KINETICS OF ACID-DISSOLUTION OF PALYGORSKITE (ATTAPULGITE) AND SEPIOLITE

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Abstract — Palygorskite and sepiolite were digested with large excesses of hydrochloric acid at constant temperature for various periods of time. The reaction was found to be of first order with respect to the magnesium, aluminum and iron components in the clay and also with respect to hydrochloric acid concentration.

The reaction rate constant for Mg is larger than for Fe, which is larger than for Al. Approximately one-third of the octahedral-type cations are extraneous, suggesting that only three of the five octahedral positions are filled. The reaction rate constant of Mg in sepiolite is about 240 times that for palygorskite. This may be an indication that sepiolite is less stable to chemical weathering than palygorskite.

The activation energies for Mg, Al and Fe in palygorskite were found to be the same and equal to about 18·4 kcal per mole.

Treatment of partially acid-digested palygorskite with magnesium ions under alkaline conditions resulted in an increase in the X-ray intensity of the 10·5 Å spacing, suggesting some of the palygorskite was reconstituted.

INTRODUCTION

IT IS believed that Brindley and Youell (1951) were the first to determine the amounts of octahedral and tetrahedral Al using the acid attack technique. They digested magnesium chlorite (penninite) with a considerable excess of 10% HCl at constant temperatures for various time intervals. The percentage extractions of Mg, Al, and Fe were found to follow the same curve when plotted against time; in the case of Al the extraction rate is similar up to about 47 per cent, but thereafter proceeds very much more slowly. Brindley and Youell concluded that octahedral Al is represented by the first part of the curve, whereas, the second part of the curve represents the tetrahedral Al. Their results were found to agree closely with that given by the structural formula.

More detailed studies of the kinetics of acid dissolution were made by Osthaus (1954, 1956). He digested several montmorillonites and a nontronite with HCl at constant temperatures for various periods of time. Rate of dissolution curves were obtained by plotting the logarithm of the per cent of the residual ions against time. In general, the solution curves were straight lines or curves that could be resolved into two straight lines. From the interpretation of the dissolution curves, it was

possible to determine the amount of cations in octahedral and tetrahedral coordination.

The dissolution of the ions from the clay structure determined at several temperatures in one sample and at two acid concentrations in another sample was found by Osthaus to be in agreement with chemical kinetics principles and to be a first order reaction with respect to both residual ion concentration and to acid concentration. In addition, he found that Mg, Al, and Fe ions in the octahedral sheet have approximately the same reaction rate. Using the Arrhenius law, the activation energy of acid dissolution was determined and found to be 17–18 kcal per mole. This was found to be the same for both octahedral and tetrahedral ions.

Granquist and Sumner (1959) studied the various physical properties of an acid-treated Texas bentonite as a function of treatment time. They also observed a pseudo first-order kinetics. A large excess of 10% HCl was used in amount calculated to be ten times that required for complete dissolution of all catonic components of the crude material. Treatments were carried out primarily at 103°C, with some data obtained at 67°C.

In calculating the reaction rate, a new approach based on the X-ray intensity of the 17 Å spacing, rather than the results of chemical analysis, was used by Granquist and Sumner. Based on this new method, they found that the reaction rate was higher than that calculated from chemical analysis. Using the kinetics data of two different temperatures, they calculated the activation energy to be

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7·27 kcal/mole. Two different models were examined and it was concluded that a cylindrical model and an edge attack best fit their experimental data.

Gastuche (1963) studied the kinetics of acid dissolution of biotite. She examined the effect of the amorphous silica formed during the acid dissolution in limiting the diffusion of the dissolved cations and of hydrogen ions at the interface. Gastuche found that the acid dissolution of biotite is a tridimensional process which fits a first order reaction with respect to the HCl concentration. At low temperatures no limitation due to the diffusion process is noticeable; however. at 100°C. the reaction along the Z-axis is hindered by a diffusion mechanism. The activation energy of acid dissolution of biotite was calculated by Gastuche to be about 10 kcal/mole.

In this study the reaction rate of palygorskite was determined at four different temperatures and two different acid concentrations. The activation energy of the acid dissolution of Mg, Al, and Fe in attapulgite was calculated. The partially digested palygorskite residues were treated with the proper ions in an attempt to reverse the acid dissolution reaction. Also. the reaction rate for sepiolite was determined at one temperature and one acid concentration. The resistance of palygorskite and sepiolite to acid weathering were compared.

PROCEDURE

The palygorskite (Attapulgus. Georgia) specimen used in this investigation was a purified sample provided by Mr. T. D. Oulton of Englehard Minerals and Chemicals Corporation (Menlo Park. Edison. New Jersey). The sepiolite (Asia Minor. Turkey) specimen was obtained from southwest Scientific Company (Hamilton. Montana).

