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CHARACTERIZATION OF SYNTHETIC Na-BEIDELLITE

J. THEO KLOPROGGE,[1] J. BEN H. JANSEN,[1] AND JOHN W. GEUS[2]

[1] Institute of Earth Sciences, Department of Geochemistry, University of Utrecht
Budapestlaan 4, P.O. Box 80.021, 3508 TA Utrecht, The Netherlands

[2] Analytical Chemical Laboratory, Department of Inorganic Chemistry, University of Utrecht
Croesestraat 77a, 3522 AD Utrecht, The Netherlands

Abstract—Na-beidellite, a member of the smectite group, was grown hydrothermally from a gel of composition $0.35Na_2O \cdot 2.35Al_2O_3 \cdot 7.3SiO_2$ in NaOH solutions at a pH between 7.5 and 13.5, a pressure of 1 kbar, and a temperature of 350°C. The synthetic Na-beidellite was characterized by means of scanning electron microscopy, X-ray powder diffraction, infrared spectroscopy, electron microprobe, inductively coupled plasma-atomic emission spectroscopy, and thermogravimetric analysis. The unit-cell parameters of the orthorhombic cell are: a = 5.18, b = 8.96, and c = 12.54 Å. The cation-exchange capacity was determined to be 70 meq/100 g. A maximum of 40 wt. % water was present and reversibly lost by heating to about 55°C. The loss of water caused a decrease of the basal spacing to 9.98 Å. At temperatures ≥600°C, the Na-beidellite started to dehydroxylate, reaching its maximum in the range 600°–630°C. At 1100°C the remaining solid recrystallized to $Al_6Si_2O_{13}$ (mullite) and SiO_2 (cristobalite).

Key Words—Beidellite, Cation-exchange capacity, Scanning electron microscopy, Synthesis, Thermal analysis, X-ray powder diffraction.

INTRODUCTION

Cation exchange, swelling in water, and incorporation of organic and inorganic complexes to produce pillared clays have been studied to investigate the technical utilization of natural clays (Loeppert *et al.*, 1979; Breen *et al.*, 1985; Goh and Huang, 1986; Plee *et al.*, 1987; Schutz *et al.*, 1987; Sterte and Shabtai, 1987; Olivera *et al.*, 1988). The high costs of recovering the smectites, which usually have an unreliable quality and a large variation in impurity content, cause the industrial demand for natural smectites in these applications to be limited (Torii and Iwasaki, 1987).

To avoid the problems inherent to the use of natural raw materials, hydrothermal synthesis of pure smectites has been attempted (Tsunashima *et al.*, 1975; De Kimpe, 1976; Torii and Iwasaki, 1986; Torii and Iwasaki, 1987; Plee *et al.*, 1987; Schutz *et al.*, 1987). The formula for beidellite can be written as $(0.5Ca,Na,K,0.5Mg)_pAl_4Si_{8-p}Al_pO_{20}(OH)_4 \cdot nH_2O$. Natural beidellite has an average layer charge of 0.66, although theoretically values range from 0.4 to 1.2. Plee *et al.* (1987) and Schutz *et al.* (1987) synthesized under basic conditions (0.01 M to 0.1 M NaOH) Na-beidellite as a starting material for the pillaring processes; however, they did not characterize their product thoroughly. The aim of the present study was to synthesize Na-beidellite in less basic solutions from a gel of composition $Na_{0.7}Al_{4.7}Si_{7.3}O_{22} \cdot nH_2O$, following the procedure of Hamilton and Henderson (1968), and to characterize the synthetic Na-beidellite product. This clay was used to synthesize beidellite cross-linked with metal-hydroxide polymers having a Keggin structure.

