INTERSTRATIFIED LAYER SILICATES IN SOME SOILS OF NORTHERN WISCONSIN *

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ABSTRACT

Examination of the silt and clay fractions of two northern Wisconsin soils, Omega loamy sand of the Brown Podzolic group and Ahmeek loam of the Brown Forest group, revealed considerable quantities of interstratified layer silicates. The two soils contained montmorin, vermiculite, chlorite, and illite both as discrete and interstratified components. Regularly alternating montmorin-illite structures, which registered a 28 Å first order diffraction peak, and vermiculite-illite structures, which yielded a 24 Å first order peak, were observed in the fine silt fractions of the Ahmeek loam A_3 and B_{22} horizons, respectively. Weak diffraction peaks in general and lack of binary mixture average spacings was suggestive of ternary or quarternary interstratification of illite, chlorite, vermiculite, and montmorin in the medium and fine clay fractions of the Ahmeek loam horizons. Randomly interstratified vermiculite-chlorite and vermiculite-montmorin, as revealed by comparative basal diffraction peaks before and after heating potassium saturated samples, were evidenced in the fine silt and clay fractions of the Omega profile.

Mixed layer components in the Omega and Ahmeek soils originated from illite and chlorite in the C horizons with a progressive increase in proportions of first vermiculite and finally montmorin with proximity to the soil surface. The observed weathering transitions within the mixed layer structures illustrates the effects of accelerated leaching which has taken place in these coarse textured soil profiles, even though these soils are relatively young (late Pleistocene).

This investigation is concerned with the occurrence of interstratification of the layer silicates illite, chlorite, vermiculite, and montmorin as a function of depth in some relatively young soils (late Pleistocene) of a temperate region. The chemical weathering of the 2:1 and 2:2 layer silicates was found to follow, in general, the weathering sequence as proposed by Jackson (1948) and Jackson and Sherman (1953). The transformation proceeded from the micas, representing stages 4 (trioctahedral mica) and 7 (dioctahedral mica) through chlorite and vermiculite (stage 8) to montmorin (stage 9).

REVIEW OF LITERATURE

The weathering sequence is based on different relative stability of minerals (Jackson and Sherman, 1953). Jackson, *et al.* (1948, 1952, 1954) proposed, on the basis of X-ray diffraction and surface sorption studies,

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that weathering of micas gave "intermediate" minerals consisting of interstratified or "mixed layer" 2:1 and 2:2 (chlorite) layer silicate series, and that interstratification is of general occurrence in clays of soils and sediments as a resultant of weathering.

Interstratification of 2:1 and 2:2 layer silicates has been noted by many workers. Pauling (1930) suggested the possibility of mixed lattices of mica-like minerals and Gruner (1934) concluded that biotite and vermiculite were interstratified in hydrobiotite. Hendricks and Alexander (1939) indicated an interstratification of montmorillonite and illite in colloids of certain soils. Gruner (1948) posed the suggestion that interstratification of any or all combinations of recognized mica and clay minerals might be possible within a single crystal depending upon cation supply and composition of the chemical environment. Barshad (1948) suggested the possibility of mixtures of vermiculite-biotite, vermiculite-chlorite, and biotite-chlorite. The occurrence of mixed layer structures has also been reported by Mackenzie, *et al.* (1949) and Walker (1950). Interstratified growths in clays and clay-like minerals can apparently occur as randomly mixed structures and as regularly alternating structures (Bradley, 1950b). Bradley (1950a), investigating the alternating layer sequence of rectorite, has shown evidence to indicate that it involves the regular alternation of one pyrophyllite unit with one vermiculite unit. Basal diffraction peaks between 10 Å and 18 Å were related by Hendricks and Teller (1942) to binary mixed layer structures. Intermediate spacings have been observed by other workers (MacEwan, 1949; Brown and MacEwan, 1951; and Hsueng and Jackson, 1952).

MATERIALS AND METHODS

The two soils selected for study in this investigation include an Omega loamy sand and an Ahmeek loam, which were sampled in northern Wisconsin. The Omega profile represents a Brown Podzolic soil developed under predominantly pine forest cover on smooth glacial outwash. The Omega soils in general have been derived from very sandy water-laid material which contains a relatively high proportion of basic rock constituents. The Ahmeek profile represents a Brown Forest soil developed under a hardwood forest cover. The parent material is predominantly acidic glacial till which has a higher content of basic rocks than most of the till of northern Wisconsin. Fragments of basalt, gabbro, and diorite are abundant in the Ahmeek profile and parent material. Complete profile descriptions of the two soils are given in the U.S.D.A. Laboratory Memorandum No. 1, Beltsville, Md., 1952.

The profiles selected for study offer an opportunity to examine the mineralogical changes attendant with development of relatively young soils (late Pleistocene) whose parent materials contain appreciable percentages of basic rock constitutents. Both soils have a high content of sand and low contents of silt and clay. The sandy texture gives a high infiltration rate and low moisture holding capacity, with the result that the degree of leaching and consequent weathering of the clay fraction would be expected to be many times faster than found in associated soils of fine texture. For this reason the finer fractions were selected for the most detailed analysis.

