

EFFECT OF CHEMICAL PRETREATMENT ON X-RAY POWDER DIFFRACTION CHARACTERISTICS OF CLAY MINERALS DERIVED FROM VOLCANIC ASH

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Abstract—The effects of NaOAc removal of carbonates, H₂O₂ removal of organic materials, and sodium citrate-bicarbonate-dithionite (SCBD) removal of iron oxides upon the X-ray powder diffraction (XRD) characteristics of clay minerals present within weathered volcanic ash deposits were examined. All pretreatments of samples derived from the Ae and Bhf horizons containing volcanic ash resulted in dramatic increases in XRD peak intensities, while the treatment of IIC-horizon samples resulted in subdued XRD peak intensities. Also, the chemical treatments produced an apparent increase in the amount of expandable clay minerals within the Ae horizon, due probably to the hydrolysis and removal of interlayer Al³⁺ when H₂O₂ and SCBD were used. Hence, poorly ordered and pedogenically modified vermiculite or vermiculite/montmorillonite interstratifications became “weathered” by H⁺ attack and Al³⁺ removal in a manner similar to the natural processes which take place in the acid and eluvial A horizon. In addition, possible XRD peak modification due to the dilution of crystalline clay concentrations by amorphous clay-size material appeared to be subordinate to the laboratory-induced chemical alteration of the weathered clay minerals.

Key Words—Amorphous, Chemical treatment, Soil clay, Vermiculite, Volcanic ash, Weathering, X-ray powder diffraction.

INTRODUCTION

Procedures commonly used for X-ray powder diffraction (XRD) identification of clay minerals include some form of chemical pretreatment of clays or clay slurries. The rationale for chemically treating soil clay minerals is to obtain “clean-sharp” XRD peaks which are unobscured by extraneous organic, carbonate, or iron compounds (Jackson, 1973). In addition, chemical treatment of clay minerals is used to enhance the dispersion and separation of clay-size materials from sand and silt-size materials (Brown, 1961). Perhaps the most widespread and accepted procedures are those outlined by Jackson (1973) for the removal of organic, carbonate, and iron oxide materials.

The use of chemical treatments to remove non-clay materials is desirable because their presence may prohibit the parallel orientation of the mounted clay and, therefore, reduce XRD peak intensity. In addition, coatings of carbonate or iron oxide may act as cementing agents, possibly holding dissimilar clay materials together (Gibbs, 1967). Townsend and Reed (1971), for example, found that the removal of iron oxides exposed previously “masked” peaks and increased XRD peak intensity, as well as the number of clay-size particles in the sample. The identification of crystalline clay minerals in samples containing large amounts of amorphous material is also difficult. McKyes *et al.* (1974) concluded that the removal of amorphous materials not only produced a soil of “discrete and clean” mineral particles, but that the XRD

peaks of treated clays did not change in intensity, although they were much sharper than the XRD peaks of untreated clays.

However, such treatments prior to XRD analysis are not without question. Harward *et al.* (1962), for example, concluded that treatments for iron removal and dispersion affected the characterization of chlorite, montmorillonite-type minerals, vermiculite, and intergrade components. In addition, they found that increases in the degree of expansion upon solvation and in the degree of collapse upon K-saturation and heating were related to the severity of the treatment. Douglas and Fiessinger (1971) established that the use of H₂O₂ to oxidize organic matter causes a degradation of the XRD peaks of some standard soil clays. Harward and Theisen (1962) found that SCBD and Na₂CO₃ treatments to remove iron and disperse clays not only increased the number of clay species that could be identified, but also increased the likelihood of identifying montmorillonite. Furthermore, these authors arrived at the disconcerting conclusion that clay minerals identified by XRD techniques may be arbitrary and empirical, for they found identification to be dependent upon the particular combination of specimen carrier, saturating cation, particle-size, method of iron removal, dispersion reagents, and even the chance peeling of specimens.

Although chemical treatments of standard clay minerals have the capability of “manufacturing” clay assemblages which are not representative of soil-clay sys-

tems, a detailed examination of the effect of chemical treatments on soil clay minerals derived from the weathering of surface mixtures of volcanic ash and till materials has not been reported. Also, in view of the possibility that the striking similarity of clay mineral distributions identified within the volcanic ash-derived surface materials of the Western Cordillera may be more artificial than real (Brewster, 1979), it becomes increasingly important to determine possible laboratory-induced alterations of existing clay mineral assemblages. Even though some form of treatment may be necessary for clay dispersion and separation (Jackson, 1973), it is desirable to identify and characterize clay minerals as they exist *in situ*. In view of the possibility of clay mineral alteration resulting from treatments used to obtain "clean-sharp" XRD peaks, an experiment was devised to examine the effects of specific chemical treatments used individually and in combination upon the XRD characteristics of clay minerals present within mixtures of volcanic ash and till.