EXPERIMENTAL AND ANALYTICAL TECHNIQUES

As starting material a gel of anhydrous composition $0.35Na_2O \cdot 2.35Al_2O_3 \cdot 7.3SiO_2$ was prepared according to the procedure of Hamilton and Henderson (1968). The chemicals used in the preparation of the gel were $Al(NO_3)_3 \cdot 9H_2O$ (Merck no. 1063), Na_2CO_3 (Merck no. 6392) and tetraethyl orthosilicate (Merck-Schuchardt no. 800658). Fifty milligrams of the gel (containing 7.3% water) together with 70 μl of NaOH solution (10^{-6}, 10^{-5}, or 10^{-4} M) were placed into a gold capsule welded at one end. The capsule was closed by arc-welding, while its main body was constantly cooled in an ice-water bath. All capsules were checked for leakage before the experiment.

The synthesis was carried out in a Tuttle-type, externally heated, cold-seal pressure vessel (Tuttle, 1949) using argon as the pressure medium at 350°C and at total pressures of 0.5 and 1.0 kbar (Table 1). Temperature was measured by a chromel-alumel thermocouple, which is considered to be accurate to within 5°C. Pressure was read from a Bourdon-type pressure gauge having an accuracy of 10 bar. After quenching the pressure vessels with compressed cold air, the capsules were reweighed to check for leakage.

The morphology of the products obtained were investigated with Cambridge S150 and M600 scanning electron microscopes (SEM) equipped with energy-dispersive X-ray (EDX) analyzers. X-ray powder diffraction (XRD) patterns were recorded with a Philips PW 1050/25 diffractometer using CuKα radiation. Unit-cell dimensions were calculated using the least squares

Table 1. Experimental runs at 350°C.

Run	P (kbar)	Time (days)	pH	Starting solution	Product[3]
E414-1	1.0	5	12	Na_2CO_3[1] + H_2O	beid + qtz
E414-2	1.0	5	13.5	Na_2CO_3[1] + NaOH	beid + qtz
E415-1	1.0	5	8	NaOH	beid + qtz
E415-2	1.0	5	9	NaOH	beid + qtz
E426	1.0	5	10	NaOH	beid + qtz
E427	0.5	10	10	NaOH	beid + qtz
E432	1.0	10	7.8	H_2O	beid
E448[2]	1.0	20	10	NaOH	beid + qtz
E476	1.0	20	10	NaOH	beid + qtz

[1] Solid Na_2CO_3.
[2] Used for heating experiment at 1150°C.
[3] beid = Na-beidellite; qtz = quartz.

refinement computer program Unitcellc (Strom, 1976). Infrared (IR) absorption spectra were obtained on powdered samples in KBr tablets (sample concentration = 1 wt. %) using a Perkin Elmer 580 IR spectrophotometer. The solid product was analyzed thermogravimetrically (TGA) using a Dupont 1090 Thermal Analyzer at heating rates of 10°, 1°, and 0.2°C/min. Differential thermal analyses (DTA) were made at a heating rate of 10°C/min. Chemical analyses were made using a JEOL JXA-8600 electron microprobe. The cation-exchange capacity (CEC) was measured on a sample dried overnight at 120°C. The sample was exchanged with 1 M KCl solution. After centrifugation the solution was analyzed for Na using inductively coupled plasma-atomic emission spectroscopy.

RESULTS

SEM showed the hydrothermal products to consist of flakes exhibiting mainly (001) faces (Figure 1, upper). The (100), (010), and (110) faces were exceptionally well developed on crystals of run E426 (Figure 1, lower). Also, small aggregates were noted, which consisted mainly of anhedral material and some quartz. The presence of quartz was confirmed by XRD. In Figure 2 (lower), the EDX analyses of a small quartz particle (A) and of a large flake (B), presumably Na-beidellite, are presented. The flake yielded a relative low Na content, probably due to diffusion of Na during exposure to the electron bean (van der Pluijm *et al.*, 1988).

