THE HYDROTHERMAL TRANSFORMATION OF SEPIOLITE TO STEVENSITE AND THE EFFECT OF ADDED CHLORIDES AND HYDROXIDES

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Abstract—Hydrothermal reactions in the system sepiolite/H_2O have been examined between 149° and 316°C. Approximately 10–20% of the starting sepiolite was converted to a smectite (stevensite) at 204°C within 24 hr. Similar results were obtained when $CaCl_2$, NaOH, $Ca(OH)_2$, or $Mg(OH)_2$ was added to the system. In the presence of NaCl, about 60% of the sepiolite was converted to stevensite, whereas, only 5% stevensite formed in the presence of $MgCl_2$. Greater amounts of stevensite formed at 260°C in these systems. Above 316°C, 60–80% of the sepiolite was converted to stevensite in 24 hr, regardless of the presence or absence of salts. Within the experimental conditions used, temperature is the most important factor in the sepiolite-to-stevensite conversion.

At or below 216°C, sepiolite appears to transform into stevensite by dislocations involving $c/2$ glides that are triggered by the stresses of the hydrothermal conditions. Above this temperature, stevensite seems to form by direct precipitation after dissolution of sepiolite.

Key Words—Drilling Fluids, Hydrothermal Stability, Sepiolite, Smectite, Stevensite.

INTRODUCTION

Sepiolite-based drilling fluids have been suggested for use at the extreme temperatures and pressures encountered in deep-well drilling (Carney and Meyer, 1976). The conversion of sepiolite to smectite under these conditions may favorably alter the rheological properties of the drilling fluid. Using both a natural sepiolite and a synthetic gel of sepiolite composition, Mumpton and Roy (1958) obtained a magnesium smectite between 200° and 350°C and under 1379 bars water pressure in 8 hours to 3 weeks time. Frank-Kamenetsky *et al.* (1970, 1972) and Otsuka *et al.* (1974), however, found sepiolite to be hydrothermally stable to 325°C, and that a "hydrotalc" or "hydrated talc" phase with a basal spacing of 9.6 Å was produced above this temperature. Furthermore, Frank-Kamenetsky *et al.* (1970, 1972) found that sepiolite was stable up to 400°C in the presence of NaCl, up to 350°C in the presence of $CaCl_2$, and up to 300°C in the presence of $MgCl_2$.

The magnesium smectite obtained by Mumpton and Roy (1958) was obviously not a saponite per se, but stevensite, the Al-free member of the trioctahedral smectite group, since by definition, significant amounts of Al are present in saponite. Stevensite was proposed as a distinct species by Faust and Murata (1953). Type-locality material from Springfield, New Jersey (U.S. National Museum #R4719) was later examined by Brindley (1955) who pointed out the existence of superlattice reflections at 25 Å that had been missed by the previous investigators. Faust *et al.* (1959) then reexamined the Springfield sample and confirmed the

presence of superlattice reflections, attributing them to the coexistence of talc domains (without octahedral vacancies) and talc-*like* domains (with octahedral vacancies). Thus, the concept of stevensite as a defect trioctahedral magnesium smectite with octahedral vacancies was developed. The close chemical relationship between stevensite and sepiolite was pointed out by Randall (1959) and Imai *et al.* (1970).

To clarify the hydrothermal stability of sepiolite and to provide information on the mechanism of conversion of sepiolite to stevensite, a series of experiments was carried out under deep-well drilling conditions. Because fluids are commonly contaminated with salts of alkali and alkaline earth metals from the penetrated sedimentary formation, similar experiments were conducted in which known amounts of NaCl, $CaCl_2$, $MgCl_2$, NaOH, $Ca(OH)_2$, and $Mg(OH)_2$ were added to the system.

MATERIALS AND METHODS

Sepiolite starting material

The sepiolite sample used in the experiments was obtained from the Aschenbrenner deposit, Nye County, Nevada, courtesy of Industrial Mineral Ventures Company, Golden, Colorado. The deposit is located 9 miles northeast of Death Valley Junction, California, and 3 miles due south of Devils Hole as indicated on the U.S.G.S. map NJ 11-11: Death Valley, California-Nevada. The sample was obtained from a depth of 12.5–13.0 m at the company's grid location of 12S/8E by Jack

Table 1. Chemical composition of the Aschenbrenner sepiolite after the ignition to 1000°C.

