# **The potential of crushed rocks and mine tailings as slow-releasing K fertilizers assessed by intensive cropping with Italian ryegrass in different soil types**

Anne Kjersti Bakken<sup>1</sup>, Håvard Gautneb<sup>2</sup> & Kristen Myhr<sup>1</sup>

<sup>1</sup> The Norwegian Crop Research Institute, Kvithamar Research Centre, N-7500 Stjørdal, Norway; <sup>2</sup> Geological *Survey of Norway, P O. Box 3006, N-7002 Trondheim, Norway* 

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# **Abstract**

In search for a source for a slow-releasing K fertilizer, the plant availability of mineral K in selected crushed rocks and mine tailings was investigated by growing Italian ryegrass for six months in small volumes of peat, loamy sand or silt loam mixed with different K-sources. The K supplied as K-feldspar was nearly unavailable to the plants, whereas nearly 60% of the K supplied as biotite and nepheline in a carbonatite, was recovered in harvested plants parts. The carbonate content of the rocks and tailings seemed to be more important for the availability of the K than the specific surface of the mineral particles. It is concluded that a rock-based fertilizer containing biotite as its main K-bearing mineral and between 5 and 20% carbonate, will release K at a slower rate than soluble K fertilizers do and still supply considerably more K to the plants than is supplied from the fraction of non-exchangeable K in the soil.

## **Introduction**

There are increasing interest and demand for slowreleasing K-sources both in conventional and organic farming. The application of high loads of soluble K fertilizers one or several times during the growing season might antagonize plant uptake of other basic cations, such as Mg and Ca until the pool of highly soluble K is depleted (Jakobsen, 1993). Herbage cut or grazed shortly after being supplied with high amounts of soluble K might consequently be high in K and low in Ca and Mg and predispose ruminant animals to grass tetany (Kemp and Hart, 1957). In grasslands with frequent cuttings or grazing, the soluble K applied in spring might soon be depleted, and later plant regrowth depends on the release of native K from soils. Although the loss of K by leaching and run off from agricultural land is not regarded as an environmental problem it might be of economic significance and importance in some soil types (Johnston and Goulding, 1990; Yläranta et al., 1996). A fertilizer releasing K gradually and in closer agreement with plant demand than the mineral salts do, would for the above mentioned reasons be favourable.

Crushed rocks and minerals have been tested as plant K fertilizers in several field and pot experiments (Berthelin and Leyval, 1982; Goldschmidt and Johnsson, 1992; Hinsinger and Jaillard, 1993; Sanz Scovinio and Rowell, 1988; Weerasuriya et al., 1993; Wentworth and Rossi, 1972; Yli-Halla, 1992). Most of the investigations showed that plants are able to release significant amounts of the K bound in minerals such as biotite, phlogopite, muscovite and nepheline, whereas K supplied as K-feldspar seems to be nearly unavailable to plants without further processing. The rate of weathering and dissolution reactions releasing K from the added mineral particles are influenced by soil pH, temperature, moisture and biological activity and by the reactive surface of the mineral particles (Lasaga, 1984; Lasaga, 1995; Sparks and Huang, 1985). The plant availability of the K supplied as crushed rocks and minerals might consequently vary between different soil types and environments.

The rate and amount of native non-exchangeable K released to plants from soils depend on its mineralogy and the stage of weathering (Johnston and Goulding, 1990; Sparks and Huang, 1985). Measurements of the amount of K taken up by plants is considered to be a reliable method for assessing the availability of this fraction (Badraoui et al., 1992; Binet et al., 1984; Mengel and Rahmatullah, 1994; Møberg and Nielsen, 1983). To evaluate the potential of crushed rocks and minerals as K fertilizers their K-releasing capacity needs to be compared with the K-releasing capacity of the non-exchangeable fraction of K in different soils. To be useful sources for K on a short term basis, their K should be at least as plant available as the native K in the soil.

By intensive cropping with Italian ryegrass we have compared the K-releasing capacity of different crushed rocks and mine tailings either regarded to be candidates for, or already marketed as fertilizer products. The objective was to investigate how the plant availability of mineral bound K varied with specific surface and mineralogical composition of the rocks and tailings, within and between soil types differing in particle size and native non-exchangeable K content.

