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Abstract

Numerous studies have established that laboratory determined weathering rates are several orders of magnitude greater than rates derived from field studies at the catchment or basin scale. The laboratory rates have been determined on specimen minerals or rock isolates which were subsequently crushed. Some samples were pretreated to remove organic coatings and possible carbonate contamination. Using magnetic, density, and size separation, we isolated a plagioclase (An_{20})K-feldspar fraction from an arid zone soil (classified as Pachappa, Mollic Haploxeralf). Silica release rates decreased with time and after up to 3.7 years were in the range 0.02×10^{-12} mol m^{-2} s^{-1}, when surface area is expressed with BET surface area or 0.6×10^{-12} mol m^{-2} s^{-1}, when expressed with geometric surface area. These Si rates are in good agreement with published field rates, as are the individual mineral dissolution rates calculated from mineral abundance and cation release. These data are in contrast to faster laboratory determined rates based on crushed or ground samples, or shorter reaction times. Based on laboratory studies with and without various pretreatments, we conclude that organic and Fe oxide surface coatings due to natural weathering are not the explanation for our low reaction rates. Apparently even mild treatment such as sonification and sieving results in enhanced weathering which persists for more than 100 days.

1. Introduction

Extensive laboratory studies on the kinetics of feldspar and mica weathering of specimen minerals in the pH range 4.5-7, have led to Si dissolution rates on the order of $(2-15) \times 10^{-12}$ mol m^{-2} s^{-1}. In contrast, rates from mass balance calculations in watershed studies are approximately 100 to 1000 times slower. Velbel (1990) has calculated that differences in temperature between laboratory and field measurements account for up to a 5-fold discrepancy in these rates. It has been considered that at least part of the differences are due to preparation methods of specimen minerals (grinding, sonification) that increase surface reactivity and cause lattice strain.

Additional factors include surface coatings of secondary minerals and organic matter.

The objectives of this study are to determine the dissolution rate of a plagioclase-K-feldspar fraction separated from an arid zone soil and to compare this rate to reported values for specimen minerals and to weathering rates determined from field studies.

2. Materials and methods

2.I. *Mineral preparation*

A sample of Pachappa soil (fine sandy loam mixed, thermic, Mollic Haploxeralf) derived from

weathering of granodiorite of the southern California batholith, was collected from a site at the U.S. Salinity Laboratory in Riverside, California, U.S.A. The soil was air-dried, lightly crushed with a rubber mallet to break up soil aggregates and sieved. The $0-100$-μm fraction was saturated with 1 M NaCl, followed by deionized (DI) water washing and sedimentation to obtain a 10-100-μm fraction. This size fraction was boiled in 1 M sodium acetate at pH 5 for 8 h to remove carbonates (Jackson, 1969). This treatment was followed by extensive washing with DI water to remove adsorbed acetate. Although the soil is classified as non-calcareous, it has been suggested that all arid zone soils contain at least trace concentrations of calcite. The fraction was next boiled in 20% H_2O_2 at pH 5 (adjusted with HCl) and a soil solution ratio of 1: 1 for 8 h and allowed to react for 48 h to remove organic matter (Kunze and Dixon, 1986), and Fe- and Mn-oxide coatings, followed by washing with DI water. After further sedimentation to remove any newly produced <10-μm material, the 10–100-μm fraction was saturated with 0.5 M $CaCl_2$, washed with DI water until the electrical conductivity was below 0.05 dS m^{-1} and dried at 60°C. This treated material was subjected to magnetic and density separations to isolate the desired mineral fraction.

Magnetic material was removed by first passing a hand-held magnet wrapped with paper through the sample. The sample was then slowly passed through a strong electromagnetic field, and the material retained was removed. Further separations of-materials of varying magnetic susceptibility were performed with a Franz Isodynamic Magnetic Separator (Model Ll)[1]. Material was separated into two fractions by passing the material down an incline (forward slope), with the incline tilted (side slope). Material was successively removed at increasing amperage. Material remaining after 1.6 A and a side slope of 20°, a forward slope of 25" and vibration setting of 8 was rerun with a side slope of 9". This non-magnetic fraction was subsequently passed through the various

settings numerous times to increase the separation efficiency.