	Wt. %
SiO_2	67.1
Al_2O_3	2.70
Fe_2O_3 (total iron)	0.66
MgO	26.50
CaO	1.48
Na_2O	0.32
K_2O	0.55
Li_2O	0.11
TOTAL	99.42
(Loss on ignition: 21.91%)	

Mayhew, resident geologist. The only impurity detectable by X-ray powder diffraction was about 2–3% dolomite. The chemical composition of the sample is listed in Table 1 and was determined by atomic absorption spectroscopy as described by Lee and Güven (1975). The X-ray powder diffraction patterns of oriented slides gave strong 001 reflections characteristic of sepiolite in the air-dried state at 12.14 Å, under 40% relative humidity and at room temperature. This reflection showed a slight but reproducible expansion to 12.28 Å when the sample was saturated with ethylene glycol. A 0.1 to 0.7 Å expansion of sepiolite with ethylene glycol was consistently observed by Powe (1977). Transmission electron microscopic (TEM) examination shows that the Aschenbrenner sepiolite occurs as individual fibers up to 2 µm in length and from 200 to 300 Å in width and as birdnest-like aggregates of fibers (see Figures 1a and 1b).

Methods of analysis

Hydrothermal experiments were carried out in a specially designed high-temperature/high-pressure autoclave at the Chemical Research Laboratory of Halliburton Services in Duncan, Oklahoma. One hundred milliliters of a 4% sepiolite suspension in distilled and deionized water was heated for 24 hr between 149° and 316°C under pressure from 14 to 1379 bars. In other runs, 1 g of the chlorides and hydroxides of Na, Ca, and Mg per 4 g of sepiolite was added to the suspension. All products were centrifuged with water and dialyzed to remove excess salts.

The products were then examined by X-ray powder diffraction using a Philips Norelco X-ray diffractometer with Ni-filtered $CuK\alpha$ radiation, 1° divergence and scatter slits, a 0.005″ receiving slit, and a scanning speed of 1° 2θ/min. Oriented clay films were air-dried at room temperature under 40% relative humidity. The measured d-values were not corrected for Lorentz-polarization factors. For semiquantitative estimation of the relative amount of sepiolite and stevensite in products, mixtures of the starting sepiolite and a Cheto,
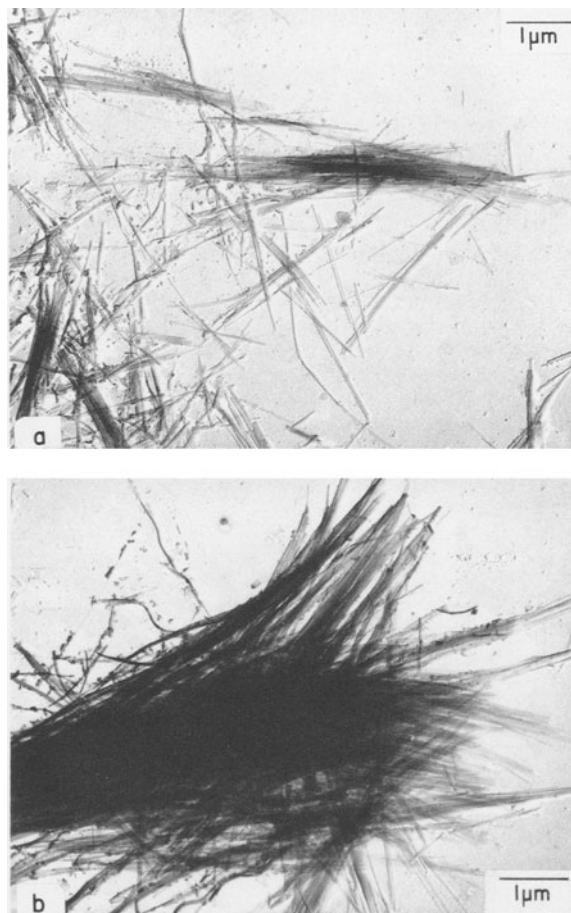

Figure 1. Transmission electron images of the original sepiolite: (a) individual sepiolite fibers, and (b) a typical birdnest-like aggregate of fibers.

