CHARACTERISTICS OF INTERLAYER MATERIALS IN NON-ALLOPHANIC VOLCANIC ASH AND RED YELLOW SOILS FROM JAPAN^{1/}

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ABSTRACT

The characteristics and properties of interlayer materials from 10 non-allophanic (Melanudand) and red yellow soils (Alfisols and Ultisols) from three locations in Japan were studied by chemical extractions and x-ray diffraction (XRD) analyses. Allophane was dissolved from clay samples using 0.15 M acid oxalate solution while silicon and aluminum were dissolved using both 0.15 M acid oxalate and 0.3 M sodium citrate solutions. XRD analyses were done for the treated and untreated samples. For the chemically extracted samples, the clay samples were treated with bicarbonate citrate solution for the removal of iron oxides after the dissolution treatments then saturated with K and Mg ions for XRD analyses. Results of the XRD patterns of the untreated and treated samples indicated the interlayer materials as a hydroxy-interlayered form of vermiculite (HIV). Between the two dissolution treatments, the 0.3 M sodium citrate treatment was found to be more effective in removing the interlayer materials than the 0.15 M acid oxalate treatment by allowing interlayer expansion and collapse.

KEY WORDS: Hydroxy - interlayered minerals. Japanese Red-Yellow. Non-allophanic soils. Chemical dissolution treatments. XRD analyses.

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INTRODUCTION

Interlayer materials play significant roles in modifying the chemical and physical properties of minerals, in particular vermiculite and smectite. Al and hydroxy-Al play an important role in modifying the surface and mineralogical properties and cation exchange capacity of clays. Modifications of these characteristics depend upon the degree to which filling of the interlayer space with hydroxy Al occurs and the relative stability of the interlayer components (Barnhisel and Bertsch, 1989). The equilibrium between Al in solution and hydroxy Al interlayered clays may influence a wide range of chemical reactions in soils, including adsorption of phosphate, formation of Al-phosphate compounds, and the formation of separate stable mineral phases such as gibbsite. They also affect the availability of phosphates in soils especially those added through fertilization.

Hydroxy-interlayered 2:1 clay minerals HIV (vermiculites) and HIS (smectites) can be considered to form a solid solution series, with "pure end – members" consisting of vermiculite or smectite at one extreme and pedogenic or aluminous chlorite at the other (Barnhisel and Bertsch, 1989). There exists various possibilities in between ranging from sparse, weakly interlayered to extensive, well-developed interlayered minerals that are arbitrarily distinguished from aluminous chlorite by their inability to maintain their 1.4 nm spacing after 550°C heat treatment (Matsue and Wada, 1988). Because these characteristics can differ widely, there is a tremendous range in the physicochemical properties of soils dominated with HIV and HIS, and this makes these mineral phases among the most intriguing for soil mineralogical investigations.

In their work with Korean Ultisols and Alfisols, Wada and Kakuto (1983 a,b) concluded that the interlayer materials present in these soils extracted using sodium citrate has a composition and structure of a 1:1 layer mineral, and that the non-exchangeable K is also present in its interlayer space together with exchangeable cations (Wada et al., 1991). Matsue and Wada (1988), on the other hand, reported that in

Dystrandepts derived from tertiary sediments, materials dissolved by citrate treatment had a Si/Al ratio ranging from 0.72 to 0.24, suggesting the formation of a Hya sheet partially bonded with Si-tetrahedra as a possible model of the interlayer structure of PIV. However, it is estimated that some interlayer materials, especially those with low Si/Al ratios, are composed of hydroxy aluminosilicates (HAS) ions and their polymers (Inoue and Satoh, 1992).

This research was conducted to study the characteristics and properties of interlayer materials from non-allophanic and red yellow soils taken from different locations in Japan. Of special interest are those soils affected by eolian dust deposition in their pedogenic development.

