PROCEEDINGS OF THE TWENTY-SIXTH ANNUAL KECK RESEARCH SYMPOSIUM IN GEOLOGY

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CLAY MINERAL ANALYSIS AND PALEOCLIMATE INTERPRETATION OF A MIDDLE MIOCENE PALEOSOL, POWDER RIVER VOLCANIC FIELD, NORTHEAST OREGON

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Research Advisor: Dr. Meagen Pollock

INTRODUCTION

Geochemical and mineralogical analysis of paleosols by techniques such as XRF, ICP-MS, XRD, SEM, and TEM have been used to quantitatively and qualitatively determine features such as paleoprecipitation, paleotemperature, and paleotopography (*e.g.* Sheldon et al., 2002; Sheldon, 2006; Chamberlain and Poage, 2000; Takeuchi et al., 2007). Clay minerals in particular can be used as weathering indices and reflect the climate conditions necessary for their formation (*e.g.* Kalm et al., 1996; Tabor et al., 2004).

Several paleosols are preserved between volcanic deposits in Northeast Oregon. Outside of Elgin, OR, exists the Cricket Flat paleosol (CFP) among volcanic outcrops of the Powder River Volcanic Field (Fig. 1 in Bader and Nicolaysen, this volume). Basalt flows about 25 km south of the CFP have been dated to ~14 Ma (Ferns et al., 2010), and the paleosol is believed to be approximately the same age.

OBJECTIVE

This study intends to propose an interpretation of climate and soil conditions present during formation of the CFP 14 Mya, in the middle Miocene. XRD and SEM analyses are used to identify clay minerals. The interpretation presented is based on the identified minerals and the conditions in which they are known to form, and the physical features of the paleosol.

METHODS

Field Methods

Observations of physical paleosol characteristics were used to determine soil horizon boundaries. Bulk hand samples from all horizons were collected, though only samples from three horizons were used for lab analysis. HCl reactivity throughout the profile was tested and recorded.

Lab Methods

Clay was extracted from paleosol samples by methods described in Moore and Reynolds (1997) and random powder mounts prepared for XRD analysis by methods described by the USGS (U.S. Geological Survey). Samples were run in a Rigaku MiniFlex II Desktop XRD at a 0.05° step size from 3 - 75° 2-theta, for 1 minute per degree. Sample spectra were compared to known mineral spectra catalogued in the PDF-2 ICDD Database, which was released in 2010. Clay powder for SEM secondary electron image analysis was affixed to sample platforms via double-sided carbon tape and coated with carbon. Samples were examined on a JEOL 5610LV SEM at a working distance of 8mm and accelerating voltage of 5 kV.

RESULTS

Field Results

Figure 1 shows the stratigraphy of the CFP. The top of the column, 0 cm depth, is at the boundary between the paleosol and overlying basalt. The olivine basalt has a minimum thickness of three meters, above which there is no exposure. The basalt is highly vesicular near the contact, indicating that it is indeed the flow bottom. Below the basalt is unit one, labeled an A horizon due to significant organic content (root traces and plant remnants present) with a mineral soil component. This pale beige horizon has a somewhat blocky structure, is comprised of silt and clay-sized particles, and is highly erodible. Clay sample NB12-10 was taken from this horizon.

The ABc unit (Ac of Lopez-Maldonado, this volume) has characteristics of both A and B horizons. Some root traces extend into this unit, and the "c" denotes the presence of red nodules among the pale pink-yellow matrix.

A sharp and wavy contact at -22 cm marks the top of the Bt horizon. Prismatic columnar peds signify high clay concentration, which is denoted by the "t". Relict clay cutans may be present; ped surfaces appear to be covered with mud cracks ≤1 mm in diameter. Prisms are consolidated but readily separate from one another and break easily. Ped exteriors are a pale pink color, while the interiors are mostly red brown with pale green and purple spots. Sample AM12-12 is from this unit.

The fourth unit begins at -48 cm and is considered a Bt2 horizon; the unit has noticeable clay accumulation and the same pale pink color, but there are differences in structure compared to the Bt horizon above. The upper boundary is a gradual transition from columnar peds to a crumbly, blocky structure, which transitions into horizontal layers 2-3 cm thick near the bottom of the unit.

The BC horizon starts 78 cm below the profile top. Sample AM12-15 was collected from the top-most 25 cm of this unit. The unit is red orange, with patches of pale orange and yellow brown. Consolidated and fist-sized blocks can be dislodged (some with difficulty) from the outcrop. This blocky structure indicates the presence of clay (hence the B horizon label) yet the appearance of weathered volcaniclastic relicts (pumice and perlite clasts) at -85 cm also warrants the C horizon classification. Further down the blocky structure fades and more evidence of volcaniclastic parent material appears.

