

CORRENSITE FROM NASŁAWICE (LOWER SILESIA, POLAND): SOME PROBLEMS OF MINERAL IDENTIFICATION AND ORIGIN

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Abstract—The corrensite from a chlorite vein-like rodingite blackwall in serpentinites has been studied. The proper identification of swelling layers in corrensite using heating at 500°C was ambiguous because of the spontaneous rehydration. Even K⁺-saturated samples heated to 500°C readily rehydrated after being cooled. This can be prevented if XRD patterns are recorded at 300°C, without cooling the sample. A standard heating at 500°C can result in partial decomposition of brucite-like sheet as evidenced by ASN simulation.

The ASN-calculated XRD patterns of contracted corrensite proved that an inhomogeneous distribution of heavy atoms (Fe, Ni, Mn, Cr etc.) between brucite-like sheet and talc-like layers and between two adjacent corrensite units in the interstratified mineral may result in the disappearance of low angle reflections (24 Å and 12 Å), which can lead to miscellaneous interpretation if distribution of heavy cations is not checked.

The corrensite occurred together with regular chlorite. However, it is assumed to be formed due to direct crystallization from late hydrothermal solutions as deduced from comparison of the Mg/(Mg + Fe) ratio in the corrensite, serpentinite and chlorite.

Key Words—Calculated X-ray patterns, Corrensite, Thermal contraction, X-ray powder diffraction.

INTRODUCTION

The term corrensite denotes a trioctahedral 1:1 regularly interstratified mineral ($R = 1$) containing chlorite and smectite layers (low-charge corrensite) and chlorite and vermiculite layers (high-charge corrensite) (Reynolds 1988). Corrensite occurs in diverse geological formations. It is widely believed to be a by product of supergene and hydrothermal chlorite alteration (e.g., Mejsner 1977, Nakamuta 1981, Ross *et al* 1982, Proust *et al* 1986, Bodine and Madsen 1987, Shau *et al* 1990), as well as a product of the aggradation of smectite or vermiculite to chlorite (e.g., April 1981b, Proust *et al* 1986, Inoue and Utada 1991, Schiffman and Fridleifsson 1991).

Corrensite is often identified on the basis of the 001 reflection present on X-ray tracings of glycol (or glycerol) saturated and heated samples, particularly in mixtures. However, in some corrensites, the 001 reflection disappeared after heating (e.g., Wiewióra and Szpila 1975, April 1981b, Lippmann and Pankau 1988), making proper identification of the corrensite and other layer silicate mixtures difficult.

The aim of this study was to determine the reason for the disappearance of the 001 reflection in heated corrensites, to develop methods suitable for proper identification of readily rehydrated samples, and to formulate a tool for deciphering the mechanism of

transition sequences involving corrensite in the context of complex geological evolution.

MATERIAL

A sample containing an interstratified smectite-chlorite mineral was found in the serpentinite quarry at Nasławice near Sobótka (Lower Silesia, SW Poland). It occurred in a chlorite rich vein-like blackwall (chlorite-rich zone formed at the expense of serpentinite during rodingitization) adjacent to a clinzoisite rodingitic body (Dubińska 1989). The sample contained chlorite and interstratified smectite-chlorite as major phases, and accessory apatite and zircon. The coarse-grained fraction was rich in chlorite IIb (according to Bailey's notation 1980). As the particle dimension decreased, the content of interstratified smectite-chlorite increased, being dominant in the fraction $<0.5 \mu\text{m}$. Both positions and relative intensities of interstratified phase reflections were constant in different grain classes. The authors failed to obtain either pure chlorite, or pure interstratified smectite-chlorite.

EXPERIMENTAL

Methods

Routine identification was performed on oriented specimens examined with a DRON-2A diffractometer

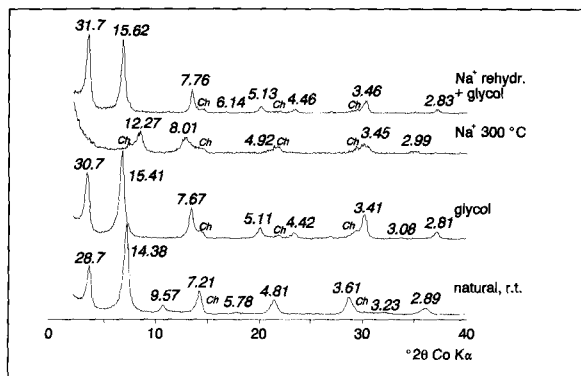


Figure 1. XRD tracings of the studied sample, oriented aggregates, ch—chlorite; Na⁺—sodium saturated sample, r.t.—room temperature (air dried); 300°C—tracing recorded by means of heating stage after one hour preheating (without cooling the specimen); glycol treatment—natural sample and sample Na⁺-saturated and rehydrated after heating.

