CLAY MINERALS IN A LIMESTONE SOIL PROFILE[1]

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ABSTRACT

The clay minerals in a soil developed from Lenoir limestone (Ordovician) in Augusta County, Va., are kaolinite and chlorite. The Lenoir limestone contains clay partings consisting of hydrous mica, and the clay in the soil is considered to be residual from that in the parent limestone and modified by soil-forming processes (podzolic). X-ray diffraction patterns of the clay fraction ($<2\mu$) from samples from different positions in the profile show a reduction in hydrous mica and an increase in kaolinite and chlorite content as the A horizon is reached. Partial chemical analyses of the whole soil and of the clay fractions are given, as well as pH and ion-exchange capacity determinations. The silica-alumina ratios are similar to those characteristic of podzolic soils, and excess silica in the A horizon accumulates as recrystallized quartz together with chert which is considered to be residual from the limestone. Mechanical analyses by the standard pipette method show that the quantity of clay in the soil varies from 17 percent at the surface to 61 percent at 26 inches (C horizon).

INTRODUCTION

The importance of the residual clay minerals in various soils from the eastern and middle-western parts of the United States south of the areas of Pleistocene glacial drift has recently been stressed by Van Houten (1953), who points out that hydrous mica, inherited from the parental rocks, characterizes the clay fraction of a great many soils. Other observers, however, have noted that where soil-forming processes, particularly those of podzolic character, have acted on soil material for a considerable period of time, kaolinite has developed at the expense of original hydrous mica and montmorillonite (Ross and Hendricks, 1945).

This paper presents the results of a mineralogical and chemical examination of a soil profile developed above the Lenoir Limestone of Ordovician age in the Shenandoah Valley of Virginia, and it confirms the findings of Ross and Hendricks (1945) that kaolinite is an end-product of clay-mineral development in limestone soils. The examination of this profile is part of the weathering and erosion studies being undertaken in the Shenandoah Valley by John T. Hack and the senior author.

The weathering of a limestone in a humid climate is dependent on the leaching action of rain and ground water, which removes the calcium and

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[1] Publication authorized by the Director, U. S. Geological Survey.

171

magnesium carbonates leaving an insoluble residue that, in the case of the limestones of the Shenandoah Valley, consists primarily of chert fragments from the abundant chert bands. Miller (1952), on the basis of his investigations in the system $CaCO_3$-CO_2-H_2O, estimated that reduction of a limestone area under humid conditions (40 inches of rain per annum) would proceed at the rate of a foot in 11,100 years; or that 3.1 tons of $CaCO_3$ per square mile per inch of rain could be removed from a limestone area each year.

Pure limestone, containing only an insignificant quantity of insoluble residue, will produce only thin soils; and where an area is subjected to active erosion by a stream system, resulting in well-defined hill slopes, very little soil will remain and the bedrock will be practically at the surface. If, however, the limestone is impure and contains chert, sandy layers, clay bands, or partings, a thick soil may develop because the residuum is relatively insoluble and provides a body of material in which soil-forming processes can act. Chert commonly acts as a skeleton holding up the weathering rock and the developing soil.

The soil profile to be described here is developed above the Lenoir limestone (Ordovician) which varies from pure, commercially valuable, high-calcium limestone in massive beds to thinly bedded cherty limestone. Much of it is characterized by a pinkish-brown clay mineral in irregular partings within the rock (Edmondson, 1945). Two chemical analyses given by Edmondson are:

	A (percent)	B (percent)
SiO_2	0.28	0.32
Al_2O_3	0.61	0.13
Fe_2O_3	0.20	0.46
$CaCO_3$	98.00	98.16
$MgCO_3$	0.83	0.76

A, Eastern environs, Staunton, Va.
B, State Sanitarium, 2 mi. SE of Staunton, Va.

Two samples of Lenoir limestone, one massive, and the other more thinly bedded and impure, collected recently, contain approximately 1 percent and 12.5 percent insoluble residue, respectively. Some idea of the amount of residual material available from the Lenoir limestone can be obtained from these figures; the pure limestone contains 22.4 and the impure limestone 280 lbs. of insoluble residue per long ton — the latter figure not including chert bands.