MATERIALS AND METHOD

For this experiment, the $<2 \mu\text{m}$ clay-size fractions were extracted by centrifugation from the mineral horizons of Pedon 6, located in the Mount Edith Cavell meadows of Jasper National Park, Alberta, Canada ($52^{\circ}42'N$, $118^{\circ}02'W$). The horizons consist of varying proportions of volcanic ash, till, and colluvial materials (Brewster, 1979). Chemical and physical characteristics of this pedon resulted in an Orthic Ferro-Humic Podzol (Typic Cryorthods) classification according to the strict application of the Canadian system of soil classification (Canadian Soil Survey Committee, 1978). Clay samples obtained from each of the four mineral horizons comprising this pedon were subjected to the following pretreatment or series of pretreatments, resulting in eight subsamples from each original clay sample:

1. Untreated.
2. Hydrogen peroxide removal of organic material (Jackson, 1973).
3. Sodium acetate removal of carbonates (Jackson, 1973).
4. Sodium citrate-bicarbonate-dithionite (SCBD) removal of iron oxides (Jackson, 1973; Mehra and Jackson, 1960).
5. Sodium acetate removal of carbonates followed by hydrogen peroxide removal of organic material.
6. Sodium acetate removal of carbonates followed by hydrogen peroxide removal of organic material followed by SCBD removal of iron oxide.
7. Hydrogen peroxide removal of organic material followed by SCBD removal of iron oxide.
8. Sodium acetate removal of carbonates followed by SCBD removal of iron oxide.

For each of these eight subsamples, XRD patterns were obtained for the following conditions: Mg-saturated and then air dried (Mg-ADPO); Mg-saturated and then saturated with ethylene glycol (Mg-GPO); K-saturated and then air dried (K-ADPO); K-saturated and then heated to 350°C for 0.5 hr (K-350°C); and K-saturated and then heated to 550°C for 0.5 hr (K-550°C). This series of pretreatments and the XRD procedures resulted in 40 XRD patterns for each mineral horizon.

All samples were mounted according to the suction method of Kinter and Diamond (1956) using the modification proposed by Rich (1969). The amount of clay-size material on each porous plate was uniform (186 mg), while the thickness and density of the mounted clay were controlled as much as possible in order to obtain a uniform distribution across the porous plate. The calculated density of the clay layer examined was approximately 14 mg/cm^2 , producing the optimum thickness corresponding to a starting angle of $35^{\circ}2\theta$ (Rich, 1975). It is important to note that for each mounted sample, the amount of clay used and the clay density were controlled as much as possible. By controlling the amount of material mounted on each porous plate and its distribution across the plate, both intra- and inter-sample examination of clay mineral XRD characteristics as revealed by peak size, shape, and location was possible. The samples were examined using Ni-filtered $\text{CuK}\alpha$ radiation and a General Electric XRD-5 diffractometer with a scanning speed of $2^{\circ}/\text{min}$. Figures 1 through 4 depict the XRD traces obtained for the Ae, Bhf, IIBf, and IIC horizons of Pedon 6.

RESULTS

Material composition

Detailed particle-size and petrographic analyses of Pedon 6 revealed that volcanic glass shards comprised approximately 53% of the material examined between 150 and $31.2 \mu\text{m}$ in size. The glass shard morphology and concentrations (clear glass = 5%; stretched glass = 4%; stretched-vesicular glass = 4%; fine-vesicular glass = 19%; and coarse-vesicular glass = 21%) are comparable to that of the St. Helens Y volcanic ash (Brewster, 1979). This ash originated from an eruption at Mount St. Helens in southwestern Washington approximately 3350 years BP (Crandell *et al.*, 1962). Other volcanic ash materials identified in the Mount Edith Cavell meadows of Jasper National Park, but not within Pedon 6, include Mazama ash and Bridge River ash, originating from eruptions dated at 6600 years BP and 2450 years BP, respectively (Fryxell, 1960; Nasmith *et al.*, 1967). The non-volcanic ash soil materials are composed of locally derived till or colluvial deposits. Deglaciation of this area of the Canadian Rocky Mountains is commonly considered to have occurred approximately 10,000 years BP (Rutter, 1972). If the

