# Ca-bearing Rectorite-type Mineral from Roseki Deposits, Japan

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Several specimens of rectorite-type regularly interstratified mineral (including Ca-rich one of hydrothermal origin) from roseki deposits together with a few rectorites were examined by chemical analysis, X-ray diffraction and IR spectroscopy.

These specimens are rich in SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and H<sub>2</sub>O in addition to considerable amounts of CaO, Na<sub>2</sub>O and K<sub>2</sub>O. The Ca content in mica-like layers ranges from a slight amount to exceeding 50% of the total interlayer population. For the Ca-bearing specimens, Al-for-Si substitution in the tetrahedral sheets of mica-like layers increases in proportion to the Ca contents. Characteristic absorption bands of 950-900 cm<sup>-1</sup> and 700-670 cm<sup>-1</sup> are observed in IR spectra. The bands are due to Al-for-Si substitution in the tetrahedral sheets. Judging from the chemical analysis and IR spectra, the component of mica-like layer rich in Ca is not that of mica but mainly that of margarite (brittle mica). Expansion characteristics of the Ca-rich mineral are similar to rectorite and expandable layer is close to beidellite. The mineral is somewhat less expandable than rectorite under the condition of RH70-80% (Na-saturation) and EG treatment (K-saturation).

The rectorite-type Ca-rich mineral reported here is similar to rectorite in its expansion characteristics, but the component of mica-like layers is different from that of rectorite. Mica-like layer of the mineral must be mainly composed of margarite-like layer. The mineral is mainly composed of a 1:1 regular interstratification of dioctahedral britlle mica (margarite) and smectite (beidellite). The mineral does not strictly correspond to rectorite, defined as a 1:1 interstratified mineral of dioctahedral mica and smectite.

Key words: rectorite, brittle mica/smectite, margarite, regular interstratification

# I. Introduction

Among regularly interstratified minerals, the occurrence of rectorite with long spacing of 25Å and corrensite and tosudite with 30Å, although small in amount, is widely known (Nagasawa, 1973). Rectorite was also described as the name of allevardite, and many documented reports have been released since the 1950s on the fact that the mineral occurres in sedimentary rocks and regions of hydrothermal alteration (Bradley, 1959; Caillère and Hénin, 1950; Heystek, 1954; Brindley and Sandalaki, 1963; Pever et al., 1980). In Japan, many investigations concerning minerals from the roseki deposits (pyrophyllite, kaolin) and various types of hydrothermal deposits (Shimoda and Sudo, 1960; Sudo et al., 1962; Yamamoto, 1965; Henmi, 1966; Tomita et al., 1981; Matsuda

et al., 1981a,b; Nishiyama et al., 1981; Matsuda, 1984) have been made since rectorite was found in the Kanakura Mine, Nagano Prefecture by Takeshi (1958).

From the studies of Brindley (1956), Brown and Weir (1963), Kodama (1966), and others, it has been known that rectorite is a regularly interstratified mineral with a mica-like layer (nonexpandable) and smectite-like layer (expandable) in a ratio of 1:1. The mica layer has a composition of paragonite, whereas the smectite layer has a beidellite-like composition.

AIPEA Nomenclature Committee (Bailey, 1982) made the following recommendation regarding the usage of species name, rectorite. The name rectorite is justified for a 1:1 regular

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interstratification of dioctahedral mica/smectite. The kind of smectite should not be specified in the definition. A pre-fix Na-, K-, or Ca- can be used to specify the dominant interlayer cation in the mica component. It is necessary for the regular interstratification that well defined series of at least ten 00*l* summation spacings are given and that the coefficient of variation (CV) of the d(00l) values is less than 0.75. In addition, the name of allevardite shall never be used in consideration of priority in designation.

The authors have encountered some types of regularly-interstratified Ca-bearing 25Å minerals in a study on the genesis of the roseki deposit. The mica-like layer of rectorite from regions of hydrothermal alteration often has a paragonite or muscovite composition. Among them, specimens containing a considerable amount of Ca have also been reported (Matsuda et al., 1981b; Nishiyama et al., 1981; Matsuda, 1991; Matsuda et al., in press). In this report, an investigation has been made with respect to Ca-bearing minerals.

