

## Research Article

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# Effect of acidulated levels and application techniques of rock phosphate on phosphorus use efficiency and yield of wheat in calcareous soil of Peshawar-Pakistan

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### Abstract

The present study investigated the effect of different levels of acidulated rock phosphate (RP), liquid or solid and application methods on phosphorus use efficiency and wheat yield under calcareous soils of Peshawar during 2015-16. Treatments included acidulation of 100 kg RP with 7.5, 15, 30 and 45 kg H<sub>2</sub>SO<sub>4</sub> equivalent to 25, 50, 100 and 150 % acidulation, applied in the form of either liquid or solid as single (no split) or two equal splits at sowing and knee height stage. The rate of P in all treatment was equal to 90 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>. Our results showed that acidulation levels significantly increased wheat yield, post-harvest soil and plant tissue phosphorus optimum at 100 % acidulation level. Solid form of the acidulated RP was more effective than liquid revealing that lesser the contact of soil, could improve P availability from the source. It was further affirmed by the split application where it significantly increased the crop yield and P availability over no split irrespective of solid and liquid form. Our results suggest that solid RP with 100 % acidulation applied in two splits improved the crop yield, P use efficiency and could substitute commercial fertilizers under the prevailing soil and agro-climatic conditions.

**Keywords:** Acidulated rock phosphate; Application methods; Calcareous soils; Solid and liquid

### Introduction

The soils of Pakistan are deficient in available phosphorus (24), mainly associated to alkalinity and calcareousness where it forms complexes with Ca and Mg (8). Nearly 90 percent of Pakistani soils are alkaline (pH >7.0) or calcareous (CaCO<sub>3</sub>> 3.0%) besides low level of phosphorus. When phosphorus fertilizers are applied, a

small amount of phosphorus becomes available to plant while the rest goes to unavailable forms. Soil solution P is readily available source [1] but it is highly vulnerable to make complexes with cations like Fe, Al, Ca and Mg or adsorbed on the soil matrix [2]. Its deficiency causes stunted growth and delay in maturity (24,31 and 20). Use of P fertilizers assures higher yield

production if the method of fertilizer application is proper and on time [3]. To alleviate adequate supply of P fertilizer, use of rock phosphate could be the most affordable option in poor farming society. Each year millions of rupees are spent on import of phosphatic fertilizers to Pakistan. It is a good idea to use local deposits of RP that will not only increase crop production but will also save money. In Pakistan deposits of rock phosphate (RP) are located in Hazara and Khyber Pakhtunkhwa. However, the direct application of RP in our alkaline calcareous condition may not be fruitful. In many areas of the world it is applied directly but its solubility is very low (<1%) in soil solution, hence unable for plant to uptake [4]. Researchers have tried to improve the solubility of RP by mixing RP with various organic and inorganic materials before application to soil. These amendments and materials include farmyard manure, composts, effective micro-organism (EM), acids, methods and time of applications. The alternate option could be converting insoluble P in RP to soluble P by treating RP with phosphoric acid, nitric acid or sulphuric acid along with sufficient amount of water to improve reaction. Promising results were obtained by applying acidulated RP to wheat and maize crops under alkaline calcareous soils [5]. However, acids cost and availability in market can also be considered for viable economical recommendations. Acidification of RP with different levels of sulphuric acid ( $H_2SO_4$ ) or nitric acids ( $HNO_3$ ) leads to the formation of mixture of water and citrate soluble and insoluble P and increase dissolution of P from RP [6]. Partially acidulated RP can be obtained by treating RP with limited acids (HCl or  $H_2SO_4$ ). However, with increase in acid levels produced more water and citrate soluble phosphate as compared to 25 and 50% acidulation [6]. After treatment of acids with RP, granules were prepared from the mixture, dried and used for application. The whole purpose of all the strategies is to improve efficiency and availability of P to

standing crops, for higher yield and food security. Thus, the objectives of the present study were to investigate, (1) the effect of different levels of acidulated rock phosphate (RP), (2) applied liquid or solid, (3) and application methods on P use efficiency and wheat yield.