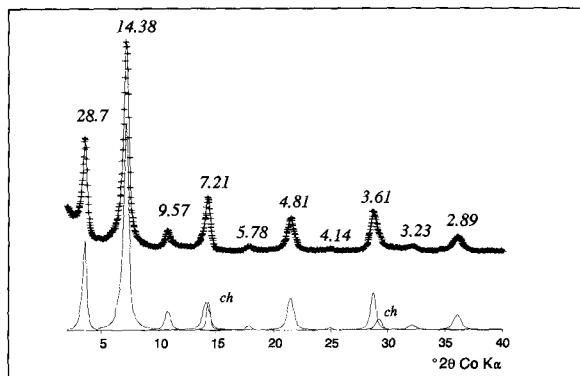
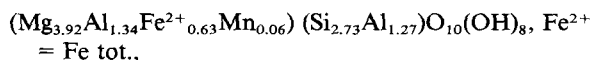


Figure 2. XRD tracing of air dried natural sample (upper, crosses represent experimental points) and its decomposition (lower); ch—chlorite; oriented aggregates.

and Co K α radiation. An untreated sample was saturated with Na⁺, K⁺, and Mg²⁺-ions. X-ray diffraction patterns were recorded conventionally (i.e. cooled specimens), after heating in a furnace (550°C), as well as by means of a homemade thermal stage when the X-ray tracings were recorded at a temperature of 300°C using the GGR Sigma 2070 reflection diffractometer. Homoionic and/or heated samples were also treated with ethylene glycol and glycerol (both liquid).

Deconvolution of the overlapped diffraction bands was performed using the NICEFIT program assuming pseudo-Voigt peak profiles. The ASN program was used to simulate XRD diagrams (Drits and Sakharov 1976, Drits and Tchoubar 1990); crystallite size limits used in the calculations are 2-10 units in equal proportions. The chemical composition of regular chlorite,



from the adjacent rodingite body was used for calculations. About 180 calculations were performed. Chlorite compositions were determined using an electron microprobe (JEOL JXA-8600-electron microscope, 20 keV, 7 mA, and beam focused at ϕ 1 μm , ZAF correction procedure, synthetic silicates and oxides as well as natural mineral standards) at the Department of Geology, Hebrew University, Jerusalem. Studied minerals are trioctahedral, indicated by their b parameter equal to 9.204 Å (corrensite) and 9.228 Å (chlorite from adjacent rodingite).

Ba-saturated coarse grained flakes were obtained by treating cleaved flakes with a 1 M BaCl₂ solution in 25 ml teflon pressure vessel at 120°C over 72 h. The saturating solution was changed daily. The flakes were washed with double-distilled water. Compositions of Ba²⁺-flakes were determined by electron microprobe.

Results and discussion

XRD patterns of untreated samples and ethylene glycol, and Mg and glycerol treated samples show regularly interstratified chlorite-smectite ($R = 1$) with rational series of 28.7 ($cv = 0.52$, cv values were calculated according to Bailey 1982), 30.7 ($cv = 0.32$), and ~ 32 Å peaks, respectively (Figures 1, 2, and 3). Experimental and ASN-calculated positions and intensity reflections of the ethylene glycol saturated sample show good agreement (Figure 4) and, the intensities did not significantly vary regardless of heavy cation distribution. Potassium saturated specimens heated at 500°C displayed a poorly rational series of 27 Å ($cv = 2.49$). After calcination at 700°C interstratified mineral reflections disappeared and only the 13.7 Å reflection of the partly decomposed chlorite admixture was present (Bailey 1975). The result of conventional heating at 500°C was ambiguous. Therefore, thermal contraction of the interstratified phase was studied using XRD patterns recorded by means of a thermal stage at 300°C (without cooling the specimens) to avoid spontaneous

