

## The Effect of Phosphate Rocks on Spring Barley Shoot Yield in a Pot Trial

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### Introduction

Phosphate rocks are of either igneous or sedimentary origin. Reactive sedimentary phosphate rocks are suitable for direct field application as P fertilizers on moderately or strongly acidic soils (KHASAWNEH & DOLL, 1978). The dissolution of phosphate rock depends mainly on the phosphate rock's properties (chemical reactivity, specific surface, particle size), as well as on soil characteristics, climate, etc. The solubility of phosphate rocks in soil is influenced favourably by low available P, exchangeable Ca, pH (<pH 6) and base saturation, high cation exchange capacity, OM content, and satisfactory water supply (HAMMOND et al., 1986). The assessment of the agronomic effectiveness of phosphate rocks as P fertilizers is based upon a comparison to water soluble P fertilizers.

Total P content is not an indicator of the agronomic effectiveness of phosphate rock. The most informative property of phosphate rock in relation to its agronomic performance is solubility (CHIEN, 1993). The chemical reactivity of phosphate rocks can be estimated by the solubility of their P content in different extractants (KHASAWNEH & DOLL, 1978; CHIEN, 1993). Mostly neutral ammonium citrate (USA, Australia), 2% citric acid (Brasil) and 2% formic acid (Europe) are used as solvents. Classification of phosphate rocks as reactive and non-reactive ones according to their solubility, is not unified all over the world. In Europe, for example, phosphate rocks with more than 55% of total P dissolved in 2% formic acid are considered reactive.

The objective of the present study was: 1. to determine the P solubility of six phosphate rocks, using different solvents; 2. to investigate the effect of different phosphate rocks, single superphosphate (SSP), and SSP+Ca treatment on soil available P contents, as well as on the shoot yields of tillering-stage spring barley (*Hordeum vulgare L.*) grown in a pot trial. Results obtained with red clover (*Trifolium pratense L.*) were published by OSZTOICS et al. (2005b).

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## Materials and Methods

### Laboratory experiment

In both the laboratory and pot experiment all phosphate rocks were applied with <math><160\ \mu\text{m}</math> particle size, as within this fraction differences in particle size do not affect their agronomic effectiveness (HAMMOND et al., 1986).

Methods for evaluating the chemical reactivity of the phosphate rocks were: 1. Extraction with 2% formic acid; 2. Extraction with 2% citric acid; 3. Extraction with neutral ammonium citrate, first extraction (ISO 7497-1984/E; OJEC, 1977. No. L 213/5.).

Total P content of P sources and soils were determined by inductively coupled plasma emission spectrometry (ICP) after microwave digestion with cc.  $\text{HNO}_3$ + $\text{H}_2\text{O}_2$  in teflon bomb.

### Greenhouse experiment

A greenhouse experiment was conducted with spring barley to determine the comparative effectiveness of six phosphate rocks applied directly on a Lamellic Arenosol with sand soil texture, strongly acidic and low in available P (Nyírlugos, Hungary), and on a Haplic Luvisol with clay loam texture, also strongly acidic and very low in available P (Ragály, Hungary). The main physical and chemical properties of the untreated soils are given in Table 1.

Table 1  
Chemical and physical properties of the soils used in the greenhouse experiment

Soil property	Acidic sandy soil (Nyírlugos)	Acidic clay loam soil (Ragály)
pH( $\text{H}_2\text{O}$ ) (1:2.5 soil: $\text{H}_2\text{O}$ )	5.0	5.7
pH (KCl) (1:2.5 soil:1M KCl)	3.8	4.5
Clay+silt (<math><0.02\ \text{mm}</math>), %	5.0	59.8
Clay (<math><0.002\ \text{mm}</math>), %	2.2	26.6
Total P, $\text{mg}\cdot\text{kg}^{-1}$	300	480
AL- $\text{P}_2\text{O}_5$ , $\text{mg}\cdot\text{kg}^{-1}$ (EGNÉR et al., 1960)	53.6	17.3
LE-P, $\text{mg}\cdot\text{kg}^{-1}$ (LAKANEN & ERVIÓ, 1971)	60.7	14.9
Olsen-P, $\text{mg}\cdot\text{kg}^{-1}$ (OLSEN et al., 1954)	16.2	6.3
Water soluble-P, $\text{mg}\cdot\text{kg}^{-1}$ (SARKADI, 1982)	8.5	3.1
Exchangeable Ca, Mg, CEC (ammonium acetate method), $\text{cmol}_c\cdot\text{kg}^{-1}$		
Exchangeable Ca	0.8	11.6
Exchangeable Mg	0.1	3.1
Cation exchange capacity (CEC)	3.0	19.6
Hydrolytic acidity, % ( $y_1$ )	11	17
Exchangeable acidity ( $y_2$ )	3.7	0
Soil organic matter, % (OM) (BUZÁS, 1988)	0.6	3.4

