

Characterization of the weathering status of feldspar minerals in sandy soils of Minnesota using SEM and EDX

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Abstract

Scanning electron microscopy was used to assess the weathering status of plagioclase and alkali feldspar grains isolated from sandy outwash soils located under three vegetation communities: mixed red and white pine, maple–basswood, and white cedar. Feldspars were selected for this study because they were the most common weatherable minerals in these sandy soils. A morphological weathering index, based on the percent of the surface of the feldspar grains covered with etch pits, was used to evaluate weathering intensity. Results revealed that significant pedogenic weathering has occurred in the surface and subsurface horizons of these soils compared to the parent material. Plagioclases were found to be much more weathered than alkali feldspars. Within the plagioclase solid solution series, the weathering index increased linearly with increasing Ca content as determined by energy dispersive X-ray analysis. Comparisons among the three sites showed that weathering environments under the pine and maple–basswood communities were relatively similar, being only slightly more intense under pine. The most intense weathering occurred under the white cedar community, particularly in the E horizon. Linear regression analyses showed that the weathering index for alkali feldspars was significantly related to soil pH ($r^2=0.30$, $p<0.001$). In the case of plagioclases, multiple linear regression analyses showed that the weathering index was significantly dependent on both Ca/(Ca + Na) molar ratio and pH ($r^2=0.83$, $p<0.001$), with 64% weight effect due to Ca/(Ca + Na) ratio, and 19% due to pH.

1. Introduction

Estimates of the relative weathering status of soils can be useful pedological indicators of the relative ages of different soils or the relative weathering intensities occurring under

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different environments; they may also provide clues to the pre-depositional weathering history of sediments.

The most direct method of determining relative weathering status is to measure the relative depletion of one or more minerals. This method has proven useful in a number of studies (St. Arnaud and Sudom, 1981; Mermut et al., 1986; Santos et al., 1986); it does not work well, however, in coarse-grained soils having low quantities of readily weatherable minerals or in the early stages of soil development where weathering losses may be masked by natural variability in the concentration of individual minerals.

Birkeland (1984) suggested that individual mineral grain morphologies may be a more sensitive indicator of relative differences in weathering than depletion ratio methods because minerals show morphological features associated with weathering (particularly surface etching) long before they are significantly depleted. It has been clearly shown that most silicate minerals do not weather uniformly (Wilson, 1975; Berner and Holdren, 1979; Petrovich, 1981); preferential weathering occurs at high energy sites associated with a number of structural features, including twinning planes, dislocations, fracture zones, and areas of mechanical abrasion. This type of preferential weathering produces etch pits and other changes in grain surface morphology. Several methods of determining the relative weathering status of mineral grains have been developed using these ideas. For example, Locke (1979) measured the depth of etching on the surface of hornblende grains of Arctic soils and calculated a mean maximum etching depth. Cremeens et al. (1992) analyzed etch pit size, density, and shape on orthoclase and pyribole grains to determine relative weathering intensities in soils of a loess catena. They concluded that size of individual etch pits was related to intensity of weathering, but that etch pit shape was specific to each mineral. The drawback to these methods is that they rely on fairly intensive measurements of etch pit characteristics. We proposed to use a much simpler technique based on visual estimation of the relative area of grain surfaces covered by etch pits.

The main objectives were to:

- (1) assess the weathering status of alkali and plagioclase feldspar grains isolated from soils by using a simple pedogenic index based on relative surface coverage of individual grains by etch pits; and
- (2) demonstrate the validity of this simple method by relating the estimated weathering status of these minerals to the soil solution environments from which they were obtained.

2. Materials and methods

2.1. Study area and soils

This study was conducted at the Cedar Creek Natural History Area (CCNHA), a reserve and field research facility located about 80 km north of Minneapolis in east-central Minnesota near the town of East Bethel. Cedar Creek is operated by the University of Minnesota and is an NSF-sponsored Long Term Ecological Research Site. The landscape is a patchwork of forest and abandoned fields located on the Anoka Sand Plain. The parent material in the area is glacial outwash sand formed during the wastage of the Grantsburg Sublobe of the

Des Moines Lobe by the Mississippi River during the latter stages of the Wisconsin (Cooper, 1935); some areas have been reworked by wind. Due to the predominance of sandy soils, much of Cedar Creek was originally dominated by fire-successional communities, mainly open oak, pine, or aspen savannas common to droughty soils of the region.

