" and "latosols" are terms used to describe the reddish-colored relic (soils which have weathered in place) soils occurring throughout the humid tropical regions of the world, although many of these soils are not lateritic. The salient characteristics that identify these soils are the presence of an abundance of the sesquioxides, plus the general absence of silica and the alkaline earths. Mineralogically, the clays are most commonly 1:1 minerals, of which kaolinite is by far the most common. Generally, the soil possesses a granular

structure due to the agglomerating effects of these sesquioxides.

General agreement exists in the literature that the sesquioxides, which consist of hydrated iron oxides are adsorbed on the surfaces of the clay minerals through the interaction of the positively charged colloidal iron and negatively charged clay minerals. However, this adsorption phenomenon is governed by the amphoteric nature of the sesquioxides and the pH of the surrounding medium (Mattson, 1927). Greenland *et al.* (1964, 1968), observed with electron microscopy that iron precipitates as surface coatings on kaolinite only at pH values of 5.0 or less. Their studies revealed that in some tropical soils the iron oxides existed as discrete particles having little association with the clay minerals. Follett (1965) indicated that colloidal iron hydroxide is adsorbed only on one basal surface of kaolinite flakes. Apparently, only the tetrahedral surface possesses sufficient charge to fix the iron particles.

Petrographic studies by Alexander and Cady

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(1962) and Sivarajasingham *et al.* (1962) have shown that the sesquioxide coatings impart a granular structure to the soil by aggregating the clay minerals into spherical clusters. The role of the sesquioxides in aggregation is generally ascribed to one or all of the following: (a) cementation due to precipitation of hydrated iron or aluminum gels 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 sesquioxides (Tutz, 1937; McIntyre, 1956).

It seems that the amorphous sesquioxides of iron and aluminum may be quite influential on the behavioral characteristics of latosols through the neutralization and agglomeration of the clay minerals. The main objective of this investigation was to ascertain the effects of the sesquioxides (crystalline) on some physico-chemical properties of a latosol. These effects were evaluated by establishing quantitative differences between natural and "sesquioxide free" soil.

MATERIALS AND METHODS

Materials

The latosol used in this investigation was obtained from a borrow pit located in Curundu, Panama Canal Zone. The soil was probably the result of normal weathering processes from a conglomerate consisting of basaltic and andesitic pebbles. Numerous small angular pebbles of quartz and chalcedony were encountered in the upper horizon where the sample was taken. The $<0.2\mu$ and $2-0.2\mu$ fractions were obtained by centrifugation with the Sharples supercentrifuge (Jackson, 1956) and the initial fractionation was obtained by gravity sedimentation. Chemical treatments intended to remove the organic material and carbonates were accomplished prior to fractionation (Jackson, 1956). Sesquioxide-free fractions were obtained by sodium dithionite-citrate-bicarbonate extraction (Mehra and Jackson, 1960).

Methods

The mineralogical identifications were based upon X-ray diffraction, DTA, scanning electron microscopy (SEM) analysis and chemical analyses. The X-ray diffraction analyses were made on Ca and K saturated oriented samples of clay with a General Electric XRD-6 apparatus with an SPG-2 spectrogoniometer using Ni filtered Cu radiation. DTA thermograms were made using samples passing the U.S. Standard No. 80 sieve. The DTA samples were exposed to a standard humidity for 4 days over a saturated solution of $Mg(NO_3)_2 \cdot 6H_2O$ in accordance with the procedure suggested by MacKenzie (1957). A Fisher Differential Therm-

alyzer-260 was used to heat the sample to $1200^\circ C$ at a rate of $10^\circ C/min$. The scanning electron micrographs were taken of gold-palladium coated specimens with a JEOLCO JSM-2 scanning electron microscope.

The percentages of free amorphous alumina and silica were determined by the selective dissolution procedure (Jackson, 1956; Hashimoto and Jackson, 1960). The percentages of kaolinite were determined following treatment for removal of amorphous silica and aluminum by the same procedure after dehydroxylation of the kaolin by heating to $560^\circ C$ for four hours. Silica was determined as the silico-molybdate complex, molybdenum blue procedure and aluminum by the aluminon method (Black, 1965).

