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SYNTHESIS OF MIXED-LAYER KAOLINITE/SMECTITE

JAN ŚRODOŃ

Institute of Geology, Polish Academy of Sciences
31-002 Kraków, Senacka 3, Poland

Abstract—Mixed-layer kaolinite/smectites containing 40–90% kaolinite layers have been synthesized from Wyoming smectite. Run times were 4 months at 156°C in solutions of $AlCl_3 \cdot 6H_2O$ and KCl or $CaCl_2$. Similar KCl runs devoid of Al^{3+} yielded mixed-layer illite/smectites. The supply of Al^{3+}, rather than pK or pH, seems to control the alteration of smectite toward kaolinite or illite.

Key Words—Interstratification, Kaolinite, Mixed layer, Montmorillonite, Smectite, Synthesis.

INTRODUCTION

The existence of mixed-layer kaolinite/smectites in nature was first suggested by Sudo and Hayashi (1956) and Altschuler *et al.* (1963). In a sense, these minerals were rediscovered in the Tertiary clays of Yucatan by Schultz *et al.* (1971) and in hydrothermal deposits of Lower Silesia by Wiewióra (1971, 1973). Since that time, more than 10 additional occurrences of kaolinite/smectites have been described, proving that these minerals are common components of many soils, weathering crusts, and hydrothermally altered rocks.

In most of the published cases it can be shown that kaolinite/smectite forms at the expense of smectite (Altschuler *et al.*, 1963; Thiry, 1973; Drits and Sakharov, 1976). A study was undertaken to simulate the alteration of smectite towards kaolinite in the laboratory, and preliminary results are reported in this paper.

EXPERIMENTAL

Teflon reaction vessels, sealed with Teflon screw-type tops and reinforced with hose clamps, were boiled as autoclaves for four months in a mixture of ethylene glycol and water at 156°C. In each run, 125 mg of powdered, <1-μm, oven-dried Wyoming smectite (separated from The Clay Mineral Society Source Clay SWy-1) and a variable amount of solid $AlCl_3 \cdot 6H_2O$ were mixed with 25 ml of 0.5 N $CaCl_2$ or KCl. The smectite was saturated with either Ca or K to duplicate the solution composition. Four blank runs (devoid of Al in solution) were also made, two of them containing clay + KCl or $CaCl_2$ and the other two acidified additionally with HCl. The pH of the final solution was measured after cooling the vessel to room temperature. The solids were then cleaned in a centrifuge by several washings with distilled water, and the supernatant liquid was analyzed for Mg and Fe by atomic absorption, and for Si colorimetrically. Chemical data on all the runs are given in Table 1. The final volume of the solution poured from the Teflon vessel after the first centrifugation is also shown in the table. It is substantially lower than the starting volume, proving that some leaking took place from all vessels. In one case (Blank II/K), a wet clay without any solution was found after opening the vessel.

The solid products were analyzed by infrared spectroscopy (IR) in KBr discs (Figure 1), and by X-ray powder diffraction (XRD) (Ni-filtered CuKα radiation) from ethylene glycol-solvated, oriented, glass-slide preparations (Figure 2). The method of the ethylene glycol solvation was that described by Środoń (1980).

RESULTS

Solid products

IR is a convenient method for detecting small admixtures of kaolinite in a smectite sample, because the sharp kaolinite band at 3690 cm^{-1} does not coincide with bands of smectite or of any other clay mineral. Figure 1 shows that Ca-runs with 0.4, 2, and 15 Al^{3+} ions per half-formula weight of smectite $[O_{10}(OH)_2]$ produced variable amounts of kaolinite, whereas the run with 50 Al and the blank runs did not produce kaolinite. The latter spectra resemble those of the starting material, but they are weaker and more diffuse. The KCl set varied analogously. As judged from the relative intensities of kaolinite and smectite bands, the products can be classified with respect to the amount of kaolinite formed:

$$15Al/Ca > 2Al/Ca > 0.4Al/Ca > 15Al/K > 2Al/K > 0.4Al/K$$

XRD data confirm this interpretation. The blank runs, when analyzed by a precise identification method which takes into account the variable thickness of the smectite-glycol complex (Środoń, 1980), prove to be pure smectites for the Ca-runs, and mixed-layer illite/smectites with about 15–20% illite layers for the K-runs. The 50-Al runs gave very poor, diffuse patterns, unsuitable for precise measurements. The approximate peak positions and peak intensities suggest, however, that the phase present is close to a pure smectite in the 50Al/Ca run, and highly expandable illite-smectite in 50Al/K run.