DESCRIPTION OF SOIL PROFILE

The soil profile on Lenoir limestone described here is exposed in a roadcut on route 742 near its junction with route 612 in Augusta County north of Staunton, Va. In the roadcut the soil is seen to be a podzolic type passing downward below a well-defined yellowish-brown horizon into weathered, impure, bedded limestone with prominent chert bands. The

soil belongs to the gray-brown podzolic group and resembles the silt loams of such soils as the Hagerstown and Frederick silt loams. Unweathered Lenoir limestone from the base of the roadcut (about 24 feet below the surface of the soil) is gray, somewhat cherty, and contains numerous partings covered with a pinkish-brown clay mineral.

The soil forms the top of a small hill which, because of its sandy character and abundant chert float, has been retained as a wood lot on a farm and apparently has never been cultivated. The profile was sampled by John T. Hack and the senior author. Details of the samples collected are as follows:

Sample No.	Depth (inches)	Horizon	Description
18A	0-10	A	Light-colored sandy layer with some clay and angular chert fragments.
18B	10-15	A-B	Yellow, less sandy layer passing downward to
18C	15-21	B	yellowish-brown more clayey layer.
18D	21-23	B-C	Brown to reddish brown with compact, dense clay and less sand.
18E	23-26	C	Reddish-brown, dense, compact clay.
18F	At 13 feet	D	Soft, altered limestone, largely clayey.

METHODS OF EXAMINATION

Mechanical Composition

Mechanical analyses using the standard pipette method of the U. S. Department of Agriculture were made by Paul D. Blackmon for each of the soil samples. The figures obtained are given in Table 1.

TABLE 1. — MECHANICAL COMPOSITION OF SOIL DERIVED
FROM LENOIR LIMESTONE

Sample No.	Horizon	Depth (inches)	pH	Stones above 2 mm	Gravel 2-1 mm	Coarse sand 1-0.5 mm	Medium sand 0.5- 0.25 mm	Fine sand 0.25- 0.1 mm	Very fine sand 0.1- 0.05 mm	Silt 0.05- 0.002 mm	Clay < 0.002 mm
				SIZE DISTRIBUTION (PERCENT BY WEIGHT)							
18A	A	0-10	4.6	12.4	2.0	2.1	1.8	3.0	5.2	56.6	17.1
18B	A-B	10-15	4.3	5.6	1.1	1.6	2.0	3.2	5.9	57.7	23.0
18C	B	15-21	4.4	2.6	0.1	1.2	1.3	2.4	4.5	44.7	42.5
18D	B-C	21-23	4.4	1.9	0.5	0.6	0.6	1.2	2.4	34.8	58.0
18E	C	23-26	4.4	0.1	0.2	0.3	0.4	0.8	2.1	33.6	61.7

Analyst: Paul D. Blackmon

Chemical Composition

Partial chemical analyses were made of the whole soil and of the finer than 2 micron clay fractions separated for x-ray examination. Silica, alumina, and iron were determined by standard methods for silicate analysis, but in addition free ferric oxide in the clay fractions was found

by Deb's (1950) hydrosulfite method. CaO, MgO, and K_2O were determined by the methods of rapid analysis developed by Shapiro and Brannock (1952) and were done in the Rapid Analysis Laboratory under Mr. Brannock's supervision.

Ion-exchange capacities were determined by Bower and Truog's (1940) colorimetric manganese method using neutral N $MnCl_2$. The concentrations of Mn in the resulting solutions were determined with a Coleman no. 14 spectrophotometer.

The chemical composition of the whole soil is given in Table 2, and that of the clay fractions in Table 3. The figures for ion-exchange capacities are added to these tables.