## Materials and methods

### Field experiment

A field experiment was conducted at Newly Developmental Farm, The University of Agriculture Peshawar, Pakistan during 2015-2016. The soil of experimental field was alkaline, strongly calcareous but non-saline in reaction with pH, lime and EC values of 7.79, 17.3 % and  $0.19 \text{ dS m}^{-1}$ , respectively. Furthermore, the soil under study was deficient in organic carbon (0.53%) and AB-DTPA extractable P ( $1.98 \text{ mg kg}^{-1}$ ). Such soils have been reported to be highly responsive to P fertilizer application [1]. Four levels of acids (25, 50, 100 and 150%) along with two forms of rock phosphate (RP) fertilizers (liquid and solid) were applied to soil either in single dose or in two split applications. The experiment was laid out in factorial Randomized Complete Block Design (RCBD) with three replications. Siran wheat variety was sown in plot size of  $3 \text{ m} \times 3.5 \text{ m}$  with row-row distance of 30 cm at optimum soil moisture condition. At sowing time, the required amount of acidulated RP was applied to soil as broadcast in case of solid and spray in case of liquid. The remaining half of the split doses (solid and liquid) were banded in the rows. The field was irrigated just after 2nd split application. In all cases, the treatments received equal net amount of acidulated RP as  $90 \text{ kg P}_2\text{O}_5 \text{ ha}^{-1}$ . All plots received basal doses of N and K as a urea and Murate of Potash (MOP). The field was irrigated according to weather condition and when needed.

### Acidification of Rock Phosphate (RP)

For acidulation of RP, the grinded 100 kg RP was treated with the require amount of 7.5, 15, 30 and 45 kg  $H_2SO_4$  along with sufficient amount of water equivalent to 25,

50, 100 and 150 % respectively. The acidification of both solid and liquid form was done at 4 levels on the basis of complete reaction/dissolution of P in the RP. Usually the SSP fertilizers are prepared on commercial scale at 100:60 RP:H<sub>2</sub>SO<sub>4</sub> water for highest dissolution of RP [6, 7].

#### Soil analysis

Soil sample from 0-20 cm depth were collected at spiking and post-harvest stage to evaluate the treatment role in P status of the soil. Soil pH and AB-DTPA extractable P in soil were determined according to previously reported methods [8].

#### Plant analysis

At harvesting time, one square meter was randomly selected in each plot for measuring dry matter yield and grain yield. Furthermore, leaves samples at plant maturity stage were collected for measuring nutrient concentrations. Leaves were washed with distilled water, air dried in oven at 60-70°C for 48 h. After grinding, the samples were digested with nitric acid (HNO<sub>3</sub>) and perchloric acid (HClO<sub>4</sub>) as described by [8]. Then, P concentration was determined by using spectrophotometer.

### Results and discussion

#### Grain yield

The wheat grain yield was significantly increased by extent of acidulation, forms of RP fertilizer (solid and liquid) and application methods; however their interactions were non-significant at  $p < 0.05$  (Table 1). When the values were averaged across the time of application and form of acidulated RP, acidulated RP of 100 and 150% produced grain yield (3857 kg ha<sup>-1</sup>) and (3966 kg ha<sup>-1</sup>) which were higher than 25% acidified RP with grain yield of 2884 kg ha<sup>-1</sup>. When the values were averaged across acidulation levels and time of application, the granular solid fertilizer produced higher grain yield (3611 kg ha<sup>-1</sup>) over the liquid fertilizer (3393 kg ha<sup>-1</sup>). In application methods, split doses of RP lead to comparatively more yield 3681 kg ha<sup>-1</sup> than no split application (3324 kg ha<sup>-1</sup>) on average basis. As the yield parameters are directly associated with availability of

phosphorus, acidulated RP could release P in a better way for plant growth. Similar results were also reported by [9, 10] that acidulation of RP resulted in better grain yield. The higher performance of the solid RP could be attributed to less contact of granular RP against the liquid acidified RP where much of it would have been adsorbed on the matrix or formed complexes with Ca and Mg in the prevailing alkaline soil [11]. The performance of solid than liquid RP may be due the better placement of the P fertilizer which influences the growth parameter positively and increased grain yield, where P applied in solid form as described by Ali et al. (2004). Our results are also comparable with [12-14] who reported that P applied as a split dose at planting stage or different growth stages by the method of side dressing, or 2nd application with irrigation, resulted more grain yield as compared to full application at sowing time. Similar findings were also reported by [15-18].