Free iron oxides were extracted from the soil samples by the dithionite-citrate method (Aguilera and Jackson, 1953) prior to separation of fractions. Then the fine silt (5 to 2 μ), coarse clay (2 to 0.2 μ), medium clay (0.2 to 0.08 μ), and fine clay (less than 0.08 μ) fractions of selected horizons of the two soils were dispersed and separated by the method of Jackson, *et al.* (1950). The separated fractions were subjected to X-ray diffraction, electron microscopic, specific surface, cation exchange capacity, elemental, and ignition loss analyses. For the objective herein, only diffraction and electron microscope data are presented.

The random powder samples were magnesium saturated and glycerol solvated according to the procedure of White, *et al.*, as reviewed by Jeffries and Jackson (1949), and X-rayed with a General Electric XRD-1 diffraction unit equipped with cylindrical cameras. Parallel oriented samples of each fraction were analyzed with General Electric XRD-3 diffractometer. Two specimens of each fraction were prepared with parallel orientation (Jackson, *et al.*, 1954). One specimen was magnesium saturated and glycerol solvated as a means of detecting expanded layer silicates. The other was potassium saturated and analyzed after drying at room temperature and after heating to various temperatures as a means of distinguishing between collapsible and noncollapsible high spacing layer silicates. Specific gravity separates of the coarse medium silt (50-20 μ) and (20-5 μ) were also X-rayed in some cases by the diffractometer.

LAYER SILICATES PRESENT AND EVIDENCE OF INTERSTRATIFICATION

The (hk0) Diffraction

The random powder X-ray diffraction patterns (not shown) of the Omega fractions revealed the presence of a relatively high concentration of layer silicates in both the Omega and Ahmeek horizons by a strong diffraction peak at a 4.5 Å spacing. In the B_{22} horizon medium clay fraction of the Omega profile, the 4.5 Å peak was the only diffraction peak observable, and in many patterns the 4.5 Å peak was the strongest present. In all cases, the strong 4.5 Å peak was accompanied by considerable small angle scattering. The resolution of a strong 4.5 Å diffraction maximum, in the absence of layer silicate basal diffraction and accompanied by considerable small angle scatter, is suggestive of interstratification of the layer silicate members present. The (hk0) spacing of near 4.5 Å is very nearly the same in all the common 2:1, 2:2, and 1:1 layer silicates normally found in soils and is independent of Z-zonal extent of each species in an interstratified mixture. The basal peaks of such an interstratified series may not be resolved, however, if the Z-zonal extent of each member is limited (Jackson, *et al.*, 1952, 1954).

The presence of a strong (hk0) diffraction peak in the absence of basal spacings cannot necessarily be taken as a positive criteria for interstratification, however, since decrease in particle size can conceivably cause the same effect. One might well expect the basal spacing intensity to decrease relative to (hk0) peak with decreasing particle size. The dimensions of a layer silicate particle along the Z-axis is much smaller than the dimensions along the X and Y axes. If a layer silicate particle be sufficiently large, the X, Y, and Z dimensions are of sufficient extent to yield both basal and (hk0) diffraction peaks. If the extent of X, Y, and Z dimensions of each crystal decrease proportionately with decrease in particle size a point may be reached at which there will be too few basal planes to allow complete cancellation of destructive interference and basal diffraction peaks will decrease in intensity. At this point, however, the X and Y axes would still be of sufficient extent to allow intense diffraction from (hk0) planes.

The Omega Loamy Sand, Brown Podzolic Soil

Fine Silt. — The X-ray diagrams of magnesium saturated-glycerol solvated 5 to 2 μ parallel oriented specimens of the Omega profile (Fig. 1, left four diagrams) reveal relatively high concentrations of a near 14 Å component, with the concentration appearing highest in the B_{22} and C_{21} horizons. Some of this material has a tendency to collapse when saturated with potassium and dried at room temperature, as evidenced by broadening of the 14 Å peak accompanied by a general decrease in intensity. Such an effect is similar to that noted by Barshad (1948) who found that vermiculite collapsed and attained properties very similar to ordinary biotite upon saturation with potassium. Heating of the potassium saturated samples at 500° C for two hours caused a further decrease in intensity of the 14 Å diffraction peaks, accompanied by an enhancement of a near 10 Å diffraction peak, showing that some of the original 14 Å material was actually collapsing to 10 Å. This effect, in accordance with conclusions of Gruner (1934), Barshad (1948), and Walker (1951, p. 203), is interpreted as sufficient evidence of the presence of vermiculite.

The increase in 14 Å peak between 500° C and 600° C, accompanied by extinction of the 7 Å peak is taken as a property of chlorite present. This effect is commensurate with observations of Brindley and Ali (1950) and Brindley and Youell (1950), who found that some chlorites show an increase in first-order and a decrease in second order basal diffraction intensity as a result of heating to 600° C. The heating results in decomposition of some of the brucite layer, and hence a decrease in electron density at one-half unit cell distance between the octahedral sheets of the 2:1 layers.

