# HYDROTHERMAL REACTIVITY OF SAPONITE

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Abstract—Saponite crystallizes from amorphous gel having an ideal saponite composition within 7 days at all experimental temperatures between 300° and 550°C at 1 kbar pressure. Reactions subsequent to this initial crystallization vary in type and degree, depending on the temperature of reaction and the type of interlayer cation. Above 450°C, the initially crystallized K-saponite dissolves, and talc and phlogopite nucleate and grow as discrete phases. At 450°C the initial K-saponite reacts to form talc and phlogopite layers, but the reaction proceeds via intracrystalline layer transformations rather than via dissolution and precipitation, producing a mixture of fully ordered, interstratified talc/saponite and fully ordered saponite/phlogopite. The K-saponite shows subtle signs of reaction at 400°C after 200 days: this temperature is at least 150°C lower than experimental reaction temperatures observed below 400°C. K-saponite reacts more rapidly than either Na-saponite on Ca-saponite above 400°C, and the Na-saponite and Ca-saponite produce no mica layers during their transformation to mixed-layer clays. Interstratified talc/saponite formed in the Na-saponite system, and the Ca-saponite system produced both talc/saponite and chlorite/saponite.

Key Words—Chlorite/saponite, Hydrothermal stability, Potassium, Saponite, Saponite/phlogopite, Talc/ saponite.

## INTRODUCTION

Saponite is the most abundant of the trioctahedral smectites and is found in a number of Mg-rich geologic environments. For example, it is an important mineral in ocean basins, where it forms as both a high- and a low-temperature alteration product of basalt. During the seawater alteration of basalt, saponite forms both as a discrete phase and as a component in mixed-layer clays (Andrews, 1980; Banks, 1972; Bass, 1976; Humphris and Thompson, 1978; Kristmannsdottir, 1976, 1978; Scheidegger and Stakes, 1977; Sevfried et al., 1978). Judging from the magnitude of the submarine alteration processes and their influence on ocean chemistry (e.g., Wolery and Sleep, 1976), the reactivity of saponite is important and must be understood over a broad range of temperatures if we are to understand the reactions in the ocean basins which affect the seawater chemistry.

The purpose of this study was to investigate the nature and extent of the reactions of iron-free saponite under experimental hydrothermal conditions. Although nearly all natural saponites contain some iron, the iron-free system was studied as a first step in understanding the reactivity of saponite under relatively simple conditions. Saponite has been synthesized previously, but the earlier experimental studies dealt only with saponites containing sodium, magnesium, calcium, or lead as the interlayer cation (Ames and Sand, 1958; Mumpton and Roy, 1956; Koizumi and Roy, 1959; Iiyama and Roy, 1963a, 1963b). The K-saponite system was chosen for study because potassium is a key element in many natural saponite-rich environments, and because no previous experimental studies of saponite included potassium as an interlayer cation. Some Nasaponite and Ca-saponite runs were also made to provide a comparison with reactions observed in previous experiments.

## EXPERIMENTAL TECHNIQUES

The starting material for all experimental runs was a noncrystalline gel, prepared by the method of Hamilton and Henderson (1968). The cation ratios in the gel correspond to the ideal saponite formula,  $Mg_3Si_{3.67}Al_{0.33}O_{10}(OH)_2 \cdot M_{0.33}^+$ , where M is K<sup>+</sup>, Na<sup>+</sup>, or  $\frac{1}{2}Ca^{2+}$ . Each run was prepared by putting 30 mg of gel into a gold tube containing 30  $\mu$ l of triply distilled, deionized water. Each capsule was welded shut and weighed before and after treatment to detect leaks. The samples were heated in rod-type reaction vessels in horizontal resistance furnaces. Temperatures were maintained within 5°C of the reported temperatures by on-off controllers.

The reaction paths were monitored by allowing samples of each gel composition to react for progressively longer periods of time at each temperature. Each run represents a unique combination of composition, temperature, and time, and no sample was subjected to further hydrothermal treatment once it had been removed for examination. Runs were made for each composition at 300°, 350°, 400°, 450°, 500°, and 550°C for 7, 30, 50, 120, and 200 days, except that no runs were made above 450°C for the Na- and Ca-saponite compositions. Pressure was held constant at 1 kbar for all runs. Table 1 lists all of the run conditions and run products.

