WEATHERING RELATIONSHIPS BETWEEN GIBBSITE, KAOLINITE, CHLORITE, AND EXPANSIBLE LAYER SILICATES IN SELECTED SOILS FROM THE LOWER MISSISSIPPI COASTAL PLAIN*

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ABSTRACT

Clays from two Reddish-Brown Lateritic soils from the southern Mississippi Coastal Plain were fractionated and the mineral associations and weathering transformations were examined. The clays were found to be dominated by well-chloritized expansible layer silicates, gibbsite and kaolinite, with smaller amounts of quartz, mica and anatase. The abundance of gibbsite appeared to be related to the presence of expansible layer silicates and to soil pH variations. This mineral and kaolinite were found in appreciable amounts in even the finest clay fractions. Quartz and mica decreased to insignificant amounts with soil depth and decreasing particle size whereas gibbsite content of the clays rose suggesting an interrrelationship between these minerals.

INTRODUCTION

DOMINANT mineral assemblages in soil clays in Mississippi generally conform to major geologic stratigraphic boundaries. Often, however, the soil evironment favors intense weathering with marked alteration of original minerals and formation of new mineral associations. Weathering alterations are most evident in areas of high temperature and rainfall and where movement of water through the soil is maximum. These conditions exist in the area around Lucedale in south Mississippi where soils were developed from loose Coastal Plain sands and loams on Pliocene–Pleistocene marine terraces. Annual rainfall here averages about 60 in. Where slopes are 1 per cent or less, runoff is practically zero and soil development has advanced to the Reddish-Brown Lateritic stage, whereas on slopes greater

than 2 per cent, enabling some runoff, soil development is less advanced and profiles have distinct A_2 horizons. Associated with advanced soil formation had been advanced mineral weathering to considerable depth. It was the objective of the present study to determine the intensity of mineral weathering and the interrelationship 'between the mineral phases found under these soil-forming conditions. The study included two profiles of the Red Bay soil sampled to a depth of 100 in. or deeper. Red Bay is a well-drained Reddish-Brown Lateritic soil by present classification and a Typic Rhodudult by the 7th Approximation.

MATERIALS AND METHODS

The two Red Bay soils used were sampled in George County, Mississippi, in co-operation with members of the Soil Conservation Service and will be referred to as profiles I and II. Sampling was done in opened pits with six of the major layers of each profile being selected for study. Acidity values were measured on field moist samples, and again after air drying, using the saturated paste method and a Beckman Model G pH meter. For mineralogical investigations 50 and 100-g samples of materials were freed of organic matter and iron oxides (Jackson, 1956) and dispersed with sodium carbonate at pH 10. The sodium carbonate boiling treatment (Jackson, 1956) prior to dispersion was omitted because of presence of high amounts of soluble aluminum. Clays were then fractionated at the 2–0.2, 0.2–0.08, and less than 0.08 micron levels by centrifuging techniques.

Mineral identification and estimation employed X-ray, DTA and chemical methods. X-ray analysis was done on a Norelco diffractometer with copper target using parallel oriented specimens on glass slides. Two specimens, one magnesium exchange saturated and one potassium exchange saturated were used. After exchange saturation and removal of excess salts with methyl alcohol each specimen was mixed with 5 ml of 20 per cent glycerine, in ethyl alcohol, and allowed to solvate for 5 hr. The excess glycerine was then removed by centrifuging and the clays were mixed with water and poured on slides to air dry. The K saturated specimens were X-rayed after heating for 4-hr intervals at 350°C, 525°C and 650°C in addition to the room temperature pattern. Differential thermal analysis specimens were magnesium saturated and air-dried from benzene. The thermograms were run by Dr. C. I. Rich at Virginia Polytechnic Institute.

Various chemical techniques were employed for estimating mineral components of the clays. Elemental analyses were done by rapid semi-microchemical methods (Jackson, 1958) with modifications for aluminum to include mercaptoacetic acid to reduce iron interference. The mica component of clays was calculated from K_2O present on a 10 per cent K_2O basis. Quartz was determined gravimetrically after pyrosulfate fusion (Kiely and Jackson, 1962). Amorphous colloids, gibbsite, and kaolinite were

measured as elements dissolved by selective dissolution in boiling alkali after preheating at 110°C and 525°C, respectively (Hashimoto and Jackson, 1960). All analyses reported are based on 110°C oven-dry basis.

RESULTS AND DISCUSSION

Soil Properties

Properties significant to the mineral composition of the two Red Bay soils are given in Table 1. The soils have reddish-brown surface layers and red or dark red subsoils. On drying the color of profile I lightened one or two Munsell chips while profile II showed no change in color. The change

TABLE 1.—MECHANICAL ANALYSIS AND pH OF TWO PROFILES OF RED BAY SOILS
FROM GEORGE COUNTY, MISSISSIPPI

Horizon and depth, in.	Percentage of size classes			Sat. paste pH	
	Sand 2000–50μ	Silt 50–2μ	Clay <2μ	Field moist soil	Air-dry soil
Red Bay—Profile I					
A$_p$: 0–7	68.9	18.5	8.8	4.93	4.59
B : 15–31	58.9	20.3	17.5	5.36	5.25
C : 92–112	77.9	10.3	10.3	5.04	4.90
Red Bay—Profile II					
A$_p$ 0–7	73.1	16.7	8.2	5.31	4.92
B : 20–36	55.4	17.3	23.3	5.42	5.15
C : 82–99	67.9	9.3	19.7	5.75	5.52

in color of profile I suggests some change in states of oxidation or hydration of free iron mineral. Both profiles had sandy loam surface layers over sandy clay loam subsoils with clay contents below 24 per cent. These textures are very favorable for water infiltration on the less than 1 per cent slopes where the soils were found. Practically all rainfall can be assumed to have entered the soil system causing maximum leaching of soluble weathering products.