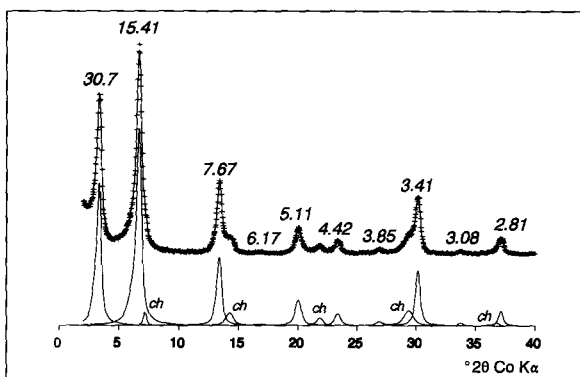


Figure 3. XRD tracing of glycolated sample (upper, crosses represent experimental points) and its decomposition (lower); ch—chlorite; oriented aggregates.

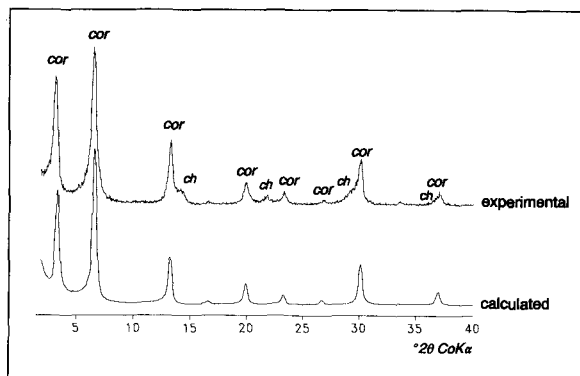


Figure 4. Experimental (upper) and ASN-calculated (lower) XRD tracing of the Mg^{2+} -saturated sample, heated and glycolated after rehydration; oriented aggregates; ch—chlorite, cor—corrensite.

rehydration of the expanding layers in the interstratified phase. The phase contracted to $\approx 24 \text{ \AA}$ (Na-sample— $cv = 1.26$, K-sample— $cv = 1.19$, Figures 1 and 5) readily rehydrated overnight (K-sample— $\approx 26 \text{ \AA}$, $cv = 1.89$). It expanded after glycol saturation and an almost rational series of X-ray reflections was obtained ($cv = 0.99$ or 0.64 for Na- and K-samples, respectively). Hence, conventional heating at 500°C of the K-sample probably produced a one-sheet hydrate of K-saponite layers in the interstratified phase (Kawano and Tomita 1991).

Deconvolution of XRD patterns of a stage-heated, rehydrated and glycolated sample suggested an admixture of ambiguous phase diffraction maxima (newly formed phase— nfp, Figures 5 and 6). These maxima can be tentatively ascribed to an interstratified swelling chlorite-smectite-chlorite using Méring (1949) diagrams modified by Drits and Sakharov (1976) and the procedure of Dubínska (1984) and Jelitto *et al* (1993).

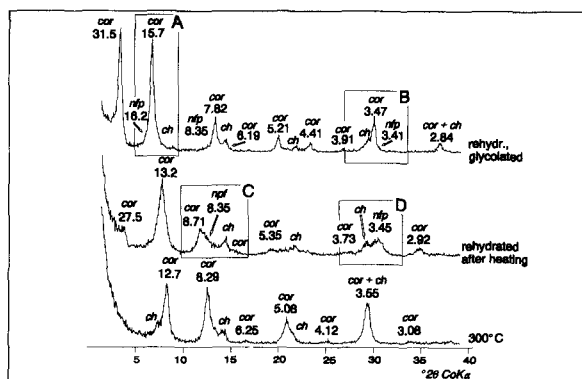


Figure 5. XRD tracings of K^+ -saturated sample, 300°C —tracing recorded by means of heating stage after one hour preheating (without cooling the specimen); ch—chlorite, cor—corrensite, nfp—newly formed phase; oriented aggregates. Areas A, B, C, and D are expanded in Figure 6.

