

Acidulated pegmatitic mica: A promising new multi-nutrient mineral fertilizer

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Abstract

Scrap grade pegmatitic phlogopite mica contains 5–7% K (~8% K₂O), 10–14% Mg (~23% MgO), 1–2% Ca (~2.9% CaO), 0.03% Mn and 109 ppm Zn. On acidulation upto 65% of K and Mg and 15–100% Mn and Zn were recovered. Less than 13% of Ca was recovered in solution. Water soluble and NH₄OAc extractable K and Mg of acidulated mica of pegmatitic origin increased a 10² to 10³ times compared to untreated mica. Acidulated mica remained non-hygroscopic even when mixed with acids at a 2:1 mica to acid ratio. X-ray diffraction analyses demonstrated that interlayer cations were easily leached from the mica structure leaving behind a kaolinitic residue, compared to the more stable tetrasilicate feldspars.

The most significant achievement through these experiments was the yield increase obtained in the greenhouse experiment with rice by using the lowest application rate (200 kg ha⁻¹) so far reported for mica, – an exponential decrease from tonnes/ha previously reported. Acidulated phlogopite mica chips (200 kg ha⁻¹– 4 kg K, 8 kg Mg, trace elements Mn, Zn etc.) gave a yield increase of over 41% compared to a control with recommended muriate of potash and dolomite (17 kg K, 6 kg Mg). The response to acidulated feldspar (500 kg ha⁻¹– 1.5 kg K) and an acidulated feldspar-dolomite combined fertilizer (250 kg ha⁻¹– 0.6 kg K and 6 kg Mg) was not significant.

The response to mica clearly shows a multinutrient deficiency in highly weathered tropical soils. The relatively high solubility of the acidulated mica, its range of nutrient element supply, its nonhygroscopic nature and its extremely simple manufacturing process makes mica, a cheap but effective fertilizer for the tropical regions where these nutrients are deficient, especially in highly metamorphosed crystalline terrains.

Introduction

The possibility of using rock powder directly as fertilizer has intrigued some workers (Keller, 1948; Gillman, 1980; Kronberg, 1977; Fyfe et al., 1983). 'Agriculture Alchemy: Stones into Bread' (Chesworth et al., 1983) characterises this attempt. Conventional NPK fertilizers lack many of the vital micronutrients and their continued application may deplete the natural nu-

trient supply in intensely cultivated, heavily weathered soils found in the tropics (Leonardos et al., 1987). The key factor that makes rock powder attractive when compared to highly soluble commercial fertilizer, is its potential for supplying an array of nutrients, among them the major elements K, cheaply, over a long period of time so that plants can make the most use of it, especially in the tropics. Godefroy et al., (1970) have computed that 60–70% of highly soluble

commercial K-fertilizer applied to tropical soils is subject to leaching during the first year. In Sri Lanka, Jayakody and Kendaragama (1989) reported that 50% of the recommended K needed for rice cultivation in the dry zone could be obtained from irrigated waters in the Mahaweli system B area. This amount in the irrigated waters is gained through leaching from applied K fertilizers in the intensely cultivated terrains of the catchment areas of the Mahaweli river in Sri Lanka. At present rock powder research spans several continents from Europe (Blum et al., 1989), South America (Salinas et al., 1986; Leonardos, 1987) to Eastern Africa (Chesworth et al., 1989).

The value of volcanic lava and ash in furnishing highly fertile soils is well known (Fyfe et al., 1983). For instance Blum et al. (1989) have shown that volcanic ash can be one of the best rock powder additives for soil amelioration. Smectite rich volcanic ash had a C.E.C. of $45.9 \text{ cmol kg}^{-1}$ compared to 3.0 cmol kg^{-1} in a podsol(Ap) and 2.5 cmol kg^{-1} in a pelosol(Ap). Volcanic ash has the ability to provide a high percentage of secondary nutrients, because it is porous and amorphous or finely crystalline. Thus it weathers relatively fast and provides a natural fertilizer (Kronberg, 1977; Salinas et al., 1986).

However, the rejuvenating conditions of periodic volcanism are not felt, in some countries dependent on agricultural economies situated in regions of tropical weathering. Sri Lanka is such a country. It is underlain by a highly metamorphosed, crystalline shield that has remained

stable during the past 500 million years, and as such poses a problem regarding the choice of rock powder for use as a cheap but effective fertilizer. Rocks found in crystalline terrains contain significant amounts of quartz which dilute the effectiveness of rocks as a source of K, Ca and Mg from minerals such as feldspars and mica. Nivas et al. (1987) reported the potassium supplying capacity of several commonly found rock types in Sri Lanka. Among them pink granite, granulitic gneiss and migmatitic gneiss had relatively high exchangeable K contents (Table 1). In pegmatites, however, feldspar and mica occur as relatively pure concentrates. Hence the current authors decided to test deposits of alkali-feldspar [$\text{K}(\text{Al}, \text{Si})_3\text{O}_8$] and phlogopite mica [$\text{K}(\text{Mg})_3\text{AlSi}_3\text{O}_{10}(\text{OH}, \text{F})_2$] of pegmatitic origin also found in Sri Lanka.