Cation exchange capacity data (CEC) were obtained by calcium saturation and sodium replacement and exchange calcium was determined by the EDTA-versenate titration procedure (Jackson, 1958). After the CEC values had been determined for the "natural" soil, the sesquioxides of iron and alumina were removed (Mehra and Jackson, 1960) and the CEC of the "sesquioxide free" soil was determined.

Grain size distribution curves were obtained utilizing a hydrometer method of analysis as suggested by Lambe (1951). The objective of this analysis was to determine the particle size distribution curve of the untreated soil and of the sesquioxide-free soil.

RESULTS AND DISCUSSION

X-ray diffraction

The X-ray diffraction patterns for the coarse clay fraction (Fig. 1) reveal that the primary constituent of the clay fraction is a kaolin. The term "kaolinite intermediate" has been used to identify a mixed layered mineral in Panamanian soils Brown and Wolfschoon (1960) having basal spacings of 7 \AA – 10 \AA , but the material is probably kaolin or mixed metahalloysite kaolinite. The presence of sesquioxide or silica coatings on the natural soil particles is strongly suggested by the occurrence of an X-ray diffraction quartz peak (3.35 \AA) after sesquioxide extraction. This diffraction peak was not present in untreated coarse clay fractions. Concurrently, the increased sharpness of the kaolin peaks (7.31 \AA and 3.47 \AA) upon removal of the sesquioxide suggests the existence of coatings on the soil clay particles.

Other diffraction patterns obtained in this study revealed that the soil has been very strongly weathered to poorly crystalline kaolin with some resistant quartz grains in addition to the poorly crystalline kaolin remaining as the identifiable minerals. Some secondary quartz as cristobalite is

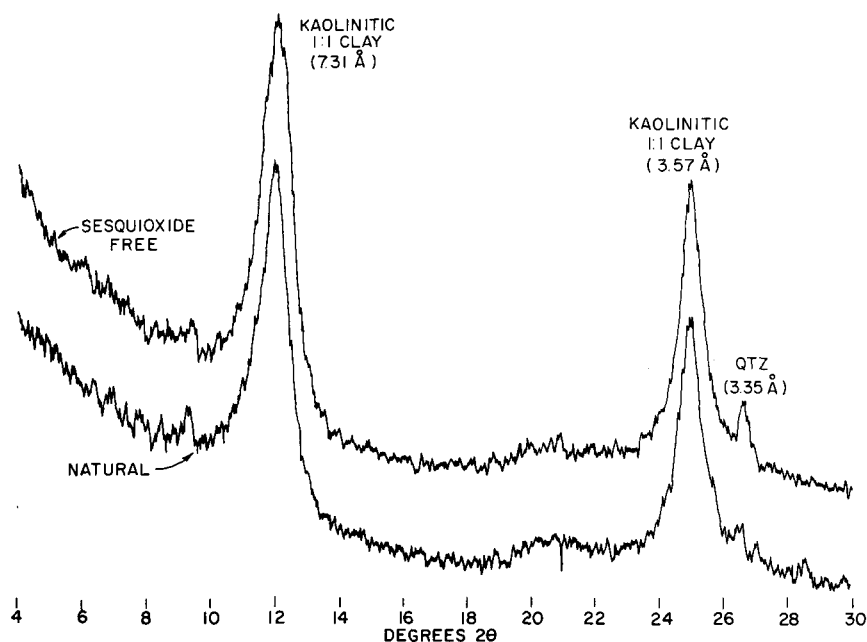


Fig. 1. X-ray diffractograms of the untreated and sesquioxide free K-saturated coarse clay fraction of a Panamanian latosol (Curundu, Panama Canal Zone).

suspected to be present. The absence of a broad (020) kaolin peak (4.45 \AA) in the clay fraction which was observed in the silt fraction suggests amorphous materials in the clay fraction (Jackson, 1956). The sesquioxide coatings on soil particles must exist in microcrystalline or amorphous forms as no X-ray diffraction peaks were observed for goethite, hematite, or gibbsite.

Differential thermal analysis (DTA)

The DTA thermograms of the natural and sesquioxide free samples (Fig. 2) indicate the presence of an amorphous material, which is not detected by X-ray diffraction. The large endothermic peak at 100°C in the untreated soil is an important feature of thermograms for halloysite and allophane, as kaolinite only exhibits a relatively small peak (Jackson, 1956; MacKenzie, 1957). The marked decrease in intensity of this low temperature endotherm in the sesquioxide-free sample indicates the presence of an allophanic material in the original sample. Had this endotherm been attributed solely to halloysite, it would still exist at an appreciable magnitude after sesquioxide extraction. However, the chemical treatment could have dehydrated the remaining soil minerals.

