

SHORT COMMUNICATION

AGRONOMIC EFFECTIVENESS OF NOVEL PHOSPHATE FERTILISERS DERIVED FROM AN IGNEOUS ZIMBABWE PHOSPHATE ROCK

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ABSTRACT

A review of literature shows that work on non-conventional phosphate fertilisers has been done exclusively on sedimentary phosphate rocks. The potential of using novel phosphate fertiliser materials derived from unreactive igneous Dorowa (Zimbabwe) phosphate rock was investigated in a greenhouse experiment. Three phosphate fertiliser materials; (1) finely ground (0.150-mm screen) Dorowa phosphate rock (DPR), (2) partially acidulated Dorowa phosphate rock (PADPR), and (3) a compacted mixture of DPR + triple superphosphate + urea + potassium chloride (DTUK) with half of P from DPR and half from triple superphosphate (TSP) were made from Dorowa rock and their relative agronomic effectiveness compared with that of single superphosphate (SSP) on an acid Hartsells silt loam (pH 4.8) with maize (*Zea mays* L.) as the indicator crop. Direct application of DPR was found to be ineffective in increasing phosphorus (P) uptake and dry matter yield of maize. The relative agronomic effectiveness of DPR partially acidulated with 50% of the sulfuric acid (H₂SO₄) required for complete acidulation, in increasing P uptake and dry matter yield was 60% and 75%, respectively. The compacted fertiliser product, DTUK, was equally effective in increasing P uptake and dry matter yield as SSP.

Key Words: Acidulation, agronomic effectiveness, fertiliser compaction, *Zea mays*

RÉSUMÉ

Une revue de la littérature montre que le travail sur engrais phosphatés non conventionnels a été fait exclusivement sur les roches (substances) phosphate sédimentaires. Le potentiel d'utilisation des nouveaux matériaux fertilisants phosphatés dérivés de réactifs éruptifs Dorowa (Zimbabwe) ont été examinés dans une serre expérimentale. Trois matériaux fertilisants phosphate ; finement moulu (0, 150 mm tamis) Dorowa roche phosphatée (DRP), et 2) DRP acidulé (DRPA), et 3) un mélange compacté de DRP et le triple superphosphate+urée+ chlorure de potassium (DTUK) avec la moitié de P du DRP et la moitié de triple superphosphate (TSP) était faite à partir de la roche Dorowa et leur efficacité agronomique relatives comparées avec celle du superphosphate (SSP) sur un dépôt d'acide Hartsells terreau (BH 4, 8) avec le maïs (*Zea mays* L.) comme indicateur de plante. Une application directe du DRP était trouvée être non effective dans la croissance de l'assimilation du phosphore (P) et production de matière sèche de maïs. L'efficacité relative agronomique du DRP partiellement acidulé, avec 50% d'acide sulfurique (H₂SO₄) demandée pour une complète acidulation, pour l'accroissement de l'assimilation et la production de matière sèche de P était respectivement de 60% et 75%. Le produit fertilisant compacté DTUK était également effectif dans accroissement de l'assimilation et la production de matière sèche de P comme le SSP.

Mots Clés: Acidulation, efficacité agronomique, compaction de fertilisants, *Zea mays*

INTRODUCTION

Phosphate rock reserves in Zimbabwe amount to 37 million Mg at Dorowa deposit, 18 million Mg at Shawa, and a "considerable amount" at Chishanya (McClellan and Notholt, 1986). The Dorowa, Shawa, and Chishanya phosphate rocks contain an average of 8 %, 3.8 %, and 7.5 % P_2O_5 , respectively. The Dorowa deposit is the only one currently used in the production of water-soluble fertilisers. Current production is 130,000 Mg of concentrate (33 % P_2O_5) annually. The concentrate is processed to produce single superphosphate (SSP) and triple superphosphate (TSP). Dorowa phosphate rock approximate composition: $Ca_{10}(PO_4)_6(F_{1.08}, OH_{0.92})$ (Van Kanwenbergh, 1989) is an igneous hydroxy-fluorapatite with MgO/P_2O_5 , CaO/P_2O_5 , $(Fe_2O_3 + Al_2O_3)/P_2O_5$, and $(Fe_2O_3 + Al_2O_3 + MgO)/P_2O_5$ weight ratios which are higher than the levels considered desirable for the production of conventional phosphorus (P) fertilisers such as SSP and TSP (Roy and McClellan, 1985). The higher these ratios the greater the sulfuric acid (H_2SO_4) consumption for wet-process phosphoric acid production and the greater the post precipitation of sludge in phosphoric acid, scale formation on equipment during phosphoric acid concentration, insoluble phosphoric compounds in liquid or solid ammonium phosphate products, and unwanted agglomeration in non solid ammonium polyphosphate (Becker, 1989).

