

Dissolution Kinetics of Milled-Silicate Rock Fertilizers in Organic Acid

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ABSTRACT

Dissolution Kinetics of Milled-Silicate Rock Fertilizer in Organic Acid (J. Priyono and R.J. Gilkes): A dissolution experiment was carried out to identify the effects of milling condition on dissolution kinetics of silicate rock fertilizers. Initially ground materials ($\phi < 250 \mu\text{m}$ for basalt, dolerite, gneiss, and $\phi < 150 \mu\text{m}$ for K-feldspar) were further milled with a ball mill (Spex 8000) under dry and wet conditions for 10, 60, and 120 minutes. The rock powders were dissolved in a mixture of 0.01M acetic-citric acid at a rock powder/solvent ratio of 1/1000, and the solution was agitated continuously on a rotary shaker at 25° C. The concentrations of dissolved Na, K, Ca, Mg, Al, and Si from the milled rocks were determined at intervals from 1 hour up to 56 days. Results indicated that the relationships of quantity of dissolved rock and elemental plant nutrients (E_t) with time (t) were well described by a power equation: $E_t = E_0 + at^n$ with reaction order (n) of 0.3 – 0.8. Milling increased quantity of total and individual dissolved element (E_t), dissolution rate (R_t), the proportion of rapidly soluble rock or element (E_0), and dissolution constant a . The increases in dissolution due to dry milling were larger than for wet milling. Although further proves should be provided, results of this dissolution experiment clearly indicates that SRFs may be used as multinutrient fertilizers as well as remedial materials for acidic soils; and dry milling may be applied as an appropriate method for manufacturing effective SRFs.

Keywords: Dissolution kinetics, milling, organic acid silicate rocks

INTRODUCTION

The dissolution of plant nutrient elements from rocks and minerals has been studied for decades through dissolution experiments under laboratory conditions. In many dissolution experiments, the ground solid materials are pre-treated prior to investigation in order to eliminate very fine particles produced by grinding. The pre-treatments include successive washing with deionized water (Huang and Keller, 1970; Barman *et al.*, 1992; Harley, 2002), acid washing (Bloom and Erich, 1987), ultrasonic cleaning (Zhang and Bloom, 1999), and wet sieving (Lundström and Öhman, 1990). These treatments were related to the purposes of the researches which were mostly to identify steady dissolution rate, mechanisms, pathways and processes of primary minerals in solutions, requiring very fine- and amorphous-free solid materials.

Various organic acids are known to occur and involve in numerous reaction processes in the

rhizosphere, including dissolution of nutrient elements from silicate minerals and rocks into soil solution (Jones, 1988). For this reason, organic acids have been used in many dissolution studies. The acids are responsible for a substantial complexation of metal cations, especially for the polyvalent cations such as Si and Al, originating from dissolution of aluminosilicates (Huang and Keller, 1970; Huang and Kiang, 1972; Song and Huang, 1988; Eick *et al.*, 1996) and these acids may produce higher dissolution rates than for dilute inorganic acids or water (Welch and Ullman, 1996; Blake and Walter, 1999; Zhang and Bloom, 1999; Oelkers and Gislason, 2001).

Among organic acids commonly present in soils, oxalic, citric, and acetic acids at concentrations varied from 0.001 to 0.01M were mostly used as solvents for studying the dissolution of silicate minerals and rocks under laboratory and Huang, 1988; Lundström and Öhman, 1990 conditions (Huang and Kiang, 1972; Song; Barman

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et al., 1992; Stillings *et al.*, 1996; Eick *et al.*, 1996; Blake and Walter, 1999; Zhang and Bloom, 1999; Hamer *et al.*, 2003). Huang and Kiang (1972) found that acetic and citric acids (0.01M) are the weakest and strongest respectively among the organic acids they evaluated for dissolving metallic elements from silicate minerals.

We conducted a dissolution experiment as a part of a research on the evaluation for the effectiveness SRFs subjected to high-energy milling. The main objective of the dissolution experiment was to evaluate the effect of milling methods on plant nutrients released from silicate rocks in dilute acetic-citric acid. In this experiment, cleaning rock powders to eliminate fines as mentioned above was avoided as such treatments may cause loss of readily soluble nutrients from the materials and will not be applicable to manufactured SRFs. In stead of using individual acid as the solvent, mixture of the acids is simple and presumed will be more representative for various combinations of main organic acids in soils.

