FORMATION OF HYDROXY-Al AND -Fe INTERLAYERS IN MONTMORILLONITE AND VERMICULITE: INFLUENCE OF PARTICLE SIZE AND TEMPERATURE

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Abstract—Hydroxy-Al and -Fe interlayers were prepared in “mono-mineralic” fine and coarse clay montmorillonite and coarse clay and silt vermiculite at 3°C and 21°C. The formation of hydroxy interlayers was evaluated by X-ray diffraction, cation exchange capacity, and chemical analyses.

At comparable particle size and regardless of temperature, the amounts of hydroxy-Al and -Fe interlayers in montmorillonite exceeded by far those formed in vermiculite. Likewise, the aluminum systems exhibited a higher degree of interlayering than iron systems.

Within montmorillonite, the amount of hydroxy-Al and -Fe interlayers increased as the particle size decreased, regardless of temperature. The aluminum interlayered montmorillonite equilibrated at 3°C was characterized by basal spacings of about 17 Å after Ca-saturation plus 54 per cent relative humidity. These spacings are larger than those normally observed for smectites.

Within vermiculite systems equilibrated at 3°C, more hydroxy-Al interlayers were recorded in coarse clay than in silt fraction, whereas at 21°C about equal amounts of interlayers were formed. By contrast, hydroxy-Fe interlayer was favored by the silt fraction at both temperature levels.

The formation of aluminum interlayers in both minerals increased with increasing temperature. The formation of hydroxy-Fe interlayers in montmorillonite was generally not temperature dependent, whereas the formation of such interlayers in vermiculite increased slightly with increasing temperature.

These data may partially explain the formation of chloritic intergrades in soils as a function of type of minerals, kind of ions, and thermal variations.

INTRODUCTION

The 14 Å spacing 2:1-2:2 chloritic intergrades (Jackson, 1963) occur in soils under a wide set of environmental conditions. The chlorite-like intergrade minerals in soils have originated from montmorillonite (Singleton, 1966; Tamura, 1957, 1958; Dixon and Jackson, 1960), chlorite (Drost, 1956), beidellite (Singleton, 1966) or vermiculite (Brown, 1953; Sawhney, 1960; Dixon and Jackson, 1962). Their preferential formation in one or the other mineral matrix would depend primarily upon the intensity and duration of the weathering forces, mineralogical nature of the parent material, acidity of the medium and chemical environment.

It is expected that chlorite-like structures would readily form in vermiculite because of its similarity to chlorite with respect to degree and location of isomorphous substitution. Dixon and Jackson (1962) postulated that more hydroxy interlayer development occurs in the coarser clay fraction of soils. Further, they suggested that the hydroxy interlayers may be preferentially formed in vermiculite rather than montmorillonite because of the higher charge of the former. Later, Jackson (1963) stated that natural hydroxy interlayers form in montmorillonite as well as in vermiculite. However, he implied that the formation of hydroxy interlayers in montmorillonite is countered by a rapid disappearance of the mineral itself as a result of acid weathering forces.

Pawluk (1963) minimized the argument that chloritic intergrades are mostly formed in vermiculite because of their frequent occurrence in the coarse clay fraction. He found that the hydroxy interlayers in some podzolized soils of Alberta, Canada, bound the fine clay particles of montmorillonite into aggregates of coarse clay intergrade. The coarse clay intergrade was converted into fine clay montmorillonite by removing the binding interlayers.

Numerous investigators have been successful in synthetizing intergrade minerals in the laboratory (Sawhney, 1960; Rich, 1960; Slaughter and Milne, 1960; Shen and Rich, 1962; Barnishel and Rich, 1963; Hsu and Bates, 1964; Carstea, 1965,
The resulting information contributed toward a better understanding of the natural "chloritization" process. It is generally agreed that the influence of pH, salt concentration, kinds of ions, layer charge and time, are among the essential factors responsible for the genesis of hydroxy interlayers in expansible layer silicates.

Although the importance of temperature and crystal size in determining the weathering rate of minerals is well recognized (Jackson et al., 1948; Jackson, 1963), these factors have received relatively little attention in the synthetic studies. Rich (1960) reported that the hydrolysis and polymerization of Al-vermiculite showed a considerable increase when boiled for 2 hr in an aqueous media. Drying experiments, some in conjunction with wetting cycles, were also conducted with hydroxy interlayered materials.