Except for some minor points. the method used in this investigation was essentially that of Osthaus (1956). The clay specimens were ground and the particle size smaller than one micron separated with the centrifuge. washed several times with distilled water and then dried at room temperature. This material was left at room temperature for sufficient time to assure its equilibrium with air. One gram samples were used for each run. To avoid the effects of changing humidity. samples sufficient for all the runs were weighed within a short period of time. The 1 g sample was transferred to a 500-ml round-bottomed pyrex flask. Then 200 ml hydrochloric acid. which was heated to the reaction temperature, was added and the flask was quickly connected to water-cooled reflux condensers by ball and socket ground glass joints. The bulb section of the flask was submerged in a constant-temperature water bath for various periods of times. The water-bath temperature was controlled by a coil of copper tubing which was connected to the LAUDA constant temperature circulator model K-2 manufactured by Brinkmann Instruments (Westbury. New York). Uniformity of the bath temperature was achieved by using a magnetic stirrer, while the water level in the bath was kept constant by adding water dropwise from a large water jar using a stop-cock in order to make up for the water lost by evaporation. At the end of each digestion period. the flask was removed and quickly cooled with ice. The clay–acid suspension was centrifuged. then filtered. and the filtrate was retained for chemical analysis. For about 50 per cent digestion or less. the filtrate was analyzed for soluble Mg, Al, and Fe. The amount of each of the residual ions was calculated by subtracting the per cent soluble from the total percentage in the sample. For considerably higher than 50 per cent digestion. it was advantageous to determine the small amounts of residual Mg. Al. and Fe directly from the insoluble residues. The attapulgite was digested in 2·5 N and 5·0 N HCl. 200 ml 5·0 N HCl is equivalent to about 85 times that required for complete digestion of the cations in 1·0 g attapulgite. HCl concentration of 1·0 N was used to digest the sepiolite. This is equivalent to about 19 times that required for the complete digestion of the cations.

Mg, Al, and Fe analyses were made with an atomic absorption spectrophotometer. Each of the residues of the digested polygorskite made with 5·0 N HCl at 70·3°C were divided into two portions. One portion was treated with very dilute NaOH solution (pH = 9) at 110°C for 2 days in a nickel pressure bomb. The other portion was treated under similar conditions with a weak solution of $MgCl_2$ (about 1000 ppm Mg) after adjusting the pH to 9 with a few drops of NaOH solution. The samples were cooled. centrifuged and washed many times with distilled water. The X-ray intensity of the 10·5 Å spacing was compared with that of the initial residue.

RESULTS AND DISCUSSION

Partial chemical analysis of air dried palygorskite and sepiolite are given in Table 1.

The amount of HCl used for dissolution was

Table 1. Partial chemical analysis of palygorskite and sepiolite

Per cent	Palygorskite	Sepiolite
SiO_2	56·00	52·00
MgO	12·40	23·350
Al_2O_3	7·98	0·404
Fe_2O_3	2·51	0·215

equivalent to about 85 times the amount needed for the complete reaction with the Mg. Al. and Fe in palygorskite; for sepiolite it was about 19 times the amount required to react with the Mg. Therefore, the HCl concentration remained virtually constant during the reaction.

Acid-dissolution curves were obtained by plotting the residual cations, expressed as the oxide, vs. time on a semilogarithmic paper. A straight line relation represents a first order reaction with respect to the cations, Mg, Al, and Fe. This assumes that the volume of the clay remains constant during the reaction. This assumption seems to be reasonable as the molecular structure does not collapse until most of the metal ions are extracted.

Data on the acid-dissolution of palygorskite were obtained at temperatures of 60·0, 70·3, 80·1 and 91·2°C using 5·0 N HCl. The data are plotted in Figs. 1–4.

The curves for Mg, Al, and Fe have high slopes at the start of the reaction, then level off and form straight lines. The straight line portions for the Mg

must represent the octahedral ion, since Mg does not occur in the tetrahedral layer. By analogy, the straight line portions for the Al and Fe curves presumably represent octahedral Al and Fe.

The first parts of the curves for Mg, Al, and Fe represent some extraneous materials which possess high reaction rate and which are completely reacted in short time. Extrapolation of the straight line portion of the curves indicates the early soluble material amounts to approximately 4 per cent MgO, 2 per cent Al_2O_3 and 1 per cent Fe_2O_3. A small portion of this material is present as exchange material but most of it must occur on the clay surface or in the palygorskite channels.