Three sites were selected for this study, each located under different vegetation communities: (Site 1) mixed white pine (*Pinus strobus* L.) and red pine (*Pinus resinosa* Ait.); (Site 2) mixed maple (*Acer saccharum* Marsh.) and basswood (*Tilia americana* L.); and (Site 3) a white cedar (*Thuja occidentalis* L.) community. All sites were located on outwash sand. Site 1 is located on a nearly level portion of a spur ridge above a small creek; clear evidence of old fires (charcoal, burn scars) is preserved on the landscape and in the soils at this site. Site 2 is located on the summit of Crone's knoll, a small rounded hill (< 10 m high) completely surrounded by cedar swamp; no evidence of past fires is present. The presence of the maple–basswood community on this site is somewhat puzzling, both because neither species is fire resistant and also because they generally occur on finer-textured soils. It has been hypothesized (E. Cushing, pers. commun.) that the maple–basswood community persisted because the knoll has been protected from fire by the surrounding cedar swamps. Pollen analyses from Cedar Creek Bog (< 100 m from Crone's knoll) indicate that both species have been present in this area for several thousand years.

The soils at Sites 1 and 2 are deep and well-drained, and are morphologically very similar; both were originally mapped in Zimmerman (Alfic Udipsamments) mapping units (Grigal et al., 1974), but the sum thickness of individual lamellae was sufficiently thick (> 15 cm) at both sites for them to be reclassified as either Psammentic Eutroboralfs or Psammentic Glossoboralfs. The presence of lamellae, which greatly improve the water holding capacity of the soil, may also help explain the occurrence of the maple–basswood community at Site 2. Abbreviated profile descriptions for the soils at Sites 1 and 2 are given in the Appendix.

Site 3 is located on the edge of Crone's knoll in a footslope position, has a slope of about 5–8%, and has a water table at about 70 cm, close to the average water table depth. The water table at this site fluctuated 0.41 m during a 2.5 year period encompassing this study, mainly the result of one exceptionally dry summer (Basiletti, 1994). The soil at Site 3 also fell within a Zimmerman mapping unit (Grigal et al., 1974), but was described and reclassified using field criteria and micromorphology as a Typic Endoaquod. This soil occurs in a narrow band (< 10 m wide) at the base of the knoll; its areal extent is too limited for mapping. A profile description of the soil at Site 3 is given in the Appendix.

2.2. Sample preparation and analyses

Three horizons from each site were selected for sampling: the A, Bw2, and E6 horizons from Sites 1 and 2, and the A, E, and C horizons from Site 3. The fine sand fraction (0.1–0.25 mm) was separated for use in this study because it is the dominant size fraction in these soils (Table 1). The fine sand fraction was obtained from a 40 g soil sample by wet sieving following removal of organic matter using 30% H₂O₂ (Kunze and Dixon, 1986), removal of Fe oxides using dithionite–citrate–bicarbonate (Mehra and Jackson, 1960), and dispersion in Na-pyrophosphate.

Table 1
Physical and chemical properties of the soil horizons selected for this study