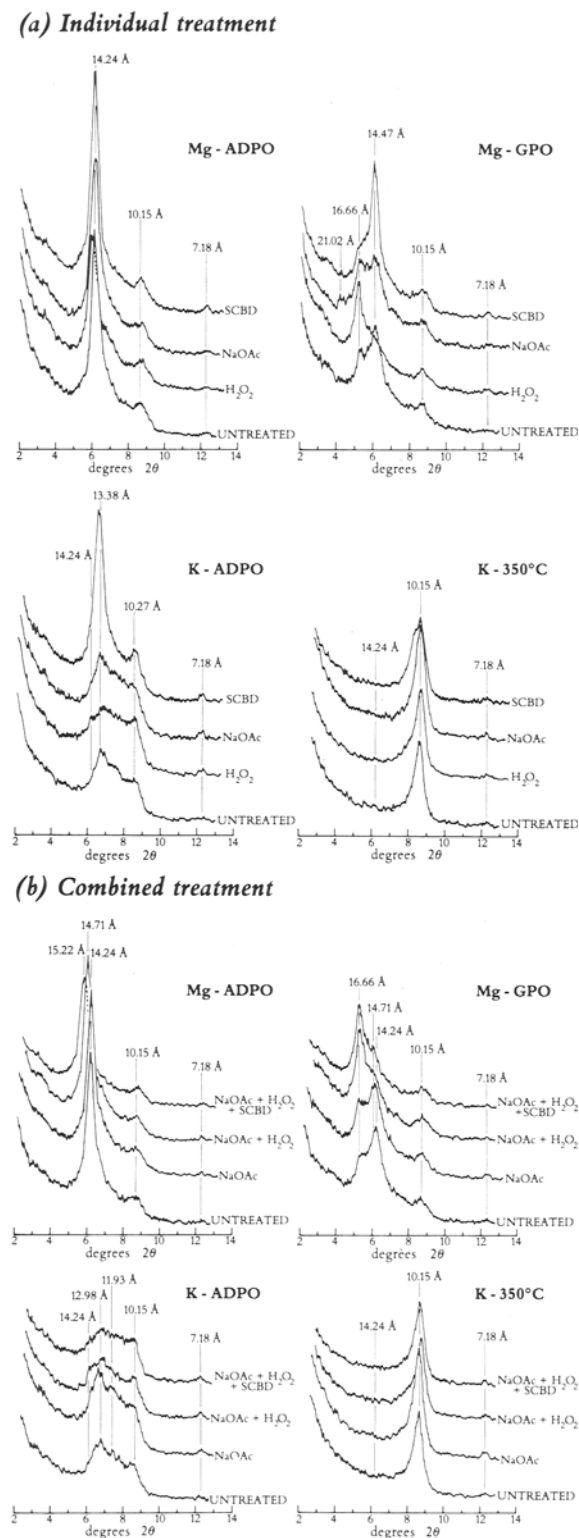


Figure 1. The effect of (a) individual treatment and (b) combined treatment upon clay mineral XRD characteristics of the Ae horizon, Pedon 6.

subsoil materials are colluvium, these materials are of variable age, having been deposited between the time of deglaciation (10,000 years BP) and the time of the oldest ashfall identified at the site (St. Helens Y, 3350 years BP).

Ae horizon

The volcanic ash-derived Ae horizon was estimated to contain only about 33% volcanic glass within the total soil material, although approximately 55% of the particles examined between 150 and 31.2 μm in size were identified as volcanic glass shards (Brewster, 1979). The XRD traces of the untreated clay sample obtained from this horizon (Figure 1a) showed the presence of illite (10.15 Å) and a variable expandable and collapsible 14-Å clay mineral. Following Mg-saturation and glycolation, this mineral expanded from 14.24 to 16.66 Å (Figure 1a, Mg-GPO); following K-saturation it partially collapsed to a variable 13.38–10.15-Å spacing (Figure 1a, K-ADPO). The spacing collapsed to 10.15 Å on heating the sample 350°C for 0.5 hr (Figure 1a, K-350°C). The behavior of this 14-Å mineral is thought to represent a mixture of montmorillonite, vermiculite, and Al-interlayered vermiculite. Thus, the XRD characteristics of the untreated sample indicate that the clay mineral suite of this horizon is dominated by Al-interlayered vermiculite.

Individual chemical treatment. Removal of organic material (4.7% organic carbon) by H_2O_2 treatment produced a significant expansion of the 14.24-Å peak to 16.66 Å following Mg-saturation and glycolation (Figure 1a, Mg-GPO). Asymmetry of the 16.66-Å peak to the high angle side suggests that expansion was not complete. The XRD patterns of the K-saturated and heated samples (Figure 1a, K-ADPO and K-350°C) suggest that although a montmorillonite-type clay mineral dominates the mineral suite, both vermiculite and Al-interlayered vermiculite are also present. The apparent dominance of the clay assemblage by the montmorillonite-like, expandable mineral in the H_2O_2 -treated sample contrasts with the subordinate role of this mineral in the untreated sample.

The XRD traces of samples treated with NaOAc to remove carbonates are characterized by a clay mineral which produced a sharp and intense 14.24-Å peak upon Mg-saturation (Figure 1a, Mg-ADPO). Saturation of this sample with ethylene glycol resulted in a variable expansion of the 14-Å peak to a 16.66-Å spacing and the creation of a broad plateau between 14.47 and 16.66 Å (Figure 1a, Mg-GPO). Partial collapse to 13.38 Å and asymmetry to the high angle side of the XRD peak produced by the K-saturated sample suggest the presence of Al-interlayered vermiculite (Figure 1a, K-ADPO). The NaOAc-treated sample did not exhibit as drastic a modification of the untreated-sample XRD traces as did the H_2O_2 -treated sample; however, the montmoril-

lonite-like expandable mineral appeared to be more abundant in the NaOAc-treated sample than in the untreated sample. Also, the characteristics of the XRD peaks of the NaOAc-treated sample contrast sharply with those of the untreated sample (Figure 1a).

