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High-Energy Milling Improves the Effectiveness of Silicate Rock Fertilizers: A Glasshouse Assessment

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Abstract: The effectiveness of intensively milled gneiss and potassium (K-feldspar) as K fertilizers was evaluated through a glasshouse experiment with ryegrass. Plant were grown on two soil types for 12 months. Results show that the agronomic effectiveness of milled gneiss was nearly as great as potassium sulfate (K_2SO_4), but milled K-feldspar was much less effective because of the relatively small extent of dissolution of K-feldspar in the soil. The positive effects of K-silicate rock fertilizers (K-SRFs) included increases in plant biomass, uptake of K and silicon (Si), and soil pH with increasing application rate of the K-SRFs. The application of K-SRFs will be most advantageous for amending K-deficient soils, and high-energy milling provides a simple method for manufacturing effective multinutrient SRFs.

Keywords: Ameliorant, high-energy milling, liming effects, multinutrients, silicate rock fertilizers

INTRODUCTION

Evaluations of the effectiveness of silicate rock fertilizers (SRFs) have produced conflicting results. For example, applications of SRFs, including

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basalt (de Villiers 1961; Gillman 1980; Barak, Chen, and Singer 1983; Leonardos, Fyfe, and Kronberg 1987; Leonardos, Theodoro, and Assad 2000; Priyono 1991; Gillman, Burkett, and Coventry 2001, 2002; Coventry et al. 2001), gneiss (Wang et al. 2000a, 2000b), granite (Coroneos, Hinsinger, and Gilkes 1996; Hinsinger, Bolland, and Gilkes 1996), and fly ash (Pathan, Aylmore, and Colmer 2003) and pure silicate minerals such as feldspars (Sanz Scovino and Rowell 1988), hornblende, microcline, and biotite (Harley 2003), or micas (Weerasuriya, Pushpakumara, and Cooray 1993) have positively affected soil properties and stimulated plant growth. On the other hand, granite quarry dust provided a minor amount of soluble potassium (K) and reduced wheat yield by up to 65% (Bolland and Baker 2000) relative to standard fertilizers. Bakken, Gautneb, and Myhr (1997) and Bakken et al. (2000) concluded that applied crushed rocks and mine tailings containing biotite, K-feldspar, and nepheline contributed a very small amount of K for the growth of ryegrass. Despite conflicting experimental agronomic results, it appears that the very small and slow release of nutrients from SRFs into soil solution is the main limiting factor (Hinsinger, Bolland, and Gilkes 1996). Efforts should therefore be made to accelerate the release of nutrients from SRFs to soil solution.

Responding to this need, Harley (2003) accelerated the release of plant nutrients from several silicate mineral fertilizers by employing high-energy milling, as did Lim, Gilkes, and McCormick (2003) for rock phosphate fertilizers. This milling method increased the quantities of major plant nutrients dissolved from basalt, dolerite, gneiss, and K-feldspar SRFs in a neutral-salt solution (Priyono, Gilkes, and McCormick 2002) and soils (Priyono and Gilkes 2004). However, it is necessary to evaluate effects of the application of these intensely milled SRFs in the soil–plant system to provide appropriate evidence of their effectiveness for agricultural purposes. Whether the SRFs act only as nutrient sources and/or are general ameliorants of soil conditions remains unclear. Thus, a glasshouse experiment was conducted using several soils and SRFs, and ryegrass (*Lolium multiflorum* cv. Richmond) was grown for 1 year as an indicator plant. The objectives of this research were to (1) identify the effects of the applications of high-energy milled gneiss and K-feldspar as sources of K on the growth of ryegrass and nutrient status for two soils and (2) compare the initial and residual agronomic effectiveness of the SRFs relative to standard (chemical) fertilizers for the test plant nutrient (K).