METHODOLOGY

Ten selected soil samples from the A and B horizons of non-allophanic and red yellow soils from different locations in Japan were used (Table 1). Only soils that showed distinct properties of non-allophanic Andisols, Alfisols and Ultisols were chosen for this study. These soil samples were air dried and fractionated into fine earth using a 2 mm sieve. The clay fraction ($< 2 \mu\text{m}$) was separated from the fine earth by washing with 1 N NaCl, water, methanol and acetone through a series of dispersion, ultrasonification and sedimentation procedure. The particle size distribution and chemical analyses of some of these samples are presented in Table 2. For particle-size analyses, the soil samples were first treated with 6% H_2O_2 to remove organic matter and then dispersed in deionized water by sonification at 20 kHz (150w, pH 10) for 10 min. The clay (less than $2 \mu\text{m}$) and silt (2 to $20 \mu\text{m}$) fractions were obtained by sedimentation and the fine (20 to $200 \mu\text{m}$) and coarse (200 to $2000 \mu\text{m}$) sand fractions were separated by wet sieving. The pH was measured using a 1:2.5 soil:water/KCl ratio after 1 h reciprocal shaking (ISRIC, 1987). Organic carbon (C) was measured using the Walkley-Black procedure (Walkley and Black, 1934). Cation exchange capacity (CEC) and exchangeable bases using 1 N buffered ammonium acetate at pH 7.0 (Chapman, 1965).

Removal of Free Iron Oxides from Clay

Free iron oxides were removed from clay following the procedure of Mehra and Jackson (1960) using 0.3 M Na citrate solution and 0.5 ml of 1 M NaHCO₃ solution added to 100 mg air dried clay samples in a 10 ml glass centrifuge tube.

Preparation of Mg- and K- Saturated Clay Specimens

The clay samples were saturated with K and Mg by treating them with 1 N KCl and MgCl₂ solution, respectively. Mg-saturated clay samples were solvated with glycerol (Mg-gly) and 20°C (Mg-20) while the K-saturated clay specimens were subjected to 20 (K-20) 110 (K-110), 300° (K-300) and 550°C (K-500) heat treatments. X-ray diffraction (XRD) patterns of these mounted clay specimens were determined using the Rigaku Geigerflex X-ray machine.

Table 1. Brief description and location of the soil samples used in the study.

Sample Code	Horizon	Depth (cm)	Soil Color	Location	Soil Order
Aki-1	A	0-34	5 YR 3/2	Yamaguchi	Typic Hapludult
	B ₁	34-70	5 YR 3/4	"	Typic Hapludult
	B ₂	70-110+	10 R 4/8	"	Typic Hapludult
Aki-2	A	0-14	5 YR 3/4	"	Typic Hapludult
	B ₁	14-40	5 YR 3/4	"	Typic Hapludult
	B ₂	49-100+	10 R 4/6	"	Typic Hapludult
M-2	B1	25-50	7.5 YR 5/8	Okinawa	Typic Hapludult
I-2-3	IIB21	17-38	6.25 YR 5/6	Iwate	Alic Melanudand
Z-5	BC	65-100	7.5 YR 5/8	Okinawa	Typic Hapludult
I-3-3	IIA1	23-35	10 YR 1.7/1	Iwate	Alic Melanudand

Table 2. Physical and chemical properties of some soil samples used in the study.

Sample Code	Particle size distribution analysis (%)				
	C. sand	F. sand	Silt	Clay	Textures
Aki-1 A	4.6	7.8	51.5	36.1	SiC
Aki-1 B ₁	5.5	7.6	52.1	34.8	SiC
Aki-1 B ₂	2.5	6.1	32.8	58.6	HC
Aki-2 A	3.4	5.5	56.6	34.5	SiC
Aki-B ₁	2.5	6.2	48.4	42.9	SiC
Aki-2B ₂	0.4	2	12.3	85.3	HC
I-2-3	0.3	6.1	31.1	62.5	HC
I-3-3	0.7	22.9	31.5	44.9	LiC