Arizona, montmorillonite were prepared at 10% intervals. The Cheto material was chosen because of its morphological similarity to the stevensite formed in the experiments. Glycol-saturated samples were prepared for the semiquantitative estimates, and the 011 and 001 reflections of sepiolite and stevensite, respectively, were compared.

Electron microscopic studies were made with a JEM-7 transmission electron microscope with accelerating voltages of 80 and 100 kV. Small drops of very dilute suspensions were air-dried on 200-mesh copper grids that had been previously coated with Formvar films from a 0.3% Formvar solution in chloroform. The grids were then coated with gold and carbon.

EXPERIMENTAL RESULTS

The results of all runs in the system sepiolite/water and the systems containing chlorides and hydroxides of Na, Ca, and Mg are listed in Table 2. The significant portions of the X-ray powder diffraction patterns of key
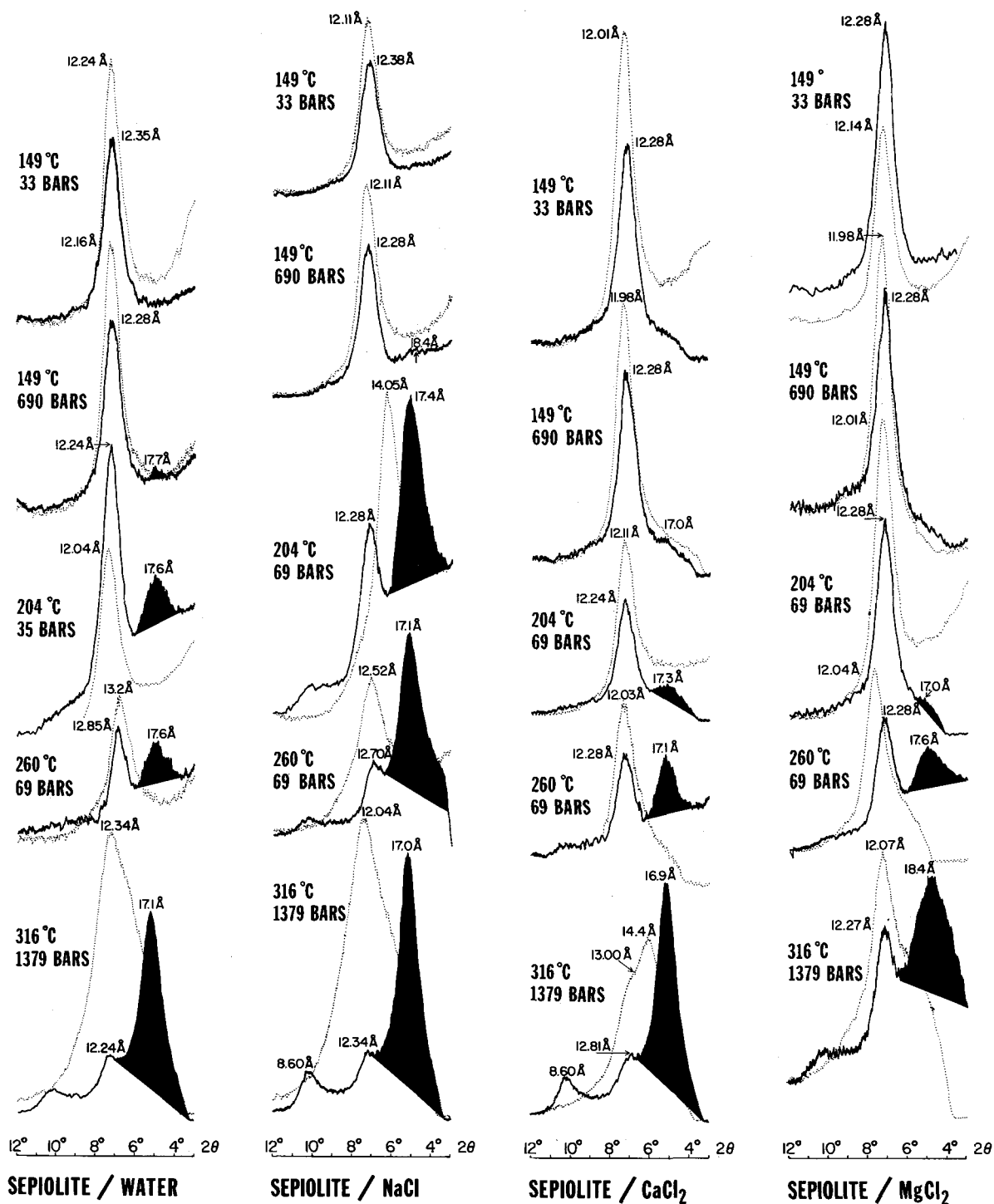
Figure 2.   X-ray powder diffraction patterns of the sepiolite after various hydrothermal treatments. The pattern with dotted lines was obtained from the sample in air-dried state. The pattern with full lines was obtained from the glycol saturated sample, where the smectite's 17 Å reflection is represented by shaded areas.

