

Response of Italian Ryegrass to Phosphorus in Organic-acid Treated Phosphate Rocks

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Abstract Two phosphate rocks (PRs) from Sri Lanka and Togo were acidulated with 5 M of citric, oxalic or tartaric acid at 2:1 acid:rock ratio for three days. The resulting materials treated with oxalic and tartaric acid were easily dried up and nearly all the P was in water soluble form. The PRs treated with citric acid were rather difficult to dry and contained about 50% of the total P in water soluble form. The effectiveness of the materials as phosphorus sources were compared with that of single superphosphate in acidic granitic regosol with Italian ryegrass as the test crop. All the acid treatments were as effective as single superphosphate in increasing the dry matter yield of Italian ryegrass at 168 days after planting except the citric-acid treated Sri Lanka PR. Phosphorus recovery by the plants from tartaric-acid treated Togo PR was 58%, which was significantly greater ($P < 0.05$) than those from single superphosphate (52%) and tartaric-acid treated Sri Lanka PR (53%). Significantly higher levels of available P (OLSEN P) and pH were found in the cropped soils which received the organic-acid treated PRs than those of the soils which received single superphosphate and control at the end of the experiment. No adverse effect of the organic-acid treated PRs on the growth and nutrient uptake by Italian ryegrass was observed throughout the growth period. These results indicate that at least the tartaric-acid treated PRs could be better sources of P to crops growing on acid soils than single superphosphate.

Key words: citric, organic acids, oxalic, phosphate rock, phosphorus availability, tartaric

INTRODUCTION

Phosphorus (P) is a key nutrient to improve crop production in many developing countries, especially under acidic soil conditions. The major source of P is phosphate fertilizers which are produced exclusively from phosphate rocks (PRs). Although large deposits of PRs occur in many parts of the world, many of them have low reactivity and therefore show poor performance when directly applied to soils. To improve P availability of PRs to crops, PRs are usually acidulated with such mineral acids as sulfuric, nitric, and phosphoric acids to increase water solubility of the P compounds. Many of the areas where PR deposits occur also lack elemental sulfur for making sulfuric acid, another key raw material in making phosphate fertilizers. If some forms of simple treatments of PRs to increase the P

availability without using mineral acids are available, they could be used in the local areas where the cost of the usual phosphatic fertilizers is beyond the reach of many subsistent farmers.

Organic acids have been reported as being able to release P from insoluble P compounds including apatites (STEVENSON, 1967; ASEA *et al.*, 1988), and also facilitate weathering of minerals and rocks (SONG and HUANG, 1988). Almost all of these reported works involved culture solutions and very low acid concentrations. TERMAN *et al.* (1969) granulated PR with oxalic and citric acids at different PR:acid ratios and they observed increases in the P availability of the PR to maize. Higher proportions of the acids in the mixtures increased the plant availability and, oxalic acid was found to be more effective than citric acid. The related data, however, on the plant availability of phosphate rocks that has been acidulated with organic acids are scarce.

In our laboratory, we have been studying the stoichiometric relations in solubilization of PRs by organic acids under various conditions (SAGOE, 1995; SAGOE *et al.*, 1995). As a result, we found high concentrations of tartaric and oxalic acids were very effective in solubilizing the low reactive PRs and to obtain the powdered products which contained high concentration of water soluble P.

The purpose of this study is to assess P availability of the PRs that have been acidulated with 5M citric, oxalic or tartaric acid to Italian ryegrass in comparison with commercial single superphosphate in a greenhouse pot experiment. The adverse effects of the organic-acid treated PRs on crop growth and nutrient uptake were also examined. The results obtained indicated that at least the tartaric-acid treated PRs were as effective as single superphosphate in P uptake by Italian ryegrass.

MATERIALS AND METHODS

Organic acids treatment of phosphate rock (PR)

Phosphate rocks from Togo (total P 163 g/kg, total Ca 349 g/kg) and Sri Lanka (total P 172 g/kg, total Ca 359 g/kg) were treated with 5 M of commercial reagent grade citric, oxalic or tartaric acids at 2:1 acid:rock ratio. Both PRs were of less than 0.015 mm particle size. The mixture was allowed to stand at room temperature for three days. This ratio and acid concentration were arrived at since in our previous experiments the resulting materials were easier to handle and also contain at least 50% water soluble P (SAGOE, 1995). Samples of the mixtures were analyzed for the water soluble P (AOAC, 1984). The treated phosphate materials were aged for at least a week before application to the pots.