Sample Code	Chemical Analysis							
	pH		Org. C	CEC	Exch.	Bases	(me/100g)	BS
	H2O	KCl	(%)	(me/100g)	Ca.	Mg	K Na	(%)
Aki-1A	5.0	4.4	1.60	7.4	1	0.6	0.2	26
Aki-1 B ₁	5.5	4.4	0.63	9.5	0.5	0.2	0.1	9.0
Aki-1 B ₂	5.4	4.4	0.33	4.5	1.5	0.1	0.2	48
Aki-2 A	6.7	5.7	5.23	20.7	15.9	1.4	0.1	86
Aki-B ₁	5.5	4.3	0.70	10.9	2.9	0.5	0.2	34
Aki-2B ₂	5.9	4.4	0.54	28.4	12.6	1.8	0.5	53
I-2-3	5.2	4.0	0.81	18.4	0.5	0.9	0.8	14
I-3-3	5.2	4.2	14.11	46.8	0.04	0.1	0.4	2.0

Dissolution of Allophane, Si and Al by Oxalate Solution

Allophane, Si and Al were dissolved from clay samples using 0.15 M acid oxalate solution (Higashi and Ikeda, 1974). A hundred mg clay sample was placed in 500 ml polyethylene bottle, to which 200 ml of acid oxalate solution was added. The bottle was covered and shaken for 4 or

24 hours in a horizontal reciprocating shaker in the dark at 30°C. The content was then centrifuged for 10 min at 10,000 rpm and the clear supernatant was decanted in polyethylene bottles. The smaller clay particles in the supernatant were further filtered by ultrafiltration using 0.2 mm membrane filter.

Silicon was determined colorimetrically following the modified blue silicomolybdous acid procedure by Weaver et al. (1968). Aluminum was determined using the procedure of Davenport (1949), after the destruction of oxalate following the method of Higashi and Ikeda (1974).

Dissolution of Si and Al by Citrate Treatment

Aside from the acid oxalate solution, Si and Al were dissolved from clay minerals following the procedure of Tamura (1958). One hundred mg of clay samples were treated with 100 ml of 0.3 M sodium citrate at 100°C for 4 or 24 hours in a tetrafluoroethylene-hexafluoro-propylene (Teflon) flask with a reflux condenser. The amount of Si dissolved was determined by the modified blue silicomolybdous acid procedure of Weaver et al. (1968) as in the acid oxalate treatment. The amount of Al was also analyzed colorimetrically using the method of Davenport (1949).

X-ray Diffraction of Treated Samples

After the dissolution treatments with hot 0.3 M sodium citrate and 0.15 M acid oxalate, clay samples remaining in the centrifuge tubes after collecting the supernatant solutions were set aside for XRD analysis. The clay samples were first treated with bicarbonate citrate solution for the removal of iron oxides (Mehra and Jackson, 1960) then saturated with K and Mg ions by following the procedure for the preparation of Mg- and K-saturated specimens as previously presented.

RESULTS AND DISCUSSION

XRD Patterns of Untreated Samples

Figure 1 shows the XRD patterns of representative clay samples exhibiting the presence of interlayer materials. A small 1.0 nm peak in the Mg-saturated patterns of some soil samples indicate that these samples contain some mica. Distinct appearance of 1.4 nm peak in both the unheated K (K-20) – and Mg (Mg-20) – saturated samples indicate a hydroxy interlayer form of vermiculite, or smectite or chlorite. As the K-saturated samples was heated to 110 and then to 300°C, the 1.4 nm peak shifted toward 1.0 nm, indicating a hydroxy interlayer form of vermiculite or smectite. The 0.71 nm peak shows the presence of kaolinite which was further confirmed by the disappearance of the 0.71 nm peak when the samples were heated to 550°C because it would make the kaolinite amorphous to X-ray diffraction at 550°C and the peaks would disappear at this temperature. The presence of vermiculite in the clay samples was detected by the 1.4 nm peak of Mg-saturated samples which did not shift to 1.77 nm upon solvation with glycerol. The presence of chlorites was indicated by the 1.4 nm peak of K-saturated samples which remained present although at lower intensity, even after heating the samples to 550°C. The hump or shoulder on the low angle side of the 1.0 nm reflection may be from the hydroxy interlayered form of vermiculite. In addition to interlayer minerals, the clay samples also contained vermiculite, chlorite and kaolinite as indicated by XRD patterns.

The results of the XRD analyses implied that the dominant interlayer material found in these soils is a hydroxy-interlayered vermiculite or smectite. These findings are similar to the results obtained by Inoue and Naruse (1987) for some eolian dust derived soils taken from different locations in Japan.