The R horizon begins 184 cm below the profile top and extends downward; the base of this unit is not exposed. This volcaniclastic material is unweathered, consolidated and massive with a fine-grained matrix and many coarse to pebble sized clasts, mostly pumice and perlite. The color is grey and yellow grey. This unit is considered the parent material of the paleosol.

At no point in the profile did hand samples react with hydrochloric acid (HCl).

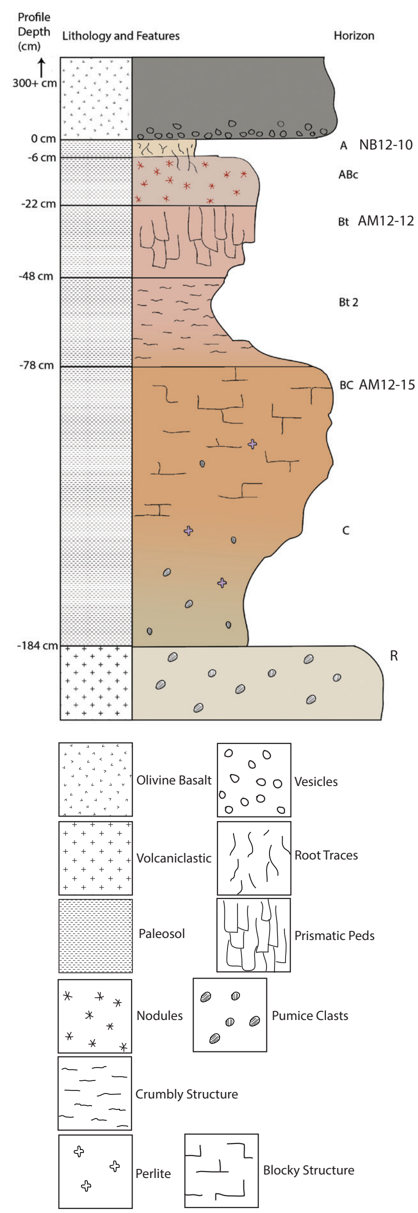


Figure 1. Stratigraphic column of the Cricket Flat paleosol summarizing soil horizons and physical features, and colors.

XRD Results

For XRD analysis, the unknown clay mineral identifications were chosen on the basis of fit of measurement (FOM) numbers and best-fitting peaks, and whether the phase identification made sense in the context of field observations. Kaolinite and montmorillonite clay standards were used as calibrators to judge the accuracy of the sample preparation and XRD measurements. Because we know the identity of the clay standards, the FOM numbers for correct phase identifications serve as reference points to which unknown clay identifications' FOM numbers can be compared. An FOM closest to 0 - 0.2 is ideal, and anything approaching 3.0 is likely a bad match (Rigaku PDXL software manual). The standards' FOM numbers ranged from 0.621 to 1.209, so obtaining FOM results under or close to 1.0 for unknown clay mineral matches is reasonable and can be accepted as a good match.

XRD analysis identified halloysite in all three samples and calcian montmorillonite in samples AM12-12 and AM12-15 (Fig. 2). The atomic spacing in the halloysite changed from 14Å to 7Å to 10Å in samples NB12-10, AM12-12, and AM12-15, respectively.