Microprobe analyses gave a composition of 56.76% SiO_2, 30.96% Al_2O_3, 2.44% Na_2O, and 9.84% H_2O, corresponding to $Na_{0.61}Al_{4.70}Si_{7.32}O_{20}(OH)_4$. Here, a relative low Na content was also observed, probably due to the diffusion of Na. The hydrothermal solution after synthesis contained 157 ppm Na, 188 ppm Al, and 1833 ppm Si. The CEC of the solid was determined to be 70 meq/100 g.

The *hkl* values of the synthetic Na-beidellite were indexed mainly according to Weir and Greene-Kelly (1962), except for the 001, 002, and 004 reflections. Good agreement between the calculated and observed values was observed, as long as the 024 reflection was changed to 004 (Table 2). The unit-cell parameters based on the revised indices are compared with data for Na-beidellite from Brindley and Brown (1980) and for Ca-beidellite from Weir and Greene-Kelly (1962) in Table 3. The Na-beidellite expanded to d(001) =

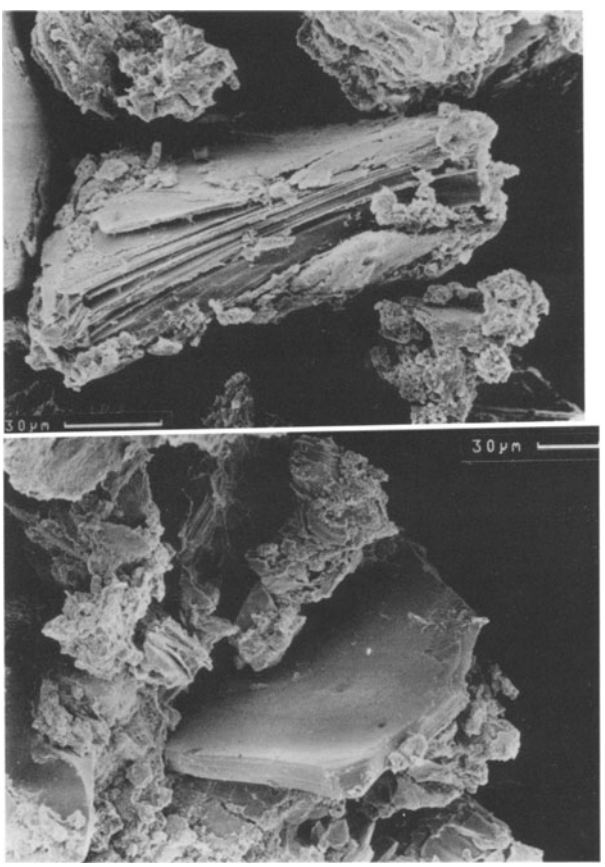

Figure 1. Scanning electron micrographs of (upper) Na-beidellite crystal synthesized at 350°C and 500 bar (run E427); (lower) Na-beidellite crystal synthesized at 350°C and 1 kbar (run E426).

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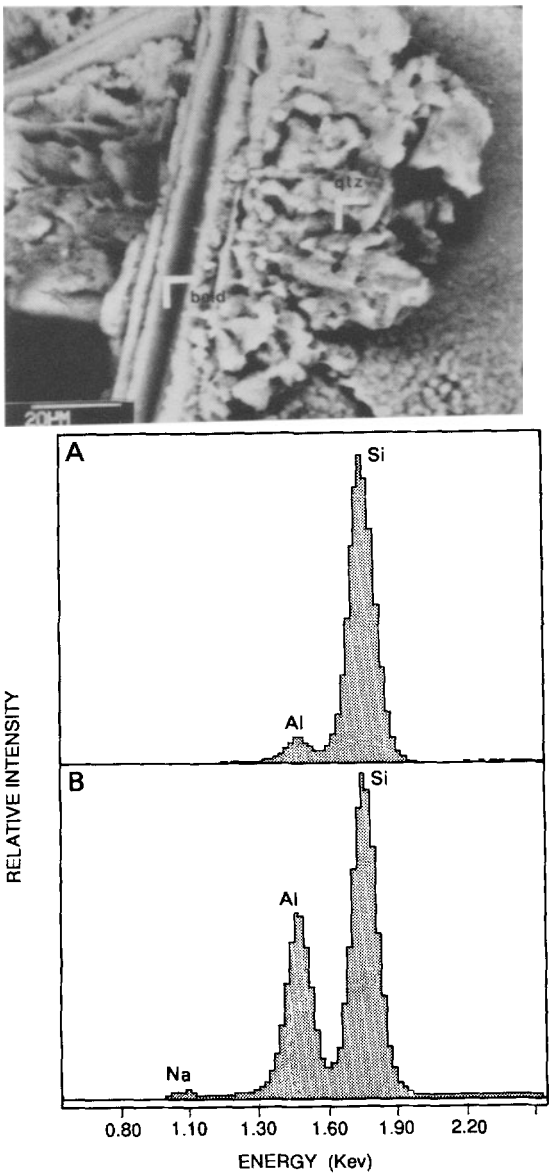