## **Materials and methods**

#### *Rocks and minerals*

The rocks and minerals tested in the experiments are listed in Table 1. The carbonatite is from the Lillebukt Alkaline Complex at Stjernøy in Norway (Mjelde, 1983; Strand, 1981). Lurgi and FilterlI are tailings from the nepheline-syenite production (North Cape Minerals) located at Stjernøy. The epidote schists is from a quarry at Inderøy in Norway, and the commercial product Adularia is from the fucoid beds near Ullapool in Scotland.

Whole rocks were crushed in a jaw crusher and a ball mill. Crushed rocks, Adularia and tailings were sieved through 0.59 mm. Tailings Lurgi was further milled by a ball mill several times to obtain batches with different specific surfaces. The total mineralogical and chemical composition (Tables 1 and 2) was analysed by X-ray diffractometry (XRD) and X-ray fluorescence spectrometry (XRF), and the content of acid soluble K by ICP-spectrometry after digestion in  $7 N HNO<sub>3</sub>$  for 30 min at 120 $\degree$  C, at the Geological Survey of Norway. The specific surface of the crushed and sieved rocks and minerals was analysed by the BET-

method (Brown - Emmet- Teller) at SINTEF Rock and Mineral Engineering, Trondheim, Norway.

#### *Growth experiments*

In two separate experiments *Lolium multiflorum italicum* var. Turilo was grown in peat moss (Floralux, Nittedal Torvindustrier A/S, Norway, mixed with 6 kg  $m^{-3}$  of a 1:3 mixture of dolomite and limestone), loamy sand or silt loam in perforated polyethylene boxes  $(56.5 \text{ cm} \times 36.5 \text{ cm} \times 17.0 \text{ cm})$  with a non-woven cloth lining. Specifications of the loamy sand and silt loam are given in Table 3. The pregerminated seedlings (35 per box) were grown for 6 months (Experiment 1 : 18 April - I November in 1994 and Experiment 2:20 February - 21 August in 1995) in an acrylic greenhouse at Kvithamar Research Centre (63°30'N). The natural daylight was supplemented with light from highpressure sodium lamps (200  $\mu$ mol m<sup>-2</sup> s<sup>-1</sup>at plant height, 16 h photoperiod). The plants were supplied with tap water containing 0.2 mg K per litre. Drainage water was collected continuously from each box separately. Day and night temperature were  $16^\circ$  and  $13^\circ$  $\rm{^{\circ}C}$ , with mid-day maxima between 20 $\rm{^{\circ}}$  and 30 $\rm{^{\circ}C}$  in summer.

At the start of the experiments 20 g  $Ca(NO<sub>3</sub>)<sub>2</sub>$ . 4  $H_2O$ , 8 g NH<sub>4</sub>NO<sub>3</sub>, 6 g MgSO<sub>4</sub>. 7 H<sub>2</sub>O, 22 g Superphosphate (9% P) (Hydro Agri Norway) and 4 g Fritted Trace Elements 36 (Ferro Holland BV) were added to the growth medium in each box. On the basis of the total content of K as analysed by  $XRF$  (Table 1), K (25 g per box) was supplied from different crushed rocks/minerals or from KC1. In the first experiment, the rocks/minerals Adularia, tailings FilterlI and carbonatite were evaluated and in the second experiment, tailings Lurgi at three different specific surfaces, carbonatite and epidote schists were evaluated. Treatments without K fertilizer were also included. Potassium was added to the growth medium only at the start of the experiments, whereas other macro and micro nutrients were resupplied regularily. After 4, 8, 12, 14, 16, 18, 20 and 22 weeks of growth,  $1.4$  g NO<sub>3</sub>-N,  $0.1$  g NH<sub>4</sub>-N, 1.8 g Ca, 0.3 g P, 0.3 g Mg, 0.2 g S, 0.6 mg Zn, 0.7 mg B, 4.0 mg Fe, 2.3 mg Mn, 0.1 mg Cu and 0.05 mg Mo dissolved in water were added to each box.

