

ON DEHYDRATION OF BOUND WATER OF SEPIOLITE

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(Received 20 August 1973)

Abstract—Bound water of sepiolite dehydrates in two steps in the temperature range of 250–650°C, as shown in the TG-curve. These steps are described here as steps II and III. At step II, half of the bound water is removed; other half at step III. From step II to III, discontinuous changes are confirmed in such properties as activation energy of dehydration, *a*-dimension, axial ratio, and intensities and spacings of X-ray powder reflections. A structural state at step II may be recognized as a distinct phase in the dehydration process.

INTRODUCTION

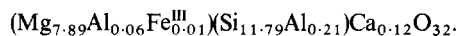
Two kinds of structure models of sepiolite were reported, one by Nagy and Bradley (1955) and the other by Brauner and Preisinger (1956); the latter has been favored (Brindley, 1959; Zvyagin, 1964; Gard *et al.*, 1968; Rautureau *et al.*, 1972). In both models the crystal-chemical state of water molecules has been grouped into three: zeolitic water, bound water and hydroxyl water (Caillère and Hénin, 1957, 1961; Martin-Vivaldi and Hach-Ali, 1970; Martin-Vivaldi and Robertson, 1971).

It is well known that removal of bound water transforms sepiolite into a dehydrated form. The crystal structure model of this dehydrated form ('sepiolite anhydride') was proposed by Preisinger (1959, 1963). On the other hand, it has been reported that bound water is removed in two steps at elevated temperatures (Kulbicki and Grim, 1959; Otsuka *et al.*, 1966; Hayashi *et al.*, 1969). Martin-Vivaldi and Cano-Ruiz (1956) reported three steps of dehydration: (1) loss of 1 molecule of zeolitic water below 200°C; (2) loss of 2.5 molecules in the region of 250–350°C; and (3) loss of 0.5 molecules between 350–650°C. It was noticed, however, that a dehydrated phase occurred at 330°C, when about 1 molecule of water still existed in dehydrated structure. Nathan (1969) and Otsuka, Hayashi and Imai (1970) have shown that the formation of the 'sepiolite anhydride' seems to be non-uniform, part of the structure being dehydrated, while part remaining intact. Their conclusions cannot explain the dehydration mechanism shown by two endothermic peaks in the DTA-curve and two steps in the TG-curve due to removal of bound water.

The purpose of the present study is to make clear the dehydration behavior of sepiolite with respect to the removal of bound water.

EXPERIMENTAL

Sepiolite from Kuzuu, Tochigi Prefecture, Japan was used for the present study. Its mineralogical and geological properties have been studied in detail by Imai and his collaborators (Imai *et al.*, 1966; Otsuka *et al.*, 1968; Hayashi *et al.*, 1969; Imai *et al.*, 1969). The structural formula was given by these workers as follows:



The calculation was based on the assumption of Caillère and Hénin (1961) that the dehydrated half-cell contains 32 oxygens. This formula is close to the ideal formula presented by Brauner and Preisinger (1956).

DTA and TG curves were recorded simultaneously by a Rigaku Thermoflex. The mean heating rate was used in the following three ways: 2.5°C/min, 5°C/min and 10°C/min. X-ray powder patterns were recorded by a Rigaku Geigerflex. The experimental conditions were as follows: CuK α radiation, 30 kV–15 mA or 35 kV–20 mA; split systems used, 1°–0.3 mm–1° or 1/6°–0.1 mm–1/6°; time constants used, 1 or 2. I.R. absorption spectra were recorded by the KBr method with a Japan Spectroscopic model i.r.-G spectrometer.

The sample powder (about 0.2 g) was pre-heated at 300, 400, 500 and 600°C for certain times. The notation 300–120 means the sample was pre-heated at 300°C for 120 hr. The heated samples were stored in a desiccator.