Coarse Clay. — The 2 to 0.2 μ fractions of the Omega horizons (Fig. 1, right four diagrams) all show considerable quantities of 14 Å material which only partially collapsed to near 10 Å upon heating. There is evidence of the 10 Å peak of illite in the magnesium saturated samples from the B_{22}, C_{21}, and C_{22} horizons, but none in the A_3 horizon fraction. A small

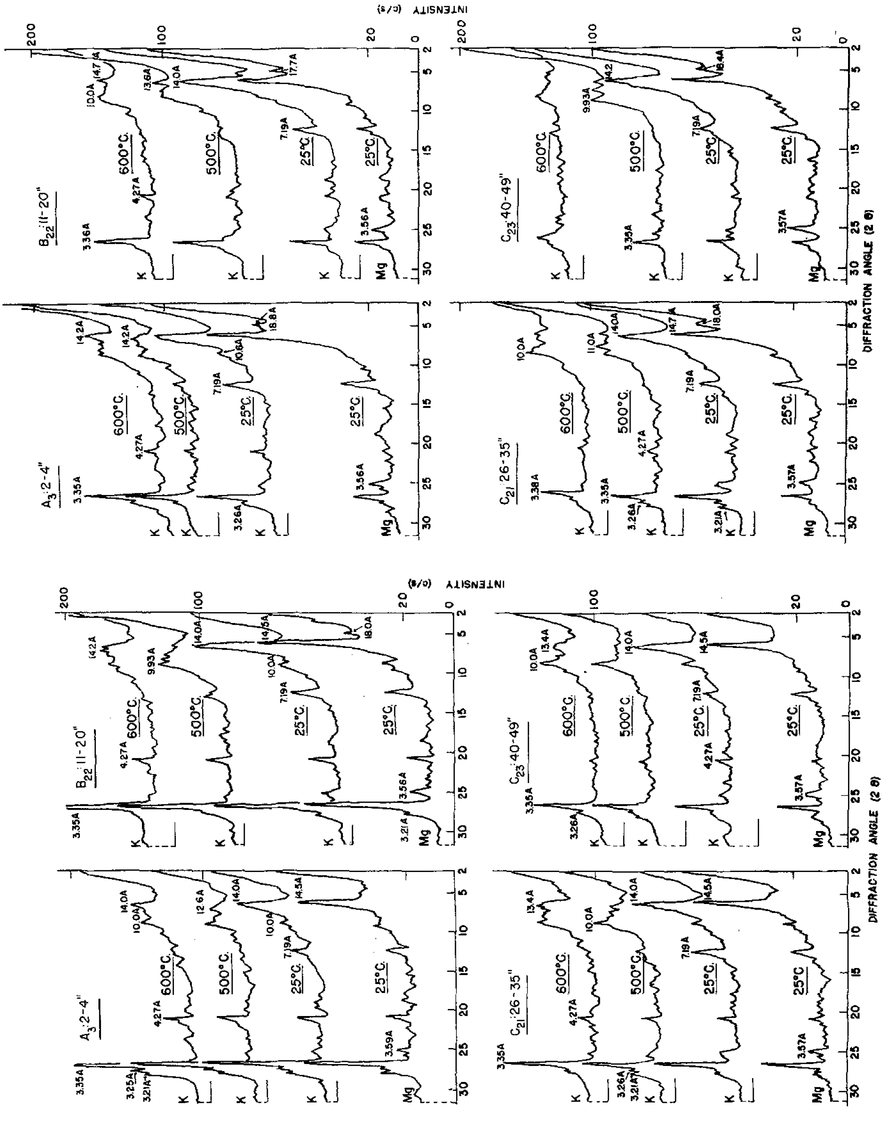

FIGURE 1. — Parallel oriented sample X-ray diffraction diagrams of the 5 to 2 μ (left) and 2 to 0.2 μ fractions of the Omega profile horizons showing the effects of magnesium saturation-glycerol solvation and potassium saturation-heat treatments.

amount of montmorin was also detected in each horizon, as evidenced by the near 18 Å peak obtained from glycerol solvated samples, but these peaks in no instance were resolved in the film-recorded patterns.

Interstratification. — That the 2:1 and 2:2 layer silicates of the 5 to 2 μ and 2 to 0.2 μ fractions are interstratified to a certain extent is supported by the X-ray diagrams of Figure 1. The 10 Å and 14 Å diffraction intensities, after heating the samples to 500° C, were much lower when taken together than the original 14 Å intensity before heating. If chlorite and vermiculite were present entirely as discrete particles, the summation of the 10 Å and 14 Å peaks after heating should be comparable to the original 14 Å intensity. Further, appearance of peaks between 10 Å and 14 Å after heating is suggestive of some random interstratification of 10 Å and 14 Å materials.