Acidity values (Table 1) were significantly different between the two profiles and appear to have important bearing on mineral composition to be discussed later. It was noted that pH values obtained from saturated pastes of field moist samples were higher than values obtained two weeks later after the samples were air dried. Lower pH values after drying have

been found to be common in highly weathered soils in Mississippi. Profile I had pH values between 4.7–5.3 when field moist, which dropped to 4.6–5.2 on air drying. Profile II had much higher values, in the range of 5.3–6.0, which dropped to 4.9–5.6 on air drying. Values measured in 1 N KCl (salt pastes) were one to two units lower than values measured in water, with profile II showing more change in KCl pH than profile I.

X-ray Diffraction Analysis

Diffraction patterns in Figs. 1 through 6 include fractions of the surface and deepest layers of both Red Bay profiles. Minerals present were generally well resolved even in finest fractions. Principal peak intensities in 2–0.2μ
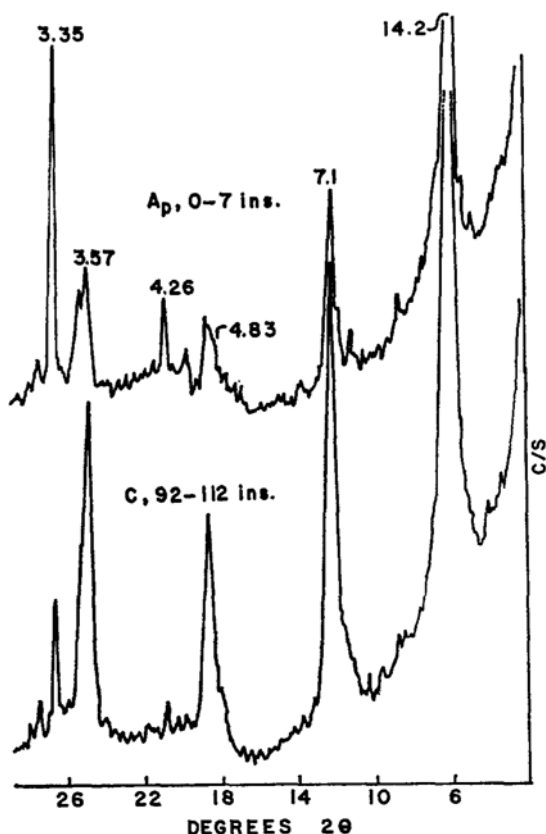

FIGURE 1.—X-ray diffraction patterns of parallel oriented specimens of 2–0.2μ clays from the surface and 92–112 in. layers of Red Bay profile I. Specimens were magnesium-saturated, glycerol solvated, and dried at room temperature. Spacings are given in angstroms.
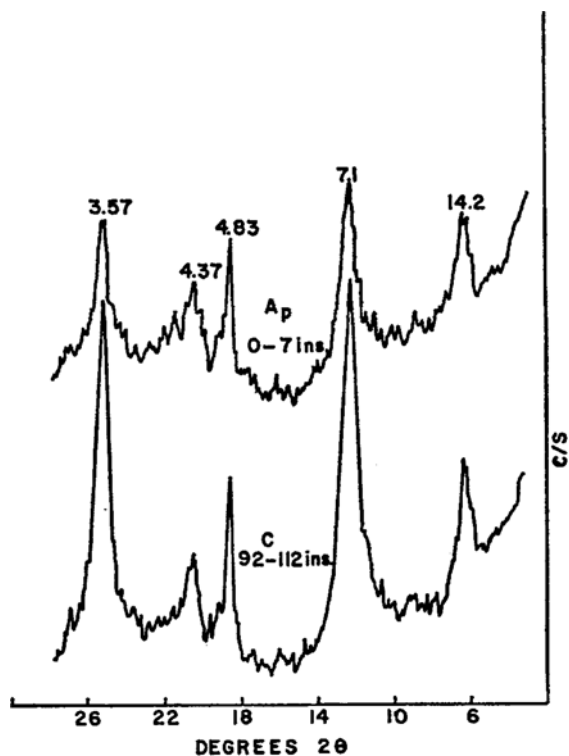
FIGURE 2.—X-ray diffraction patterns of parallel oriented specimens of
0.2–0.08μ clays from the surface and 92–112 in. layers of Red Bay
profile I. Specimens were magnesium-saturated, glycerol solvated, and
dried at room temperature. Spacings are given in angstroms.