The run products were gently disaggregated in water with a mortar and pestle, pipetted onto glass slides, and examined by X-ray powder diffraction (XRD) using Nifiltered CuK $\alpha$  radiation. Samples were X-rayed after air

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Figure 1. X-ray powder diffraction patterns of the products of seven-day runs of the K-saponite gel treated hydrothermally at the temperatures indicated. These products represent the starting material for the longer runs. All X-ray powder diffraction patterns are of glycolated samples,  $CuK\alpha$  radiation.



Figure 2. X-ray powder diffraction patterns of K-saponite, Ca-saponite, and Na-saponite after 200 days at 400°C. This was the lowest temperature that any reaction was observed beyond the crystallization of saponite. All samples are glycolated. S = saponite, T/S = mixed-layer talc/saponite. CuK $\alpha$ radiation.

drying and after equilibration with ethylene glycol vapor. XRD patterns of mixed-layer minerals were calculated using a modified version of the computer program of Reynolds and Hower (1970).

### RESULTS

The reaction of the gel-water mixture proceeded in two stages. The first stage was the crystallization of the amorphous starting materials to saponite. Except for the highest-temperature run (550°C), all samples of the K-saponite gel crystallized to a pure K-saponite after seven days (Figure 1). At 550°C the products contained an unidentified, possibly mixed-layer phase.

Subsequent to the initial crystallization, the K-saponite underwent progressive alteration at temperatures of 400°C or higher. The nature and extent of the reactions were temperature dependent. For example, at 400°C reactions were quite subtle, producing only a small amount of mixed-layer talc/saponite in addition to the discrete saponite (Figure 2). At 450°C the reac-

Table 1. Hydrothermal run results.<sup>1</sup>

Sample	Т (°С)	Time (days)	Run products			
K-saponite gel						
S-1	450	7	sap			
S-2	500	7	sap + t/s (?)			
S-3	550	7	sap + t/s (?)			
S-4	300	30	sap			
3-3 8-6	400	7	sap			
S-0	350	7	sap			
S-8	500	50	sap			
S-9	550	50	t + p + sap			
S-10 <sup>2</sup>	150	50	sap (poorly crystalline)			
S-11 <sup>2</sup>	150	120	sap			
S-12 <sup>2</sup>	150	200	sap			
S-13	450	7	sap			
S-14	450	7	sap			
S-15	450	20	sap			
5-10	350	30	sap			
S-17 S-18	550	30	sap sap + t + p			
S-19	300	50	sap			
S-20	300	120	sap			
S-21	550	120	t + p + sap			
S-22	300	200	sap			
S-23	350	50	sap			
S-24	350	120	sap			
S-25	350	200	sap			
S-20 S-27	400	 120	sap			
S-27 S-28	400	200	sap + t/s			
S-29	450	50	$\frac{1}{1/8} + 2$			
S-30	450	120	t/s + p/s			
S-31	450	200	t/s + p/s			
S-32	500	120	t + sap + p			
Ca-sapon	ite gel					
611	350	50	sap			
612	350	120	sap			
613	350	200	sap			
614	400	50	sap			
621	400	120	sap			
622	300	120	sap			
624	300	200	sap			
631	400	200	sap			
632	450	120	sap + t/s			
633	450	120	sap + t/s			
634	450	200	sap + t/s + c/s			
641	350	75	sap			
643 644	400 450	/5 75	sap sap + t/s			
0.11	.50	75	Sup 1 45			
Na-sapon	ite gel					
811	350	50	sap			
812	350	120	sap			
813	350	200	sap			
014 871	400	120	sap			
822	300	50	$sap \pm vs(i)$			
823	300	120	sap			
824	300	200	sap			
831	400	200	sap + t/s			
832	450	50	sap + t/s			
833	450	120	sap + t/s			
834	450	200	sap + t/s			
041 847	330 400	15	sap			
843	450	75	sap sap			
· · ·			July			

tion was pronounced, even after only 30 days, as indicated by the shift in peak positions (Figure 3). After 200 days the reaction products include two types of mixed-layer clay: a fully ordered (R = 1) talc/saponite with 66% talc layers, and a fully ordered (R = 1) phlogopite/saponite with 20% phlogopite layers (Figure 4). The formation of this complex mixture of mixed-layer clays occurred only at 450°C within the 200-day duration of the experiments.