MATERIALS AND METHODS

Fertilizers and Soils

The silicate rocks used in this experiment were gneiss and K-feldspar, which were used in our earlier chemical experiments (Priyono, Gilkes, and

McCormick 2002; Priyono and Gilkes 2004), and in those publications the properties of the rocks were discussed in detail. The initially milled rocks (Ø < 250 μm for gneiss and Ø < 150 μm for K-feldspar) were further milled using a vertical stirred ball mill for 1 hour under dry condition. This milling time and condition produced maximum amounts of cations [calcium (Ca), magnesium (Mg), K, and sodium (Na)] extractable from the rocks in 1 M ammonium acetate (NH_4Ac), pH 7 (Priyono, Gilkes, and McCormick 2002). The mill is constructed from stainless steel with an effective volume of 7 L. Rock sample (1 kg) and steel balls Ø 0.25″ (23 kg) were loaded and milled at a speed of 1500 rpm for 1 h. The milled gneiss and K-feldspar were used as K fertilizers, and the reference fertilizer was potassium sulfate (K_2SO_4).

Two soils used in this experiment were the top 15 cm, excluding organic layers from Busselton (BSN-1) and Waroona (SCP-11), southwestern Australia [numbers are for Western Australia (WA) reference soil sites (McArthur 1991)]. Soil BSN-1 and SCP-11 were presumed to be K-deficient soils based on the interpretation of values of exchangeable K (see Table 1).

Experimental Design

A glasshouse experiment was prepared to provide the response curves of ryegrass to the application of K-SRFs (milled gneiss and K-feldspar) and a reference fertilizer (K_2SO_4). Three rates of each fertilizer and a control (zero rate) were applied in triplicates for each nutrient and soil. The maximum application rates of SRF were based on the amount of nutrient dissolved in 56 days in 0.01 M acetic–citric acid for the 60-min-dry-milled rocks (i.e., 40 and 20% K, respectively, from gneiss and K-feldspar, unpublished data). The three rates of application of K-SRFs were based on the maximum, 1/2 maximum, and 1/4 maximum of these dissolution values. The rates of application of SRFs based on this assumption are 25, 50, and

Table 1. Several properties[a] of the soils used for the glasshouse experiment

Soil code	Classification[b]	pH (1:5)		EC (μS cm^{-1})	Exch. cations ($cmol_c\ kg^{-1}$)			
		H_2O	$CaCl_2$		Ca	Mg	K	Na
BSN-1	Plinthic Eutrudox	5.7	4.8	57	1.27	0.75	0.78	0.09
SCP-11	Dystric Xeropsamment	6.1	5.0	21	3.84	0.71	1.08	<0.01

[a]For analytical methods, refer to [Priyono and Gilkes (2004)].
[b]Refers to [McArthur (1991)].

100 g SRF kg^{-1} (for milled gneiss) and 5, 10, and 20 g SRF kg^{-1} (for milled K-feldspar). The basal fertilizer (Harley 2003) excluding test nutrients was applied to all pots: 23 mg nitrogen (N), 20 mg phosphorus (P); 40 mg Ca, 2 mg Mg, 7.6 mg sulfur (S), 0.5 mg zinc (Zn), 1 mg copper (Cu), 2.6 mg iron (Fe), 2.6 mg manganese (Mn), 0.06 mg cobalt (Co), 0.08 mg molybdenum (Mo), and 0.12 mg boron (B) per kg soil.

For each pot, 1 kg air-dried soil (<2 mm) in a plastic bag was thoroughly mixed with the appropriate fertilizer, watered to field capacity with deionized (DI) water, and placed in a nondraining plastic pot. The plastic bag was closed to prevent evaporation loss. After a week of equilibration, 15 seeds of ryegrass were sown in each pot, and 10 uniform plant -2ts were grown for 12 months. During the experiment, the pots were randomized each fortnight within treatments for the same soil and were watered daily to maintain the soil water content at about field capacity. Nitrogen solution [ammonium nitrate (NH_4NO_3)] at 1/3 rate of basal N fertilizer was applied fortnightly for all pots, and complete basal fertilizer was applied after each harvest. The plants were harvested four times (H1–H4) at intervals of 3 months by cutting the tops at about 1 cm above the soil surface. After the end of experiment (12 months), a subsample of soil was taken from each pot (i.e., residual soil), and samples for the three replicates were combined and analyzed for amounts of NH_4Ac-extractable K, pH, and EC.