Removal of iron oxides (0.55% dithionite-extractable Fe) by SCBD treatment changed the 14-Å peak in a manner which is contrary to the behavior of this peak with H₂O₂ treatment. After saturation with ethylene glycol, the 14.24-Å peak became asymmetrical to the low angle side, and a definite 16.66-Å spacing was not produced (Figure 1a, GPO). K-saturation produced an intense 13.38-Å peak which was asymmetrical on the high angle side and an increase in the intensity of the 10.27-Å spacing. Heating the sample to 350°C for 0.5 hr completely collapsed the spacing to 10.15 Å. The behavior of the Ae horizon samples subjected to SCBD treatment indicates that the samples are totally dominated by Al-interlayered vermiculite with minor amounts of vermiculite, illite, and vermiculite/montmorillonite interstratifications.

It can be seen from the examination of the Ae horizon clay minerals that individual treatments can change interpretations of the nature of the clay mineral assemblage. Although the preceding discussion primarily involved 14-Å clay mineral characteristics, it should be noted that illite (10.15 Å) is also present and that the XRD characteristics of illite are not changed by any of these treatments. The 7.18-Å spacings shown in Figure 1 were identified as kaolinite by prior analysis (Brewster, 1979).

Combined treatment. Chemical treatments used in combination markedly influenced the interpretation of the clay mineral assemblage in the Ae horizon of this pedon. The XRD traces of the untreated sample and the maximum-treated sample (NaOAc + H₂O₂ + SCBD) may be interpreted quite differently. The XRD traces of the Mg-saturated and glycolated samples (Figure 1b, Mg-GPO) show a progressive increase of expandable minerals with the increasing severity of treatment. K-saturation of these samples resulted in a variable collapse of the 14-Å spacing from 14.24 to 10.15 Å (Figure 1b, K-ADPO). With increasing severity of treatment the XRD peak intensity of the K-saturated samples decreased. This reduction may be partially due to clay mineral degradation, as evidenced by a broad plateau between 14 and 10 Å (Figure 1b, K-ADPO). Heating the K-saturated sample to 350°C collapsed the 14-Å spacing to 10.15 Å (Figure 1b, K-350°C).

Summary of effects upon Ae horizon samples. Within the Ae horizon of this pedon, pretreatments, both individually and in combination, created a variable interpretation of the clay mineral assemblage. If the untreated sample represents the clay mineral suite as it exists *in situ*, the assemblage appears to be dominated by Al-

interlayered vermiculite with subordinate amounts of vermiculite, illite, and kaolinite. The XRD traces of treated samples suggest that the clay mineral suite is dominated by montmorillonite-like, expandable clays. The nature of the clay species present in the Ae horizon did not change as a result of the treatments, but the relative abundance of some individual species increased as the severity of the treatment increased.

Bhf horizon

The Bhf horizon was estimated to contain approximately 29% volcanic glass within the total soil material. Volcanic glass shards accounted for approximately 53% of the particles between 150 and 31.2 μm in size (Brewster, 1979). The mineral assemblage of the untreated sample is dominated by a poorly ordered, Al-interlayered vermiculite (Figure 2a) with lesser amounts of illite and vermiculite. The XRD peaks of the untreated sample are irregular and weak, characteristics commonly associated with either poorly crystalline clay minerals or an abundance of amorphous materials. Unlike the alteration of Ae horizon clays, treatment of the Bhf horizon clays did not change the estimates of clay mineral species concentration, although XRD peak characteristics of these minerals were changed. The clay-size materials in the untreated sample (Figure 2a) are poorly ordered and probably contain considerable amorphous material, whereas the materials in the SCBD-treated sample have intense and symmetrical XRD peaks, characteristics which suggest well-crystallized and/or abundant clay minerals.

Individual treatment. NaOAc-treated samples have XRD characteristics which are similar to the XRD characteristics of the untreated samples. However, SCBD treatment (1.60% dithionite-extractable Fe) caused the greatest variation in the XRD peak characteristics between the treated and untreated samples (Figure 2a). The XRD traces of the H₂O₂- and SCBD-treated samples revealed a 14-Å clay component having a sharp and intense diffraction peak which did not collapse with K-saturation (Figure 2a, K-ADPO). The peak collapses to 10.04 Å on heating to 350°C (Figure 2a, 350°C). The SCBD-treated sample contained a minor 14.24-Å component which was stable after heating to 350°C and 550°C, a characteristic of chloritic clay minerals. The presence of chlorite within the Bhf horizon was only indicated in samples which had been treated with SCBD (Figure 2a).