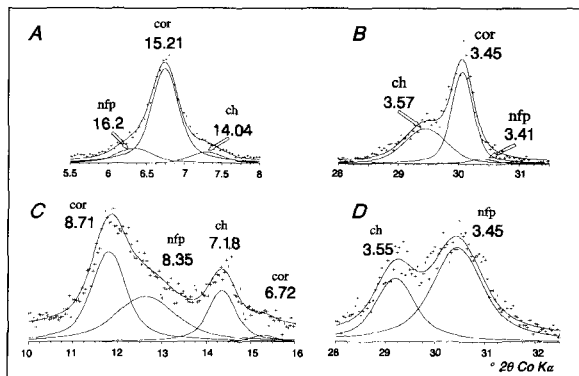


Figure 6. Sections of diffractograms from Figure 5, crosses—experimental points, solid lines represent sum of decomposed diffraction maxima, for other explanations see Figure 5.

Probably the nfp maxima reflect inhomogeneous smectite layer charge distribution (Suquet *et al* 1977), or interlayer contaminants (intergrade-like, MacEwan and Wilson 1980). The interpretation is equivocal because of probable artifacts produced by partial decomposition of the brucite-like sheet.

The 24 \AA -reflection was not present in X-ray tracings of the stage-heated sample (Figure 1 and 5). This reflection is often weak or absent in tracings of 350°C heated corrensite minerals, whereas it was present in tracings of corrensites heated at $\sim 550^\circ\text{C}$ (e.g., April 1981b, Figure 2; Lippmann and Pankau 1988, Figure 12). We suppose that partial thermal decomposition of chlorite layers in corrensite is responsible for appearance of $\sim 24 \text{ \AA}$ reflection in XRD patterns of overheated samples (Figure 7). Neither X-ray pattern of a contracted corrensite calculated by Reynolds (1988) reveals the 24 \AA reflection.

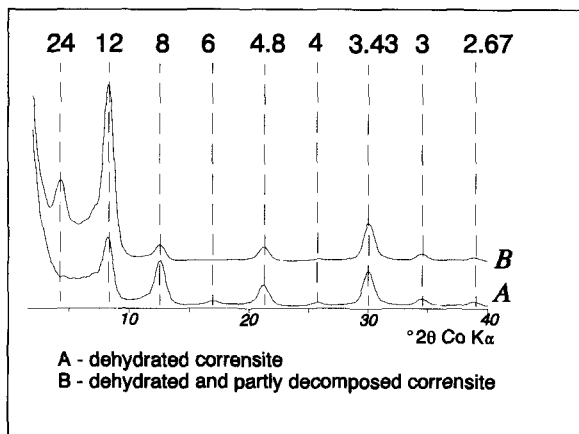


Figure 7. ASN-calculated X-ray tracings of dehydrated (10 \AA K-smectite/ 14.2 \AA chlorite) and dehydroxylated and partly decomposed corrensite, where brucite-like sheet is thermally reduced to 66% as compared with those of regular chlorite outer hydroxide sheet; both XRD patterns are at the same scale.