Allophane, an amorphous hydrous aluminosilicate (van Olphen, 1971) occurs in highly

weathered latosols and is believed to be a weathering relic (Jackson, 1956) or precursor (Alexander and Cady, 1962) (Henricks and Whittig, 1968) of kaolinite and halloysite. The presence of allophane in this soil as determined by DTA, is consistent with other studies of Panamanian latosols in which allophane has been reported (Brown and Wolfschoon, 1960; Martini, 1970).

Free silica and alumina composition

The percentages of free silica and alumina as determined by selective dissolution of the several soil fractions are presented in Table 1. These percentages confirm the results of the mineralogical analyses, in that considerable quantities of amorphous material, are present in this latosolic soil. Generally, latosolic soil is considered to be low in free silica and relatively inactive chemically. However, the presence of amorphous materials undoubtedly contributes greatly to the chemical behavior of this soil. High pH-dependent-cation-exchange-capacities, high water retention capabilities and large specific surfaces are characteristic of amorphous colloids.

Kaolinite composition

The approximate percentages of kaolin present in the various fractions of the soil are listed in Table 2. Approximate percentages are listed,

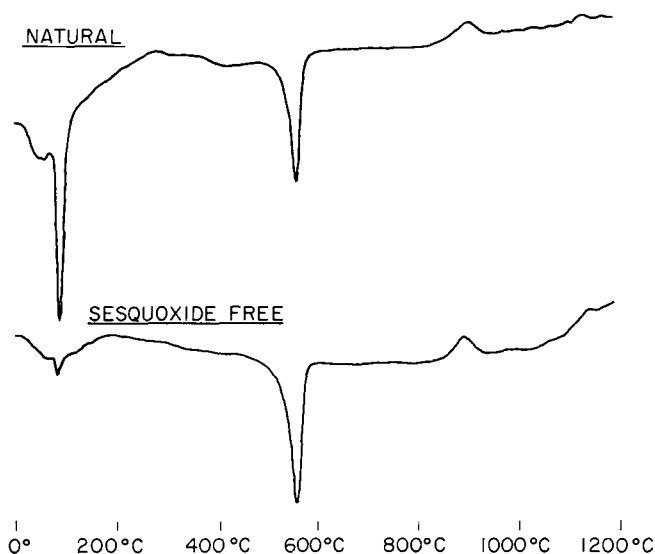


Fig. 2. DTA thermogram of the untreated and sesquioxide free combined silt and clay fractions of a Panamanian latosol (Curundu, Panama Canal Zone).

Table 1. Free amorphous SiO_2 and Al_2O_3 percentages of the silt and clay fraction of a Panamanian latosol (Curundu, Panama Canal Zone)

Soil fraction	SiO_2 (%)	Al_2O_3 (%)
Silt (50–2 μ)	8.6	3.5
Coarse clay (2–0.2 μ)	17.0	6.3
Fine clay (< 0.2 μ)	12.6	4.0

Table 2. Kaolin content of the silt and clay fractions of a Panamanian latosol (Curundu, Panama Canal Zone) based on the silica and alumina composition (Dixon and Jackson, 1960)

Soil fraction	Based upon silica (SiO_2) composition (%)	Based upon alumina (Al_2O_3) composition (%)	av. (%)
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	43.6

because the mineralogical analyses revealed that the kaolin is poorly crystalline and the formula weight of poorly crystallized kaolin may not be equal to that of well crystallized kaolin.

The high percentage of kaolin, 40%, in the silt fraction suggests that cementation by amorphous silica and sesquioxides agglomerates the clay minerals into coarse particles. As expected, the percentage of kaolin increases as the soil fractions become finer. The close agreement between the kaolin percentages of the coarse clay fraction as determined from the silica and alumina composition and the wide variation in the kaolin content as estimated from the silica and alumina composition indicates that the kaolin in the fine clay fraction is poorly crystalline and, therefore, inconsistent with the assumed combination ratios. The close agreement between the reported percentages of kaolin in the coarse clay fraction (based on the silica and alumina content) indicates a more well crystallized material. A comparison of the X-ray patterns of the coarse and fine clay fractions showed somewhat greater peak intensities for the coarse clay fraction which also indicates a greater degree of crystallinity for the coarse clay.