Horizon	Depth (cm)	Particle size distribution (%)						pH (H ₂ O)	OC ^b (%)	Fe ^f (%)	Exchangeable bases (mol _c /kg)			ECEC ^d (mol _c /kg)	BS ^e (%)			
		Sand ^a			Coarse silt	Fine silt	Clay				Ca	Mg	Na			K		
		vcs	cs	ms													fs	vfs
<i>Site 1 (mixed pine)</i>																		
A	0–5	0.1	1.3	21.6	53.7	11.1	4.2	6.0	2.0	4.89	0.64	0.48	1.74	0.30	0.05	0.00	3.47	52.4
Bw2	30–88	0.2	1.0	22.3	54.9	14.4	2.7	3.1	1.4	5.56	0.07	0.38	0.82	0.20	0.01	0.00	1.80	57.2
E6	164–195	0.2	0.2	22.2	54.3	16.8	2.1	2.0	2.2	5.76	0.05	0.43	0.57	0.20	0.00	0.00	2.10	36.7
<i>Site 2 (maple-basswood)</i>																		
A	0–8	0.1	1.3	24.1	51.1	10.8	4.5	5.7	2.4	5.05	2.56	0.42	3.90	0.20	0.00	0.00	6.91	59.3
Bw2	60–110	0.1	1.2	24.7	52.1	12.2	2.8	4.5	2.4	5.79	0.10	0.38	0.08	0.10	0.00	0.00	1.40	64.3
E6	200–223	0.2	1.4	21.3	58.1	12.4	2.6	1.5	2.4	6.08	0.02	0.43	0.82	0.30	0.00	0.00	2.15	51.2
<i>Site 3 (white cedar)</i>																		
A	0–10	0.0	1.2	24.9	49.3	8.3	5.6	6.7	4.0	5.12	3.58	0.15	6.40	0.50	0.00	0.00	8.15	84.7
E	10–20	0.0	1.2	22.8	53.5	12.0	4.6	5.2	0.7	4.67	0.34	0.10	0.90	0.10	0.00	0.00	1.83	54.6
Cg	60–100	0.3	1.3	25.6	56.3	11.4	2.0	2.1	1.0	7.48	0.07	0.13	0.70	0.00	0.00	0.00	0.97	72.2

^avcs (very coarse sand) = 1–2 mm; cs (coarse sand) = 0.5–1 mm; ms (medium sand) = 0.25–0.5 mm; fs (fine sand) = 0.1–0.25 mm; vfs (very fine sand) = 0.05–0.1 mm.

^bOrganic carbon.

^cCBD extractable iron.

^dEffective cation exchange capacity.

^eBase saturation = (Σ bases/ECEC) × 100.