Recent laboratory studies by Carstea (1965, 1967) have shown that the formation of hydroxy-Al, -Fe and -Mg interlayers should be looked upon as a dynamic process which exhibits significant changes with time. He showed that initially it was much easier to form stable chlorite-like minerals in montmorillonite than in vermiculite. The layer charge density and pH conditions were cited as primary factors to explain this behaviour. However, the role of particle size has not been fully assessed because the size fractions of the experimental minerals were not comparable. Consequently, there is a need for a study designed to investigate the role of particle size with respect to hydroxy interlayer formation.

It is the objective of the present study to examine and assess the influence of temperature and particle size upon the formation of synthetic hydroxy-Al and -Fe interlayers in montmorillonite and vermiculite.

**MATERIALS AND METHODS**

**Materials**

Montmorillonite 21 from Chisholm Mine, Polkville, Mississippi, was supplied by Ward's Natural Science Establishment. The < 0-2 μ and 2-0-2 μ fractions were obtained by centrifugation following chemical treatments intended to remove organic matter, free iron oxides, allophane and free alumina and silica (Jackson, 1956; Hashimoto and Jackson, 1960).

African vermiculite obtained from Perlite Co., Portland, Oregon was mainly a trioctahedral vermiculite with 1:1 regularly interstratified 10:14 Å component. Preliminary X-ray diffraction analysis suggested the presence of some hydroxy interlayer and possible kaolinite. The 2-0-2 μ and 50-2 μ size fractions were obtained by the wet sieve method in conjunction with centrifugation (Jackson, 1956). The selected samples were subjected to appropriate treatments in order to remove interlayer K (Carstea, 1965), "free iron" (Jackson, 1956) and amorphous, hydroxy interlayer and kaolinite components (Dixon and Jackson, 1960).

Prochlorite 6 from Chester, Vermont was obtained from Ward's Natural Science Establishment. The 2-0-2 μ and 50-2 μ fractions were separated by centrifugation and wet sieving with no additional chemical treatments. The introduction of this mineral was mainly to serve as a standard for comparison. The properties of minerals are shown in Table 1. Calcium-saturated montmorillonite formed a duo-interlayer with ethylene glycol and

<table>
<thead>
<tr>
<th>Particle size</th>
<th>CEC me/100 g</th>
<th>Ca 54%RH</th>
<th>Basal spacings (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Ca K 105°C</td>
</tr>
<tr>
<td>Montmorillonite</td>
<td></td>
<td></td>
<td>solv.</td>
</tr>
<tr>
<td>&lt; 0-2 μ</td>
<td>127.5</td>
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<td>2-0-2 μ</td>
<td>121.3</td>
<td>15.0</td>
<td>17.0</td>
</tr>
<tr>
<td>Vermiculite</td>
<td></td>
<td></td>
<td>14.5</td>
</tr>
<tr>
<td>2-0-2 μ</td>
<td>149.0</td>
<td>14.4</td>
<td>14.0</td>
</tr>
<tr>
<td>50-2 μ</td>
<td>177.9</td>
<td>14.7</td>
<td>14.0</td>
</tr>
<tr>
<td>Chlorite</td>
<td></td>
<td></td>
<td>14.3</td>
</tr>
<tr>
<td>2-0-2 μ</td>
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<td>14.3</td>
<td>14.2</td>
</tr>
<tr>
<td>50-2 μ</td>
<td>4.3</td>
<td>14.2</td>
<td>14.2</td>
</tr>
</tbody>
</table>
expanded to near 17 Å, whereas vermiculite formed only a single-sheet complex. The K + 105°C treatment in conjunction with dry air collapsed the crystal layers of montmorillonite and vermiculite in the vicinity of 10 Å. In contrast, chlorite showed no changes in basal spacings regardless of the treatment imposed.