The treatments of the experiment set up in split-split plot design included three replicates of each of the phosphate rocks, single superphosphate (SSP) (Linz, Austria), and SSP+CaCO<sub>3</sub> mixed into the soil at rates of 100, 400 and 1600 mg total P<sub>2</sub>O<sub>5</sub>·kg<sup>-1</sup> soil, plus a NK treatment without P. In the SSP+CaCO<sub>3</sub> treatment 1910 and 4960 mg CaCO<sub>3</sub>·kg<sup>-1</sup> soil was applied to the acidic sandy soil and acidic clay loam soil, respectively, to neutralize acidity (FILEP, 1999). All pots received 100 mg N and 200 mg K<sub>2</sub>O·kg<sup>-1</sup> soil for adequate N and K supply. All P sources and the N and K fertilizers were mixed into each pot containing 1.6 kg soil on the day the spring barley test crop was planted. Moisture levels were initially maintained at about 60% of field capacity, after which soils were watered according to the requirement of plants during the growing period. The weight of air-dried shoots was determined for each pot. The results from the tillering-stage cut are evaluated in the present paper. The second half of the trial was harvested at flowering stage, but due to paper volume limitations, these results are not included. Available soil P was estimated by the Ammonium-Lactate (AL) (EGNER et al., 1960), Lakanen-Erviö (LE) (LAKANEN & ERVIÖ, 1971), Olsen (OLSEN et al., 1954), Bray1 (BRAY & KURTZ, 1945) and water (SARKADI, 1982) methods.

## Results and Discussion

### Laboratory experiment

Specific surface, CaCO<sub>3</sub> content, total P and the reactivity scales of the investigated six phosphate rocks – originating from Algeria (ALG), North Florida (FLO), North Carolina (NCA), Senegal (SEN), Morocco (MO) and Hyperphosphate (HYP) – as measured by the chemical extractions are shown in Table 2. Among the phos-

Table 2  
Properties and solubility tests (reactivity scales) of the phosphate rocks (PR) (<160 µm) as measured by conventional reagents (OSZTOICS et al., 2005a,b)

Phosphate rock	Properties			Soluble P <sub>2</sub> O <sub>5</sub> %, extracted by					
	Specific surface, m <sup>2</sup> ·g <sup>-1</sup>	CaCO <sub>3</sub> %	Total P <sub>2</sub> O <sub>5</sub> , %	2% formic acid		2% citric acid		neutral NH <sub>4</sub> <sup>+</sup> citrate	
				% of PR	% of total P	% of PR	% of total P	% of PR	% of total P
ALG	14.2	18.3	27.5	17.6	63.9	10.8	39.4	8.2	29.8
FLO	12.7	6.9	25.4	6.7	26.3	6.5	25.6	2.7	10.6
NCA	20.0	12.8	23.8	17.9	75.2	11.6	48.9	5.1	21.4
SEN	5.7	4.3	33.0	9.5	28.8	8.6	26.0	2.5	7.6
MOR	12.0	14.4	26.1	15.2	58.2	9.7	37.3	3.1	11.9
HYP	15.4	13.3	22.5	14.7	65.2	10.9	48.3	5.0	22.2

Note: ALG: Algeria; FLO: North Florida; NCA: North Carolina; SEN: Senegal; MOR: Morocco; HYP: Hyperphosphate

phate rocks ALG, NCA and HYP had the highest P solubility. On the other hand, the less P soluble phosphate rocks were SEN and FLO (Table 2).