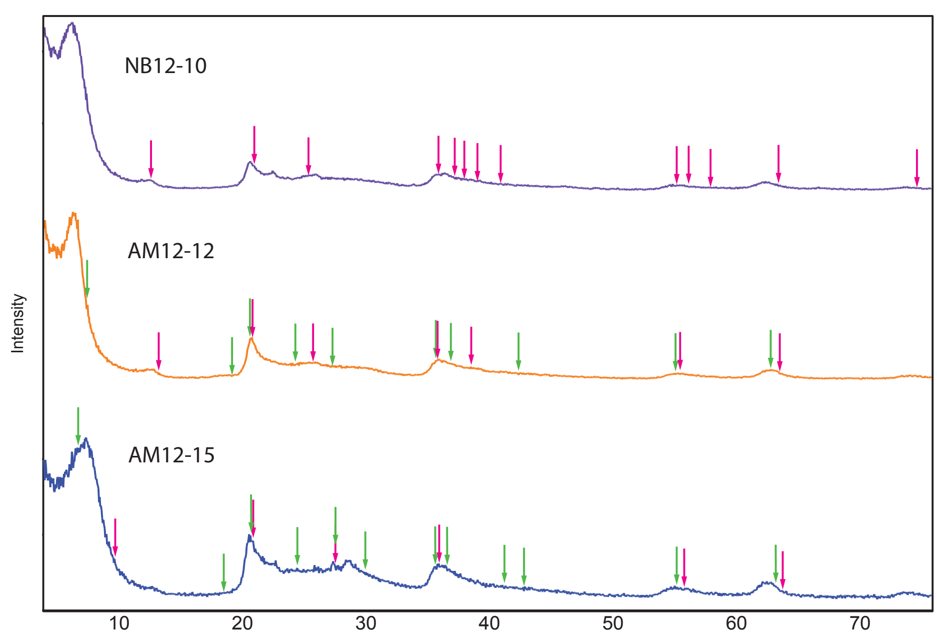
SEM Results

NB12-10 Oval aggregates occur and range in diameter from <10 μm to 50 μm. Some anhedral to euhedral platy crystals (≤5 μm) can be seen on these aggregates, though their edges appear to be ragged (Fig. 3A). Many plates exhibit hexagonal or pseudo-hexagonal shapes. In Figure 3A a packet of clay sheets can be seen on its side, with the edges facing up. Surrounding the packet are smaller spheroids of clay.

AM12-12 Clay particles in this sample form aggregates ~10 μm to 60 μm in diameter, though some clumps of clay measure <5 μm across (Fig. 3B). Subhedral to euhedral plates exhibiting a hexagonal or pseudo-hexagonal shape are seen in Figure 3B. In the center of the figure are two stacked hexagonal plates with visible growth lines. Some aggregates are larger than 5μm in one dimension and look slightly rhombic in shape and three-dimensional form. These larger aggregates in Figure 3B appear stacked and layered; on several aggregate edges, the outlines of horizontally laying clay sheets are visible.

AM12-15 In general, the clay aggregates appear fluffier and less compact than previous samples. In Figure 3C, ≤1 μm sized spherical clumps of clay spot the aggregate surface, though these spots may also be anhedral clay plates. The clay plates in this sample are less defined than in previous samples, but anhedral and subhedral hexagonal platelets can still be found.

Figure 2. XRD spectra of clay samples from the CFP. Pink arrows denote identified halloysite peaks and green arrows denote montmorillonite peaks. FOM for each sample are NB12-10) halloysite 14Å: 1.049; AM12-12) halloysite 7Å: 0.849, montmorillonite: 0.632; AM12-15) halloysite 10Å: 0.611, Ca-montmorillonite: 0.829.