Methods

Synthetic hydroxy-Al and -Fe interlayers were prepared according to a method described by Carstea (1965). The OH/cation solution ratio, expressed on a milliequivalent basis, was maintained constant, namely 0.85 throughout this study. However, the resulting pH values varied for different systems and they were measured with a Beckman model G pH meter after 1 min and 10 days of equilibration. The preparation and equilibration of interlayered systems were made at two levels of temperature, namely 3°C and 21°C. Following a 10-day period with daily shaking, the samples were centrifuged and the liquid and solid phases were analyzed. X-ray diffraction and CEC data of resulting chloritic intergrade were compared with those of standard clay minerals.

X-ray analyses were made with a Philips X-ray diffractometer equipped with a proportional counter and pulse-height analyzer using CuKα radiation. The oriented Ca- and K-saturated samples were prepared according to the method suggested by Theisen and Harward (1962). Following a two-day equilibration period at 54 per cent relative humidity, the Ca-saturated samples were first irradiated in the presence of a similar relative humidity and again after solvation with ethylene glycol (Kunze, 1955). There has been no attempt to control the relative humidity during irradiation of glycolated samples. The K-saturated samples were analyzed in the presence of dry air after heating for 2 hr at 105°C, 300°C and 550°C.

Cation exchange capacity data (CEC) were obtained by using Rich's method (1961) with the modification that sodium was substituted for magnesium as the replacing cation. Aliquots of the NaCl extractions were titrated with cyclohexanediamine tetraacetic acid (CyDTA) using calcine as an indicator (Carlson and Johnson, 1961).

In the extract, aluminum was determined by the aluminon method (Hsu, 1963) with a slight modification. One drop of thio-glycolic acid was added prior to color development to reduce any ferric to ferrous iron and thus eliminate its effect on the color of the aluminum-aluminon complex (Robertson, 1950). Iron was determined with the aid of a Perkin-Elmer 303, atomic absorption spectrophotometer.

RESULTS

Aluminum interlayered systems

At comparable particle size and regardless of temperature level, the degree of aluminum interlayering in montmorillonite was greater than in vermiculite (Table 2). This is primarily shown by the decrease in CEC and by the resistance to collapse of chloritic intergrades on K-saturation and heating. Further, more hydroxy interlayer formed at 21°C than at 3°C in both minerals. Although chemical data showed more aluminum fixation by clay vermiculite at 21°C than at 3°C, X-ray diffraction patterns exhibited no differential behavior as a function of temperature. This may suggest that some aluminum was adsorbed on the edge surface presumably hindering the passage of cations into the interlayer space. This is also consistent with previous findings (Carstea, 1967).

Within montmorillonite, the degree of interlayering increased as the particle size decreased regardless of temperature. Furthermore, the amount of hydroxy interlayers showed a slight but consistent increase for both size fractions with an increase in temperature. The coarse clay apparently displayed more sensitivity to temperature than the fine clay.

The greatest degree of interlayering was exhibited by montmorillonite equilibrated at 21°C which showed a diagnostic X-ray reflection and CEC of chlorite. This resulting material would meet the requirements of a 2:1-2:2 intergrade of 14 Å spacings (Jackson, 1963). On the other hand, the systems equilibrated at 3°C resulted in a swelling 2:1-2:2 intergrade. This chloritic intergrade exhibited a basal spacing of about 17 Å when equilibrated with 54 per cent relative humidity or ethylene glycol, in spite of its relative high thermal resistance. The chemical data suggested that the threshold between swelling and non-swelling intergrades in a montmorillonite matrix is very narrow. Caillère and Hénin (1949) reported that magnesium interlayered montmorillonite exhibited basal spacings between 17.6 Å and 14.1 Å as a function of relative humidity. The appearance of larger than normal spacings, similar to those observed in this study, has been reported in aluminum interlayered montmorillonite after solvation or hydration (Slaughter and Milne, 1960; Barnishel and Rich, 1963).