Table 2.   Hydrothermal treatments of sepiolite fluids.

Temperature/Pressure		Sepiolite d(011) (Å) before and after glycolation		% smectite in product
°F/psi	°C/bars			
Sepiolite/H$_2$O System				
300/475	149/33	12.24	12.35	[2]
300/10,000	149/690	12.16	12.28	5
400/500	204/35	[1]	12.28	20
500/1000	260/69	[1]	12.85	40
600/20,000	316/1379	[1]	12.24	80
Sepiolite/NaCl System				
300/475	149/33	12.11	12.38	[2]
300/10,000	149/690	12.11	12.28	[3]
400/1000	204/69	[1]	12.28	60
500/1000	260/69	[1]	12.70	70
600/20,000	316/1379	[1]	12.34	80
Sepiolite/NaOH System				
300/200	149/14	11.90	12.14	[2]
400/500	204/35	[1]	12.38	20
500/1000	260/69	[1]	12.84	70
Sepiolite/CaCl$_2$ System				
300/475	149/33	12.01	12.28	[2]
300/10,000	149/690	11.98	12.28	[3]
400/1000	204/69	[1]	12.24	20
500/1000	260/69	[1]	12.28	50
600/20,000	316/1379	[1]	12.81	80
Sepiolite/Ca(OH)$_2$ System				
300/200	149/14	12.01	12.28	[2]
400/500	204/35	[1]	12.28	10
500/1000	260/69	[1]	12.27	30
Sepiolite/MgCl$_2$ System				
300/475	149/33	12.14	12.28	[2]
300/10,000	149/690	11.98	12.28	[2]
400/1000	204/69	[1]	12.28	5
500/1000	260/69	[1]	12.28	40
600/20,000	316/1379	[1]	12.27	60
Sepiolite/Mg(OH)$_2$ System				
300/200	149/14	12.14	12.28	[2]
400/500	204/35	[1]	12.28	10
500/1000	260/69	[1]	12.55	70

[1] Sepiolite and smectite reflections overlap.
[2] Not detectable with X-ray powder diffraction.
[3] Present but at a level below 5%.

products are shown in Figure 2. Stevensite was not detected in runs made at 149°C and 33 bars in the sepiolite/H$_2$O system with or without the addition of chlorides. Similar results were obtained in the sepiolite/hydroxide systems at 149°C and 14 bars. When the pressure was increased to 690 bars at 149°C, very small quantities (5% or less) of smectite formed in the sepiolite/H$_2$O system and in the sepiolite/NaCl and sepiolite/CaCl$_2$ systems. As indicated in Figure 2, these smectite products gave broad, weak reflections at 17–18 Å. No smectite, however, formed at 149°C and 690 bars in the sepiolite/MgCl$_2$ system. Thus, increasing pressure at 149°C had little or no effect on the sepiolite-to-stevensite conversion.