clays were recorded at 14.2, 7.1, 4.83, 4.37, 4.26, 3.57, 3.53 and 3.34 Å
with smaller peaks at 10 and 4.97 Å. The 14.2 Å peaks were identified as
aluminous chlorite and chloritized expansible 2 : 1 layer silicates. Potassium
saturation of specimens failed to alter the intensity of this peak observed
in Mg-saturated specimens. Heating of the K-saturated specimens to
550°C resulted in partial breakdown of the 14 Å material with shifts of
intensities to the range of 10 to 12 Å (see Fig. 7). Boiling clays in sodium
citrate for 12 hr (used by Tamura, 1958, to remove Al interlayers) followed
by K saturation, and heating to 550°C resulted in collapse of most of the
14 Å material to 10 Å, although amounts of 14 Å material still remained.
Boiling NaOH (Dixon and Jackson, 1959) after preheating at 550°C per-
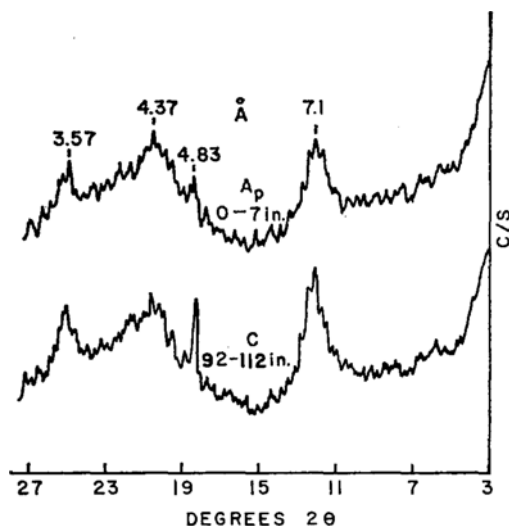mitted complete collapse of the 14 Å material to 10 Å on heating. Removal

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FIGURE. 3.—X-ray diffraction patterns of parallel oriented specimens of less than 0.08μ clays from the surface and 92–112 in. layers of Red Bay profile I. Specimens were magnesium-saturated, glycerol solvated, and dried at room temperature. Spacings are given in angstroms.

of gibbsite by NaOH boiling was done before the removal of interlayer aluminum was attempted. The X-ray patterns showed that this was accomplished by boiling 5 min in 0.5 N NaOH without preheating of samples.

The 7 Å lines, which were primarily kaolinite with some contribution from chlorite, were stable at 350°C but disappeared from the patterns when specimens were heated at 550°C. The 7 Å peaks showed considerable increase in intensity with increasing soil depth. Prominent peaks at 4.83 and 4.37 Å were identified as gibbsite and were removed from patterns when specimens were heated at 350°C or boiled in 0.5 N NaOH for 5 min without preheating. Intensities of the gibbsite lines were highest in the deepest soil layers indicating that considerably more gibbsite was present at depths. Profile II apparently contained much more gibbsite than profile I.

Quartz was identified from the 4.26 Å line which was observed only in the 2–0.2μ fractions. Amounts of quartz, based on X-ray intensity, and later confirmed by pyrosulfate fusion and weighing, were highest in the surface layer of soil and progressively decreased in deeper layers. The finer clays appeared to contain principally gibbsite and kaolinite with moderate amounts of chlorite-like material in 0.2–0.08μ fractions and only trace amounts in the less than 0.08μ clays. Both gibbsite and kaolinite lines were strong in less than 0.08μ clays. Presence of anatase was indicated by a heat
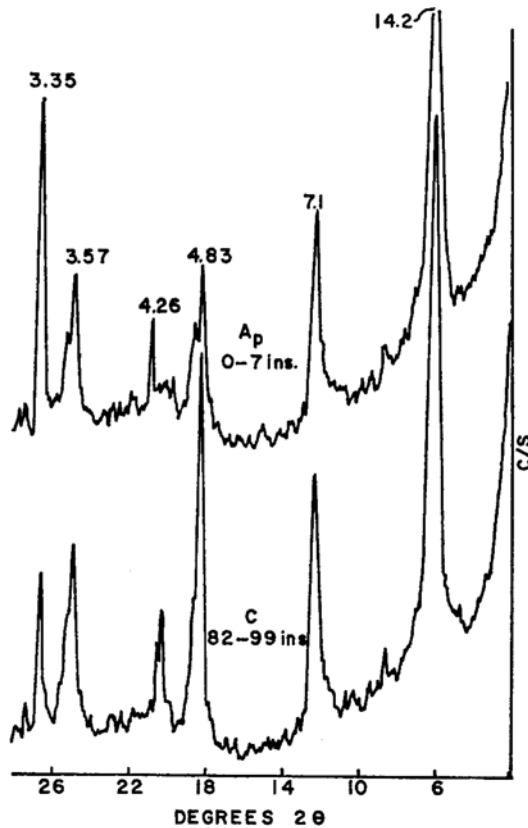
FIGURE 4.—X-ray diffraction patterns of parallel oriented specimens of 2–
0.2μ clays from the surface and 82–99 in. layers of Red Bay profile II.
Specimens were magnesium-saturated, glycerol solvated, and dried at
room temperature. Spacings are given in angstroms.

stable 3.53 Å peak which was observed only in 2–0.2μ clays and was later
confirmed by elemental analysis.