Pure mica and feldspar have been tested individually, for their fertilizer value previously, though the results on its efficacy are conflicting. Preliminary unpublished studies using mica and feldspar as straight fertilizer by the present authors have indicated that massive dosages had to be applied to use untreated mica and feldspar as substitute fertilizer for rice cultivation. A 20% yield increase was obtained at an application rate of 10 tons ha^{-1} , but not at lower rates in a field experiment. However without any additional mica the same field was able to provide a 20% yield increase in the next growing season, with only an additional input of N and P. Sanz-Scovino and Rowell (1988) report that finely ground sanidine feldspar gave nonsignificant

Table 1. K_2O content, exchangeable K and C.E.C. of some rocks tested as potential fertilizer in Sri Lanka

Rock Material	Total K (%)		Exchangeable K (meq/100 g)		CEC (meq/100 g)
	(1)	(2)	(1)	(2)	
Phlogopite Mica Dust	4.47	4.51	0.16	0.15	8.17
Kaikawela Feldspar		10.37	0.33	n.a.	1.11
Pink Granite	5.70	5.15	0.10	0.10	0.77
Graphite			0.02	0.02	0.18
Granulitic Gneiss			0.31	n.a.	4.10
Charnockite			0.14	0.13	1.17
Marble			0.01	n.a.	1.08
Bt. Gn.		1.31	0.33	n.a.	2.31
Granulitic Gneiss ^a			0.36		
Migmatitic Gneiss ^a			0.314		
Pink Granite ^a			0.351		

^a Nivas et al. (1987).

response in a pasture experiment in Columbia. Kahnt et al. (1986) report an increase in barley grain yield of upto 67% by using 10% w/w rock powder. This however corresponds to approximately 350 tons ha⁻¹ of crushed rock as calculated by the present authors. Salcedo et al. (1982) obtained 4.54 g dry matter pot⁻¹ by using biotite which was 32.4% of the yield produced by a control using soluble K. Application rate cited was 200 kg ha⁻¹ K₂O added as biotite which should correspond to 2.5 tons ha⁻¹ biotite (at 8% K₂O in biotite). Arkhipov et al. (1988) report yield increase of 10–150% with reference to potatoes, cereals and an oats-vetch mixture by using phlogopite applied either alone or combined with lime, peat and mineral fertilizers.

Thus research shows that rock powder, with the exception of volcanic rocks, can only act as a soil ameliorant or slow-release fertilizer unless it is supplied in massive dosages of the order of several tonnes per hectare. Hence experiments have been designed to increase solubility by thermal treatment of phlogopite combined with a wide range of products (Aitta et al., 1987); through acidulation with organic acids, at varying mica (feldspar): acid ratios (Fragstein and Vogtmann, 1983; Song and Huang, 1988; Lundstrom and Ohman, 1990) and by using rhizosphere microflora (Berthelin and Leyvel, 1982). Microbicidal and nematocidal properties of acidulated mica group minerals have also been reported. Use of inorganic acids to increase solubility or orthoclase as well as biotite and muscovite mica is reported by Borsch (1990) and for phlogopite mica and alkali feldspar by Weerasuriya et al. (1991).

This paper reports results of acidulation trials with inorganic acids aimed to increase nutrient releasing capacity of fresh and waste grades of phlogopite mica and alkali-feldspar of pegmatitic origin as well as a green house experiment with acidulated mica to grow rice. Feldspar and mica are mined for the insulating and ceramic industries respectively in Sri Lanka. The feldspar deposit is capable of supplying the ceramic industry for another 100 years or more. The extent of pegmatitic mica has not been evaluated and at present it is mined at a cottage industry level in the Matale and Elahara regions.

Green house experiments with acidulated mica

and feldspar to grow rice have been conducted in Sri Lanka using application rates of 1–10 tons for acidulated mica and 10 tons ha⁻¹ for acidulated feldspar (Pushpakumara et al., 1991; Kithsiri, 1991). An increase in panicle and tiller number over 45% compared to the control was obtained with the lowest application rate used till then, – 1 ton ha⁻¹ acidulated mica (K₂O approximately 55 kg). In the greenhouse experiment reported here application rates were partially based on commercial considerations (100 kg acidulated mica and 100 kg MOP cost approximately US \$18 and 22).