Greenhouse pot experiment

The pot experiment was conducted in the greenhouse of Fac. of Appli. Biol. Sci., Hiroshima University from November 1995 to March 1996. Undrained Wagner pots, a/5000 in size, were filled with granitic regosol. The soil was collected from an uncultivated part of the Hiroshima University Farm and had the following properties; pH, 5.9 (1:2.5 soil:water); Olsen P, 2.8 mg P/kg; organic carbon, 0.3g C/kg dry soil. The air-dried soil (4 kg/pot, <2 mm fraction) was mixed with 300 mg N (NH₄NO₃), 300 mg CaCO₃, 100 mg K (KCl), 100 mg Mg (MgSO₄.7H₂O) and the required amount of the acid treated PRs or commercial single superphosphate to give 200 mg P/pot. A treatment without P application was

included as a control. Approximately 50 seeds of Italian ryegrass (*Lolium multiflorum* Lam.) were planted per pot. The nutrients except P were supplemented when necessary. A total of 1.5 g N, 200 mg K and 200 mg Mg per pot was applied throughout the growth period. The moisture content of the pots were maintained at near field capacity by regular watering with deionized water. Each treatment had three replicates and the pots were arranged in a random fashion.

The plants were cut at about 1 cm above the soil surface at 58, 132, days after planting (DAP) and allowed to grow again after each cut. Whole plants were harvested at 168 DAP. The plant materials were dried at 80°C for 48 hours, weighed and ground. Plant samples were digested with 1:1 H₂SO₄:HNO₃ mixture, and phosphorus in the sample was measured by the molybdenum-blue method described by MURPHY and RILEY (1962). Nitrogen contents were determined by the micro-Kjeldahl method, Ca and Mg by atomic absorption spectrophotometry and K by flame photometry. Soil samples from each pot were taken at the end of the experiment and were analyzed for pH (1:2.5, water) and OLSEN P (0.5 M NaHCO₃ extractable P).

Statistical Analysis

One way ANOVA and Duncan's Multiple Range Test (DMRT) were performed. The difference between treatment means were tested at 5% for all sets of data analyzed.

RESULTS

Water soluble P content of the organic-acid treated PRs

Although the original PRs did not contain water soluble fractions of P, the organic acid treatments considerably increased water soluble forms of P in the PRs. The treatments with tartaric acid converted almost all the P in both PRs into water soluble form, while oxalic acid caused 100% water solubility in Sri Lanka PR, but only 56% of the total P in Togo PR. The citric acid treatment of Sri Lanka PR and Togo PR resulted in about 50 and 56% water solubility, respectively. The PRs treated with tartaric and oxalic acid were easily dried up within a few days, but the citric-acid treated PRs were rather difficult to dry within a week under room conditions.

Dry matter yield

The shoot dry matter yield of Italian ryegrass at 58, 132, and 168 DAP are shown in Table 1. The initial growth of Italian ryegrass which received untreated PRs was not much different from that of the control. With time, however, both untreated PRs significantly increased dry matter yield over the control, except Togo PR at 132 DAP, though the effects were much smaller than those of the organic-acid treated PRs and superphosphate.

Application of the organic-acid treated PRs resulted in remarkable increases in the growth and yield of Italian ryegrass throughout the experiment. Among the organic acids, tartaric acid was most effective to increase shoot yield throughout the growth period. At 58 DAP the tartaric- and oxalic-acid treated Togo PR produced the same amounts of dry matter as that of single superphosphate, but the same treatments applied to Sri Lanka PR produced less dry matter than the single superphosphate. At 132 and 168 DAP, the dry matter yield of Italian ryegrass that received organic-acid treated PRs were not different significantly regardless of the acid treatment and PR, and were comparable to single super

Table 1 Shoot dry matter yield (g/pot) of Italian ryegrass 58, 132 and 168 days after planting. The phosphate rocks (PR) were treated with 5M organic acids for three days.