At 204°C, appreciable amounts of smectite formed in the sepiolite/NaCl system. At 260°C and 69 bars, 40–70% of the sample was converted to stevensite. When the pressure was raised to 1379 bars in the sepiolite/CaCl$_2$ system, similar results were obtained. These data again show that pressure had only minor influence on the conversion of sepiolite to stevensite in these hydrothermal systems. At 260°C, a strong reflection was noted at 12.70 Å in the X-ray pattern of the sepiolite/NaCl product and at 12.85 Å in the pattern of the sepiolite/H$_2$O product. A similar reflection was noted at 12.81 Å for the sepiolite/CaCl$_2$ product at 316°C and 1379 bars, but not for the corresponding sepiolite/MgCl$_2$ product (Figure 2). Higher order reflections were
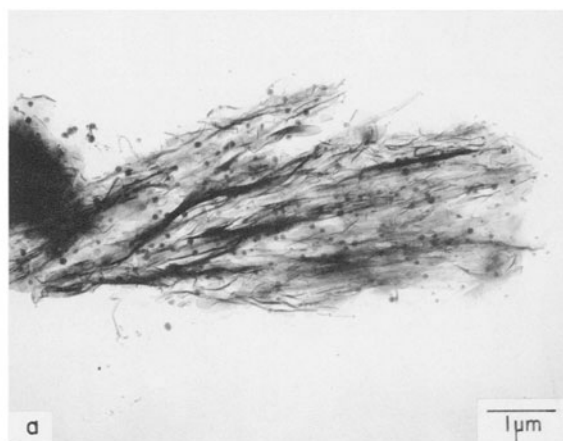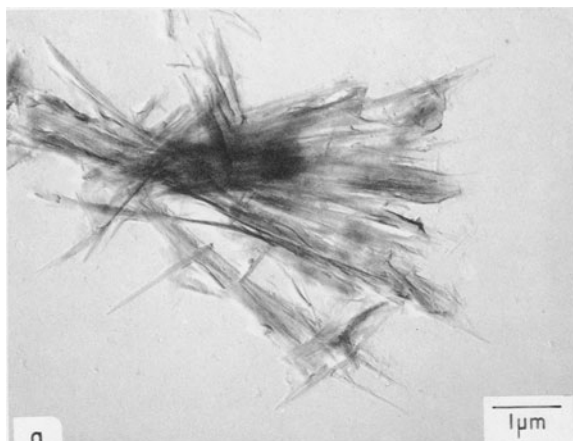
Figure 3. Typical smectite particles formed during the hydrothermal treatment at 260°C and 69 bars (a) in the system sepiolite/CaCl$_2$ and (b) in the system sepiolite/NaCl.

Figure 4. Typical smectite aggregates formed during the hydrothermal treatments at 316°C and 1379 bars (a) in the system sepiolite/CaCl$_2$, and (b) in the system sepiolite/NaCl.

not observed, and the 12.7–12.8 Å reflection does not seem to have been caused by particle-size effects. In fact, electron microscopic examination of these products showed no observable decrease in the size of the sepiolite fibers. No explanation can be offered for this reflection, although it may be the result of a sepiolite/stevensite mixed-layering. At 316°C and 1379 bars, most of the sepiolite was converted to stevensite independent of whether or not chlorides or hydroxides were present in the system.

Hydrothermal treatment of 149°C caused no detectable change in the morphology or structure of the sepiolite starting material as indicated by transmission electron microscopy. Discrete particles of smectite were not observed even when the pressure was raised to 690 bars. At 260°C and 69 bars, however, typical smectite particles could be seen in the hydrothermal products in the sepiolite/CaCl$_2$ and sepiolite/NaCl systems as shown in Figures 3a and 3b, respectively. Here the transition from the original fibrous morphology of

sepiolite to the flaky (lamellar) morphology of smectite can be seen. The original fiber outlines are still visible, and the fibers do not seem to have been dissolved. Similar smectites were formed during the treatments at 204°C.

The smectites formed by hydrothermal treatment of sepiolite at 316°C and 1379 bars are morphologically different from those described above. These smectites occur as irregular, flaky aggregates (Figures 4a and 4b) in which the original fiber forms are not discernible. These irregular flakes were probably formed by direct precipitation after dissolution of the parent sepiolite. A large number of spherical particles of silica(?) associated with the stevensite products can also be seen in Figures 4a and 4b.

