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CELADONITE AND ITS TRANSFORMATION TO SMECTITE IN AN ENTISOL AT RED ROCK CANYON, KERN COUNTY, CALIFORNIA

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Abstract—A green, Lithic Torriorthent soil derived from a celadonite-rich, hydrothermally altered basalt immediately north of the Mojave Desert region in southern California was studied to investigate the fate of the celadonite in a pedogenic weathering environment. Celadonite was found to be disseminated in the highly altered rock matrix with cristobalite, chalcedony, and stilbite. X-ray powder diffraction (XRD) showed the soil material to contain celadonite having a d(060) value of 1.510 Å, indicative of its dioctahedral nature. Very little smectite was detected in the parent material, whereas Fe-rich smectite was found to be abundant in the soil. The Fe-smectite and celadonite were identified as the sole components of the green-colored clay fraction (<2 μm) of all soil horizons. The soil clay showed a single d(060) value of 1.507 Å, indicating that the smectite was also dioctahedral and that its *b*-dimension was the same as that of the celadonite. Mössbauer spectroscopy showed that the chemical environments of Fe in the rock-matrix celadonite and in the smectite-rich soil clay were also nearly identical. These data strongly suggest a simple transformation of the celadonite to an Fe-rich smectite during soil formation.

Supporting evidence for this transformation was obtained by artificial weathering of celadonite, using sodium tetraphenyl boron to extract interlayer K. The intensity of the 001 XRD peak (at 10.1 Å) of celadonite was greatly reduced after the treatment and a peak at 14.4 Å, absent in the pattern of the untreated material, appeared. On glycolation of the sample, this peak expanded to 17.4 Å, similar to the behavior of the soil smectite. The alteration of celadonite to smectite is a simple transformation requiring only the loss of interlayer K. The transformation is apparently possible under present-day conditions, inasmuch as the erosional landscape position, shallow depth, and lack of significant horizonation indicate that the soil is very young.

Key Words—Celadonite, Entisol, Mica, Mössbauer spectroscopy, Smectite, Soil, Weathering, X-ray powder diffraction.

INTRODUCTION

Celadonite is a green, dioctahedral mica common in hydrothermally altered or low-grade metamorphic (zeolite facies) volcanic rocks (Wise and Eugster, 1964). In basalts of the Red Rock Canyon area in southern California (Figure 1), celadonite occurs in amygdules and veins and in the matrix of highly altered parts of the flows. Celadonite in the amygdules is dark bluish green, whereas in the veins and highly altered rock matrix, it is lighter green and is intimately mixed with cristobalite, chalcedony, and stilbite.

Although occurrences of celadonite are well documented (Hendricks and Ross, 1941; Wise and Eugster, 1964; Kimbara and Shimoda, 1973; Odom, 1984), this mineral has not been described in a pedologic setting, nor have its weathering products been reported. The objective of the present study was to determine the fate of celadonite in a soil weathering environment on the Red Rock Canyon basalts. Artificial weathering experiments were also used to test the proposed pedogenic transformation mechanism.

MATERIALS AND METHODS

The soil and rock samples were obtained from a hydrothermally altered basalt flow of the Dove Spring Formation (Loomis, 1984) near Red Rock Canyon State Park, immediately north of the northwestern margin of the Mojave Desert, Kern County, California (Figure 1). The occurrence of celadonite in basalt vesicles in the study area was reported previously by Pemberton (1983). The mean annual air temperature at the study site is 17°C and the mean annual precipitation is 150 mm (personal communication, Mark Faull, State Park Ranger, Red Rock Canyon State Park, Cantil, California 93519, December 1986). A loamy, mixed (calcareous) Lithic Torriorthent on a 35% slope nearly barren of vegetation was sampled using standard procedures (Soil Conservation Service, 1984).