Treatment	Days after planting		
	58	132	168
Sri Lanka PR			
Untreated	1.9e	12.4b	8.1cd
Citric acid	9.4d	23.2a	9.3bc
Oxalic acid	12.3c	22.7a	11.1ab
Tartaric acid	12.5bc	24.6a	11.3ab
Togo PR			
Untreated	1.3ef	6.0c	6.3d
Citric acid	11.8c	22.9a	10.8ab
Oxalic acid	13.4ab	22.5a	10.6ab
Tartaric acid	13.6a	24.6a	12.2a
Superphosphate	13.7a	25.2a	11.5a
Control	0.8f	1.4c	1.0e

Column values followed by the same letter are not significantly different (DMRT, 0.05 level).

phosphate application except the citric-acid treated Sri Lanka PR.

Total shoot dry weight (sum of three cuts) and the root dry weight are shown in Fig. 1. The shoot dry weight ranged from 13.6 g/pot (Togo PR) to 50.5 g/pot (tartaric-acid treated Togo PR). Total dry matter accumulation for all the acid treatments except citric-acid treated Sri Lanka PR were not significantly different from that for single superphosphate. Untreated Sri Lanka PR produced more dry matter than the Togo PR, which tended to show better performance in the organic-acid treated PRs. The root dry weight showed a similar pattern to shoot dry weight.

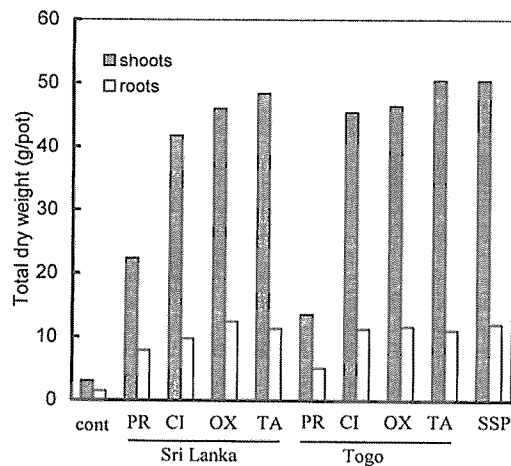


Fig. 1 Total dry matter yield of Italian ryegrass 168 days after planting. The phosphate rocks were treated with 5 M citric (CI), oxalic (OX) or tartaric (TA) acid for three days. SSP: single superphosphate.

Phosphorus concentration

All the P application treatments increased P concentration in the shoots of Italian ryegrass at 58 DAP (Table 2). During this time, P concentration increased from about 1.4 g/kg in the untreated rocks to about 4.5 g/kg in the tartaric acid treatments, which were not significantly different from that in single superphosphate. For Sri Lanka PR, P concentration of the shoots in the oxalic and tartaric acid treatments were not different, but greater than that of the citric acid treatment. In Togo PR, however, the shoot P concentration was in the order of tartaric > oxalic > citric acid. The shoot P concentration decreased with time, except the untreated Togo PR. There were no significant difference among the treatments for both shoot and root P concentration at 168 DAP.

Table 2 Phosphorus concentration (g P/kg dry matter) in shoots and roots, and uptake by shoots of Italian ryegrass at 58, 132 and 168 days after planting.

Treatment	P in shoots			P in roots	P uptake (mg P/pot)		
	Days after planting			168	Days after planting		
	58	132	168		58	132	168
Sri Lanka PR							
Untreated	1.4de	1.1cd	0.8	0.7	2.8e	12.1e	6.2bc
Citric acid	1.9cd	0.7cd	0.6	0.6	17.5d	16.0de	5.7c
Oxalic acid	4.3a	1.7ab	1.0	0.8	52.9b	37.9b	10.5a
Tartaric acid	4.5a	1.7ab	1.0	0.8	56.1b	41.5b	10.8a
Togo PR							
Untreated	1.3e	1.9a	0.9	0.9	1.7e	11.4e	5.4c
Citric acid	2.1c	0.8cd	0.6	0.5	24.5d	17.7de	5.9c
Oxalic acid	3.3b	0.9cd	0.5	0.6	43.9c	20.9d	5.4c
Tartaric acid	4.4a	2.1a	0.7	0.8	60.2ab	51.7a	8.2b
Superphosphate	4.9a	1.2bc	0.7	0.8	66.3a	30.3c	8.2b
Control	0.7f	0.6d	0.4	0.8	0.5e	0.8f	0.4d

Column values followed by the same letter are not significantly different (DMRT, 0.05 level).