RESULTS

TG-curves and kinetics

Figure 1 shows DTA and TG-curves of the original and preheated samples. Four steps of weight losses, I, II, III and IV are shown clearly. The values of weight losses read from TG-curves are shown in Table 1 and

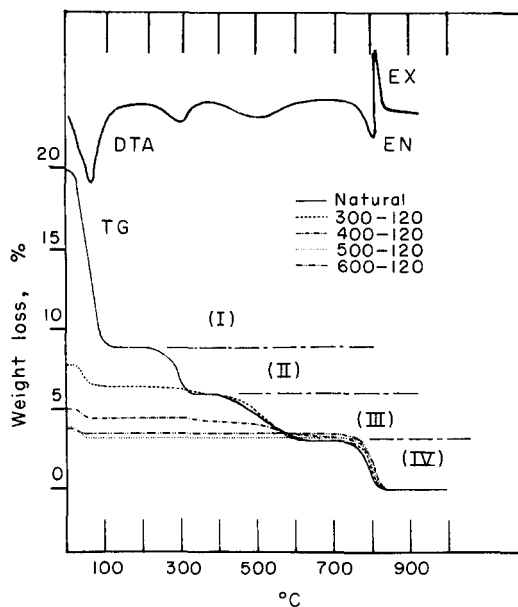


Fig. 1. DTA and TG curves of sepiolite; untreated and pre-heated sepiolite.

are compared with values calculated from two ideal structural formulas.

According to Freeman and Carroll (1958), activation energy and order of reaction for the reaction, $aA(s) = bB(s) + cC(g)$, can be obtained from TG-curve. Now, the rate of reaction for the disappearance of reactant A is expressed as $-dX/dt = kX^n$, where $X =$

amount of reactant A , $k =$ specific rate and $n =$ order of reaction with respect to A . k may be expressed as

$$k = Z \exp(-E^*/RT).$$

Therefore,

$$Z \exp(-E^*/RT) = -(dX/dt)/X^n$$

where, $Z =$ frequency factor, $E^* =$ energy of reaction, $R =$ gas constant and $T =$ absolute temperature.

Then,

$$\frac{-(E^*/R)(1/T)}{\ln X} = \frac{\ln(-dX/dt)}{\ln X} - n.$$

The plots of

$$\frac{(1/T)}{\log X} \text{ vs } \frac{(-dX/dt)}{\log X}$$

should result in straight lines with slopes of $+$ or $-E^*/2.3R$ and intercepts of $-n$.

The values of the activation energy and the order of the dehydration reaction at steps II and III were obtained from the plots as shown in Fig. 2 (Table 2). Slight differences were only shown among the values of the activation energy. This is no difference between the kinetic data shown at step III from a natural sample and its preheated counterpart (300-550).

Effects of heat upon X-ray powder patterns

Figure 3 shows the effect of heat on X-ray powder patterns. A noticeable effect is the appearance of new

Table 1. Weight losses of sepiolite; untreated and pre-heated samples and calculated values

	R.T. - 200°C (%)	200-380°C (%)	380-680°C (%)	680-900°C (%)	Total loss (%)
Natural	11.1	2.90	2.90	3.38*	20.28
	10.4	3.04	2.99	3.37*	19.76
300- 50	6.64	2.04	2.63	3.51*	14.82
300-120	0.70	0.08	2.79	3.12*	6.69
400- 50	3.48	0.32	2.00	4.07*	9.87
400-120	0.69	0.23	0.92	3.26*	5.10
400-200	0.56	0.16	0.46	3.31*	4.51
500- 50	1.04	0.48	0.84	3.75*	6.11
500-120	0.63	0.0	0.0	3.16*	3.79
600- 50	2.46	0.82	0.96	3.69*	7.93
600-120	0.21	0.0	0.0	3.46*	3.67
	Zeolitic water	Bound water		Hydroxyl water	
N.B	8.19	5.46		4.10	17.75
B.P	11.11	5.56		2.78	19.45

Mean heating rate, 10°C/min. Mean sample weight, 20 mg.