Figure 1. X-ray diffractograms of untreated soil clays representing three soil orders (Hapludults, A-12 and Z-5; Hapludalf, A-22; Melanudand, I-3-3).

XRD Patterns of Oxalate Treated Samples

Figure 2 shows the XRD patterns of oxalate treated clay samples dissolved with 0.15 M acid oxalate solution in the dark for 4 or 24 hrs. periods. We could see a similar pattern as in the untreated samples wherein no shift in the Mg-saturated clay was exhibited even after solvating with glycerol. For the K treated samples, the 1.4 nm peak did not shift to 1.0 nm with air drying but was gradually shifted towards 1.0 nm with increasing heat treatment indicating only minimal and incomplete interlayer collapse. This suggests that oxalate could not effectively allow interlayer expansion and collapse even after the removal of aluminosilicate ions from the interlayer spaces. This was probably due to the inability of the oxalate reagent to dissolve large amounts of Si and Al ions as compared to the citrate treatment even after the removal of amorphous aluminosilicates (Inoue and Satoh, 1992).

XRD Pattern of Citrate Treated Samples

Unlike in the previous figures for the untreated and oxalate treated samples, the citrate treated samples show interlayer expansion and collapse after extraction of Al and Si (Figure 3). This was clearly shown by the XRD patterns of Mg-saturated I-3-3 sample, in which the 1.77nm peak indicating the presence of smectite became apparent after glycerol solvation. Interlayer collapse was evident even in low temperatures of K-saturated samples, wherein at air dried K samples, the 1.4 nm completely disappeared and shifted to 1.0 nm in most of the clay samples. From the XRD patterns, a sharp shift of the 1.4 nm peak to 1.0 nm was detected and the virtual absence of a shoulder in some patterns suggests the destruction of the hydroxy-interlayer materials in the clay. The sharp

reflection produced at 1.0 nm, indicates that HAS ions adsorbed on the interlayer spaces of Vt were completely dissolved by the citrate treatment (Inoue and Satoh, 1992). However, the Mg saturated (air dry, 20°C) and glycerol solvated samples did not shift to 1.8 nm, indicating that the mineral is an HIV (hydroxy interlayered vermiculite). A similar result was reported by Matsue and Wada (1988) in Dystrandepit derived from tertiary sediment. These results suggest that citrate treatment could dissolve the interlayer materials from smectite and vermiculite thereby allowing interlayer expansion and collapse. The efficiency of the citrate treatment as compared to the oxalate treatment in removing hydroxy-interlayer materials was indicated by the large quantities of Si and Al dissolved by citrate (Tables 3 and 4).

Table 3. Dissolution of Si and Al from clay samples by oxalate treatment.

Sample Code	Amounts of Si and Al (g/kg) dissolved					
	4 hrs. treatment			24 hrs. treatment		
	Si	Al	Si/Al ratio	Si	Al	Si/Al ratio
Aki-1A	0.6	1.6	0.38	1.5	5.1	0.29
Aki-1 B ₁	0.7	1.8	0.39	1.8	5.2	0.35
Aki-1 B ₂	0.7	6.8	0.10	1.5	7.6	0.20
Aki-2	0.8	6.2	0.13	1.7	6.8	0.25
Aki-2B ₁	0.6	2.5	0.24	1.6	6.2	0.28
Aki-2B ₂	0.7	5.1	0.14	1.4	5.2	0.27
M2	0.6	10.5	0.06	1.3	1.5	0.87
I-2-3	1.2	5.2	0.23	2.2	1.1	5.8
Z-5	0.11	5.8	0.02	1.1	5.8	0.19
I-3-3	0.6	18	0.03	6.6	24.1	0.27

Figure 2. X-ray diffractograms of oxalate treated soil clays representing three soil orders (Hapludults, A-12 and Z-5; Hapludalf, A-22; Melanudand, I-3-3).

Figure 3. X-ray diffractograms of citrate treated soil clays representing three soil orders (Hapludults, A-12 and Z-5; Hapludalf, A-22; Melanudand, I-3-3).