Materials and methods

Samples of fresh phlogopite mica [K(Mg)₃AlSi₃O₁₀(OH, F)₂] and corresponding waste grades were collected in Sri Lanka from a mica processing center in Matale, and waste grades of K-feldspar [K(Al, Si)₃O₈] were collected from the main feldspar deposit at Kaikawela. Representative samples were crushed to pass through a 149 micron diameter sieve. Samples were digested with a mixture of nitric/perchloric/hydrofluoric acids (tri-acid digests – TAD) and filtered. The filterates were analysed using inductively coupled plasma spectroscopy to determine total concentration of 7 major elements for mica and feldspar (Table 2A). Mica samples crushed at two different mills (Mica-1 and Mica-2 vs. Mica-3) were analysed by X-ray fluorescence for 8 major elements and 15 trace elements (Table 2B).

Preparation of acidulated mica(feldspar)

Three 50 g portions of crushed mica were mixed separately at room temperature, with 25 ml of 10 M analytical grade, HNO₃, HCl and H₂SO₄ acids respectively, to give a 2:1 ratio of mica:acid. Similarly three 5:1 feldspar:acid mixtures were prepared separately with the three acids. In addition 5:1, and 10:1 ratios of mica:H₂SO₄ acid and a 10:1 ratio of feldspar:H₂SO₄ acid were also prepared. For comparative purposes samples of waste grade (weathered) phlogopite mica and biotite mica [K(Mg, Fe⁺²)₃(Al, Fe⁺³)Si₃O₁₀(OH)₂] were also included. The acidulated products were crushed

Table 2A. Major element composition of some rocks tested as potential fertilizer in Sri Lanka

Major Element Oxide	Pink Granite		Weathered Phlogopite		Gt.Bt.Gn ^a	Kaikawela Feldspar
	(1)	(2)	(1)	(2)		
Al ₂ O ₃	12.25	13.04	12.7	12.89	15.30	13.85
Fe ₂ O ₃	2.7	2.7	5.36	6.34	9.15	0.38
CaO	1.38	1.49	3.9	4.27	8.16	0.069
MgO	0.34	0.39	9.59	9.03	4.7	0.01
TiO ₂	0.22	0.18	0.72	0.63	0.82	0.0046
Na ₂ O	2.18	2.11	0.11	0.18	1.76	1.07
K ₂ O	5.15	5.70	4.47	4.51	1.31	10.37

^a Garnet biotite gneiss.

Table 2B. XRF analysis of pure and acidulated phlogopite mica

	Mica-1 (n = 1)	Mica-2 (n = 1)	Mica-3 (n = 3) Avg.	Std.D.	Acidulated Mica (n = 1)
SiO ₂	37.71	39.20	38.47	0.28	15.48 (43.08*)
TiO ₂	0.95	0.92	1.04	0.02	0.30 (0.83)
Al ₂ O ₃	15.95	15.41	16.67	0.19	5.84 (16.25)
Fe ₂ O ₃	6.17	6.33	6.06	0.00	2.32 (6.45)
MnO	0.05	0.07	0.04	0.00	0.01 (0.03)
MgO	22.44	20.82	23.30	0.12	8.07 (22.46)
CaO	1.94	2.86	1.11	0.24	0.52 (1.45)
K ₂ O	7.70	6.49	8.67	0.21	2.90 (8.07)
P ₂ O ₅	0.05	0.00	0.05	0.07	0.00 (0.00)
Na ₂ O	0.00	0.00	0.00	0.00	0.00 (0.00)
L.O.I.	6.31	7.11	3.73	0.38	64.07 (-)
Total	99.27	99.21	100.84		99.50 (100.00)
Trace Elements (ppm)					
Nb	29	55	32	1	13
Zr	55	79	40	5	27
Y	17	18	15	0.5	4
Sr	107	105	118	3.1	36
Rb	538	452	568	7.4	247
Pb	5	6	<5	-	<5
Zn	126	147	109	3.3	56
Cu	40	53	19	2.6	11
Ni	49	47	44	0.5	20
Co	12	16	15	1.7	6
Cr	40	51	48	2.6	16
Ba	6922	6010	7461	124.7	3020
V	83	78	81	3.4	41
As	<5	<5	<5	-	<5
Ga	14	16	15	1.3	7

* Anhydrous basis.

using a mortar and pestle prior to analysis. A 2:1 acidulated mica sample was heat treated at 500°C for 30 minutes to produce a heat-treated acidulated mica product.