Approximately one-third of the cations that normally are assumed to be in the octahedral layer are apparently present as impurities. This indicates that fewer of the five octahedral positions are filled than the four suggested by Bradley (1940). Calculations based on the original analysis (assuming 21 oxygens per formula unit indicate that 4·16 octahedral positions are filled. Calculations based on
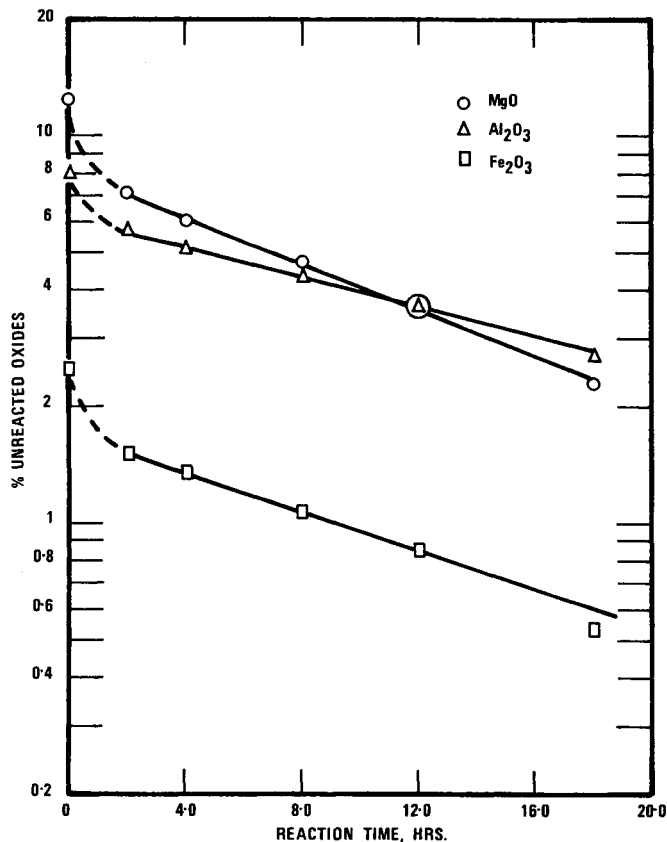

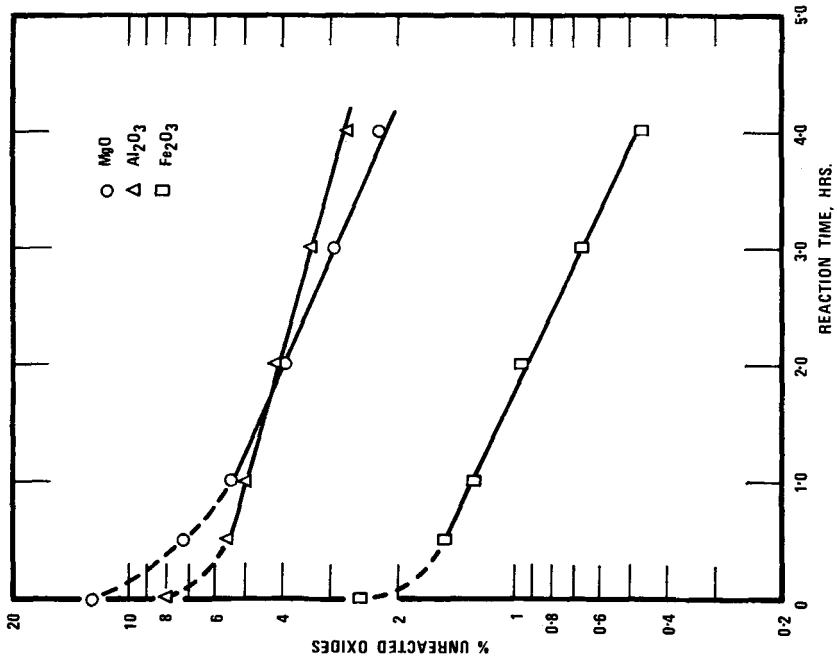Fig. 1. Reaction of palygorskite. Attapulgus. Georgia. with 5·0 N HCl at 60°C.

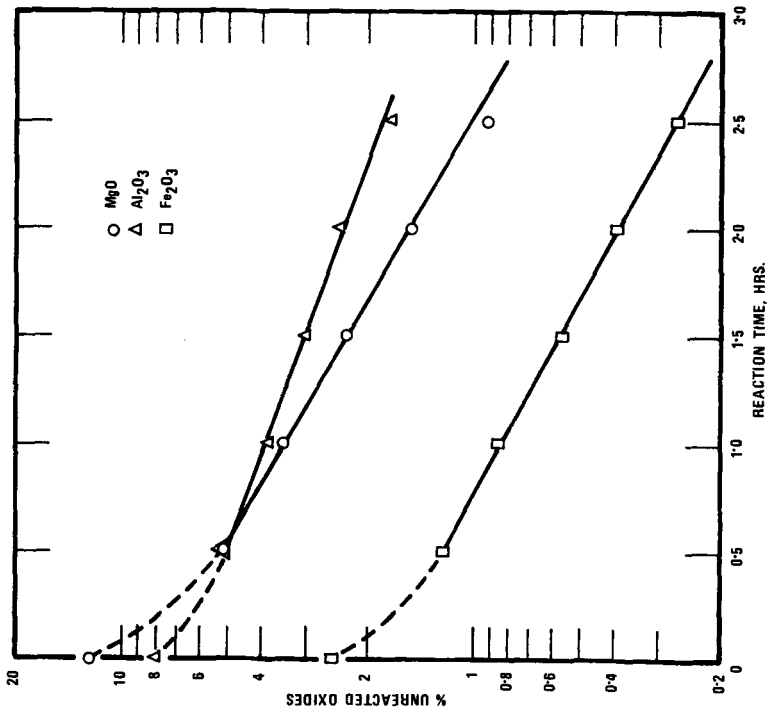Fig. 3. Reaction of palygorskite, Attapulgus, Georgia, with 5·0 N HCl at 80·1°C.

