

RELATION OF POTASSIUM EXCHANGE AND FIXATION TO DEGREE OF WEATHERING AND ORGANIC MATTER CONTENT IN MICACEOUS CLAYS OF PODZOL SOILS*

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(Received 2 November 1970)

Abstract—The rates of K exchange of untreated and peroxide-treated micaceous clays from five Podzol soils of increasing degrees of weathering were determined using sodium tetraphenyl boron. In addition, the amounts of K fixed against NH_4 were measured. The Ae horizon clays of these soils contained mainly interstratified mica-vermiculite-montmorillonite whereas the C horizon clays contained mica of a more discrete nature.

As was expected, there was an inverse relationship between rates of K exchange and degrees of weathering of the Ae horizon clays. However, in the samples from each of these soils except in those of the most weathered one, the K exchange rate of the interstratified mica from the Ae horizon was higher than that of the less weathered, more discrete mica from the corresponding C horizon. The abundance of hydrated edges and layers exposing K exchange sites in the Ae horizon micas probably contributed to their higher rates of K exchange. Amounts of K fixed in the Ae horizon clays were not related to degrees of weathering.

Removal of organic matter from the Ae horizon clays by peroxide considerably increased both the rates of K exchanged and the amounts of K fixed. These increases were attributed to the elimination of a blocking effect of adsorbed organic matter on K exchange and fixation.

INTRODUCTION

STUDIES IN the artificial weathering of micas show that their K may be exchanged with hydrated cations and that this results in a swelling mineral resembling vermiculite. During this process the rate of K exchange decreases as the front of the exchanged region approaches the center of the particle. Diffusion appears to be the rate-limiting step in this reaction (Mortland and Ellis, 1959; Rausell-Colom *et al.*, 1965; Reed and Scott, 1962).

The natural weathering process apparently is not this uniform. Micas in soils generally become interstratified to varying degrees of randomness with hydrated layers. Also some of the hydrated layers may show montmorillonite-like characteristics (Kodama and Brydon, 1968; Lisitsa and Tikhonov, 1969; Sokolova and Shostak, 1969).

However, considering the results of the studies cited thus far, it seems reasonable to assume that it becomes more difficult to exchange K from mica in soil as weathering progresses. The validity of this assumption might be tested by comparing K exchange rates of micaceous clays obtained from

soils of similar genesis but in advancing stages of weathering.

Another aspect that may be studied by using these micaceous soil clays is the effect on K exchange of material adsorbed on clay surfaces. Adsorption of hydrous aluminum oxide on outer and interlayer surfaces of weathered micaceous clays is well substantiated and has been shown to inhibit K release and fixation to varying degrees (Rich, 1960a; Rich, 1960b; Rich and Obenshain, 1955). Laboratory experiments have demonstrated also adsorption of soil humic compounds on outer and interlayer surfaces of expanding clays (Greenland, 1956; Kodama and Schnitzer, 1969; Schnitzer and Kodama, 1966). This process seems to occur at least in some soils as indicated by the presence in these soils of clay-organic complexes in which charge sites are blocked by organic matter (Dudas and Pawluk, 1970; Greenland, 1965; Syers *et al.*, 1970). However, notwithstanding this evidence and the evidence obtained by Mortland (1961) who showed that certain organic compounds greatly inhibited the adsorption of K on vermiculite, the effect of organic matter on K exchange and fixation in soil micaceous clays has not been specifically studied.

In view of these considerations, this investiga-

*Contribution No. 352.

tion was aimed at clarifying the effect of degree of weathering and of organic matter content on K exchange and fixation. Hydrated micaceous Podzol clays were used which varied chiefly in degree of weathering and organic matter content and which contained very small amounts of free aluminum oxide.