MECHANISMS OF THE SEPIOLITE-TO-STEVENSITE CONVERSION

The experimental evidence presented here on the hydrothermal stability of sepiolite and subsequent for-
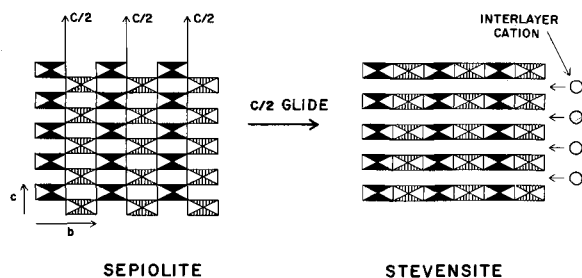
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Figure 5.   Schematic representation of sepiolite structure as projected along the *a*-axis (parallel to fiber direction) and its transformation to stevensite by the *c* glide mechanism.

Figure 6.   Distribution of the octahedral cations in sepiolite structure and formation of vacancies, ⊡, following the *c* glides in stevensite structure.

mation of stevensite allows a model to be proposed for the transformation of a chain silicate to a layer silicate and also provides information on the nature of the defects in the structure of stevensite.

The crystal structure of sepiolite is shown schematically in Figure 5 as projected along the fiber direction. In accordance with Zvyagin *et al.* (1963), the crystallographic *a*-axis of sepiolite is set parallel to the fiber direction. The rectangular envelope-like blocks in Figure 5 represent the projections of talc-like ribbons in the sepiolite structure. The hatched and dark triangles indicate the directions in which the silica tetrahedra point in these ribbons. The sepiolite structure may thus be described as talc-like ribbons that are displaced with respect to each other by a *c*/2 glide. As these ribbons are attached to each other only by Si–O–Si bonds at their four corners, the sepiolite structure is a rather delicate one. At higher temperature and pressure the ribbons may change to a more stable configuration by undergoing a *c*/2 glide, e.g., in a series of dislocations, and by establishing additional bonds between their octahedral networks. Alternatively, sepiolite may break down into talc-like ribbons, e.g., by partial dissolution, which in turn may reassemble in the same manner as achieved by *c*/2 glides. As indicated in Figure 6, the new phase, formed by either mechanism, cannot be talc. The magnesium octahedra in the talc-like ribbons are located at z = ¼ in one ribbon and at z = ¾ at the next ribbon in the sepiolite structure. Magnesium positions along the edges of the ribbons at z = ¼ are designated by M1 and M2, and those along the edges of the ribbon at z = ¾ are similarly designated by M1′ and M2′. Each of the M1 and M1′ metals have two water ligands, whose positions are marked by X in the *c*-projection of the structure (Figure 6). After the *c*/2 glides (or when the ribbons are reassembled), the H_2O ligands superimpose, and a reaction such as that formulated in Figure 7 should occur, thereby generating layer charge in the smectite structure. Depending on the number of water molecules that undergo this reaction, the sepiolite-to-stevensite conversion can be formulated as:
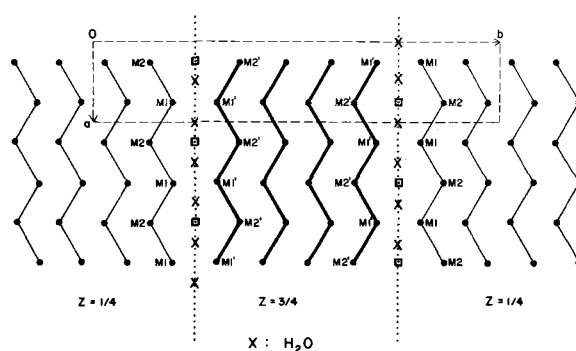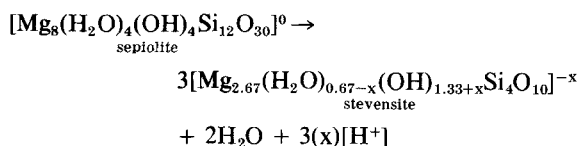
$$[Mg_8(H_2O)_4(OH)_4Si_{12}O_{30}]^0 \rightarrow$$
$$\text{sepiolite}$$