Within vermiculite, more hydroxy-Al interlayers were formed in coarse clay than in silt fraction at 3°C, whereas about equal amounts of interlayers were formed in the silt fraction at 21°C. The silt fraction was more temperature dependent than the clay fraction. Although the amount of aluminum adsorbed by the silt fraction at 21°C increased almost three times compared to the
Table 2. Physical and chemical properties of hydroxy-Al interlayered montmorillonite and vermiculite after 10 days of equilibration

<table>
<thead>
<tr>
<th>Particle size (μm)</th>
<th>pH</th>
<th>CEC</th>
<th>Basal spacings (Å)</th>
<th>X-ray Pretreatments</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 min</td>
<td>10 days</td>
<td>me/100g</td>
<td>(%) red'n Al adsorbed</td>
</tr>
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<td></td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>Montmorillonite</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>&lt; 0.2</td>
<td>5.6</td>
<td>5.3</td>
<td>18.8</td>
<td>85.2</td>
</tr>
<tr>
<td>2-0.2</td>
<td>5.5</td>
<td>5.1</td>
<td>25.6</td>
<td>78.9</td>
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<tr>
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</tr>
<tr>
<td>2-0.2</td>
<td>5.6</td>
<td>5.3</td>
<td>95.9</td>
<td>35.8</td>
</tr>
<tr>
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<td>134.2</td>
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<td>&lt; 0.2</td>
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<td>5.3</td>
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<td>50-2</td>
<td>5.7</td>
<td>4.8</td>
<td>100.3</td>
<td>43.4</td>
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</table>

*Initially, 72 mg of aluminum per 0.5 g of mineral were added.
†Where two peaks were observed, the more prominent one is underlined.

amount adsorbed at 3°C, the X-ray diffraction measurements revealed only slight differences between the two systems. This was interpreted as an indication of an increased aluminum adsorption on the external surface by silt vermiculite with increasing temperature.

The 12 Å diffraction peaks observed after K-saturation suggested the occurrence of mixed systems of 14 Å Al-polymer vermiculite and 10 Å K-vermiculite. All interlayered vermiculite samples responded easily to heat at 550°C. An intergrade structure was therefore achieved in vermiculite, but the degree of interlayering was lower than in montmorillonite.

Iron interlayered systems

Like aluminum interlayered systems, more iron hydroxy interlayer was formed in montmorillonite than in vermiculite, regardless of particle size and temperature (Table 3).

The X-ray diffraction patterns and CEC measurements of interlayered montmorillonite showed essentially no difference in the degree of interlayering between comparable size fractions at two levels of temperature. However, about 30 per cent more iron was fixed at 21°C by both size fractions than at 3°C. Since the additional amount of iron adsorbed was not reflected in X-ray diffraction patterns, it was inferred that most of the iron was adsorbed externally rather than between layers. The iron interlayers did not inhibit expansion on solvation of the clay matrix, but the interlayered systems exhibited a moderate resistance to collapse on K-saturation and heating. The diffraction patterns were characterized by diffuse peaks and plateaus suggesting poorly organized and mixed layer systems. In addition, the diffuse patterns and high backgrounds may be caused by a poor orientation of the particles due to irregular masses of precipitate between and around the platelets.

Unlike montmorillonite, the CEC would indicate that more hydroxy iron interlayers were formed in vermiculite at 21°C than at 3°C regardless of particle size. The silt fraction favored a slightly higher degree of interlayering than coarse clay at 21°C. The iron interlayered vermiculite systems exhibited relatively distinct peaks which suggested a higher degree of orientation and crystallization of the iron interlayers in vermiculite than in montmorillonite.

**DISCUSSION**

**Layer charge**

The higher degree of interlayering in montmorillonite as compared to vermiculite in similar size fractions is primarily attributed to the differences in the amount and location of charge between the...
Table 3. Physical and chemical properties of hydroxy-Fe interlayered montmorillonite and vermiculite after 10 days of equilibration