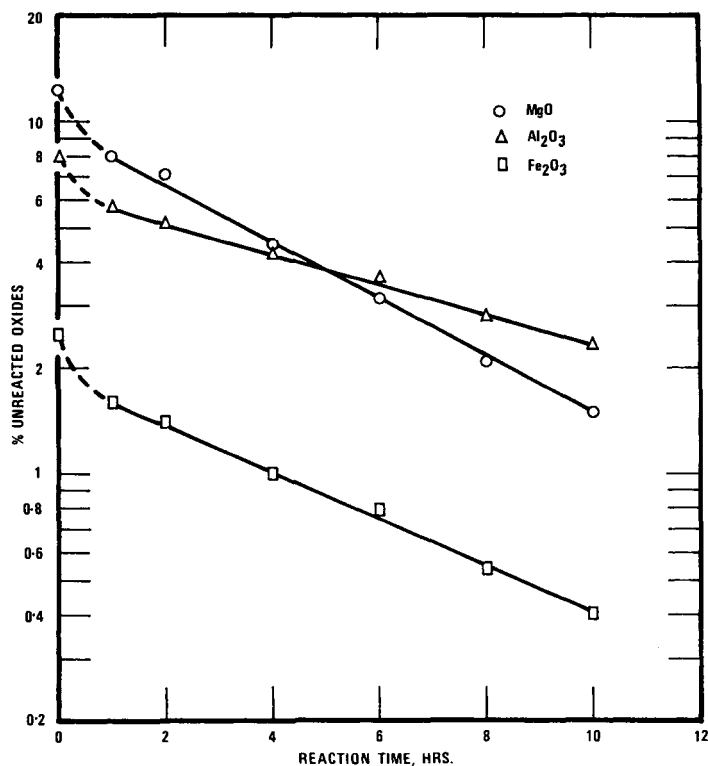Fig. 2. Reaction of palygorskite, Attapulgus, Georgia, with 5·0 N HCl at 70·3°C.

Fig. 4. Reaction of palygorskite. Attapulgus, Georgia, with 5·0 N
HCl at 91·2°C.

the extrapolated data obtained from the 60°C curves (6·10% Al_2O_3, 1·70% Fe_2O_3, 8·10% MgO) indicate 3·18 of the possible 5·00 octahedral positions are filled. The calculations also indicate excess Si is present; however it is unlikely the Si excess is enough to cause a major increase in the calculated octahedral cation population. The SiO_2 value would have to be lowered to approximately 45 per cent to allow for a calculated value of 4·0 filled octahedral positions. Approximately two octahedral positions are filled with the large Mg and Fe ions and one by the small Al ion. On the basis of the dissolution data and the strain induced by the difference in cation size it could be speculated that the Mg and Fe ions fill the two edge positions and the Al ion the center position, with the two intermediate positions being vacant. Charge deficiencies would be made up by H ions.

This is in accordance with the conclusion of Bradley (1940), who said that the distribution of Al ions among the five available octahedral positions in a chain can be accomodated either by vacancies in the octahedral positions or by some replacement of Si by Al. The former seems more likely for the present sample. When larger percentages of

Al and Fe than shown in the graphs were dissolved, the palygorskite structure collapsed and the assumption of constant volume of clay during the reaction became invalid. This break-up of the palygorskite is reflected by an increase in the slopes of the dissolution curves. As the slopes for the tetrahedral cations are lower than their corresponding octahedral cations as reported by Brindley and Youell (1951) and by Osthaus (1954), no evidence was obtained to suggest the presence of tetrahedral Al and Fe.

The results of Osthaus (1954, 1956) for montmorillonite and nontronite indicate approximately equal reaction rate constants for Mg, Al, and Fe. The slopes of the curves in Figs. 1–4 show that Mg and Fe have a higher reaction rate constant than Al. This would tend to substantiate the suggestion that the Al is concentrated in the interior positions and the Mg and Fe in the edge positions. As the reaction rate constants for Mg, Al, and Fe are identical for montomorillonite it could be concluded that they are distributed randomly in the octahedral positions.