Medium Clay. — Montmorin, vermiculite, chlorite and illite compose the major portion of the 0.2 to 0.08 μ fractions of the Omega horizons with vermiculite dominant (Fig. 2, left four diagrams). An 18 Å montmorin peak together with much vermiculite (10 Å on heating) were registered in the A_3 horizon, while illite and chlorite were present in increasing quantities in the B_{22}, C_{21}, and C_{23} horizons. An illustration of interstratification is afforded by the C_{21} horizon. The glycerol solvated sample showed weak 14 Å and 17 Å peaks. Upon potassium saturation of the sample, however, a strong 14 Å peak was resolved which was more intense than the original 14 Å and 17 Å peaks combined. Upon heating to 500° C, a strong but broad 10-11 Å peak was resolved. This occurrence suggests that montmorin is interstratified with vermiculite and probably some chlorite is interstratified with them. The interstratified structure contains too few repetitions of any one of the components in a given zone (Jackson, *et al.,* 1952, 1954) to yield strong diffraction peaks. When potassium is applied to the material, however, the montmorin collapses to coincide with the 14 Å peak produced by the vermiculite and chlorite. Upon heating to 500° C, both the montmorin and the vermiculite collapsed to yield a strong 10 Å peak. Specific surface and cation exchange capacity measurements confirm a considerable vermiculite and montmorin content.

Fine Clay. — The less than 0.08 μ fractions of the Omega horizons (Fig. 2, right four diagrams) gave no distinct peaks for any of the layer silicates found in coarser fractions. In general, there was high diffraction background in the low angle region and a broad peak at 3.5 Å which suggests (Jackson, *et al.,* 1954) that there are materials present with intermediate spacings in the range from 10 Å to 18 Å. It is felt, however, that lack of discrete diffraction peaks for the less than 0.08 μ fractions is due in large part to fineness of the individual layer silicate particles which are established to be present by the (hk0) peaks (previous section). That interstratification also contributes to the diffraction effects obtained is illustrated by the patterns obtained from the A_3, B_{22}, and C_{21} horizons. A distinct, though broad peak was resolved after heating where no peak had existed before, showing that expanded layer silicates had been shifted to a

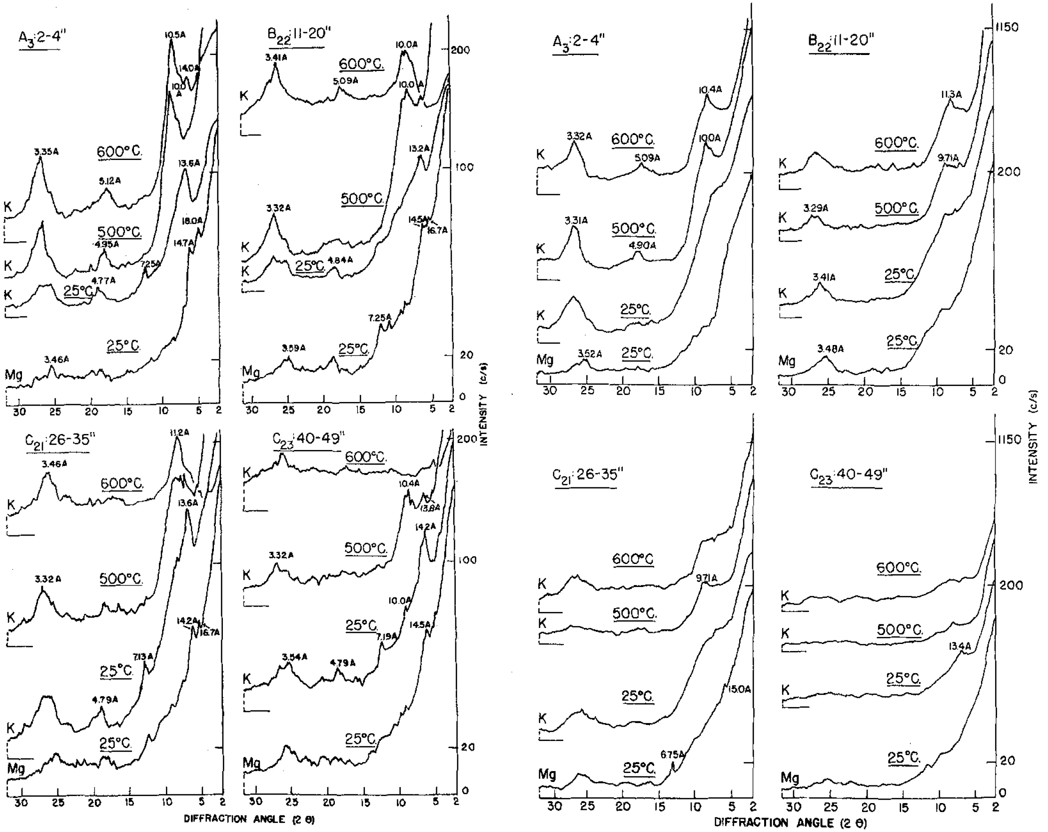

Figure 2. — Parallel oriented sample X-ray diffraction diagrams of the 0.2 to 0.08 μ (left) and less than 0.08 μ fractions of the Omega profile horizons showing the effects of magnesium saturation-glycerol solvation and potassium saturation-heat treatments.

common spacing by the treatment. Even in the C_{23} horizon a 13.4 Å spacing was resolved by K saturation. The over-all low diffraction intensity suggests the presence of some x-amorphous layer silicate weathering relic material, as also evidenced by electron micrographs (below).