Solubility check

Water soluble base and trace element concentrations of the acidulated products were analysed by shaking 5 g of acidulated fertilizer with 100 ml

distilled deionised water for 2 hours. A non-acidulated sample was included for comparison. Exchangeable bases were extracted from 5 g similarly with 1.0 M NH_4OAc . The resultant filtered solutions were analyzed for K, Mg, Ca, Fe, Mn and Zn in solution using atomic absorption spectrophotometry and Cu, Al, Si and B by inductively coupled plasma spectroscopy (Figs. 1–4, Tables 3, 4). Analyses were repeated 2 or 3 times with the acidulated fertilizers as starting material [(1), (2), and (3) in Table 3]. The percentage of elements released due to acidulation (Table 5) was calculated using averages of

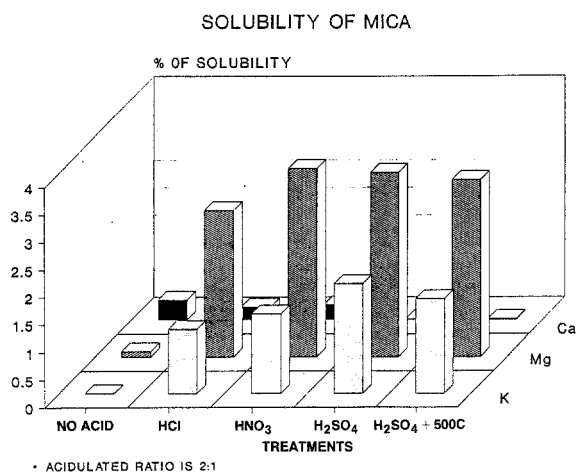


Fig. 1. NH_4OAc extractable contents of K, Ca and Mg of pure mica and 2:1 acidulated mica.

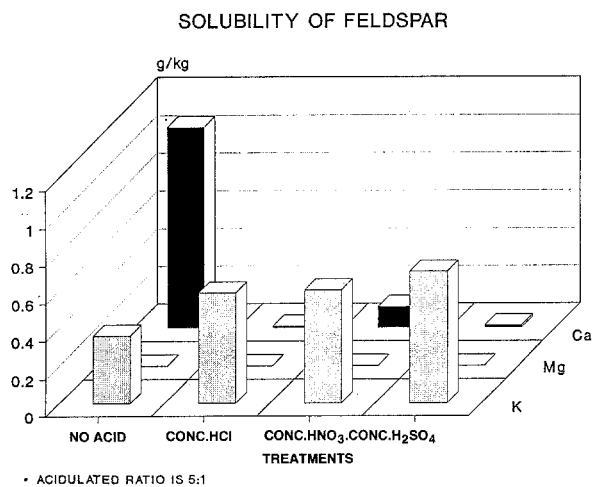


Fig. 2. NH_4OAc extractable contents of K, Ca and Mg of pure feldspar and 5:1 acidulated feldspar.

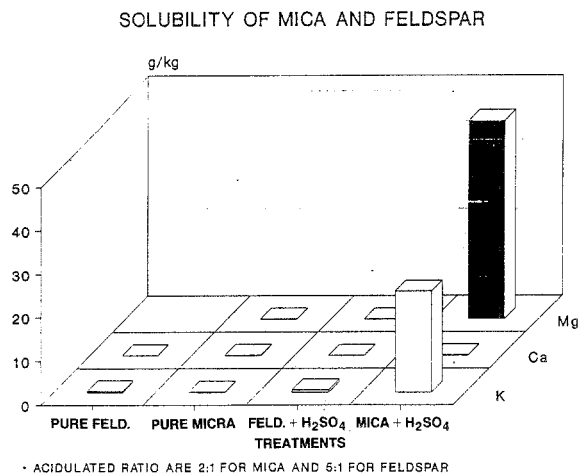


Fig. 3. Water soluble contents of K, Ca and Mg of pure and 2:1 acidulated mica and pure and 5:1 acidulated feldspar.

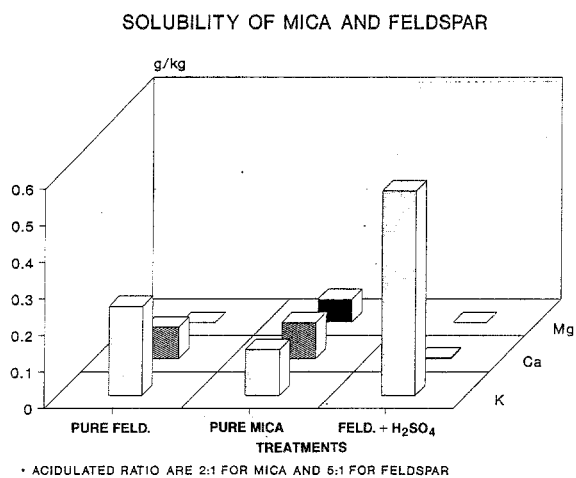


Fig. 4. Water soluble contents of K, Ca and Mg of pure mica and feldspar and 5:1 acidulated feldspar on an expanded Y-scale.