EXPERIMENTAL

The main results were obtained from samples taken from the Ae and C horizons of five Podzol soil series in New Brunswick. Chemical and structural analyses of the coarse and fine clays from portions of these samples had been made previously by Kodama and Brydon (1968) and pertinent data are shown in Table 1. The Poitras (Podzol) and Breton (Grey Luvisol) soils were included for purposes of comparison. The five Podzol soils from New Brunswick were arranged according to increasing degrees of weathering as measured by TiO_2 accumulation in the Ae horizons and expressed in Ae/C TiO_2 ratios (Table 1). These soils followed the same sequence when arranged according to the kind and degree of interstratification of the clays in the Ae horizons (Kodama and Brydon, 1968). The Podzol Ae horizon clays consisted of dioctahedral interstratified mica-vermiculite-smectite components whereas the C horizon clays contained mainly dioctahedral mica and trioctahedral chlorite (Table 1). The mixing ratios of the three components (mica-vermiculite-smectite) in the $< 0.2 \mu$ clay were 0.53: 0.27: 0.20 for the least intensively weathered SMJ1 sample and 0.40: 0.38: 0.22 for the most intensively weathered SMJ9 sample. The determination of these mixing ratios was based on X-ray diffraction data and not on K contents of the clays (Kodama and Brydon, 1968). The Poitras Ae horizon clay has a large proportion of vermiculite-like layers whereas the expanding layers in the Breton clay are montmorillonitic (Brydon *et al.*, 1968; Kodama and Brydon, 1964).

Portions of the air-dried samples were dispersed in twice-distilled H_2O by ultrasonic vibration for $1\frac{1}{2}$ min and the 2–0.2 μ fraction was separated by combined sedimentation and centrifugation methods (Jackson, 1956). The clays were then calcium saturated using $CaCl_2$ solution and washed free of chloride. Then part of each clay was boiled for at least 24 hr with 6 per cent H_2O_2 without addition of HOAc and the untreated and peroxide-treated clays (after washing to remove H_2O_2) were freeze-dried.

The carbon contents of the clays were determined by a micro-dry combustion method using WO_3 to ensure complete combustion (Steyermark, 1961). Total K of each fractionated and differ-

ently treated clay was determined using the HF- $HClO_4$ decomposition method of Jackson (1958). Potassium was extracted at 25°C with NaTPB as described previously by Ross and Kodama (1970) using essentially the method of Reed and Scott (1966). Total and extracted K was determined by atomic absorption spectrophotometry (Ross and Kodama, 1970).

The procedure used to measure K fixation was adapted from a method described by Alexiades and Jackson (1965). Twenty ml of 1N KCl were added to 100 mg air-dry clay in a 50 ml plastic centrifuge tube. The contents were shaken overnight and the clay was washed with twice-distilled H_2O until free of chlorides (usually 4 washings) in a highspeed centrifuge. Next, 20 ml of 1N NH_4Cl were added and after overnight shaking the clay was centrifuged and washed once with 1N NH_4Cl . The clay was then decomposed using the HF- $HClO_4$ method of Jackson (1958) and the K determined by atomic absorption spectrophotometry. The increase in K content as compared to the K content prior to the K fixation procedure was taken as the amount of K fixed in the clay.

RESULTS

Comparing the rates of K exchange of the Ae horizon clays in Fig. 1A with each other, it is evident that the rates decreased sharply with increased degrees of weathering. In fact, after 1 and 2 days treatment time the per cent of total K exchanged from the least weathered SMJ1 clay was about 10 times that exchanged from the most weathered SMJ9 clay. After removal of organic matter from these clays, the rates of K exchange increased considerably (Fig. 1B). These increases were especially pronounced for the most weathered SMJ7 and SMJ9 clays which contained 1.5–2 times more organic matter than the remaining ones. However, the inverse relationship between rates of K exchange and degrees of weathering shown in Fig. 1A was also expressed in Fig. 1B, except for the reverse order of the SMJ5 and SMJ7 clays. This inverse relationship did not apply in the comparison of the K exchange from the Ae and corresponding C horizon peroxide-treated clays (Fig. 2). On the contrary, with the exception of the most weathered SMJ9 clay, the rates of K exchanged from the weathered interstratified Ae horizon clays were higher than those from the corresponding C horizon clays containing less weathered and more discrete mica.