X-ray Examination

Each sample was dispersed in distilled water to which sodium metaphosphate was added as a dispersing agent. The clay fraction (less than 2 microns equivalent settling diameter) was separated by repeated centrifuging and decanting. Portions of the clay suspensions were placed on $1 \times 1\frac{3}{4}$ in. glass slides and allowed to dry at room temperature to give oriented aggregates. The water was removed from the remaining suspen-

TABLE 2. — PARTIAL CHEMICAL ANALYSES OF SOIL PROFILE
ON LENOIR LIMESTONE

Sample No.	Hori- zon	Depth (inches)	SiO_2 (per- cent)	Al_2O_3 (per- cent)	Fe_2O_3 (per- cent)	$H_2O\pm$[1] (per- cent)	$\dfrac{SiO_2}{Al_2O_3+Fe_2O_3}$	Ion- exchange capacity meq/100 g
18A	A	0-10	86.30	3.84	1.00	3.47	32.8	1.9
18B	A-B	10-15	83.89	6.89	1.71	3.56	16.8	4.7
18C	B	15-21	75.28	10.55	3.45	5.41	10.0	13.8
18D	B-C	21-23	64.28	15.38	6.09	8.52	7.3	12.5
18E	C	23-26	63.36	15.91	5.50	8.48	9.0	12.1
18F	D	13 ft	76.83	9.56	4.81	4.00	10.4	7.6

[1] Loss on ignition.
Analyst: D. Carroll.

TABLE 3. — PARTIAL CHEMICAL ANALYSES OF THE CLAY FRACTION (LESS THAN
2 MICRONS) SEPARATED FROM THE PROFILE ON LENOIR LIMESTONE

Sample No.	Hori- zon	Depth (inches)	SiO_2 %	Al_2O_3 %	Fe_2O_3 free %	Fe_2O_3 com- bined %	FeO %	CaO %	MgO %	K_2O %	$H_2O\pm$* %	$\dfrac{SiO_2}{Al_2O_3}$	Ion- exchange capacity meq/100 g
18A	A	0-10	48.79	26.39	3.10	0.38	2.54	0.61	0.61	0.91	13.46	3.14	11.0
18B	A-B	10-15	46.18	24.17	5.87	0.17	1.30	1.04	1.32	1.20	13.86	3.20	15.0
18C	B	15-21	44.58	27.08	7.14	2.28	1.01	0.68	1.34	1.40	12.97	2.80	16.0
18D	B-C	21-23	42.19	28.57	9.60	2.38	0.90	0.56	1.62	1.80	13.35	2.50	18.6
18E	C	23-26	42.70	29.15	7.41	2.00	0.81	0.80	1.55	1.50	13.21	1.98	20.2
18F	D	13 ft	46.24	23.02	7.71	1.83	0.40	0.44	2.33	3.70	9.73	3.41	14.4

* During preparation the clay was dried at about 60°C; the figures for H_2O are loss on ignition of this dried clay.
Analysts: D. Carroll and J. M. Dowd.

Table 4. — Mineralogy of the Clay Fractions (<2 Microns)
of the Soil Profile on Lenoir Limestone

Sample No.	Horizon	Depth (inches)	Kaolinite	Chlorite	Hydrous mica	Montmorillonite	Quartz
18A	A	0-10	+ +	+	–	–	+
18B	A-B	10-15	+ +	+	–	–	+
18C	B	15-21	+ +	+	?+	–	+
18D	B-C	21-23	+ +	?+	+	–	+
18E	C	23-26	+ +	?+	+	–	+
18F	D	13 ft	+ +	–	+ +	–	+
18G		clay parting*	+	–	+ + +	+	?+

* Clay scraped from parting in limestone.
+ + + present in abundance.
+ + present in medium amount.
+ present in small amount.
?+ possibly present.
– absent.

sions by means of porcelain filter candles. The dry clay was ground to pass a U.S. Standard Series sieve no. 400 (less than 38 microns) and was packed into aluminum sample holders. X-ray diffractometer patterns were made with a North American Philips wide-range goniometer diffractometer using Ni-filtered CuKα radiation at 40 kvp and 20 ma. A 1-degree slit system, a rate meter scale factor of 2, and a time constant of 8 seconds were employed. The Geiger-Müller tube was operated at 200 volts over threshold. The scanning speed of the goniometer was 2 degrees 2θ per minute. For each sample four diffractometer patterns were made; these were (1) untreated clay packed into the standard aluminum holder; (2) untreated oriented aggregate on a glass slide; (3) glycerol-treated aggregate on a glass slide; and (4) oriented aggregate on a glass slide heated to 550°C for about 45 minutes.

Some of the pinkish-brown clay mineral on partings in the Lenoir limestone was scraped off, and x-ray patterns were made using both standard mounts and glycerol-treated oriented aggregates.