The Ahmeek Loam, Brown Forest Soil

The X-ray patterns from parallel oriented samples of the Ahmeek loam fractions (Figs. 3 and 4) reveal the presence of montmorin, vermiculite, chlorite, and illite, with the expanded type minerals showing an increase in concentration with proximity to the surface. Within each particle size range, the montmorin concentration is highest in the A_1 horizon, the vermiculite concentration is highest in the B_{22} horizon, and the chlorite and illite concentrations are highest in the C_1 and C_2 horizons.

Fine Silt. — The resolution of a spacing of near 29 Å in the glycerol solvated 5 to 2 μ fraction of the A_1 horizon (Fig. 3, left) suggests that there is present some regularly alternating interstratified montmorin plus illite (18 Å + 10 Å = 28 Å). The resolution of 24 Å peaks in the 5 to 2 μ fraction of the B_{22} and C_1 horizons has pronounced significance, related to the interpretation of the 29 Å peak obtained from the A_1 horizon fraction. Montmorin is virtually absent in the B_{22} horizon fraction but vermiculite is present in considerable quantity. This suggests, then, that there is present regularly alternating interstratified vermiculite plus illite (14 Å + 10 Å = 24 Å). Occurrence of the first order spacing requires that it arise from regularly rather than randomly interstratified layers. The occurrence reflects a very significant aspect of the layer silicate weathering sequence. It would appear that vermiculite present in the B_{22} horizon has further weathered to montmorin in the A_1 horizon. The decrease in chlorite concentration in the B_{22} compared to lower horizons, accompanied by an increase in vermiculite, suggests that the chlorite may be weathering to vermiculite. Some 24 Å spacing appears even in the C_2 horizon, and this horizon, of course, must not be considered to be entirely unweathered. The fact that the 24 Å peak was absent after the 500° C and 600° C heating, together with the lack of an electron density low, precludes its rise from regularly interstratified chlorite plus illite.

Coarse Clay. — The 2 to 0.2 μ fractions (Fig. 3, right four diagrams) yielded little or no higher spacings indicative of regular interstratification of layer silicate components. The lack of such interstratification within this size range may well be a manifestation of more intensive weathering as a function of particle size. The intensity of diffraction peaks of montmorin in the A_1 horizon and vermiculite in the B_{22} horizon, suggests that these layer silicate members are present largely as discrete particles rather than as interstratified mixtures. In the C_1 and C_2 horizons, however, diffraction intensity is less and it is suspected that the layer silicates are interstratified to some extent. Only in the coarse clay of the A_1 horizon, in which the montmorin was not interstratified and gave high intensity, was the 18 Å