Iron and aluminum can also cause reversion of available P to an unavailable form in superphosphate-type products (Roy and McClellan, 1985). In addition to the problem of undesirable metal oxides to P_2O_5 ratios, DPR is unreactive because as an apatite from igneous sources, it is coarsely crystalline and does not possess internal surfaces (Khasawneh and Doll, 1978; Leon *et al.*, 1986). The purpose of this study was to evaluate the agronomic effectiveness of novel fertilisers produced by partial acidulation and compaction of PR with soluble fertilisers as alternatives to produce P fertilisers from an igneous PR source that may otherwise be unsuitable for use as P fertilisers.

MATERIALS AND METHODS

Phosphate fertiliser materials. The primary P fertiliser source for this study was Dorowa phosphate rock. Fertiliser materials made from Dorowa rock were: (1) finely ground Dorowa phosphate rock (DPR); (2) partially acidulated Dorowa phosphate rock (PADPR); and (3) compacted DPR + triple superphosphate + urea + potassium chloride (DTUK).

The fertiliser materials were prepared at the International Fertiliser Development Center (IFDC), Alabama. The finely ground DPR was prepared by grinding the phosphate rock to pass a 0.150-mm screen (100-mesh). The PADPR fertiliser material was prepared by partially acidulating the finely ground DPR with 50% of the H_2SO_4 required to fully acidulate the rock to produce SSP (Schultz, 1986). The 50% level of acidulation was selected based on proportions used in previous studies (Terman and Allen, 1967) and after preliminary greenhouse tests. The compacted phosphorus fertiliser material was prepared by first grinding separately DPR, TSP, urea, and KCl to pass a 0.150-mm screen; and then mixing appropriate amounts to produce N: P_2O_5 : K_2O ratio of 1:1:1 with half the P from DPR and half from TSP. After mixing, the product was physically compacted by applying pressure equal to 9 Mg cm^{-1} using a hydraulic compactor (Lupin and Le, 1983). Both the partially acidulated DPR and the compacted mixture were crushed and screened to particle sizes smaller than 3.36 mm but larger than 1.19 mm. A commercial grade SSP was used as a standard to compare the agronomic effectiveness of DPR, PADPR and DTUK. The total P_2O_5 , water soluble P_2O_5 , and neutral ammonium citrate soluble P_2O_5 of the fertilisers used in the study are shown in Table 1.

Greenhouse study. A bulk soil sample from an Ap horizon of a Tennessee Hartsells silt loam (Typic Hapludult; fine loamy, silicious, thermic; pH 4.8 [1:1 soil: water], organic matter 48 g kg^{-1} , and CEC 9.5 $cmol_c kg^{-1}$) was air dried, and screened to less than 2 mm. Immediately before potting

and planting, each of the four fertiliser materials (DPR, PADPR, DTUK and SSP) was thoroughly mixed with the four kilograms of soil to give fertiliser treatment levels of 0, 25, 50, 75, 100 and 150 mg P kg⁻¹ of soil. Potassium chloride and urea were added to each P fertiliser treatment level to give a constant total content of 300 mg K kg⁻¹ and 300 mg N kg⁻¹ of soil. Other nutrients were added to every treatment at the rate of 125 mg Mg as MgSO₄·7H₂O, 25 mg Zn as ZnSO₄·7H₂O, 7 mg Cu as CuSO₄·7H₂O, and 3 mg B as Na₂B₄O₇·10H₂O per pot (4 kg soil per pot). No lime was applied.

The pots were placed in a greenhouse in a randomised block design with three replicates. Maize was planted as the test crop with three plants per pot after thinning. The pots were watered daily with de-ionized water to maintain soil moisture at approximately 80% of container capacity. As a precaution, saucers were placed at the bottom of the pots to collect any leachate, which would then be returned to the respective pot. The air temperature in the greenhouse ranged from 16 to 26°C. To minimize the effect of the pot location on maize growth, the pot position within each block was randomly rearranged on a weekly basis.

Six weeks after planting, the aboveground parts of the plants were harvested, dried in an oven to constant weight at 65°C, weighed, and ground to less than 3 mm. The ground plant samples were digested in 2:1 HNO₃:HClO₄ mixture, and P in

the samples was measured by the ammonium molybdate ascorbic acid method (Watanabe and Olsen, 1965).