Differential Thermal Analysis

X-ray evidence of gibbsite as a major component of Red Bay clays was
substantiated by intense endothermic reactions around 300°C in the
differential thermograms (Figs. 8–11). The peak temperature of this reaction
varied considerably, however, being close to 280°C in the 2–0.2μ clay of
profile I and 315°C in the same fraction of profile II. This temperature
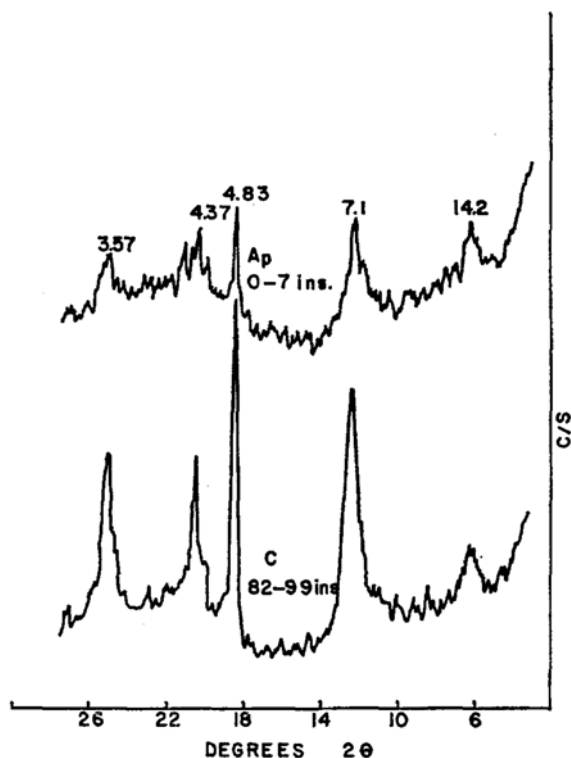variation may be a reflection of state of crystallinity. Possible thermal

FIGURE 5.—X-ray diffraction patterns of parallel oriented specimens of 0.2–0.08μ clays from the surface and 82–99 in. layers of Red Bay profile II. Specimens were magnesium-saturated, glycerol solvated, and dried at room temperature. Spacings are given in angstroms.

reactions of iron compounds in this temperature range were excluded by prior removal of these components. The intensity of the gibbsite endotherm showed a significant increase as a function of increasing soil depth and was more prominent in profile II in both 2–0.2μ and 0.2–0.08μ clays.

Other significant endothermic reactions were observed at 450°C and 505–535°C. The latter reaction was superimposed on the 450°C reaction in the 2–0.2μ clays. Little evidence of the 450°C reaction was found in the 0.2–0.08μ clays of either profile. It was noted that the 505°C endotherm in the 2–0.2μ clay of the surface layer of profile II shifted to 525°C in the deepest layer. This reaction may represent joint contribution of kaolinite and chlorite-like material and is similar to the reaction reported for
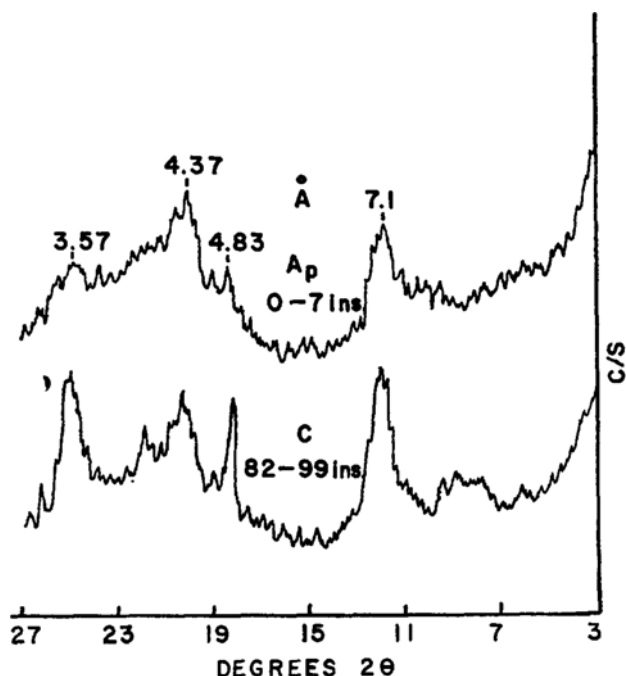
FIGURE 6.—X-ray diffraction patterns of parallel oriented specimens of less than 0.08μ clays from the surface and 82–99 in. layers of Red Bay profile II. Specimens were magnesium-saturated, glycerol solvated, and dried at room temperature. Spacings are given in angstroms.

chloritic Crosby clays (Klages and White, 1957). The rather symmetrical 535°C reaction in the 0.2–0.8μ clays was identified as kaolinite although the peak temperature of the reaction was somewhat lower than is typical of, kaolinite, which may be a result of poor crystallinity.