Palygorskite was also digested with 2·5 N HCl at 70·3°C. The dissolution data for palygorskite with 2·5 N and 5·0 N HCl are plotted in Figs. 5 and 6.
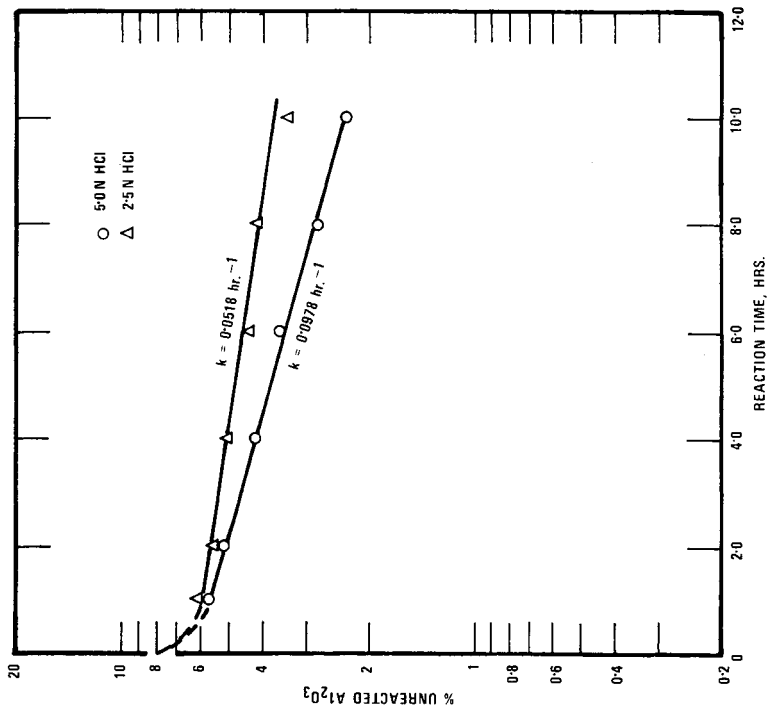
Fig. 6. Reaction of Al_2O_3 component of palygorskite. Attapulgus, Georgia, with HCl at 70·3°C.
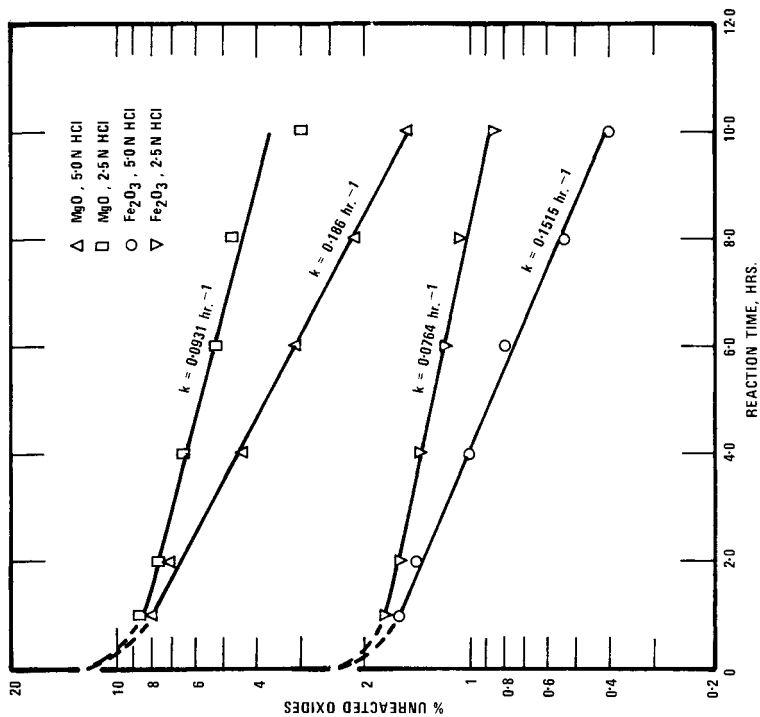
Fig. 5. Reaction of palygorskite. Attapulgus. Georgia. with HCl at 70·3°C.

For the corresponding cations, the slopes of the curves for 5·0 N HCl are about twice that for 2·5 N HCl. Therefore the reaction is also of first order with respect to acid concentration. This result is similar to that found by Osthaus (1954, 1956) for montmorillonite and nontronite and by Gastuche (1963) for biotite.

The first order rate equation states that the reaction rate is proportional to the concentration of the reactants as shown below:

$$\frac{dM}{dt} = -kM \qquad (1)$$

where M is the concentration of the undissolved metal ion, t the time and k is the apparent rate constant.

Integrating equation (1) between the limits of time t_1 and t_2 for the straight line portion of the curve, yields:

$$\ln\frac{M_1}{M_2} = k(t_2 - t_1) \qquad (2)$$

using common logarithms gives:

$$\log\frac{M_1}{M_2} = \frac{k(t_2 - t_1)}{2\cdot3}. \qquad (3)$$

Table 2 lists the apparent rate constants of attapulgite with 2·5 N and 5·0 N HCl at 70·3°C.