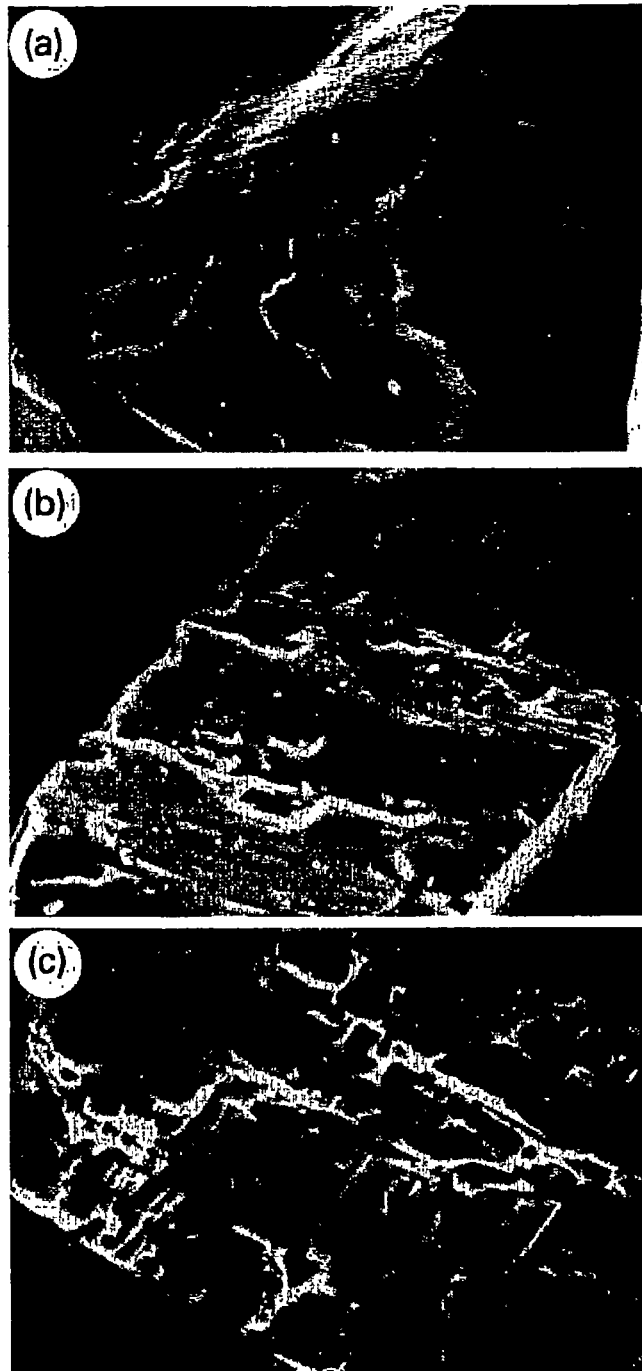


Fig. 1. Scanning electron micrographs of feldspar grains removed from soils showing a wide range of estimated weathering indices (EWI): (a) plagioclase, $\text{Ca}/(\text{Ca} + \text{Na}) = 0.40$, $\text{EWI} = 2$; (b) plagioclase, $\text{Ca}/(\text{Ca} + \text{Na}) = 0.50$, $\text{EWI} = 5$; (c) plagioclases, $\text{Ca}/(\text{Ca} + \text{Na}) = 1.00$, $\text{EWI} = 10$.

2.3. Evaluation of grain weathering status

Initially, we attempted to determine differences in weathering status by determining the relative depletion of feldspars. This technique was abandoned, however, because only slight,

Table 2

The weathering index (WI) limits as related to the percent of etch pit formation visually estimated on the surface of weathered feldspar grains

	WI									
	1	2	3	4	5	6	7	8	9	10
% surface coverage	<5	5–10	10–20	20–30	30–45	45–60	60–70	70–80	80–90	>90

but not significant, differences were found within or among horizons. It quickly became obvious that a more sensitive technique would be required. To that end, we developed a relative weathering index for individual feldspar grains based upon the visual estimation of the percentage of the mineral surface covered by etch pits.

Morphological examinations of mineral grains were performed using a scanning electron microscope (SEM) (Philips SEM 500, Philips Electronic Instruments, Inc., Mahwah, New Jersey). Samples were prepared by mounting several hundred grains of each sample on aluminum stubs using double sided tape and coating them with a thin film of carbon using a carbon evaporator. After the sample was inserted into the SEM, mineral grains were selected by random vertical and lateral movements of the microscope stage to prevent operator bias in the selection of grains. The chemical compositions of selected grains were determined by energy dispersive X-ray analysis (EDX) on 2 or more areas of each grain to minimize errors. Since we were interested exclusively in feldspar minerals, only grains that had compositions and morphologies characteristic of feldspars were evaluated for their weathering status.

Once a feldspar grain was identified, the operator visually estimated the percentage of the mineral surface covered by etch pits. All observations and evaluations of the mineral grains were made directly on the SEM monitor. This percentage was converted to a weathering index of integer values from 1 to 10 according to the limits presented in Table 2. Several reference micrographs of weathered feldspar grains, displaying a range of weathering indices, were reexamined at intervals to maintain consistency of weathering index estimations. Some of the reference micrographs are presented in Fig. 1.

3. Results and discussion

Confirmation of parent material uniformity is a necessary requirement in the evaluation of mineral weathering in soils. Semi-quantitative, random powder X-ray diffraction analyses of feldspars in the fine sand fraction and heavy minerals in the silt fraction showed no significant differences within or among sites (data not shown; Bouabid, 1992), supporting the assumption of parent material uniformity. Elemental concentrations of Zr were also not significantly different within or among sites. The particle size and chemical data presented in Table 1 also show broad similarities between soils.

Plagioclase grains were about 2 to 3 times more abundant than alkali feldspar grains. The plagioclase feldspars had a wide range of Ca:Na molar ratios, with Na-rich plagioclases being more abundant, as shown by the relative frequency distribution presented in Fig. 2.