Analytical Methods

Subsamples of finely ground plant tops were analyzed for nutrient content [i.e., Ca, Mg, K, Na, Cu, Zn, Mn, Fe, silicon (Si), P, S, and chloride (Cl)] using x-ray fluorescence (XRF) (Phillips 1400) after pressing the sample into a boric acid backing at about 350 bar. For measurement of the amount of NH_4Ac-extractable K of residual soils, 5 g soil were transferred to 25 mL of 1 M NH_4Ac in a 50-mL plastic vial, shaken on an end-over-end shaker for 1 h, and filtered, and the concentrations of K in the filtrate were determined using flame emission. The pH_{H_2O} of the residual soils at a soil–water ratio of 1:5 was measured using a pH meter (Cyberscan 2000, Eutech Cybernetics, Singapore).

Analysis of Data

The agronomic effectiveness (AE) of SRF relative to reference fertilizer was calculated using the values of cumulative uptake of the nutrient concerned (Mackay, Syers, and Gregg 1984). The relative agronomic effectiveness (RAE) of SRF was defined as the ratio of the effectiveness of SRF over that for the reference fertilizer. The RAE values for K fertilizers were calculated based on the best-fit equations describing response curves of cumulative K uptake as a function of the application rate of K fertilizers. The best-fit equations for the response curves of K_2SO_4 (reference fertilizer) were linear, whereas those for K-SRFs were quadratic. If the rate for the K_2SO_4 had been extended to much higher values than 90 mg K kg^{-1}, K uptake data would also have confirmed the quadratic equation with maximum K being achieved at a much higher rate than 90 mg K kg^{-1}. Consequently, the maximum K uptake for the $+K_2SO_4$ treatment cannot be defined based on the response curves obtained in this present research. Therefore, the RAE values for the K-SRFs were defined as the ratio of the slopes for the application rate of 90 mg K as K-SRF kg^{-1} (i.e., the highest rate of K for the $+K_2SO_4$ treatment).

RESULTS AND DISCUSSION

Plant Growth and Nutrient Uptake

The plot of cumulative plant yield and K uptake versus the application rate of K fertilizers are presented in Figures 1 and 2. Application of all K fertilizers greatly affected plant yield and K uptake for both soils.

Ryegrass grew well on soils BSN-1 and SCP-11 for the first three months (H1). However, K-deficiency symptoms appeared after the first cut for the $+K_2SO_4$ treatment associated with low concentrations of K in dry tops (i.e., $<1\%$). For the +K-SRF treatments, values of K uptake for the first cut were two- to four-fold larger than those for the $+K_2SO_4$ treatment. However, these high values of K uptake for the +K-SRF treatments were not associated with large increases in yield as all the plants were K sufficient.

As shown in Figure 1, for the first cuts there were only small increases in yield for all K fertilizers. There were large increases in K uptake with increasing application rate of all K fertilizers, and for K_2SO_4 where about 50% of added K was utilized by the plants. By the second harvest for $+K_2SO_4$, most K had been utilized by plants so that there was little additional K uptake by subsequent plant growths. This was not the case for the two K-SRFs where additional K was utilized by plants for up to 12 months (H4). In summary, application of K-SRFs increased plant yield and K uptake for ryegrass grown on soils BSN-1 and SCP-11 that were deficient in K (i.e., exchangeable K values were 0.78 and 1.08 $cmol_c\ kg^{-1}$, respectively; see Table 1).

Concentration of Plant Nutrients in Dry Tops

The application of SRFs may greatly affect concentrations of the major nutrient applied (K) but may also affect the concentrations of nontreatment