Table 2. Apparent rate constants of octahedral Mg, Al, and Fe in palygorskite with HCl at 70·3°C

HCl Concentration	Apparent rate constant (hr⁻¹)		
	Mg	Al	Fe
2·5 N	0·0931	0·0518	0·0764
5·0 N	0·186	0·0978	0·1515

As the reaction is also of first order with respect to acid concentration equation (1) can be rewritten to give account of the acid concentration as shown below:

$$\frac{dM}{dt} = -k^*AM \qquad (4)$$

where A is the acid concentration and k^* is the true rate constant, which is independent of acid concentration. From equations (1) and (4):

$$k = k^*A. \qquad (5)$$

Therefore, the true rate constant k^* is calculated by dividing the apparent rate constant k

by the acid concentration A in moles/l. The true rate constants for the octahedral cations in palygorskite are calculated at the four different temperatures employed.

From the Arrhenius equation

$$k = A\,e^{-E/RT} \qquad (6)$$

where k is the rate constant, hr^{-1}, A is constant, E is the activation energy in calories per mole, R is the gas constant in cal/deg/mole, and T is the absolute temperature in degrees Kelvin.

The logarithmic expression of equation (6) is

$$\log k = -E/2\cdot303\,RT + C. \qquad (7)$$

Therefore, a plot of the rate constant versus the reciprocal of the absolute temperature on a semilogarithmic paper gives a straight line with a slope equal to $-E/2\cdot303\,R$.

A plot of the true rate constants for the octahedral cations in palygorskite versus the reciprocal of the absolute temperature is shown in Fig. 7. This figure shows that the 3 straight lines for Mg, Al, and Fe are approximately parallel indicating equal activation energies for the above cations. The activation energy was calculated to be 18·4 kcal per mole. This value is essentially in the same range as that found by Osthaus (1956) for montmorillonite and nontronite (17–18 kcal per mole).

The acid-dissolution data for sepiolite (Asia Minor), obtained at 30·5°C for 1·0 N HCl, was used to make a semilogarithmic plot of the residual magnesium oxide versus time (Fig. 8). Unlike palygorskite the curve for sepiolite is a straight line throughout its extent. This indicates that all the magnesium ions are in the octahedral layer. The true rate constant at 30·5°C was calculated to be 0·553/hr. Compared to palygorskite, the rate constant for sepiolite is larger by a factor of about 240. This indicates that sepiolite is less stable to acid weathering than palygorskite.

Model for palygorskite and sepiolite reactions

Osthaus (1956) suggested that the acid-dissolution of a completely expandable mineral can be considered as essentially analogous to a liquid–liquid reaction. In the case of montmorillonite and nontronite, he suggested that the metal ions can migrate through the edges and particularly the surfaces of the clay platelets and that the solid–liquid boundary has little or no differential effect on the transfer of ions. Osthaus suggested that if the clays are not expandable or only partially expandable, the transfer of metal ions may take place only at the edges of the platelets, in which case the
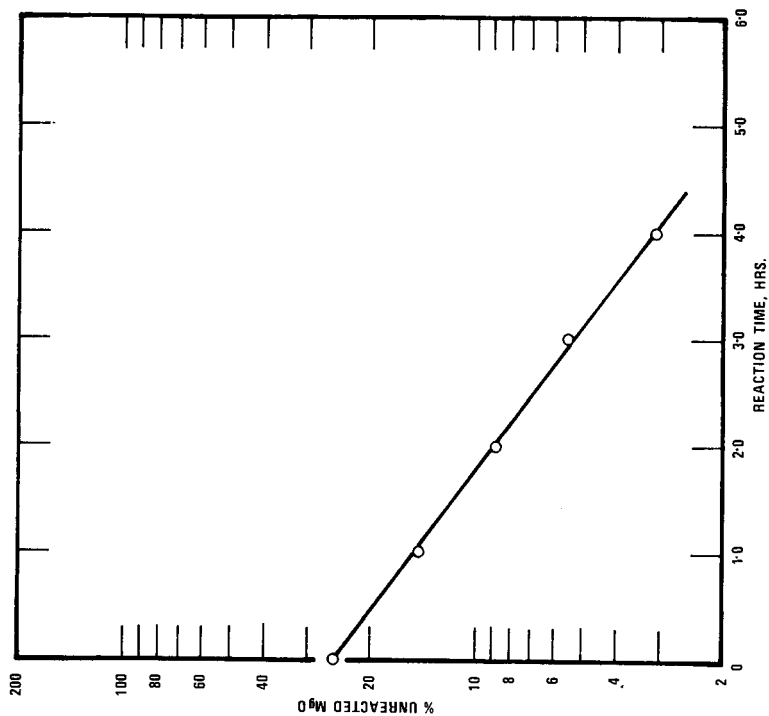
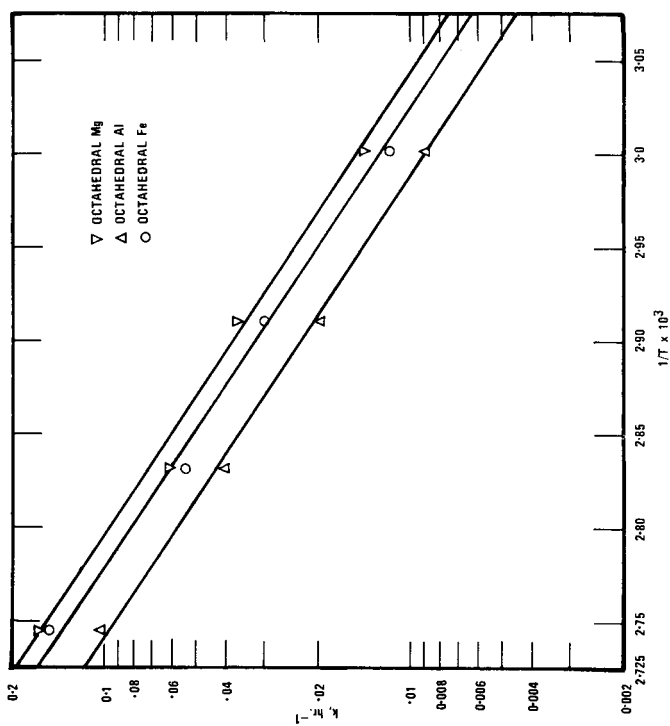Fig. 8. Reaction of sepiolite, Asia Minor, Turkey, with 1·0 N HCl at 30·5°C.