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Table 1. Chemistry and volume of solutions before and after 120 days at 156°C and at autoclave pressure.

Run	Starting composition $AlCl_3 \cdot 6H_2O$ (mg)	pH	Final composition pH	SiO_2 (mg)	Fe_2O_3 (mg)	MgO (mg)	Volume (ml)
0.5 N $CaCl_2$ starting solution, 25 ml							
Blank I	0	6.2	4.45	1.9	0.28	0.50	12
Blank II	0	2.8	3.96	3.8	0.23	0.50	16
0.4Al/Ca	34	3.7	1.93	5.6	1.85	1.50	14
2Al/Ca	170	3.5	1.62	4.4	2.55	2.00	16
15Al/Ca	1275	2.7	1.45	3.7	2.70	2.00	19
50Al/Ca	4250	2.0	2.10	5.5	1.40	1.00	17
0.5 N KCl starting solution, 25 ml							
Blank I	0	6.1	4.56	4.1	0.03	0.75	17
Blank II	0	2.6	—	—	—	—	—
0.4Al/K	34	3.9	2.40	3.4	1.25	1.05	12
2Al/K	170	3.6	2.16	2.6	1.40	1.10	18
15Al/K	1275	2.9	2.06	3.8	1.55	1.35	20
50Al/K	4250	2.3	2.44	6.2	1.40	0.90	18

XRD patterns of the 0.4-, 2-, and 15-Al products (Figure 2) show an increase in kaolinite content in the same sequence as that found from the IR data. With increasing kaolinite layers, the mixed-layer reflections evolving from the 002 and 005 smectite reflections become stronger, relatively to the reflections evolving from the 003 and 004 of smectite, and migrate toward the positions of the 001 and 002 reflections of kaolinite. Additionally, a small reflection migrates from the 003 of smectite towards the 001 of kaolinite. The 001 reflection of smectite becomes broader and weaker, but does not migrate. Weak reflections persist at positions corresponding to the (003) and (004) planes of smectite,

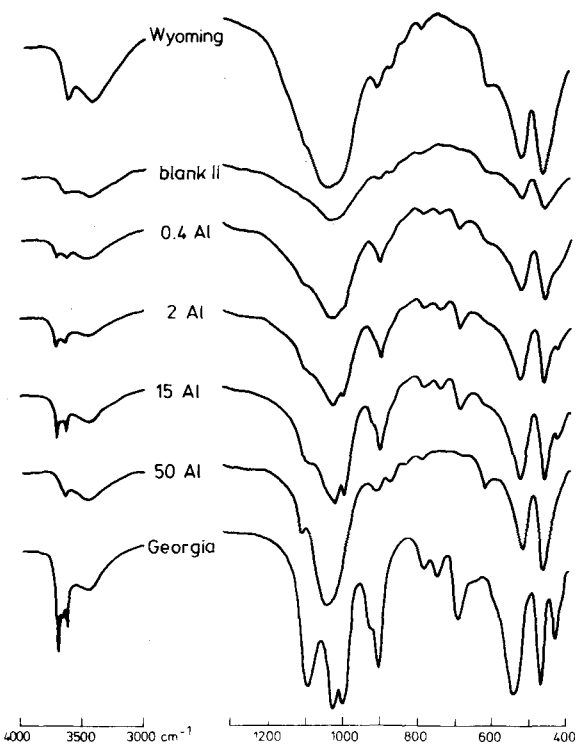


Figure 1. IR spectra of the starting Wyoming smectite (The Clay Mineral Society Source Clay SWy-1) and solid products of hydrothermal runs in 0.5 N $CaCl_2$ with variable amounts of $AlCl_3 \cdot 6H_2O$. Concentration of Al^{3+} expressed as number of ions per formula weight of smectite based on $O_{10}(OH)_2$. Blank runs are those without $AlCl_3 \cdot 6H_2O$ added. Spectrum of Georgia kaolinite (The Clay Mineral Society Source Clay KGa-2) is included for comparison.