A positive identification of the 450°C reaction was not made, although it probably relates to OH loss from partial chlorite-like interlayers in expansible 2 : 1 silicate minerals. Similar reactions of this nature have been previously reported (Rich and Obenshain, 1955; Shen and Rich, 1962). Further studies of this reaction are needed, however, for positive identification.

Loss of discrete water layers from freely expansible 2 : 1 layer silicates in the temperature range of 50 to 280°C was not resolved, although considerable loss of water in this range was evident. This suggested that some water, other than external surface water, was evolved but not at any discrete temperature as usually occurs with Mg-saturated 2 : 1 layer clays.
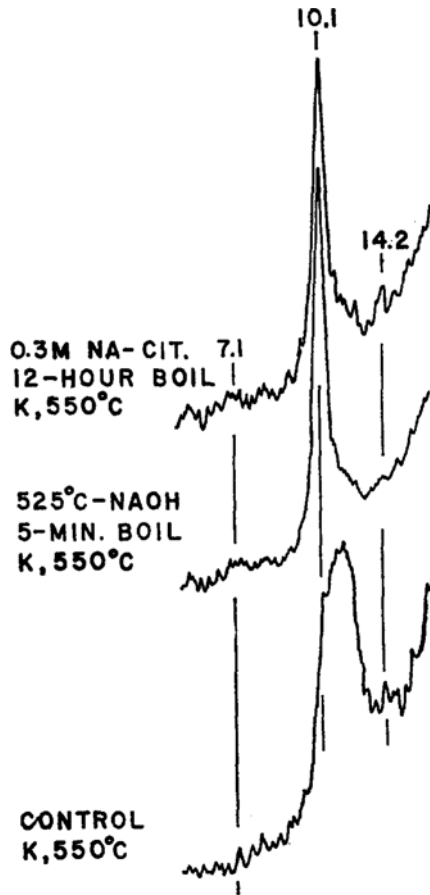
FIGURE 7.—X-ray diffraction patterns showing effects of boiling sodium citrate and boiling sodium hydroxide on aluminum interlayers in 2–0.2μ clays of the 92–112 in. layer of Red Bay profile I. Specimens were boiled 5 min in 0.5 N NaOH prior to treatments shown to remove gibbsite. Spacings are given in angstroms. Note collapse of the 14Å peak.

Elemental Analyses

Total elements in Red Bay clays are given in Tables 2 and 3 and analyses of silica and alumina dissolved from the clays by rapid NaOH boilings for allocation of amorphous material, gibbsite and kaolinite are presented in Tables 4 and 5. The elemental results show that Red Bay subsoil clays are high in aluminum and relatively low in silica, indicating low content

Figure 8.—Differential thermograms of 2–0.2μ clays from the surface and 92–112 in. layers of Red Bay profile I. Specimens were magnesium-saturated and dried from benzene prior to analysis.

FIGURE 9.—Differential thermal analysis of 0.2–0.08μ clays of the surface and 92–112 in. layer of Red Bay profile I. Specimens were magnesium-saturated and dried from benzene prior to analysis.