Fig. 7. Rate constant in HCl of octahedral Mg, Al and Fe in palygorskite, Attapulgus, Georgia, versus the reciprocal of the absolute temperature.

migration of ions would be affected by the rate of diffusion through the crystal structure.

As palygorskite and sepiolite are not expandable. a mechanism different from that for montmorillonite and nontronite must be operative.

The continuous reaction model may apply. It is assumed that the reactant fluid enters and reacts throughout the solid particle at all times. most likely with different rates at different locations within the particle. Thus. solid reactant is converted continuously and progressively throughout the particle. This model is reported by Levenspiel (1962) to be the most reasonable representation of the actual phenomenon for slow reaction of a very porous solid. The above requirements for the continuous reaction model seem to be satisfied for the acid-dissolution of palygorskite and sepiolite. as both minerals are highly porous due to the presence of open channels and the reaction is relatively slow at moderate temperatures.

Reversibility of the clay-acid reaction

Mering (1949) studied the activation of montmorillonites by treating them with hot concentrated acids. He found that when the octahedral ions had been largely removed and an amorphous product formed it was possible. by treating the residue with a solution containing magnesium ions. to reconstitute a montmorillonite-type product. He deduced that the Si–O network was not fully destroyed when the octahedral ions had been extracted.

Brindley and Youell (1951) likewise. found that it was possible to reconstitute the Si–(Al)–O networks from a chlorite into a mica-like product with a characteristic 10 Å spacing.

In this work, the reconstitution of palygorskite was examined. Each of the residues of the palygorskite digested with 5·0 N HCl at 70·3°C was divided into two portions. One portion was treated with very dilute sodium hydroxide solution (pH = 9). The mixture was placed in a stainless steel pressure bomb. which was heated in the oven at 110°C for 2 days. The other portion was treated under similar conditions with magnesium chloride

solution containing 1000 ppm Mg. The pH was also adjusted to 9 with sodium hydroxide. It was noticed that there is a small decrease in pH in the first case. while in the second case, the decrease in pH was quite appreciable. The X-ray intensities of the 10·5 Å spacing for the treated samples were compared with that of the initial residue. The results are shown in Table 3.

It is believed that the increase in the X-ray intensity of the 10·5 Å spacing after the NaOH treatment is mainly due to the dissolution of amorphous silica which was formed during the palygorskite acid reaction. The sodium ions are too large to enter in the octahedral sheet and occupy a vacant site. However, the large increase in the X-ray intensity of the 10·5 Å spacing after the $MgCl_2$ treatment could be interpreted to indicate there was some reconstitution and some Mg had entered the octahedral sheet. In the acid dissolution, the Mg, Al, and Fe ions in the octahedral sheet are supposed to be displaced by H ions.

If Mg ions are able to enter the octahedral sheet and displace the H ions an appreciable decrease in the pH of the solution would occur. That this has occurred is shown by the pH data in Table 3.

In another experiment. the original palygorskite was treated with $MgCl_2$ solution under the same conditions as above. It was found that the X-ray intensity of the 10·5 Å spacing increased from 56 to 90 (0·1 in.) after the $MgCl_2$ treatment. The residue was analysed and found to contain higher ratio of Mg than in the original sample. Only H ions were found in the reacting fluid. The initial pH was 9·4 and the final pH 7·5. It was calculated that the increase in Mg was equal to about 72 per cent of that needed to completely fill the vacant sites in the octahedral sheet. It seems quite likely that the Mg has entered the octahedral sheet.