#### Dry matter yield

Dry matter yield of wheat was significantly increased over control with acidulation levels of RP, forms of P fertilizers and application methods at  $P < 0.05$  (Table 2). The interactions were non-significant. The plots receiving 100 and 150 % acidified RP gave higher dry matter yield of 8534 kg ha<sup>-1</sup> and 8748 kg ha<sup>-1</sup> respectively, while lowest was obtained 5362 kg ha<sup>-1</sup> in control plots. It is suggested that 100 % acidulation was sufficient for full acidification of RP in term of plant growth. Furthermore, liquid RP produced lower dry matter yield (7476 kg ha<sup>-1</sup>) while solid RP produced higher dry matter yield (8135 kg ha<sup>-1</sup>) respectively. Regarding application methods, split application yielded 7969 kg ha<sup>-1</sup> dry matter yield as compared to 7643 kg ha<sup>-1</sup> with no split application when values were averaged over acidulation levels and forms of RP fertilizers. Application of RP+50 % acid enhanced biomass yield of wheat crop as compared with other P fertilizer suggesting that acidulation could be the best option under prevailing soil situation

[9]. The best performance of 100% acid level with RP could be due to more release of P from RP for plants as concluded by [19-21] that increase H<sub>2</sub>SO<sub>4</sub> concentration in RP, releases more P for plants. Solid fertilizer was found superior in improving wheat yield and yield components. This may be because of better availability of P to

wheat roots, as RP granules were properly incorporated into soil through deep placement, and irrigation was done on time [22, 23]. Split application of P fertilizers with deep placement method (banding) increased the biomass yield of wheat crop [1, 24].

**Table 1. Grain yield of wheat as affected by different acid levels, forms of rock phosphate fertilizer and application methods**

Application Methods	Grain Yield (kg ha <sup>-1</sup> )			
	% Acidity	Fertilizer Forms		Mean
		Liquid	Solid	
	25	2802	2966	2884 c
	50	3116	3487	3301 b
	100	3767	3947	3857 a
	150	3888	4045	3966 a
No Split		3194	3454	3324 b
Split		3592	3769	3681 a
	Mean	3393 b	3611 a	Significance
Planned Mean Comparison				***
Control	2571			
Rest	3502			
LSD (0.05)		Interactions		
Fertilizer Forms (F)	***	F x AM		NS
App. Methods (AM)	***	F x L		NS
Acid Levels (L)	155	AM x L		NS
		F x AM x L		NS

**Table 2. Dry matter yield of wheat crop as influence by various degrees of acidulation, forms of RP fertilizers and application methods**

Application Methods	Dry matter Yield (kg ha <sup>-1</sup> )			
	% Acidity	Fertilizer Forms		Mean
		Liquid	Solid	
	25	6012	6392	6202 c
	50	7410	7822	7616 b
	100	8168	8597	8382 a
	150	8427	8781	8604 a
No Split		7163	7508	7336 b
Split		7846	8287	8067 a
	Mean	7504 b	7898 a	
Planned Mean Comparison				
Control	5362			
Rest	7701			
LSD(0.05)		Interactions		
Fertilizer Forms (F)	***	F x AM		NS
App. Methods (AM)	***	F x L		NS
Acid Levels (L)	300	AM x L		NS
		F x AM x L		NS