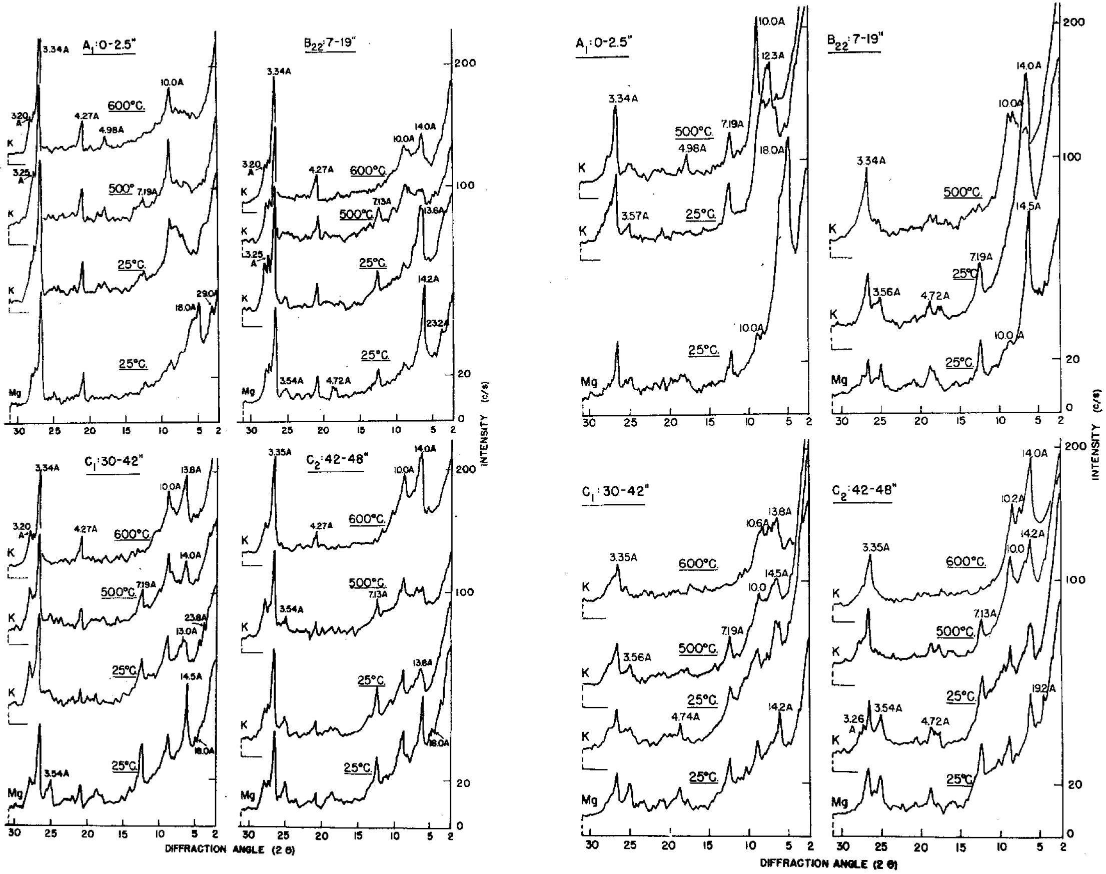

FIGURE 3. — Parallel oriented X-ray diffraction diagrams of the 5 to 2 μ (left) and 2 to 0.2 μ fractions of the Ahmeek profile horizons showing the effects of magnesium saturation-glycerol solvation and potassium saturation-heat treatments.

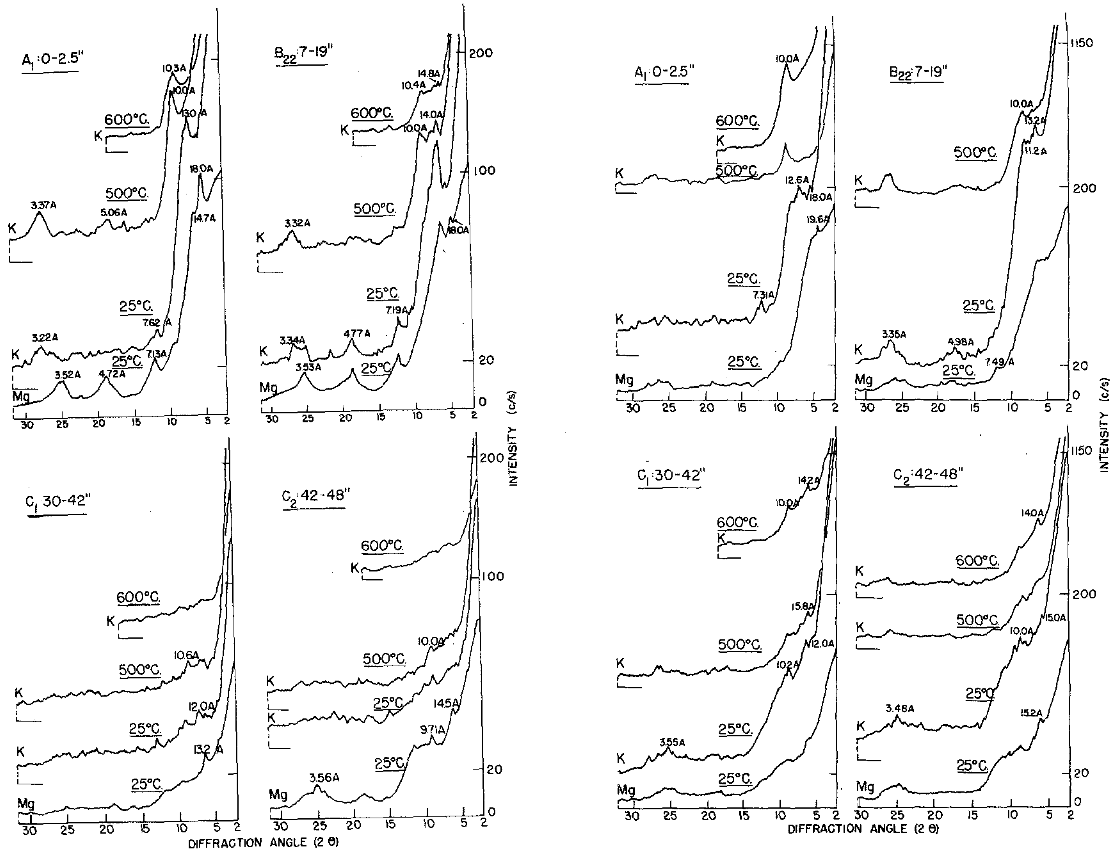

FIGURE 4.—Parallel oriented sample X-ray diffraction diagrams of the 0.2 to 0.08 μ (left) and less than 0.08 μ fractions of the Ahmeek profile horizons showing the effects of magnesium saturation-glycerol solvation and potassium saturation-heat treatments.

peak resolved and distinguishable in the film-recorded diffraction patterns.