The increase in K exchange after organic matter removal was investigated in more detail in the SML2 sample. The results of Fig. 3 show that the main increase in K exchange occurred when

Table 1. Clay mineral and chemical analysis of the 2-0.2 μ fraction †‡

Interstratified minerals															
Soil type	Horizon	Sample No.	Chlorite	Mica	Kaolinite	M-Ch	V-Ch	Ch-S	M-V-S	M-V-Ch	TiO ₂ (per cent)	TiO ₂ Ae/C ratio	K ₂ O (per cent)	Carbon (per cent)	Carbon (after peroxide treatment) (per cent)
Kedgewick	Ae	SMJ1		*					***		1.97	1.26	3.29	2.64	0.41
	C	SMJ2	*	***	tr.	*					1.56		5.46		
Monquart	Ae	SMJ3		*					***		2.12	1.85	2.81	3.24	0.63
	C	SMJ4	*	***	*					*	1.14		4.98		
Glacier	Ae	SMJ5		*					***		3.00	1.92	2.72	3.13	0.97
	C	SMJ6	**	**							1.56		4.13		
Serpentine	Ae	SMJ7		*	tr.				***		3.39	2.17	2.94	5.15	0.55
	C	SMJ8	**	**	*		*	**			1.25		3.13		
Tuadook	Ae	SMJ9		tr.	*				***		5.14	2.92	1.71	5.48	0.83
	C	SMJ10	***	**	*		*				1.73		3.17		
Poitras	Ae	SML2		***	*				***		1.70	1.31	3.62	3.99	0.46
	C	SML13	***	***	*		**				1.30		4.65		
Breton	C	SML12		*	*				***		0.80		1.93	0.72	0.73

***Abundant; **Moderate; *Minor. M—Mica; V—Vermiculite; Ch—Chlorite; S—Smectite; tr.—Trace.

†Clay mineral data and TiO₂ contents for the first 10 samples from Kodama and Brydon (1968). Data for TiO₂ in per cent of ignited weight of samples and for K₂O and carbon in per cent of air-dry weight of samples not treated with peroxide.

‡Clay mineral data for SML12 from Brydon *et al.* (1968). The author thanks Dr. J. E. Brydon for the use of the clay mineral and TiO₂ data for the SML2 and SML13 samples.

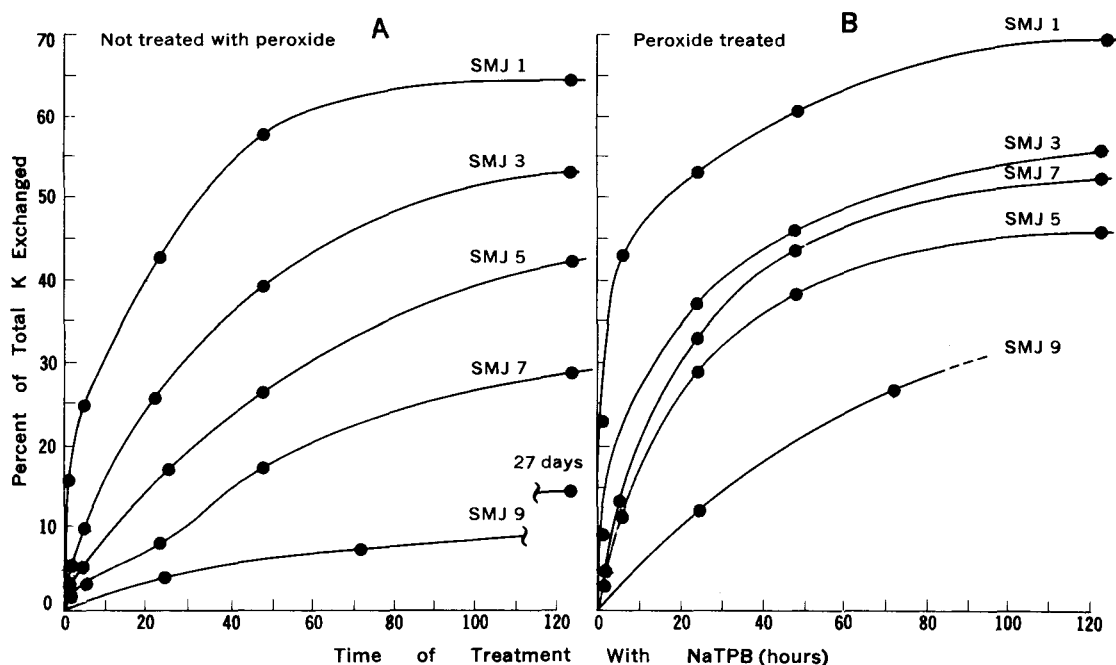


Fig. 1. Per cent of total K exchanged from the Ae horizon clays of five podzol soils of increasing degrees of weathering. Degrees of weathering increase from SMJ1 to SMJ9. Total K for part A refers to K contents of untreated samples. Total K for part B refers to K contents of peroxide-treated samples.

between 20 and 50 per cent of the organic matter was removed.