MATERIALS AND METHODS

Samples Preparation

Bulk samples of basalt, dolerite, gneiss, and K-feldspar respectively were from Bunbury,

Jarralidale, Northam, and Port Hedland Western Australia. The first three rocks were washed with H₂O and then broken with a hammer to about 1-cm diameter, ground with a Tema ring mill for 3 minutes, and sieved to pass a 250 µm sieve. The K-feldspar was obtained from Commercial Mineral Co. Perth, Western Australia, and had been ground to $\phi < 150 \mu\text{m}$. These materials were designated as initially milled rocks.

Five grams of initially ground rocks (T0) were further milled with a ball mill (Spex-8000) using a 80 cm³ steel vial and 50 g of $\phi 5 \text{ mm}$ steel balls for 10, 60, and 120 minutes (T10, T60, and T120). For wet milling, 15 ml H₂O was added to the samples prior to milling. The dry-milled samples were transferred to plastic containers, whereas the wet-milled samples were centrifuged for 10 minutes at 11,000 rpm, the supernatant was discharged and the powder was oven dried at 60° C for 48 hours.

Total element of the rocks was determined by XRF (Phillips PW 1400) after fusing the samples with lithium metaborate flux (Karathanasis and Hajek, 1996). The mineralogical compositions of the silicate rocks were identified using XRD (Phillips PW-3020). Results of these analyses are presented in Table 1.

Table 1. Total elemental and mineral compositions of the silicate rocks at their initial condition (T0).

Rock	Element ^a (oxides) %											Total ^f
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃ ^b	CaO	MgO	K ₂ O	Na ₂ O	TiO ₂	MnO	P ₂ O ₅	SO ₃	
Basalt	47.95	19.15	11.25	11.11	6.08	0.18	2.10	1.76	0.16	0.19	0.05	100
Dolerite	49.92	13.94	15.22	11.37	6.32	0.37	1.21	1.89	0.21	0.13	0.12	100
Gneiss	74.01	15.02	2.64	2.01	0.74	1.09	4.06	0.31	0.02	0.08	0.01	100
K-feldspar	61.53	19.05	0.02	0.05	0.01	11.56	2.51	0.05	0.01	0.22	0.01	100
	Mineral Composition^d											
Basalt	: Mg-Hornblende (5 - 6), Pargasite (5 - 6), Augite (5 - 6), Albite (6 - 6.5), Anorthite (6 - 6.5), Biotite (2.5 - 3), Phlogopite (2 - 2.5)											
Dolerite	: Mg-Hornblende, Pargasite, Augite, Albite, Anorthite, Biotite, Diopside (5.5 - 6.5)											
Gneiss	: Quartz (7), Albite, Biotite, Chlorite (2 - 3)											
K-feldspar	: Microcline (6 - 6.5), Albite, Anorthite											

^a Average values of 4 replicates.

^b Total iron is expressed as Fe₂O₃, much Fe is present as Fe²⁺.

^c Normalized to total of 100 % to exclude structural H₂O.

^d Duplicate measurements, and minerals listed in order of decreasing abundance, followed by relative hardness numbers (Deer *et al.*, 1992).

Dissolution Experiment

A mixture of 0.01M acetic acid + 0.01M citric acid at ratio of 1:1 was used as the solvent. Four hundred milligrams of rock powder and 400 ml of solvent were transferred into a 500 ml plastic container, tightly capped, then continuously agitated on a rotary shaker at 130 rpm and an ambient temperature of 25° C. Subsamples of suspension were taken periodically from 1 hour up to 56 days by removing the containers from the shaker, the content was homogenised by hand-shaking for 5 seconds, and immediately 20 ml of suspension was taken by a pipette. The samples of suspension were centrifuged at 4000 rpm for 20 minutes, and about 17 ml of clear solution was transferred by a pipette into a plastic tube. The concentrations of Ca, Mg, and Al in the solution were measured using AAS, Na and K determined using flame emission, and Si using a colorimetric method described by Rayment and Higginson (1992). The pH and EC of solutions were measured with pH and EC meters (Cyberscan 2000). At the end of the dissolution experiment, the remaining rock powder was centrifuged, oven-dried at 60° C (two weeks) and used for analyses of XRD patterns and the quantity of amorphous constituents.