N.B—Calculated values from Nagy and Bradley (1955).

B.P—Calculated values from Brauner and Preisinger (1956).

* These values are not corrected by base line.

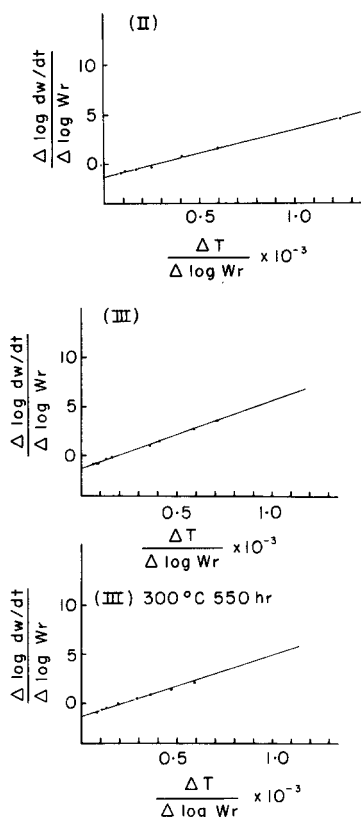


Fig. 2. Kinetics of dehydration of sepiolite. (II) and (III) show steps, II and III. $*w$ = total weight loss up to time, t . Wr = remaining weight at time, t , in total weight loss.

reflections at 10.2 and 8.1 Å in 300–120, which are persistent up to 600–120, though the spacings and intensities are slightly modified by the elevated temperatures.

Further, as shown in the data recorded by the high temperature X-ray diffractometer (Fig. 4), the (110) and (120) reflections of the dehydrated phase show a discontinuous change before and after step III (400°C).

On the other hand, Fig. 5 shows the comparison of the X-ray pattern of 300–120 with that of an artificial mixture of natural sepiolite and completely dehydrated sample (600–120). There are clear differences with regard to line profiles, relative intensities of some reflections, and the existence of several reflections on one of these two patterns which are not on the other.

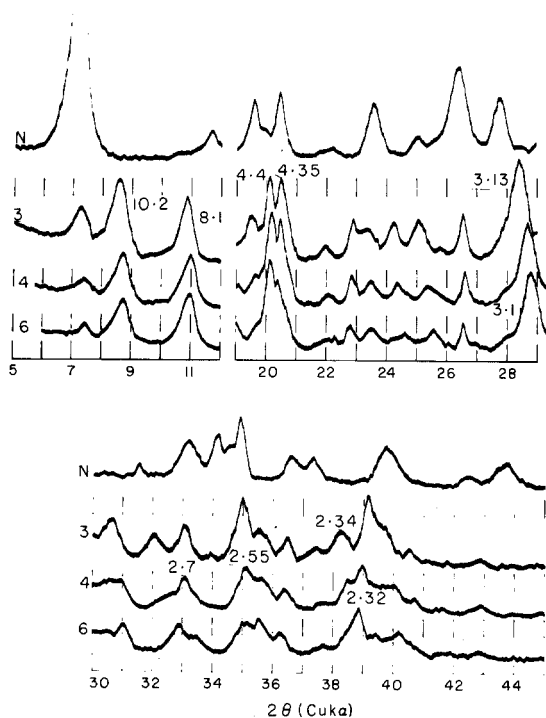


Fig. 3. X-ray powder diffraction patterns of sepiolite; untreated (N) and pre-heated samples. 3, 4 and 6 show respectively 300–120, 400–120 and 600–120. Numbers on the patterns present the d -spacing (Å).

These data suggest that the dehydrated form at step II (300–120) may exist as a distinct phase to be differentiated from that of the completely dehydrated state (600–120).