Higher percentages of silica than alumina were obtained in the dissolution analyses. These higher percentages suggest a condition of acid weathering whereby the alumina is removed and the silica remains (*Note.* pH of untreated soil is 5.1). Such conditions are consistent with finding cristobalite and chalcedony pebbles in the soil.

Grain size distribution

A comparison between the grain size distribution curves in Fig. 3 for untreated and sesquioxide-free soil shows an increase in finer sizes after removal of the sesquioxides. This increase suggests that the soil in its natural state consists of clay-sized particles bonded into microdomains by sesquioxide and siliceous cementing agents. Similar results have been reported by Winterkorn (1951) and Pearring (1969) who showed increases in clay-sized fractions and corresponding decreases in sand and silt size fraction after removal of free iron oxides from latosolic soils.

Scanning electron microscopy

Scanning electron micrographs of untreated and sesquioxide-free soil are shown in Fig. 4a and b respectively. The granular flocculant clay microdomains of the "natural" soil are quite apparent in Fig. 4a. Contrasting with this aggregated structure is the platy structure of the sesquioxide-free soil. Jointly these two photomicrographs show the agglomerating effect of the sesquioxides.

Cation exchange capacity

The cation exchange capacities of the various soil fractions are presented in Table 3. Considering that this soil is of a latosolic nature the exchange values are somewhat higher than expected. Ferralitic soils contain kaolin clays which generally

Table 3. Cation exchange capacities (CEC) of the silt and clay fraction of a Panamanian latosol (Curundu, Panama Canal Zone) before and after removal of sesquioxides

Soil fraction	Untreated soil (meq/100 g)	"Sesquioxide free soil" (meq/100 g)
Silt (50-2 μ)	11.2	13.1
Coarse clay (2-0.2 μ)	16.4	19.1
Fine clay (< 0.2 μ)	25.1	21.0

exhibit CEC values from 5.0 to 10 me/100 g (Cardosa, 1964).

Although the soil in this study was not identified as a ferralitic soil the report by Cardosa (1964) indicates that kaolin in tropical soils has a low CEC. The higher values of 11.2 me/100 g to 25.0 me/100 g, therefore, must be due in part to the amorphous constituents. Brown *et al.* (1960) obtained similar values for a Panamanian latosol containing amorphous colloids.

The CEC values increase as the clay fractions become finer, the high CEC values measured in the silt fraction, which ordinarily should exhibit negligible exchange capacities, suggests that the amorphous silica and sesquioxides are responsible for the major portions of the exchange capacity. Although the silt fraction is known to contain

MECHANICAL ANALYSIS CHART

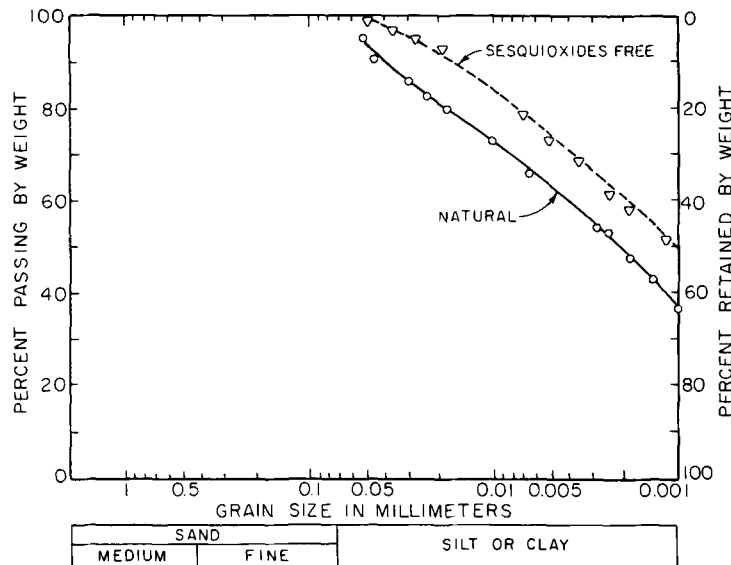


Fig. 3. Effect of sesquioxides on the particle size distribution of Panamanian latosol (Curundu, Panama Canal Zone).

microdomains of clay in silt sized particles, the sesquioxide cementing agents probably neutralize a substantial portion of the charge on these clays.