Analysis of Data

The quantity of individual element dissolved was presented as mole charge per unit mass of rock ($\text{cmol}_c \text{ kg}^{-1}$) and % to total of corresponding element in rock. The proportion of rock dissolved was estimated from total quantity ($\text{cmol}_c \text{ kg}^{-1}$) of major elements, i.e., (Na + K + Ca + Mg + Fe + Al + Si) for basalt, dolerite, and gneiss, and (Na + K + Al + Si) for K-feldspar, dissolved relative to the total of these elements in rock. This estimation is similar to the method used by Lundström and Öhman (1990).

The quantity of total element dissolved vs dissolution time was fitted to a modification of the power equation applied by Zhang and Bloom (1999). The modified equation (added E_0) is:

$$E_t = E_0 + \alpha t^n \quad \text{Eq. (1)}$$

where E_t is the quantity of element dissolved (% for total) at time t (days), E_0 is the quantity of rapidly soluble rock which is equal to E at $t = 0$, α is a constant ($\% \text{ d}^{-1}$) that relates to the amount of reactive sites on the surface of rock particles, and n is an experimental constant (reaction order). The

dissolution rate R at time t is calculated from derivative of Eq. (1):

$$R = dE_t/dt = \alpha n t^{n-1} \quad \text{Eq. (2)}$$

The quantities of total dissolved element (E) as function of dissolution time (T) were very well described by Eq. (1) as indicated by $R^2 \approx 1.00$ in most cases and the standard deviation of regression (S_{xy}) was < 1 % relative to sample mean.

RESULTS AND DISCUSSION

Dissolution Kinetics of Milled Rocks

The plots of E_t vs t and R_t vs t for all rock types are quite similar, so only those for basalt and K-feldspar are presented in Figures 1 and 2 as examples. The values of E_0 , α , and n are presented in Table 2.

This dissolution kinetic is basically consistent with results of other dissolution experiments, such as dissolution of muscovite (Lin and Clemency, 1981), yellow and black tuffs (Silber *et al.*, 1999), basaltic glass (Oelkers and Gislason, 2001), lunar basalt (Eick *et al.*, 1996), and silicate minerals (Siever and Woodford, 1979; Harley, 2002). Some of these authors consider that this form of kinetic curve indicates that the initial rapid release of elements attributed to preferential dissolution of superfine fine particles, because this effect is not obtained anymore if caution is taken to remove the smaller particles in order to conduct the dissolution experiments on a restricted range of particle size (Holdren and Berner, 1979), and dissolution mechanisms are ion exchange reactions in which soluble surface cations are exchanged equivalently by H^+ from the acidic solvent. In addition, organic acids (e.g. acetic and citric acids) are capable of extracting mainly polyvalent ions as suggested by Huang and Keller (1970), Welch and Ullman (1996), and Blake and Walter (1999). The following much slower dissolution may be attributed to ion release involving dislocation of structural bonds in the silicate structure (Oelkers and Scott, 2001; Oelkers and Gislason 2001). However, an interpretation of the present results that is different from the above. Acetic-citric acid rapidly dissolved much of the amorphous materials and the structurally disordered/microcrystalline minerals present in intensively milled rocks, followed by slower dissolution of residual crystalline materials.