#### **II. Specimen**

Seven specimens obtained respectively from roseki deposits in the Sano Mine, Nagano Prefecture and in the Ohtoge Mine, Yamagata Prefecture were used in this study. The Sano Mine was situated northeast Nagano Prefecture, and the deposit is formed by hydrothermal alteration of porphyrite in late Tertiary Period (Inoue, 1964). The Ohtoge Mine situated in the southern end of Yamagata Prefecture was formed by hydrothermal alteration of acidic brecciated tuff in the Miocene Age. In Table 1, the occurrence of

Table 1. Occurrence and Associated minerals of specimens

specimen	Occurrence	Associated minor	minerals trace	Locality
1	vein	Q	S,K	Ohtoge
2	vein	Р	Q	Sano
3	psd	Q	Р	Sano
4	psd	Q	S,K	Sano
5	$\mathbf{psd}$	K	Q,S,B	Sano
6	$\mathbf{psd}$	K,Q	B,Chl	Sano
7	psd	K	Q,S	Sano

1 - 3; rectorite, 4 - 7; Ca-rich rectorite-type mineral psd; pseudomorph after plagioclase, B; beidellite, Chl; chlorite, K; kaolinite, P; pyrophyllite, Q; quartz S; sericite.

the rectorite-type minerals of the 7 specimens and accompanying minerals are shown.

In the Sano Mine, a specimen was taken from a vein in the pyrophyllite ore body (composed of pyrophyllite, quartz, and rectorite). Five other specimens were obtained from pseudomorphs after plagioclase in the alteration zone around the Among the specimens from the pseudodeposit. morph after plagioclase, Ca content ranges from slight (specimen 3) to a considerable amount (specimens 4-7). Specimen 1 from the Ohtoge Mine was obtained from a vein in the pits. The vein is comprised mainly of quartz and rectorite.

For the sake of comparison, rectorites from Mitsuishi, Okayama Prefecture (Yamamoto, 1965), and from Baluchistan, Pakistan (Kodama, 1966), syntheytic margarite-beidellite (Matsuda and Henmi, 1983), beidellite from the Sano Mine (Matsuda, 1988) and montmorillonite from Tsukinuno (Matsuda, 1989) were also used.



Fig. 1. X-ray powder patterns of specimen(1,3,5 and 7). basal spacing (00*l*); Å. K; kaolinite, P; pyrophyllite, Q; quartz, S; sericite.

#### **III. Experimental method**

The specimens contain considerable amount of quartz, whereas kaolin, pyrophyllite, sericite, (chlorite), beidellite, etc. occur in small or trace quantities. Although hydraulic elutriation was conducted, these impurities could not perfectly be removed. An X-ray diffraction diagram of several specimens is shown in Fig. 1.

Chemical analysis was made by the wet method. For the impurities contained in the specimens, the amounts of quartz and pyrophyllite were adjusted by their contents from the X-ray diffraction by means of a standard addition method. Exchangeable cations were extracted at 0.1N HCl, and cation exchange capacity (CEC) was quantitatively measured in accordance with atomic absorption and flame methods by extracting at 0.1M SrCl<sub>2</sub> from the Mg-saturated On the other hand, chemical analysis specimen. of plagioclase (exclusively with Ab,Or, and An components) in the host rock with very weak alteration was done by means of EPMA.

X-ray diffraction was conducted by using oriented aggregates under the condition of relative humidity (RH) 30 - 90%, EG and Gly treatment, Greene-Kelly test, and 600°C after heating. The



Fig. 2 Na-K-Ca plots of interlayer cations in mica-like layers.

open circle; present data. solid circle; previous studies. Sy; synthetic margarite beidellite (Matsuda and Henmi, 1983). Other data are from Brindley (1956), Brown and Weir (1963), Rodoriguez and Peretz (1965), Cole (1966), Kodama (1966), Kodama et al. (1969), Matsuda et al. (1981a,b) and Nishiyama and Shimoda (1981). basal spacing was obtained by averaging values of the basal reflections less than  $2\theta$  30° (CuK $\alpha$ ) except for (001) reflection.

Infrared absorption spectrum was measured by means of the KBr disk method.

## **IV. Results and discussion**

1. Chemical composition

In Table 2 chemical analyses of the specimens are given, whereas cation compositions calculated on an O<sub>20</sub>(OH)<sub>4</sub> basis are shown in Table 3. These specimens are respectively comprised of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and H<sub>2</sub>O as main components and Na<sub>2</sub>O, K<sub>2</sub>O and CaO as subsidiary components. The CaO content ranges from a slight to a considerable amount. Aluminium accounts for almost the all cations occupying the octahedral site, and only a small amount of layer charges caused by the octahedral sheet is found. The Al occupation in the tetrahedral site is different from specimen to specimen, and the Si/Al ratio is 2.72 - 5.45, indicating a wider range than the values of Matsuda et al. (1981a). Since almost all the layer charges are in the tetrahedral sheets, chemical compositions of the expandable layer may be similar to beidellite.