Soil samples were air dried and sieved to remove coarse fragments (>2 mm). The particle-size distribution was determined by the pipet method, and calcium carbonate equivalent was determined using a manometric method (Soil Conservation Service, 1984). Soil pH was determined on a saturated paste. Water-soluble cations were measured on saturated paste extracts that had been allowed to stand overnight. Exchangeable cations were extracted with 1 N NH_4OAc at pH 7.0. The cation-exchange capacity (CEC) was measured by saturating samples with 1 N NaOAc at pH 8.2, washing them with 95% ethanol, and treating them with 1 N NH_4OAc to extract exchanged sodium

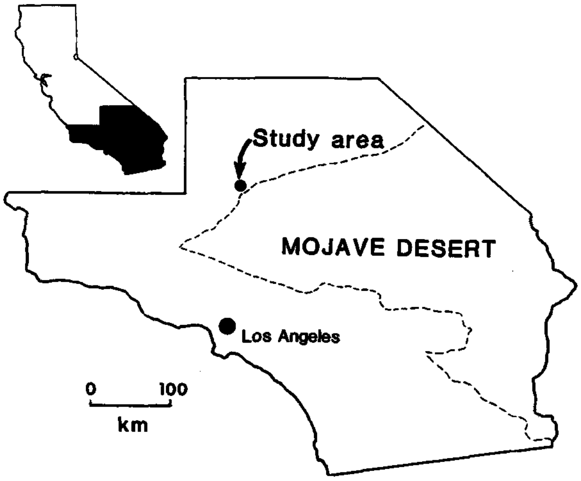

Figure 1. Location of the study area at Red Rock Canyon, Kern County, California.

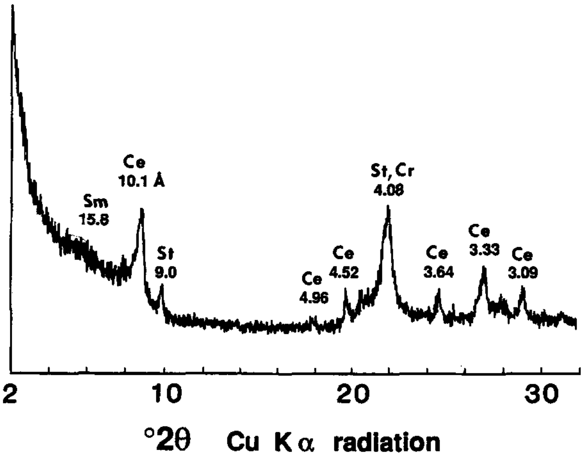

Figure 2. X-ray powder diffractogram of celadonite-containing rock matrix (<5-μm fraction). Celadonite (Ce), cristobalite (Cr), stilbite (St), and a trace of smectite (Sm) are present.

(Soil Conservation Service, 1984). Na, Ca, K, and Mg were determined by atomic absorption spectrophotometry.

To prepare samples for particle-size fractionation, carbonates and soluble salts were extracted using a 1 N NaOAc solution buffered at pH 5.0, and organic matter was removed with 30% H_2O_2 (Kittrick and Hope, 1963). The <2-μm size fraction of the soil and the <5-μm fraction of the celadonite-containing rock matrix were separated by centrifugation and sedimentation techniques (Jackson, 1979). An oriented mount of the <5-μm fraction of the celadonite-containing rock matrix was prepared for X-ray powder diffraction (XRD) by pipeting a slurry onto a glass slide. Pastes of K- and Mg-saturated clays were smeared on glass slides (Theisen and Harward, 1962). All samples were examined by XRD (CuKα radiation) using a Siemens D-500 diffractometer, equipped with a graphite crystal monochromator and a scintillation counter. Specimens in powder form were packed sideways in the shallow trough of an aluminum sample holder for measurement of d(060) values. Selected rock samples were mounted on aluminum stubs with epoxy resin, stored overnight in a dessicator, sputter-coated with Au/Pd, and examined in a JEOL scanning electron microscope.

Freeze-dried samples of the unweathered, <5-μm size fraction of the rock matrix and the soil clay (<2 μm) from the Crk1 horizon were analyzed by Mössbauer spectroscopy. For Mössbauer spectroscopic analysis, absorbers containing about 10 mg Fe/cm (based on total iron analyses) were pressed to uniform thickness in a brass ring with Al foil backing and sealed with a benzene-styrofoam suspension. Mössbauer spectra were obtained at room temperature using a Co-Rh source and an Austin Science Associates microprocessor drive. The velocity was calibrated with a laser interferometer. Spectra were fitted using non-linear least squares to a sum of doublets, after subtraction of the small peak due to Fe in the Al backing. The intensities and widths for the Lorentzian lines of a given doublet for both components were constrained to be equal, but each doublet had variable intensity, width, and, of course, isomer shift (IS) and quadrupole splitting (QS).