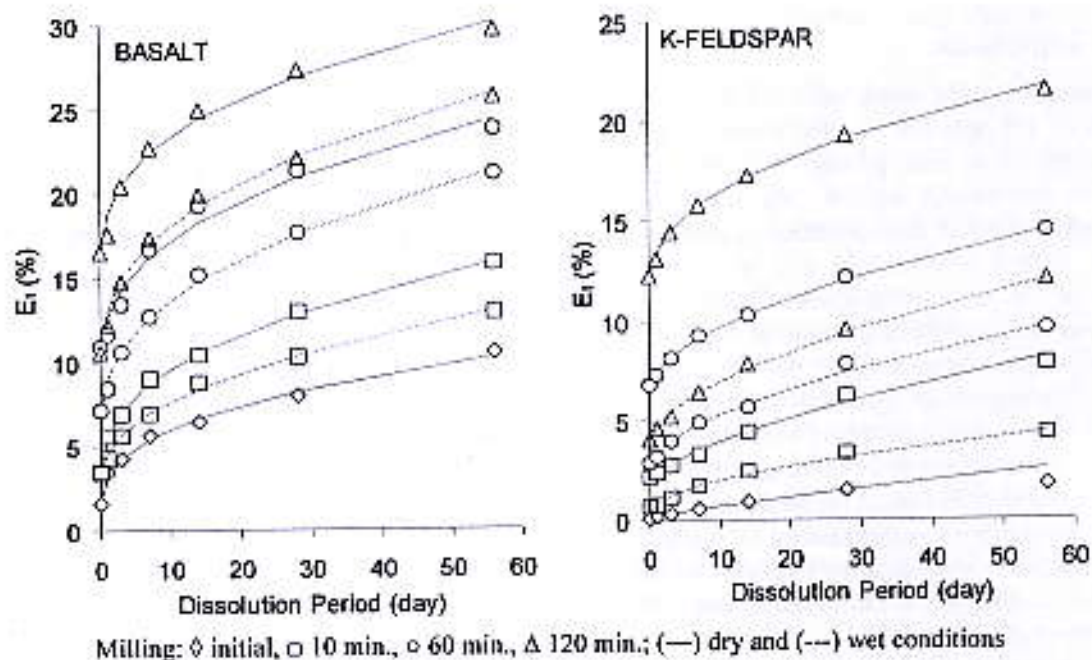


Figure 1. The quantity of dissolved rock powder (E_1), estimated from the total mole charge of major elements (Ca + Mg + Na + K + Fe + Al + Si) for basalt and (Na + K + Al + Si) for K-feldspar dissolved in 0.01M acetic-citric acid as % of the total mole charge of corresponding elements in rock powder, as functions of dissolution time (t). The rocks were milled under dry (full lines) and wet (broken lines) conditions. The lines are statistical fits to Eq. (1) for the corresponding data.

The large increase in solubility of the rocks due to milling is mostly associated with a large difference in the quantity of the rapidly dissolved fraction (E_0) which is considered to be mostly the amorphous and disordered/micro-crystalline silicate phases.

During dissolution of the silicate rocks, the pH of solution increased with increasing milling and dissolution times (Figures 3 – 4). The initial pH of the solvent was about 2.50 and this value increased by about 0.25 units (for initially milled rocks) and between 0.50 and 0.75 units (for 120 minute-milled rocks) after dissolution for 1 hour. At the end of dissolution experiment (56 days), the pH of solution had increased by about 0.75 units (for initially milled rocks) and between 1.0 and 1.2 units (for 120 minute-milled rocks). Conversely, the EC of the initial solvent was about 1.25 mS cm⁻¹ and decreased to about 0.7 mS cm⁻¹ after 56 days of dissolution which may be due to precipitation of salts.

These increases in pH indicate the occurrence of surface exchange reactions as, especially during the early stages of dissolution in which quite large amount of cations were released from the milled

rocks. The rate of dissolution of cations was greatly enhanced by milling. At the same time, the precipitation of amorphous material from solution may have occurred or alternatively dissolved ions may have neutralised the free charges on the functional groups of the organic acids (i.e., dissolution solvent), thereby decreasing the electrical conductivity of the solution. This neutralization process maybe an indication of the occurrence of a ligand-assisted dissolution mechanism as suggested by Shoty and Nesbitt (1992) and Welch and Ullman (1996) that the dissolved polyvalent cations (mainly Al) form metal-organic complexes thereby increase the concentration gradients of these cations within solution – rock particle interfaces, enhancing diffusion rate of these cations from the rock particles to bulk solution. In this present experiment, this dissolution mechanism occurred more intensively for dry- than wet-milled rocks.

In summary, the reduced particle size, the increased effective surface area and abundance of amorphous and disordered constituents due to

Table 2. The values of E_o , a , n , and R^2 for Eq. (3.1) for dissolution of rocks estimated from the total mole charge of major elements (Na, K, Ca, Mg, Fe, Al, and Si) dissolved for basalt, dolerite, and gneiss, (Na, K, Al, and Si) dissolved for K-feldspar relative to the total of these elements in rock.