The amount of K fixed in the untreated Ae horizon clays did not increase with increased degrees of weathering (Table 2). On the contrary, no K was fixed in the most intensively weathered SMJ7 and SMJ9 clays prior to removal of their organic matter. The amounts of K fixed after removal of organic matter increased considerably in all the Ae horizon clays, particularly in the SMJ1, SMJ7 and SMJ9 clays. The SML2 clay, which had a large vermiculite component and contained 4 per cent organic carbon, showed the largest increase in K fixation after organic matter removal.

Table 2. Fixation of K against NH_4 in meq/100 g

Sample No.	Before peroxide treatment	After peroxide treatment
SMJ1	3.8	12.5
SMJ3	2.8	7.7
SMJ5	7.7	12.8
SMJ7	0	11.5
SMJ9	-0.7	7.7
SML2	9.2	24.0
SML12	6.1	5.1

However, the interstratified SML12 clay, which contained no organic carbon that could be removed by peroxide, showed no increase in K fixation after peroxide treatment. The C horizon clays fixed virtually no K by the fixation procedure followed.

To make the interpretation of the aforementioned results less equivocal, additional data were obtained on the effect of the peroxide treatment on the clays. The data in Fig. 4 show no difference in rates of K exchange before and after peroxide treatment of the interstratified SML12 clay which contained no removable organic matter. Neither did the amount of K fixed in this clay increase after this treatment (Table 2). Analysis of the supernatants showed that the peroxide treatment extracted less than 0.1 per cent Al and Fe and less than 0.03 per cent K from each clay. Since there was a possibility that particles remained aggregated in the untreated 2-0.2 μ clays and segregated during the following peroxide treatment producing additional small particles, some of the peroxide-treated clays were redispersed and the 2-0.2 μ fraction was again separated. There was no difference, however, between the rates of K exchange of the once- and twice fractionated clays.

The efficiency of the K fixation procedure was also investigated using an extracted SMJ1 sample in which the K content was reduced from 2.70

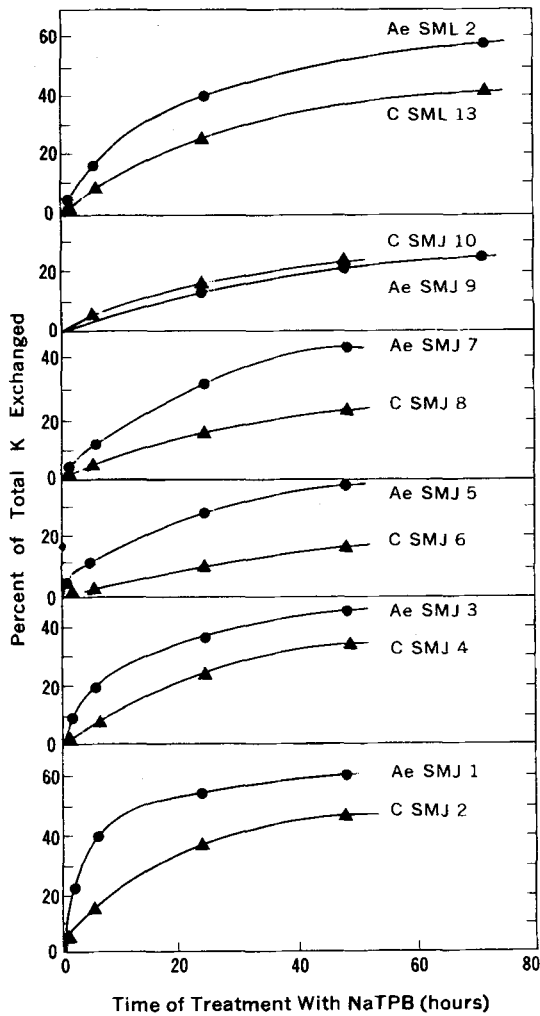


Fig. 2. Per cent of total K exchanged from the Ae and corresponding C horizon clays of Podzol soils. Both Ae and C horizon samples were treated with peroxide. Total K refers to K contents of peroxide-treated samples.

per cent to 0.70 per cent by extraction with 0.1N BaCl₂ at 100°C (Graf von Reichenbach and Rich, 1968). The K content after the K fixation procedure was 2.80 per cent, slightly higher than its original K content.

