

ALUMINUM INTERLAYERS IN LAYER SILICATES

EFFECT OF OH/Al RATIO OF Al SOLUTION, TIME OF REACTION, AND TYPE OF STRUCTURE

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Abstract—Aluminum interlayers were synthesized under the same experimental conditions in a number of vermiculites and montmorillonites from different sources to determine the effects of the degree of neutralization of Al solutions, the time of reaction, and the type of structure.

Vermiculite fixed Al as well as hydroxy-Al ions in its interlayers, producing a stable 14 Å spacing and decreasing its cation exchange capacity considerably. Heating the Al-interlayered vermiculite at 300°C produced an interstratified mixture, indicating that some interlayers collapsed while others did not. The different collapse was attributed to different charge on vermiculite layers.

Neither the aging of vermiculite in Al solutions nor their OH/Al ratios changed the stability of the interlayers appreciably.

Montmorillonites, on the other hand, did not fix Al ions but fixed appreciable amounts of hydroxy-Al ions. In addition, the stability of the interlayers in montmorillonite increased on aging in hydroxy-Al solutions and exceeded the stability of the interlayers produced in vermiculite. To explain the greater stability of montmorillonite interlayers, it was postulated that the more expanded interlayer space in montmorillonite provides a favorable locale for the organization of hydroxy-Al ions into gibbsite structure while the restricted expansion in vermiculite prevents it.

INTRODUCTION

NUMEROUS examples of a 14 Å layer mineral, more resistant to collapse on heating or K-saturation than vermiculite but less resistant than chlorite, have been found in acid soils and sediments. The stability of the 14 Å spacing has been attributed to the presence of hydroxy-Al polymers in the interlayers of expanding layer silicates, montmorillonite and vermiculite. The stable spacing has also been obtained in the laboratory by precipitating $\text{Al}(\text{OH})_3$ or $\text{Mg}(\text{OH})_2$ in the interlayers of montmorillonite (Caillère and Hénin, 1949; Slaughter and Milne, 1960) or by treating vermiculite or montmorillonite with partially neutralized Al salt solutions (Sawhney, 1960; Rich, 1960; Barnhisel and Rich, 1963; Hsu and Bates, 1964; and de Villiers and Jackson, 1967). Not only is the composition of the interlayer material which imparts stability to the interlayers a matter of speculation, but the chemical constitution of the hydroxy-Al species used for the formation of Al-interlayers is also unknown. In addition, it has been observed by Shen and Rich (1962) that when montmorillonite was treated with Al salt solutions of increased OH/Al ratio, the stable spacing varied from 12.6 Å to 16.1 Å. Thus, not only is the nature of the interlayer material responsible for the stable spacing unknown, but the role of the

type of structure is also not established.

In the present investigation, Al interlayers were synthesized under the same experimental conditions in a number of vermiculites and montmorillonites from different sources to determine the effects of the type of structure, the degree of neutralization of Al solutions, and the time of reaction.

MATERIALS AND METHODS

Vermiculites (Libby, Montana and Transvaal, South Africa) and montmorillonites (Arizona and Wyoming) were obtained from Ward's Natural Science Establishment, Rochester, New York. Llano vermiculite was obtained through the courtesy of Dr. C. J. Mankin of the University of Oklahoma and was collected from the Moss Ranch at stop 12 during the field trip of the 10th Clay Conference.

The Al solutions with different OH/Al ratios were prepared as follows. Sodium hydroxide solution was added slowly (about 5 drops/min) to a 0.5 N AlCl_3 solution which was stirred constantly and vigorously. The concentration of the NaOH solution varied from about 0.17 N to 0.27 N to give a final solution 0.2 N in Al ions in all cases. The OH/Al ratios of these solutions refer to the ratio of added OH to Al in solution. This

procedure prevented the precipitation of $\text{Al}(\text{OH})_3$ and the solutions thus obtained were clear.