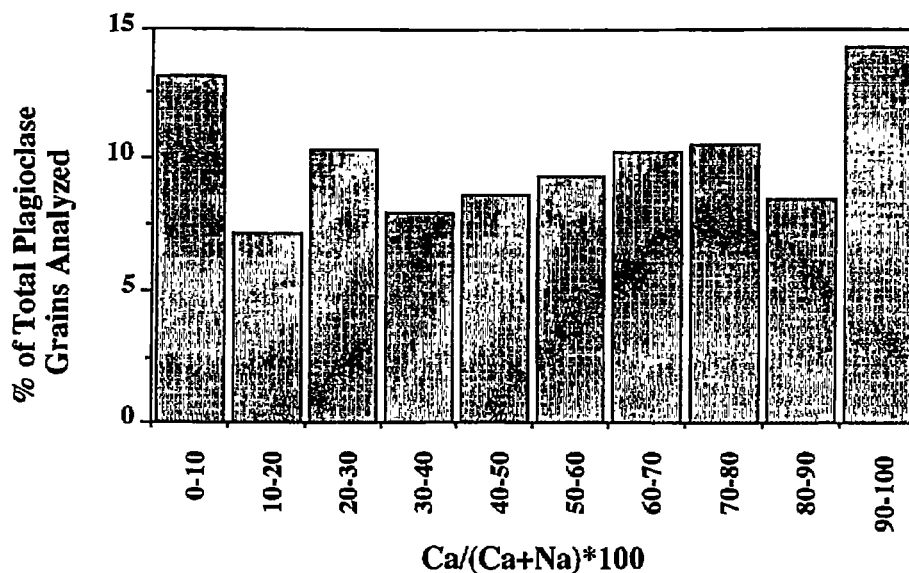


Fig. 2. Plagioclase grain frequency as a function of the $\text{Ca}/(\text{Ca} + \text{Na})$ molar ratio. Values are for all three sites.

The wide range of plagioclase compositions observed in these soils may be explained in part by the origin of the outwash sand which contains some sediments from three different glacial lobes (Cooper, 1935; Farnham, 1956). The alkali feldspars were predominantly high K feldspars with negligible Na contents.

Comparisons of the weathering index of K-feldspar grains among sites and by depth within sites were performed using a statistical multiple comparison of weathering index means (Table 3). The results show that the mean weathering index of K-feldspar grains from the A and Bw2 horizons of Sites 1 and 2 were not significantly different from each other but were significantly ($p < 0.05$) higher than that of K-feldspar grains from their

Table 3

Weathering index of alkali feldspar grains and a multiple comparison of means using a *t*-test^a

Site	Horizon	Weathering index	
		Mean	SD ^b
1	A	3.69 a	0.64
	Bw2	3.36 a	0.49
	E2	2.52 b	0.51
2	A	3.50 a	0.93
	Bw2	3.33 a	0.78
	E2	2.50 b	0.51
3	A	3.81 a	0.83
	E	4.39 c	0.61
	C	2.58 b	0.52

^aStatistical level of significance used $p < 0.05$.

^bStandard deviation.

respective deep E horizons. The mean weathering index relative to the A horizon from Site 3 was similar to those of Sites 1 and 2. However, the E horizon of Site 3 showed a distinctly higher weathering index ($p < 0.05$) than those of the A horizons of all three sites (4.4 vs. 3.7, 3.5 and 3.8). A higher degree of weathering is typical of the E horizons of Spodosols.

Interpretation of plagioclase weathering is more complex because of its compositional variability. The rate of plagioclase weathering has been shown to be dependent on the mineral Ca:Na ratio (Holdren and Speyer, 1987), with higher Ca:Na (more anorthitic) plagioclase weathering faster than low Ca:Na plagioclase. This is clearly demonstrated in Fig. 3, which shows regression relationships between the estimated weathering index and the Ca/(Ca + Na) molar ratio for each horizon sampled. In each case, the weathering index increased linearly with increasing Ca content (Fig. 3), although the slopes of the regression lines are somewhat lower in the E6 and C horizons than in the other horizons.

The weathering index values at the intercept for Ca/(Ca + Na) = 100 for the A horizons from all three sites were comparable (9.0, 8.9 and 9.3 for Sites 1, 2 and 3, respectively) and higher than those of the E6 and C horizons, indicating that weathering was more intense at the surface than at lower depths, in agreement with the K-feldspar data. The E6 and C horizons also showed evidence of weathering, indicating that the parent material was preweathered to some extent before deposition and/or has weathered in situ. Nevertheless, because of the compositional uniformity of the parent material, the degree of weathering of mineral grains from the E6 and C horizons can serve as a reference for evaluating the weathering intensity in other horizons. The weathering index of the Bw2 horizon was intermediate between those from the A and E6 horizons in Site 1 and was not much different from that of the A horizon in Site 2.

The weathering index of the E horizon from Site 3 was distinctly higher than that of the surface horizons in all three sites. High weathering intensities in eluvial horizons are a common characteristic of Spodosols and Spodosol-like soils (McKeague et al., 1982; Righi and Chauvel, 1987). Quartz/feldspar ratios in the fine sand fraction of some Spodosols and other acid soils in Canada showed marked depletion of feldspars in the E horizons (McKeague and Brydon, 1970).