At 500° and 550°C the K-saponite dissolved, and discrete talc and phlogopite nucleated and grew (Figure 5). The abundant, low-angle scatter around the 17-Å saponite peak indicates that a mixed-layer intermediate phase may have formed, but it could not be characterized. The 17-Å peak of the K-saponite did not migrate as it diminished, showing that no ordered mixed-layer mineral evolved from the smectite (Reynolds, 1981; Whitney, 1979).

After the initial crystallization of the gel, the Na-saponite and the Ca-saponite showed distinct signs of reaction at 400°C (Figure 2), but still exhibited no reactions at lower temperatures. At 450°C the K-saponite reacted more extensively after 200 days than either the Na-saponite or Ca-saponite (Figure 6). In addition, the reaction products of the Na- and Ca-saponites differ from those in the K-saponite runs. In the Na-saponite and Ca-saponite experiments there is no evidence for the formation of mica layers, either as a discrete phase or in mixed-layer clays. Only talc or mixed-layer talc/ saponite formed at 400°C (Figure 2). Talc/saponite (70% talc, 30% saponite) formed in the 450°C Ca-saponite runs. It probably formed by precipitation from solution rather than by the incremental conversion of saponite layers to talc layers within the original saponite, which would be marked by the migration of the 8.58-Å saponite peak toward lower angles (Reynolds, 1981; Whitney, 1979). Likewise, the same dissolution and precipitation mechanism apparently formed a fully ordered  $(\mathbf{R} = 1)$  chlorite/saponite (55% saponite, 45% chlorite) in the Ca-saponite 450°C samples. An interstratified talc/ saponite also formed in the 450°C Na-saponite sample, but its mechanism of formation could not be determined.

## DISCUSSION

Eberl *et al.* (1978) examined the differences in reactivity between dioctahedral and trioctahedral smectites. Dioctahedral smectites react more readily under hydrothermal conditions than trioctahedral smectites, apparently because the substitution of Al for Si in tet-

<sup>2</sup> Atmospheric pressure.

sap = saponite; t/s = mixed-layer talc/saponite; p/s = mixed-layer phlogopite/saponite; c/s = mixed-layer chlorite/saponite; t = talc; p = phlogopite; ? = unidentified; (?) = questionable.

<sup>&</sup>lt;sup>1</sup> l kbar pressure.



Figure 3. X-ray powder diffraction patterns of the time-sequence of K-saponite products treated at 450°C for the times shown. Such a sequence allows the progress of the reactions to be monitored. S = saponite, P/S = mixed-layer phlogopite/saponite, T/S = mixed-layer talc/saponite. All samples are glycolated. CuK $\alpha$  radiation.



Figure 4. X-ray powder diffraction patterns of the mixture of mixed-layer clays formed from the K-saponite gel treated hydrothermally for 200 days at 450°C. The measured diffractogram (bottom) was simulated with a composite of two calculated diffractograms for a fully ordered, 34% expandable talc/ saponite and a fully ordered, 80% expandable phlogopite/ saponite. CuK $\alpha$  radiation.

rahedral coordination, the primary charge-building mechanism, is more easily achieved in the aluminumrich dioctahedral system. In closed-system experiments, the only aluminum available is the small amount which accounts for the layer charge on the saponite (0.33equivalents per O<sub>10</sub>(OH)<sub>2</sub>). This amount of aluminum is sufficient to convert one-third of the layers to phlogopite if all of the aluminum is concentrated to form a mica charge in a few layers. Such a conversion process would require that the remaining two-thirds of the layers contain no aluminum or potassium (e.g., talc layers).