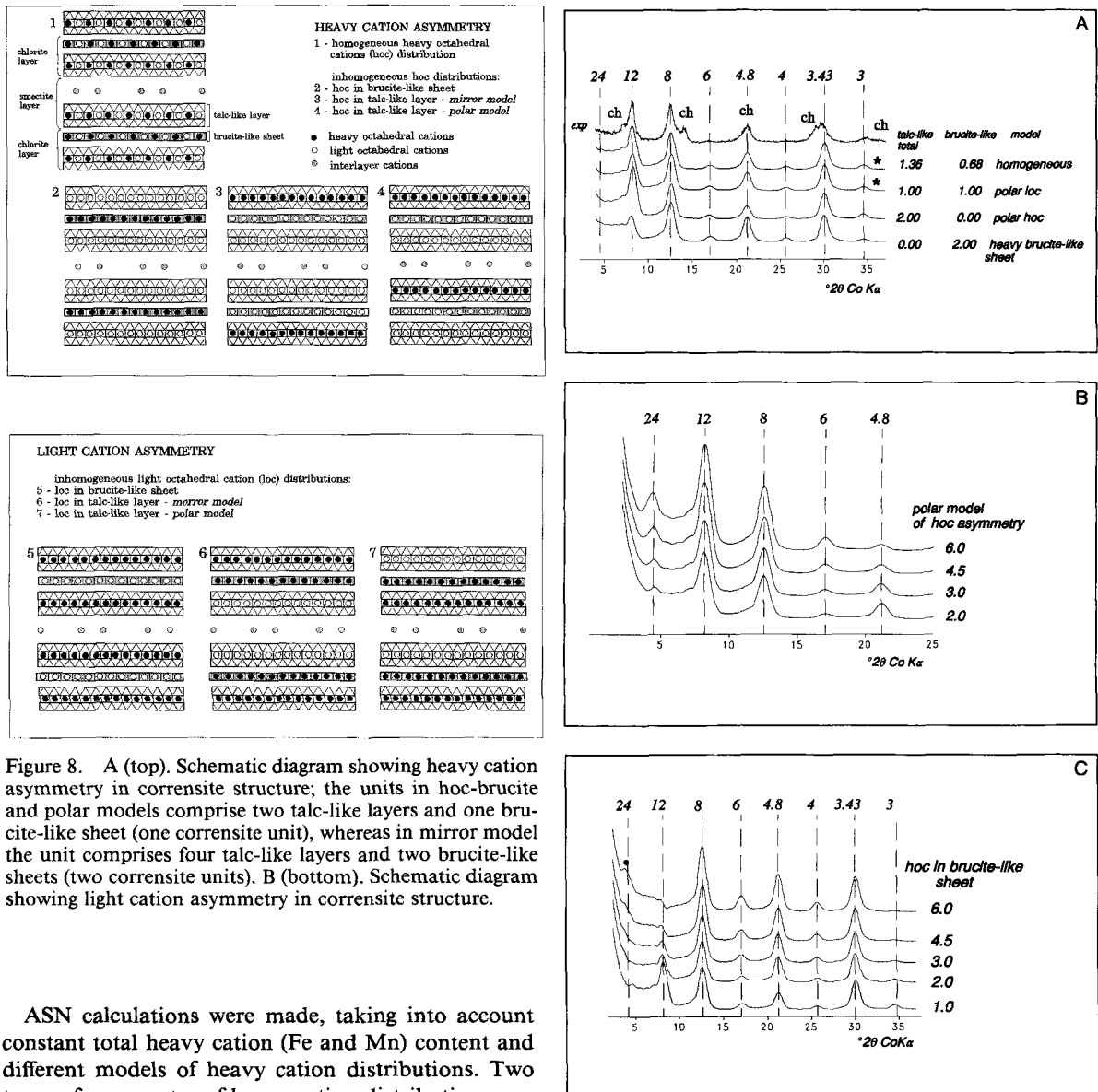


Figure 8. A (top). Schematic diagram showing heavy cation asymmetry in corrensite structure; the units in hoc-brucite and polar models comprise two talc-like layers and one brucite-like sheet (one corrensite unit), whereas in mirror model the unit comprises four talc-like layers and two brucite-like sheets (two corrensite units). B (bottom). Schematic diagram showing light cation asymmetry in corrensite structure.

ASN calculations were made, taking into account constant total heavy cation (Fe and Mn) content and different models of heavy cation distributions. Two types of asymmetry of heavy cation distribution were considered: 1) preferential concentration in one location labelled as heavy cation asymmetry (hoc, Figure 8A); and 2) preferential concentration of "light" cations (Mg and Al) at one location labelled as light cation asymmetry, i.e., heavy cation concentration in two of three possible sheets (loc Figure 8B). Two patterns show the best conformity of experimental and calculated intensities: 1) calculated for homogeneous distribution of heavy cations (Figures 8A-1 and 9A); and 2) calculated for loc polar model (Figures 8B-7 and 9A).

Furthermore, the calculation revealed that the asymmetric location of heavy cations in corrensite may result in the following changes of basal reflection intensities: 1) an increase of heavy cation concentration coupled with its location according to hoc polar model distribution augments the relative 24 Å-reflection in-