Figure 1 shows the x-ray patterns obtained from glycerol-treated oriented aggregates of the less than 2 micron fraction of the soils. Peaks occurring near 14 A were interpreted as the 002 spacing of chlorite if they persisted after heat treatment, and those near 10A as the 002 spacing of hydrous mica. A peak near 7 A was interpreted as the 001 spacing of kaolinite if it disappeared upon heat treatment. A line persisting in this region was interpreted as the 004 spacing of chlorite. In the pattern for sample 18G a small peak occurred near 14 A that moved to about 18 A upon glycerol treatment. This was interpreted as the 001 peak of montmorillonite.
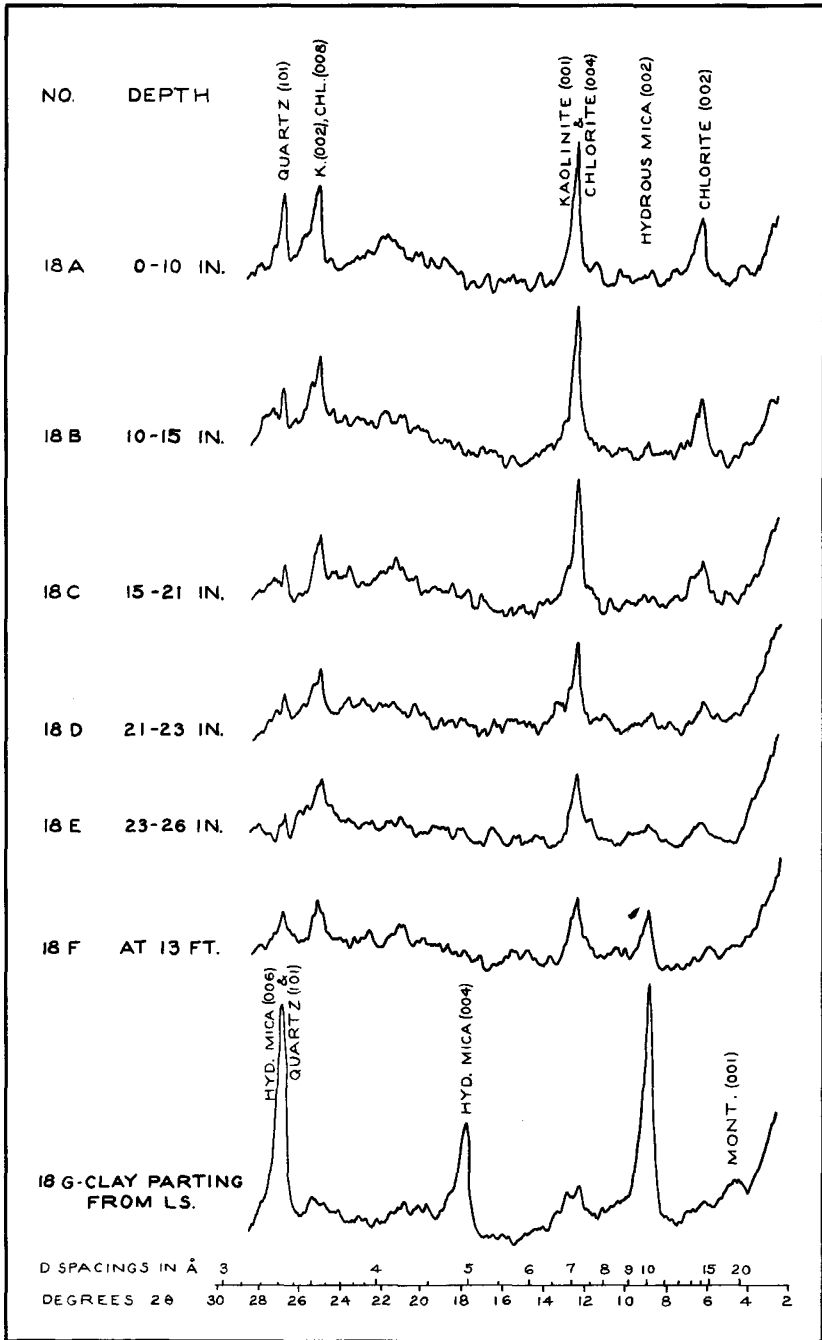
FIGURE 1. — X-ray diffractometer patterns of the clay fraction (< 2 microns) of the various horizons of the soil profile on Lenoir limestone, Shenandoah Valley.