Figure 2. Scanning electron micrograph of (upper) small particle formed from the excess SiO_2 (arrow indicates position of the energy-dispersive X-ray analyses A and B) (run E448); (lower) energy-dispersive X-ray analysis of (A) quartz-bearing particle and (B) Na-beidellite (Table 2).

16.48 Å with ethylene glycol at room temperature, which is comparable with the value of 16.7 Å given by Brindley and Brown for natural beidellite. A sample saturated with Li and glycerol and heated overnight at 300°C (Hofmann-Klemen and Greene-Kelly test) resulted in a basal spacing of 17.67 Å, confirming that the product was Na-beidellite. Heating the product to 55°C caused a gradual decrease in d(001) from 12.54 Å to 9.98 Å and a proportional decrease in d(002) due to the loss of interlayer water. The 004 reflection decreased in intensity and was replaced by the 003 reflection.

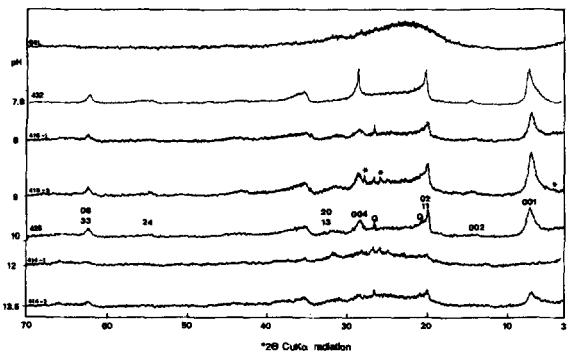

Figure 3. X-ray powder diffraction patterns of beidellite formed at different pHs. For comparison, the pattern of the gel is also given. Q = two strongest lines of quartz; * = three strongest lines of SiO_2-X_2, an intermediate phase in the crystallization of quartz from noncrystalline silica in alkaline media (Mitsyuk *et al.*, 1976).

As is apparent from Figure 3, the starting solution influenced the degree of crystallinity of the product Na-beidellite. The optimum crystallinity was obtained at pHs of 9 and 10; however, whether the degree of crystallinity was influenced by only the pH or by the Na concentration is not known. The two small peaks in Figure 3 at d = 4.26 and 3.34 Å are the 100 and 101 reflections of a trace of quartz.

The IR spectra of the starting gel, the Na-beidellite synthesized in run E415-3, and a natural Na-montmorillonite from Clay Spur, Wyoming, are shown in Figure 4. The absorption maxima of the synthetic and natural Na-beidellite (van der Marel and Beutelspa-

Table 2. X-ray powder diffraction data for hydrothermally synthesized Na-beildellite (run E415-2).