The plants were cut at intervals of four weeks (altogether 6 times). The stubble height was 4 cm at harvests 1 - 5 and 1 cm at the last harvest. The harvested material was dried at 60 °C and the yield at each harvest recorded. Herbage samples bulked for each two harvests  $(1+2, 3+4, 5+6)$  were analysed for total K.

Rock/mineral type type	Chemical composition as analysed by XRF $(\%$ , w/w)									Calcite content	Acid soluble K	Relative contribution from different minerals to total K surface	Specific	
	Si	Al	Fe	Ti.		Mg Ca Na K Mn P					LOI $(\%$ , w/w) $(\%$ of total) $(\%$ of total)			$(m^2g^{-1})$
Adularia	20.4	5.9			2.6 0.2 3.8				6.8 0.1 6.4 0.1 0.2	16.440		10	$Ksp(80\%)$ , Bt $(10\%)$ , Glt $(10\%)$	2.80
<b>Tailings FilterII</b>		24.2 11.9			$1.4 \; 0.5 \; 0.3$			3.1 5.5 6.3 0.1 0.1		$2.3 \quad 2$		25	$Ksp(77%)$ , Ne (20%), Bt (3%)	0.59
Carbonatite	7.9		3.9 10.9 1.7 1.6 19.4 0.2 3.2 0.2 1.1							20.7 40		95	$Ksp(1\%)$ , Ne (18%), Bt(80%)	1.38
Tailings Lurgi 1	22.3	9.9			5.8 1.2 1.4			5.2 4.4 4.6 0.2 0.1			$1.0 \pm 3$	26	$Ksp(41\%)$ , Bt $(40\%)$ ,	0.15
Lurgi 2												30	Ne $(14\%)$ , HbI $(5\%)$	0.69
Lurgi 3												33		1.47
Epidote schists	28.5	7.6	3.8 0.1 1.9						$3.6$ 2.0 2.2 0.1 < 0.1	4.7 10		33	Bt $(62\%)$ , Ms $(32\%)$ , $Hbl(4\%)$ , Pl $(2\%)$	0.68

*Table 1.* Composition and specific surface of crushed rocks and mine tailings tested as K-sources for Italian ryegrass. Mineral abbreviations (Kretz, 1983): Ksp- K-feldspar, Ms- muscovite, PI - plagioclase, Bt - biotite, GIt - glauconite, Ne - nepheline, Hbl - hornblende. Acid soluble K: K soluble in  $7 \text{ N HNO}_3$ . LOI: Loss on ignition

*Table 2.* The content of some selected trace elements in crushed rocks and mine tailings tested as K-sources for Italian ryegrass

Rock/mineral type	Content of trace elements as analysed by XRF											
	Сu	Zn	Ph	Ni	Co	v	Mo	Cd	Сr			
	(ppm, w/w)											
Adularia	12	14	$\leq 10$	31	< 10	71	<5	< 10	49			
<b>Tailings FilterII</b>	<5	18	< 10	8	< 10	38	$\leq$	< 10	$\leq$			
Carbonatite	16	106	72	5	19	53	<5	$\leq 10$	5>			
<b>Tailings Lurgi</b>	9	77	51	19	< 10	119	-5	< 10	37			
Epidote schists	31	70	14	79	16	111	<5	$\leq 10$	178			

After the last harvest, the weight of the growth medium (containing plant roots and stubble) in each box was recorded. Samples of this mixture of plant remainings and growth medium were dried at 35 °C and the content of exchangeable and total K and pH analysed. The collected drainage water was bulked together for intervals of 8 weeks, the total volume recorded and the content of total K in the water analysed.

#### *Soil, plant and water analyses*

The particle size and the mineralogical and total chemical composition of unfertilized samples of the loamy sand and silt loam was analysed at the Geological Survey of Norway. The particle size was determined by an automatic analyzer, and the mineralogical and chemical composition of the sand fraction ( $>63 \mu m$ ), silt fraction (2 - 63  $\mu$ m) and clay fraction (<2  $\mu$ m) by XRD and XRF after sieving and sedimentation in water. The amounts of the minerals identified by XRD were determined semiquantitatively by microscopy.