Indexing for X-ray powder diffraction peaks of 600–120 was done on the basis of the lattice parameters and diffraction intensities calculated from the structural model proposed by Preisinger (1959, 1963) as shown in Table 3. From these indices and the observed spacings, the lattice constants were again calculated (Table 4). On the basis of these calculated constants, peak spacings were calculated and compared with the observed spacings shown in Table 3.

The X-ray pattern of 300–120 is close to that of 600–120, with slight relative shifts of peak positions. Indexing of diffraction peaks of 300–120 was done assuming that each peak of 300–120 and the corresponding peak

Table 2. Activation energies and orders of reaction of the dehydration steps, II and III.

		n	E^* (kcal/mol)
Original sepiolite	Step II (200–400°C)	1.3	21.9
	Step III (400–600°C)	1.3	31.3
300–550	Step III (400–600°C)	1.3	31.1

Table 3. X-ray powder data of sepiolite; untreated and pre-heated samples

	Natural			300-120			400-120			500-120			600-120		
	h	k	l	I	d obs.	d calc.	I	d obs.	d calc.	I	d obs.	d calc.	I	d obs.	d calc.
200,	1	1	0	VS	12.08	11.90	15	11.89	11.62	25	11.89	11.62	25	11.86	11.62
	1	3	0	45	7.531	10.20	110	10.17	9.897	95	10.17	9.897	95	10.10	9.897
	0	4	0	65	6.732	8.120	100	8.094	7.964	100	8.094	7.964	100	8.085	7.964
	0	1	1	25	5.036	5.498	20	5.484	5.470	20	5.484	5.470	20	5.480	5.470
	1	5	0	85	4.515	5.166	35	5.165	5.149	40	5.165	5.149	40	5.154	5.149
	0	3	1	85	4.324	5.024	75	5.024	4.949	40	5.165	5.149	40	5.154	5.149
	1	3	1	85	3.997	4.678	30	4.660	4.659	30	4.981	4.949	30	4.970	4.949
	2	2	1	20	3.759	4.414	30	4.407	4.401	15	4.668	4.659	15	4.664	4.659
	2	3	1	100	3.542	4.355	45	4.318	4.362	110	4.410	4.401	110	4.403	4.401
	2	6	0	35	3.360	3.875	55	3.875	3.872	15	4.351	4.362	15	4.350	4.362
400,	2	4	1	180	3.198	3.790	10	3.789	3.799	15	3.898	3.872	15	3.896	3.872
	3	3	1	100	3.054	3.137	80	3.102	3.088	20	3.789	3.799	20	3.786	3.799
	0	8	0	40	2.831	3.050	115	3.024	3.002	70	3.102	3.088	70	3.098	3.088
	2	6	1	10	2.690	3.016	45	2.989	3.001	30	3.102	3.088	30	3.098	3.088
	4	1	1	90	2.623	2.931	70	2.931	2.976	3	3.102	3.088	3	3.098	3.088
	4	6	0	65	2.565	2.918	50	2.918	2.905	55	2.990	2.976	55	2.984	2.976
	5	1	0	75	2.449	2.885	40	2.885	2.869	2	2.990	2.976	2	2.927	2.976
	1	9	1	40	2.449	2.885	40	2.885	2.869	3	2.990	2.976	3	2.927	2.976
	2	0	2	40	2.449	2.885	40	2.885	2.869	0	2.990	2.976	0	2.927	2.976
	2	1	2	40	2.449	2.885	40	2.885	2.869	8	2.990	2.976	8	2.927	2.976
2	9	1	40	2.449	2.885	40	2.885	2.869	5	2.990	2.976	5	2.927	2.976	
															75