The exchangeable cations are comprised of Na, K, Mg,Ca, and Al, and a few of the specimens contain a considerable amount of Al. Rectorite



Fig. 3. Ab-Or-An composition plots of plagioclase in the host rock.

	1	2	3	4	5	6	7
$SiO_2$	48.13	47.78	44.46	43.85	44.12	43.68	41.25
$TiO_2$	0.05	0.12	0.10	0.09	0.11	0.07	0.07
$Al_2O_3$	30.43	30.98	31.85	33.96	33.48	35.54	36.48
$Fe_2O_3$	0.94	0.51	0.24	0.53	0.87	0.65	0.67
MnO	-	-	-	-	-	0.02	tr
MgO	0.07	1.11	0.45	0.76	0.74	0.42	1.27
CaO	1.56	1.53	1.27	3.32	3.51	3.37	3.97
$Na_2O$	2.20	2.01	1.84	1.19	1.60	1.02	0.99
K <sub>2</sub> O	2.41	2.84	2.01	1.72	1.23	0.76	0.83
$P_2O_5$	0.09	0.04	tr	0.01	0.02	0.03	0.01
$H_2O(+)$	5.67	6.51	6.80	6.74	7.54	7.40	7.19
$H_2O(-)$	8.16	7.10	10.67	7.11	6.30	6.01	6.99
Total	99.71	100.53	99.69	99.29	99.52	98.97	99.72
CEC(meq/100g)	59	57	49.2		46	32.2	44

Table 2. Chemical analyses and CEC values of Samples

Table 3. Cation compositions based on  $O_{20}(OH)_4$ 

	1	2	3	4	5	6	7
Tetrahedral							
Al	6.76	6.65	6.47	6.20	6.23	6.13	5.85
Si	1.24	1.35	1.53	1.80	1.77	1.87	2.15
Octahedral							
Ti	0.02	0.01	0.01	0.02	0.01	0.01	0.01
Al	3.80	3.73	3.93	3.78	3.77	3.90	3.94
${f Fe}$	0.10	0.05	0.03	0.07	0.09	0.06	0.07
Mg	0.02	0.22	0.10	0.14	0.13	0.08	0.08
Interlayer							
Fixed							
Na	0.44	0.40	0.47	0.28	0.34	0.28	0.27
K	0.37	0.41	0.34	0.28	0.17	0.13	0.15
Ca	0.05	0.13	0.19	0.46	0.51	0.51	0.59
Exchangeable							
Na	0.08	0.14	0.05	0.04	0.11		
K	0.04	0.09	0.03	0.03	0.05	0.01	
Mg		0.01	0.10	0.02	0.03	0.01	0.16
Ca	0.15	0.10	0.01	0.04	0.02		
Al				0.08	0.03	0.11	
Charges							
Tetrahedral	-1.24	-1.35	-1.53	-1.80	-1.77	-1.87	-2.15
Octahedral	-0.18	-0.18	-0.08	-0.09	-0.12	+0.08	+0.23
Interlayer							
Fixed	+0.92	+1.07	+1.19	+1.48	+1.53	+1.43	+1.87
Exchangeable	+0.42	+0.45	+0.30	+0.43	+0.35	+0.25	+0.32

reported up to now is generally comprised of Na, Mg, and Ca as exchangeable cations. K is seldom contained in the mineral. However, the material associated with the roseki deposit contains Na, K, Mg, and Ca in various ratios (Matusda, 1984,1991).

Non-exchangeable cations are comprised of Na, K, and Ca and some of the specimens dealt with in this study are rich in Ca. Interlayer cations Na, K, and Ca in the mica-like layers of the present specimens and rectorites reported so far are shown in Fig. 2. Most of the minerals reported as rectorite are rich in Na and K (Shimoda et Some minerals associated with al., 1974). roseki deposits contain a considerable amount of Ca in addition to Na and K (Matsuda et al., 1981a; Nishiyama et al., 1981; Matsuda, 1991). Although the specimens are relatively rich in Ca (eg Sa, To, and Mi, Fig.1; Matsuda, 1984), the specimen 4-7 in this study contains a greater amount of Ca. With specimen 7, it is evidently seen that Ca accounts for more than 50%. No such rectorite-type mineral with Ca exceeding 50% in the interlayer cations of the mica-like layers have been reported so far (Matsuda et al., in press).