In the diffractometer patterns no peaks for definite iron oxide minerals were present, but a diffuse raised area extending from 8 degrees to 14 degrees 2θ in the patterns produced by the standard unoriented mounts probably indicated the presence of amorphous iron oxide, as this disappeared after the sample was boiled in dilute HCl. The low iron content shown by the chemical analyses serves to explain the absence of peaks for such minerals as goethite and hematite.

The minerals identified from the diffractometer patterns are given in Table 4.

Microscopic Examination

The mineral grains in all grades of sand size were examined with a petrographic microscope. Chert and quartz were found to be the two main constituents with distribution as follows:

Grade (mm)	Description
+2	Chert in angular fragments
1 - 2	Chert
0.5 - 1	Chert
0.25 - 0.5	Irregular chert grains with some euhedral and some rounded quartz grains
0.10 - 0.25	Chert grains and euhedral quartz; some rounded quartz grains a few of which show regrowths
0.05 - 0.10	Euhedral quartz about equal in quantity to irregular chert

A bromoform (sp. gr., 2.8) separation was made of the 0.05 - 0.10 mm grade for each sample, but the quantity of heavy residue obtained was very small. The only heavy minerals present were black opaque grains, presumably ilmenite or magnetite, and a few brownish-black grains, probably limonitic.

Microscopic examination of the pinkish-brown clay mineral in the Lenoir limestone showed that the color is due to innumerable minute grains of iron oxide that impregnate the colorless micaceous plates of the mineral itself. Each flake of the mineral is made up of small overlapping micaceous plates that have an index of refraction of 1.55 and birefringence within the range recorded for the hydrous mica and for the montmorillonite groups.

The insoluble residue from the limestone collected near the soil profile consists of very fine clayey material of dark color (about 95 percent) and larger chert fragments. Loss on ignition was very small so that the limestone, although it is dark gray, contains little carbonaceous material. All the grades coarser than 0.05 mm consist mainly of chert, although a few grains of angular quartz are present.

DISCUSSION

Mechanical Composition

The mechanical analyses given in Table 1 indicate that texturally the soil consists of silt loam, with very little sand-grade material. An accumulation of gravel, mostly chert, is found in the surface layers; clay accumulates in the B and B-C horizons. This soil profile resembles that described by Alexander, et al. (1939) as Frederick silt loam from Fairfield, Va., but is more cherty. Both field examination and the mechanical analyses indicate that leaching has caused this type of profile development. It is a podzolized soil. The low pH (Table 1) supports this statement.

Chemical Composition

Partial analyses of the whole soil (Table 2) indicate the siliceous character of the A horizon, and the increase of Al_2O_3 and Fe_2O_3 in the B horizon. Increase in water content reflects the additional amount of clay present, and ion-exchange capacity also increases with clay content.

The chemical composition of the clay fractions (<2 microns) in Table 3 and the SiO_2/Al_2O_3 ratio agree with those given for similar soils by Alexander, et al., (1939). The variation in the SiO_2/Al_2O_3 ratio in the various parts of the profile is typical of podzolic weathering (Jenny, 1950).

Silica is present in excess of the requirements for the normative formation of kaolinite, hydrous mica, and chlorite, which the x-ray diffractometer patterns show to be present. This excess is approximately as follows: A horizon, 50 percent; B horizon, 30 percent; B-C horizon, 7 percent; C horizon, 20 percent. Quartz is abundant in the finer fractions of the soil and is evidently well crystallized even in the clay fractions, although some chert is probably also present.

Alumina, the next most abundant constituent, is present in amounts about 10 percent less than in analyses of typical kaolinites reported for the A.P.I. standards (Kerr et al., 1950) but is in amounts similar to those obtained for colloids of limestone soils by Alexander et al. (1939), particularly for the Frederick and Hagerstown silt loams. On a normative basis, kaolinite amounts to somewhat more than 50 percent of the clay fraction in all samples except 18B (45 percent), and 18F (25 percent). Table 3 shows that within the clay fractions there is little variation in the alumina content, but consideration of the x-ray data indicate that this alumina is distributed between kaolinite, chlorite, and hydrous mica in different proportions in different parts of the soil profile.