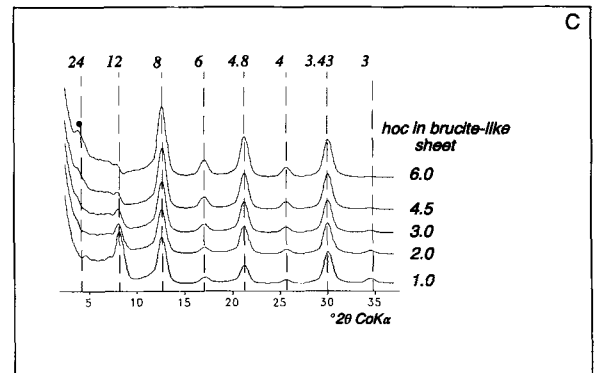


Figure 9. A. ASN-calculations for contracted corrensite displaying constant total iron content (two Fe cations on 50 oxygens) and experimental pattern of heated Na⁺-sample (300°C), talc-like total—total iron content in talc-like layers, brucite like—iron content in brucite-like sheet, model—for explanations see Figures 8A and 8B; asterisk show calculated patterns with the best conformity with experimental tracings. B. ASN-calculations for contracted corrensite displaying different heavy cation content—model polar hoc (Figure 8A, 4); iron contents calculated on 50 oxygens. C. ASN-calculations for contracted corrensite displaying different heavy cation content and its preferential location in brucite-like sheet; black filled circle marks 24 Å-reflection of very low intensity.

tensity (Figures 8A-4 and 9B); minor differences in reflection intensities at X-ray patterns calculated for hoc polar model (Figures 9A and 9B) result from different sequences of the component layers in calculations; 2)

Table 1. Representative compositions of layer silicates from naslawice.

Sample	Na47B ch	Na48D serp	Na18bl Ch > cor	Na18bl cor > ch
SiO ₂	27.47	41.08	32.25	34.77
TiO ₂	—	—	—	—
Al ₂ O ₃	22.24	3.21	12.97	13.34
Cr ₂ O ₃	0.01	0.06	—	0.19
FeOtot.	7.36	0.94	7.09	7.08
MnO	0.75	0.05	0.13	0.14
NiO	—	n.d.	—	—
MgO	26.49	41.52	30.01	25.03
CaO	0.00	0.05	—	—
BaO	—	—	0.11	5.19
K ₂ O	0.02	0.04	0.18	0.08
Na ₂ O	0.04	—	—	—
Total	84.61	86.95	82.76	85.82
	on the basis of O ₁₀ (OH) ₈			
Si	2.73	3.82	3.30	
Al ^{IV}	1.27	0.18	0.70	
Al ^{VI}	1.34	0.18	0.87	
Cr	0.00	—	—	
Fe ²⁺ tot.	0.63	0.07	0.46	
Mn	0.06	—	0.01	
Ni	—	—	—	
Mg	3.92	5.75	4.58	
Tot. oct. cat.	5.95	6.00	5.92	
Mg/(Mg + Fe)	0.86	0.99	0.91	0.86

ch—chlorite, cor—corrensite, serp—serpentine; Na18bl—corrensite-chlorite intergrowths (coarse grained flakes), Mg/(Mg + Fe)—atomic ratio.

preferential location of heavy cation in brucite-like layer combined with an increase of heavy cation content result in gradual disappearance of 12 Å- and absence of 24 Å-reflection; thus, only 8 Å-reflection is present on X-ray tracings (Figure 9C). Such minerals were reported by Wiewióra and Szpila (1975) and Dubińska (1984).

GEOLOGICAL REMARKS

Corrensite seems to be common in two geological situations:

- (1) weathered and hydrothermally altered basalts/andesites and their pyroclastic and low-grade metamorphic equivalents, where corrensite is frequently used as an indicator of geological conditions (e.g., Shirozu *et al* 1975, April 1981a, Khamkhadze *et al* 1981, Shau *et al* 1990, Bettison-Varga *et al* 1991, Drits and Kossovskaya 1990, Inoue and Utada 1991, Robinson *et al* 1993);
- (2) clastic layers in evaporitic series and in alkaline lakes (e.g. Denoyer de Segonzac 1969, Nishiyama *et al* 1979, April 1980, April 1981b, Bodine and Madsen 1987, Drits and Kossovskaya 1990).