### Post-harvest soil pH

The post-harvest soil pH as influenced by acidification levels of RP, forms of RP fertilizers and application methods showed non-significant result and their interactions were also found non-significant except only the controls vs rests showed slightly significant result (Table 3). Control plots had significantly higher pH with value of 7.70 as compared to the overall mean of all treatments with value of 7.56 indicating that P fertilizer reduced the soil pH at postharvest stage of the crop. The decrease in the pH could be associated to CaSO<sub>4</sub> by production during the reaction of RP with H<sub>2</sub>SO<sub>4</sub> as well as to surplus acid remained unreactive in the process. However, the pH changes among the treatments were non-significant that could be due to higher

changes among the replications. It has been reported that Pakistani soils are mostly alkaline in reactions with pH ranges from 7.5 to 8.5. The reason for this may be the calcareous parent materials (90 % soils of Pakistan are calcareous [25] which include Ca<sup>2+</sup>, Mg<sup>2+</sup>, SO<sub>4</sub><sup>2-</sup>, CO<sub>3</sub><sup>2-</sup> and HCO<sub>3</sub><sup>2-</sup> as well as greater proportion of OH<sup>-</sup> than H<sup>+</sup> ions in soil solution. These ions induce higher soil buffering capacity and make it difficult to change the soil pH substantially. The application of P fertilizers decrease soil pH up to some extent but due high Ca and Mg content along with high buffering capacity cannot decrease the soil pH at greater extent but there may be decreasing trend with P fertilizer as compared to no P fertilizers application [26, 27].

**Table 3. Soil post-harvest pH reading as affected by various degree of acidulation, form of RP fertilizer and application methods**

Application Methods	Post-Harvest soil pH			Mean
	% Acidity	Fertilizer Forms		
		Liquid	Solid	
	25	7.59	7.63	7.61 a
	50	7.61	7.48	7.54 a
	100	7.51	7.58	7.54 a
	150	7.58	7.56	7.57 a
No Split		7.55	7.56	7.56 a
Split		7.59	7.55	7.57 a
	Mean	7.57 a	7.56 a	Significance
Planned Mean Comparison				*
Control	7.70			
Rest	7.56			
LSD (0.05)		Interactions		
Fertilizer Forms (F)	NS	F x AM		NS
App. Methods (AM)	NS	F x L		NS
Acid Levels (L)	0.08	AM x L		NS
		F x AM x L		NS

### Soil AB-DTPA extractable phosphorus at boot stage of wheat

The soil AB-DTPA extractable P was significantly influenced by various acid levels, form of RP fertilizers and application methods and their interactions were found non-significant except between application methods and acid levels (AM x L) (Table 4). The AB-DTPA extractable

phosphorus at boot stage of wheat was increased with increasing acidulation levels of RP. At 150 % acidulation, the soil P was 4.84 mg kg<sup>-1</sup> which was similar to 4.15 mg P kg<sup>-1</sup> observed in 100 % acidulated RP on average basis. The 25 % acidulated RP produced 2.32 mg kg<sup>-1</sup> P which was higher than 1.57 mg kg<sup>-1</sup> recorded for control. Regarding the solid and liquid forms of



acidulated RP, solid fertilizers were found superior as compared to liquid fertilizer. In solid fertilizers, the P was 4.30 mg kg<sup>-1</sup> while in case of liquid acidulated RP it was 2.92 mg kg<sup>-1</sup> at boot stage of wheat crop. Among the application methods, split application showed significantly higher values of 3.92 mg kg<sup>-1</sup> as compared to no split application giving value of 3.30 mg kg<sup>-1</sup> at boot stage of wheat.

The increase in soil P concentration affirmed higher release of P with acidulation of RP as also reported by [6, 9].

The solid fertilizers increased the soil P over the liquid fertilizer that could be associated of less contact with soil colloids. Similar results were also reported by [13]. This was further affirmed by the splitting of fertilizer which further reduced the contact time between the P in solution with soil colloids and hence reduced the P fixation and complexation that usually increased with time and more contact of P fertilizer with soil. These results are in line with [6] who reported that there will be more P in soil after split application of P fertilizers.