Figure 10.—Differential thermal analysis of 2–0.2μ clays of the surface and 82–99 in. layers of Red Bay profile II. Specimens were magnesium-saturated and dried from benzene prior to analysis.
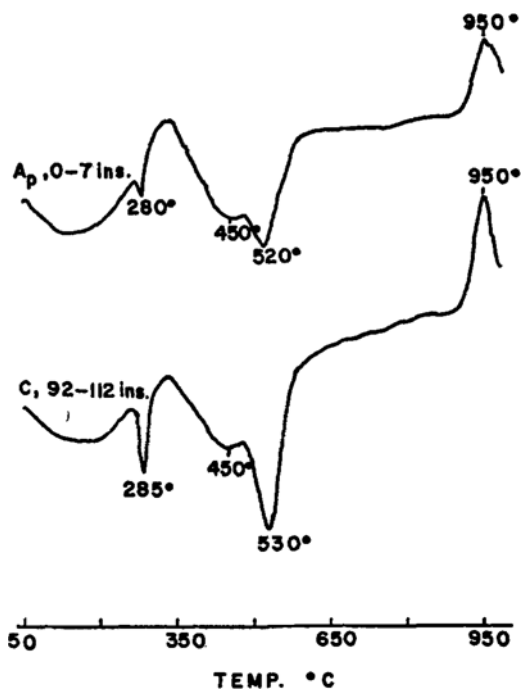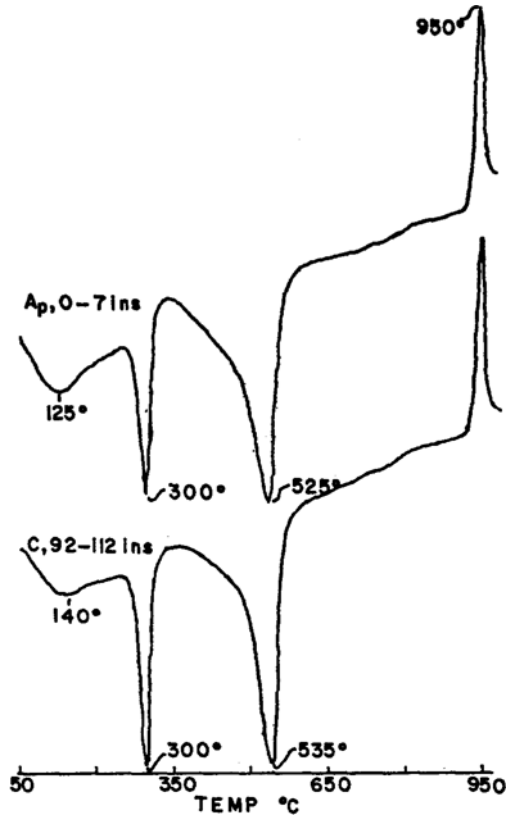
FIGURE 11.—Differential thermal analysis of 0.2–0.08μ clays of the surface and 82–99 in. layers of Red Bay profile II. Specimens were magnesium-saturated and dried from benzene prior to analysis.
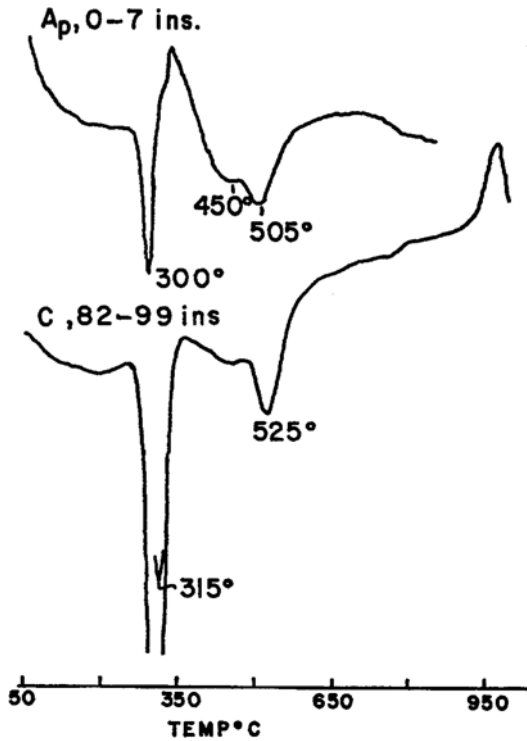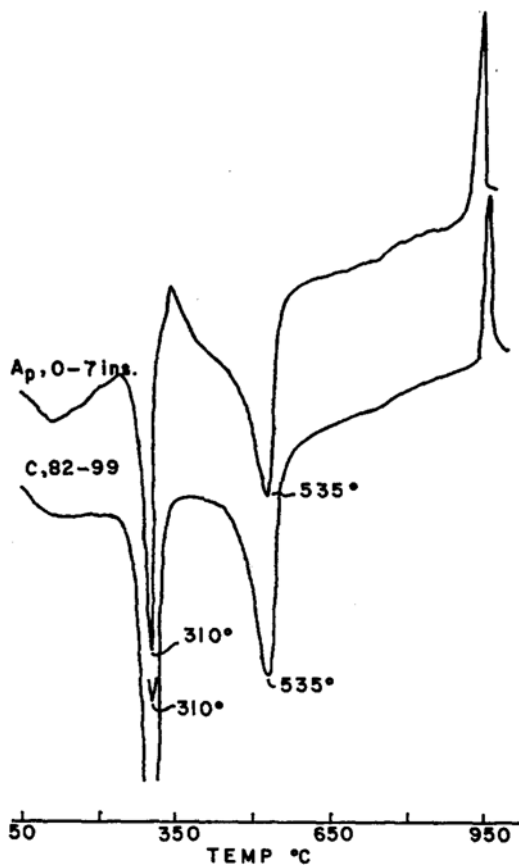
TABLE 2.—ELEMENTAL ANALYSIS OF CLAYS OF RED BAY PROFILE I

Oxide	Percentage of oxides by soil layer		
	A_p 0–7 in.	B 15–31 in.	C 92–112 in.
	2–0.2μ		
SiO_2	53.7	43.2	43.9
Al_2O_3	24.9	33.6	33.0
Fe_2O_3	2.35	1.73	1.64
CaO	0.00	0.00	0.00
MgO	1.31	0.68	0.47
K_2O	2.26	0.97	0.81
Na_2O	0.28	0.35	0.40
TiO_2	5.05	2.30	2.73
H_2O	10.4	15.8	14.9
Total Oxides	100.4	98.6	97.8
	0.2–0.08μ		
SiO_2	39.6	39.9	38.9
Al_2O_3	36.8	39.0	40.1
Fe_2O_3	1.84	1.90	1.39
CaO	0.00	0.00	0.00
MgO	0.71	0.73	0.24
K_2O	0.67	0.62	0.35
Na_2O	0.08	0.13	0.10
TiO_2	0.73	0.87	1.27
H_2O	18.2	18.9	18.1
Total Oxides	98.6	102.1	100.5

of 2 : 1 layer silicates and quartz. Amounts of alumina increased significantly from surface to subsoil layers corresponding to the high intensity gibbsite peaks observed in the X-ray and DTA data. Amounts of gibbsite present in subsoil layers were also reflected in water content of the clays, most of which was lost in the temperature range of 110–350°C. Silica was relatively high in surface layer clays because of quartz, but showed a significant decline in subsoil layers. Amounts of titanium present (3–5 per cent) agreed with the occurrence of anatase (3.53 Å) lines in the X-ray patterns. Total potassium indicates that the clays have low mica contents with significant amounts of sodium, probably proxying for potassium. Feldspar lines in X-ray patterns were too weak to account for the amounts of sodium found. Octahedral ions other than aluminum were generally low in amount, being principally magnesium. Although some iron was present (1–2 per cent), it was presumed to be associated with amorphous material rather than in octahedral positions of crystalline minerals.