Data analysis. Analysis of variance to determine the significance of treatment effects was performed using the Statistical Analysis System (SAS, 1990). The analysis for dry matter yield of and P uptake by maize was based on factorial treatment combinations in a randomized complete block design. The analysis of variance results were expressed as percentage of total sum of square (SS) to show the extent of contribution of each source of variation to the total variance (Colwell, 1985; Chien *et al.*, 1988; Hellums *et al.*, 1989). Fisher's protected least significant difference (LSD) was used to make pairwise comparisons of treatment means (Steel and Torrie, 1980). The relationships between dry matter yield or P uptake and fertiliser P rate applied were expressed as follows:

$$Y_i = \beta_0 + \beta_1 X + \Sigma_i$$

where Y_i is the dry matter yield or P uptake obtained with source i, X the rate of P applied, β₁ the regression coefficient of the response function, β₀ the intercept, and Σ_i the error term of the fitted model. A t-statistic was used to test the null hypothesis H₀: β₁ = 0, against an alternative H₁: β₁ ≠ 0 for individual regression coefficients for the different P sources. The standard errors (SEβ)

TABLE 1. Selected characteristics of P fertiliser sources used in the experiment

P Source†	Total P ₂ O ₅ , %	% of total P ₂ O ₅ Soluble in	
		Water	Citrate‡
DPR	33.3	0.0	2.4
PADPR	25.2	34.5	4.4
DTUK	16.6	39.8	16.3
SSP	19.4	77.3	18.6

†DPR = Finely ground Dorowa phosphate rock (to pass 0.150-mm screen)

PADPR = Partially acidulated Dorowa phosphate rock (50 % H₂SO₄)

DTUK = Compacted DPR + TSP + urea + KCl (N:P₂O₅:K₂O = 1:1:1)

SSP = Single superphosphate (commercial grade)

PADPR and DTUK were in granular form (<3.36, >1.19 mm) and the P ratio of DPR:TSP was 50:50 for the compacted material (DTUK)

‡Does not include water-soluble P

of estimate for regression coefficients (β_i) were used to test whether the regression coefficient of a P source was statistically different from that of the other sources (Chien *et al.*, 1986).

To evaluate the efficiency of response to fertiliser over the range of rates used, a Relative Crop Response index (RCR_i) which is defined as the ratio of two regression coefficients was used (Chien *et al.*, 1990). The ratio represents the increase of yield or P uptake as compared with a standard source per unit of P fertiliser applied (Menon and Chien, 1990). It is mathematically expressed as:

$$RCR_i = (\beta_i / \beta_{SSP}) \times 100$$

where β_i is regression coefficient of a P source and β_{SSP} the regression coefficient of the standard (SSP) used. The RCR_i for dry matter yield or P uptake for a given source of P was then calculated against SSP as 100%. Statistical differences between regression coefficient values also meant significant differences between RCR_i values.

RESULTS

The analysis of variance (Table 2) shows significant variations ($P = 0.01$) in dry matter yield and P uptake due to P fertiliser rates, P fertiliser sources and the interactions between the

rates and the sources. The effects of P sources, and P rates accounted for 89 and 88% of the variations in dry matter yield and P uptake, respectively.

For the PADPR, DTUK, and SSP treatments, dry matter yield and P uptake by maize increased as P rate increased. On average, DTUK treatment resulted in highest dry matter yield followed by SSP (Table 3). Maize P uptake was highest for SSP treatment. Dorowa phosphate rock had no effect on dry matter yield and P uptake. The regression estimates for dry matter yield and P uptake are presented in Table 4. The regression model $Y_i = \beta_0 + \beta_i X + \Sigma_i$ explained 93% of the variations in maize dry matter yield and 94% in P uptake. Since there was no response in dry matter yield or P uptake from soils treated with DPR, there was no dry matter or P uptake response function. Therefore, the RCR value for DPR was zero because its β_i was zero. By analyzing the regression coefficients of response functions of P sources, it was found that there were significant differences among P uptake response functions for PADPR, DTUK and SSP. The SSP treatment had the highest rate of P uptake increase per unit applied P; whereas the DTUK treatment had the highest rate of dry matter increase per unit applied P. There was, however, no significant difference between the dry matter response functions of SSP and DTUK (Table 4).

TABLE 2. Analyses of variance of dry-matter yield and P uptake by maize in a greenhouse experiment after six weeks†

Source of Variation	df	Dry matter yield		P Uptake	
		—	— % of total adjusted SS	—	—
Replicates	2	0.07		0.10	
Rates	5	33**		34**	
Check vs P rate	1	7.4**		7.0**	
P rates	4	26**		27**	
P Sources	3	56**		54**	
P Sources x P Rates	12	8.8**		9.8**	
Error	40	1.9		2.0	
CV(%)		12		14	
General Mean		14 g pot ⁻¹		30 mg pot ⁻¹	
Standard Error		1.7 g pot ⁻¹		4.2 mg pot ⁻¹	
R ²		0.98		0.98	

†The results are expressed as a percentage of the adjusted total variation by using a Type 1 sum of squares.