**Table 4. Soil AB-DTPA extractable phosphorus at boot stage of the wheat crop as affected by various degree of acidulation, form of RP fertilizer and application methods**

AB-DTPA Extractable P (mg kg <sup>-1</sup> )				
Application Methods	% Acidity	Fertilizer Forms		Mean
		Liquid	Solid	
	25	1.98	2.66	2.32 d
	50	2.23	4.03	3.13 c
	100	3.65	4.64	4.15 b
	150	3.81	5.87	4.84 a
No Split		2.42	4.17	3.30 b
Split		3.41	4.43	3.92 a
	Mean	2.92 b	4.30 a	Significance
Planned Mean Comparison				***
Control	1.57			
Rest	3.61			
LSD (0.05)		Interactions		
Fertilizer Forms (F)	***	F x AM		NS
App. Methods (AM)	*	F x L		NS
Acid Levels (L)	0.66	AM x L		*
		F x AM x L		NS

#### Post-harvest soil AB-DTPA extractable P

Like the AB-DTPA extractable P in boot stage of the crop, the postharvest soil AB-DTPA extractable P was also significantly affected by acidulation levels while non-significantly by forms of RP fertilizer, application methods and their interactions produced non-significant result. When control was compared, the treated plots had significantly higher AB-DTPA extractable P concentration at postharvest stage than control receiving non-acidulated RP suggesting that acidulation increased the P

release from the RP (Table 5). The minimum value 1.26 mg kg<sup>-1</sup> was observed in controls plots while maximum value 2.28 mg kg<sup>-1</sup> was recorded in treated plots. This increase from RP increased with acid levels and higher value of post-harvest soil AB-DTPA extractable P as 2.77 mg kg<sup>-1</sup> was noted in 150 % acidulated RP when averaged across the forms and application method. This was followed by 100 % levels 2.61 mg kg<sup>-1</sup> while lower value of 1.67 mg kg<sup>-1</sup> was recorded for 25 % acid levels. The soil AB-DTPA extractable P decreased gradually from boot stage of the crop to

post-harvest stage indicating reduction with time associated with plant uptake and fixation that increased with time. The

increased in soil AB-DTPA with RP acidulation was also reported by [6] in the same soils of Peshawar.

**Table 5. Post-harvest soil AB-DTPA extractable phosphorus as affected by various degree of acidulation, form of RP fertilizer and application methods**

Post- Harvest Soil AB-DTPA Extractable P (mg kg <sup>-1</sup> )				
Application Methods	% Acidity	Fertilizer Forms		Mean
		Liquid	Solid	
	25	1.55	1.79	1.67 c
	50	2.07	2.10	2.09 b
	100	2.47	2.74	2.61 a
	150	2.56	2.97	2.77 a
No Split		2.10	2.30	2.20 a
Split		2.23	2.51	2.37 a
	Mean	2.16 a	2.40 a	
Planned Mean Comparison				Significance
Control	1.26			***
Rest	2.28			
LSD (0.05)		Interactions		
Fertilizer Forms (F)	NS	F x AM		NS
App. Methods (AM)	NS	F x L		NS
Acid Levels (L)	0.337	AM x L		NS
		F x AM x L		NS

### P concentration in plant leaves

The P concentration in plant leaves was significantly influenced by various acidulation levels and application methods while effect of forms of RP fertilizers (solid and liquid), increased P concentration in plant leaves over control but the difference between the two forms was statistically non-significant (Table 6). Among the acid levels, 100 and 150 % were found superior and more P concentration was recorded in plant leaves which are 0.146 and 0.148 % respectively. When averaged across the forms of fertilizers and application technique, the minimum P concentration 0.116 % was observed at 25 % acidified RP. In case of application methods, split application showed more P concentration 0.145 % while lower P accumulation in leaves 0.124 % was noted in single application. All treated plots produced higher P concentration in wheat leaves at harvesting stage over the control which received the non-acidulated RP suggesting that acidulation of RP substantially

improved the P release from RP and its availability to plant. The interactions of the treatments produced no significant result. Our results are in line with [6], that acidulation of RP increased the P concentration in plants leaves.