Soil, plant and water samples from all experimental treatments were analysed at The Chemical Analytical laboratory, Holt Research Centre, Troms¢, Norway. After dry ashing at 500 °C, the samples were digested in a mixture of concentrated  $HNO<sub>3</sub>$  and  $HCl$  $(1:2)$  before analysis for K by a flame photometer. The content of exchangeable K in soil samples was analysed after extraction in ammonium lactate (Egnér et al., 1960).

### *Experimental design and statistical analyses*

Three replicates of each of the 15 treatments (14 in Experiment 2) were organized in a randomized block design within one greenhouse compartment. The data for each experiment were analysed separately by ANO-VA with block, soil type and K-source as class vari-

*Table 3.* Potassium added at the start and recovered at the end of an experiment with Italian ryegrass (Experiment 1) grown in peat, loamy sand or silty clay loam with different K-sources. Fertilizer K is denoted as analysed by X-ray fluorescence spectrometry, and soil, water and plant K as K soluble in a mixture of concentrated HNO<sub>3</sub> and HCl. The amount of fertilizer K soluble in  $7/N$  HNO<sub>3</sub> and the amount of exchangeable K in the soil are given in brackets. Means within soil types marked with different letters were significantly different ( $p < 0.05$ ) in a Ryan-Einot-Gabriel-Welsch multiple comparison test

			K added at start of experiment		K recovered at end of experiment				
Soil type		Soil K	Fertilizer K	Sum	K in drainage water	K in plant shoots	K in soil and plant roots	Sum	
			$(g K box^{-1})$			$(g K box^{-1})$			
Peat	No K	0.2(0.1)	0.0	0.2	0.1a	0.7a	$0.3a$ $(0.2)$	1.0	
	Adularia	0.2(0.1)	25.0(2.2)	25.2	0.1a	1.4a	1.0 ac $(0.3)$	2.5	
	FilterII	0.2(0.1)	25.0(6.4)	25.2	0.1a	1.4a	8.7 abc (1.6)	10.2	
	Carbonatite	0.2(0.1)	25.0(24.0)	25.2	0.1a	15.6 <sub>b</sub>	13.5 <sub>b</sub> (1.4)	29.2	
	KCI	0.2(0.1)	25.0(25.0)	25.2	0.7 <sub>b</sub>	18.1c	10.5 <sub>b</sub> (7.5)	29.3	
Loamy	No K	8.6(1.1)	0.0	8.6	0.2a	3.1a	5.7a (0.3)	9.0	
sand	Adularia	8.6(1.1)	25.0(2.2)	33.6	0.2a	4.6a	6.7 ab $(0.3)$	11.5	
	FilterII	8.6(1.1)	25.0(6.4)	33.6	0.2a	4.3a	$9.5$ ab $(1.8)$	14.0	
	Carbonatite	8.6(1.1)	25.0(24.0)	33.6	0.4a	17.4 <sub>b</sub>	11.7 <sub>b</sub> (1.3)	29.5	
	KCI	8.6(1.1)	25.0(25.0)	33.6	1.6 <sub>b</sub>	19.7c	11.2 <sub>b</sub> (5.3)	32.5	
Silt	No K	28.2(0.6)	0.0	28.2	0.3a	3.9a	21.4 a (0.8)	25.6	
loam	Adularia	28.2(0.6)	25.0(2.2)	53.2	0.3a	4.4a	$23.5$ ab (3.0)	28.2	
	FilterII	28.2(0.6)	25.0(6.4)	53.2	0.5a	5.2a	27.8 <sub>b</sub> (1.7)	33.4	
	Carbonatite	28.2(0.6)	25.0(24.0)	53.2	0.3a	16.6 <sub>b</sub>	37.3 c (1.5)	54.2	
	KCI	28.2(0.6)	25.0(25.0)	53.2	2.2 <sub>b</sub>	18.6 <sub>b</sub>	24.7 ab (2.2)	45.5	