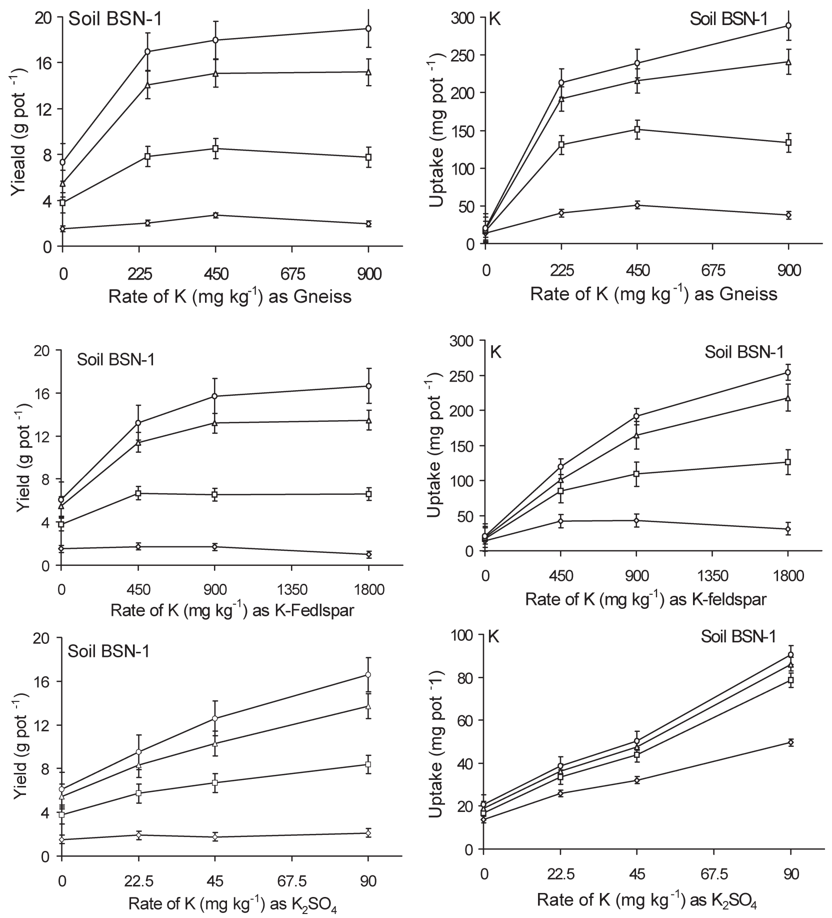

Figure 1. Effects of the application of K as gneiss, K-feldspar, and K_2SO_4 on cumulative yield (oven-dried plant tops) and cumulative K uptake for the first, second, third, and fourth harvests (H1, H2, H3, and H4) of ryegrass grown on soil BSN-1. Error bars are the standard error of mean.

nutrients (Mg, Na, Cu, Zn, Mn, Fe, Si, P, S, and Cl) in plants, causing plant nutrient disorders (deficiency or toxicity). There were two significant points interpreted from very large data set relating to the general nutrition status of the plants (data are not shown), as follows:

1. The concentration of K in dry tops of increased with increasing application rate of K fertilizers. For the $+K_2SO_4$ treatment, this effect occurred only for plant tops from H1 and H2, whereas for the +SRF treatments, it occurred for H1–H4. Concentrations of other