d(obs)[1]	d(calc)[1]	I/I_0[1]	*hk* + 001[1]	d(calc)[2]	I[2]	*hkl*[2]
12.44	12.54	100	001			
6.19	6.27	5	002[3]			
4.43	4.48	75	02,11	4.48	10	020,110
	4.22			4.35		021
				3.56	10	023
3.12	3.14	55	004[4]	3.15	2	024[4]
2.59	2.59	15	20,13	2.59	—	200,130
2.54	2.54	20		2.56	8	201
				2.48	10	202
1.670	1.679	5	24	1.687	6	241
				1.664	8	242
1.490	1.495	15	33	1.495	10	060,330
	1.493		06			

[1] Na-beidellite from run E415-2.
[2] Brown (1961); Weir and Greene-Kelly (1962).
[3] 002 not given by Brown (1961), Weir and Greene-Kelly (1962).
[4] 004 instead of 024 as given by Weir and Greene-Kelly (1962).

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Table 3. Unit-cell parameters (Å) of hydrothermally synthesized Na-beidellite (run E415-2), based on an orthorhombic unit-cell.

	a	*b*	*c*
This study	5.18 ± 0.0044	8.96 ± 0.0076	12.54 ± 0.0106
Weir and Greene-Kelly (1962), Brown (1961)[1]	5.179	8.970	17.57
Brindley and Brown (1980)	—	—	12.5

[1] Sample Ca-saturated and glycerol expanded.

cher, 1976) are listed in Table 4. The maxima for the synthetic Na-beidellite are shifted by about 2% towards higher wavenumbers compared with natural beidellite (van der Marel and Beutelspacher, 1976). The very small shoulders at 1200 and 460 cm^{-1} are probably due to the trace of quartz.

TGA and DTA plots depicting dehydration and dehydroxylation processes are shown in Figure 5. The results presented below are based on a heating rate of 0.2°C/min. During heating to 55°C, 40 wt. % of absorbed water was lost. The DTA curve showed a strong endothermic maximum. The weight loss due to dehydration ranged from 30% to 40% after 14 days drying in air before the measurement.

In the range 55° to 600°C the sample gradually lost 2.5% weight (3.4% based on water-free Na-beidellite). This loss is attributed to the removal of interlayer water and to the dehydroxylation, which began at about 400°C. Mackenzie (1970) and Gerard and Herbillon (1983) proposed an overlap of dehydroxylation and the last part of the dehydration between 150° and 600°C. Another possibility is that the weight loss was derived partially from unreacted noncrystalline material. Between 600° and 630°C the dehydroxylation reached a maximum, and the sample lost 2.0% weight (2.6% based on water-free Na-beidellite).

Theoretically, the complete dehydroxylation of Na-beidellite involves the production of two moles of H_2O and corresponds to a weight loss of 4.9%. The experimental weight loss between 55° and 630°C was about 6.0%, however, which is more than the theoretical value for dehydroxylation.

TGA experiments at relatively slow heating rates showed lower temperatures of dehydration and dehydroxylation (Figure 5). At a heating rate of 0.2°C/min most of the water was lost <55°C, instead of 130°C at 10°C/min. The maximum dehydroxylation was at 600°–630°C at 0.2°C/min, vs. 630°–740°C at a heating rate of 10°C/min. At temperatures >630°C no further weight loss of H_2O was observed.

The small S-shaped endothermic-exothermic peak system at 1045°–1095°C (Figure 5, DTA curve) was probably due to the breakdown of the anhydrous beidellite to a X-ray-amorphous material from which the new phases mullite and cristobalite crystallized.

DISCUSSION

In all experiments the synthesis of Na-beidellite was successful (Table 1). The degree of crystallinity apparently depended on pressure, temperature, the length of time at the hydrothermal conditions, and the pH or NaOH concentration of the starting solution (unpublished results in this laboratory). The most highly crystallized Na-beidellite was synthesized at 350°C, 1 kbar, 5 days, and a pH of 10 of the starting solution. Plee *et al.* (1987) and Schutz *et al.* (1987) produced Na-beidellite at pressures of 600 and 130 bar, but provided no information on crystallinity or morphology. The

Table 4. Infrared absorption maxima (cm^{-1}) of hydrothermally synthesized Na-beidellite (run E415-3) and natural beidellite.