DISCUSSION

The inverse relationship between rates of K exchange and degrees of weathering was evident only in the comparison of the rates of K exchange of the Ae horizon clays. Smith *et al.* (1968) also observed that the rates and amounts of K replaced from micaceous clays, although obtained from soils of diverse genesis, decreased with increasing soil

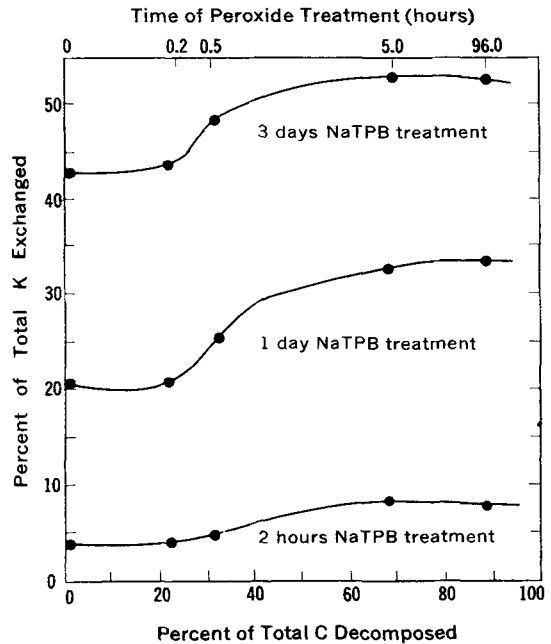


Fig. 3. Per cent of total K exchanged from a Podzol Ae horizon clay (SML2) at increasing times of peroxide treatment corresponding to increasing per cent of total carbon decomposed. Total K refers to K contents of samples determined after indicated periods of peroxide treatment.

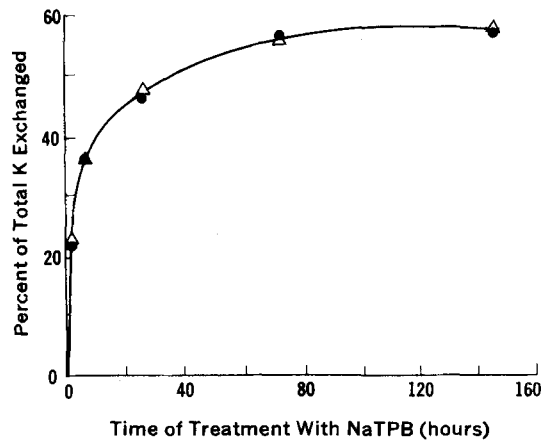


Fig. 4. Per cent of total K exchanged from a Gray-Luvisol C horizon clay (SML12) not treated with peroxide (Δ) and treated with peroxide (\bullet). Total K refers to K contents of untreated and peroxide-treated samples.

development. This inverse relationship did not apply to the comparison of Ae and corresponding C horizon clays. The lower rates of the C horizon clays in the latter comparison could not be attri-

buted to a higher content of potassium feldspars in the C horizon clays because X-ray data (Kodama and Brydon, 1968) indicated that the feldspar minerals in these clays belonged to the plagioclase series. The greater abundance of hydrated edges and layers exposing K exchange sites in the Ae horizon clays probably contributed to their higher rates of K exchange. This explanation derives support from the work of Newman and Brown (1969). They showed that K exchange was considerably delayed at ordered natural crystal edges, whereas this delay was eliminated at roughly-cut disrupted edges. This explanation is also supported by unpublished results which show that the rate of K exchanged from muscovite-type layers of interstratified mica-montmorillonite was much higher than that from discrete muscovite. However, the presence of adsorbed hydrous oxides of aluminum and iron in the C horizon clays cannot be ruled out and, if present, they could also have contributed to a retarded K exchange.