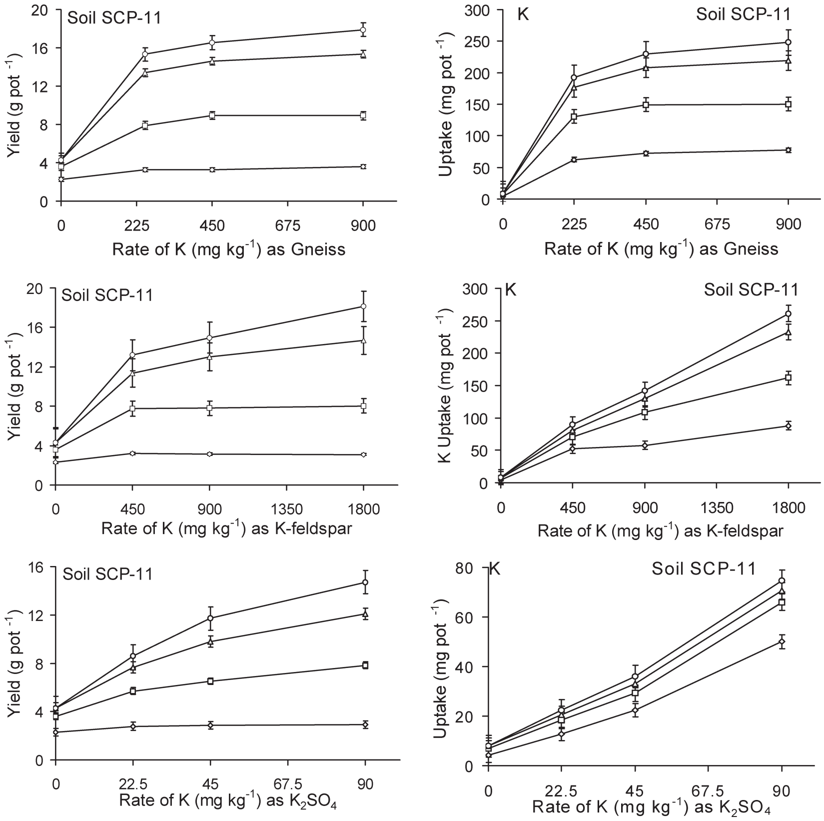

Figure 2. Effects of the application of K as, gneiss, K-feldspar, and K_2SO_4 on cumulative yield (oven-dried plant tops) and cumulative K uptake for the first, second, third, and fourth harvests (H1, H2, H3, and H4) of ryegrass grown on soil SCP-11. Error bars are the standard error of mean.

nutrients (i.e., Ca, Mg, P, Cl, Cu, and Zn) in dry tops for most harvests decreased with increasing application rate of all K fertilizers. Antagonistic interactions of Ca and Mg with K have been reviewed by Munson (1968) and Marschner (1986) in which high concentrations of K inhibit the uptake and physiological availability of Ca and Mg for plants, and these mechanisms may have operated in the present experiment.

2. The concentration of Si in dry tops of plants increased with increasing application rate of K-SRFs due to dissolution of the K-SRFs. Large amounts of plant-available Si in soil may stimulate plant growth and the quality of plant products (Epstein 1999), reduce toxicity effects of Mn (Marschner 1986) for several plant species, reduce Al-induced inhibition of corn roots (Ma, Sasaki, and Matsumoto 1997; Corrales,

Table 2. The best-fit equations describing relationships among cumulative uptake of K (Y, mg K kg^{-1}), application rate of K fertilizers (X, mg K kg^{-1}), and RAE values

K-Fertilizers	Harvest	Equation	RAE (%)
Soil BSN-1			
K_2SO_4	H1	$Y = 15.1 + 0.39X$ ($R^2 = 0.99$)	100
	H2	$Y = 16.6 + 0.68X$ ($R^2 = 1.00$)	100
	H3	$Y = 18.2 + 0.74\ X$ ($R^2 = 0.99$)	100
	H4	$Y = 19.9 + 0.77\ X$ ($R^2 = 1.00$)	100
Gneiss	H1	$Y = 14.1 + 0.14X - 129\ 10^{-5}\ X^2$ ($R^2 = 1.00$)	43
	H2	$Y = 20.3 + 0.50X - 425\ 10^{-5}\ X^2$ ($R^2 = 0.96$)	86
	H3	$Y = 30.4 + 0.70X - 519\ 10^{-5}\ X^2$ ($R^2 = 0.95$)	107
	H4	$Y = 34.3 + 0.75X - 526\ 10^{-5}\ X^2$ ($R^2 = 1.00$)	110
K-feldspar	H1	$Y = 15.5 + 0.06X - 289\ 10^{-5}\ X^2$ ($R^2 = 0.92$)	17
	H2	$Y = 19.8 + 0.15X - 521\ 10^{-5}\ X^2$ ($R^2 = 0.99$)	24
	H3	$Y = 18.3 + 0.21X - 558\ 10^{-5}\ X^2$ ($R^2 = 1.00$)	30
	H4	$Y = 20.1 + 0.25X - 666\ 10^{-5}\ X^2$ ($R^2 = 1.00$)	34
Soil SCP-11			
K_2SO_4	H1	$Y = 2.1 + 0.52X$ ($R^2 = 0.99$)	100
	H2	$Y = 4.1 + 0.66\ X$ ($R^2 = 0.99$)	100
	H3	$Y = 5.4 + 0.70\ X$ ($R^2 = 0.99$)	100
	H4	$Y = 5.9 + 0.74X$ ($R^2 = 0.99$)	100
Gneiss	H1	$Y = 7.8 + 0.24X - 185\ 10^{-5}\ X^2$ ($R^2 = 0.95$)	53
	H2	$Y = 14.5 + 0.52X - 412\ 10^{-5}\ X^2$ ($R^2 = 0.95$)	90
	H3	$Y = 17.8 + 0.71X - 548\ 10^{-5}\ X^2$ ($R^2 = 0.96$)	116
	H4	$Y = 18.4 + 0.78X - 588\ 10^{-5}\ X^2$ ($R^2 = 0.96$)	119
K-feldspar	H1	$Y = 8.2 + 0.08X - 225\ 10^{-5}\ X^2$ ($R^2 = 0.95$)	17
	H2	$Y = 8.3 + 0.14\ X - 327\ 10^{-5}\ X^2$ ($R^2 = 0.99$)	23
	H3	$Y = 9.9 + 0.15\ X - 158\ 10^{-5}\ X^2$ ($R^2 = 1.00$)	22
	H4	$Y = 10.6 + 0.17\ X - 150\ 10^{-5}\ X^2$ ($R^2 = 1.00$)	23