Phosphorus uptake

At 58 DAP, the maximum amount of P uptaken by the plants was 66.4 mg P/pot in single superphosphate (Table 2). Only the tartaric-acid treated Togo PR was comparable to this amount. At 132 DAP, however, all the tartaric acid treatments and oxalic-acid treated Sri Lanka PR showed significantly higher P uptake than single superphosphate. At 168 DAP, the amount of P uptake in the oxalic- and tartaric-acid treated Sri Lanka PR were greater than that in single superphosphate. It is noteworthy that the amount of P uptake by the plants from the untreated PRs was comparable to those from the citric-acid treated PRs at 132 and 168 DAP.

The total P uptake by Italian ryegrass at the end of the experiment is shown in Fig. 2A. The highest amount of total P uptake was from the tartaric-acid treated Togo PR (118.3 mg P/pot). All the acid treatments except citric acid were statistically not different from single superphosphate. Generally the pattern of P uptake by the roots was similar to that of the shoots, though the amounts were much smaller than those in shoots.

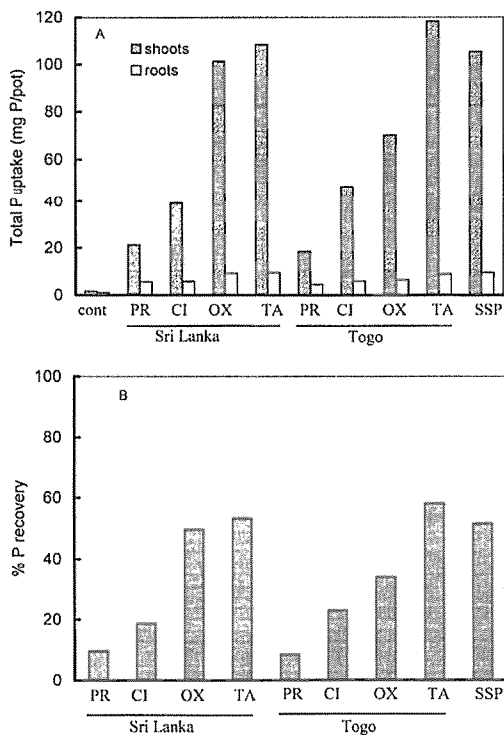


Fig. 2 Phosphorus uptake (A) and recovery (B) by Italian ryegrass 168 days after planting. The phosphate rocks were treated with 5 M citric (CI), oxalic (OX) or tartaric (TA) acid for three days. SSP: single superphosphate.

Phosphorus recovery

The proportion of the applied P that was recovered by Italian ryegrass was 19, 50, and 53% for citric-, oxalic-, and tartaric-acid treated Sri Lanka PR, respectively (Fig. 2B). In Togo PR the percentage recovery was 23, 34, and 58%, respectively. A significantly higher (0.05 level) P recovery rate was found in the tartaric-acid treated Togo PR than single superphosphate (52%) and other organic-acid treated PRs. In the untreated PRs, only about 10% of the applied P was recovered by the plants during 168 days of growth.

Nutrient status of the plants

The concentration of N, K, Ca and Mg in the shoots of Italian ryegrass is shown in Table 3. Concentrations of these nutrients in the plants that received the organic-acid treated PRs were generally similar to those that received single superphosphate. The plants in the untreated PRs and control, which showed poor growth, tended to show higher N concentration.

DISCUSSION

The incubation of the powdered PRs mixed with 5 M tartaric, oxalic or citric acid at 2:1 acid: PR ratio at room temperature for three days considerably increased water soluble form of P in the PRs. Although an equimolar concentration of the organic acids was used,

Table 3 N, K, Ca and Mg concentration (g element/kg dry weight) in the shoots of Italian ryegrass at 58, 132 and 168 days after planting.