Rock Type	Milling		E_o (%)	a	n	R^2
	Condition	Time (min)				
Basalt	Dry	0	1.09	2.13	0.36	0.99
		10	2.56	3.04	0.37	1.00
		60	9.16	3.46	0.37	0.98
		120	14.58	4.14	0.33	0.98
	Wet	10	2.86	1.76	0.43	1.00
		60	5.93	3.11	0.40	1.00
120		8.92	4.07	0.35	0.99	
Dolerite	Dry	0	1.12	1.04	0.47	1.00
		10	5.08	2.02	0.50	1.00
		60	13.52	3.40	0.41	1.00
		120	19.44	5.15	0.30	0.99
	Wet	10	2.05	1.71	0.46	1.00
		60	4.83	2.77	0.42	0.99
120		7.17	3.31	0.41	0.99	
Gneiss	Dry	0	0.56	0.45	0.55	0.99
		10	2.87	1.28	0.44	1.00
		60	8.55	0.35	0.71	1.00
		120	15.20	1.36	0.43	0.98
	Wet	10	1.44	0.56	0.57	1.00
		60	5.66	1.28	0.45	1.00
120		8.35	2.31	0.42	0.96	
K-feldspar	Dry	0	0.00	0.19	0.56	0.98
		10	1.29	0.46	0.64	0.99
		60	4.74	0.94	0.54	1.00
		120	8.31	1.57	0.47	1.00
	Wet	10	0.26	0.43	0.55	0.99
		60	1.85	0.62	0.60	0.99
120		2.53	0.87	0.57	1.00	

milling (Priyono *et al.*, 2002) resulted in an increase in quantity of dissolved cations. These changes also resulted in an increase in the dissolution rate (R_d), the proportion of rapidly soluble rock (E_o), and the amount of reactive sites on the surface of particles (a). The changes in dissolution due to milling were greater for dry than for wet milling.

Elemental Dissolution of Plant Nutrients

The above discussion of dissolution relates to the dissolution of the whole rock particles based on the sum of cations dissolved. However, dissolution may not be congruent as several mineral species may be present in a rock and may dissolve at

different rates, additionally some dissolved ions may precipitate so that these laboratory data can not be used directly to predict the release of plant nutrient ions to soil solution. The main objective of this dissolution experiment was to provide an indication of the potential utility of silicate materials as Na, K, Mg or Ca and Mg fertilizers. Therefore, it is necessary to present and discuss the results of dissolution measurements on a single element (plant nutrient) basis. Clearly, the rapid release of nutrients (Table 3) is directly related to the rapidly soluble fraction (E_o) of the individual plant nutrient cations (i.e., Ca, Mg, K, and Na). These values of elemental E_o , calculated using Eq. (1) in units of $\text{cmol}_c \text{ kg}^{-1}$ and % to total in rock