*Table 4.* Potassium added at the start and recovered at the end of an experiment with Italian ryegrass (Experiment 2) grown in peat or loamy sand with different K-sources. Fertilizer K is denoted as analysed by X-ray fluorescence spectrometry, and soil, water and plant K as K soluble in a mixture of concentrated HNO<sub>3</sub> and HCl. The amount of fertilizer K soluble in 7 N HNO<sub>3</sub> and the amount of exchangeable K in the soil are given in brackets. Means within soil types marked with different letters were significantly different ( $p < 0.05$ ) in a Ryan-Einot-Gabriel-Welsch multiple comparison test





*Figure 1.* The yield of K in harvested plant parts in three successive growth periods in an experiment with crushed rocks and mine tailings as K-sources for Italian ryegrass (Experiment 1). The plants were grown in peat, loamy sand or silt loam. ANOVA showed significant differences  $(p < 0.05)$  between soil types and between K-sources in all periods and a significant interaction between soil type and K-source in the first period.

ables. Treatment means were compared by the Ryan-Einot-Gabriel-Welsch multiple comparison test.

#### Results

In all types of growth medium the crushed rock Adularia and the tailings FilterlI and Lurgi supplied small amounts of K to the plants compared to KC1 and the carbonatite (Figures 1 and 2). In the treatments with peat moss, less than 10% of the added fertilizer K was recovered in above-ground plant parts at the end of the experiments compared to more than 60% for carbonatite and KCI treatments (Tables 3 and 4). In peat moss the total dry yield was, however, higher in treatments with Adularia, FilterlI and Lurgi than in the treatment with no K added (Table 5). The K added as epidote schists was more plant available than the K added as tailings and Adularia and less available than the K added as carbonatite and KC1 (Figure 2, Table 4). The plant availability of the K in Lurgi, and the harvested plant production, were about the same in the treatments with three different specific surfaces of this type of tailings (Figure 2, Tables 4 and 5). At the end of the last growth period, the content of K in plant dry matter was more than 1.4% in all treatments with carbonatite and KCI, and 0.5% and 0.6% in treatments with epidote schists in peat and loamy sand, respectively (data not shown).

In the treatments with no fertilizer K, less than 15% of the total K in silt loam and less than 40% of the K in loamy sand were recovered in harvested plant parts (Tables 3 and 4). Further, the recovered K constituted less than 1.5 and 2.7% of the initial K as analysed by XRF (115 and 256 g in loamy sand and silt loam, respectively). On the basis of the present results it is not possible to deduce which size fractions and minerals (Table 6) the plant K was taken from. In both soil types the plants had taken up more K than initially was present in the soil as exchangeable K (Tables 3 and 4).

In all treatments with FilterlI, Lurgi and Adularia the sum of the total recovered K at the end of the trial was far less than the sum of added K at the start of the experiments (Tables 3 and 4). This deficit indicates



*Figure 2.* The yield of K in harvested plant parts in three successive growth periods in an experiment with crushed rocks and mine tailings as K-sources for Italian ryegrass (Experiment 2). The plants were grown in peat or loamy sand with crushed rocks and mine tailings as K-sources. ANOVA showed significant differences ( $p < 0.05$ ) between K-sources in all periods, between soil types in the first and the second period and a significant interaction between soil type and K-source in the first period.

*Table 5.* Total dry yield in experiments with Italian ryegrass grown for 6 months in different soil types with different Ksources. Means within experiments and soil types marked with different letters were significantly different in a Ryan-Einot-Gabriel-Welsch multiple comparison test



that some of the K initially analysed by XRF was not digested in a mixture of concentrated acids (cf. Materials and methods section) and thereby not recovered in the soil samples at the end of the experiment. In Experiment 1 there was a corresponding deficit for the KCI treatment in silt loam (Table 3). KCI is highly soluble and K from this source should consequently be detected in drainage water, dried plant material and soil unless it is fixed in soil minerals.

The leaching of K was higher in treatments with KC! than in the other treatments (Tables 3 and 4). Even in the KC1 treatments, it did not account for more than 5% of the total amounts of K added at the start of the experiment.