Treatment	N			K			Ca			Mg		
	58	132	168	58	132	168	58	132	168	58	132	168
Sri Lanka PR												
Untreated	42.2a	23.5b	14.8bc	18.2b	11.8b	10.7d	4.4bc	4.4c	5.2ab	4.3ab	4.4ab	4.3
Citric acid	30.1b	11.0c	17.9b	21.0a	9.5bcd	17.2abc	2.2c	2.2b	5.6ab	3.4cd	2.8b	3.8
Oxalic acid	23.8c	10.5c	15.4bc	15.1c	8.4cd	12.2bcd	2.2c	5.6ab	4.4ab	3.3cd	4.1ab	4.4
Tartaric acid	20.1c	10.8c	14.3bc	14.8c	8.4cd	12.0cd	5.6b	2.2b	4.4ab	3.8bc	3.8b	4.6
Togo PR												
Untreated	34.6b	35.2a	26.9a	13.9cd	17.7a	18.2ab	3.7bc	4.5b	9.0a	3.3cd	4.1ab	5.1
Citric acid	20.7c	11.1c	14.8bc	14.6c	10.6bc	13.1bcd	2.2c	2.2b	2.2b	2.6b	3.3b	3.8
Oxalic acid	21.6c	12.0c	15.1bc	12.8cd	9.4bcd	8.7d	2.2c	2.2b	2.2b	3.3cd	3.4b	4.6
Tartaric acid	20.3c	12.0c	12.0c	12.9cd	9.5bcd	10.2d	5.6b	4.5b	5.9ab	3.3cd	3.1b	4.8
Superphosphate	21.6c	11.4c	12.5c	13.1cd	7.4d	10.2d	9.0a	2.2b	2.2b	4.9a	3.1b	4.6
Control	34.3b	36.9a	19.1b	11.6b	16.2a	19.4a	3.7bc	10.0a	9.0a	3.8bc	5.6a	5.2

Column values followed by the same letter are not significantly different (DMRT, 0.05 level).

the levels of water soluble P in the PRs were different among the organic acids. Only tartaric acid converted almost all the P in both phosphate rocks into water soluble form. Oxalic acid was also effective to solubilize Sri Lanka PR, but not Togo PR. Citric acid converted about half of the total P in both PRs into water soluble form. This solubility pattern is consistent with that observed when the same acids were used at lower concentrations (SAGOE, 1995; SAGOE *et al.*, 1995). Citric acid is less effective to solubilize the PRs, possibly because this acid has lower ability to precipitate Ca in the solutions (unpublished data). The reason why Togo PR is resistant to resolution by oxalic acid solution is not known.

Acidulation of the PRs with the organic acids considerably increased the P availability of the PRs to Italian ryegrass by significant margins over the control and the untreated PRs. The treatments of Sri Lanka PR with oxalic and tartaric acid and Togo PR with tartaric acid resulted in similar accumulation of dry matter, P uptake and P recovery rates by Italian ryegrass to application of single superphosphate. The oxalic-acid treated Togo PR and citric-acid treated PRs, however, were less effective in increasing P uptake by the plants. Since a high positive correlation ($r=0.92^*$) was found between water solubility of the organic-acid treated PRs and P uptake by plants, it may be possible to assess P availability of the organic-acid treated PRs from the water solubility.

The citric acid treatment of both PRs resulted in the lowest amount of water soluble P among the organic acids. The resulting 50% water soluble P material was, however, enough to produce about 80% (Sri Lanka PR) and 90% (Togo PR) of the possible dry matter yield by single superphosphate. The P supply by citric-acid treated PRs to the plants might be almost enough for the P requirement of the plants under the tested conditions.

On the other hand, the untreated PRs could not supply enough amount of P to the plants, especially during the initial growth period. The two phosphate rocks are reported to be poor in supplying P when applied directly to the soil (PATHIRATNA *et al.*, 1989; CHIEN and FRIESEN, 1992). In order to use these PRs more effectively for especially the initial growth of plants, some forms of acid treatment of the PRs will be necessary. After the first cut, however, P concentration and uptake of the plants increased remarkably. The dry matter production was also increased by the application of the untreated PRs as compared with the control after the first cut. This may indicate that the untreated PRs are gradually solubilized in the soil and/or that Italian ryegrass might develop the ability to solubilize the PRs. If these mechanisms of PR solubilization work in the soils, it may not be necessary to convert all the P in PRs to water soluble forms.