A triangular diagram indicating the Ab-Or-An composition of the plagioclase in the host rock of the Sano Mine is shown in Fig. 3. Plagioclase containing the Ab and An components of almost the same degree and the one containing more than 80% of the An component are found. Ca-bearmineral ing rectorite-type occurs as а pseudomorph after plagioclase in the Sano Mine. No clear relation can be observed between the occurrence and the Ca content of the mineral. Carich mineral may have formed from plagioclase high content of the An component.

A slight amount of Al occupies the tetrahedral sites in the beidellite layer since the expandable layer of the rectorite is similar to beidellite, but the tetrahedral sites in the mica-like layer has the cation component of almost the same as  $Si_3Al_1$  (Brown and Weir, 1963; Kodama, 1966). However the Ca-rich rectorite-type mineral (specimens 4-7) dealt with in this study has a much greater amount of Al than rectorite reported so far (Table 2). In Fig. 4, a relation between Ca/Na+K+Ca of the interlayer cations in the micalike layers and the Al of the whole tetrahedral sites is shown. The broken line A gives the amount of Al substitution on the assumption



that the mica-like layer is regarded as paragonite (or muscovite) and the smectite-like layer as beidellite. On the other hand, the broken line B shows the amount of Al substitution on the assumption that the mica-like layer is regarded as margarite of dioctahedral brittle mica, and the smectite-like layer as beidellite. The specimen whose mica-like layer is rich in Na and K is placed in the neighborhood of the broken line A. In within line A and B (Fig. 4), the specimen containing Ca approaches the broken line B as its Al content increases. Furthermore, accompanied with the increase of Ca, it is perceived that the amount of the tetrahedral A1 increases. This fact might indicate, it is assumed, that the mica-like layer of the rectorite-type mineral is comprised not only of mica but also of the solid solutions of mica (paragonite, muscovite) and brittle mica (margarite). From the above observation. it is assumed that more than 50% of the mica-like layer of Ca-rich rectorite-type minerals (speci-





Fig. 5. Behaviors of basal spacing in Na saturated specimens with relative humidity. open circle; present specimen (1,2,5), solid circle; rectorite from Mitsuishi (A, Matsuda et al., 1981) and Pakistan (B, Kodama, 1966). triangle; Tsukinuno montmorillonite (C), Sano beidellite (D).

men 7) are of margarite composition.

#### 2. X-ray diffraction

Rectorite, which is a 1:1 regular interstratification of dioctahedral mica and smectite, shows basal reflection of integral series as shown in Fig. 1. With the condition of EG, Gly treatment, (00l) reflections of more than 10 are obtained. From comparison of the strengths of the (001) and (002) reflection (Fig. 1), it is distinctly conceived that the (002) reflection of specimen 1 is greater than that of specimens 5 and 7. The above phenomenon might be related to the chemical compositions of the mica-like layer.

In Fig. 5, changes of basal spacings are shown in the relative humidity (RH) 30-90% of specimens 1, 2, and 5 (this study) in comparison with Ca-bearing rectorite from Mitsuishi (Matsuda et al., 1981b), rectorite from Pakistan (Kodama, 1966), beidellite, and montmorillonite (Matsuda, 1988). In Table 4, the basal spacing after ethylene glycol (EG) and glrycerol (Gly) treatment, Greene-Kelley test, and 600°C heating is The expandability of the rectorite is less shown. than montmorillonite under conditions of each relative humidity, and EG and Gly treatment. The value is either greater than or almost the same that of beidellite (Matsuda, 1989). The specimens used in this study indicate that the swelling almost the same as that of rectorite.

Specimen	EG	Gly	Greene-	Heated			
-		Kelly test					
1	26.36	27.61	27.10	19.5			
2	26.94	27.51	27.04	19.4			
5	( 26.8	27.25	26.94	19.3			
	24.5						
Mi	<sub>1</sub> 26.13	$^{\prime} 27.2$	26.85	19.2			
	23.34	23.8*					
Bal	26.60	27.53	27.20	19.4			
Ts	17.28	17.80	9.82	9.8			
			<sup>\</sup> 17.32*	k			
Sa	14.06	17.80	17.26	9.9			

Table 4. Basal spacings (Å) of various treatments

EG; treatment with ehtylene glycol (saturated with  $K^+$ ). Gly; tretment with glycerol (saturated with  $Mg^{2+}$ ) Heated; after heateing at 600 °C for 3 hours (saturated with Na<sup>+</sup>).