The reconstitution of palygorskite is possible up to about 90 per cent dissolution of the octahedral cations. A skeleton framework must exist up this point. Complete dissolution of the octahedral cations resulted in destruction of the structure. The product was amorphous to X-ray, and it was not possible to reconstitute the material to palygorskite by treating it with $MgCl_2$.

Table 3. Reconsititution data

Time of treatment of palygorskite with 5 N HCl at 70°C (hr)	X-ray intensity. 10·5 Å (0·1″ units)			pH. NaOH-treatment		pH. MgCl₂ Treatment	
	Initial residue	After NaOH-treatment	After MgCl₂-treatment	Initial	Final	initial	final
2	35	50	69	9·3	8·5	9·0	6·2
4	15	28	47	9·2	8·9	9·0	6·2
8	13·5	18	36	9·2	8·6	9·0	6·1
10	9	14·5	20	9·0	8·4	9·0	6·2

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Résumé—De la palygorskite et de la sépiolite ont été digérées avec d'importants exédents d'acide chlorhydrique à une température constante pendant des périodes de temps variables. La réaction obtenue est apparue de premier ordre par rapport aux constituants de magnesium, d'aluminum et de fer présents dans l'argile et également par rapport à la concentration d'acide chlorhydrique.

Le taux constant de reaction pour Mg est relativement plus élevé que pour Fe, qui est lui-meme plus élevé que pour Al. Approximativement un tiers des cations du type octaedrique sont étrangers, suggérant par la que seules trois des cinq positions octaedriques sont occupées. Le taux constant de reaction de Mg dans la sépiolite est 240 fois supérieur à celui dans la palygorskite. Ceci peut indiquer que la sépiolite est moins stable aux altérations chimiques que la palygorskite. Les énergies d'activation pour Mg, Al et Fe dans la palygorskite paraissent sensiblement identiques et egales a environ 18,4 kcal par molécule-gramme.

Un traitement de palygorskite partiellement digérée par acide par des ions de magnesium à resulté en une augmentation d'intensité de rayon X dans l'écartement 10,5 Å, suggérant qu'une partie de la palygorskite a été reconstituée.

Kurzreferat—Palygorskit und Sepiolit werden bei konstanter Temperatur über verschiedene Zeiträume hinweg mit grossen Überschüssen von Salzsäure behandelt. Es wurde festgestellt, dass die Reaktion in Bezug auf die Magnesium-, Aluminum- und Eisenbestandteile im Ton, sowie in Bezug auf die Salzsäurekonzentration, erster Ordnung ist.

Die Reaktionsgeschwindigkeitskonstante für Mg ist grösser als für Fe. die wiederum grösser als für Al ist. Ungefähr ein Drittel der Kationen von oktaedrischem Typ sind äusserlich, was darauf hindeutet, dass nur drei der fünf oktaedrischen Stellungen bestzt sind. Die Reaktionsgeschwindigkeitskonstante des Mg im Sepiolit ist etwa 240 mal die für Palygorskit. Das mag ein Zeichen dafür sein, dass Sepiolit der chemischen Verwitterung gegenüber weniger beständig ist als Palygorskit.

Es wurde festgestellt, dass die Aktivierungsenergien für Mg, Al und Fe im Palygorskit gleich sind und circa 18,4 Grosskalorien pro Mol betragen.

Behandlung von teilweise mit Säure aufgeschlossenem Palygorskit mit Magnesiumionen unter basischen Bedingungen führte zu einer Zunahme in der Röntgenintensität des 10,5 Å Abstandes, was auf eine teilweise Rückbildung von Palygorskit hindeutet.

Резюме—Палыгорскит и сепиолит подверглись различной по продолжительности обработке соляной кислотой (в большом избытке) при постоянной температуре в течение разных периодов времени. Установлено, что взаимодействие протекало по реакции первого порядка по отношению к магнезиальному, алюминиевому и железному компонентам и по отношению к концентрации соляной кислоты. Константа скорости реакции для магния больше, чем для железа и для железа больше, чем для алюминия. Приблизительно одну треть катионов октаэдрического типа можно отнести к посторонним; это позволяет предполагать, что заполнены только три из пяти октаэдрических позиций. Константа скорости реакции магния в сепиолите приблизительно в 240 раз больше, чем в палыгорските, что может служить указанием на меньшую химическую устойчивость сепиолита по сравнению с палыгорскитом к химическому выветриванию. Энергии активации для магния, алюминия и железа в палыгорските оказались одинаковыми и равными 18,4 килокалорий на моль.

После частичного разложения палыгорскита кислотой, его обработка щелочными растворами с ионами магния вызвала увеличение интенсивности рефлекса при 10,5 А на порошкограмме; это указывает на частичное восстановление стуруктуры палыгорскита.