Density separation was accomplished by placing material in a separatory funnel containing diiodomethane and benzene mixed to a desired density and stirred slowly. The material retained from the magnetic separation was split at a density of 2.6 x 10^6 g m^{-3}. The material $d < 2.6$ was used in the weathering studies described below. After density separation, the materials were washed with benzene and acetone and then dried. This isolated mineral fraction constituted 50.5% of the original soil.

Subsamples were taken for elemental analysis, surface area determination, and X-ray diffraction (XRD). Weighed samples were digested using the hydrofluoric acid in a closed vessel method (Lim and Jackson, 1982), in which first aqua regia, then hydrofluoric acid and then boric acid are added. The digests were analyzed for total Ca, Mg, Na, K, Fe, Al and Si by Atomic Absorption Spectroscopy (AAS) and Inductively Coupled Plasma Emission Spectroscopy (ICP-ES). Surface area was determined by single-point BET using a Quantsorb Jr. (Quantachrome Corp., Syosset, New York, U.S.A.) with 30% nitrogen and a 100% nitrogen calibration. Standards of known surface area (TiO_2 and Al_2O_3) were used as a control for the calculated surface area values. For XRD analysis, we selected the areas of the largest peak for each mineral to estimate individual mineral composition using an external quartz standard (Pawloski, 1985).

2.2. Weathering studies

The mineral isolate was thoroughly rinsed in DI water, dried and weighed into 0.06-l polyethylene bottles. We added 1.36 g of material (BET surface area of 3.0 m^2) in each bottle. Next we added 0.03 l of filtered (0.45 μm) DI water (pH 5) to each bottle. Eight replicates were prepared. The bottles were stored in the dark at 24.4 $\pm$ 0.1°C and hand-shaken daily. The pH was measured and 25-ml samples collected from the replicates were filtered through a 0.2-μm filter on day 1, 3, 7, 14, 21, 28, 35 and 43. Filtered samples were placed into separate polyethylene bottles. All samples were analyzed for Ca, Mg, Na and K by AAS and for Si by Technicon Autoanalyzer (Technicon, Buffalo Grove, Illinois,

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[1] Trade names and company names are included for the benefit of the reader and do not imply endorsement or preferential treatment of the product by the USDA.

U.S.A.). Ion release rates were calculated from the differences in mass between the replicate bottles sampled at the different time intervals.

In a second set of experiments, 1.36 g (BET surface area of 3.0 m^2) of the mineral isolate was placed into 0.04-l polycarbonate tubes with screw caps, and 0.03 1 of DI water (pH 5) was added. Three replicates and one blank were prepared. The tubes were stored in the dark at $23 \pm 1°C$ and hand-mixed daily. Periodically, 5 ml were sampled, filtered through prerinsed 0.2-μm filters and stored as before. DI water was added back to the samples to replenish the original volume after each sampling. Ions released were calculated from the changes in mass with time after correcting for mass removed during sampling. Samples for Si and Ca were taken 6 times for the first 28 days, then every 7 to 11 days for 213 days. The tubes were stored for 3 years, then sampled for Si, Ca, Mg, Na, and K. The material was washed with water then brought back to a 30-ml volume. They were sampled again at intervals of 2 to 3 weeks. Silica was analyzed using the heteropoly blue method (Clesceri et al., 1989) and cations were analyzed by AAS. At the conclusion of this long-term experiment, the supernatant was removed, the sample tubes dried and reweighed to check for loss of sample, and the solids were removed for a redetermination of BET surface area.

To determine if the pretreatments to remove carbonates and organics affected the reaction rates, 0.050 kg of 0- 100 μm Pachappa soil was placed in each of four l-l beakers, washed with DI water, settled and siphoned to obtain a fraction of 10- 100 pm. One sample was used as a control, the second received the acetate treatment, the third received the H$_2$O$_2$ treatment, and the fourth received both the acetate and H$_2$O$_2$ treatments. Newly generated material of O-10 μm was removed by washing and decantation after pretreatments. Each of the four samples was washed in DI water, treated with 0.5 *M* CaCl$_2$, next washed in DI water and finally settled to remove the < 10 pm fraction by decantation and dried at 60°C. Surface area was determined as described above. Subsamples of exactly 1.00 g were placed into each of eight 0.06-l polyethylene bottles and 0.03 1 of DI water was added. The bottles were sampled over a period of 40 days as described above.