The <5-μm fraction of the celadonite-containing rock matrix was used for the artificial weathering studies. Interlayer K in celadonite samples from the basalt was extracted with sodium tetraphenyl boron (NaTPB), using the method described by Scott and Reed (1962). The effectiveness of this procedure for the removal of K from micas is related to the insolubility of the potassium salt of tetraphenyl boron. This technique increased the rate at which K was removed from the mica, inasmuch as K in micaceous minerals was in equilibrium with K in solution, which was maintained at low levels by its precipitation as potassium tetraphenyl boron.

RESULTS

Celadonite in the hydrothermally altered basalt

Celadonite in the hydrothermally altered basalt was identified in the field by its color and mode of occurrence as described by Pemberton (1983), and its presence was confirmed in the laboratory by XRD and Mössbauer spectroscopy. The celadonite in the rock matrix had a d(001) value of 10.1 Å; the d(002) peak was obscured by destructive interference (Figure 2), as is characteristic of high Fe micas, including celadonite (Odom, 1984). The dioctahedral nature of the celadonite was indicated by its d(060) value of 1.510 Å (Figure 3a).

The Mössbauer spectrum of the celadonite-containing rock matrix (<5 μm) sample is shown in Figure 4a

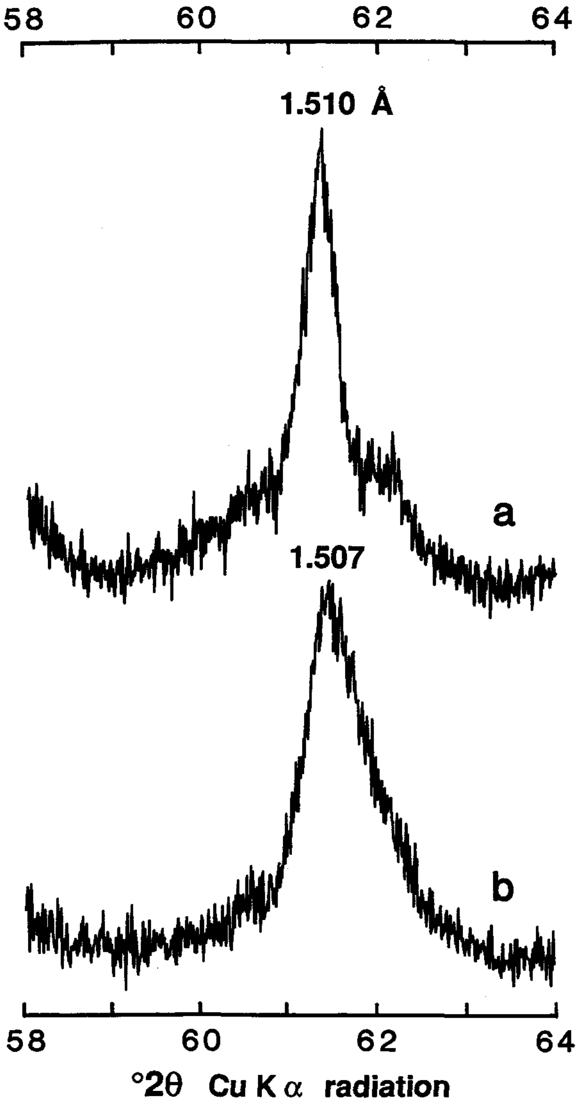

Figure 3. d(060) values (a) from celadonite-containing rock matrix (<5-μm fraction), and (b) from Crk1 horizon soil clay (<2-μm fraction).

and the parameters are listed in Table 1. Three of the four doublets are in good agreement with the values reported by Heller-Kallai and Rozenson (1981) for celadonite. The ideal structure has Fe^{3+} in M2 (cis-OH) sites only. The two Fe^{3+} doublets observed in the present sample had similar parameters to the two Fe^{3+} doublets reported by the above authors (Table 2). These doublets are interpreted as resulting from some occupancy of the trans-OH (M1) sites (QS ~1.9 mm/s) and M2 (QS ~2.6) sites by Fe^{3+}, although the literature is not clear on the QS assignments for Fe^{2+} in micas (Levillain *et al.,* 1982; Heller-Kallai, 1982).