Natural		300-120			400-120			500-120			600-120		
h	k	I	d obs.	I	d obs.	I	d obs.	I	d obs.	I	d calc.	I	
3	8	40	2:405	20	2:718	40	2:720	15	2:718	35	2:721		
0	6			30	2:706						2:716		
3	1	55	2:260	55	2:558	70	2:538	65	2:558	75	2:551		
2	10				2:559						2:509		
3	9			35	2:516						2:478		
3	10	20	2:124	35	2:509	55	2:319	30	2:509	55	2:329		
4	0	65	2:072	35	2:340						2:319		
4	6	15	1:879	35	2:320						2:317		
6	8		1:759								2:273		
6	0				2:281			15	2:285	25	2:241		
0	12			30	2:264	50	2:248	10	2:248	40	2:234		
6	1	30	1:700		2:240				2:219		2:226		
6	9			10	1:978						1:960		
6	6	60	1:594								1:959		
7	8		1:589								1:959		
8	2				1:964						1:956		
3	15			25	1:931	15	1:908	15	1:908	15	1:905		
6	12		1:580	15	1:739	20	1:720	20	1:720	25	1:727		
4	11				1:730						1:705		
8	4	30	1:550		1:567	10	1:548	30	1:548	20	1:545		
1	8				1:549	10	1:531	30	1:531	10	1:531		
7	1				1:508	40	1:499	35	1:499	35	1:499		
4	12				1:378	30	1:380	10	1:379	30	1:379		
2	8	20	1:519		1:365	15	1:363	15	1:363	30	1:364		
0	9				1:313	25	1:312	25	1:312	40	1:312		
9	3	55	1:415										
		15	1:350										
		75	1:299										

* The intensity at 8:1 Å peak (120) of each run is adjusted as 100 because of that the intensity of this peak does not vary on heating. VS—very strong.

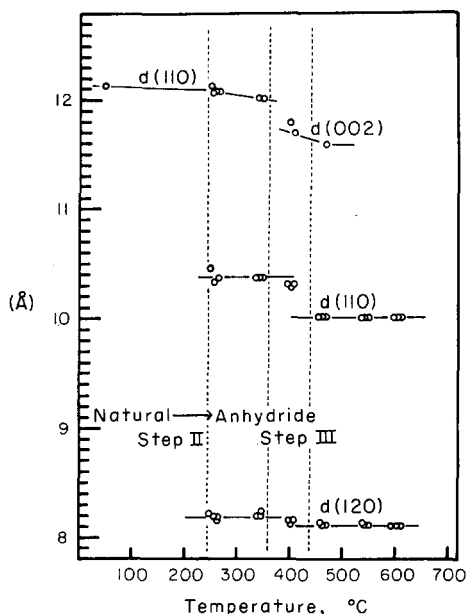


Fig. 4. The change of d -spacings for sepiolite at elevated temperatures as revealed by high temperature X-ray powder diffraction. Mean heating rate was $10^{\circ}\text{C}/\text{min}$. At the required temperatures, the temperature was kept for 1 hr to record the X-ray diffraction data.

of 600–120 in terms of relative positions of peaks have the same index. From these indices and the observed spacings, the lattice constants were again calculated (Table 4). Table 3 shows the observed and calculated spacings of the peaks of 300–120.

As shown in Table 4, a - and b -values clearly decrease in the sample heated at 300°C and a -values successively decrease from step II to step III. The decrease of a -values gives rise to the decrease of the axial ratio (a/b). This result corresponds to that obtained by high temperature X-ray diffraction (Fig. 4).

Infrared absorption spectra

The effects of heat on i.r. absorption patterns are shown in Table 5 and Fig. 6.

The spectra in the region $1400\text{--}400\text{ cm}^{-1}$ are grouped into three: (1) the absorption shown by the

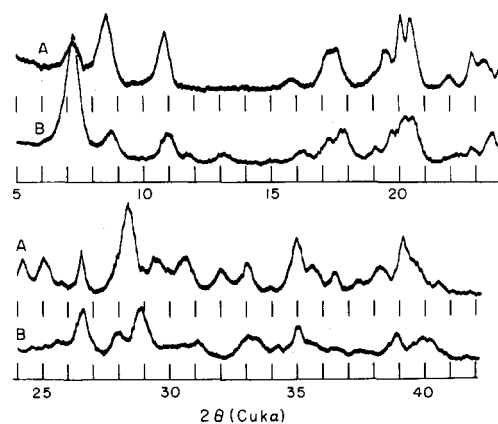


Fig. 5. X-ray powder diffraction patterns of 300–120 (A) and of a mechanical mixture of unheated sample and 600–120 (B).