This study	van der Marel and Beutelspacher (1976)		Stubican and Roy (1961); Farmer (1974)
3655 strong	3625	Al–O–H	stretching
3450 strong	3420–3415	H–O–H	stretching
3240 weak (shoulder)	3220	H–O–H	stretching
1638 medium	1625	H–O–H	bending
1081 weak (shoulder)	1100–1098	Si–O	stretching
1047 strong	1030–1028	Si–O–Si	stretching
935 medium	914	Al–O–H	bending
883 medium	875	Al–O–H	bending
800 medium	796	Al–O–H	bending
700 weak	698–693	Si–O–Al	bending
627 weak	640–622	Al–O–H	bending
531 strong	530–520	Si–O–Al	bending
470 strong	471–467	Si–O	bending
440 weak (shoulder)	419	Si–O	bending

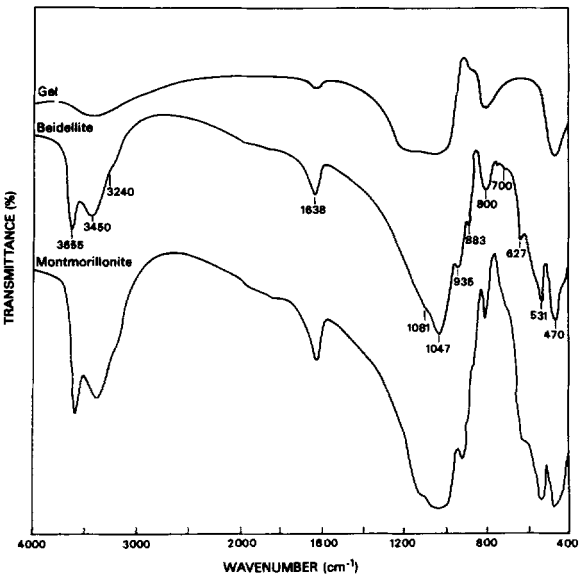

Figure 4. Infrared spectrum of Na-beidellite (run E415-3). Spectra of gel and natural Na-montmorillonite are included as references.

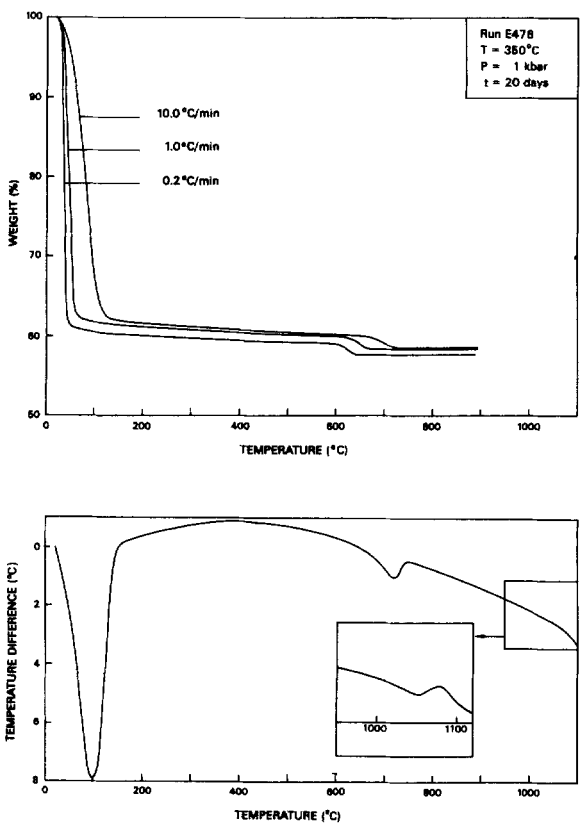

Figure 5. Thermogravimetric analyses (heating rate = 10, 1, and 0.2°C/min) and differential thermal analysis (10°C/min) of Na-beidellite (run E476).