Table 3.—Elemental Analysis of Clays of Red Bay Profile II

Oxide	Percentage of oxides by soil layer		
	A_p 0–7 in.	B 20–36 in.	C 82–99 in.
2–0.2μ			
SiO_2	49.2	39.3	29.1
Al_2O_3	28.3	35.8	44.8
Fe_2O_3	2.30	1.99	1.33
CaO	0.00	0.00	0.00
MgO	1.71	0.90	0.34
K_2O	1.39	1.22	0.38
Na_2O	0.36	0.40	0.27
TiO_2	3.44	3.60	3.10
H_2O	13.6	17.6	21.4
Total Oxides	100.3	100.8	100.7
0–2–0.08μ			
SiO_2	46.1	35.3	31.6
Al_2O_3	32.5	40.2	45.3
Fe_2O_3	4.74	1.56	0.88
CaO	0.00	0.00	0.00
MgO	0.71	0.70	0.81
K_2O	1.08	0.61	0.27
Na_2O	0.18	0.14	0.14
TiO_2	0.87	0.97	1.07
H_2O	13.30	19.2	22.1
Total Oxides	99.5	98.7	102.2

Attempts to selectively dissolve gibbsite and kaolinite were only partially successful, primarily because of the fact that the solubility of gibbsite in boiling NaOH decreased markedly following dehydroxylation at 300°C and 525°C. This was most noticeable in the data of the 2–0.2μ clays of profile II (Table 5). Dissolution treatments applied to a separate sample of gibbsite showed that solubility after dehydroxylation at 535°C decreased to one-third of that observed when the material was preheated at 110°C. The loss of gibbsite solubility on heating thus explains the fact that primarily silica was obtained in the alkali dissolutions after the 525°C preheat treatment. Kaolinite therefore was allocated according to differences in silica dissolved after the 110°C and 535°C treatments. Amorphous material was assumed to have the silica:alumina molar ratio of kaolinite, which appears typical of amorphous material in Mississippi soil clays, and all of the silica dissolved by NaOH after preheating at 110°C was allocated to amorphous material with adequate aluminum to give a 2 : 1 molar

TABLE 4.—MATERIALS DISSOLVED FROM CLAYS OF RED BAY PROFILE I BY RAPID NaOH BOILING AFTER CLAYS WERE PREHEATED TO 110° AND 525°C.

Horizon and depth, inches	Oxides dissolved after 110°C		Oxides dissolved after 525°C		Difference in oxides dissolved (525°–110°C)	
	SiO_2 %	Al_2O_3 %	SiO_2 %	Al_2O_3 %	SiO_2 %	Al_2O_3 %
2–0.2μ						
A_p : 0–7	8.5	5.4	20.5	13.8	12.1	8.4
B_2 : 15–31	6.7	6.4	18.3	14.9	11.6	8.5
C : 92–112	6.7	8.6	24.5	20.4	17.7	11.8
0.2–0.08μ						
A_p : 0–7	16.7	19.1	30.8	28.2	14.1	9.1
B_2 : 15–31	16.2	20.2	32.7	26.3	16.5	6.1
C : 92–112	23.6	28.5	34.7	28.5	10.7	0.0

TABLE 5.—MATERIALS DISSOLVED FROM CLAYS OF RED BAY PROFILE II BY RAPID NaOH BOILING AFTER CLAYS WERE PREHEATED TO 110° AND 525°C.

Horizon and depth, inches	Oxides dissolved after 110°C		Oxides dissolved after 525°C		Difference in oxides dissolved (525°–110°C)	
	SiO_2 %	Al_2O_3 %	SiO_2 %	Al_2O_3 %	SiO_2 %	Al_2O_3 %
2–0.2μ						
A_p : 0–7	8.6	10.1	19.8	17.4	11.2	7.3
B_2 : 20–36	6.6	19.7	17.3	19.6	10.7	−0.1
C : 82–99	4.7	25.5	17.3	25.5	12.6	0.0
0.2–0.08μ						
A_p : 0–7	16.8	12.6	29.2	18.7	12.4	6.1
B_2 : 20–36	14.8	24.3	30.1	25.0	15.3	0.7
C : 82–99	12.7	29.9	30.0	24.4	17.3	−5.5

ratio. The alumina remaining was assumed to be gibbsite. Amounts of gibbsite obtained in this manner were in general agreement with X-ray and DTA data.