The clay fraction contains a higher percentage of iron than does the soil as a whole; both ferric and ferrous iron are present. The ferric iron occurs as oxide and combined with other elements in minerals. The interpretation of the distribution throughout the profile is interesting from the point of view of podzolic weathering. Much of the ferric iron occurs as free ferric oxide in all parts of the profile, but at about 15 inches, in the B horizon, ferric oxide in a combined state becomes notice-

able and persists to the lowest horizon sampled. Insufficient quantities of iron-bearing minerals such as goethite or hematite have developed to cause peaks in the diffractometer patterns, but, as mentioned under "Methods," a broad diffuse area extending from 8 degrees to 14 degrees 2θ probably indicates the presence of amorphous iron oxide. Ferrous iron is present in the A and B horizons, and its presence may be due to the downward percolation of acid waters containing organic acids conducive to keeping iron in the ferrous state (Byers, et al., 1938). Where conditions farther down in the profile are more conducive to oxidation, ferric iron is found. Ferrous iron is an essential constituent of certain varieties of chlorite, and the presence of this mineral in the clay of the surface horizons may be due, in part, to the conditions which allow iron to remain in the ferrous state there.

Of the remaining constituents, H_2O is probably the most interesting from the mineralogic point of view, for it is an essential constituent of kaolinite and hydrous mica as well as of chlorite. The water, represented by loss on ignition, is somewhat in excess of the amount required for the formation of clay minerals, but, owing to the indefinite water content of hydrous mica, some of this excess is probably located there. The water content is similar to that obtained for impure kaolinites and for the colloids of limestone soils.

Potassium is an essential constituent of hydrous mica, and the percentage found varies throughout the profile. Magnesium is present in small amounts, but calcium is a very minor constituent.

The total ion-exchange capacity of the whole soil is very low for the upper part of the profile, where there is little clay (Table 1), but it rises with increase in clay content. In the clay fractions the ion-exchange capacity varies from 11 meq per 100 g in the A horizon to 20 meq per 100 g in the C horizon. This variation is caused by differences in the type of clay mineral and by the dilution of the soil colloid with silica as shown in the analyses (Table 3). The hydrous mica in the Lenoir limestone has an exchange capacity of 11 meq per 100 g, which is much lower than the figures given by Grim, et al. (1937), but approaches that given for muscovite. The x-ray patterns show that the hydrous mica is contaminated by calcite, which may account, in part, for the low cation-exchange capacity.

Distribution of Clay Minerals

There are several points to be noted in the distribution of the minerals in the clay fraction (<2 microns) of the different horizons of the soil profile as shown by x-ray examination (Table 4). The Lenoir limestone contains hydrous mica with some montmorillonite and very small amounts of kaolinite, but in the soil profile kaolinite is the principal clay mineral throughout, with subordinate amounts of chlorite near the surface (A, A-B, and B horizons) and hydrous mica in the lower parts of the profile,

where it undoubtedly persists as a residual mineral from the weathering of the parent limestone. Silica in excess of that present in the clay minerals is shown in the diffractometer patterns to be quartz, and microscopic examination has shown euhedral quartz grains to be most abundant in the finest fractions. Chert grains are prominent in only the coarser grades. In some of the limestones and dolomites of the area, notably in members of the Beekmantown formation, minute quartz euhedra are a common feature, but they do not seem to be present in the two samples of Lenoir limestone examined. The development of euhedral quartz, therefore, represents one of the mineralogic changes that has taken place during profile development, probably as a result of the solution of chert, but possibly also during reconstruction of some of the clay minerals such as montmorillonite.

Gedroiz (quoted by Kelley, 1948) showed many years ago that as H ions replace metallic cations, the exchange material tends to become increasingly unstable. This is particularly true of montmorillonite, and also of hydrous mica, as shown for the latter mineral by studies made by Jeffries, Rolfe, and Kunze (1953). Ross and Hendricks (1945) pointed out that hydrous mica and montmorillonite are not the end products of weathering and that they will persist only in areas of slow weathering under alkaline conditions. Under acid conditions kaolinite replaces montmorillonite and hydrous mica. More recently, Griffiths (1952) and Frederickson (1952), have shown that kaolinite is generally regarded as an end product of weathering.