$$3[Mg_{2.67}(H_2O)_{0.67-x}(OH)_{1.33+x}Si_4O_{10}]^{-x}$$
$$\text{stevensite}$$

$$+ 2H_2O + 3(x)[H^+]$$

where the zeolitic water of sepiolite is disregarded. If all of the crystalline water ligands become hydroxyls, the layer charge of stevensite will reach a maximum value of +0.67 per formula unit. In addition, certain octahedral positions (Figure 6) will be vacant. These octahedral defects are located on the *c* glide plane and make up $^1/_9$ of the total octahedral positions. Additional magnesium to fill these positions can be supplied only by a decomposition of some of the sepiolite in the system. Such a decomposition would also generate free silica. Additional magnesium in stevensite (i.e., when Mg is greater than 2.67 in the formula) decreases its layer charge. For example, about half of the vacant positions are filled in the structure of stevensite from Springfield, New Jersey, in which the total octahedral occupancy is 2.92 per formula unit (Faust and Murata, 1953). As these vacant octahedral positions fill with magnesium, talc domains form within the stevensite structure, and may be randomly mixed with smectite domains within a layer. Also, the talc domains may be randomly stacked in successive layers in the form of mixed-layering. This situation continues until all of the vacant octahedral sites are filled and talc is formed.

The smectites shown in Figures 3a and 3b were probably formed by the proposed *c*/2 gliding mechanism. Here, the transition from fibrous morphology to a lamellar one is well displayed. The formation of irregularly folded smectite flakes, however, such as those in Figures 4a and 4b, is probably related to a direct precipitation of smectite after total dissolution of the sepiolite. In fact, when the temperature of runs was raised to 316°C, most of the smectite seemed to form by the dissolution-reprecipitation mechanism, independent of additives.
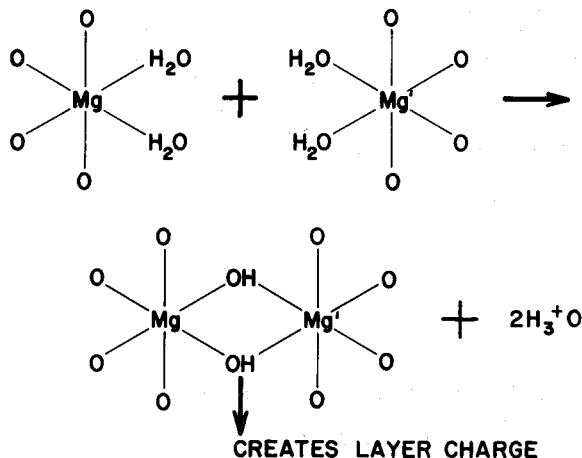
CREATES LAYER CHARGE

Figure 7. Reaction mechanism between the magnesium octahedra along the edges of talc-like ribbons after c glides (magnesium at the edge of one ribbon is designated by Mg and magnesium at the edge of the other ribbon is designated by Mg').

CONCLUSIONS

(1) The effect of pressure within the range applied in this study is negligible for the conversion of sepiolite to stevensite.

(2) Temperature is the major factor in sepiolite-to-stevensite conversion within the period of 24 hr. Appreciable amounts (>10%) of smectite first develop at 204°C within this period. At 316°C, most of the sepiolite converts to smectite regardless of the presence or absence of salts or hydroxides, demonstrating the paramount importance of temperature in this converson.

(3) Chlorides of Na and Ca are more effective than their hydroxides in converting sepiolite to smectite in temperatures up to and including 260°C; however, $Mg(OH)_2$ promotes the conversion more than does $MgCl_2$. NaCl is by far the most effective agent in this conversion up to 260°C.

(4) In fresh water, sepiolite converts to smectite as easily as it does in the presence of calcium.