In such situations, the chlorite/smectite (or vermiculite) ratio in the interstratified mineral is widely used as a geological indicator (e.g. Proust *et al* 1986, Bet-

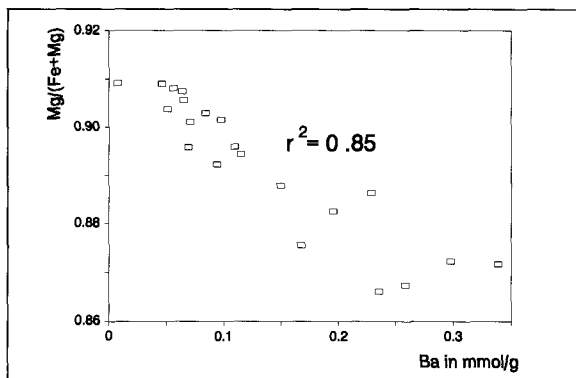


Figure 10. Ba vs. Mg/(Mg + Fe) ratio for coarse grained flakes (chlorite with corrensite intergrowths) treated with 0.5 M BaCl₂ solution; r² represents linear correlation coefficient.

tison-Varga *et al* 1991, Bevins *et al* 1991, Inoue and Utada 1991, Robinson *et al* 1993).

The origin of corrensite as a transitional phase is commonly reported as follows:

- (1) chlorite to smectite (via corrensite) sequences:
 - chlorite/corrensite ratios in geological or soil profiles (Ducloux *et al* 1976, Nakamuta 1981, Proust *et al* 1986, Shau *et al* 1990),
 - admixture of discrete chlorite in corrensite-bearing samples (Wiewióra and Szpila 1975, Brigatti and Poppi 1984),
 - decrease of chlorite layers in fine-grained interstratified minerals as compared with coarse-grained fractions (Johnson 1964, Herbillon and Makumbi 1975),
 - chemical evolution of layer silicates i.e., decrease of Mg, Al, and Fe contents (Buurman *et al* 1988, Ducloux *et al* 1976);
- (2) smectite or vermiculite to chlorite (via corrensite) sequences:
 - geological setting (Denoyer de Segonzac 1969, April 1980, April 1981b, Bodine and Madsen 1987, Inoue and Utada 1991, Schiffman and Fridleifsson 1991),
 - fine-grained fractions rich in mixed-layer minerals with chlorite layers (Drits and Kossovskaya 1990),
 - chemical evolution of layer silicates i.e., enrichment in Mg, Al, Fe etc. as related to the geological evolution of the studied area (Bodine and Madsen 1987, Inoue and Utada 1991, Robinson *et al* 1993).

Experimental alterations of chlorites into corrensite or smectite (vermiculite) were successful for iron-rich chlorite varieties (Ross and Kodama 1976, Senkayi *et al* 1981), whereas Mg-rich chlorites were persistent during experiments (Ross 1975). However, Mg-rich corrensites, believed to be products of chlorite alteration are known (e.g., Brigatti and Poppi 1984).

A transitional path of chlorite degradation or smectite aggradation to corrensite formation would result in the development of chlorite-smectite mixed-layer

minerals with different chlorite/smectite ratios in different grain fractions. The uniform XRD characteristics of corrensite from Nasławice in all grain fractions suggests its non-transitional origin.

The Mg/(Mg + Fe) ratio of chlorite indicates a general change in fluid chemistry and mineral composition (Shikazono and Kawahata 1987, Bettison-Varga *et al* 1991, Bevins *et al* 1991). The corrensite from Nasławice occurred together with regular chlorite; however their Mg/(Mg + Fe) ratios differs (Table 1, Figure 10). In chlorite/corrensite intergrowths rich in chlorite this ratio is close to the Mg/(Mg + Fe) ratio in serpentines from adjacent serpentinites (av. 0.97, Dubińska, unpublished data), whereas in the intergrowths rich in corrensite the Mg/(Mg + Fe) ratio is similar to interstitial chlorite from rodingite (0.86, Dubińska 1989). The changes in the ratio in the corrensitic materials with increasing Ba uptake (Figure 10) reflect increasing amounts of smectite layers. Probably the compositions of both interstitial chlorite and corrensite reflect increasing Fe activity in late hydrothermal solutions. This is shown by zoned epidotes and vesuvianites with rims containing more Fe than the cores as well as hydroandradite occurrence in rodingites from the studied area (Dubińska 1995).

Both iron distribution and unvarying chlorite/smectite ratios in all grain fraction suggest the corrensite should be considered as a direct, newly formed mineral rather than a product of chlorite transformation (according to Drits and Kossovskaya 1990). An analysis of iron distribution in corrensite-group minerals may be important for distinguishing transformation vs. neoformation mechanisms in weathering zones, hydrothermally altered and low-grade metamorphic mafic rocks, etc.

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