Clay fractions of the minerals were obtained as described previously (Sawhney, 1967). The clays were then saturated with Ca, suspended in water, and stored as 2 per cent stock suspensions. Aliquots containing about 0.2 g colloid were suspended in 15 ml of the 0.2 N AlCl_3 solutions of OH/Al ratio 0.0, 2.0 and 2.4. The suspensions were centrifuged and washed with water once only to in 50 ml of the same Al solution and allowed to age. The suspensions were occasionally shaken.

After different intervals, aliquots from these suspensions were withdrawn: one aliquot was centrifuged and washed with water once only to avoid possible hydrolysis of Al on the clay by excessive washing. The colloid was then suspended in a minimum amount of water, dried on a glass slide and analysed by X-ray diffraction, using a Norelco wide angle goniometer and a flat specimen holder. The slide was then heated overnight at different temperatures and analysed again. Another aliquot was saturated with K by five centrifuge washings with a 1N KCl solution followed by two water washings and then after heating at different temperatures, the colloid was analysed by X-rays.

The cation exchange capacity (CEC) of the minerals was determined on a third aliquot by the method of Sawhney *et al.* (1959). The colloid on which the CEC had been determined was given five centrifuge washings with 0.2 N HCl to extract the Al from the interlayers (Shen and Rich, 1962). The Al removed by the acid was determined by the aluminon method (Jackson, 1958). Appropriate corrections were made for Al extracted from samples untreated with Al. After the acid extraction, the CEC of the colloid was determined again. The amount of Al removed by the acid and the subsequent increase in CEC were used to determine the apparent charge on the Al blocking the exchange sites after the various Al treatments.

RESULTS AND DISCUSSION

Aluminum solutions

The aluminum solutions neutralized to different degrees remained clear for several months. In addition, the pH of these solutions did not change significantly after the first three weeks (Table 1), indicating no significant change in the hydrolysis of Al or hydroxy-Al ions in these solutions. Similarly, only slight changes occurred in the pH of mineral suspensions in the various Al solutions, again suggesting only slight changes in the hydrolysis status of the Al or hydroxy-Al ions remaining in solution as well as those sorbed on the clay.

Table 1. pH of 0.2 N AlCl_3 solution of different OH/Al ratio and of mineral suspensions in these solutions

Aging time weeks	OH/Al		
	0	2.0	2.40
	pH of Al-solutions		
0	3.35	4.15	4.70
3	3.20	3.95	4.50
10	3.30	4.02	4.55
	pH of Libby vermiculite suspension		
0	3.30	4.15	4.70
3	3.10	3.95	4.35
10	3.15	4.00	4.58
	pH of Llano vermiculite suspension		
0	3.25	4.15	4.70
3	3.05	3.95	4.25
10	3.05	4.00	4.58
	pH of Arizona bentonite suspension		
0	3.35	4.15	4.70
3	3.10	3.95	4.25
10	3.05	4.00	4.30

Stability of 14 Å spacing in vermiculites

When saturated with either unneutralized or partially neutralized Al salt solutions, vermiculites exhibited a 14 Å *c*-axis repeat. The 14 Å spacing was stable at 100°C and did not collapse completely to 10 Å even at 300°C. Obviously, the water or the hydroxyl ions associated with the interlayer Al are not removed completely, even at 300°C. Because the vermiculites treated with unneutralized as well as partially neutralized Al salt solutions were equally stable, the data on vermiculites (Libby and Llano) treated with unneutralized Al solutions (Fig. 1) are representative of the other treatments also. Similar results were obtained with vermiculite from Transvaal.