Combined treatment. Subjecting samples to a combination of treatments gave mixed results (Figure 2b). The clays in these samples include Al-interlayered vermiculite, vermiculite, illite, and traces of kaolinite. Samples which underwent NaOAc + H₂O₂ treatment produced sharper and more intense peaks than those of the untreated sample, although the peak spacings are

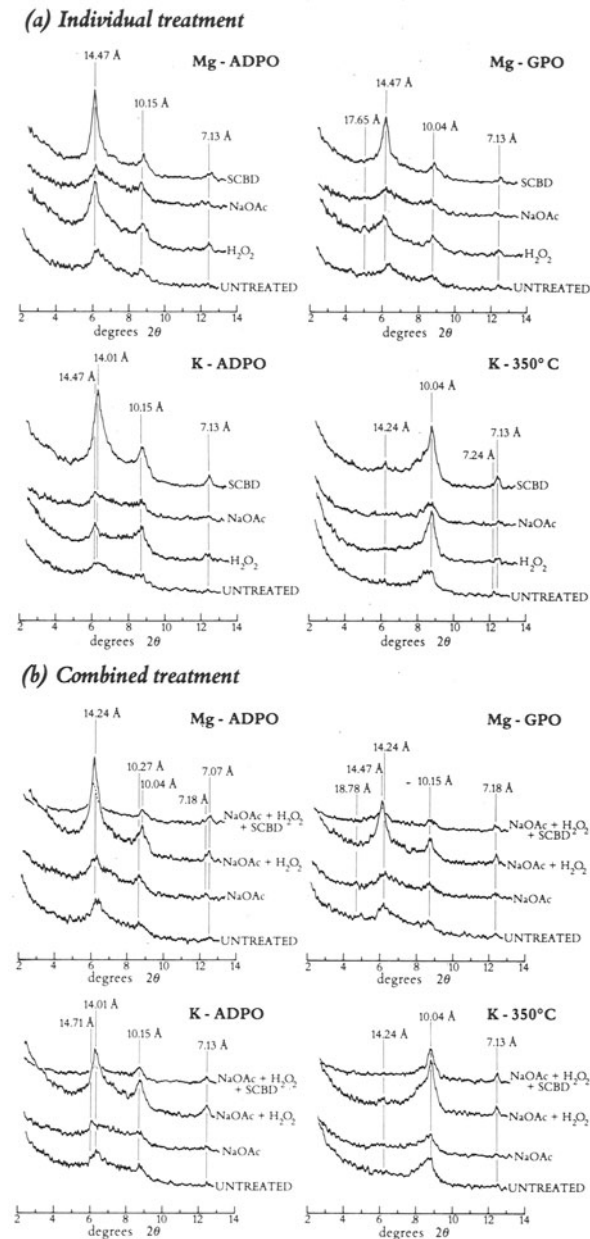


Figure 2. The effect of (a) individual treatment and (b) combined treatment upon clay mineral XRD characteristics of the Bhf horizon, Pedon 6.

the same (Figure 2b, Mg-ADPO). The sharp, intense XRD peaks of the NaOAc + H₂O₂-treated samples were unchanged by Mg-saturation and glycolation (Figure 2b, Mg-GPO), K-saturation (Figure 2b, K-ADPO), and heating of the K-saturated sample (Figure 2b, K-350°C).

Bhf horizon samples which were subjected to the most severe treatment (NaOAc + H₂O₂ + SCBD) did

not produce the greatest alteration (Figure 2b). Samples treated with NaOAc + H₂O₂ contain more crystalline clays (or less amorphous material) than those samples which were treated with NaOAc + H₂O₂ + SCBD (Figure 2b). Unlike the Ae horizon samples, the composition and concentration of Bhf horizon clay mineral species does not change in accordance with increasing pretreatment severity. The treatment of the volcanic ash-derived B horizon clay minerals does, indeed, result in "clean-sharp" XRD peaks; however, these peaks may not be representative of the *in situ* character of the clay-size material.

IIBf horizon

Individual treatment. Untreated samples from the till or colluvial materials of the IIBf horizon of this pedon produced XRD peaks characteristic of illite and iron-rich chlorite (Figure 3a). These XRD peaks are symmetrical, but rather subdued. H₂O₂ treatment (1% organic carbon) did not alter the 14-Å chlorite peak or the 10-Å illite peak, except for an increase in the peak intensity of Mg-saturated samples (Figure 3a). NaOAc-treated samples produced XRD peaks which were similar to those of the untreated sample. The greatest change in the XRD characteristics of these samples was produced by the SCBD treatment (2.07% dithionite-extractable Fe). The XRD peaks of these products are considerably more intense than those of the untreated samples (Figure 3a). Although the XRD peaks of the SCBD-treated sample are "cleaner" and "sharper" than those of the untreated sample, the interpretation of the illite-chlorite ratio or the evaluation of the concentration of these two minerals was not altered by individual treatment. The clay minerals in the SCBD-treated sample appear to be more crystalline, or conversely, less altered than those of the untreated sample. Alternatively, crystalline clay-size material is more abundant in the latter (Figure 3a).