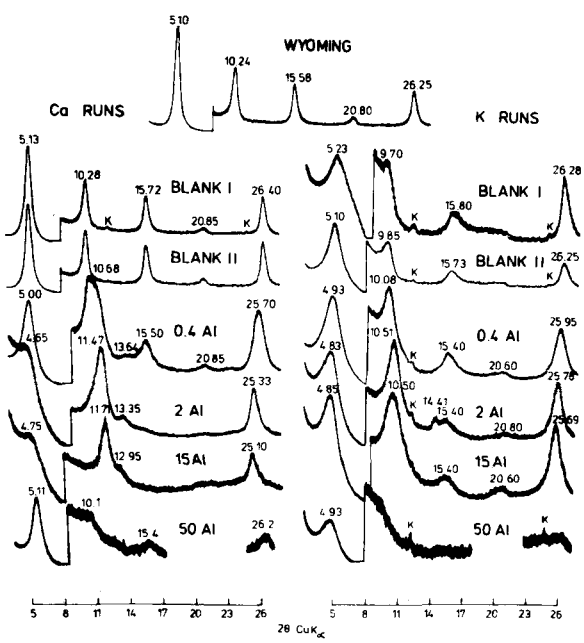


Figure 2. X-ray powder diffraction patterns (2θ, CuKα, ethylene glycol) of the starting material and run products. K = traces of discrete kaolinite. Other symbols as in Figure 1. Blank/Ca and 50Al/Ca runs are pure smectites; Blank/K and 50Al/K runs are illite/smectites; and 0.4-, 2-, and 15-Al runs are mixed-layer kaolinite/smectites + unreacted smectite.

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except for the two most kaolinitic samples 2Al/Ca and 15Al/Ca.

The above XRD data are interpreted as resulting from a mixture of interstratified kaolinite/smectite, with varying layer ratios, and a small amount of unreacted smectite. Mixed-layer kaolinite/smectite is responsible for the weak reflections between 14.41° and 12.95°2θ, gives most of the intensity to the reflections between 10.08–11.71° and 25.95–25.10°2θ, and contributes to some extent to the reflection between 4.65° and 5.00°2θ. Discrete smectite gives the 15.40–15.50°2θ and 20.60–20.85°2θ reflections, most of the 4.65–5.00°2θ reflection, and contributes some intensity to the reflections at 10.08–11.71° and 25.95–25.10°2θ. The presence of discrete smectite explains why the reflections at 10.68°2θ (run 0.4Al/Ca) and 11.47°2θ (run 2Al/Ca) are asymmetrical.

The smectite 003 and 004 reflections in run 0.4Al/Ca are displaced toward higher angles as compared with K-runs. This shift results from a thinner ethylene glycol complex for Ca-smectites than for K-smectites (Środoń, 1980).

Surprisingly low-angle position of the mixed-layer reflection at 10.08°2θ in run 0.4Al/K can be partially accounted for in the same way. Additionally, the mixed-layer kaolinite/smectites formed in KCl solutions may contain a small percentage of illite layers, shifting toward low angles the position of the reflection in question. K-exchange of SWy-1 smectite in room conditions produces about 5% of interstratified illite layers (Środoń, 1980).

An approximate estimation of kaolinite:smectite ratios in the mixed-layer minerals can be made by assuming a homogenous random model of two-component interstratification and using the reflection around 25–26°2θ for determination. The position of this reflection is much less influenced by domain thickness than that of the strong reflection around 11–12°2θ (Reynolds and Hower, 1970).

A computer program, KAOL, developed by R. C. Reynolds (Dartmouth College, Hanover, New Hampshire), was used to produce theoretical XRD patterns of mixed-layer kaolinite/smectites which were used to estimate the kaolinite content of products (Table 2). The calculated patterns for clays which contain more than 50% kaolinite layers do not have a reflection around 5°2θ, whereas such a reflection is present in the experimental patterns. The presence of this reflection suggests a phase with a lower kaolinite content than that estimated from the position of the reflection at 25–26°2θ. As mentioned above, this problem in identification is caused by the presence of unreacted smectite which persists even in the most kaolinitic products (2Al/Ca and 15Al/Ca). XRD patterns of the 0.4Al/K and 2Al/K products show the presence of traces of discrete kaolinite in addition to discrete smectite.

Table 2. Weight-amounts of smectite dissolved from the original 125 mg and of kaolinite crystallized.[1]

	Smectite from		Kaolinite from		Kaolinite from	
Run	Fe_2O_3 (mg)	MgO (mg)	Fe_2O_3 (mg)	MgO (mg)	Chemistry (%)	XRD (%)
$CaCl_2$						
0.4Al/Ca	40	40	39	40	40	40–50
2Al/Ca	55	53	61	59	55	85
15Al/Ca	58	53	67	61	57	>90
KCl						
0.4Al/K	27	28	27	29	29	<40
2Al/K	30	29	33	32	33	<40
15Al/K	33	36	35	38	37	40–50

[1] Calculated from the final solution composition and expressed also as the volume percent of kaolinite in the product for comparison with XRD estimation. Details of the calculations in the text.