Table 3 (K, Mg, Ca) and water soluble trace element concentrations also recovered after shaking 5 g in 100 ml water (Table 4). Allowance was made for the weight of acid (specific gravity \times volume) in each 5 g portion of product. Recovery of elements in the acidulated product is expressed as a percentage of the XRF analysis Mica-3. X-ray diffraction analysis of the untreated mica and the H_2SO_4 acidulated product was carried out using a Shimadzu X-ray diffraction machine at 2° per minute, Cu K-alpha radiation, and 1° degree diffraction grating.

Table 3. Water soluble contents of K, Mg and Ca of varying rock powder: H₂SO₄ ratios

Rock powder: H ₂ SO ₄ ratio	K (ppm)		Mg (ppm)		Ca (ppm)	
	(1)	(2)	(1)	(2)	(1)	(2)
Feldspar: H ₂ SO ₄ ratio						
5:1	(a) 27.6 (b) 28.3		(a) 0.6 (b) 0.6		(a) 0.17 (b) 0.09	
5:1*	(a) 31.1 (b) 31.9		(a) 1.3 (b) 1.2		n.d. n.d.	
10:1*	(a) 20.0 (b) 19.7		(a) 1.6 (b) 3.0		n.d. n.d.	
Pure Feldspar (n = 8)	(a) 12.2 ± 3.4 (b)		(a) – (b) –		(a) 4.33 ± 0.8 (b)	
Fresh Phlogopite Mica: H ₂ SO ₄ ratio						
2:1	(a) 1145.5 (b) 1166.6		(a) 2343.0 (b) 2197.0		(a) 6.3 (b) 6.3	
5:1	(a) 811.0 (b) 892.5	(a) 609.5 (b) 672.0	(a) 1210.0 (b) 1272.5	(a) 1405.0 (b) 1485.0	(a) 8.2 (b) 7.7	
10:1	(a) 600.0 (b) 623.0	(a) 505.5 (b) 467.0	(a) 1085.5 (b) 1024.5	(a) 1236.0 (b) 1158.0	(a) 10.0 (b) 5.3	
Fresh Phlogopite Mica	(a) 6.4 (b) 6.1	(a) 6.4 (b) 6.1	(a) 3.3 (b) 3.1	(a) 3.3 (b) 3.1	(a) 4.6 (b) 5.3	
Weathered Phlogopite Mica: H ₂ SO ₄ ratio						
2:1	(a) 539.5 (b) 535.5	(a) 591.8 (b) 558.1	(a) 1563.0 (b) 1518.0	(a) 2001.0 (b) 1992.0	(a) 8.2 (b) 8.2	(a) 5.1 (b) 5.5
5:1	(a) 167.9 (b) 193.5	(a) 195.5 (b) 194.1	(a) 1028.5 (b) 1010.5	(a) 1269.0 (b) 1283.0	(a) 19.0 (b) 17.5	(a) 8.8 (b) 8.7
10:1	(a) 38.0 (b) 36.2	(a) 61.4 (b) 134.3	(a) 592.0 (b) 716.0	(a) 777.0 (b) 1160.5	(a) 62.8 (b) 49.2	(a) 18.0 (b) 13.3
Weathered Phlogopite Mica	(a) 2.4 (b)	(a) 3.9 (b) 2.9	(a) 2.5 (b) 2.1	(a) 3.3 (b) 3.5	(a) 5.3 (b) 6.1	(a) 3.0 (b) 4.5
Weathered Biotite Mica: H ₂ SO ₄ ratio						
2:1	(a) 1127.0 (b) 1191.0		(a) 72.7 (b) 71.6		(a) – (b) –	
5:1	(a) 604.0 (b) 633.0		(a) 32.7 (b) 32.6		(a) – (b) –	
10:1	(a) 478.5 (b) 515.3		(a) 26.5 (b) 26.7		(a) – (b) –	
Weathered Biotite Mica	(a) 5.5 (b) 8.4		(a) 0.4 (b) 0.5		(a) – (b) –	

* Ammonium Sulphate added to reduce hygroscopy

Agromimic evaluation

Acidulated mica and feldspar were applied to a tropept (Inceptisol Order – with 4.31 meq/100 g exchangeable Mg, 0.17 meq/100 g exchangeable K, and 7.07 meq/100 g exchangeable Ca) in a greenhouse pot experiment to grow rice using the lowest application rates so far tested in Sri Lanka (Fig. 6, Table 6). Pots were filled with 7 kg ha⁻¹ soil. Concentrated nitric acid was used to prepare 5:1 acidulated mica and feldspar. The pot experiment was designed to test whether the response to acidulated mica noted in previous

experiments was due to Mg and/or K supplied by phlogopite mica. Thus an attempt was made to prepare a Mg-K fertilizer by mixing dolomite separately with muriate of potash (MOP) and feldspar.