The major Fe^{3+} doublet with QS ~0.4 mm/s is caused by the cis-OH octahedral species, labeled "A" by Daynyak and Drits (1987), and corresponds to the ordered arrangement of M^{2+} nearest neighbors. No unambiguous evidence of the disordered sites reported by these authors with QS ~0.6 mm/s was found, although peaks corresponding to some such sites might have been hidden beneath the other Fe^{3+} peaks. A distinct Fe^{3+} doublet having QS ~1.2 mm/s was, however, noted. This doublet was not reported for the celadonite samples examined by Heller-Kallai and Rozenson (1981), but it was reported for celadonite by Coey (1980) (Table 2). Daynyak and Drits (1987) also reported a doublet having QS ~1.1 mm/s for celadonite samples at ~10% abundance and attributed it to a dehydroxylated site. Indeed, thermal treatment of celadonite produces such a doublet as the major component of the Mössbauer spectrum following dehydroxylation (Heller-Kallai and Rozenson, 1980).

Celadonite in a basalt vesicle (Figure 5) showed the typical sheet-like morphology reported by Beutelspacher and Van der Marel (1968) and Kimbara and Shimoda (1973). The celadonite flakes in the vesicle were similar in size to those in the soil. Celadonite occurred entirely in the fine silt and clay fractions (<5 μm) of the soil.

Soil characteristics

Selected physical and chemical properties of the soil are presented in Tables 3 and 4. Unweathered hard rock (R horizon) was encountered at a depth of 31 cm, and the shallow soil lacked significant horizonation.

Table 1. Mössbauer parameters of celadonite from the rock matrix (RMC) and soil clay from Crk1 horizon.[1]

			Fe^{3+}				Fe^{2+}			
Sample (run)	Fit	χ^2	W (mm/s)	IS (mm/s)	QS (mm/s)	RA (%)	W (mm/s)	IS (mm/s)	QS (mm/s)	RA (%)
Celadonite (rock matrix)	4D	481	0.37	0.36	0.40	64	0.42	1.13	1.74	12
			0.40	0.41	1.16	15	0.35	1.12	2.64	9
Celadonite (soil clay)	4D	430	0.44	0.35	0.41	80	0.44	1.17	1.73	5
			0.39	0.41	1.15	11	0.31	1.11	2.71	4

[1] W = full width at half maximum; IS = isomer shift relative to metallic iron; QS = quadrupole splitting; RA = relative peak area. χ^2 is normalized to number of data points, i.e., 432. Estimated errors are 1–3 in last digit quoted.

Table 2. Mössbauer parameters of celadonite, glauconite, and nontronite reported in the literature.[1]

Sample	Fe^{3+} IS (mm/s)	Fe^{3+} QS (mm/s)	Fe^{3+} RA (%)	Fe^{2+} IS (mm/s)	Fe^{2+} QS (mm/s)	Fe^{2+} RA (%)
Celadonite[2]	0.38 (0.01)	0.35 (0.03)	—	1.11 (0.02) 1.13 (0.02)	1.88 (0.08) 2.64 (0.06)	—
Glauconite, celadonite[3]	0.36 (0.03) 0.35 (0.03)	0.4 (0.1) 1.0 (0.2)	70 (20) 20 (30)	1.15 (0.10)	1.7–2.7	10 (10)
Nontronite[2]	0.35 0.35	0.28 0.70	— —			
Nontronite[3]	0.36 (0.02) 0.36 (0.02) 0.16 (0.02)	0.30 (0.06) 0.62 (0.06) 0.54 (0.07)	55 (15) 25 (10) 20 (15)			

[1] IS = isomer shift relative to metallic iron; RA = relative peak area. The numbers in ( ) give the approximate range observed for multiple samples.

[2] Heller-Kallai and Rozenson (1981).

[3] Coey (1980).

The distinctive green color of the soil was similar to that of the underlying rock. The Munsell designations for the Ck and Crk1 horizons were not found in the standard Munsell soil color charts, but were obtained from the Munsell Book of Color (Munsell Color Company, 1970). The A horizon is not as green as subsurface horizons, because it contains wind-blown sand from neighboring tuffaceous cliffs. These wind-blown sands are also responsible for the higher sand content in the A horizon relative to the underlying horizons.