Combined treatment. A combination of chemical treatments on samples of the IIBf horizon yielded more intense XRD peaks with increasing severity of treatment (Figure 3b). The detection of individual clay minerals was not changed by such treatments; however, the patterns of the most severely treated samples were similar to those of unaltered crystalline clays which were uncontaminated by amorphous materials.

IIC horizon

Individual and combined treatment. Illite and iron-rich chlorite comprise the clay mineral suite of the IIC horizon (Figure 4a). The XRD peaks of the untreated sample are extremely sharp and intense. H₂O₂ treatment of these samples produced broader and weaker XRD peaks (Figure 4a). The intensity and symmetry of

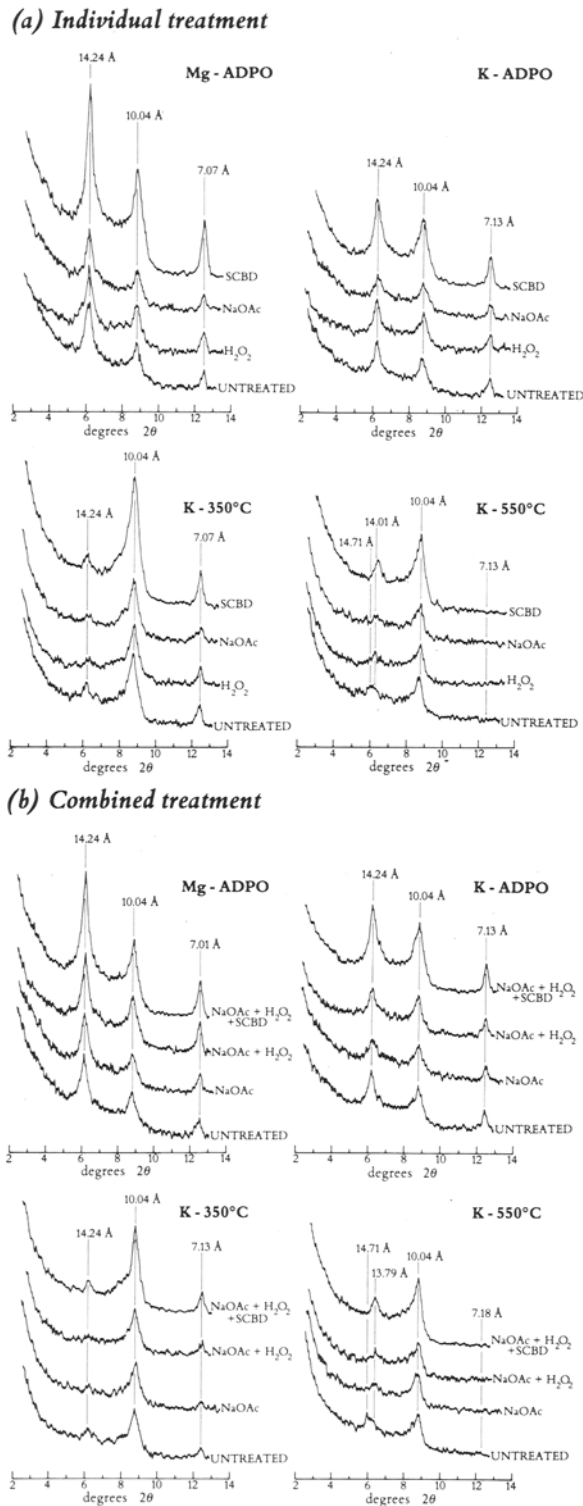


Figure 3. The effect of (a) individual treatment and (b) combined treatment upon clay mineral XRD characteristics of the IIBf horizon, Pedon 6.