When the vermiculites treated with Al solutions (Curve 1, Fig. 1) were saturated with K (curve 2, Fig. 1), no change in their diffraction patterns occurred. However, when K-saturated samples were heated at 100°C, the *c*-axis repeat was reduced to 12.6 Å (curve 3, Fig. 1). The 12.6 Å spacing represents a randomly interstratified mixture of a 14 Å and a 10 Å spacing, because no integral orders of this spacing were recorded. The collapse of the Al-interlayered structures to spacings intermediate between 14 Å and 10 Å has, heretofore, been attributed to a partial collapse of the 14 Å spacing. When the K-saturated samples were heated at 300°C, the Libby vermiculite collapsed to 10.7 Å, while the Llano vermiculite collapsed to 11.3 Å (curve 4, Fig. 1). Thus, Llano vermiculite contained a larger number of stable

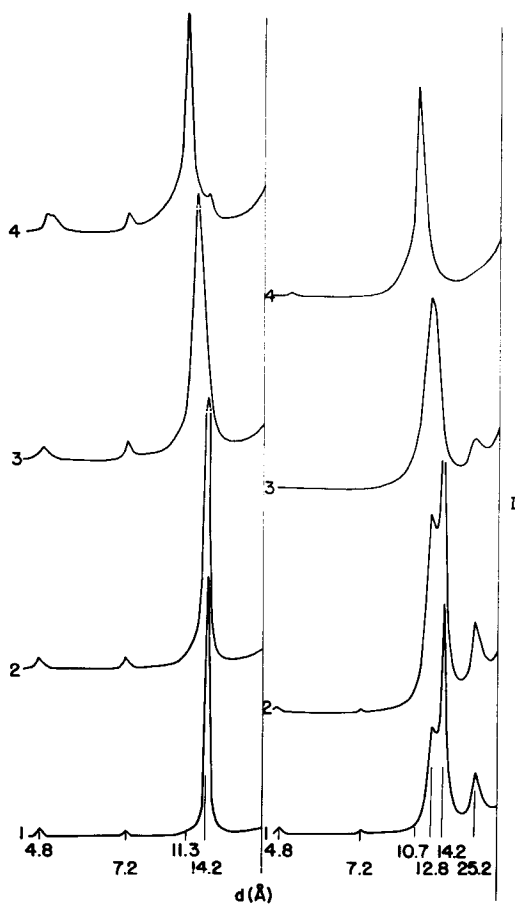


Fig. 1. X-ray diffraction patterns of Llano (left) and Libby (right) vermiculites after aging for 3 weeks in Al solution of OH/Al ratio 0.0 [1], followed by K-saturation, then air-dried [2], then heated at 100°C [3] and then at 300°C [4].

layers with a 14 Å spacing than the Libby vermiculite. Heating at 500°C, however, collapsed both vermiculites to 10 Å.

The formation of the interstratified phase in vermiculites could be ascribed to the removal of Al from some layers and not from others. However, the quantities of Al removed by KCl were too small to produce the observed changes in the spacings. Furthermore, the interstratified mixture did not appear in the K-saturated and air-dried samples but developed on heating these samples.

The interstratified mixture was also formed when vermiculites treated with Al solutions were heated without K-saturation. The temperature of the formation of interstratified phase was, however, lower in K-saturated samples, indicating that the removal of small amounts of Al by K facilitated

the collapse of some layers. The formation of the interstratified mixture on heating the Al-interlayered vermiculite, thus, shows that some layers collapsed more readily than others. The different collapse suggests an uneven distribution of the charge on vermiculite layers so that the layers with a greater charge collapsed more readily than the layers with a smaller charge.