<table>
<thead>
<tr>
<th>Particle size (μm)</th>
<th>pH 1 min</th>
<th>pH 10 days</th>
<th>CEC (mg Fe+/me of blocked CEC)</th>
<th>X-ray Pretreatments</th>
<th>Basal spacings (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>10 days</td>
<td>mg Fe* (%)</td>
<td>Ca 105°C</td>
<td>K 300°C</td>
</tr>
<tr>
<td>&lt;0.2</td>
<td>4.1</td>
<td>3.6</td>
<td>39.3</td>
<td>69.2</td>
<td>62.6</td>
</tr>
<tr>
<td>2-0.2</td>
<td>3.4</td>
<td>3.2</td>
<td>45.6</td>
<td>62.4</td>
<td>49.6</td>
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<tr>
<td>2-0.2</td>
<td>4.1</td>
<td>4.3</td>
<td>118.7</td>
<td>20.3</td>
<td>25.8</td>
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<tr>
<td>50-2</td>
<td>3.4</td>
<td>3.4</td>
<td>128.9</td>
<td>27.3</td>
<td>11.9</td>
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<tr>
<td>&lt;0.2</td>
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<td>3.6</td>
<td>39.6</td>
<td>68.9</td>
<td>85.7</td>
</tr>
<tr>
<td>2-0.2</td>
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<td>45.6</td>
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<tr>
<td>2-0.2</td>
<td>4.0</td>
<td>3.6</td>
<td>100.2</td>
<td>32.8</td>
<td>36.4</td>
</tr>
<tr>
<td>50-2</td>
<td>3.6</td>
<td>3.4</td>
<td>100.8</td>
<td>43.3</td>
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<td>Vermiculite</td>
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<td>Montmorillonite</td>
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<tr>
<td>2-0.2</td>
<td>4.0</td>
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<td>50-2</td>
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<td>3.4</td>
<td>100.8</td>
<td>43.3</td>
<td>11.0</td>
</tr>
</tbody>
</table>

*Initially, 148.9 mg of iron (III) per 0.5 g of mineral were added.
†b stands for broad.

minerals. Montmorillonite has a lower CEC than vermiculite (Table 1). Further, montmorillonite has a substitution of magnesium for aluminum in the octahedral layer whereas vermiculite has a substitution of aluminum for silicon in the tetrahedral layer. According to Coulomb's law, the interlayer cation or hydroxy polymers will be held almost four times more strongly by the tetrahedral charge than by the octahedral charge. Hsu and Bates (1964) and Schwertmann and Jackson (1964) reported that the OH-Al polymers were more tightly held by vermiculite than by montmorillonite. However, (Carstea, 1967) found that hydroxy-Al, -Fe and -Mg interlayers in montmorillonite aged for 6 months were more stable to sequential treatments than those in vermiculite. These findings are consistent with those reported by Tamura (1957, 1958) and show that other factors, in addition to charge density, play an important role with respect to hydroxy interlayer formation and stability.

It is expected that vermiculite would have a smaller interlayer space than montmorillonite at the time of hydroxy interlayer preparation because the charge density of vermiculite is higher. The strength of the electrical field would influence, among other physico-chemical phenomena, the diffusion rate of cations, degree of hydrolysis of adsorbed cations, polymerization of aluminohydronium, and the repulsion of anions. Thus, vermiculite is expected to have greater effectiveness in decreasing the hydrolysis of adsorbed Al\(^{3+}\) and Fe\(^{3+}\), lower rate of polymerization and greater repulsion for hydroxyls than montmorillonite. The results of this study seem to be consistent with the above theoretical considerations.

The iron interlayers were slightly less effective in reducing the CEC than the aluminum interlayers. The amount of fixed aluminum and iron per 1 me. of blocked CEC varied widely. In aluminum systems, this amount was between 62 and 114 mg of aluminum, whereas in iron systems it varied from 42 to 290 mg. These values are considerably higher than those reported by Shen and Rich (1962) who found a decrease in CEC of 1 me/24.6 mg of aluminum when synthetic interlayers were formed in montmorillonite. However, the values of this study are comparable with those reported by Singleton (1966) who found values as high as 135 mg of aluminum/1 me increase in CEC after removal of interlayer from B and C horizons of Wren soil from Oregon.

**Particle size**

The CEC values of montmorillonite and chlorite increased as the particle size decreased (Table 1). This is to be expected because the edge surface and a proportional number of broken bonds increased with a decrease in particle size (Jackson, 1956). In contrast, silt vermiculite showed higher CEC.
than coarse clay. This behavior can be partially explained by a physical deformation of vermiculite crystals, particularly at the edges, as a result of grinding. The deformation presumably prevented the exchangeable cations to reach the interlayer sites. In addition, the amorphous components formed during grinding presumably concentrated in finer size fractions and acted as a diluent for CEC. The degree of the erratic closure and the amount of amorphous components increased as the particle size decreased. These explanations seem to be consistent with those reported by Mackenzie and Milne (1953).