#### *The effect of P sources and rates on soil available P contents*

After completing the pot trial, the dissolution of the phosphate rocks in soil was assessed by water, Olsen, Bray1, Lakanen-Erviö (LE), and ammonium lactate (AL) soluble P tests (Table 3). In Hungary the AL method has been used officially as the extractant for assessing soil P availability in the case of using water soluble P sources as P fertilizers (EGNER et al., 1960). The applicability of the AL method for determining the basic reactive phosphate rocks as P sources was also studied in the pot trial.

On the acidic sandy soil (Nyírlugos), over all P sources, as compared to the NK control, the 1600 mg·kg<sup>-1</sup> total P<sub>2</sub>O<sub>5</sub> dose caused a five-fold increase in the H<sub>2</sub>O-P and Olsen-P, a 15-fold increase in the Bray1, and a 23–24-fold increase in the LE-P and AL-P values. The same methods on the acidic clay-loam soil (Ragály) resulted in 8–9, 20 and 46–64 times higher values, respectively (Table 3).

When low water-soluble fertilizers like phosphate rocks are applied, soil testing using strongly acidic AL (pH=3.75), or LE (pH=4.65) extractants can dissolve a substantial amount of the unsolubilized phosphate rock in the soil. Therefore they can overestimate the available P from PR-treated soils as compared to soils treated with SSP. In an incubation experiment with Algerian phosphate rock and SSP, OSZTOICS et al. (2003) obtained similar results with the AL extractant.

#### *The effect of P sources and rates on spring barley shoot yields*

In the case of both soils, the higher the phosphate rock solubility was in formic acid, citric acid or neutral ammonium citrate, the higher the P responses obtained in spring barley were (Table 4). On the average of the P forms and doses, spring barley shoot yield was 95% higher on colloid-rich acidic clay loam soil (3.69 g·pot<sup>-1</sup>) than on colloid-poor acidic sandy soil (1.90 g·pot<sup>-1</sup>). In the acidic sandy soil, on the average of P forms, as an effect of the 1600 mg total P<sub>2</sub>O<sub>5</sub>·kg<sup>-1</sup> P-dose, spring barley yields were 4.4-times greater, as compared to the NK treatment, while in the acidic clay loam soil the yield values were 4.9-times higher.

On the average of the P doses, in the case of the acidic sandy soil (Nyírlugos), the highest spring barley shoot yields (2.25 and 2.23 g·pot<sup>-1</sup>) were obtained, among the phosphate rocks, with the highly soluble Algerian (ALG) and the Hyperphosphate (HYP) PRs. Treatments with the Senegal and North Florida phosphate rocks – having the lowest P solubility – produced the smallest average spring barley shoot yields (0.88 and 0.92 g·pot<sup>-1</sup>). Spring barley grown on the acidic clay loam soil (Ragály) gave the highest average shoot yields (4.25 and 3.81 g·pot<sup>-1</sup>) in the treatments, among the phosphate rocks, with PRs from Algeria and North Carolina. A previous study (NÉMETH et al., 1996; OSZTOICS et al., 2001) also showed that on strongly acidic soils from the Carpathian Basin, Algerian phosphate rock could be considered a P source as effective as SSP. When all of the P sources were taken into

Table 3  
Effect of P sources on the soil P test values (mg·kg<sup>-1</sup>) of the acidic sandy soil (Nyírtlugos) and the acidic clay loam soil (Ragdly) at the end of the pot trial