Higher rates of acidulated feldspar were used because of its lower K supplying capacity. Application rates of 200 kg ha⁻¹ (T₄) for acidulated mica chips (2–3 mm in diameter), 500 kg ha⁻¹ (T₅) for acidulated feldspar together with a combined acidulated feldspar – dolomite rate of 250 kg ha⁻¹ (T₆) were compared against controls

Table 4. Average water soluble contents of major and trace element contents of varying rock powder:H₂SO₄ ratios

Sample	K (ppm) Avg.	Std.D	MG (ppm) Avg.	Std.D	CA (ppm) Avg.	Std.D.	AL (ppm) Avr.	Std.D.	pH	N (Replicate)
Mica-3	71914.00	155.24	139820.00	694.40	5475.91	4224.86	93174.38	1057.8		3
FM 2:1	1156.00	10.50	2270.00	73.00	6.31	0.00	n.d.			2
FM 5:1	746.25	111.57	1343.13	108.01	7.92	0.23	1046.01	33.9		4
FM 10:1	548.88	64.60	1126.00	79.16	9.86	0.09	850.80	1.2		4
FM	6.25	0.15	3.20	0.10	4.95	0.35	0.00			2
WM 2:1	623.82	97.61	1883.00	247.09	6.16	1.45	1315.00	55.6	1.38	6
WM 5:1	209.35	32.06	1172.65	110.68	12.21	4.30	788.00	8.9	1.82	6
WM 10:1	83.59	39.88	825.42	175.17	28.55	19.84	428.80	67.2	2.47	6
WM	3.14	0.52	3.18	0.71	4.77	0.95	0.00		7.29	2
Sample	FE (ppm) Avg.	Std.D.	ZN (ppm) Avg.	Std.D	MN (ppm) Avg.	Std.D.	CU (ppm) Avg.	Std.D.		N (Replicate)
MICA-3	42420.00	0.00	109.00	3.30	300.00	0.00	19.00	2.16		3
FM 5:1	586.61	34.82	2.18	0.01	2.96	0.01	0.76	0.03		4
FM 10:1	500.01	1.24	1.82	0.02	2.93	0.02	0.76	0.00		4
FM	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d		
WM 2:1	627.92	75.57	1.40	0.04	7.47	0.95	0.43	0.05		4
WM 5:1	353.86	36.33	0.94	0.10	7.60	1.23	0.39	0.08		4
WM 10:1	191.56	69.51	0.70	0.18	8.35	1.88	0.39	0.09		4
WM	-	-	-	-	0.01	0.01	-	-		4

FM – Fresh Mica;

WM – Weathered Mica;

Mica-3 Calculated from XRF analyses (Table 2A).

Table 5. Element percentage recovered in water extract and recovery compared to XRF analysis of mica

Sample	K %	Recovery %	Mg %	Recovery %	Ca %	Recovery %	Al %	Recovery %
MICA-3	7.19		13.98		0.55		9.32	
FM 2:1	4.62	64.31	9.08	64.95	0.03	4.61	n.d	
FM 5:1	2.04	28.36	3.67	26.25	0.02	3.95	2.86	41.60
FM 10:1	1.30	18.05	2.66	19.04	0.02	4.26	2.01	29.30
FM	0.03	0.35	0.01	0.09	0.02	3.62	-	-
WM 2:1	2.50	34.70	7.53	53.88	0.02	4.50	4.94	71.90
WM 5:1	0.57	7.96	3.20	22.92	0.03	6.09	2.15	31.30
WM 10:1	0.20	2.75	1.95	13.96	0.07	12.33	1.01	14.70
WM	0.01	0.17	0.01	0.09	0.02	3.48	-	-
SAMPLE	Fe %	Recovery %	Zn %	Recovery %	Mn %	Recovery %	Si %	Recovery %
MICA-3	4.24		0.01		0.03		17.95	
FM 5:1	1.59	37.50	0.01	55.21	0.01	26.67	n.d	
FM 10:1	1.18	27.83	0.00	36.81	0.01	23.33	n.d.	
FM	n.d		-					
WM 2:1	2.51	59.24	0.01	55.21	0.03	99.57	0.67	3.72
WM 5:1	0.97	22.80	0.00	27.61	0.02	69.21	0.60	3.32
WM 10:1	0.45	10.68	0.00	15.15	0.02	65.81	0.34	1.91
WM	-		-		0.00	0.05	0.26	1.45

FM – Fresh Mica;

WM – Weathered Mica;

MICA-3 Calculated from XRF analyses (Table 2A).