Increasing amounts of organic matter adsorbed on clay surfaces evidently acted in conjunction with increasing degree of weathering to reduce the rate at which K was exchanged. The reduction in K exchange caused by adsorbed organic matter was more pronounced at increased degrees of weathering. From the data in Fig. 3, the organic matter which decomposed in the range from 20 to 50 per cent of the total carbon had the greatest effect on the rate of K exchange. The data suggest also that at high levels of organic matter K fixation in these clays may be completely inhibited. Some K was fixed in all the untreated Ae horizon clays except in the SMJ7 and SMJ9 clays which contained 1.5–2 times more organic matter than the remaining ones. However, increased amounts of organic matter removed by peroxide treatment did not seem to be related to increased amounts of K fixed. The SMJ1 clay had as much increase in K fixation as the SMJ7 and SMJ9 clays but it had contained only half as much organic carbon. Apparently, in some of these clays the organic matter was more effective in blocking highly charged layers than in others.

This "blocking" effect of organic matter on both K release and fixation indicates the adsorption of organic compounds on charge sites of interlayer surfaces of micaceous clays. This adsorption apparently is sufficiently strong to inhibit the diffusion of K from and into the interlayer spaces. The organic compounds in soil organic matter which produce this inhibiting effect may well include some of the nitrogen compounds shown by Mortland (1961) to be very effective in inhibiting K adsorption in vermiculite.

Several alternative interpretations which could

explain the effect of peroxide treatment on K exchange and fixation were considered and found inadequate. The increase in rate of K exchange and extent of K fixation did not seem to be caused by exfoliation of silicate layers resulting from the peroxide treatment since this treatment did not affect the K release and fixation of an interstratified micaceous clay containing no organic matter. Neither could this increase be explained by removal of interlayer or otherwise adsorbed hydrous oxides of aluminum or iron because only small amounts of free aluminum or iron were present initially (Kodama and Brydon, 1968) and negligible amounts were extracted by the peroxide treatment. Nor could the increase be attributed to an increased number of small particles which could have resulted from a dispersion of aggregates by the peroxide treatment. Such dispersion, if present, did not affect subsequent K release as was indicated by refractionation of peroxide-treated samples.

Apparently only the highly charged layers in the interstratified clays fixed K in the procedure used since the amount of K fixed in one of these clays exceeded only slightly the amount of K extracted from it prior to the K fixation procedure. According to diagnostic criteria based on X-ray diffraction data, the mica components comprised about 40–55 per cent of the interstratified clays of the soils in the weathering sequence used. If it is assumed that the hydrated layers in these clays largely originated from mica, the relatively small amounts of K fixed suggest a considerable reduction in charge during the weathering of mica in these soils.

The evidence obtained in this investigation shows that the natural process of mica alteration, such as occurs in Podzol soils, is much more complicated than is indicated by laboratory experiments on the alteration of pure micas. The main features of this natural process may be sketched as follows.

When mica weathers in Podzol soils, the exchange of K for a hydrated cation in some mica layers is more rapid than in others resulting in an interstratified structure. The charge is also reduced following and perhaps during this exchange. Hydrous oxides of aluminum and iron are adsorbed in expanded interlayers and on outer surfaces but are eventually removed. Organic matter is retained, however, and contains compounds which are adsorbed on charge sites of interlayer surfaces with sufficient energy to impede K exchange and fixation. As weathering progresses increasing amounts of organic matter are adsorbed and eventually exchange and fixation reactions of K are virtually blocked. The evidence suggests that this weathering process may be delayed initially until a sufficient number of mica layers and

edges have become hydrated to provide readily accessible K exchange sites.

Acknowledgments—The author thanks Mr. M. Jaakkimainen for his technical assistance and Drs. J. E. Brydon, H. Kodama and J. A. McKeague for reviewing the manuscript.

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Résumé—On a déterminé, à l'aide du tétraphénylborate de sodium, les vitesses d'échange de K dans des argiles micacées, traitées et non traitées à l'eau oxygénée, provenant de cinq sols podzoliques ayant subi une altération naturelle à des degrés croissants. En plus, les quantités de K fixées ont été mesurées par rapport à NH_4 . Les argiles de l'horizon Ae de ces sols contenaient principalement un interstratifié mica-vermiculite-montmorillonite, alors que les argiles de l'horizon C contenaient un mica de nature plus distincte.