Solution chemistry

The pH of the final solutions follows similar patterns in both sets of runs (Table 1). In the blank runs, the final pH is 4–4.5, due to an increase in the initially low-pH runs and a decrease in the high-pH runs. In the 50-Al runs, the pH is almost unchanged. In all kaolinite-producing runs pH drops significantly, falling in proportion to the amount of kaolinite formed. This pattern is obeyed also by the Fe and Mg contents whereas the Si content is irregular (Table 1).

These data, combined with the mineralogical data, suggest the following reaction mechanism:

1. Both tetrahedral and octahedral sheets of smectite dissolve; $(Al,Fe,Mg)_2Si_4O_{10}(OH)_2 + 4H_2O + 6H^+ \rightarrow 2(Al,Fe,Mg)^{3+} + 4H_4SiO_4$.
2. Amorphous silicic gel does not precipitate; if present, a non-coincident 1220 cm^{-1} band would have shown on the IR spectra (van der Marel and Beutelspacher, 1976). H_4SiO_4 then reacts with Al^{3+} to form kaolinite; $2H_4SiO_4 + 2Al^{3+} + H_2O \rightarrow Al_2Si_2O_5(OH)_4 + 6H^+$.
3. The net reaction is; $(Al,Fe,Mg)_2Si_4O_{10}(OH)_2 + mAl^{3+} + 6H_2O \rightarrow 2Al_2Si_2O_5(OH)_4 + 6H^+ + aFe^{3+} + bMg^{2+}$ where $m = 2 + a + b$.

This reaction results in the lowering of pH, as was observed during the experiments.

If this reaction describes closely the process actually taking place, Fe and Mg contents of the starting material and of the final solution (Table 1) can be used to calculate the amounts of dissolved smectite. Both calculations should give close results, and the amount of crystallized kaolinite can be calculated from the amount of silica released by dissolution of smectite minus the amount of silica in solution.

The chemical composition of the starting material

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(<1-μm fraction of Wyoming smectite SWy-1) was calculated from the chemical analysis of the bulk sample (van Olphen and Fripiat, 1979) by subtracting 12% quartz (XRD estimation by the author), 3% calcite from the CO_2 percentage (2% according to XRD measurement), and 3.13% K-feldspar from the K_2O percentage (K-feldspar detected by XRD). It was shown (Środoń, 1980) that the SWy-1 sample contains 100% expandable smectite, so all the potassium should be allocated to the feldspar. The <1-μm fraction contains pure smectite, as checked by XRD.

The composition and formula of the SWy-1 smectite obtained from the above data are:

60.1% SiO_2, 23.4% Al_2O_3, 0.11% TiO_2, 4.14% Fe_2O_3, 0.39% FeO, 3.75% MgO, 1.88% Na_2O, 6.27% H_2O.

$$Na_{0.23}Mg_{0.11}(Al_{1.54}Fe^{3+}_{0.20}Fe^{2+}_{0.02}Mg_{0.24})(Si_{3.80}Al_{0.20}O_{10})(OH)_2.$$

It is believed that the above data characterize the starting material close enough for the purpose of the outlined calculations. It is also assumed that no significant amount of the dissolved matter has been lost from the vessels. This assumption is legitimate because the major solution loss, evidenced by a drop in the boiling point of the water–ethylene glycol mixture, took place at the very beginning of the experiment.

The calculations using Fe and Mg contents gave very similar results (Table 2), thereby supporting the proposed mechanism of the congruent dissolution of smectite. To compare this estimation with that from XRD data, the amount of kaolinite formed must be expressed as percentage of layers (volume %), instead of weight-amount:

$$\%\ K = \frac{K/FW_k}{(125\ mg - S)/FW_s + K/FW_k} \times 100 = \frac{\text{kaolinite layers crystallized}}{\text{smectite layers remaining} + \text{kaolinite layers crystallized}}$$

K and S are weight-amounts of crystallized kaolinite and dissolved smectite, and FW_k and FW_s are the formula weights of the unit cells of these minerals, respectively (FW_k = 516; FW_s = 744). Average values from Fe- and Mg-based calculations were used for K and S.