Table 4. Dissolution of Si and Al from clay samples by citrate treatment.

Sample Code	Amounts of Si and Al (g/kg) dissolved					
	4 hrs. treatment			24 hrs. treatment		
	Si	Al	Si/Al ratio	Si	Al	Si/Al ratio
Aki-1A	17.5	30.2	0.55	22.4	28.6	0.78
Aki-1 B ₁	11.8	36.20	0.32	21.3	48.9	0.44
Aki-1 B ₂	18.5	25.6	0.69	23.8	52.00	0.46
Aki-2 A	24.3	25.6	0.92	22.9	38.5	0.59
Aki-2B ₁	22.7	36.5	0.60	18.7	37.5	0.50
Aki-2B ₂	17.5	29.7	0.57	22.7	34.4	0.66
M-2	15.00	30.9	0.47	16.5	47.9	0.34
I-2-3	19.7	39.6	0.46	23.2	45.7	0.51
Z-5	15.5	20.10	0.75	18.8	36.10	0.52
I-3-3	22.7	39.6	0.55	27.2	36.10	0.75

Dissolved Si and Al and Si/Al Ratios

To further determine the nature of interlayer materials in the clay samples, dissolution of Si and Al was conducted by acid oxalate and citrate treatments. Table 3 shows the amounts of Si and Al dissolved by the acid oxalate treatments at 4 and 24 hours extraction periods. These 2 extraction periods were used to ascertain their effects on the amount of Si and Al dissolved. From the results, it was apparent that more Al and Si were dissolved at 24 h than at 4 hr extraction period. The results show that in comparing the 2 dissolution treatments, more Si and Al were extracted by citrate treatments (Table 4) as compared to oxalate treatments (Table 3). In majority of the samples, more Al were extracted as compared to Si regardless of the dissolution treatment used. This is very evident in Red-Yellow soils (Aki-1 and 2 samples and M-2 and Z-5) which usually contain higher amounts of oxides and hydrous oxides of Fe

and Al. On the other hand, higher Si and Al were extracted in non-allophanic Andisols (I-2-3 and I-3-3) compared to Red-Yellow soils in the oxalate treatments. This could be due to the weathering of volcanic ash soils during pedogenic development which releases hydroxyaluminosilicate ions. Chloritized vermiculite (Vt) could be pedogenically formed by interaction of expansible 2:1 layer silicates weathered from micas with HyA or HAS ions, which are transformed from volcanic ash during weathering process (Inoue and Satoh, 1992).

The Si/Al molar ratios of both the citrate and oxalate treated samples were likewise low with average values of 0.28 and 0.56 for oxalate and citrate samples, respectively. The low Si/Al ratios indicate that the interlayer materials are composed of hydroxy-interlayer materials and their polymers (Inoue and Satoh, 1992 and 1993).

These results also support the suggestion of Inoue and Satoh (1992) which stated that development of chloritized-Vt in the non-allophanic Andisols could be formed by the interaction of Vt, transformed from muscovite and transported as eolian dust from China, with HAS and/or HyA ions formed in soil solutions during weathering and pedogenic alteration of volcanic ash. Bautista and Inoue (1993) also reported that loess or eolian dust deposited on the surface of volcanic ash soils influence the Andic properties of non-allophanic Andisols.

The findings of this study are further supported by the higher amounts of Al dissolved at lower Si/Al ratios in the citrate treated samples (Figure 4). Higher amounts of Al were extracted by citrate at approximately 0.3-0.6 Si/Al ratio. On the other hand, the amounts of Al extracted increases with Si/Al ratio with most of the Si extracted at 0.4-0.6 Si/Al ratios. Higher amounts of Al were extracted in Andisols as compared to Ultisols and Alfisols.

Amount of Si and Al Dissolved and Extent of Interlayering

The extent of interlayering was estimated by following the XRD intensity ratio as expressed by:

$$\frac{(I_{14}/(I_{14} + I_{10}))^K}{(I_{14}/I_{14} + I_{10}))^{Mg}}$$

where K and Mg denote K⁺ and Mg²⁺ saturate and air dried samples, respectively (Matsue and Wada, 1988). This ratio was used to determine the extent of interlayering of hydroxy-interlayered vermiculite which is dominant in our samples, since the contribution of chlorite and smectite is minimal as indicated by the XRD patterns. The amount of Al and Si dissolved increased with interlayering and higher amounts of Al were dissolved as compared to Si (Fig.5). This indicates that dissolution of Al is always accompanied by Si. On the other hand, the Si/Al ratio decreased from approximately 0.75 to 0.30 with increasing interlayering.