Comme on pouvait l'espérer, il y a une relation inverse entre les vitesses d'échange du K et les degrés d'altération des argiles de l'horizon Ae. Toutefois, dans les échantillons provenant de chacun de ces sols, à l'exception de ceux relatifs au plus altéré, la vitesse d'échange du K du mica interstratifié de l'horizon Ae est plus élevée que celle du K du mica moins altéré et plus distinct, appartenant à horizon C correspondant. L'abondance de bords hydratés et de feuillets dotés de sites d'échange de K dans les micas de l'horizon Ae contribue probablement à conférer à l'échange du K une vitesse plus élevée. Les quantités de K fixé dans les argiles de l'horizon Ae ne sont pas reliées au degré d'altération.

La destruction de la matière organique des argiles de l'horizon Ae par le peroxyde a considérablement augmenté et les vitesses d'échange de K et les quantités de K fixé. Ces augmentations ont été attribuées à l'élimination de l'effet de blocage que la matière organique adsorbée exerce sur l'échange et la fixation de K.

Kurzreferat— Die Geschwindigkeiten des K-Austausches von unbehandelten und peroxyd-behandelten glimmerhaltigen Tonen aus fünf Podzorböden mit zunehmendem Ausmass an Verwitterung wurden unter Verwendung von Natriumtetraphenylbor bestimmt. Darüber hinaus wurden die Mengen von gegen NH_4 festgehaltenem K gemessen. Die Ae-zone Tone dieser Böden enthielten hauptsächlich zwischengeschichteten Glimmer-Vermiculit-Montmorillonit während die C-zone Tone Glimmer einer mehr abesonderten Art enthielten.

Wie erwartet, bestand eine umgekehrte Beziehung zwischen den Geschwindigkeiten des K-Austausches und dem Ausmass der Verwitterung der Ae-zonen Tone. In den Proben aus jedem dieser Böden, mit Ausnahme derjenigen aus dem am meisten verwitterten, war die K-Austauschgeschwindigkeit des zwischengeschichteten Glimmers jedoch höher aus der Ae-zone als diejenige des weniger verwitterten, mehr abesonderten Glimmers aus der entsprechenden C-zone. Die Vielzahl hydratisierter Kanten und Schichten, die K-Austauschstellen in den Glimmern aus den Ae-zonen blosslegen, trugen wahrscheinlich bei zu den höheren Geschwindigkeiten des K-Austausches. Die in den Ae-zone Tonen festgehaltenen Mengen von K standen in keiner Beziehung zum Ausmass der Verwitterung.

Eine Entfernung der organischen Stoffe aus den Ae-zone Tonen durch Peroxyd erhöhte beträchtlich die Geschwindigkeiten des K-Austausches und der festgehaltenen Mengen von K. Diese Erhöhungen werden dem Fortfall einer blockierenden Wirkung der absorbierten organischen Stoffe auf den K-Austausch und die K-Festhaltung zugeschrieben.

Резюме — С использованием натрий-тетрафенил-бора определена степень обмена ионов K^+ для исходных и обработанных перекисью водорода слюdistых глин из пяти подзолистых почв. Кроме того определялось количество фиксированного K по отношению к NH_4 . Глины горизонта Ae подобных почв содержали, главным образом, смешанно-слоистый слюда-вермикулит-монтмориллонит, в то время как глины горизонта C содержали слюду более дискретной природы.

Как и ожидалось, была установлена обратная зависимость между долей обменного K и степенью выветривания глин горизонта Ae; однако во всех образцах изученных почв, за исключением одного (наиболее выветрелого), степень обмена K в смешанно-слоистой слюде из горизонта Ae была выше, чем в менее выветрелой, более дискретной слюде из горизонта C. Обилие в слюдах горизонта Ae гидратированных краев и слоев с обнаженными позициями обменного K, возможно, и обуславливает их более высокую степень обмена K. Количество K, фиксированного, в слюдах горизонта Ae, не зависит от степени выветривания.

Удаление органического вещества из глин горизонта Ae при обработке перекисью водорода приводило к значительному увеличению как степени обмена K, так и количества фиксированного K. Это увеличение способствовало ликвидации блокирующего влияния поглощенного органического материала на обмен и фиксацию K.