The diffusion of ions is directly related to the particle size. At comparable charge densities, the time required for cations or polymers to reach the middle of the interlayer space will increase with increasing particle size. Data of the present study indicated that more hydroxy-Al and -Fe interlayers formed in fine clay than in coarse clay montmorillonite at both temperature levels.

Unlike montmorillonite, a definite pattern was not observed for vermiculite systems. The present data would indicate that the formation of hydroxy interlayers in vermiculite was a net result of an interaction between kind of cations, charge density, temperature, and particle size with none displaying a dominant role. At 21°C, equal amounts of hydroxy-Al interlayers were formed in both size factions. In contrast, more hydroxy-Fe interlayer formed in silt faction than coarse clay faction at 21°C in spite of a relatively low amount of iron adsorbed. The low adsorption of iron by silt vermiculite may be due to low pH values (Carstea, 1967). Nevertheless, the iron was quite effective in forming hydroxy interlayers, and the iron polymers were preferentially adsorbed in the interlayer position. The difference in the degree of coating between coarse clay and silt vermiculite was very apparent from the visual inspection of reacting bottles.

**Temperature**

The temperature levels, namely 3°C and 21°C, were selected to approach those reported for soil temperature classes in the American Soil Classification (1960) and the January and July mean temperatures observed for certain regions of North American continent. Thus, it was hoped that some information may be obtained with regard to formation of chloritic intergrades in soils as a function of seasonal thermal variations.

It is generally agreed that heat and pressure were important during formation of chlorite which is widely distributed in low grade metamorphic rocks. Likewise, the formation of chlorite from ferromagnesian minerals in igneous rocks is related to the hydrothermal alteration. In contrast, the processes leading to the formation of chloritic intergrade seem to be confined to "open" environments which are characteristic for soils (Jackson, 1963).

In the present study, the formation of aluminum interlayers in both minerals increased with increasing temperature. Except for silt vermiculite, the formation of hydroxy-Fe interlayers was generally not temperature dependent. The hydrolysis and polymerization of 

\[ \text{AlCl}_3 \] and \[ \text{FeCl}_3 \] solutions increased as the temperature increased (Rich, 1960; Thomas and Coleman, 1964). Data reported have shown that the thermal variations in natural conditions influence the degree of interlayering in expanding clay minerals.

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FORMATION OF HYDROXY-Al AND -Fe INTERLAYERS


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Résumen-Le couches intermédiaires d’oxy-Al et -Fe étaient préparées dans des montmorillonites “monominerale” d’argile fine et grosse et de vermiculite d’argile grosse et de limon à 3°C et à 21°C. La formation de couches intermédiaires d’hydroxyde a été évaluée par diffractions de rayons X, par la capacité d’échange de cations, et par des analyses chimiques.

Pour des dimensions particulières comparables et indépendamment de la température, les quantités de couches intermédiaires d’oxy-Al et -Fe étaient nettement plus importantes dans la montmorillonite que dans la vermiculite. De même, les systèmes d’aluminium avaient un degré de couches intermédiaires plus élevé que ceux de fer.

Pour la montmorillonite, la quantité de couches intermédiaires d’oxy-Al et -Fe augmentait à mesure que la taille de la particule diminuait, indépendamment de la température. La montmorillonite à couches intermédiaires d’aluminium en équilibre à 3°C se caractérisait par des écartements de base d’environ 17 Å après saturation de Ca avec une humidité relative de 54%. Ces écartements sont plus gros que ceux que l’on note en général pour les smectites.

Pour les systèmes de vermiculite en équilibre à 3°C, on a noté plus de couches intermédiaires d’oxy-Al dans l’argile grosse que dans la fraction de limon, alors qu’à 21°C les couches intermédiaires formées étaient à peu près équivalentes. La couche intermédiaire d’oxy-Fe, par contre, était favorisée par la fraction de limon aux deux niveaux de température.

La formation de couches intermédiaires d’aluminium pour les deux minéraux augmentait avec l’augmentation de la température. Si la formation de couches intermédiaires d’oxy-Fe dans la montmorillonite ne dépendait pas de la température, celle de telles couches dans la vermiculite augmentait légèrement avec l’augmentation de la température.