The percentages of kaolinite calculated in this way are systematically lower than those obtained from the XRD data (Table 2). This is consistent with the XRD-based interpretation of the mineral composition of the products (a mixture of mixed-layer kaolinite/smectite and discrete smectite). The chemical data give the percentage of kaolinite in the whole mixture, whereas the XRD data give the percentage of kaolinite in the mixed-layer mineral.

DISCUSSION AND CONCLUSIONS

At least three attempts have been made to synthesize kaolinitic material starting from a smectite (Poncelet and Brindley, 1967; Kittrick, 1970; Oberlin and Couty, 1970). In all cases the products were reported as kaolinite. Poncelet and Brindley, and Oberlin and Couty worked with Al-OH-smectites at 175–240°C in a pH range of 1–6. Their experiments lasted from one week to one month. Poncelet and Brindley (1967) reported a mineral with d(001) = 8.8–8.1 Å, when treated with ethylene glycol, which evolved into a 7.2 Å phase, whereas the final product of Oberlin and Couty (1970) had spacings of 7.44 and 3.54 Å. These data, as well as IR spectra published in the former paper, suggest that the intermediate product of Poncelet and Brindley and the final product of Oberlin and Couty were mixed-layer kaolinite/smectites instead of a "chlorite-like phase" or "poorly crystallized kaolinite" as suggested by the authors. If the product was halloysite, it should have collapsed to 7.2 Å on heating to 200–300°C (Poncelet and Brindley, 1967), and it should have d(002) > 3.58 Å (Oberlin and Couty, 1970).

Kittrick (1970) treated smectites for 3–4 years at 25°C in solutions supersaturated with respect to kaolinite by additions of HCl, $AlCl_3$, and $Na_2SiO_3 \cdot 9H_2O$. His XRD data leave no doubt that pure kaolinite was crystallized directly, without a mixed-layer intermediate stage.

Thus, experimental evidence exists for two possible paths of smectite alteration toward kaolinite: direct or through a mixed-layer stage. Both paths are followed in nature. Examples of the mixed-layer type of alteration were cited in the introduction. A case of direct alteration was well documented by Morgan *et al.* (1979). Further experimentation is needed to discover which factors control the reaction path.

Previously, the transformation to a mixed-layer through the removal of one tetrahedral sheet from a smectite layer was advocated by Altschuler *et al.* (1963). Drits and Sakharov (1976) suggested simultaneous crystallization of both types of layers due to oscillatory changes of chemical parameters in the microenvironment surrounding the crystallizing particle. The data presented in this paper cannot be explained by the mechanism proposed by Altschuler *et al.* (1963); an excess of amorphous silica should be present if one of the tetrahedral sheets was removed. On the other hand, the mixtures of mixed-layer kaolinite/smectite and discrete smectite in the run products do not necessarily imply the crystallization of mixed-layer kaolinite/smectite from solution. Some smectite might have been left untouched either due to slow diffusion (the pressure vessels were not shaken during the experiment), or because of some characteristic of its interlayer region. The mixed-layer mineral might have formed through the dissolution of some smectite crystallites and subsequent crystallization of kaolinite in the inter-

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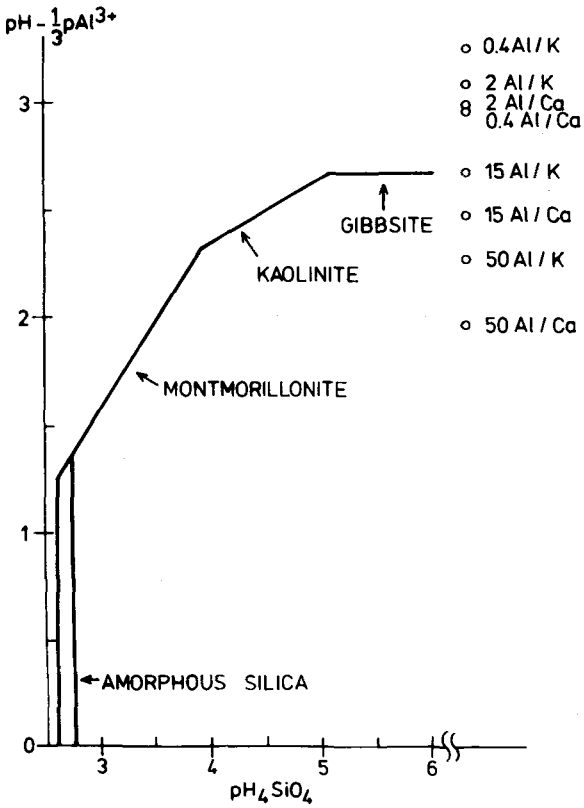