3. Results

XRD analysis, summarized in Table 1, indicated the presence of the following minerals and estimated abundance: plagioclase (40%) alkali feldspar (35%) quartz (26%) and biotite (trace). Based on the XRD analysis we determined the plagioclase to be of oligoclase-andesine composition. These results are consistent with the findings of Larsen (1948) that the composition of the plagioclase in granodiorite in the Southern California batholith is oligoclase. The results of the chemical analysis, expressed as g kg^{-1}, was as follows: SiO$_2$, 691; Al$_2$O$_3$, 152; CaO, 14; MgO, 1.6; Na$_2$O, 45; K$_2$O, 35; Fe$_2$O$_3$, 7.9. Based

Table 1
Mineral composition and abundance in the isolated fraction

Mineral	Chemical formula	XRD abundance (g g^{-1})	Abundance (chemical composition) [a] (g g^{-1})	Geometric surface area [a] (m^2)	BET S.A. in 1.36 g sample [b] (m^2)	Relative S.A. in isolate
Plagioclase (oligoclase)	Na$_{0.8}$Ca$_{0.2}$Al$_{1.2}$Si$_{2.8}$O$_8$	0.40	0.340	0.0223	0.940	0.313
Orthoclase	KAlSi$_3$O$_8$	0.35	0.195	0.0 120	0.539	0.179
Anorthoclase	NaAlSi$_3$O$_8$	0.35	0.131	0.0086	0.32	0.120
Quartz	SiO$_2$	0.26	0.250	0.0 164	0.69 1	0.230
Biotite	K(Fe$_{0.7}$Mg$_{0.3}$)$_3$AlSi$_3$O$_{10}$(OH)$_2$	tr.	0.030	0.0079	0.332	0.1 10
Unknown			0.054	0.0035	0.147	0.049
Σ		*OVERLI $\neq$ 1.00* 1.00		*OVERLI $\neq$ 0.0715 OVERLINE3.01*		*OVERLINE 1.00*

[a] Expressed per gram of soil isolate from abundances; calculated from chemical composition of digest.
[b] Calculated from total BET surface area, mineral distribution, and assumption of constant roughness for all minerals.

on this analysis and the minerals identified we calculated mineral abundances. We assigned all the Ca and the required Na to the plagioclase (assuming oligoclase of An,,,). The remaining Na was assigned to anorthoclase. The Fe and Mg was assigned to biotite, along with the required small amount of K. After accounting for the K in biotite the remaining K (almost all) was assigned to orthoclase. The remaining Si, after accounting for the Si in the above minerals, was assumed to be present as quartz. Minor adjustments were made to minimize the absolute difference between the elemental composition of the digest and the calculated mineral distribution. The mineral compositions and quantities calculated in Table 1 result in a 6% deficit in Al and 6% excess of Na as compared to the bulk isolate analysis. These small differences are within the experimental error of the digest procedure. As seen in Table 1, these mineral abundance distributions, based on the chemical composition, are in good agreement with the XRD determinations. We conclude that our sample was 67% feldspar by weight, with 25% quartz and a very minor (3%) amount of biotite.

The measured BET surface area of the starting material was 2.2 $m^2 g^{-1}$. A geometric surface area was calculated, using the assumptions of Swoboda-Colberg and Drever (1993) that the quartz and feldspar can be approximated by spheres and mica by platlets with a diameter/height ratio of 10. The geometric surface area contribution of each mineral (based on the mass distribution calculated from the chemical analysis of the digest) is listed in Table 1. We calculated a surface roughness factor of 31 by dividing the measured BET surface area by the total geometric surface area. This value is comparable to those determined by Anbeek (1993) on much larger material (200-800 μm), indicating that surface roughness is still appreciable for silt-sized material. The relation determined by Anbeek (1993) between particle size and roughness decreased linearly with particle size. When extrapolated to material of 10 and 100 μm in size, the relation predicts surface roughness values of 5.3 and 9.6, respectively, considerably smaller than those measured in this study.

3.1. Weathering studies

Pretreatments to remove trace levels of carbonates or organic matter increased the reaction rates by

about a factor of 2 beyond the first day of reaction, as shown in Fig. 1. Based on the similarity among the results from the peroxide, Na-acetate, and peroxide plus Na-acetate treatments, we conclude that this size fraction of the soil was free of even trace amounts of carbonates and that the solvents used to perform density separations may also have served to remove organic coatings. Extensive prewashings of the mineral fractions was sufficient to remove adsorbed acetate which has been shown to enhance feldspar dissolution rates (Huang and Kiang, 1972). From this experiment we conclude that our treatments resulted in relatively minor changes in reaction rates.