Weathering Trends Associated with Soil Development

Minerals in clays of the Red Bay soils were found to be primarily aluminous types with kaolinite, gibbsite and chlorite, or expansible 2 : 1 layer silicates in advanced stages of chloritization, dominating the association. Also present were smaller amounts of less resistant quartz and mica and highly resistant hematite and anatase. Mineral weathering and soil development, therefore, appear to be well synchronized. The observed distribution of minerals within each profile, however, varied contrary to the common belief that weathering intensity is greatest at the soil surface and diminishes with depth. Expressed on a percentage of clay bases, gibbsite, generally considered representative of advanced weathering in acid soils, increased in profile II from 5–8 per cent in the surface layer to 30–35 per cent at the 82–99 in. depth. Similarly, kaolinite, also a relatively resistant mineral and indicator of an advanced weathering stage, and composing 20–40 per cent of Red Bay clays, was of greater abundance in the deeper soil layers. Quartz and mica, minerals of only moderate weathering resistance, and observed principally in the $2-0.2\mu$ fractions, diminished progressively from the surface downward. Actual amounts of quartz in $2-0.2\mu$ fractions of profile II ranged from 20 per cent in the surface layer to 4 per cent in the 82–99 in. layer. Mica likewise decreased in amount downward in the profile from 15–18 per cent to about 4 per cent. The same sequence of distribution of minerals was observed in profile I, although gibbsite was less common, while quartz, mica, kaolinite and chlorite, or chloritized expansible silicates, were more evident. Anatase was uniformly distributed throughout profile II, but was higher in the surface layer of profile I.

Mineral abundance in accord with weathering stability as a function of particle size was much in evidence and followed the expected trend, that is, less resistant quartz, mica, and chloritized expansible 2 : 1 layer minerals progressively diminished in importance in fractions finer than $2-0.2\mu$, whereas kaolinite, gibbsite and amorphous material became more prominent. Fractions finer than 0.08μ contained only trace amounts of chlorite-like material. Amorphous colloids comprised as much as 40 per cent of less than 0.2μ fractions in the deeper layers of soil.

The interrelationships of minerals present as a function of progressive weathering were somewhat complex, but generally appear to follow Jackson's proposed weathering sequence (1959) with possible inclusion of aluminous chlorite and chlorite-like materials between montmorillonite and kaolinite as has been suggested (Glenn et al. 1960). The lower degree of weathering stability of aluminous chlorite and chlorite-like minerals is

indicated by the fact that they became less evident as particle size decreased while amounts of kaolinite increased.

The anti-gibbsite effect of expansible 2 : 1 layer silicates (Jackson, 1962a, b), was examined in relation to gibbsite formation in Red Bay and was found to apply. According to this concept, positive charged aluminum polymers at pH values below the isoelectric pH (4.8) of the gibbsite molecule would respond to exchange charges of montmorillonite and vermiculite minerals entering their interlayer positions to form chlorite-like structures and prevent gibbsite nucleation. At pH values above the isoelectric pH the gibbsite molecule would be negative and subject to repulsion from interlayer positions permitting nucleation of a crystalline gibbsite phase. Profile I of Red Bay was found to be at or slightly above (4.8 to 5.3) the isoelectric pH of gibbsite and gibbsite appeared in the clays to the extent of 2–7 per cent depending on soil depth. Profile II had somewhat higher subsoil pH values (5.3 to 5.6) with gibbsite appearing in amounts of 30–35 per cent of the clays. Both profiles had low gibbsite content in the surface where the pH was around 4.9. Gibbsite formation in the surface layers is also subject to inhibition by presence of organic chelates as well as unfavorable pH.

Quartz and mica content of clays appeared to be related to amount of gibbsite present. Presumably these minerals provided much of the silica functioning as the proton sink (Jackson, 1962) in aluminum hydrolysis which was later leached from the soil as silicic acid. Chloritization of expansible layer silicates was likely extensive during periods when soil solution pH was probably down in the vicinity of 4.5. Later rises of pH, accompanying reduced exchange properties of expansible clays by interlayer formation, and also through formation of mineral phases of low exchange charge, favored gibbsite nucleation, along with silica consumption and leaching. With continued weathering and rise in pH, further desilication and destruction of quartz or mica would occur with their eventual disappearance. The desilication process would likely proceed next through chloritized expansible silicates and chlorite, and finally through kaolinite. The soils then would be approaching the Latosol stage of development. The weathering interrelationship of the non-quartz minerals through the entire process thus would appear to be as follows:

Mica ────→ Expanded K-depleted mica ──── pH = 4.5 ────→ chloritized expansible 2 : 1 silicates ────→ Kaolinite pH > 5.0 ────→ gibbsite
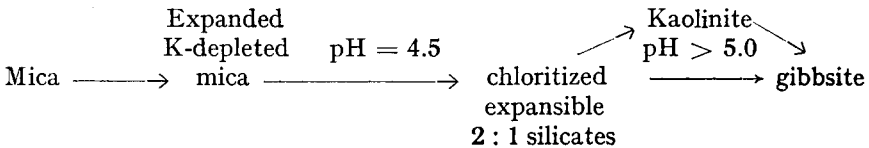
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