т/s 29.9 Å



Figure 5. X-ray powder diffraction pattern of the time-sequence of K-saponite run products treated hydrothermally at 550°C for the times shown. The saponite which predominates in the shortest run diminished and gave way to discrete talc and discrete phlogopite with increasing run times. S = saponite, P = phlogopite, T = talc. All samples are glycolated. CuK $\alpha$  radiation.

The reactions in the K-saponite system support such a scheme. The reaction products of the K-saponite at 450°C were two mixed-layer phases, talc/saponite and phlogopite/saponite. If the conversion of saponite layers to talc and phlogopite layers were ideal, the reaction could be written:

## 3 saponite $\rightarrow$ 2 talc + 1 phlogopite.

Hence, the products would contain twice as many talc layers as phlogopite layers. Although the ratio of the different types of layers in the reaction products of K-saponite at  $450^{\circ}$ C could not be measured precisely, more talc layers than phlogopite layers appeared to be present.

At 500° and 550°C, the conversion of saponite layers



Figure 6. Comparison of X-ray powder diffraction patterns of run products of the K-saponite with the Ca-saponite and Nasaponite after 200 days of hydrothermal treatment at 450°C. The reactions of the saponite with different interlayer cations differed both in extent and in the nature of the reaction products. All samples are glycolated. CuK $\alpha$  radiation.

to talc and phlogopite layers must have proceeded via a dissolution-precipitation mechanism because no intermediate mixed-layer phases appeared at these temperatures. K-saponite dissolved and the talc and phlogopite crystallized and grew as discrete phases. Although the amounts of each of the layer types could not be measured, it appeared that more talc layers were produced.

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Authors	Mineral <sup>1</sup>	Run time (days)	Stability limit <sup>2</sup> (°C)
Ames and Sand (1958)	Na-saponite	1–8	750
Mumpton and Roy (1956)	Na-saponite Mg-saponite Ca-saponite Pb-saponite	>14 (1.5 kbar)	560 565 570 570
Koizumi and Roy (1959)	Na-saponite	7	550
liyama and Roy (1963a)	Na-saponite	3-10	575
liyama and Roy (1963b)	"special trioctahedral sodium smectite"	3–10	850
Whitney (this study)	K-saponite Na-saponite Ca-saponite	200 200 200	400 400 400

Table 2.	Saponite	stability	studies.

<sup>1</sup> Mineral names are those used by the authors cited.

<sup>2</sup> Pressure = 1 kbar unless specified otherwise.

It is notable that the mechanism by which saponite layers are transformed to talc or phlogopite changed between 450° and 500°C. Although at higher temperature the reactions proceeded by the dissolution-precipitation mechanism, at 450°C the reactions took place via topotactic transformations, which require less energy because much of the mineral structure remains intact (Spry, 1969). Even if the absolute experimental temperatures do not apply to geologic situations, it may be geologically significant that the mixed-layer clays formed only near the thermal boundary between discrete saponite and discrete talc + phlogopite. Kinetic barriers may have precluded the dissolution-precipitation mechanism at 450°C, thus the formation of mixed-layer intermediate phases provided the easiest reaction path at that temperature.

The role of the interlayer cation in the reactions is revealed by comparing the reactivity of the K-saponite with that of the Na-saponite and Ca-saponite. At 450°C the K-saponite reacted further than the Na-saponite and Ca-saponite. Eberl (1978) related the rapid reaction of dioctahedral K-smectite to the low hydration energy of potassium. Dioctahedral Na-smectites react more sluggishly to mica-type layers than do the dioctahedral K-smectites because the hydration energy of sodium is higher than that of potassium. Thus, a greater layer charge must be built on the 2:1 layer in order to dehydrate the interlayer sodium and make the layer collapse. Calcium has an even higher hydration energy and reacts even more slowly. Although it is difficult to compare the rates of the reactions of the Na-saponite and Ca-saponite in the present experiments, neither reacted as rapidly as the K-saponite, thus conforming to the trends observed for the dioctahedral smectites. Of course, the absolute reaction rates are much slower for the trioctahedral smectites than for the dioctahedral smectites (Eberl et al., 1978).