The intense weathering observed in the A horizons from all three sites, and the more intense weathering observed in the E horizon from Site 3 shows a clear relationship to soil pH. A linear regression analysis between K-feldspar weathering indices and the pH of the horizon from which they were extracted was highly significant ($r^2 = 0.30$, $p < 0.0001$). A multiple linear regression analysis of weathering index versus the Ca/(Ca + Na) molar ratio of plagioclase grains and horizon pH was also significant ($R^2 = 0.83$, $p < 0.001$), with 64% of the variability explained by the Ca/(Ca + Na) ratio, and 19% by pH. For both alkali and plagioclase feldspars, the regression coefficient of pH was negative, indicating that higher weathering rates were associated with lower pH, as expected.

Parent material uniformity among the three sites was also supported by the weathering index estimations for the E6 and C horizons. Mean weathering indices for alkali feldspars (2.52, 2.50, and 2.58 for the E6 and C horizons for Sites 1 to 3, respectively) did not show any significant differences among sites (Fig. 3). Likewise, the plagioclase weathering index values at the intercept for Ca/(Ca + Na) = 100 for the E6 and C horizons were also comparable (6.7, 6.6 and 6.8 for Sites 1 to 3, respectively). This is particularly interesting considering that Sites 2 and 3 are well- or excessively well-drained and the C horizon at