Although we used high organic acid concentrations to treat the PRs, we did not detect any adverse effect of the acids on the growth and nutrient uptake of the plants throughout the experiment. The growth, yield and nutrient uptake characteristics of the plants that received the organic-acid treated PRs were generally similar to that of the single superphosphate. The organic acids applied might be easily metabolized by soil microorganisms.

High P recovery rates were observed for all the P applications except the untreated PRs under the tested conditions. This will be partially due to the low P retention characteristic of the granitic regosol used and also due to the prolonged growth period in this experiment. Since a larger amount of the applied P will be taken up by plants under these

conditions, the P availability of the organic-acid treated PRs should be reexamined by using the soils of high P fixation characteristics.

Reactions of the organic-acid treated PRs in soils were also different from that of single superphosphate. Available P (OLSEN'S P) concentrations of the cropped soils that received the oxalic- and tartaric-acid treated PR were either equal to or higher than that of single superphosphate (Table 4). The pHs of the cropped soils that received the organic-acid treated PRs were higher than those of the soils that received single superphosphate. These effects indicate a favorable condition especially in soils with low pH and high P-retention capacity. These observations however need to be looked at further in order to fully understand the reactions involved.

Table 4 Olsen P and the pH of the soil 168 days after cropping with Italian ryegrass.

Treatment	Olsen P (mg P/kg dry soil)	pH (1:2.5, water)
Sri Lanka PR		
Untreated	3.9g	6.00a
Citric acid	6.9e	6.25a
Oxalic acid	11.6bc	6.06a
Tartaric acid	12.5ab	6.07a
Togo PR		
Untreated	6.9e	5.32b
Citric acid	8.5d	6.07a
Oxalic acid	10.4c	6.07a
Tartaric acid	13.8a	6.07a
Superphosphate	11.3bc	5.53b
Control	3.3f	5.23b

Column values followed by the same letter are not significantly different (DMRT, 0.05 level).

The results of this experiment do suggest that the organic-acid treated PRs are better sources of P for plant use. At least the tartaric acid-treated PRs resulted in the better performance characteristics and residual effects as compared with single superphosphate. By quite simple procedures, just mixing powdered PRs with the organic acid and leaving them for several days, the P availability of the PRs to plants were greatly improved. Therefore, if we can obtain enough amount of these organic acids at reasonable costs, such organic-acid treated PRs will help the farmers to improve crop production in many developing countries.

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有機酸で処理したリン鉱石中のリンに対する イタリアンライグラスの反応

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スリランカとトーゴ産のリン鉱石に5Mのクエン酸，シュウ酸，酒石酸溶液を1:2の割合で加え，3日間反応させた。シュウ酸と酒石酸で処理したリン鉱石は容易に乾燥し，ほとんど全てのリン酸が水溶性となった。クエン酸で処理したリン鉱石は乾燥しにくく，また水溶性リン酸の割合も全リン酸の50%程度であった。これら有機酸で処理したリン鉱石のリン源としての有効性を，イタリアンライグラスを供試作物とし，マサ土(花崗岩風化土壌)を用いた土耕ポット試験で過磷酸石灰と比較した。クエン酸処理したスリランカ産リン鉱石を除き，他の全ての有機酸処理リン鉱石は播種後168日目のイタリアンライグラスの乾物収量を過磷酸石灰と同程度に増加させた。酒石酸で処理したトーゴ産リン鉱石におけるリンの回収率は58%で，過磷酸石灰(52%)や酒石酸処理スリランカ産リン鉱石(53%)より有意に高かった。有機酸処理したリン鉱石を与えた区の跡地土壌の可給態リン(Olsen P)濃度とpHは，過磷酸石灰施与区や無リン酸区(対照区)より有意に高かった。実験期間を通じてイタリアンライグラスの生育と養分吸収に及ぼす有機酸処理リン鉱石施与の悪影響は認められなかった。以上の結果から，酸性土壌に生育する作物に対し，少なくとも酒石酸処理したリン鉱石のリン源として有効性は過磷酸石灰より良好であると判断された。

キーワード：クエン酸，有機酸，シュウ酸，リン鉱石，リン酸可給性酒石酸