The very small amount of ordered chlorite/smectite

in the 450°C Ca-saponite run after 200 days indicates that some dissolved magnesium precipitated as interlayer hydroxide sheets to form the chlorite-like layers. Despite the fact that calcium was intended to be the interlayer cation, the presence of chlorite/saponite shows that other dissolved species compete for that role and points out the need for monitoring solution compositions in future experiments.

## HYDROTHERMAL STABILITY OF SAPONITE

The high-temperature stability limit of saponite has been investigated in the earlier experiments, mentioned above, and is listed in Table 2. Previous workers used short run times (less than 30 days) and generally used sodium as the interlayer cation. All of the experiments employed either noncrystalline gels or oxide mixtures as starting materials. The temperatures at which saponite began to react towards other phases, as determined in the earlier experiments, ranged from 550° to 850°C. The lowest temperature of reaction (550°C) is still at least 150°C higher than the lowest reaction temperature observed in the present experiments (400°C).

The major differences between the present study and the earlier work are the long duration of the experiments and the use of potassium as an interlayer cation. The results of the shortest runs in the present experiments were similar to those reported previously. Saponite is the dominant phase in the run products at all temperatures after only seven days, and the Ca-saponite and Na-saponite remained largely unreacted even after 200 days. The K-saponite, on the other hand, reacted extensively toward mixed-layer clays after only 50 days, and after 200 days no discrete saponite remained at 450°C. Clearly, long run times are required to allow these sluggish reactions to proceed. Furthermore, equilibrium was never achieved in any series of runs. Identical products in two consecutive long runs would indicate that equilibrium may have been acheived, but this was never observed. Even the most reactive composition, K-saponite, did not completely react at any temperature in the longest runs, as shown by the continued, although diminished, presence of a saponite peak at 17 Å in the 550°C runs (Figure 5). Much longer run times are required to carry these reactions to completion, and much longer run times may reveal reactions at lower temperatures than those observed in these experiments.

## CONCLUSIONS

Saponite reacts hydrothermally at temperatures as low as 400°C, which is at least 150°C lower than previously observed. At 500° and 550°C, K-saponite reacts to talc + phlogopite by a dissolution-precipitation mechanism, whereas at 450°C it reacts to a mixture of two mixed-layer clays. The ultimate products of the reaction at 450°C are not known because the reaction is not complete even after 200 days. Below 400°C, K-saponite shows no signs of reaction after 200 days.

The interlayer cation has a noticeable effect on the reaction rate and the reaction type. K-saponite reacts more rapidly than either Na-saponite or Ca-saponite. Furthermore, the Na-saponite and Ca-saponite reaction products contain no mica layers, either as discrete phases or in mixed-layer phases because the hydration energy for Na and Ca is so large that a much larger layer charge is required on the 2:1 layer to dehydrate the Na or Ca to produce a mica layer. The saponite system contains insufficient aluminum to build such a charge.

These experiments suggest that the temperature at which saponite reacts to form other phases is considerably lower than previous experimental results had indicated. In fact, the suggested hydrothermal stability limit of 200°C which Tomasson and Kristmannsdottir (1972) inferred from hydrothermally altered basalts may be close to the true value if the system is given sufficient time to reach equilibrium. The present experiments say nothing about the effect of substituted iron on the hydrothermal stability limit of saponite, and the iron-bearing system must be understood in order to apply experimental results to natural systems.