original unheated sample; (2) the pattern shown by samples 300–120 and 300–250; and (3) the pattern shown by samples 400–120, 400–200, 500–120 and 600–120. Group (2) is composed of its own characteristic bands in addition to weak bands which appear in groups (1) and (3). Occurrence of these characteristic bands in group (2) is due to change from the structures giving rise to the patterns of groups (1) and (3).

As already reported, the heat-induced changes of peaks in regions $4000\text{--}3000\text{ cm}^{-1}$ and $1700\text{--}1600\text{ cm}^{-1}$ are mainly due to dehydration of zeolitic and bound water (Otsuka *et al.*, 1968; Hayashi *et al.*, 1969). The patterns in these regions can be only grouped to correspond to (1) and (3) in the region $1400\text{--}400\text{ cm}^{-1}$.

Though assignment of all these peaks is not possible at present, except in the OH-stretching region, the absorptions in the region $1400\text{--}400\text{ cm}^{-1}$ which appear in 300–120 and 300–250 are due to an intermediate state at step II.

Rehydration

Sample 300–120 rehydrates at room humidity as revealed by the increase in intensity of the 12.2 \AA reflection. On the other hand, 600–120 does not so

Table 4. Lattice parameters (\AA) of preheated and original sepiolite and axial ratios a/b

	a	b	c	a/b
Natural	13.43	26.88	5.281	49.96
300–120	11.2	23.1	5.3	48.5
400–120	10.95	23.2	5.28	47.1
500–120	10.94 ₅	23.2	5.28	47.1
600–120	10.94	23.23	5.28	47.09
Sepiolite anhydride Preisinger (1960)	10.9	23.3	5.28	46.8

rehydrate, nor in water, nor under hydrothermal conditions.

Preisinger (1963) suggested that under low water vapor pressure the phase transformation sepiolite to 'sepiolite anhydride' is reversible. Nathan (1969) reported that the irreversible transformation occurs at about 500–550°C. However, the rehydration ability to original sepiolite is concerned with two stages of removal of bound water.

DISCUSSION

Bound water is removed in two steps, II and III. The former is due to removal of half of the bound water, the latter to removal of the other half.

At step II, dehydration occurs rapidly as shown by the steep slope of the TG-curve. Along with this dehydration, the original structure is modified as shown by the changes in the X-ray powder reflections.

Table 5. Infrared absorption bands (cm^{-1}) of sepiolite; untreated and pre-heated samples

Natural	300–120	300–250	400–120	400–200	500–120	600–120
3688 w	3688 w		3669 m	3670 m	3670 m	3673 m
	3673 w					
3650 w	3650 w					
3623 w	3630 w		3593 m	3595 m		
	3598 m					
3563 m	3525 m		3525 m	3525 w		
1657 b	1657 b					
1620 b	1610 m		1610 m			
	1208 b	1208 b	1208 b			
1197 b				1153 m	1153 m	1153 m
	1145 m	1145 m	1150 m			
				1125 b	1127 b	1129 b
				1098 b	1100 w	1100 m
1073 m	1073 m	1073 m	1092 w	1077 b	1078 b	1078 b
		1050 b	1075 b	1057 b	1057 b	1057 b
			1058 b	1022 St	1022 St	1025 St
1017 St	1010 St	1010 St	1017 St			
974 m	973 m	973 m	975 w	975 w	975 b	975 m
				960 w	957 w	957 w
	937 m	937 m				
			898 m	897 m	897 m	897 m
			813 m	814 m	815 m	815 m
	810 m	809 m				
			800 w	800 w	800 w	800 w
783 m						
	766 m	765 m				
			756 m	753 m	752 m	752 m
727 w			724 b	723 b	726 m	727 m
	720 w	720 w				
690 m	688 m	688 m	688 m	687 m	687 m	685 m
	655 w	655 w	655 m	657 m	657 m	658 m
648 m	644 m	643 w				
			632 w	633 b	633 w	633 w
	618 m	618 m				
			585 w	588 w	585 w	585 w
			567 w	565 w	565 w	565 w
			550 w	550 w	550 w	550 w
533 w	535 w	535 w	531 w	533 m	532 m	532 m
				487 w	485 w	487 w
			470 b	474 m	473 m	473 m
467 m	465 b	465 m		460 w	460 w	460 w
			443 b	443 m	440 m	442 m
432 w	437 b	438 b				