**Significant at 0.01 probability level

Agronomic effective of novel phosphate fertilisers

The efficiency of response to P sources across the range of P rates studied was indicated by Relative Crop Response Index (RCR_i) values (Table 4). The RCR_i values for PADPR, DTUK and SSP in increasing dry matter yield of and P uptake by maize show that partially acidulating DPR or compacting a mixture of DPR + TSP + urea + KCl, significantly increased the effectiveness of DPR.

DISCUSSION

The reason for the agronomic ineffectiveness of DPR as a fertiliser material can be explained in terms of the inherent chemical and mineralogical composition of the igneous Dorowa rock. Apatites from igneous sources have low reactivity as evidenced by their low neutral ammonium citrate P₂O₅ solubilities of below 2% of rock compared to

TABLE 3. Dry matter yield and P uptake of maize obtained with DPR, PADPR, DTUK, and SSP

P Source	Rate of P application (mg P kg ⁻¹ soil)						Mean†
	0	25	50	75	100	150	
----- Dry matter yield (g pot ⁻¹) -----							
Check	2.23						2.2d
DPR		2.2	2.6	2.0	2.2	2.8	2.4d
PADPR		6.4	10.6	15.3	18.8	24.0	15.1c
DTUK		10.6	16.8	23.7	25.0	29.7	21.2a
SSP		9.7	12.7	21.4	24.1	29.2	19.4b
----- P uptake (mg pot ⁻¹) -----							
Check	1.94						1.9d
DPR		2.2	2.5	2.1	2.2	2.9	2.4d
PADPR		10.6	20.3	32.3	41.3	47.6	30.4c
DTUK		18.9	33.8	47.4	55.7	65.2	44.2b
SSP		19.4	31.4	51.2	61.5	75.5	47.8a

†Mean values followed by the same letter are not significantly different at 0.05 probability level

Dry matter yield LSD_{0.05} = 1.26; P uptake LSD_{0.05} = 3.20

TABLE 4. Response equations and Relative Crop Response Index (RCR_i) values obtained with applied P rates (X) from different sources

P Source	Response Equations and RCR _i Values			
	Dry matter yield (DMY)		P Uptake (PUPT)	
	(g pot ⁻¹)	RCR _i †	(mg pot ⁻¹)	RCR _i
PADPR	DMY = 4.01 + 0.139X(b)‡	74.7b	PUPT = 5.73 + 0.307X(c)	60.0c
DTUK	DMY = 4.01 + 0.120X(a)	107.5a	PUPT = 5.73 + 0.455X(b)	89.0b
SSP	DMY = 4.01 + 0.186X(a)	100.0a	PUPT = 5.73 + 0.511X(a)	100.0a

†Same lower case letter within columns indicates no significant difference between regression coefficients of the given equations, at 0.05 probability level

‡DMY and PUPT equations followed by the same letter in brackets within columns are not significantly different from each other (SE_β = 0.009 for DMY; SE_β = 0.021 for PUPT; Number of observations = 60)

sedimentary sources such as North Carolina phosphate rock; one of the most reactive rocks known that has P_2O_5 solubility as high as 7.2% of rock (Lehr and McClellan, 1972).

Partial acidulation of phosphate rock is one way to increase water solubility and citrate-solubility (McLean and Wheeler, 1964; McLean *et al.*, 1965; McLean and Balan, 1967; McLean and Logan, 1970; Hammond *et al.*, 1986). The acidulation of Dorowa rock with 50% H_2SO_4 increased its water solubility from 0 to 34.5% of total P_2O_5 and increased citrate solubility from 2.4 to 4.4 % of total P_2O_5 (Table 1). These changes in solubility resulted in increased dry matter yield of and P uptake by maize from 0 to 75% and 60%, respectively, with respect to SSP. In other words, partial acidulation at the 50% level with H_2SO_4 resulted in agronomic effectiveness that was greater than 50% of SSP which is 100% acidulated with H_2SO_4 . For example, the dry matter yield of SSP at 50 mg is less than that of PADPR at 100mg P kg^{-1} of soil even though at those rates the PADPR and SSP theoretically have the same amount of water plus citrate soluble P.