The soil pH of the A horizon was 7.58 and increased with depth to 8.33 in the Crk1 horizon. The calcium carbonate equivalent increased from 0.9% in the A horizon to 5.0% in the Crk1 horizon. No discrete carbonate nodules or concretions were observed. Calcium was the dominant exchangeable cation in all horizons, although some of this Ca probably originated from the dissolution of $CaCO_3$. Exchangeable Na increased 18-fold from the A horizon to the Crk1 horizon. Sodium was the dominant water-soluble cation in all horizons, and its concentration increased with depth. Exchangeable K also increased with depth, but not to the level reached by Na. Water-soluble K was low and exchangeable and water-soluble Mg were less abundant than other cations in all horizons.

The CEC of the soil (<2 μm fraction) increased with depth; however, not all of the CEC could be attributed

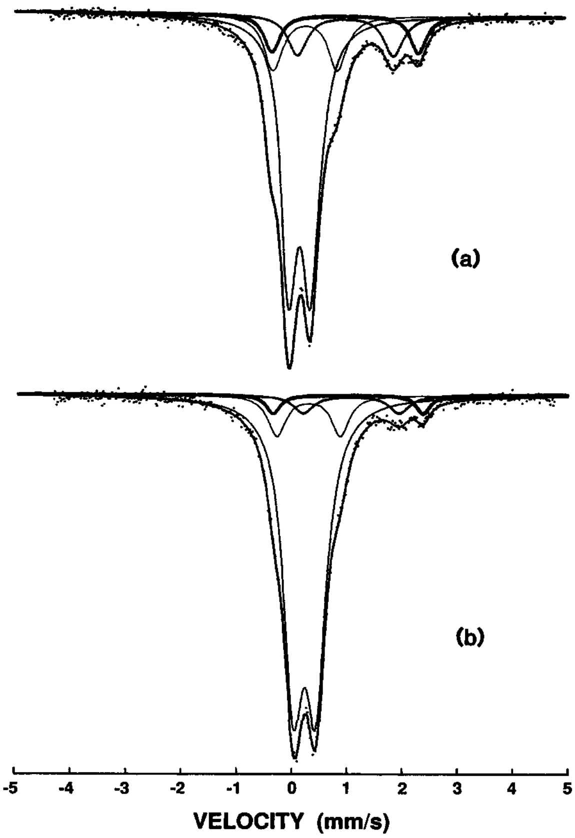

Figure 4. Mössbauer spectra (a) from celadonite-containing rock matrix (<5-μm fraction), and (b) from Crk1 horizon soil clay (<2-μm fraction).

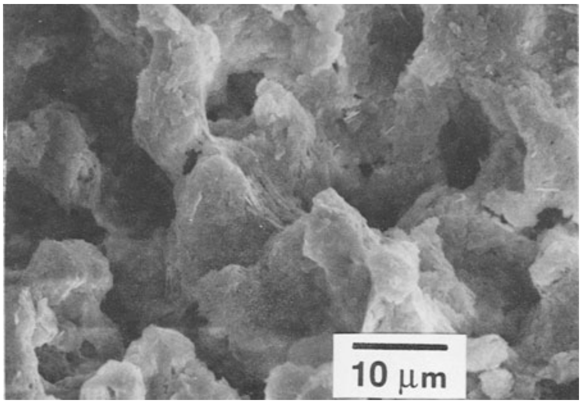

Figure 5. Scanning electron micrograph of celadonite in basalt vesicle.

Table 3. Selected physical characteristics of soil from Red Rock Canyon, Kern County, California.

Horizon	Depth (cm)	Moist color	Sand (2–0.05 mm) (%)	Silt (0.05–0.002 mm) (%)	Clay (<0.002 mm) (%)
A	0–2	2.5Y 4/2	75.7	15.5	8.8
Ck	2–9	10Y 4/2	52.7	27.4	19.9
Crk1	9–21	5GY 4/2	46.4	25.9	27.7
Crk2	21–31	Highly fractured, weathered rock			
R	>31	Green, hydrothermally altered rock			

to the clay fraction, because smectite was also identified in the fine silt (2–5 μm) fraction of all horizons.