Table 6. Grain response to acidulated mica and feldspar

Treatment	Grain Yield (g/pot)	Duncan Grouping
T ₄ . 200 kg ha ⁻¹ A.M. ¹ Chips	93.3	A ³
T ₅ . 500 kg ha ⁻¹ A.F. ² Powder	69.8	B
T ₁ . 33 kg ha ⁻¹ MOP	65.9	B
T ₆ . 200 kg ha ⁻¹ A.F. + 50 kg ha ⁻¹ dolomite	60.5	B
T ₂ . 33 kg ha ⁻¹ MOP + 50 kg ha ⁻¹ dolomite	55.9	B C
T ₃ . 0 kg ha ⁻¹ MOP + 50 kg ha ⁻¹ dolomite	52.0	B C
T ₀ . 0 kg ha ⁻¹ MOP	41.1	C

¹ A.M. = Acidulated Mica;

² A.F. = Acidulated Feldspar;

³ Treatments with the same letter are not significantly different at the 95% confidence interval.

without (T₀) and with muriate of potash (T₁ – 33 kg ha⁻¹). A combination of MOP and dolomite (T₂) as well as dolomite by itself (T₃) was also tested. The recommended dose of N and P were applied to all pots. Tiller and panicle numbers were counted and yield weighed. Soil solutions collected every fortnight was analysed for pH, K, Ca, and Mg. The shoot nutrients were analysed after harvest. In this paper results of tiller, panicle and yield measurements are reported.

Results and discussion

Physical properties of fertilizer

Acidulated feldspar was found to be excessively hygroscopic even after oven drying and feldspar:acid ratio could not be increased beyond 5:1. In contrast mica remained dry and granular (2–4 mm in size) even with mica:acid ratios of 2:1. It was not necessary to oven dry the product which could be stored in capped glass jars for any length of time. This augers well for its commercial preparation.

Chemical properties

Tri-acid digest and XRF results (Table 2A, 2B) indicate that the total K, Mg and Ca comprise 5–7%, 10–14% and 1–2% of the fresh mica. Upto 4.6% K and 9% Mg was recovered (Table 5) in solution from the 2:1 fresh sample:acid mixture. With the weathered (scrap) grade, the recovery rates were less. It implies either that

there is a remainder which could act as a buffer or that part of K and Mg has already leached out from the scrap grade. The increase in recovered Fe, Al, and Mn % in the weathered product may support the latter contention. In a parallel study, with muscovite and biotite micas containing 7–14% total potash, Borsch (1990), recovered 2–4% potash with 2% ammonium citrate solution after nitric acid treatment of the mica and upto 9.6% (from biotite) with sulphuric acid.

Recovery of K from feldspar was poor. 0.2% or less was made available with acid attack (Table 3) from a total K content of 8–12% K (10–15% K₂O). Similarly Borsch (1990) recovered 0.2–0.3% potash after rigid acid treatment, from orthoclase, with a total potash content of over 10%.

Treatment with concentrated HNO₃, and H₂SO₄ proved slightly more effective than treatment with HCl (Figs. 1, 2), as shown by the base cation content when extracted with NH₄OAc.

The pH of the water extracts of acidulated mica measured 1.4 to 2.5 units (Table 4). As with NH₄OAc extractable K and Mg, the water soluble K and Mg contents of H₂SO₄ treated mica were several 10² to 10³ orders of magnitude greater than similarly treated feldspar (Figs. 1–3). Treatment with acid did not increase water soluble contents of K to any great extent in the feldspar (Figs. 3, 4).

An increasing trend in the water soluble contents of K, Mg, Fe, Zn, Mn and Cu were observed with increasing acid concentrations, i.e. lower mica:acid ratios (Tables 3, 4). The reverse was true for Ca especially in the weathered mica where the recovery of Ca was higher 12% in the

10:1 WM product (Tables 4, 5). NH_4OAc extractable Ca in the acidulated mica was also less than that obtained from pure mica excepting with HNO_3 (Fig. 1). Decrease in the recoverable Ca content with increasing acidity may result from the precipitation of Ca compounds such as gypsum.

The content of bases that went into solution was markedly less in the waste grade (weathered) phlogopite mica compared to fresh phlogopite mica. Compared to weathered biotite, however the weathered phlogopite released higher Mg and lower K contents. Biotite mica did not release any measurable Ca. These differences directly reflect the chemical composition of the two micas.

Structural changes

Since acidulated mica gave best results, an X-ray diffractogram of the product was obtained (Fig. 5). The untreated mica gives sharp clearly defined peaks characteristic of mica. The treated product shows a diminution of the mica peaks and a broad hump in the region of 7 \AA likely corresponding to disordered kaolinitic material or poorly crystalline or "amorphous" material showing the band diffraction around 7 \AA . In a recent TEM study of muscovite weathering Robertson and Eggleton (1991) observed 10 \AA kaolinite d-spacing contiguous with typical mica lattice spacing and concluded that muscovite can weather directly to kaolinite. It would appear that acidulating mica has a similar effect, in that the acid leaches out the interlayer bonding cations and gives rise to kaolinite and con-