Stability of 14 Å spacing in montmorillonites

When montmorillonites were treated in the same manner as vermiculites with Al solutions of different basicities, the results differed from those obtained with vermiculites. The spacing of the montmorillonite lattice containing Al interlayers varied with the OH/Al ratio of the Al solution. When the Arizona bentonite was aged in AlCl₃ solution of OH/Al ratio 2.0 or 2.4, a superlattice of 18.4 Å spacing was formed (curves 2 and 3, right, Fig. 2)

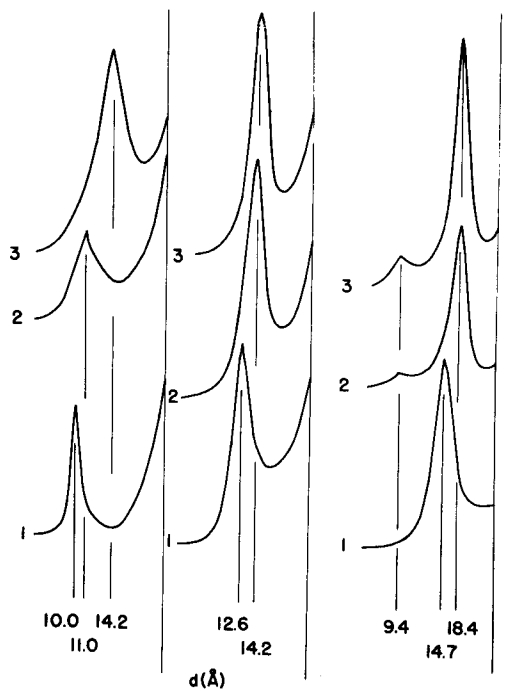


Fig. 2. X-ray diffraction patterns of Arizona montmorillonite after aging for 3 weeks in Al solutions of OH/Al ratio 0.0 [1], 2.0 [2] and 2.4 [3], right column; same saturated with K and heated at 100°C [middle column] and then at 500°C [left column].

compared to the 14.2 Å spacing of vermiculites. Similar results were obtained with Wyoming bentonite. The larger spacing of Al-interlayered montmorillonite than of vermiculite is associated with its smaller charge originating in its octahedral

layer as compared to the larger vermiculite charge originating in its tetrahedral layer. Under different experimental conditions, Shen and Rich (1962) obtained montmorillonite with spacings from 14.7 Å to 16.7 Å in Al solutions of OH/Al ratio 0.0–1.35. Apparently, Al interlayers in montmorillonite can be hydrated to different degrees.

Also, whereas the spacing of Al-interlayered vermiculite did not change on aging, the montmorillonite spacing increased on aging (data not shown). In a solution of OH/Al of 2.0, the spacing increased from 18.4 Å at 3 weeks to 19.0 Å at 10 weeks, and in a solution with OH/Al of 2.4, the spacing increased to 19.6 Å. Furthermore, montmorillonites aged for 10 weeks gave sharp integral orders of the basal spacing while the integral orders given by the 3-week samples were diffuse.

When montmorillonites aged for 3 weeks in Al solutions were saturated with K and air dried or heated at 100°C, the montmorillonite treated with Al solution of OH/Al 0.0 collapsed to 12.6 Å and then to 10.0 Å at 500°C. On the other hand, the large spacing montmorillonite formed at OH/Al of 2.0 and 2.4 was dehydrated to only 14.2 Å upon heating at 100°C (middle column, Fig. 2). On further heating to 500°C, the montmorillonite treated with Al solution of OH/Al 2.0 collapsed to about 11.0 Å, while that treated with OH/Al of 2.4 remained at 14.2 Å (left column, Fig. 2). Whereas, aging for 10 weeks did not change the montmorillonite in Al solutions of OH/Al 0.0, the montmorillonite treated with Al solutions of OH/Al 2.0 and 2.4 retained the stable 14.2 Å spacing after K-saturation and heating up to 600°C (not shown).

Thus, the hydroxyl-Al interlayers occur in more

hydroxy-Al solutions of OH/Al ratio 2.25, also shows that the montmorillonite structure facilitates the formation of the gibbsite structure. Although X-ray diffraction of montmorillonite aged for 10 weeks showed some gibbsite, no gibbsite was observed either in the vermiculite suspensions or in the hydroxy-Al solutions during this period.