These results signify the occurrence of hydroxy-Al and/or-Fe interlayering in 2:1 layer silicate minerals present in Alfisols, Ultisols and nonallophanic Andisols in Japan.

Amount of Al Dissolved and Interlayer Collapse

The failure of the interlayer to collapse was measured by comparing the ratio $I_{14}/(I_{14} + I_{10})$ for K – saturated (K) and air – dried samples, where I_{14} and I_{10} stand for the XRD intensities at $d=1.4$ and 1.0 nm, respectively (Wada and Matsue, 1988). High I_K ratios show very low degree of interlayer collapse and lower I_K ratios show high degree of interlayer collapse. In comparing the values obtained in the three soil orders, higher values were obtained in non-allophanic Andisols while lower values were obtained in Ultisols (Fig. 6).

In establishing the relationship between the amount of Al dissolved and interlayer collapse, the results show that the dissolution of Al both by citrate and oxalate treatments is directly related to interlayer collapse. This suggests that interlayer collapse was accompanied by the removal of hydroxyaluminosilicate and hydroxy-Al materials in the interlayer spaces of 2:1 silicate minerals.

Implications to Soil Development and Soil Properties

The presence of hydroxyinterlayered vermiculites (HIV) in Japanese soils derived from eolian dust will probably affect soil development and the various soil chemical and mineralogical properties. This will lead to the modifications of various reactions that will take place in the soil. Inoue and Higashi (1988) and Inoue (1990) reported that the active Al components in chloritized vermiculite can contribute to the accumulation and stabilization of humus and to the retention of phosphate by acidic and nonallopahnic Andisols. Inoue and Satoh (1992,1993) reported that the fixation of hydroxyaluminosilicate (HAS) and hydroxy-Al ions in the interlayer spaces of vermiculite and montmorillonite greatly changed the electrical charge and the surface properties of soils containing them. Aside from this, the cation exchange capacity of the soil was also diminished by hydroxyaluminosilicate ions and their polymers, which will greatly affect the chemistry and fertility of the soils. Sakurai and Huang (1998) reported significant reduction in the CEC of montmorillonite after HAS ion adsorption. They added that from the pedological point of view, the intercalation of HAS ions within permanent-charge minerals can be considered as a weathering process in subtropical and temperate regions. This weathering process would lead to the alterations of the surface chemistry of soil colloids pertaining to transformation and transport of nutrients and pollutants in the environment.

Figure 4. Relationships between the amounts of Si and Al dissolved and 4 h citrate (A) and 24 h citrate after 4 h oxalate (B) treatments and the Si/Al atomic ratios of Alfisols, Ultisols and Andisols.

Figure 5. Relationship between the amounts of Si and Al dissolved by 24 h citrate treatment after 4 h oxalate and Si/Al atomic ratios with the extent of interlayering in Alfisols, Ultisols and Andisols.

Figure 6. Relationship between the amount of Al dissolved by 24 h citrate treatment after 4 h oxalate and interlayer collapse in Alfisols, Ultisols and Andisols.

CONCLUSION

This study provides evidence for the existence of hydroxyinterlayer minerals in non-allophanic Andisols and Red-Yellow Alfisols and Ultisols from Japan. The presence of hydroxy interlayer minerals was confirmed by selective dissolution treatments using 0.15 M acid oxalate and 0.30 M sodium citrate solutions and XRD analyses. Results of XRD analyses showed that the dominant hydroxyinterlayer minerals present in these soils consist mainly of hydroxyinterlayer vermiculite (HIV), smectite and chlorite. Selective dissolution analyses indicated that the 0.3 M sodium citrate treatment was more effective in removing the interlayer materials as compared to the 0.15 M acid oxalate treatment.

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