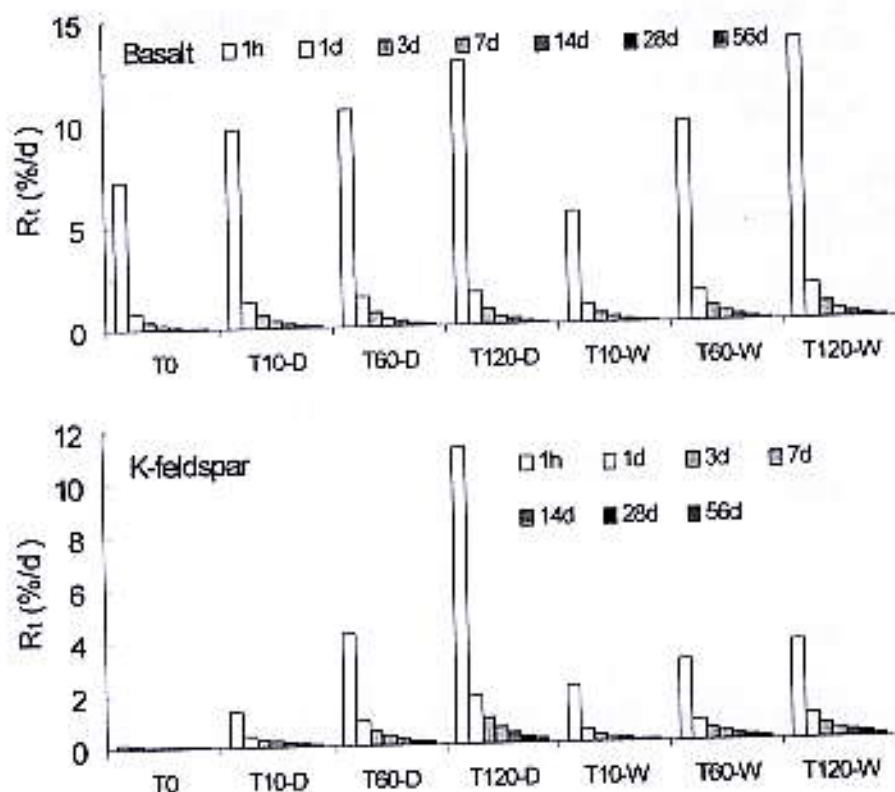


Figure 2. Dissolution rate of rock (R_t) at various contact times estimated from the total mole charge of major elements (Ca + Mg + Na + K + Fe + Al + Si) for basalt and (Na + K + Al + Si) for K-feldspar dissolved in 0.01M acetic-citric acid per day relative to the total mole charge of corresponding elements in rock. The values of R_t were calculated using Eq. (2) for the rocks milled for 0, 10, 60 and 120 min (T0, T10, T60, and T120) under dry (D) and wet (W) conditions.

(Table 3) may be used to estimate the quantity of nutrients made available for plant growth by the rapid dissolution of SRFs in soil.

The proportions of the rapidly soluble plant nutrients (Ca, Mg, K, and Na) (Table 3) were increased by milling and the increases due to dry milling were higher than for wet milling. Milled mafic rocks (basalt and dolerite) rapidly released Na (up to 42% of total Na) and K (up to 73%), which were higher proportions than for felsic rocks (up to 34% Na and 54% K). The total content of K in K-feldspar (12% K_2O , see Table 1) is much higher than for the other silicate materials (4% K_2O for gneiss and < 1% K_2O for basalt and dolerite), so that K-feldspar is potentially an effective K

fertilizer. The milled felsic rock (gneiss) rapidly released 100% of its Mg which was a much higher proportion than for milled mafic rocks (e.g., 27 and 39% respectively for basalt and dolerite). However, gneiss (with total content < 1% MgO) may only supply small quantities of Mg for plant growth, whereas basalt and dolerite (with 6% MgO) are likely to be better Mg fertilizers. The proportion of the rapidly released Ca for milled-mafic and felsic (gneiss) rocks was similar (up to about 16%). Although this proportion of rapidly released Ca is quite low, mafic rocks are likely to be better sources of Ca than felsic rocks. It is notable that these rocks may act as multi-element or compound fertilizers.