To explain the presence of gibbsite in montmorillonite and not in vermiculite, it is postulated that the more expanded interlayer space of montmorillonite permits the reorganization of the hydroxy-Al ions into a gibbsitic structure whereas the restricted expansion in vermiculite interlayers does not permit gibbsite formation. Gibbsite may then be extruded from the interlayers and restore the CEC of montmorillonite as observed by Barnhisel and Rich (1963).

Effect of Al-interlayers on cation exchange capacity of vermiculites and montmorillonites

All the Al solutions used decreased the cation exchange capacity of vermiculites drastically (Table 2). However, the hydroxy-Al solutions reduced the CEC somewhat more than the Al solution to which no base was added: The CEC of Libby vermiculite decreased from 140 me to about 40 me/100 g after 3 weeks in hydroxy-Al solutions, while the CEC decreased to only 62 me after 3 weeks in Al solution to which no base was added (column 1). Furthermore, the CEC of vermiculite continued to decrease slightly with time in all Al solutions.

The cation exchange capacity of montmorillonites, on the other hand, did not change significantly in 0.2 N $AlCl_3$ to which no base was added.

Table 2. Cation exchange capacity, me per 100 g, of vermiculite and montmorillonite minerals as affected by the OH/Al ratio of the Al solution and the time of reaction

Treatment	Libby vermiculite		Llano vermiculite		Arizona bentonite	
	3 wk	10 wk	3 wk	10 wk	3 wk	10 wk
Untreated	140	140	146	146	137	137
OH/Al = 0.0	62	50	59	48	137	138
OH/Al = 2.0	40	31	51	35	114	20
OH/Al = 2.4	41	33	51	36	41	16

organized structural units in aged montmorillonites than in vermiculites. The presence of gibbsite was indicated by a 4.85 Å and a 4.50 Å diffraction peak in montmorillonite aged for 10 weeks in Al solutions of OH/Al ratio 2.0 and 2.4. The observation of Barnhisel and Rich (1963) that gibbsite was formed in montmorillonite aged for 3 months in

But, the CEC of montmorillonite decreased appreciably with increasing OH/Al ratio, and the CEC decreased further on aging in hydroxy-Al solutions. The CEC of Arizona bentonite decreased from 137 me/100 g to 114 and 41 after aging for 3 weeks in Al solutions with Al/OH of 2.0 and 2.4 respectively (column 6). The CEC decreased

further to 20 and 16 me after aging for 10 weeks (column 7).

Thus, although vermiculites with greater layer charge and tetrahedral charge sites hold the Al as well as the hydroxy-Al ions more tightly than montmorillonites, aging in hydroxy-Al solutions decreased the exchange capacity of montmorillonites more than vermiculites. The stability of the 14 Å spacing of montmorillonite aged in hydroxy-Al solutions (Fig. 2) was also greater than the stability of similarly treated vermiculite (Fig. 1). The larger decrease in CEC and the more stable 14 Å spacing in montmorillonite suggest that the interlayers of montmorillonite, which are more expanded than vermiculite, provide a more favorable locale for the organization of a gibbsite interlayer structure.

Nature of aluminum interlayers

Interlayer Al was extracted with 0.2 N HCl (Shen and Rich, 1962) from the samples on which CEC was previously determined (Table 2). Immediately after the acid extraction, the CEC of the samples was determined again. Since the H-saturated clays are known to change to Al-clays on aging, the CEC was determined without letting the samples age. Although the extraction with HCl increased the CEC of vermiculite and montmorillonite containing Al-interlayers, the original CEC was not regained completely, suggesting that the HCl did not remove all the Al from the interlayers. Nonetheless, a definite trend in the exchange charge blocked by the Al and the hydroxy-Al ions is obvious. The Al removed per me of exchange capacity was about 9 mg in the samples aged in Al solution of OH/Al ratio 0.0 (Table 3). The Al removed from vermiculite aged in hydroxy-Al solution of OH/Al 2.0 varied from 24 to 33 mg/me while the Al removed from samples in solution of Al/OH 2.4 was about 45–51 mg per me. Thus, the