Medium Clay. — The weak diffraction intensity from the 0.2 to 0.08 μ fractions of the C_1 and C_2 horizons (Fig. 4, left four diagrams) is concluded to be due largely to interstratification of the layer silicate members, since more intense peaks were obtained from A_1 and B_{22} horizon samples of the same size. It is suspected that there may be a quaternary interstratification involving mica, chlorite, vermiculite, and montmorin in these deeper horizons. Resolution of distinct peaks at 14 Å and 18 Å in the A_1 and B_{22} horizon fractions indicate that weathering has proceeded far enough to produce x-crystalline zones of sufficient extent to yield good peaks.

Fine Clay. — Montmorin and vermiculite were the major constituents of the less than 0.08 μ fractions of the A_1 and B_2 horizons, respectively (Fig. 4, right four diagrams). A small amount of chlorite (non-collapsible) was detected in the C_1 and C_2 horizon fractions. In general, the diffraction patterns were very weak, suggesting interstratification of the layer silicate members. Particle size, however, undoubtedly also contributes to lack of diffraction intensity in these fine fractions.

Electron Micrographic Evidence

Representative electron micrographs (Fig. 5) of fractions of the Omega and Ahmeek profiles reveal that the mineral particles are predominantly of a layer silicate habit as shown by their thin platey structure. The apparent crystalline nature of the minerals of the 0.2 to 0.08 μ fraction shown in Figure 5, which yielded relatively weak X-ray diffraction peaks, suggests the presence of interstratified mixtures of layer silicates. The fine clay particles less than 0.08 μ also showed similar thin platey habit, differing only in being fleecy and poorly defined in outline and thus suggesting that the fine clay contains x-amorphous layer silicate relic material.

Significance

The observed transitions from one to another layer silicate illustrates the effects of accelerated leaching in relatively young soils, and contributes to knowledge of the mechanism of weathering of illite and chlorite to vermiculite and this to montmorin. The depth function (more weathering with proximity to the soil surface) is illustrated. The direct evidence of interstratification was due almost exclusively to the high resolution of the X-ray diffractometer applied to parallel oriented samples. But specific internal surface and cation exchange capacity measurements concord.

Percolating rain waters have had free passage through these sandy profiles, and pass in large amounts because of the low water holding capacity of these sandy soils. As a result, the minerals have been subjected to many times more extensive leaching than finer textured soils in the same region. Great changes in layer silicate clay mineral weathering stage within the profile depth are the result. In a medium or fine textured soil, under the

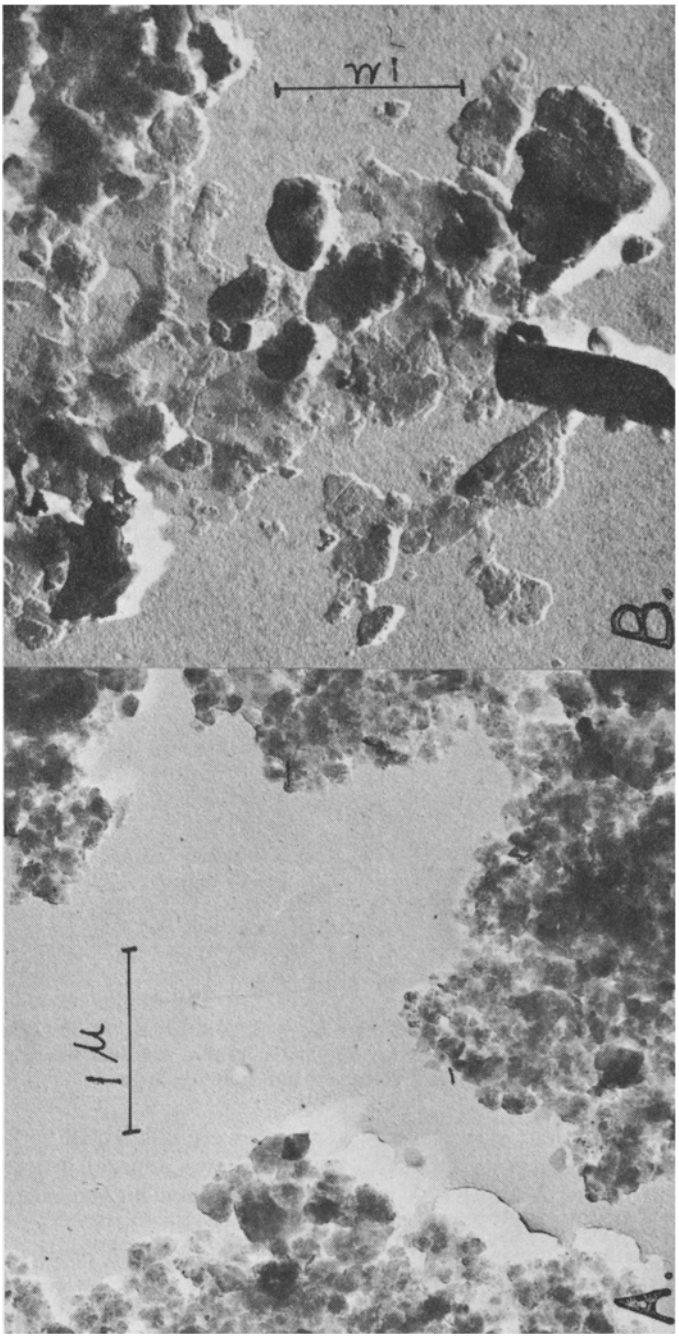

FIGURE 5. — Representative electron micrographs of fractions of the Omega and Ahmeek profiles. (A) Omega, B_{22} horizon, 0.2 to 0.08 μ. (B) Ahmeek, A_1 horizon, 0.2 to 0.08 μ.

same environmental conditions, leaching action would be less effective and less mineral changes would be expected.