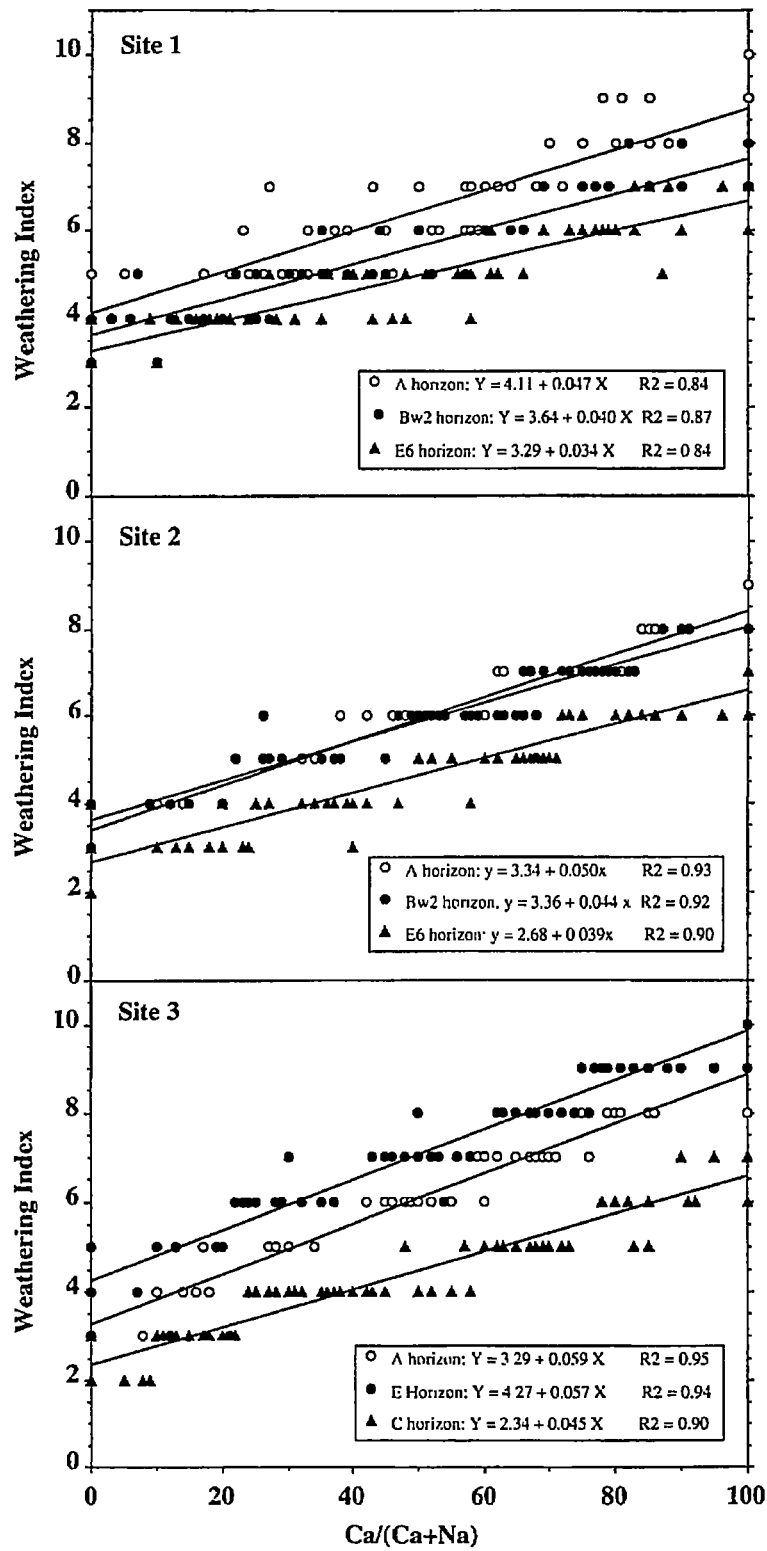


Fig. 3. Weathering indices of plagioclase grains as a function of Ca/(Ca + Na) molar ratio.

Site 3 is continuously saturated, suggesting that little weathering of deep E6 and C horizon materials has occurred at these three sites.

Plagioclase weathering indices showed that the weathering environment under Site 1 was slightly more intense than that in Site 2, in agreement with soil solution chemistry and mineral stability data for these horizons (Bouabid, 1992); soil solutions from Site 1 were somewhat more undersaturated with respect to the feldspar minerals than at Site 2. These two environments differ markedly from that occurring at Site 3, which displayed slightly more intense weathering in the A horizon than was observed in the other two sites (Table 3). Weathering in the E horizon at Site 3 was even more pronounced, probably due to the presence of cedar leaf litter, which produced much larger quantities of low molecular weight dissolved organic compounds than were measured on the other two sites (Bouabid, 1992). These low molecular weight organic compounds have been shown to induce rapid and intense weathering of primary minerals (McKeague et al., 1982).

Our findings regarding the weathering trends observed in these soils are based on the assumption that the plant communities at each site have persisted for sufficiently long periods of time to have imprinted their effects on the soils. The forest communities in the area are known to be at least 200 years old, but are probably much older. Similar types of vegetation are believed to have existed in this area at least since the hypsithermal period (~5000 years ago) (D.F. Grigal and E. Cushing, pers. commun.). Good correlations between the soil chemistry at each site and the weathering index of grains obtained from those sites suggests that a steady state has been reached for sufficiently long periods to cause the weathering differences observed.

4. Conclusions

The significant correlations observed between the estimated weathering indices versus soil pH for the alkali feldspars, and versus soil pH and Ca content of individual grains for plagioclase demonstrate the validity of this relatively simple method for estimating weathering intensity.

Estimated weathering indices indicate that significant pedogenic weathering has occurred in the surface and subsurface horizons of the soils studied. The less intense weathering observed in the deep E6 and C horizons from all sites may indicate pre-depositional weathering and/or in situ weathering. Plagioclases displayed more intense weathering features than alkali feldspars, and the degree of weathering of plagioclase grains increased linearly with increasing Ca content. Both of these trends are in agreement with laboratory studies of feldspar weathering rates.

Appendix 1. Abbreviated profile descriptions for the studied soils*Site 1*

- A 0–5 cm: very dark gray (10YR 3/1)¹ fine sand; weak, very fine, granular structure; very friable; many roots; strongly abrupt; smooth boundary
- BW1 5–30 cm: yellowish brown (10YR 5/4) fine sand; single grained; loose; acid; gradual irregular boundary
- BW2 30–88 cm: yellowish brown (10YR 5/4) fine sand; single grained; loose; abundant medium brown (10YR 4/4) mottles; acid; clear smooth boundary
- Bt1 88–90 cm: dark brown (7.5YR 4/4) loamy fine sand; angular and subangular blocky fragments; hard and weakly cemented when dry; slightly acid; abrupt irregular boundary; occasional yellowish brown (10YR 5/4) tongues
- E2 90–98 cm: yellowish brown (10YR 5/4) fine sand; single grained; loose; acid; abrupt, irregular boundary
- Bt2 98–100 cm: dark brown (7.5YR 4/4) loamy fine sand; angular and subangular blocky fragments; hard and weakly cemented when dry; slightly acid; abrupt, smooth boundary