apparent charge per Al ion fixed in the vermiculite interlayers varied from about +3 for Al solutions of OH/Al ratio 0.0 to about +1 for samples treated with a solution of OH/Al ratio 2.0 and about +0.5 for samples treated with a solution of OH/Al ratio 2.4. In montmorillonites aged for 10 weeks in hydroxy-Al solutions, Al fixed per me varied from 72 to 86, and the charge per Al ion fixed in the interlayers (stable to 600°C) was about 0.2–0.3 (column 8). The estimates of the charge per unit Al ion in the interlayers are in the range of some of the polymeric forms of Al proposed by Hsu and Bates (1964).

Although the scheme proposed by Hsu and Bates may be one of the possible mechanisms for the development of gibbsite structure from hydroxy-Al ions, the assignment of a specific polymeric form to the interlayer Al on the basis of the charge alone is uncertain.

CONCLUSIONS

Laboratory syntheses of Al interlayers in different vermiculites and montmorillonites under the same experimental conditions showed that the stability as well as the composition of the interlayers are controlled not only by the OH/Al ratio of the Al solution but also by the mineral structure.

Vermiculite fixed Al as well as hydroxy Al ions in its interlayers, reducing the cation exchange capacity considerably. The Al-interlayers in vermiculite were of different stability attributed to different charge on its layers. Neither the aging of vermiculite in Al solutions nor their OH/Al ratios changed the stability of the interlayers appreciably.

Montmorillonites, on the other hand, did not fix Al ions but fixed appreciable amounts of hydroxy-Al ions. When aged in partially neutralized Al solutions, the stability of interlayers in montmorillonite increased and exceeded the stability of the interlayers produced in vermiculite.

Table 3. Increase in cation exchange capacity (Δ CEC, me/100 g) and Al removed (mg/me) by 0.2 N HCl from vermiculite and montmorillonite treated with Al solutions of different OH/Al for different time period

OH/Al	Change with HCl	Libby vermiculite		Llano vermiculite		Arizona bentonite	
		3 wk	10 wk	3 wk	10 wk	3 wk	10 wk
0.0	Δ CEC	23	48	44	60	0	0
	Al removed	10	9	8	8	—	—
2.0	Δ CEC	50	69	52	65	24	86
	Al removed	33	29	24	31	84	72
2.4	Δ CEC	40	57	55	62	59	84
	Al removed	47	45	50	51	87	80

To explain the formation of more stable interlayers in montmorillonite, it was postulated that the more expanded interlayer space in montmorillonite provides a favorable locale for the organization of hydroxy-Al ions into gibbsitic structure while the restricted expansion in vermiculite prevents it.

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Résumé—La synthèse de couches intermédiaires d'aluminium a été faite dans les mêmes conditions expérimentales, sur un nombre de vermiculites et de montmorillonites de différentes sources, afin de déterminer les effets du degré de neutralisation de solutions d'Al, le temps de réaction et le type de structure.

La vermiculite a fixé Al ainsi que les ions hydroxy-Al dans des couches intermédiaires, en produisant un espacement stable de 14 Å et en diminuant considérablement sa capacité d'échange de cations. En chauffant à 300°C la vermiculite avec couche intermédiaire d'Al, il s'est produit un mélange entre les couches, indiquant que quelques couches intermédiaires se sont effondrées tandis que les autres n'ont pas bougé. Cet effondrement a été attribué à la charge différente sur les couches de vermiculite.

Ni le vieillissement de la vermiculite en solutions Al, ni leurs rapports OH/Al, n'ont changé pas de façon appréciable la stabilité des couches intermédiaires.