OCCURRENCE OF AN UNUSUAL HEAVY MINERAL IN THE SILT FRACTIONS OF THE OMEGA AND AHMEEK SOILS

A mineral with unusual properties was detected in the medium and coarse silt fractions of the two soils studied. A considerable portion of these fractions was composed of a mineral of greater than 2.95 specific gravity. When the heavy material was analyzed with the XRD-3 diffractometer, it yielded a relatively strong peak at approximately 8.6 Å. Representative X-ray diagrams of the material from the Omega profile are shown in Figure 6. The most common occurrence is exemplified by the 20 to 5 μ fraction of the A_3 horizon, the spacings of which were unaffected by heating. The 50 to 20 μ fraction of the C_{23} horizon shows unusual properties in that very strong 10 Å and 8.6 Å peaks and associated higher orders at 4.3, 4.1, 3.56, 3.26, and 3.15 Å were resolved after heating to 500° C. The response to heating suggests strongly the presence of random ternary and quaternary interstratification. The 10 Å peak would form by the joint effects of montmorin, vermiculite, and illite having this common spacing after heating; occurrence of merely a binary interstratified mixture of two

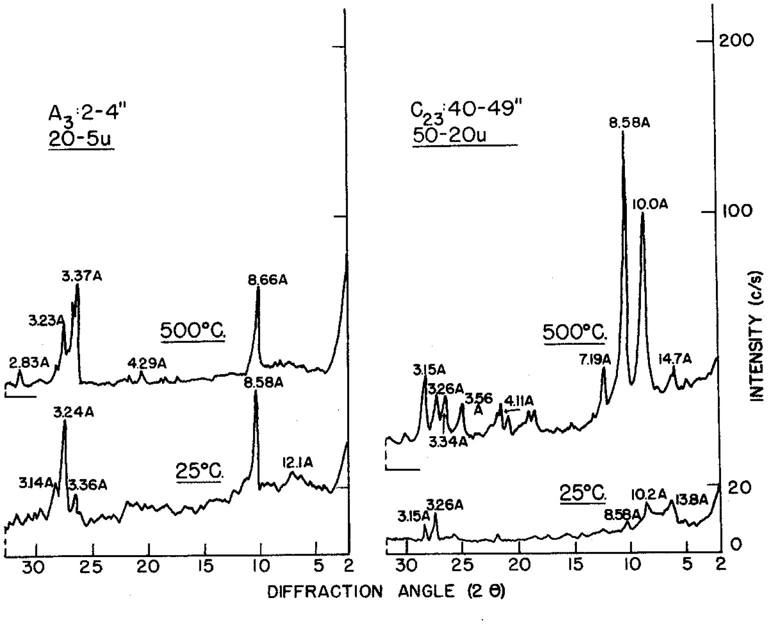

FIGURE 6. — Parallel oriented sample X-ray diffraction diagrams of the greater than 2.95 sp. gr. concentrate of fractions of the Omega profile horizons showing the effects of magnesium saturation, glycerol solvation, and heat treatment.

of these minerals is precluded by lack of Hendricks and Teller mixed spacings before heating, suggesting that the three minerals are present as a ternary mixture. The 8.6 Å peak could arise from randomly interstratified 7 Å and 10 Å minerals (Brown and MacEwan, 1951, p. 276). The small 14.7 and 7.19 Å peaks after heating may be attributed to chlorite and antigorite. Antigorite without chlorite is indicated in Figure 3 (left B_{22} horizon) by a 7.13 Å spacing without a 14 Å spacing. The presence of antigorite (and possibly chlorite, providing an electron density distribution like a 1 : 1 structure in the mixture, which its 2 : 2 structure well might) interstratified with two or three of the minerals which give a 10 Å spacing on heating to 500° C could explain the 8.6 Å spacing after heating. The standard amphibole spacings in the region of 8.40 to 8.48 Å do not concord with the 8.6 Å spacing which occurs without heating, and does not explain the striking effect of heating on the C_{23} horizon sample.

SUMMARY

Illite and chlorite, more abundant in the fine silt and clay fractions in the C horizon, were found to weather to vermiculite and montmorin with proximity to the soil surface in two very sandy soils of northern Wisconsin in which leaching is severe. Evidence was found of the occurrence of interstratification of these four layer silicates in binary-regular (24 and 29 Å spacing) and binary-, ternary-, and quaternary-random mixtures. An unusual heavy mineral was found which gave low background and low diffraction peaks before heating and gave very acute 10 Å and 8.6 Å peaks after heating to 500° C, explainable by ternary and quaternary interstratification.

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