The concentration data shown in Figs. 2 and 3 are based on the experiment where different tubes were sampled with time. The solutions were initially around pH 5, but after the first few samplings pH values were in the range 5-6. The Si concentration data, shown in Fig. 2 for the first 40 days of reaction time, show the characteristic parabolic concentration curves with time. Dissolved species concentrations were below those required for possible solid phase precipitation. There was no grinding, harsh pretreatments or sonification, thus we consider that these data provide evidence that parabolic kinetics are not just an artifact of sample preparation. Na and Ca were the predominant cations released from the solid. Fig. 3 shows the parabolic release of Ca. K, and Mg
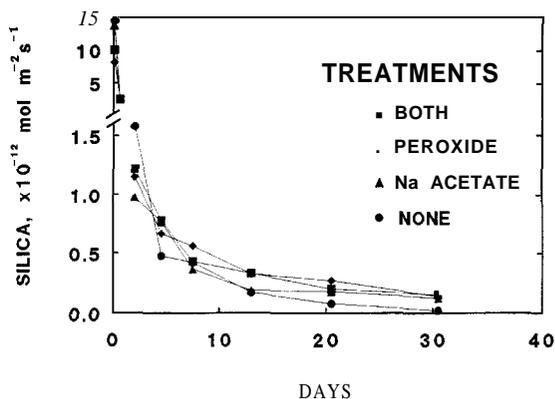
Fig. 1. Silica weathering rates as a function of time for Pachappa soil of IO-100 μm in size with and without pretreatments. The Na-acetate treatment removes carbonates and the peroxide treatment is used to remove organic matter.
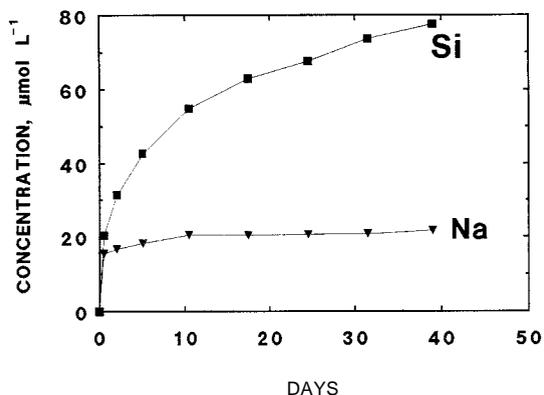
Fig, 2. Silica and Na concentration as a function of time for the plagioclase-K-feldspar isolate.

Fig, 4. Silica and cation dissolution rates as a function of time for the plagioclase-K-feldspar isolate. Samples were taken at selected times from replicates.

with time, for the initial 40 days interval. After 40 days the (Na + Ca + K + Mg)/Si ratio in solution is 0.43, greater than the ratio of 0.216 determined in the solid. If we consider that all the minerals except quartz have a cation/Si ratio ~0.33, then regardless of the relative dissolution rates of the individual minerals, the overall solution cation/Si ratio must be smaller than 0.33 for stoichiometric dissolution to occur. Based on this reasoning we conclude that there was initially preferential leaching of cations relative to Si. Similar results have been observed in numerous earlier studies. The rate data shown in Fig. 4 indicate that the rate decreased throughout the reaction time but that there was still preferential release of cations relative to Si. The final Si dissolution rate was 0.06×10^{-12} mol $m^{-2} s^{-1}$.

Silica release rates for the long-term experiment, presented in Fig. 5, show that the rates decreased initially, and after a reaction time of 40 days, the rate of 0.07×10^{-12} mol $m^{-2} s^{-1}$ was comparable to that found in the first experiment (in which products were allowed to accumulate in solution). After 200 days of reaction the Si rates give an appearance of steady state with a reaction rate of 0.035×10^{-12} mol $m^{-2} s^{-1}$. This is an order of magnitude lower than the rate of 0.35×10^{-12} mol $m^{-2} s^{-1}$ obtained by Anbeek (1993) whose data also appears to have stabilized after ~200 days. It should be noted that this mineral isolate is similar in mineralogy (and