Removal of the sesquioxides from the soil would be expected to, (a) increase the overall number of individual clay particles due to a breakdown of the aggregated clay clusters through loss of natural cementing agents, and (b) remove ionized iron and aluminum oxide from blocking positions on the surfaces of the clay particles. Both of these actions should contribute to an increase in the CEC of the soil. However, the extraction procedure also alters the properties of the amorphous and crystalline materials and their contribution to the CEC values. Consequently, the extraction of the free iron and aluminum oxides can either enhance or diminish the CEC depending upon the clay minerals involved, the amount of cation exchange blockage, cementing by the sesquioxides (Davidtz and Sumner, 1965) and the percentage of amorphous colloids present.

An examination of the CEC of the silt fraction reveals that no significant difference ($\alpha = 0.5$) in CEC is due to sesquioxide extraction. Apparently the presence of 40% (Table 2) of "cleaned" kaolin in the silt fraction through the removal of blocking and cementing agents is insufficient to counteract the alteration of amorphous materials and cause an increase in CEC. Sesquioxide extraction of the fine clay fraction probably contributes little to disaggregation of the cemented clay clusters. The increase in CEC of the coarse clay fraction could be due to the removal of iron and aluminum oxides from blocking positions on the surfaces of the clay minerals. However, this contribution is not sufficient to offset the alteration of the amorphous constituents, which results in a significant ($\alpha = 0.05$) decrease in the CEC in the fine clay fraction upon sesquioxide extraction. This is interpreted to be due to alteration of amorphous constituents in an attempt to obtain "cleaned" clay particles.

SUMMARY AND CONCLUSION

The results of this investigation have revealed that the amorphous silica and sesquioxides of iron and aluminum greatly influence the properties of this latosol. These influences are summarized as follows:

(1) X-ray diffraction analyses suggested the presence of sesquioxide coatings on the clay mineral surfaces. Removal of the free iron and aluminum oxides increased the diffraction peak intensities and exposed previously "masked" diffraction peaks.

(2) The sesquioxides function as cementing agents and aggregate the clay minerals into silt sized particles. Extraction of these sesquioxides

increased the amounts of clay size particles and decreased the silt and sand sized particles.

(3) Contrary to expectations, considerable quantities of free amorphous silica, possibly as a component of allophane, were present in the soil. Extraction of the free iron and aluminum by sodium dithionite-citrate-bicarbonate procedures (Mehra and Jackson, 1960) altered the water retention capabilities and CEC of the remaining amorphous constituents.

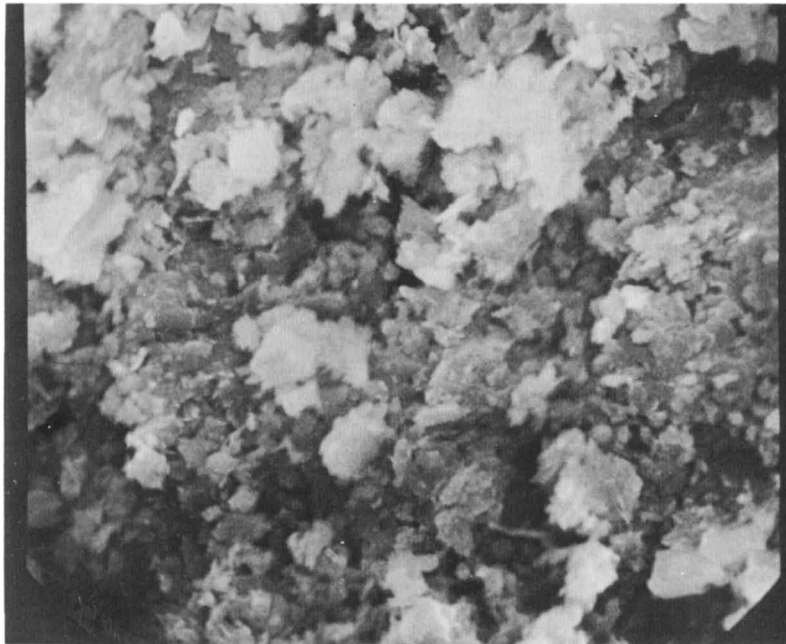
(4) The CEC of the soil fractions was dominated by the amorphous constituents since the sesquioxides probably occupy the exchange sites of the clay minerals.