* The notations are as follows: *b*—broad, *m*—medium, *St*—strong, *w*—weak absorption.

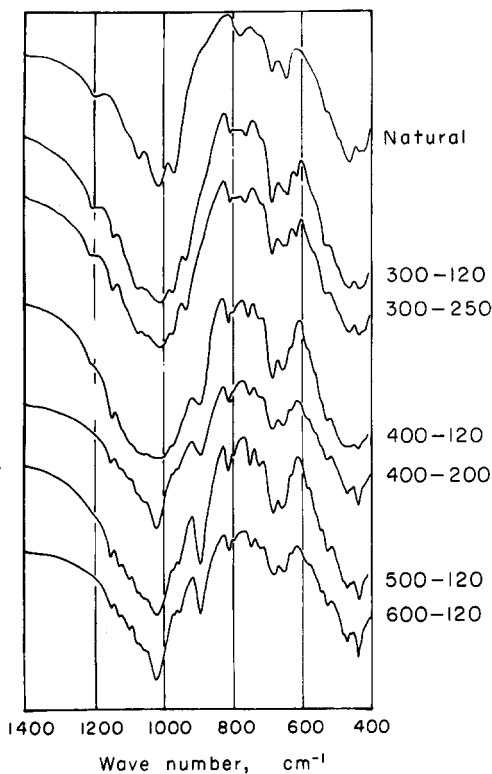


Fig. 6. Infrared absorption spectra of sepiolite C1400-400 cm^{-1} ; untreated and pretreated samples.

At step III, the slope of the dehydration step is more gentle than that of step II. Corresponding to this dehydration, discontinuous changes are confirmed in

such properties as a -parameter, axial ratio, and spacings and intensities of X-ray reflections.

The variation of the axial ratio on heating suggests that slight distortion has arisen within each unit of the dehydrated structure; that is, if the dehydrated structure is only deformed as a whole by the rotation of undistorted units from the original sepiolite structure, a - and b -values would decrease slightly but the axial ratio would not change.

The discontinuity of the change in properties from dehydration step II to III eliminates the possibility that the properties of step II are due to an interstratification of the original sample and its completely dehydrated phase, because such an assumed interstratified structure would display a gradual, not an abrupt, change of properties.

Thus finally, the dehydration behavior of sepiolite may be shown schematically as in Fig. 7.

Acknowledgements—The authors wish to thank Dr. T. Watanabe of Kyushu University and Mr. Y. Nakajima of Osaka University for their valuable suggestions and discussions. We are also indebted to Dr. M. Sato of Gumma University and Dr. Y. Ishikawa for the use of their computer programs.

Part of the expense of this work was defrayed by a grant for scientific research from the Ministry of Education of the Japanese Government.