The significant increase in agronomic effectiveness of PADPR compared to unreacted phosphate rock supports the suggestions by Terman and Allen (1967) and Hammond *et al.* (1980) that water solubility of a partially acidulated PR is the factor responsible for increased crop response. McLean *et al.* (1965) concluded that acidulation solubilises part of the P in phosphate rock and, in addition, more P is solubilised from the rock by the acidity (H_3PO_4) produced when monocalcium phosphate undergoes hydrolysis in the soil. Another explanation for the increased effectiveness of PADPR with respect to DPR is that the water-soluble P components of PADPR promote early plant-root development, which enables the plant to use the unacidulated PR component more effectively than plants treated with PR alone. Such "starter effect" of the water-soluble P on the utilization of PR by the plant was demonstrated by Chien and Hammond (1988).

Although the agronomic effectiveness for PADPR was not as high as that of DTUK or SSP in this greenhouse study, partial acidulation of unreactive Dorowa rock did greatly increase dry matter yield of and P uptake by maize grown on an acid soil. Partial acidulation at 50% therefore

represents a technology that can improve the agronomic value of Dorowa PR at a lower cost than would be required to manufacture the conventional 100% acidulated fertiliser from the rock. Similar conclusions were made about other phosphate rocks evaluated by Chien and Hammond (1988), Hammond *et al.* (1986) and Schultz (1986).

There are several possible reasons why compacting a phosphate rock with soluble P fertilisers makes the compacted product more agronomically effective. First, compacting the PR with TSP is equivalent to partial acidulation processes. Both processes produce the same major ingredients, namely, acid-reacted PR and water-soluble P. When the TSP dissolves, the formation of H_3PO_4 induced by the hydrolysis of monocalcium phosphate leads to further acidulation of the unreacted rock. Secondly, the neutralization of acidity (H_3PO_4) by the PR reduces the solubilization of iron and aluminum oxides resulting in reduced fixation of water-soluble P in soils when the oxides are significantly present. Third, the presence of urea in the compacted product is believed to enhance PR dissolution. In the presence of organic matter, urea hydrolysis can hydrolyze soil organic matter because of the increase in pH. Soil organic matter, upon hydrolysis, may supply more organic functional groups or anions such as citrate and oxalate that can effectively chelate Ca^{2+} ions and thus lower Ca^{2+} activity in soil solution and drive the PR dissolution process (Chien, 1979). Fourth, the presence of more than one nutrient in the granule, or briquettes as is the case with DTUK product may increase crop response due to "starter" and "synergistic" effects. The simultaneous presence and availability of multinutrients in a granule has greater agronomic effect on early plant-root development. Leikam *et al.* (1983) proposed that the presence of ammoniacal N fertiliser increases P availability because of the synergistic effect of mixing N and P together. Compacting N and P together should therefore increase the synergistic effect on N and P uptake by enhancing the interaction between N and P in the granules and in soil. The resultant early plant-root development enables the plant to use the unacidulated PR component more effectively (Chien and Hammond, 1988).

The compaction technology has great potential because of several advantages (Lupin and Le, 1983; Govere, 1993; Menon and Chien, 1996). Some of the advantages are:

1. Reduction of energy and capital cost by eliminating use of water, steam, and subsequent drying of moist granules or briquettes.
2. Reduction in the use of acid when compared to SSP, TSP and partial acidulation processes.
3. Compaction reduces the hazard from the dust and it prevents fertilisers from caking in bags and containers.
4. Incorporation of secondary nutrients and micronutrients can be accomplished without difficulty, thus making it easier to apply very small amounts of micronutrients as components of the compacted material.
5. Compaction process can be readily designed for the relatively low production rates attainable with the small compaction units that are available, thus it can be more easily adapted in a developing country.
6. Multi-nutrient compacts tend to have greater agronomic effectiveness than single-nutrient fertiliser sources or partially acidulated phosphate rocks.
7. The compaction can enhance P supply for phosphate rocks that have low reactivity and are high in iron and aluminum oxides.
8. Compaction technology requires minimal technical skills, thus it is most suited for developing countries where skilled manpower is in short supply.

CONCLUSION

The direct application of finely ground DPR as a P source was ineffective. However, when the rock was partially acidulated (50% H_2SO_4) the agronomic effectiveness of the rock in increasing dry matter yield of maize increased to 75% with respect to SSP. Mixing and compacting the rock

with TSP (at P ratio of 50:50), urea and KCl made the product as agronomically effective as SSP in increasing dry matter yield of maize. Therefore, partial acidulation with sulfuric acid, and compaction of a mixture of nonreactive Dorowa rock with soluble fertilisers such as TSP and urea, are technologies that can improve the agronomic value of the DPR.

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