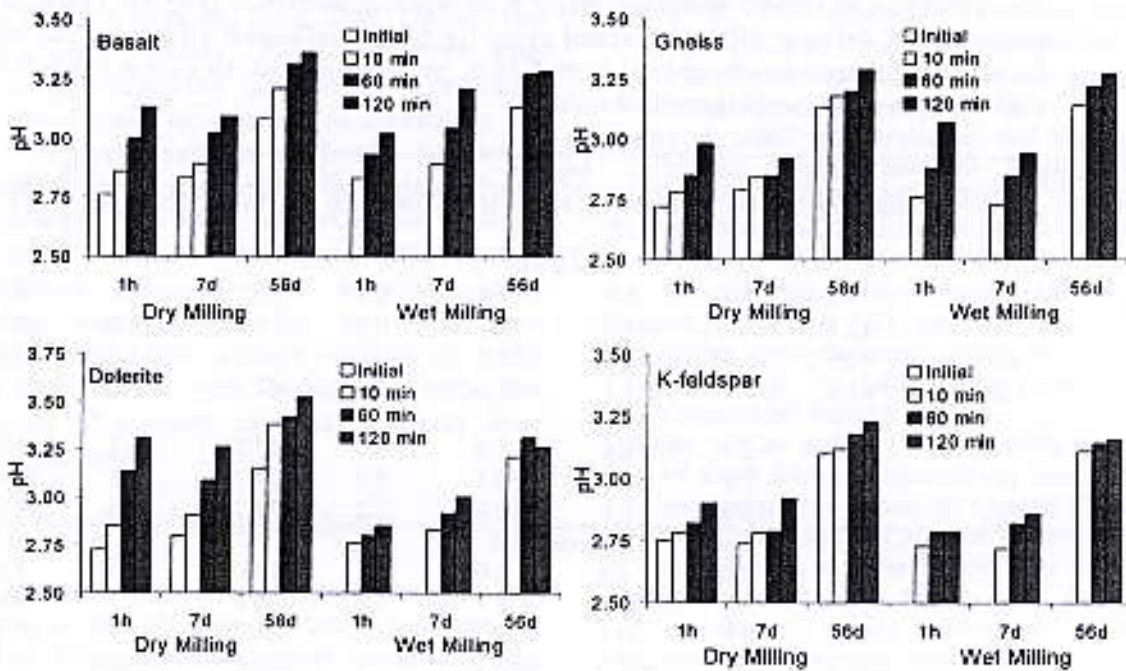


Figure 3. pH of solution after dissolution of silicate rock powders for 1 hour, 7, and 56 days (1h, 7d, and 56d) as affected by milling time of the rocks. The pH of solution before dissolution was 2.50.

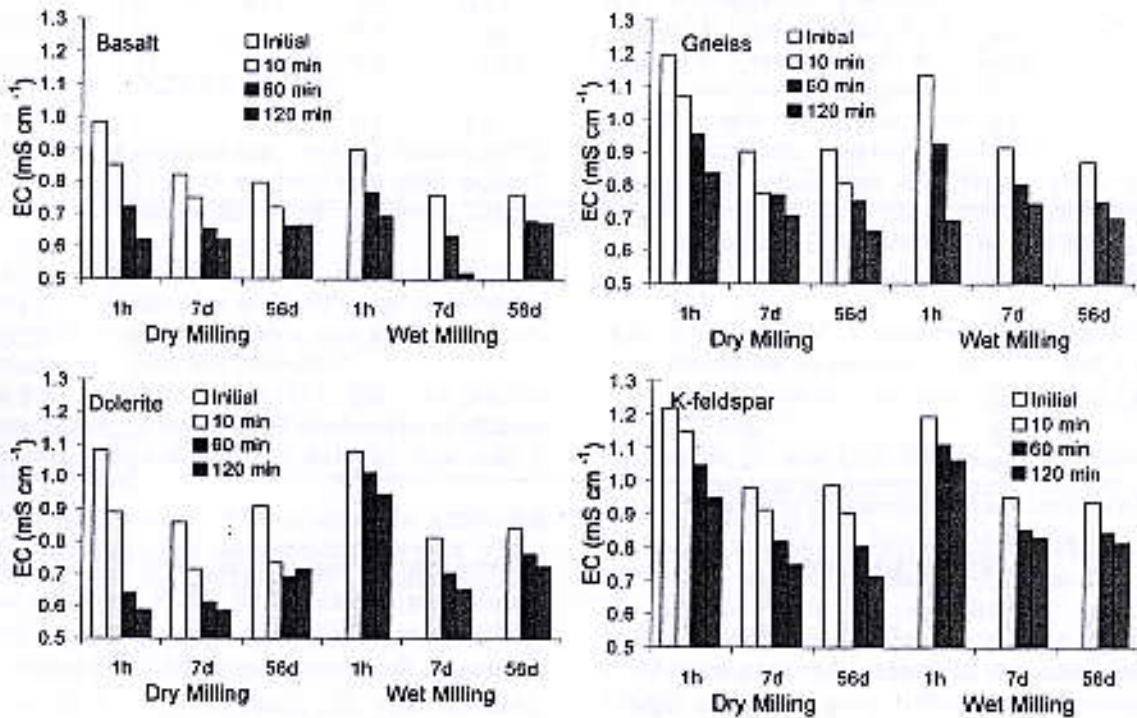


Figure 4. EC of solution after dissolution of silicate rock powders for 1 hour, 7, and 56 days (1h, 7d, and 56d) as affected by milling time of the rocks. The EC of solvent before dissolution was 1.3 mS cm^{-1} .