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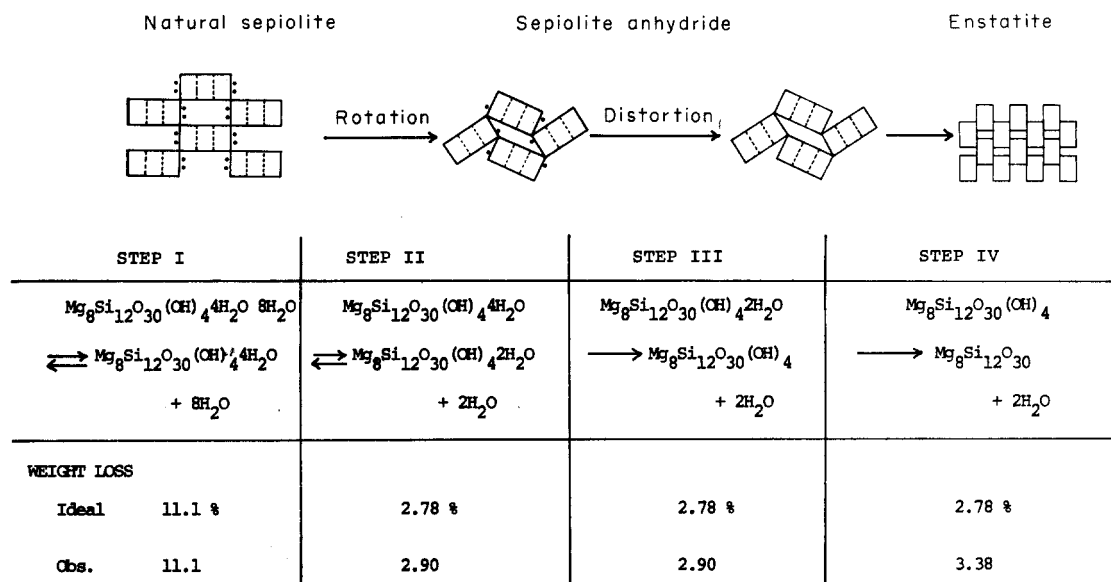


Fig. 7. Schematic figures of structural changes of sepiolite on heating. Small circle: bound water.

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Résumé—L'eau liée de la sépiolite part en deux stades dans le domaine de températures de 250–650°C, comme le montrent les courbes d'ATG. Les étapes sont décrites ici comme les stades II et III. Au stade II, la moitié de l'eau liée est éliminée; l'autre moitié est éliminée au stade III. Du stade II au stade III, des changements discontinus sont confirmés dans des propriétés telles que l'énergie d'activation de la déshydratation, la dimension a , le rapport axial, les intensités et les espacements des diagrammes de diffraction X de poudre. Un état structural au stade II peut être reconnu comme une phase distincte au cours du processus de déshydratation.

Kurzreferat—Gebundenes Wasser wird von Sepiolit in zwei Stufen im Temperaturbereich von 250–600°C abgegeben, wie an TG-Kurven gezeigt wird. Diese Stufen werden hier als Stufen II und III bezeichnet. Bei Stufe II wird die Hälfte des gebundenen Wassers entfernt, die andere Hälfte bei Stufe III. Zwischen Stufe II und III werden diskontinuierliche Veränderungen in solchen Eigenschaften wie Aktivierungsenergie der Dehydratisierung, a -Dimension, Achsenverhältnis und den Intensitäten und Abständen der Röntgenpulverreflexe festgestellt. Der Strukturzustand bei Stufe II kann als eine bestimmte Phase im Entwässerungsprozeß angesehen werden.

Резюме — Связанная вода сепиолитов дегидратируется по двум этапам в пределах температур от 250 до 650°C, как показано на кривой ТГ. Эти этапы мы называем ступенями II и III. На II ступени удаляется половина связанной воды, а другая половина на III. От II ступени до III, подтверждаются прерывистые изменения в таких свойствах как: энергия активации дегидратирования, a -размер, отношение осей и интенсивности и расстояние между рентгеновскими отражениями на порошкограммах. Структурное состояние на ступени II можно считать определенной фазой процесса дегидратирования.