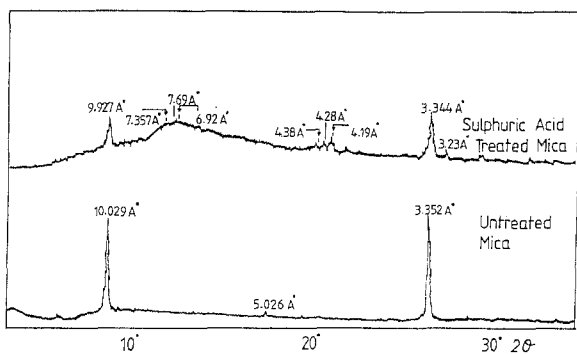


Fig. 5. X-ray diffractogram of pure and sulphuric acid treated mica.

sequent high concentrations of cations in solution. The marked contrast between mica and feldspar towards acidulation is explained by the difference in crystal structure between the sheet silicate structure found in mica which likely lends itself to acid attack more easily and the more durable framework silicate structure found in feldspar.

Agronomic evaluation

The response to 200 kg ha^{-1} of 5:1 acidulated mica chips is clearly significant in the greenhouse experiment (T_4 - Table 6, Fig. 6) compared to the currently recommended dose of MOP and MOP with dolomite (T_1 and T_2). An increase of 32% and 41% in panicle number and seed weight compared to the control with recommended MOP was obtained by using acidulated mica. The authors have not come across reference to pot experiments or field trials that

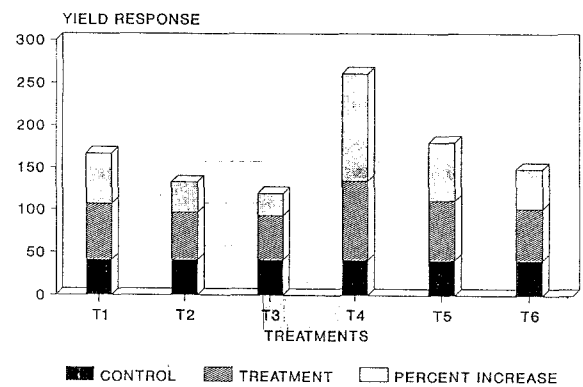
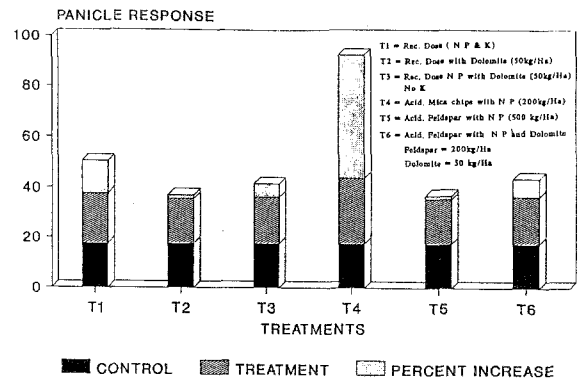


Fig. 6. Panicle and yield response to acidulated mica and feldspar

produced a high demand crop such as rice, with such low levels of mica fertilizer. Application rates have been in tonnes ha⁻¹ in the literature cited. Feldspar however did not produce a significant response at the levels applied (Table 6). The response to 200 kg ha⁻¹ of acidulated mica (4 kg ha⁻¹ K at 2% immediately soluble K) cannot only be to the K supplied since the treatment with MOP (T₁) was supplied with 17 kg ha⁻¹ or 4 times as much. The factors responsible for response to mica could be a combination of elements – K, Mg, S, Si, and trace elements since even the amount of Mg supplied (8 kg ha⁻¹) is low. Addition of dolomite together with MOP suppressed yield (T₂ – Table 6) implying that acidulated mica containing K-Mg-S cannot be equated to K-Mg-Ca-CO₃ given by the MOP-dolomite combination. This result has far reaching repercussions on the application of dolomite in the presence of MOP to address Mg deficiency.

Conclusion

A green house pot experiment with acidulated mica gave promising results with a yield increase of 41% compared to a control with MOP. The moderate solubility of the acidulated mica, its range of nutrient element supply, its non-hygroscopic nature and its extremely simple manufacturing process makes mica, a cheap but effective fertilizer for the tropics.

These results also have important bearing on future rock powder research in that it helps to narrow down the possible choices among rock types that need be tested for their potential as fertilizer. Treatment with acid will increase the solubility of any kind of rock powder, but rocks containing a greater percentage of sheet silicates such as biotite gneisses are likely to prove more successful compared to rocks containing high percentages of quartz and feldspar (both being framework silicates) such as granite. In a high grade gneissic terrain as found in Sri Lanka, devoid of recent volcanic activity and minerals such as zeolites, the next best sources are pegmatitic micas, followed by mica bearing rocks.

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