The horization from the Bt2 horizon to 3.5 m (the depth of sampling) consists of alternating interlamellar (E) horizons and lamellae (Bt). Typically, the lamellae were thicker and the interlamellar horizons were thinner with depth. With the exception of thickness, their other features are similar to the E2 and Bt2 horizons described. The depth of the C horizon was > 3.5 m.

Below the Bt2, the horization consists of: E3 (100–115 cm); Bt3 (115–120 cm); E4 (120–134 cm); Bt4 (134–140 cm); E5 (140–158 cm); Bt5 (158–164 cm); E6 (164–195 cm); Bt6 (195–203 cm).

Site 2

- A 0–8 cm: very dark gray (10YR 3/1) fine sand; weak, very fine, granular structure; very friable; heavily matted with roots; strongly acid; abrupt, smooth boundary
- Bw1 8–60 cm: pale brown (10YR 6/3) and brown (10YR 5/3) fine sand; single grained; loose; slightly acid; common faint yellowish brown (10YR 5/6) mottles; gradual irregular boundary
- Bw2 60–110 cm: yellowish brown (10YR 5/4) fine sand; single grained; loose; few dark brown (7.5YR 4/4), 0.2 to 0.5 cm thick, irregular, weakly cemented bands; common medium brown (7.5YR 4/4) mottles; acid; gradual irregular boundary
- Bt1 110–112 cm: reddish brown (5YR 4/4) loamy fine sand; massive breaking to angular and subangular blocky fragments; hard and weakly cemented when dry; slightly acid; abrupt, irregular boundary
- E2 112–140 cm: light yellowish brown (10YR 6/4) fine sand; medium subangular blocky structure parting to single grained; loose; abrupt, smooth boundary

¹ Colors are for moist soils.

Bt2 140–143 cm: reddish brown (5YR 4/4) loamy fine sand; massive breaking to angular and subangular blocky fragments; hard and weakly cemented when dry; slightly acid; abrupt, irregular boundary

The horizonation from the Bt2 horizon to 3.5 m (the depth of sampling) consists of alternating interlamellar (E) horizons and lamellae (Bt). Typically, the lamellae were thicker and the interlamellar horizons were thinner with depth. With the exception of thickness, their other features are similar to the E2 and Bt2 horizons described. The depth of the C horizon was > 3.5 m.

Below the Bt2, the horizonation consists of: E3 (143–158 cm); Bt3 (158–161 cm); E4 (161–170 cm); Bt4 (170–174 cm); E5 (174–195 cm); Bt5 (195–200 cm).

Site 3

A 0–10 cm: very dark gray (10YR 3/1, 3/2) fine sand; weak, very fine granular structure; very friable; heavily matted with roots; acid; abrupt, smooth boundary

E 10–20 cm: white (10YR 8/1) or light gray (10YR 7/1) fine sand; single grained; loose; many fine medium and large roots; acid; clear wavy boundary

Bs 20–47 cm: brown (7YR 5/4) and yellowish brown (10YR 5/6) loamy fine sand; fine and medium subangular blocky fragments; friable; common distinct reddish brown (5YR 2.5/2) mottles; very few tongues from E horizon; abrupt boundary

Bhs1 47–55 cm: red (2.5YR 4/4) and yellowish red (5YR 5/8) loamy fine sand; common prominent reddish brown (5YR 2.5/2) and black (5YR 2.5/1) iron–manganese concretions; few grayish brown (10YR 5/2) mottles; clear boundary

Bhs2 55–60 cm: dark red (2.5YR 3/4 and 2.5YR 2.5/4) loamy fine sand; massive; firm; hard; clear smooth boundary

B/Cg 60–70 cm: grayish brown (10YR 5/2) fine sand; common coarse, distinct reddish brown (5YR 5/4) and gray (2.5YR N6/) mottles; few black (5YR 2.5/1) concretions; gradual wavy boundary

Cg 70+ cm: gray (2.5YR N6/) fine sand; single grained; loose

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