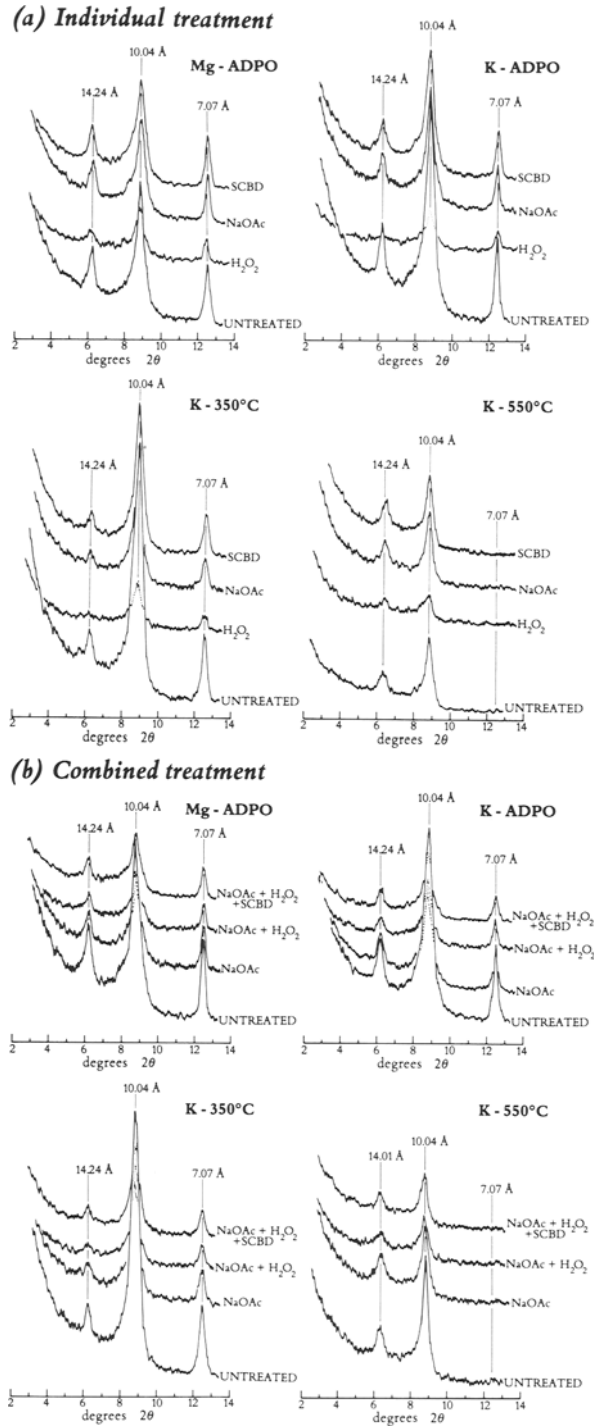


Figure 4. The effect of (a) individual treatment and (b) combined treatment upon clay mineral XRD characteristics of the IIC horizon, Pedon 6.

the XRD peaks of the NaOAc- and SCBD-treated samples are comparable in intensity and symmetry to those of the untreated sample. Samples which were subjected to combinations of treatments produced weaker and broader XRD peaks compared to those of the untreated sample (Figure 4b).

DISCUSSION AND CONCLUSIONS

The results of this experiment demonstrate the need for caution when interpreting clay mineral assemblages, species concentration, or crystallinity when treatments are used to produce "clean-sharp" XRD peaks. Treatment of the weathered Ae, Bhf, and IIBf horizon samples resulted in rather dramatic improvements in the XRD peak intensity, while treatment of IIC horizon samples resulted in subdued peak intensities. The contrasting effect which these treatments had on the XRD peak intensities of weathered and unaltered clay samples suggest that some form of mineral modification occurs following treatment; however, the modification is masked by marked improvements in XRD peak intensities.

A problem arises when attempting to determine the XRD characteristics of the natural (*in situ*) clay system. In this study, chemically untreated samples were assumed to be representative of the natural clay system. The XRD peak characteristics of these untreated samples were then compared to the XRD characteristics of the treated samples in an attempt to establish treatment effect. In view of the possibility of non-parallel orientation of clay minerals, carbonate and iron oxide bonding, and masking by amorphous materials, the XRD patterns of the untreated samples may not represent the so-called "natural clay system." The problem of XRD characterization of the *in situ* clay is difficult to overcome when using naturally occurring, weathered clay samples; however, evaluations of treatment effect are best made using an untreated sample as a reference.

A more pronounced effect of such treatment is the apparent production of expandable clay minerals within the Ae horizon. Increasing the severity of treatment increases the concentration and crystallinity of montmorillonite-like expandable minerals. This finding is not new; Harward *et al.* (1962) observed that the likelihood of identifying montmorillonite increases and the degree of dispersion increases as the severity of the treatment for iron removal increases. This phenomenon is probably due to the hydrolysis and removal of interlayer aluminum ions during H₂O₂ and SCBD treatments, causing the degradation of the clay minerals. Consequently, the poorly ordered vermiculite or vermiculite/montmorillonite interstratifications become "weathered" by hydrogen attack and aluminum removal, in a manner similar to the weathering processes in the acid and eluvial A horizon. The fact that this reaction takes place only with Ae horizon samples supports this general conclusion, because these samples

contain weathered vermiculite and vermiculite/montmorillonite interstratifications which would serve as substrata for further laboratory modification.

The possible dilution of untreated samples by amorphous clay-size materials may also be significant. Within other pedons examined from Jasper National Park, volcanic ash-derived B horizons were found to contain approximately 50% more amorphous clay-size material than the overlying A horizons (Brewster, 1979). However, the volcanic ash-derived B horizons examined in this study did not exhibit the degree of variation in species composition, concentration, and crystallinity as did the samples obtained from the A horizon. This suggests that the effect of dilution by amorphous materials is subordinate to the chemical modification of pre-existing weathered clay minerals.