Celadonite and smectite in the soil

XRD showed that celadonite and Fe-rich smectite comprised the entire 5-0.2-μm fraction of the soil in all horizons. Few particles <0.2-μm in size were found. Celadonite in the <2-μm fraction had a d(001) value of 10.0 Å (Figure 6). The d(001) value of Mg-saturated smectite was 14.3 Å, and the d(001) value of glycolated smectite was 17.3 Å. Air-dried, K-saturated samples showed a partial collapse of the smectite, producing a broad peak at 11 Å. Heating to 350°C caused further collapse to about 10.7 Å, and the sample collapsed completely to 10.1 Å on heating to 550°C. The soil clay showed a single d(060) value of 1.507 Å (Figure 3b), indicating that both its components, celadonite and smectite, were dioctahedral and had nearly identical *b*-dimensions.

The Mössbauer spectra show that the chemical environments of Fe in rock matrix celadonite (Figure 4a) and in the soil clay (Figure 4b) were very similar. The only obvious difference in the soil is that the proportion of iron in the Fe^{3+} oxidation state was greater (about equally for the M1 and M2 sites, Table 1). This oxidation is important, because it indicates a reduction in structural charge. The doublet having QS ~0.4 mm/s (Figure 4b, Table 1) could well include some contribution from more than one type of Fe^{3+} site, as are present in Fe-rich smectites, such as nontronite (Table 2), because the XRD data indicate that Fe-rich smectite was present in the soil clay (Figure 6). No evidence of tetrahedral Fe (the doublet with IS ~0.16 mm/s in Table 2), however, was found, and the oxidation does not seem to have affected the dehydroxylated sites (QS ~1.2 mm/s, Table 1).

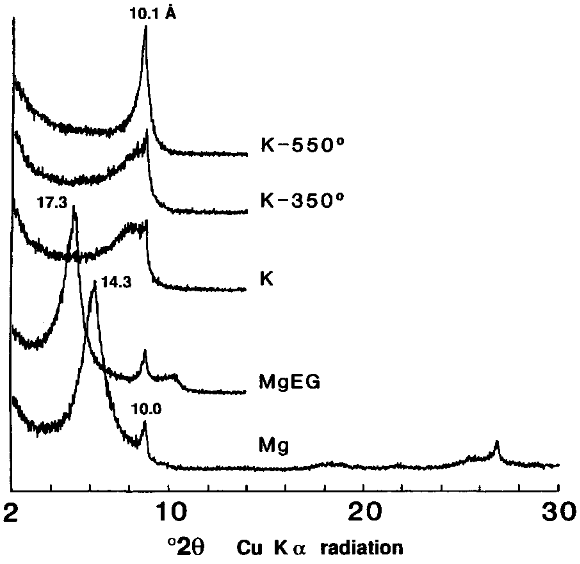

Figure 6. X-ray powder diffractograms of the Crk1 horizon clay (<2-μm fraction) after various treatments. Mg = Mg-saturated; MgEG = Mg-saturated ethylene glycolated; K = K-saturated; K-350° = K-saturated, heated to 350°C for 2 hr; K-550° = K-saturated, heated to 550°C for 2 hr.

Artificial weathering of celadonite

Artificial weathering of celadonite using NaTPB to extract interlayer K greatly reduced the intensity of the 001 XRD peak, and a peak at 14.4 Å, absent prior to weathering, became apparent (Figure 7). Upon glycolation of the sample, this peak expanded to 17.4 Å, similar to the behavior of the soil smectite (Figure 6).

DISCUSSION

The parent hydrothermally altered basalt contained celadonite, but very little smectite, whereas the overlying soil was found to contain both dioctahedral Fe-smectite and celadonite as the sole components of its clay fraction. The d(060) values and Mössbauer parameters of the smectite and celadonite in the soil clay are indistinguishable and are nearly identical to those of the celadonite in the parent rock. These observations strongly suggest a simple transformation of celadonite to smectite during soil formation.

Weathering of micas in soils is typically initiated by a loss of interlayer K (Scott and Reed, 1966). Artificial weathering experiments have provided most of the in-

Table 4. Selected chemical characteristics of soil from Red Rock Canyon, Kern County, California.