Table 3. The quantities of rapidly dissolved major plant nutrient elements¹ (E₀) for basalt, dolerite, gneiss, and K-feldspar SRFs, calculated using Eq. (1) for individual elements. The values of E₀ are also presented as % of total in rock (e.g., percentage cmol_c an element dissolved over total cmol_c the element in the milled rock).

Milling		Ca		Mg		K		Na	
Condi- tion	Time (min)	(cmol _c kg ⁻¹)	(%)	(cmol _c kg ⁻¹)	(%)	(cmol _c kg ⁻¹)	(%)	(cmol _c kg ⁻¹)	(%)
Basalt SRF									
Dry	0	9.4	2.4	5.4	1.8	0.8	21.7	2.4	3.5
	10	13.7	3.5	44.0	14.7	0.2	5.0	6.0	8.9
	60	42.6	11.4	62.9	22.2	1.6	43.5	18.2	27.3
	120	61.8	16.1	75.2	26.6	1.5	42.3	27.2	42.1
Wet	10	15.6	3.9	23.4	8.0	0.5	13.2	2.7	4.0
	60	30.4	7.9	28.8	10.1	0.9	24.2	7.8	12.0
	120	40.7	10.9	31.3	10.9	1.2	32.2	9.6	14.0
Dolerite SRF									
Dry	0	0.0	0.0	6.4	2.0	2.3	28.6	0.4	1.0
	10	13.7	3.4	27.4	8.7	3.6	46.8	2.2	5.6
	60	43.1	10.8	74.6	23.7	4.7	61.3	7.9	21.0
	120	89.3	22.8	120.9	38.6	5.4	72.6	16.0	40.4
Wet	10	5.1	1.3	9.5	3.0	2.6	35.2	0.8	1.9
	60	8.0	2.0	16.4	5.2	2.8	38.4	1.6	3.9
	120	7.6	2.0	26.8	8.7	3.3	48.2	3.2	8.2
Gneiss SRF									
Dry	0	2.4	3.3	2.3	6.2	0.9	4.0	0.6	0.5
	10	5.2	7.2	7.0	19.0	3.2	13.6	6.5	5.0
	60	6.8	9.7	36.6	100	9.0	39.6	16.1	12.7
	120	7.9	11.5	36.6	100	12.0	53.8	41.2	33.7
Wet	10	4.7	6.6	3.0	8.1	1.2	5.4	1.2	1.0
	60	5.3	7.4	5.2	14.8	1.1	4.6	6.8	5.4
	120	7.5	11.6	6.2	16.9	3.5	16.8	11.5	10.1
K-feldspar SRF									
Dry	0	-	-	-	-	0.9	0.4	< 0.1	0.1
	10	-	-	-	-	15.4	6.6	3.2	3.8
	60	-	-	-	-	44.9	19.5	9.2	11.7
	120	-	-	-	-	53.3	23.4	16.7	23.6
Wet	10	-	-	-	-	4.5	1.9	0.7	0.9
	60	-	-	-	-	19.7	8.6	4.2	5.5
	120	-	-	-	-	26.3	12.7	8.3	8.9

¹ It is assumed that Na is essential to (some) plants.

CONCLUSIONS

Milling time and condition affected the values of quantity and rate of dissolved cations from SRFs in acetic-citric acid, the proportion of rapidly soluble rock and elemental plant nutrient (E₀), and the amount of reactive sites on the surface of particles (α). These values increased with increasing milling time, and these effects for dry milling were

greater than for wet milling. Data of this dissolution experiment indicate that SRFs may be used as multinutrient fertilizers and liming material. However, those potencies need to be tested in soil-plant systems.

The general trends of rock or elemental dissolution in this present study were consistent with those found by other researchers (Huang and Keller, 1970; Welch and Ullman, 1996; Blake and

Walter, 1999; Oelkers and Scott, 2001; Oelkers and Gislason, 2001; Harley, 2002). However, pre-treatment by removing amorphous constituents from ground rocks or minerals as applied by the above workers results in different dissolution mechanisms. In particular the high initial dissolution rate was not so important for 'cleaned' materials. In this present study, the dilute organic acid mixture dissolved more amorphous than crystalline material, whereas the mechanism suggested by the cited authors involves an initial higher dissolution rate associated with the detachment of outmost structural elements from ordered structures.

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