The split application of RP to reduced contact time between the soil colloids and soil particles increased the P release and enhanced the P concentration in wheat leaves when compared to no-split. However the P concentration in leaves did not differs much in solid and liquid fertilizers, which could be concerned to growth variations. Similar results were obtained by [28]. Higher P concentration was recorded when P was applied in split doses to wheat crop [29].

### P concentration in grains

The P concentration in wheat grains was significantly increased with increasing acidification level of RP and split application while the difference between the liquid and solid acidulated RP was found non-significant while the interactions

were found non-significant (Table 7). Results indicated that P concentration in grain significantly increased with increasing acidification levels. More P concentration 0.17 % was recorded in 150 % acid levels followed 100 % acid level while lower P concentration 0.14 % was observed in 25 % acidification level. Among the application methods, split application produced more P concentration 0.16 % in grains over no split which was recorded 0.15 %. Similarly treated plots showed more P accumulation 0.15 % in grains as compared to controls plots 0.11% when averaged across all factors.

The higher P concentration in grain reflected the higher availability of P in soil which would have been increased with acidulation levels and time of application. Similar to P concentration in wheat leaves, the P concentration in grains also increased in split application which may be due to the deep placement of RP fertilizers in the 2nd application time that would have reduced the roots and granule distances and facilitated the P availability. Split application of phosphorus fertilizers caused higher P concentration in grains of wheat [29].

**Table 6. Plant leaves P concentrations as affected by various degree of acidulation, form of RP fertilizer and application methods**

Application Methods	% Acidity	Plant leaves P concentration (%)		
		Fertilizers Forms		Mean
		Liquid	Solid	
	25	0.111	0.120	0.116 b
	50	0.117	0.138	0.127 b
	100	0.141	0.151	0.146 a
	150	0.150	0.146	0.148 a
No Split		0.122	0.126	0.124 b
Split		0.138	0.151	0.145 a
	Mean	0.130 a	0.139 a	
Planned Mean Comparison				Significance
Control	0.100			**
Rest	0.134			
LSD (0.05)		Interactions		
Fertilizer Forms (F)	NS	F x AM		NS
App. Methods (AM)	**	T x L		NS
Acid Levels (L)	0.0171	AM x L		NS
		F x AM x L		NS

**Table 7. Wheat grain P concentrations as affected by various degree of acidulation, form of RP fertilizer and application methods**

Application Methods	% Acidity	Grain P Concentration (%)		
		Fertilizer Forms		Mean
		Liquid	Solid	
	25	0.145	0.139	0.142 c
	50	0.153	0.158	0.155 bc
	100	0.156	0.175	0.166 ab
	150	0.165	0.176	0.171 a
No Split		0.143	0.157	0.150 b
Split		0.167	0.167	0.167 a
	Mean	0.155 a	0.162 a	Significance
Planned Mean Comparison				***



Control	0.117			
Rest	0.158			
LSD (0.05)		Interactions		
Fertilizer Forms (F)	NS	F x AM		NS
App. Methods( AM)	**	F x L		NS
Acid Levels (L)	0.014	AM x L		NS
		F x AM x L		NS

## Conclusions

The wheat grain and biomass yield and plant P and soil AB-DTPA extractable at postharvest stage improved with acidification suggesting that P release from RP was enhanced through acidification. Solid acidulated RP showed higher efficiency as compared to the liquid fertilizer which could be attributed to less exposure and contact with soil matrices. Furthermore, split application of both solid and liquid acidified RP improved the yield and P uptake by wheat crop. Conclusively, solid 100 % acidulated RP, applied in two equal splits is recommended for higher wheat yield and P uptake under calcareous conditions with improved technology and agronomic practices.

## Authors' contributions

Conceived and designed the experiments: M Rahman, D Muhammad, M Sharif & M Mussarat, Performed the experiments: M Rahman & D Muhammad, Analyzed the data: M Rahman, D Muhammad & M Sharif, Contributed reagents/ materials/ analysis tools: M Rahman, D Muhammad, RJ Ahmad & F Ishaq, Wrote the paper: M Rahman, D Muhammad, Rafiullah & M Irfan.

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