Fig. 3. Calcium. Mg and K concentrations as a function of time for the plagioclase-K-feldspar isolate.
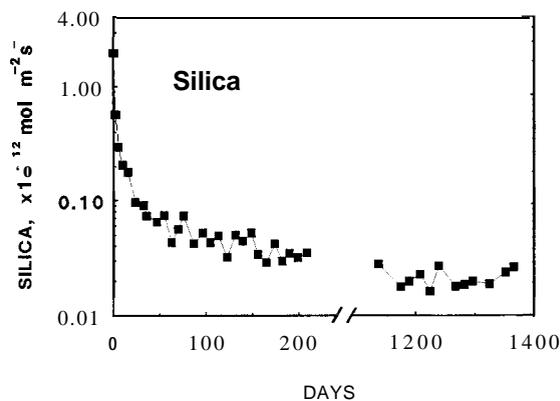
Fig. 5. Silica dissolution rates as a function of time for the plagioclase-K-feldspar isolate. Sampled solution was replenished with DI water to maintain a constant volume. Rates were calculated from the change in mass since the last sampling, after correction for the mass removed during the last sampling.

relative distribution) to that examined by Anbeek (1993).

After reaction of our samples for an additional 3 years the solution was discarded, samples were rinsed in DI water and sampling was resumed. As seen in Fig. 5 dissolution rates decreased further to levels of 0.02×10^{-12} mol m^{-2}s^{-1}. There is no assurance that "steady state" rates have been achieved, nonetheless the change in reaction rate is at least quite low (factor of 1.8 after 3 years). At the conclusion of the experiment the BET of the reacted material had increased to 3.0 m^{2}g^{-1} (despite the loss of 2% of the sample mass). An increase in surface area after reaction has also been observed by others, although it has been attributed to generation of fines caused by fracturing along lattice defect planes as a result of grinding. Increase in surface area cannot be attributed to grinding in our study but the fine-grained material appeared to be feldspar rather than a weathering product (based on lack of detection of any new minerals by XRD). Thus the average Si release rate at the end of the experiment was 0.015×10^{-12} mol m^{-2} s^{-1}, based on the final BET values.

The cation release rates at the end of the experiment were as follows (in mol m^{-2} s^{-1} units, based on the initial BET surface area): Ca, 0.204×10^{-15}; Mg, 0.034×10^{-15}; Na, 3.0×10^{-15}; K, 1.19×10^{-15}. At the end of the experiment the cation/Si ratio was 0.219, in excellent agreement with the digest analysis of 0.216, indicating that preferential leaching of cations was no longer occurring.

Comparison of our Si release rates to those in previous feldspar weathering studies is shown in Table 2. Previous laboratory rates are based almost exclusively on experiments using ground, specimen minerals. Minor differences in pH exist in these experiments but there is a negligible effect of pH on feldspar dissolution in the pH range 4.5-7.5 (Amrhein and Suarez, 1988) so these results can be compared. The Si dissolution rates in this study are initially comparable to published rates of 1.5-4 $\times$ 10^{-12} mol m^{-2}s^{-1}, but after 3.7 years our rates decreased to 0.02×10^{-12} mol m^{-2}s^{-1}. Our release rates are thus about two orders of magnitude lower than most laboratory studies and more than an order of magnitude lower than the data of Anbeek (1993).

The overall Si release rates reported by Swoboda-Colberg and Drever (1993) of 0.24×10^{-12} mol g^{-1}s^{-1} for laboratory dissolution of a quartz-feldspar soil isolate at pH 4.5 was based on reaction with 75-150 μm size material with a calculated geometric surface area of 0.02 m^{2}g^{-1}. This rate corresponds to a rate of 12×10^{-12} mol m^{-2}s^{-1}, with the rate expressed in terms of geometric surface area. This rate is 20 times larger than our overall release rate, when our release rate is expressed in terms of geometric surface area.

The field rates, also shown in Table 2, either need to be corrected by the surface roughness factor, λ, or we need to divide our rates by 31, since field rates were based on geometric surface areas rather than

Table 2

Silica release rates (pmol m^{-2}s^{-1}) from feldspar dissolution

Mineral	Treatment	Rate	Reference
Evje albite	grinding	4	Busenberg and Clemency, 1976
Albite	grinding	3	Nickel, 1973
Amelia albite	grinding (fluidized bed)	15	Chou and Wollast, 1984
Amelia albite	grinding (fluidized bed)	5	Chou and Wollast, 1985
Specimen Na-feldspar	grinding	2	Lagache, 1976
Specimen Hybla albite	grinding	2	Holdren and Speyer, 1985
Orthoclase	grinding	1.7	Busenberg and Clemency, 1976
Microcline	grinding	1.5	Buaenberg and Clemency, 1976
Albite-microcline plagioclase (40%)	mineral and size separation	0.35	Anbeek, 1993
Oligoclase (Bohemian Maasif)	field	0.03	Paces, 1983
Plagioclase (Blue Ridge, N.C.)	field	0.9	Velbel, 1985
Plagioclase-K-feldspar	mineral and size separation	0.02	This study