Cas données peuvent aider à expliquer la formation de degrés intermédiaires de chlorite dans des sols en fonction du type de minéraux d’ions et de variations thermiques.

Bei vergleichbarer Teilchengrösse und unabhängig von der Temperatur überwogen die Hydroxy-AI und -Fe Zwischenschichten in Montmorillonit mengenmässig bei weitem die im Vermiculit gebildeten Zwischenschichten. Ferner wiesen das Aluminum-System ein höheres Mass an Zwischenschichtung auf als das Eisen-Systeme.

Innerhalb des Montmorillonits nahm die Menge der Hydroxy-Al und -Fe Zwischenschichten mit abnehmender Teilchengrösse zu, und zwar unabhängig von der Temperatur. Das bei 3°C im Gleichgewicht befindliche Montmorillonit mit Aluminum-Zwischenschichten war gekennzeichnet durch basische Abstände von ca. 17 Å nach Ca-Sättigung plus 54% relative Feuchtigkeit. Diese Abstände sind grösser als die normalerweise in Smekrite beobachteten.

Innerhalb der bei 3°C im Gleichgewicht befindlichen Vermiculit-Systeme wurden im groben Ton mehr Hydroxy-Al Zwischenschichten festgestellt als in der Schlickfraktion, während beim 21°C ungefähr gleiche Mengen von Zwischenschichten gebildet wurden. Im Gegensatz dazu wurden mehr Hydroxy-Fe Zwischenschichten gebildet. Im Gegensatz dazu wurden mehr Hydroxy-Fe Zwischenschichten an beiden Temperaturhöhen in der Schlickfraktion festgestellt.

Die Bildung von Aluminum-Zwischenschichten in beiden Mineralen nahm mit steigender Temperatur zu. Die Bildung von Hydroxy-Fe Zwischenschichten im Montmorillonit war im allgemeinen nicht temperaturabhängig, während die Bildung solcher Zwischenschichten im Vermiculit mit zunehmender Temperatur leicht anstieg.

Diese Ergebnisse könnten eine teilweise Erklärung für die Bildung von chloritischen Zwischenstufen in den Böden als eine Funktion der Art des Minerals, der Ionenart und thermischer Schwankungen darstellen.

Резюме—Прослоёки гидроксі-Al і -Fe були приготовлені в "мономінеральному" мелком і грубом глиністом монтморіллоніті, а також в грубій глини і залишкові вермікуліти при 3°C і 21°C. Образування прослоєк гидроксі оцінювалось, позаявляючи дифракційні рентгеновських ліній, катионобмінніюючою спроміжністю і хімічними аналізами.

При щільності часцісти і незалежному від температури, кількість промежуточних шарів гидроксіалюміній і железа в монтморіллоніті в значній ступені превищило кількість таких шарів, образованихся в вермікуліті. Східним образом системи алюмінії показали більшу кількістю промежуточних шарів, і шар системи железа.

В монтморіллоніті кількість прослоєк гидроксі-Al і -Fe зростала в межах умовного розміру частці, незалежно від температури. Монтморіллоніт з алюмінійними прослоєками, который уравновешивался при 3°C, отличался базальным расположением прибл. в 17 Å после насыщения кальцием плюс 54 % относительно влажности. Расстояния эти большие тех, которые обычно встречаются для сукумовых глин.

В системах вермікуліті, уравновешенных при 3°C, замечено было большее количество прослоек гидрокси-Al в грубої глине, чем в залишковій фракції, причем при 21°C образовывались почти равные количества промежуточных слоев. Как контраст, промежуточный слой гидрокси-Fe был более приемлемым для залишковій фракції на обоих уровнях температуры.

Образование алюмінійних прослоек в обоих минералах увеличилось по мере повышения температуры. Образование прослоек гидрокси-Fe в монтморіллоніті не было в обшим зависимым от температуры, в то время как образование таких промежуточных слоев в вермікуліті несколько повысилось при росте температуры.

Данные эти могут в некоторой степени объяснить образование хлоритных промежуточных слоев в грунтах как функции типа минералов, рода іонов і термічних змінінь.