D'autre part, les montmorillonites n'ont pas fixé les ions Al, mais ont fixé par contre des quantités considérables d'ions hydroxy-Al. De plus, la stabilité des couches intermédiaires dans le montmorillonite a accrue le vieillissement des solutions hydroxy-Al et a dépassé la stabilité des couches intermédiaires produites dans la vermiculite. Pour expliquer la plus grande stabilité des couches intermédiaires de montmorillonite, il a été postulé que l'étendue plus grande des couches intermédiaires dans le montmorillonite fournit une scène favorable pour l'organisation des ions hydroxy-Al dans la structure de gibbsite, tandis que l'expansion restreinte dans la vermiculite l'empêche.

Kurzreferat—In einer Anzahl von Vermiculiten und Montmorilloniten verschiedener Herkunft wurden unter den gleichen Versuchsbedingungen Aluminiumzwischen-schichten künstlich hergestellt, um die Wirkung des Neutralisations-grades von Al-Lösungen, der Reaktionszeit und der Gefügeart zu bestimmen.

Vermiculit hielt Al sowie Hydroxyl-Al Ionen in den Zwischenschichten fest unter Bildung einer stabilen 14 Å Abstand und beträchtlicher Verminderung des eigenen Kationenaustauschvermögens. Erwärmung des mit Al-Zwischenschichten versehenen Vermiculits auf 300°C führte zu einer zwischengeschichteten Mischung, was darauf hindeutet, dass manche der Zwischenschichten zusammenbrachen, während andere erhalten blieben. Das unterschiedliche Zusammenbrechen wurde auf Verschiedenheiten in der Ladung der Vermiculitschichten zurückgeführt.

Die Stabilität der Zwischenschichten wurde weder durch die Alterung des Vermiculits in Al-Lösungen, noch durch die OH/Al Verhältnisse derselben wesentlich verändert.

Im Gegensatz hierzu hielten die Montmorillonite keine Al Ionen fest, sondern jedoch beträchtliche Mengen von Hydroxyl-Al Ionen. Darüber hinaus erhöhte sich die Stabilität der Zwischenschichten im Montmorillonit bei der Alterung in Hydroxyl-Al Lösungen und überstieg die Stabilität der im Vermiculit gebildeten Zwischenschichten. Zur Erklärung der grösseren Stabilität der Montmorillonit-Zwischenschichten wurde angenommen, dass der erweiterte Zwischenschichtabstand im Montmorillonit einen günstigen Ort für die Anordnung von Hydroxyl-Al Ionen in ein Gibbsit Gefüge schafft, während der beschränkte Raum im Vermiculit das verhindert.

Резюме—Аллюминиевые промежуточные слои синтеризованы в тех же экспериментальных условиях в ряде вермикулитов и монтмориллонитов из различных источников для определения степени нейтрализации растворов Al , продолжительности реакции и типа структуры.

Вермикулит фиксирует ионы Al , а также гидроксиды— Al в промежуточных слоях, что дает устойчивый промежуток 14 \AA и значительно понижает катионообменную способность. Нагрев вермикулита с промежуточными слоями алюминия при 300°C дает рассланцованную смесь, указывая тем самым, что некоторые промежуточные слои рухнули, в то время как другие нет. Разность разрушения приписывают разнице заряда в слоях вермикулита.

Старение вермикулита в растворах Al , или соотношения OH/Al в заметной степени не меняют устойчивости промежуточных слоев.

С другой стороны, монтмориллониты не фиксируют ионов Al , но зато фиксируют крупные количества ионов гидроксидов Al . Кроме этого, устойчивость промежуточных слоев в монтмориллоните увеличивается при старении в растворах гидроксидов— Al и превышает устойчивость промежуточных слоев, возникших в вермикулите. Для того, чтобы объяснить большую устойчивость монтмориллонитовых промежуточных слоев, допускается, что более увеличенное пространство промежуточных слоев предоставляет благоприятное место для группировки ионов гидроксидов— Al в гиббитовые структуры, в то время как ограниченное увеличение в вермикулите предотвращает именно это.