BET measurements. Expressing our Si release rate in terms of geometric surface area we obtain the rate of 0.62×10^{-12} mol m^{-2}s^{-1}, which is only 30 and 0.6 times greater than the field rates of Paces (1983) and Velbel (1985), respectively.

Mineral dissolution rates, shown in Table 3, were calculated based on the cation release rates, assuming stoichiometric dissolution, and that all Ca (and 4 times as much Na) was dissolved from An,, plagioclase, the remaining Na was released from anorthoclase, 98% of the K was released from K-feldspar, and all the Mg was released from biotite. The calculations are based on the initial surface area and the assumption that the surface roughness factor is the same for all minerals. The contribution of quartz dissolution to the Si release can be neglected, as it is orders of magnitude smaller than the rates determined for feldspars. For example, Bennett et al. (1988) determined that at pH 5 and 25°C the quartz dissolution rate was $10^{-22.06}$ mol m^{-2}s^{-1}. Nonetheless a Si phase dissolution rate can be calculated by subtracting the Si release rate from each of the other minerals from the overall measured Si release rate, assuming stoichiometric dissolution of the other minerals. Based on these assumptions we calculate that 38% of the Si released came from a Si phase. These values result in a dissolution rate of 0.0076×10^{-12} mol m^{-2}s^{-1}, which may be attributed to dissolution of an XRD amorphous Si phase. We conclude that most of the Si release was in fact from feldspar dissolution.

Individual mineral dissolution rates from this study are compared in Table 3 to the laboratory and field study of Swoboda-Colberg and Drever (1993). The values presented are all based on calculated geometric surface areas. Values in parentheses are based on measured BET values for this study and estimated BET values for the data of Swoboda-Colberg and Drever (1993). The relative mineral weathering rates from the field and from both laboratory studies indicate that the biotite weathering rate is slower than the feldspar weathering rate. In addition our laboratory rates suggest that anorthoclase (Na-feldspar) weathers faster than Na-rich plagioclase.

As seen in Table 3 our laboratory rates and the Swoboda-Colberg and Drever (1993) field rates differ only by a factor ranging from 1.2 to 4, with their field rates being slower. These differences are well within the experimental uncertainties, including calculations of mineral surface area in the field experiments. In contrast, there is a large discrepancy between the two laboratory studies, with our rates being up to 300 times slower than those of Swoboda-Colberg and Drever (1993). Among the differences in the experiments, we reacted the materials for longer times, we did not treat our samples by ultrasonification, and our pH values were slightly higher. It is likely that all three factors influenced the reaction rates, but the pH differences would result in only small differences in reaction rates.

4. Conclusions

Our laboratory Si release rates from a plagioclase-K-feldspar mineral isolate, after 3.7 years of reaction time, are in general agreement with data from field studies, within the uncertainties of both experimental measurements and field estimates of reactive surface area. In contrast, previous laboratory rates, determined on either crushed specimen minerals or relatively short reaction times, were orders of magnitude greater than dissolution rates determined from field studies. Exact agreement between labora-

Table 3
Mineral dissolution rates (pmol m^{-2}s^{-1}) [a]

2 13 Mineral	Swoboda-Colberg and Drever (laboratory)	Swoboda-Colberg and Drever (field study)	This study (laboratory)
Plagioclase (oligoclase)	13.6 (0.44)	0.052 (0.0016)	0.102 (0.0033)
K-feldspar (orthoclase)	16.7 (0.54)	0.052 (0.0016)	0.205 (0.0066)
Anorthoclase			0.55 (0.0177)
Biotite	3.8 (0.12)	0.009 (0.0003)	0.01 (0.00036)

[a] Values are expressed in terms of geometric surface area. Values in parentheses are based on BET surface area.

tory rates and field rates is not necessary, considering that field rates are expected to be reduced as a result of non-wetted surface area, and surface coatings, as well as enhanced by microbial activity, and organic ligands.

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