(5) The textural and structural characteristics of the soil can be greatly influenced by the amorphous constituents.

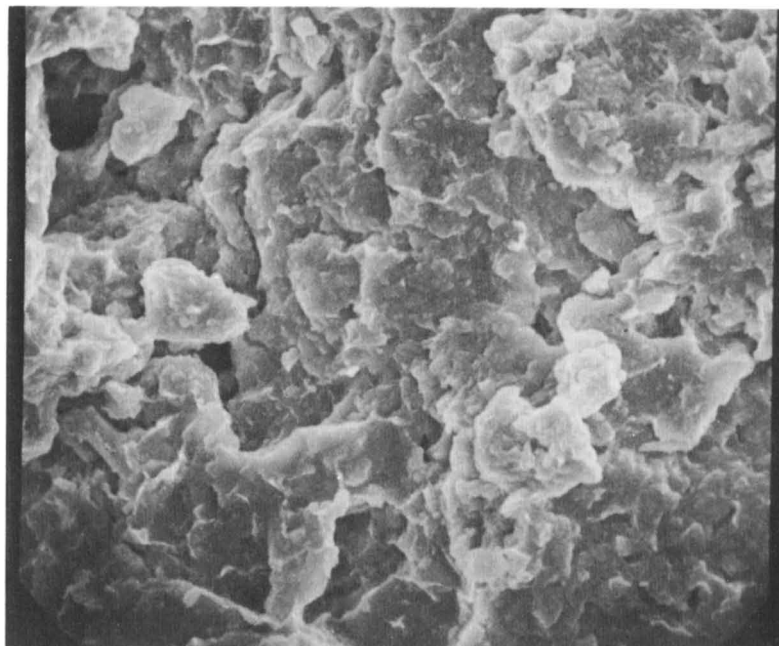
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10 μ



10 μ

Fig. 4. Scanning electron micrographs (SEM) of the effect of sesquioxides on the arrangement of particles in the whole soil of a Panamanian latosol (Curundu, Panama Canal Zone). (4a) Upper portion untreated soil; (4b) Lower portion sesquioxide free soil.

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Résumé—Ce travail est consacré à l'étude des effets que les constituants sesquioxydes exercent sur certaines propriétés minéralogiques et physicochimiques d'un latosol de Panama.

Les latosols sont des sols caractérisés par de fortes concentrations en oxydes de fer et d'aluminium, et une absence générale de silice libre et d'alcalino-terreux.

La diffraction X a montré que des revêtements de sesquioxydes existent sur la surface des minéraux argileux. Au point de vue minéralogique le sol était composé de kaolin, de minéraux amorphes, d'oxydes de fer et d'aluminium hydratés, de silice libre, de grains de quartz et de magnétite. Les résultats de l'ATD suggèrent la présence de colloïdes amorphes dans le sol. Cette suggestion a été confirmée par la suite lors d'une analyse par dissolution sélective qui a révélé la présence inattendue de 17% de silice amorphe dans la fraction granulométrique argile grossière. L'analyse granulométrique et la microscopie électronique à balayage ont montré que les minéraux argileux sont probablement agglomérés par les sesquioxydes en petits grains de la taille du limon. Les valeurs obtenues pour la C.E.C. ont été attribuées essentiellement aux colloïdes amorphes plutôt qu'aux minéraux cristallisés du fait que les sesquioxydes bloquent probablement en partie les sites d'échange des argiles.

L'élimination des oxydes de fer et d'aluminium par les méthode dithionite–citrate–bicarbonate (Mehra et Jackson, 1960) a eu les conséquences suivantes: (a) affinement et révélation de pics de diffraction X qui étaient au préalable "masqués", (b) désagrégation des agglomérats d'argile ce qui produit une quantité plus élevée de particules de la dimension de l'argile, (c) modification de la capacité d'échange de cations (C.E.C.) et des caractéristiques de la rétention d'eau pour les constituants du sol.

Ce travail démontre que la silice amorphe et les oxydes de fer et d'aluminium influencent notablement les propriétés de ce latosol en recouvrant et agrégeant les minéraux argileux. Ces recouvrements de sesquioxydes font disparaître les caractéristiques du comportement ordinaire des minéraux argileux en place, et en conséquence, on observe pour le sol un comportement dominé par les constituants amorphes.