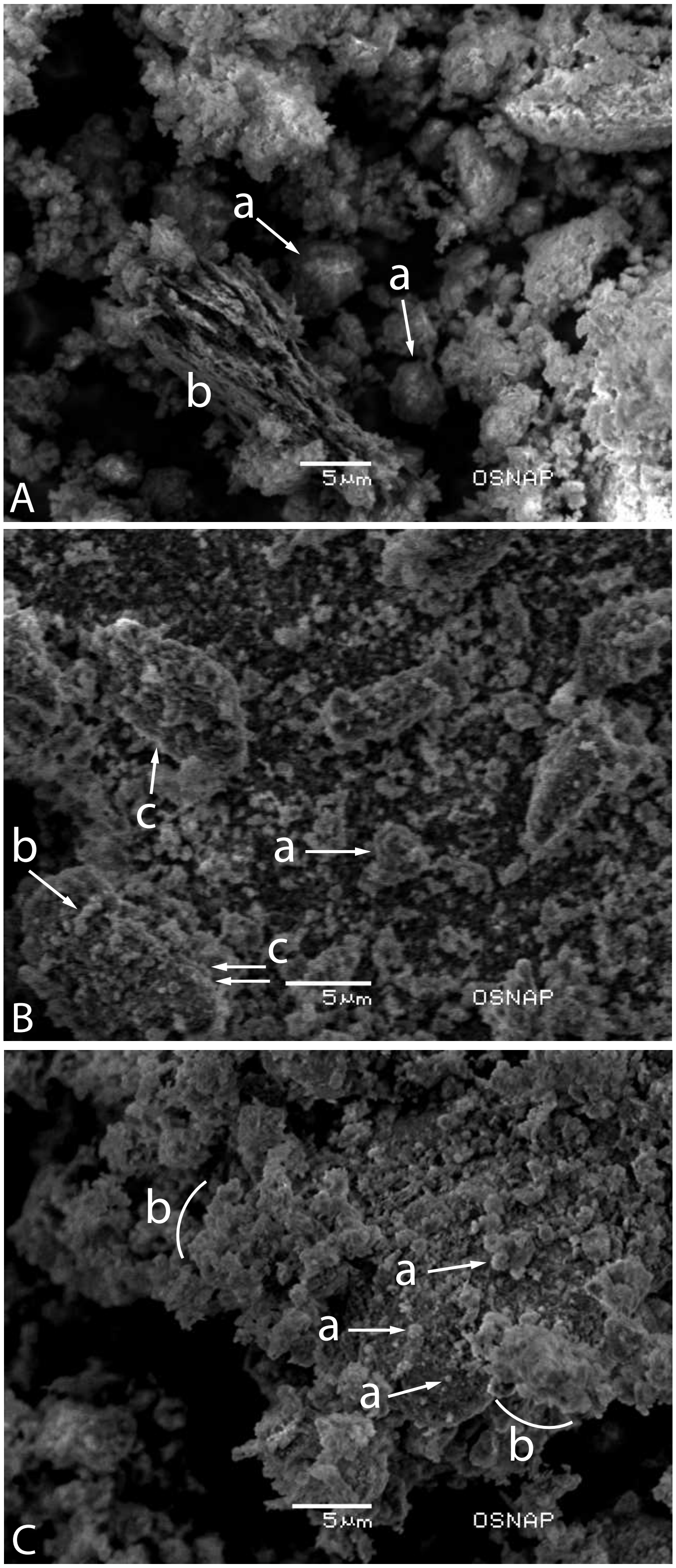


Figure 3. SEM secondary electron images of clay samples from the CFP. A) NB12-10: clay spheroids (a) and packets of clay sheets (b) are the dominant clay particle morphologies. B) AM12-12: stacked euhedral hexagonal plates exhibiting growth lines (a) as well as subhedral hexagonal plates in a stepped cluster (b) are present. The edges of horizontally laying clay sheets are visible on some aggregates (c). C) AM12-15: small (<5 µm) spheroids (a) sit on and among packets of anhedral clay sheets (b).

DISCUSSION

Given the FOM and values of 2-theta and d-spacing for the kaolinite and montmorillonite standards, we have established that the methods of analysis are reliable. Thus, XRD analysis of the sample clays indicates that the minerals present in this paleosol are montmorillonite (calcian) and halloysite (14Å, 10Å, 7Å). SEM and field observations support our XRD identifications. Halloysite can display platey and spheroidal morphologies, not just the commonly known tubes, as we observed by SEM (Wada and Mizota, 1982). Montmorillonite, too, is typically flakey and sometimes occurs as rosettes (Fiore et al., 2001). Many studies have discovered these minerals as weathering products of glassy volcanic material (*e.g.* Abdioglu and Arslan, 2005; Hover and Ashley, 2003; Churchman, 2000). The columnar peds in the Bt horizon can denote the presence of swelling clays such as montmorillonite. Additionally, halloysite can form synonymously and/or directly from allophane (Joussein et al., 2005), which is also common in volcanic ash soils.

These minerals, when developing on the same parent material, also form under similar climates. Halloysite formation requires a high moisture regime, i.e. a humid or water saturated environment, though the soil needs to be well-drained (Joussein et al., 2005). Parfitt (1990) found that moderate rainfall favored halloysite formation, compared to high rainfall, which favored allophane formation. In general, halloysite is common in wet and warm climates with a short dry season (Joussein et al., 2005).

Montmorillonite forms where leaching is minimal and water does not continuously flow through the soil, for example in areas with low rainfall or in soils with a restrictive layer (Allen and Hajek, 1989). This may seem contradictory to the conditions necessary for halloysite formation, but montmorillonite was not found in the topmost horizon of the CFP. Leaching may have occurred in the two uppermost horizons but the downward flow of water would have been impeded by the argillic ped horizon, below which leaching would be minimal. Although smectite clays are most prolific in cooler temperate environments, when forming from volcanic ash they have been found in warm temperate climates with a distinct dry season

(Allen and Hajek, 1989).

The conditions of formation for these clay minerals lead us to the interpretation that this paleosol formed under a humid, warm temperate climate with low to moderate rainfall and a distinct dry season. Although halloysite and montmorillonite can form in tropical and warm temperate climates, the lack of kaolinite, a clay that dominates tropical soils, leads us to believe that climate conditions were neither warm nor wet enough to be considered tropical. Furthermore, high rainfall would not favor halloysite or montmorillonite formation. The presence of the columnar ped layer indicates seasonal desiccation of clay in the soil, which supports the proposal of a dry season.

CONCLUSIONS

XRD identifications are supported by SEM and field observations. Halloysite and montmorillonite both form from the weathering of volcanic glass under similar climatic conditions. Thus, the CFP is interpreted as having developed under a humid, warm temperate climate that experienced a yearly dry season and low to moderate rainfall.

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