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Резюме—Изучались воздействия удаления карбонатов с помощью NaOAc, удаления органических материалов с помощью H₂O₂ и удаления окисей железа с помощью натриевого цитрат-бикарбоната-дитионита (НЦБД) на рентгеновско-дифракционные (РД) характеристики глинистых минералов, присутствующих в выветренных отложениях вулканического пепла. Все предварительные обработки образцов, отобранных из горизонтов Ae и Bhf, содержащих вулканический пепел, вызывали резкое повышение интенсивностей РД пиков, в то время как обработка образцов из горизонтов IC вызвало сглаживание РД пиков. Кроме того, химические обработки вызывали явное увеличение количества расширяющихся глинистых минералов в горизонте Ae, вероятно из-за гидролиза и удаления межслойного Al³⁺ когда использовались H₂O₂ и НЦБД. Следовательно, плохо упорядоченные и измененные в почвенных условиях вермикулитные или вермикулито/монтмориллонитные перемежающиеся пропластки стали “выветренными” под воздействием H⁺ и удаления Al³⁺, т.е. путем, подобным естественным процессам, которые происходят в кислотном и элювиальном горизонте A. К тому же, возможные изменения РД пиков из-за разубоживания кристаллических глинистых концентраций аморфным материалом с размерами, соответствующими глинам, оказались второстепенными по сравнению с индуцированным в лаборатории химическим изменением выветренных глинистых минералов. [N.R.]

Resümee—Es wurden die Auswirkungen auf die Röntgendiffraktometer-Charakteristika von—in Vorkommen verwitterter vulkanischer Aschen auftretenden—Tonmineralen untersucht, die entstehen, wenn man Karbonate mit NaOAc, organisches Material mit H₂O₂, und Eisenoxide mit Natriumcitrat-Bicarbonat-Dithionit (SCBD) entfernt. Alle Vorbehandlungen führen bei den Proben, die aus den vulkanische Asche führenden Ae und Bhf Horizonten stammen, zu einem ungeheuren Anwachsen der XRD-Peakintensitäten. Die Behandlung der Proben aus dem IC Horizont führt dagegen zu einer Verminderung der XRD-Peakintensitäten. Außerdem bewirken die chemischen Behandlungen, daß der Gehalt an quellfähigen Tonmineralen in Proben des Ae Horizontes sichtbar zunimmt, was wahrscheinlich auf die Hydrolyse und das Entfernen von Zwischenschicht-Aluminium zurückzuführen ist, wenn H₂O₂ und SCBD verwendet wurden. Daher werden schlecht kristallisierte und durch Bodenbildung beeinflusste Vermiculite und Vermiculit/Montmorillonit-Wechsellagerung durch den H⁺-Angriff und das Entfernen von Al³⁺ in einer Art verwittert, die den natürlichen Prozessen ähnlich ist, wie sie in sauren und eluvialen A-Horizonten stattfinden. Auch schien eine mögliche Beeinflussung der XRD-Peaks durch die Verdünnung der Konzentration des Kristallisierten Tons durch amorphes Material von Tongröße der durch die Behandlung bedingten chemischen Veränderung der verwitterten Tonminerale untergeordnet zu sein. [U.W.]

Résumé—Les effets sur les caractéristiques XRD des minéraux argileux présents dans des dépôts de cendres volcaniques altérées de l'enlèvement par NaOAc de carbonates, par H₂O₂ de matières organiques, et par dithionite-bicarbonate-citrate de sodium (SCBD) des oxides de fer, ont été examinés. Tous les prétraitements d'échantillons dérivés des horizons-Ae et -Bhf contenant de la cendre volcanique ont résulté en un accroissement dramatique des intensités de sommet XRD, tandis que le traitement des échantillons d'horizons-IC ont résulté en des intensités de sommet XRD amoindries. Il a aussi été remarqué que les traitements chimiques produisaient un accroissement apparent de la quantité de minéraux argileux expansibles dans l'horizon-Ae, sans doute dû à l'hydrolyse et à l'enlèvement de l'inter-couche Al³⁺ lorsque H₂O₂ et SCBD étaient employés. Ainsi, la vermiculite ou les interstratifications vermiculite/montmorillonite pauvrement ordonnées ou pédogéologiquement modifiées sont devenues “altérées” par l'attaque d'H⁺ et par l'enlèvement d'Al³⁺ d'une manière semblable aux procédés naturels qui se passent dans l'horizon-A acide et éluvial. De plus, la modification possible de sommet XRD due à la dilution de concentrations d'argile cristalline par une matière amorphe de taille argileuse semblait être subordonnée à l'altération chimique induite dans le laboratoire des minéraux altérés. [D.J.]