The RAE value is defined as the ratio of slope of the equation for SRF relative to that for K_2SO_4 at the application rate (X) of 90 mg total K kg^{-1}.

Poschenrieder, and Barceló 1997), and increase the resistance of plants to the attack of several pathogens (Volk, Kahn, and Weintraub 1958). Coventry et al. (2001) associated the beneficial effects of plant-available Si from application of Minplus^{TM} (crushed basalt) to the reduction of Al toxicity in acidic soils. These effects indicate an advantage for the use of SRFs rather than conventional fertilizers for plants that require Si (e.g., grasses).

In summary, application of K-SRFs increased concentrations of K for soils that were K deficient. Plant response was sometimes associated with either a decrease or increase in the concentration of other nutrients, which extended to nominally deficient or toxic levels for some treatments.

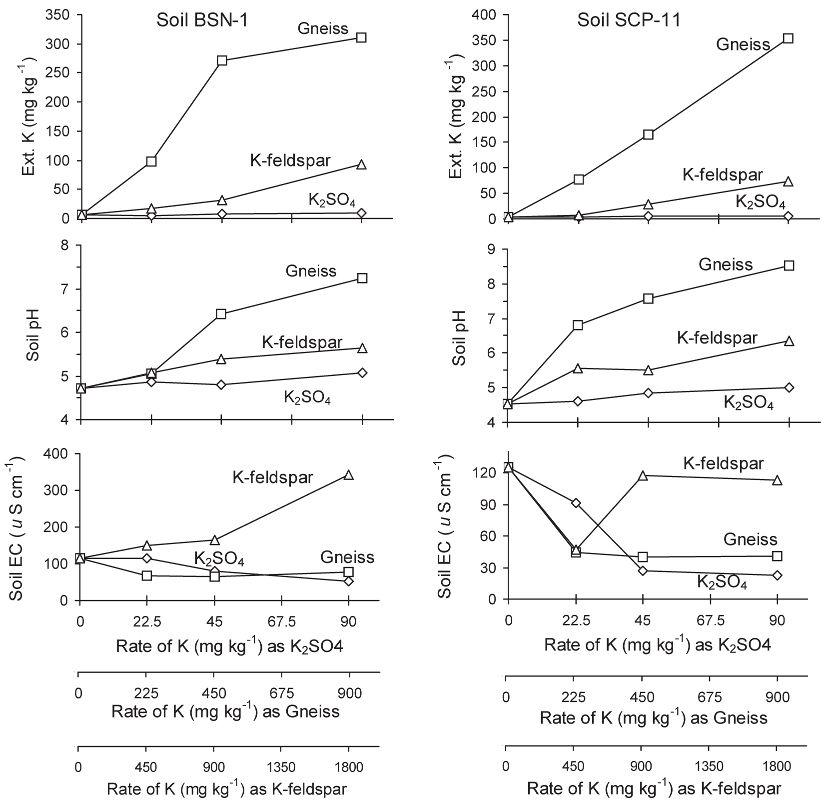

Figure 3. Amount of NH_4Act-extractable, K, pH, and EC of soils BSN-1 and SCP-11 after the fourth harvest as affected by application rate of K fertilizers.