Figure 3. Starting solution compositions plotted on the stability diagram of Kittrick (1971). pAl^{3+} and pH_4SiO_4 are negative logarithms of activities. Starting compositions differ only along the vertical axis because they were initially silica-free. These compositions move to the left as dissolved silica increases during the runs.

layer space of other smectite crystallites. If this last mechanism is correct, the two paths of alteration of smectite toward kaolinite discussed above would differ from each other by the site of nucleation of kaolinite layers: whether inside or outside of the smectite crystallite.

The question of why the 50-Al runs did not yield kaolinite can probably be answered by plotting the data from Table 1 on Kittrick's stability diagram (1971), although this diagram is calculated for 25°C and 1 bar (Figure 3). The starting solution compositions differ only in pH $-$ ⅓ pAl^{3+} (no silica was introduced into the solution). During the course of the experiment, pH_4SiO_4 should decrease due to the dissolution of smectite and amorphous silica, which is known to be present in Wyoming bentonite (Mering, cited by Oberlin and Couty, 1970). This aqueous silica would bring the solution composition into the montmorillonite and kaolinite stability fields. pH $-$ ⅓ pAl^{3+} values were lowest for the 50-Al compositions, perhaps low enough to stabilize smectite.

Increasing the amount of $AlCl_3 \cdot 6H_2O$ relative to smectite increased the amount of kaolinite layers that formed during treatment. This is analogous to the formation of mixed-layer pyrophyllite/smectites from the same starting materials at higher temperatures (Eberl, 1979). The type of alkali or alkaline earth cation present also seems to influence the rate of alteration of smectite towards kaolinite; much more kaolinite was formed in Ca-runs than in the corresponding K-runs.

A comparison of Blank/K- and Al/K-runs gives information on the genetic relationship between mixed-layer kaolinite/smectites and illite/smectites. Both minerals form abundantly in nature at the expense of smectite. In the experiments, the minerals were produced from the same smectite, at the same temperature and in solutions of similar pK/pH ratio (compare Blank II/K and run 15Al/K in Table 1). The sole difference was that Al^{3+} was present in the kaolinite-producing runs. In terms of the stability diagram found in Helgeson *et al.* (1969), the Blank I/K run had a starting solution composition which plots in the field of microcline, and the Blank II/K had a composition which plots in the field of kaolinite. In both runs similar mixed-layer illite/smectites were formed. Evidently, pAl, and not pK/pH, determines whether kaolinite/smectite or illite/smectite will form from smectite, but further experiments are needed to clarify this problem.

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Резюме—Смешанно-слойные каолиниты/смектиты, содержающие 40–90% каолинитовых слоев, были синтезированы из смектитов Вайоминга. Процесс продолжался 4 месяца при 156°C в растворах $AlCl_3 \cdot 6H_2O$ и KCl или $CaCl_2$. Подобные процессы с KCl но без Al^{3+} приводили к формированию смешано-слойных иллит/смектитов. Поступление Al^{3+}, а не pK или pH, по-видимому, контролирует превращение смектита в каолинит или иллит. [N.R.]

Resümee—Aus dem Smektit von Wyoming wurden Kaolinit/Smektit-Wechsellagerungen mit 40–90% Kaolinitlagen synthetisiert. Die Versuchszeiten betrugen 4 Monate bei 156°C mit Lösungen aus $AlCl_3 \cdot 6H_2O$ mit KCl oder $CaCl_2$. Ähnliche KCl-Versuche ohne Al^{3+} lieferten Illit/Smektit-Wechsellagerungen. Das Angebot an Al^{3+} scheint die Umwandlung von Smektit in Kaolinit oder Illit in stärkerem Maß zu kontrollieren als pK oder pH. [U.W.]

Résumé—Des smectites/kaolinite à couches melangées contenant de 40–90% de couches de kaolinite ont été synthétisées à partir de smectite du Wyoming. Les périodes étaient de 4 mois à 156°C dans des solutions d'$AlCl_3 \cdot 6H_2O$ et de KCl ou $CaCl_2$. Des séries de KCl semblables sans Al^{3+} ont fourni des smectites/illite à couches melangées. La provision d'Al^{3+}, plutôt que le pK ou le pH semble contrôler l'altération de la smectite vers une kaolinite ou une illite. [D.J.]

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