However, under the condition of RH70-80% and EG treatment, specimens 1 and 2 are liable to expand as in the case of rectorite from Pakistan, and is still apt to expand rather than specimen 5 and Ca-bearing rectorite from Mitsuishi. The phenomenon may supposedly be related to the amount of Ca in the mica-like layer. With the regularly interstratified mica/smectite, the authors consider that the structure of the layer is derived from model B (Nagasawa and Tsuzuki,1974; where the Al-Si compositions of the tetrahedral sites of the mica-like layer and smectite-like layer are different from each other). In the case of the Ca-rich mineral, not only the mica-like layer but also the expandable layer (the smectite-like layer) become greater in their Al-for-Si substitution of the tetrahedral sites because the mineral was formed in an environment rich in Al. Thus small expandability is believed to be exhibited.

#### 3. Infrared absorption spectrum

In Fig. 6, the infrared absorption spectrum of specimens 2 and 5 and the synthetic regularly interstratified margarite/beidellite (Matsuda and Henmi, 1983) is shown.

For the synthesized material as well as the specimen 6, distinct absorptions appear in the region of 950-900cm<sup>-1</sup> and 700-670cm<sup>-1</sup>. A separate peak occurs at 490-470cm<sup>-1</sup>. Such absorptions do not come out in the case of minerals



Fig. 6. IR absorpton spectra. 2,5; present specimen, Sy; synthetic margarite/beidellite (Matsuda and Henmi, 1983).



Fig. 7. Relationship between Ca/(Ca+Na+K) of mica like layers and relative intensity ratio  $I930cm^{-1}/I1050cm^{-1}$ .

open circle; present data, solid circle; previous data from Matsuda et al.(1981, in press) and Sy; synthetic margarite/beidellite. such as muscovite, paragonite, beidellite, montrillonite, etc. (Farmer and Russell, 1964). The absorption in the region of 950-900cm<sup>-1</sup> and 700-670cm<sup>-1</sup> are believed to be caused by the Al-Si substitution of the tetrahedral site deriving from the margarite structure (Farmer and Russell, With specimen 2, the absorption in this 1964). region is quite scanty. In the case of specimen 5, the absorption in these regions is a little smaller than that of the synthesized material. With the synthesized regularly interstratified margarite /beidellite, the mica-like layer has the compositions of margarite because almost all the interlayer cations are occupied by Ca. This allows the tetrahedral site to possess almost ideal Al-Si compositions with which the margarite and beidellite are comprised in a ratio of 1:1 (Matsuda and Henmi, 1983).

Peak intensities of absorption bands in the regions of 900-930 cm<sup>-1</sup> and 670-700 cm<sup>-1</sup> increase as Ca content in the mica-like layers increase (Matsuda, 1991). The relationship between relative intensity of absorption bands at 900-930cm<sup>-1</sup> and Ca content of mica-like layers of rectoritetype minerals is shown in Fig.7. Similarly, the relationship between relative intensity of absorption bands at 670-700 cm<sup>-1</sup> and tetrahedral



Fig. 8. Relationship between tetrahedral Al/O20(OH)4 and relative intensity ratio I700cm<sup>-1</sup> / I550cm<sup>-1</sup>. Symboles are same as for Fig. 7.

Al is given in Fig.8. Positive correlations between absorption band intensities and chemical compositions (Ca contents in the mica-like layers and Al contents of tetrahedral sheets) are observed. From the above observation, it can be suggested that the Al-for-Si substitution of the tetrahedral sites increases as the amount of interlayer Ca of the mica-like layer increases.

# V. Conclusions

As a result of the chemical analysis, the interlayer cation of the mica-like layer ranging from a slight amount of Ca to an amount exceeding 50% is observed with the rectorite-type minerals as discussed above. With this mineral, the Al-for-Si substitution in the tetrahedral sites increases as the content of Ca increases.

On the infrared absorption spectrum, characteristic absorption bands accompanied with the tetrahedral Al-Si substitution appear in the region of 950-900cm<sup>-1</sup> and 700-670cm<sup>-1</sup>. From the chemical analysis and infrared absorption spectrum, it is explained that a greater part of the mica-like layer of these minerals is not Ca mica but a brittle mica (margarite).

The expandability of this mineral obtained from the X-ray diffraction is almost the same as that of rectorite, but is slightly smaller than that of rectorite under the condition of the EG treatment (K-type), RH70-80% (Na-type).

Rectorite is defined as a 1:1 regularly interstratified mineral of the dioctahedral-mica /dioctahedral-smectite. In this study, however, the mica-like layers of Ca-rich rectorite-type mineral possess more than 50% of the dioctahedral brittle mica component. This appearantly contradicts the definition of rectorite. Thus it remains to be seen whether this mineral should be regarded as a new species of the mineral or the definition of rectorite should be changed.

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