The present work found that sepiolite converts hydrothermally to smectite at 204°C, in agreement with the results by Mumpton and Roy (1958). Discrepancies exist between the results of this study and those by Frank-Kamenetsky *et al.* (1970, 1972) and by Otsuka

et al. (1974) (*vide supra*). At the present time no explanation can be offered for these discrepancies.

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Резюме—Изучались гидротермальные реакции в системе сепиолит/H_2O при температуре от 149° до 316°C. При 204°C в течение 24 часов примерно 10–20% начального сепиолита было превращено в смектит (стевенсит). Аналогичные результаты были получены при добавлении $CaCl_2$, NaOH, $Ca(OH)_2$, или $Mg(OH)_2$ в систему. В присутствии NaCl около 60% сепиолита было преобразовано в стевенсит, в то время как в присутствии $MgCl_2$ образовалось только 5% стевенсита. Большее количество стевенсита в этих системах было получено при 260°C. При температурах, превышающих 316°C в течение 24 часов 60–80% сепиолита преобразовывалось в стевенсит независимо от присутствия или отсутствия солей. Было установлено, что при соблюдавшихся условиях экспериментов, температура является наиболее важным фактором в преобразовании сепиолита в стевенсит.

При температуре 216°C или ниже сепиолит, повидимому, переходит в стевенсит в результате перемещений, включающих скольжения $c/2$, которые возникают под влиянием гидротермальных условий. При более высоких температурах стевенсит, повидимому, формируется путем непосредственного осаждения после растворения сепиолита.

Resümee—Hydrothermische Reaktionen im Sepiolit/Wasser System sind zwischen 149° bis 316°C untersucht worden. Ungefähr 10–20% des eingesetzten Sepiolit wurde innerhalb von 24 Stunden bei 204°C zu einem Smektit (Stevensit) umgesetzt. Ähnliche Resultate wurden erhalten, als $CaCl_2$, NaOH, $Ca(OH)_2$, oder $Mg(OH)_2$ zu dem System gegeben wurden. In Gegenwart von NaCl ungefähr 60% des Sepiolit wurde zu Stevensit umgesetzt, wohingegen nur 5% Stevensit in der Gegenwart von $MgCl_2$ geformt wurden. Größere Mengen von Stevensit wurden bei 260°C in diesen Systemen geformt. Über 316°C, 60–80% des Sepiolit wurde innerhalb von 24 Stunden umgesetzt zu Stevensit, ob Salze anwesend waren oder nicht. Im Rahmen der angewendeten Bedingungen ist Temperatur der wichtigste Faktor in der Umwandlung von Sepiolit zu Stevensit.

Bei 216°C und darunter scheint das Sepiolit in Stevensit überzugehen durch Verschiebungen, die $c/2$ Verrutschungen einbeziehen, welche durch Spannungen von den hydrothermischen Bedingungen ausgelöst werden. Über dieser Temperatur scheint Stevensit durch direktes Ausfallen nach Auflösen von Sepiolit geformt werden.

Résumé—Les réactions hydrothermales dans le système sépiolite/H_2O ont été examinées entre 149° et 316°C. Approximativement 10–20% de la sépiolite d'origine a été convertie en une smectite (stevensite) à 204°C en 24 heures. Des résultats semblables ont été obtenus lorsque $CaCl_2$, NaOH, $Ca(OH)_2$, ou $Mg(OH)_2$ étaient ajoutés au système. En présence de NaCl, environ 60% de la sépiolite a été convertie en stevensite, alors que seulement 5% de stevensite était formée en présence de $MgCl_2$. De plus grandes quantités de stevensite ont été formées à 260°C dans ces systèmes. Au dessus de 316°C, 60 à 80% de la sépiolite a été convertie en stevensite en 24 heures, sans égards à l'absence ou à la présence de sels. Sous les conditions expérimentales employées, la température est le facteur le plus important dans la conversion de sépiolite en stevensite.

A 216°C ou en dessous, la sépiolite a l'air de se transformer en stevensite par dislocations impliquant des glissements $c/2$ qui sont mis en mouvement par les forces des conditions hydrothermales. Au dessus de cette température, la stevensite semble se former par précipitation directe après la dissolution de la sépiolite.