Horizon	Depth (cm)	pH	$CaCO_3$ eq. (%)	CEC (meq/100 g clay)	Exchangeable cations (meq/100 g)				Water-soluble cations (mmole/liter)			
					K	Na	Ca	Mg	K	Na	Ca	Mg
A	0–2	7.58	0.9	16.9	1.4	0.6	18.0	1.0	0.4	2.8	2.7	0.2
Ck	2–9	8.02	1.6	33.7	3.4	4.4	32.8	1.1	0.3	5.3	0.8	0.04
Crk1	9–21	8.33	5.0	41.0	4.0	11.3	33.8	0.7	0.2	9.2	0.3	0.04

formation concerning the natural processes by which K is removed and the associated structural changes in the mica during chemical weathering (Scott *et al.*, 1960; Scott and Reed, 1962, 1966; Scott and Smith, 1966). The transformation of micas to vermiculite or smectite-like minerals has been accomplished in the laboratory by simply extracting interlayer K (Scott *et al.*, 1960; Scott and Reed, 1962; Scott and Smith, 1966; Robert, 1973; Abudelgawad *et al.*, 1975). The degree to which dioctahedral micas expand upon interlayer K depletion depends primarily on their structural charge, inasmuch as the strength with which K and other ions are bonded to 2:1 layers increases with increasing structural charge (Scott and Smith, 1966). In addition, structural charge originating in the tetrahedral sheet is more effective for holding K than charge originating in the octahedral sheet (Scott and Smith, 1966). Robert (1973) reported that low levels of tetrahedral Al in micas, resulting in lower structural charge, facilitates direct transformation to smectite without an intermediate vermiculite state. Apparently, on interlayer K depletion, micas having low structural charge that originates chiefly in the octahedral sheet expand more than micas with structural charge originating in the tetrahedral sheet.

In the present study, the artificial removal of interlayer K from celadonite resulted in a transformation product having an expansion behavior similar to that of smectite and similar to that of the soil smectite. Robert (1973) reported that glauconite (an Fe-rich dioctahedral mica similar in composition to celadonite) from Cormes, France, also expanded to 18 Å following K-depletion, Mg-saturation, and glycolation. A similarly treated glauconite from Villers, France, however, expanded only to 14 Å and required oxidation of octahedral Fe^{2+} before it exhibited a smectite-like swelling behavior (Robert, 1973).

Direct transformation of celadonite to smectite without an intermediate vermiculite state appears to be favored by the structural characteristics of celadonite. Tetrahedral substitution of Al for Si is minimal in celadonite, ranging from 0.0 to 0.2 atoms per formula unit (Bailey *et al.*, 1979). Thus, the structural charge originates largely in the octahedral sheet, and the interlayer K is not strongly held.

The small particle size of celadonite (generally <5 μm) may also have facilitated its weathering in the soil, inasmuch as several artificial weathering experiments have shown that small mica particles release their K more rapidly than large particles (Fanning and Keramidas, 1977). Scott (1968) reported that the extraction of most of the K from 0.2–0.7-μm-size muscovite particles required 10 days, whereas 1000 days was required for the extraction of K from 50–60-μm-size muscovite particles.

The transformation of celadonite to smectite required only the loss of interlayer K and is apparently possible under present-day conditions, inasmuch as the erosional landscape position, shallow depth, and lack of significant horizonation indicate that this is a very young soil. Ca and/or Na, both abundant on the exchange sites, may be the cations displacing interlayer K in the soil.

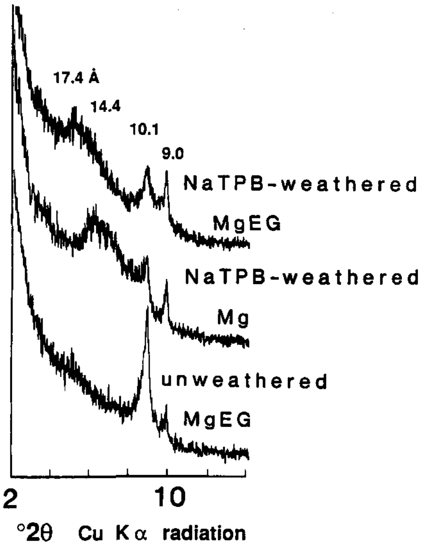

Figure 7. X-ray powder diffractograms of unweathered and NaTPB-weathered celadonite-containing rock matrix (<5-μm fraction). Samples were Mg-saturated (Mg) or Mg-saturated and ethylene glycolated (MgEG).

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