Kurzreferat—Es wurde eine Untersuchung der Wirkungen von Sesquioxid—bestandteilen auf einige mineralogische und physikalisch—chemische Eigenschaften eines panamanischen Latosols durchgeführt.

Latosole sind Böden, die durch eine hohe Konzentration von Eisen- und Aluminiumoxyden sowie eine allgemeine Abwesenheit freier Kieselsäure und Erdalkalien gekennzeichnet sind.

Röntgenbeugungsstudien ergaben, dass sich Sesquioxidbeläge auf den Oberflächen der Tonminerale befanden. Mineralogisch bestand der Boden aus Kaolin, amorphen Mineralen, hydratisierten Eisen- und Aluminkumoxyden, freier Kieselsäure, Quarzkörnern und Magnetit. Die Ergebnisse der DTA Werte deuteten die Gegenwart amorpher Kolloide im Boden an. Dies wurde in der Folge bestätigt durch selektive Lösungsanalyse, die die unerwartete Anwesenheit von 17% amorpher Silica in der groben Tongröße fraktion aufzeigte. Korngrößenanalyse und Untersuchungen mit dem Abtastelektronenmikroskop zeigten, dass die Tonminerale wahrscheinlich durch die Sesquioxide zu Nestern von Schlickgröße agglomeriert sind. Die erhaltenen CEC-Werte wurden in erster Linie amorphes Kolloiden eher als den kristallinen Tonmineralen zugeschrieben weil die Sesquioxide wahrscheinlich die Austauschstellen der Tone teilweise blockieren.

Die Entfernung von Eisen- und Aluminiumoxyden durch Natrium-dithionit-Citrat-Bikarbonat Verfahren (Mehra und Jackson, 1960) hatte zur Folge (a) die Schärfung und Aufdeckung vorher

“maskierter” Röntgenbeugungsspitzen; (b) Zerlegung der Tonester unter Bildung grösserer Mengen von Teilchen von Tongrösse, und (c) Veränderung der Kationenaustauschleistung und des Wasserfesthaltens der Bodenbestandteile.

Die Untersuchung ergab, dass amorphe Kieselsäure sowie Eisen- und Aluminiumoxyde die Eigenschaften dieses Latisols durch Beschichtung und Agglomerisierung der Tonminerale stark beeinflussen. Diese Sesquioxymbeschichtungen unterdrücken die normalen Kennzeichen der einheimischen Tonminerale und aus diesem Grund wird das beobachtete Verhalten des Bodens durch die amorphen Bestandteile dominiert.

Резюме — Изучено влияние отдельных полуторных окислов на некоторые минералогические физико-химические свойства латозолей Панамы. Латозоли — почвы с высоким содержанием окислов железа и алюминия, обычно не содержащие свободного кремнезема и щелочных земель. Рентгеновское изучение показало, что на поверхности глинистых частиц имеются пленки полуторных окислов. Изученные почвы состоят из каолина, аморфных минералов, гидратированных окислов железа и алюминия, свободного кремнезема, зерен кварца, магнетита. Данные ДТА указывают на присутствие в почвах аморфных коллоидов. Это подтверждается применением методики селективного растворения; неожиданным оказалось наличие в грубой глинистой фракции 17% аморфного SiO_2 . Механический анализ и применение сканирующего электронного микроскопа показали, что глинистые минералы, вероятно, сцементированы полуторными окислами в агрегаты, размер которых характерен для частиц силта. Определенные емкости обмена катионов скорее следует приписать аморфным коллоидам, чем кристаллическим глинистым минералам, так как полуторные окислы, вероятно, частично, блокировали обменные позиции глинистых минералов.

Удаление окислов железа и алюминия с помощью натриевого дитионит-цитрат-бикарбонатного раствора (методика Мира и Джексона; 1960) привело к: (а) выявлению ставших более четкими (ранее «замаскированных») дифракционных пиков; (б) дезагрегации скоплений глинистых частиц с повышением количества частиц глинистой фракции; (в) изменению емкости катионного обмена и способности компонентов почвы удерживать влагу. Доказано большое влияние аморфного кремнезема и окислов железа и алюминия на свойства изученных латозолей вследствие образования пленок и агрегирования частиц глинистых минералов. Пленки полуторных окислов подавляют влияние на особенности почвы глинистых минералов и, как следствие этого, свойства почвы определяются ее аморфными составными частями.