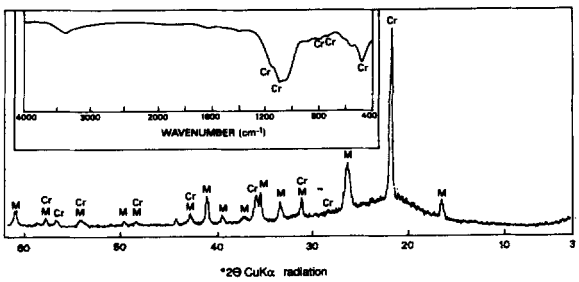

Figure 6. X-ray powder diffraction pattern and infrared spectrum of the products of run E448 after heating at 1150°C. C = cristobalite; M = mullite.

CEC value of 70 meq/100 g is low compared with the expected CEC of 97 meq/100 g for Na-beidellite having a composition of $Na_{0.7}Al_{4.7}Si_{7.3}O_{20}(OH)_4 \cdot xH_2O$, probably because the CEC was determined on a mixture of Na-beidellite and unreacted material, which could not be separated from each other.

XRD yielded a basal spacing of 12.54 Å, which is consistent with the theoretical value of 12.50 Å (Weir and Greene-Kelly, 1962; Brindley and Brown, 1980). Indexing based on the calculated lattice parameters revealed that an 024 index for the peak at 3.15 Å reported by Weir and Greene-Kelly (1962) is incorrect. An assignment of 004 is more consistent with the other reflections. The indexing given here is in accord with the data given by Nadeau *et al.* (1985) for the Unterrupsroth beidellite.

The decrease in basal spacing from 12.54 at 25°C to 9.98 Å at 55°C and the corresponding weight loss in the TGA experiment demonstrate a loss of absorbed water. Na-beidellite having a basal spacing of 9.98 Å should contain a maximum of 1.1% water in the interlayer, next to the Na ions, as evidenced by the fact that between 55° and 630°C 6% water was lost, which can not be ascribed to the loss of hydroxyl groups alone. Realistically, some water was probably absorbed by the unreacted noncrystalline material. The microprobe analyses indicate (by difference) a water content of 9.84% (adsorbed water plus hydroxyl groups), which is much less than the TGA results, thereby suggesting that some water was absorbed by the unreacted starting material. At temperatures >600°C 2.2% weight was lost. This bipartitional dehydroxylation probably took place, because the hydroxyl groups in the Al octahedral layer of beidellite are not equivalent, inasmuch as 8.8%, based on microprobe analyses (9.6% based on ^{29}Si magic angle spinning-nuclear magnetic resonance, unpublished results in this laboratory) of the Si in the Si tetrahedral layer has been substituted by Al. This substitution may have distorted the hexagonal rings of tetrahedra and the Al octahedra.

Slow heating rates during the TGA led to low temperatures of dehydration and dehydroxylation. These processes were probably controlled by diffusion. A

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standard heating rate of 10°C/min is normally used for weight-loss determinations, but not for the determination of decomposition temperatures, if diffusion plays an important role.

The presence of quartz in the products of runs having NaOH solution as the reaction medium indicate that the kinetics of dissolution of silica in basic conditions was too rapid compared with the formation of beidellite. For the liquid/solid ratio used in these experiments supersaturation of silica should have occurred, and the silica should have segregated as quartz. In pure water, the dissolution of silica would have been slower, thereby avoiding supersaturation in silica and yielding no quartz.

The two peaks at 1045° and 1095°C in the DTA curve (Figure 5) are probably due to the breakdown of the anhydrous Na-beidellite to a noncrystalline phase, from which new phases crystallized (Grim and Bradley, 1940; Bradley and Grim, 1951; Greene-Kelly, 1957). XRD of the product of run E448 heated for 18 hr at 1150°C revealed the presence of mullite and cristobalite (see Figure 6).

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