Relative Agronomic Effectiveness (RAE) of SRF

The best-fit equations for cumulative K uptake versus application rate of K fertilizers together with values of RAE are presented in Table 2. Gneiss SRF was about half as effective as K_2SO_4 for growing ryegrass up to 6 months (H1–H2), but was slightly more effective for plants harvested at 9 and 12 months (H3–H4). This interpretation should be treated with some caution as the plants supplied with K_2SO_4 had removed most ($\approx 3/4$) of applied K from soil by H2. K feldspar SRF was much less effective than K_2SO_4 for all growth periods.

Extractable K, pH, and EC of Residual Soils

The effects of K-SRF application on changes in NH_4Act-extractable K, pH, and EC of soils sampled after the experiment are presented in Figure 3.

This evaluation was aimed to identify whether the application of K-SRFs has long-term beneficial effects.

The application of K as gneiss SRF greatly increased the amounts of extractable K in both soils; there were smaller increases for K_2SO_4 and K-feldspar. The application of these K-SRFs greatly increased soil pH, whereas the application of K as K_2SO_4 did not affect soil pH. Soil pH increased by about 2.5 and 4 respectively for soils BSN-1 and SCP-11 receiving gneiss SRF. The increase of soil pH due to the K-feldspar SRF was about 1.5 for both soils. However, the efficiency of K-feldspar SRF as a liming material (ratio of increase of soil pH/application rate for unit mass of the milled rock) was two to five times higher than that of gneiss SRF.

For the experiment using soil BSN-1, the application of K as K-feldspar SRF increased soil EC by about 375 $\mu S\ cm^{-1}$, but there was no effect for gneiss SRF and K_2SO_4. The plants for the no-K treatment for soil SCP-11 did not survive after the third harvest, so that the basal fertilizer salts added to the soil after the third harvest remained in the soil, causing a higher EC value (i.e., about 120 $\mu S\ cm^{-1}$) relative to initial EC value (i.e., 21 $\mu S\ cm^{-1}$, see Table 1) for soil SCP-11. In summary, the trends of EC values versus the application rate of K fertilizers for soil SCP-11 seem to be similar to those for soil BSN-1 as described before.

CONCLUSIONS

The application of gneiss and K-feldspar SRFs to the K-deficient soils increased plant yield, K concentration and K uptake of plant tops, amounts of NH_4Ac-extractable K, and soil pH. The agronomic effectiveness of gneiss milled for 1 h was nearly as great as K_2SO_4, indicating that gneiss could be used as a potassium SRF. On the other hand, the milled K-feldspar was much less effective, mostly because of the relatively low dissolution of K-feldspar in the soil.

Results of this experiment indicate that all SRFs tested were suitable for use as soil ameliorants. In addition to being sources of plant nutrient elements, the SRFs are quite effective liming materials. These results are consistent with other findings: Sanz Scovino and Rowell (1988) used gneiss dust, and Bakken et al. (2000) used several K-bearing minerals as K fertilizers. In summary, it appears that application of SRFs will be most advantageous for amending strongly acidic and nutritionally impoverished soils.

Milling greatly improved the agronomic effectiveness of SRFs in supplying K relative to values reported for other pot experiments (Coroneos, Hilsinger, and Gilkes 1996; Hinsinger, Bolland, and Gilkes 1996) for field experiments with granite dust (Bolland and Baker 2000), mine tailings (Bakken, Gautneb, and Myhr 1997; Bakken et al. 2000), and feldspars (Sanz Scovino and Rowell 1988). In these published experiments, the sources of K were relatively coarse particles. Clearly, high-energy milling provides a simple method for manufacturing effective SRFs. However, further research to identify optimum milling times on an industrial scale is needed. A cost–benefit evaluation for manufacturing SRF is a major requirement prior to establishing a SRF industry. The economic effectiveness of SRF also needs to be assessed on the basis of SRF being a multinutrient fertilizer and liming agent.

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