The pH in the growth medium varied significantly between soil types and fertilizer treatments (Table 7). All tailings/crushed rocks contained  $CaCO<sub>3</sub>$  (calcite) (Table 1) and caused an increase in pH in all soil types.

*Table 6.* The initial particle size, the content of total K and K-bearing minerals in the clay, silt and sand fractions of loamy sand and silt loam soils. The minerals listed in descending order were: Bt - biotite (6-7% K), Chi - chlorite  $(0-0.5\% K)$ , Ill - illite  $(7-8\% K)$ , Ka - Kaolinite  $(0-0.5\% K)$ , Ms - Muscovite  $(8-9\% K)$ , PI - plagioclase (0-1% K). The K-content of different minerals is according to Deer et al. (1992)

	Particles size distribution $(vol\%)$	Median particle size $(\mu m)$	K content as analysed by <b>XRF</b> $(\%$ , w/w)	Fractional distribution of K $(\%$ of total K)	K-bearing minerals
Loamy sand		200	0.9		
clay fraction	2		1.4	3	$Ch$ $>$ $Il$ $l$
silt fraction	28	30	0.9	28	Pl > Chl > Ms
sand fraction	70	400	0.9	69	Pi > Chl > Bt
Silt loam		10	2.5		
clay fraction	8		3.7	14	Pi > III > Kln
silt fraction	88	7	2.1	84	Pl > Chl > Ms > Ill
sand fraction	4	400	1.4	2	Pi > Chl > Ms > Bt

*Table 7.* The pH in the growth medium at the end of two experiments with Italian ryegrass grown for 6 months in peat, loamy sand or silt loam (in Experiment 1 only) with KCI, rocks or mine tailings as K-sources. Means within experiments and soil types marked with different letters were significantly different in a Ryan-Einot-Gabriel-Welsch multiple comparison test



## **Discussion**

In agreement with the findings in previous pot and field experiments (Berthelin and Leyval, 1982; Goldschmidt and Johnsson, 1922; Hinsinger and Jaillard, 1993; Sanz Scovinio and Rowell, 1988; **Yli-Halla,** 

1992) the K supplied as K-feldspar from Adularia and tailings FilterlI was nearly unavailable to the plants, irrespective of soil environment and mineral particle surface. Considerable amounts K were, however, taken up from the crushed rocks carbonatite and epidote schists containing nepheline and/or biotite as their main K-bearing minerals. The K bound in carbonatite seemed to be nearly as plant available as the K in KCI, both in peat, loamy sand and silt loam. This rock, at least at the particle size used in the present experiments, is unlikely to be used as a slow-releasing K fertilizer. The epidote schists, on the other hand, might correctly be characterized as a slow-releasing K-source. At the end of the experiment, the concentration of K in the plant tissues was, however, well below what is regarded as a critical level for ryegrass (Barraclough and Leigh, 1993). This fact together with the low concentration of K in the rock itself indicate that the epidote schists is not the ideal source for a fertilizer either.

The nepheline-K and the biotite-K in the tailings Lurgi was harder to extract for the plants than the K bound in these minerals in the carbonatite and epidote schists. This could not be related to differences in the specific surface of the fertilizer particles as the performance of Lurgi was nearly the same within a range of surface from 0.15 to 1.47  $m^2$  g<sup>-1</sup>. Lurgi contained, however, less carbonate, manifested as a low loss on ignition and a low calcite content (Table 1), than the epidote schists and far less than the carbonatite. Carbonates dissolve faster than silicates in most environments (Laronne, 1986), and other minerals (e.g. biotite

and nepheline) in rock- particles with a high content of carbonate will consequently be more exposed to weathering and release K at a higher rate than minerals in rocks containing mainly silicates do.

In conclusion, a rock-based fertilizer containing biotite as its main K-bearing mineral and between 5 and 20% carbonate, will probably release K at a slower rate than K-salts do and still supply considerably more K to the plants than is supplied from the fraction of non-exchangeable K in the soil.

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