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#### Whitney

Резюме—Сапонит кристаллизировался из аморфного геля, имеющего идеальный сапонитовый состав, в течение 7 дней при всех экспериментальных температурах в диапазоне от 300° до 550°С и при давлении 1 кбар. Реакции, следующие за этой первоначальной кристаллизацией, различаются типом и степенью в зависимости от температуры реакции и типа межслойного катиона. Выше 450°С, первоначально выкристаллизированный К-сапонит растворяется, а тальк и флогопит образуют центры кристаллизации и растут как дискретные фазы. При 450°С первоначально образованный К-сапонит реагирует, образуя слои талька и флогопита. Эта реакция происходит скорее путем внутрикристаллических трансформации слоев, чем путем растворения и осаждения. Продуктом реакции является смесь полностью упорядоченных талька/сапонита и сапонита/ флогопита. К-сапонит проявляет слабые признаки реакции при 400°С после 200 дней. Температура 400°С является по крайней мере на 150°С ниже, чем экспериментальные температуры реакции, ранее опубликованные для сапонитов. Ниже 400°С не наблюдались никакие реакции кроме начальной кристаллизации сапонита. Выше 400°С К-сапонит реагирует более быстро, чем Naсапонит или Са-сапонит. Na-сапонит и Са-сапонит не формируют слои слюды во время их трансформаций в смешано-слойные глины. Переслаивающийся тальк/сапонит, формировался в системе Na-сапонит, а оба, тальк/сапонит и хлорит/сапонит формировались в системе Са-сапонита. [E.C.]

**Resümee**—Saponit kristallisiert aus einem amorphen Gel, das eine ideale Saponitzusammensetzung hat, innerhalb von 7 Tagen bei allen experimentellen Temperaturen zwischen 300° und 550°C und einem Druck von 1 kbar. Die Reaktionen nach dieser anfänglichen Kristallisation variieren in Art und Ausmaß je nach Reaktionstemperatur und Art der Zwischenschichtkationen. Über 450°C löst sich der anfänglich kristallisierte K-Saponit wieder auf, und Talk und Phlogopit wachsen als diskrete Phasen. Bei 450°C reagiert der ursprüngliche K-Saponit und bildet Talk- und Phlogopit-Lagen. Diese Reaktion verläuft jedoch über intrakristalline Lagenumwandlung und weniger über Auflösung und Ausfällung. Dabei bildet sich ein Gemenge aus einer vollständig geordneten Talk/Saponit-Wechsellagerung und einer vollständig geordneten Saponit/Phlogopit-Wechsellagerung. Der K-Saponit zeigt bei 400°C nach 200 Tagen gewisse Anzeigen einer Reaktion. Diese Temperatur ist mindestens um 150°C niedriger als experimentelle Reaktionstemperaturen, die bisher für Saponit berichtet wurden. Unter 400°C wurde nach der anfänglichen Saponit-Kristallisation keine weitere Reaktion beobachtet. K-Saponit reagiert über 400°C schneller als Na-Saponit oder Ca-Saponit. Außerdem bilden sich aus Na-Saponit und Ca-Saponit keine Glimmerlagen während ihrer Umwandlung zu Wechsellagerungen. Talk/Saponit-Wechsellagerungen, die im Na-Saponit-System und im Ca-Saponit-System gebildet wurden, führten zur Bildung einer Talk/Saponit- und einer Chlorit/Saponit-Wechsellagerung. [U.W.]

**Résumé**—La saponite se cristallise à partir d'un gel amorphe ayant une composition saponite idéale endéans 7 jours à toutes les températures expérimentales entre 300° et 550°C sous une pression d'1 kbar. Les réactions subséquentes à cette cristallisation initiale varient de type et de degré, dépendant de la température de la réaction et du genre de cation intercouche. Au dessus de 450°C, la saponite-K initialement cristallisés es dissout, et du talc et de la phlogopite deviennent nucléés et croissent en phases discrètes. A 450°C la saponite-K initiale réagit pour former des couches de talc et de phlogopite, mais la réaction se produit via des transformations de couche intracristallines plutôt que via une dissolution et une précipitation, produisant un mélange de talc/saponite interstratifié complètement ordonné et de saponite/phlogopite complètement ordonné. La saponite-K montre des signes subtiles de réaction expérimentales rapportées auparavant pour des saponites. On n'a observé aucune réaction au-delá de la cristallisation initiale de saponite-Na et la saponite-Na reagit plus rapidement que soit la saponite-Na ou la saponite-Ca au dessus de 400°C et la saponite-Na et la saponite-Ca ne produiseint pas de couche de mica pendant leur transformation en argiles à couches mélangées. Le talc/saponite interstratifé a été formé dans le système saponite-Na, et le système saponite-Ca au produi tà la fois du talc/saponite et de la chlorite/saponite. [D.J.]