In the profile described here, hydrous mica seems to have been converted into chlorite, which remains in the surface soil. The FeO shown in the analyses probably is present in the chlorite, and the fact that conditions are not oxidizing may be conducive to its retention as the principal ion in the brucite layers of chlorite. Should the iron change to the ferric state, the brucite layers would break down and the structure would revert to that of kaolinite. It should not be overlooked, however, that this chlorite may be the chloritic variety of chamosite and may even have developed from kaolinite in the environment in which it is found.

The structures of hydrous mica and chlorite are somewhat similar, but, whereas the double sheets in mica are linked by K ions, those of chlorite are linked by $Mg_2Al(OH)_6$ in 6-coordination — the brucite layer. Jenny (1950) has suggested that on alteration of minerals by weathering, the various units, such as Si-Al tetrahedra, are released as composite units and are available for reconstitution. With removal of K, mica eventually changes to kaolinite, but it is possible that where conditions are favorable, mixed mineral types may be constructed. Chlorite in very small quantities may be intergrown with hydrous mica in the Lenoir limestone, and, by removal of the more easily weatherable mica, may become concentrated in the soil towards the top of the profile; in other words, chlorite could be a residual mineral rather than a reconstructed one.

The small amount of montmorillonite in the Lenoir limestone may result from slight weathering of the limestone whereby release of Mg and Ca may allow these ions to enter into the crystal lattice of hydrous mica producing montmorillonite (Keller and Frederickson, 1952). It is more likely, however, that montmorillonite was the clay mineral in the limestone and that it is in the process of reversion to hydrous mica, which is apparently a more stable mineral.

Geologic Significance of Profile Formation

The detailed examination of this profile has, first of all, confirmed field evidence of weathering processes in limestones and the soils developing from them in this part of the United States. It has demonstrated that kaolinite is the end-product of clay mineral weathering under conditions of acid leaching, probably continued for considerable periods of time. Furthermore, observations on other limestone soils that are less maturely developed than this profile indicate why they contain hydrous mica and/or montmorillonite, and this in turn explains the presence of K_2O, MgO, and CaO in analyses of them although the $CaCO_3$ of the parent limestone has been removed. Immaturity of profile development in the particular environment allows residual montmorillonite and hydrous mica to remain unchanged in such soils.

Of significance, too, is the fact that rapidity of erosion in areas like the Shenandoah Valley prohibits the large-scale development of completely leached soils and encourages the deposition of alluvium in which the soil material is incompletely weathered, thereby greatly increasing fertility from an agricultural point of view. The deposits laid down by the rivers draining such areas will not contain much kaolinite, but mostly hydrous mica, which may later convert to kaolinite as the soil profiles start to develop and become mature. Millot (1952) has pointed out that the clay minerals derived from the removal of soil from areas of acid leaching will be kaolinitic and that the sediments formed will have kaolinite as a major constituent. Taylor (1952) has modified this statement to take into account the erosion factor, and the conditions obtaining today in the Shenandoah drainage area provide an example of the removal of largely nonkaolinitic material from an area, which potentially, should provide kaolinite to future sediments.

The above statements refer to weathering in a humid climate. Where no acid leaching occurs, the clay mineralogy will reflect that of the parent rock, and under these circumstances montmorillonite and hydrous mica may persist for very long periods of time in soils. The final end-product of leaching in a humid climate will be bauxite with the original silica and iron wholly or partially removed. That this does occur, given satisfactory topographic conditions and sufficient time, is shown by the occurrence of a few bauxite deposits in the southeastern part of the Shenandoah Valley in Virginia.

CONCLUSIONS

Examination of a podzolized soil profile developed on Lenoir limestone in the Shenandoah Valley has shown that the original clay minerals of the limestone, hydrous mica and montmorillonite, are changed during profile development under leaching conditions to kaolinite and chlorite. X-ray and chemical studies indicate the changes that have taken place in various parts of the profile. Excess silica has accumulated in the form of euhedral quartz crystals (generally less than 0.05 mm long), and chert is residual from the limestone.

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