Soil P test method	P source									
	ALG	FLO	NCA	SEN	MOR	HYP	SSP	SSP+lime		
	<i>Acidic sandy soils (Nyírtlugos)</i>									
AL-P <sub>2</sub> O <sub>5</sub>	Mean* 445	376	491	224	474	527	387	438		
	Range 44-1358	51-1024	49-1494	53-526	45-1345	48-1556	57-1121	63-1244		
LE-P <sub>2</sub> O <sub>5</sub>	Mean* 374	235	307	89	356	471	188	312		
	Range 35-1163	39-618	36-888	37-182	31-1015	36-1428	42-468	38-923		
Bray-1 P	Mean* 191	181	203	201	207	219	185	177		
	Range 37-551	49-496	38-590	47-548	36-601	35-654	30-525	28-522		
Olsen-P	Mean* 29.9	31.8	36.3	27.0	31.0	29.6	84.6	77.6		
	Range 16.4-38.4	18.9-44.3	20.1-52.2	18.1-38.3	18.2-38.5	17.4-42.7	21.1-205.5	14.6-204.8		
Water-P	Mean* 16.7	15.2	13.7	12.3	14.5	14.6	34.8	38.3		
	Range 8.5-25.9	8.1-20.9	8.5-20.5	8.3-18.1	7.7-20.2	8.0-25.3	8.5-91.6	9.3-100.4		
	<i>Acidic clay loam soil (Ragdly)</i>									
AL-P <sub>2</sub> O <sub>5</sub>	Mean* 270	209	310	66	259	288	180	213		
	Range 16-893	17-609	15-991	15-160	18-762	14-866	13-577	10-695		
LE-P <sub>2</sub> O <sub>5</sub>	Mean* 250	134	261	39	222	292	159	207		
	Range 8-855	11-403	11-856	10-93	8-694	10-941	9-534	10-709		
Bray-1 P	Mean* 26.4	12.4	17.8	6.2	18.3	23.1	67.6	71.5		
	Range 4.0-74.0	4.3-27.9	4.4-37.8	4.7-11.0	5.1-44.6	5.0-63.5	3.8-233.6	5.6-239.2		
Olsen-P	Mean* 14.5	9.4	13.5	6.3	12.0	9.2	34.0	49.0		
	Range 4.1-32.1	4.7-17.8	5.5-24.4	4.7-9.1	5.4-22.4	4.3-15.5	4.7-100.7	6.2-154.6		
Water-P	Mean* 14.9	6.4	7.3	4.0	7.5	7.0	18.4	20.5		
	Range 3.3-37.2	3.2-10.8	1.9-13.2	3.1-6.8	3.7-13.0	3.1-12.6	3.2-54.7	3.5-64.0		

Note: Soil P test methods: See Table 1. P sources: see Table 2; SSP: single superphosphate (Austria). Mean: Average of 0 (NIK), 100, 400 and 1600 mg P<sub>2</sub>O<sub>5</sub>·kg<sup>-1</sup> treatments

Table 4

Shoot weights ( $\text{g}\cdot\text{pot}^{-1}$ ) of spring barley grown in the pot experiment with acidic sandy soil (Nyírlugos) and acidic clay loam soil (Ragály) treated with different P sources (at the beginning of shooting)

Treatments	P source								LSD <sub>5%</sub>	Mean
	ALG	FLO	NCA	SEN	MOR	HYP	SSP	SSP+Ca		
<i>A. Acidic sandy soil (Nyírlugos)</i>										
NKP <sub>0</sub>	0.51	0.47	0.48	0.45	0.53	0.50	0.36	2.68	0.67	0.75
NKP <sub>100</sub>	1.26	0.67	0.97	0.64	0.69	1.13	0.74	3.94		1.25
NKP <sub>400</sub>	3.00	0.95	1.68	0.76	2.52	2.94	2.06	4.43		2.29
NKP <sub>1600</sub>	4.23	1.59	2.85	1.67	3.74	4.36	3.68	4.19		3.29
LSD <sub>5%</sub>					0.67					0.24
Mean	2.25	0.92	1.50	0.88	1.87	2.23	1.71	3.81	0.33	1.90
<i>B. Acidic clay loam soil (Ragály)</i>										
NKP <sub>0</sub>	1.16	1.08	1.28	1.03	1.03	0.98	1.11	1.26	1.11	1.12
NKP <sub>100</sub>	4.11	1.85	2.47	1.12	2.55	2.87	6.92	4.56		3.31
NKP <sub>400</sub>	5.64	2.86	5.18	1.80	5.34	4.65	6.29	7.10		4.86
NKP <sub>1600</sub>	6.08	4.62	6.31	3.20	5.93	5.12	6.20	6.32		5.47
LSD <sub>5%</sub>					1.11					0.39
Mean	4.25	2.60	3.81	1.79	3.71	3.41	5.13	4.81	0.55	3.69

Note: ALG: Algeria; FLO: North Florida; NCA: North Carolina; SEN: Senegal; MOR: Morocco; HYP: Hyperphosphate; SSP: single superphosphate (Linz, Austria)

account it was found that on the acidic sandy soil (Nyírlugos) the single superphosphate and lime (SSP+CaCO<sub>3</sub>) treatment ( $3.81 \text{ g}\cdot\text{pot}^{-1}$ ), on the acidic clay loam soil (Ragály) the SSP and SSP+CaCO<sub>3</sub> treatments ( $5.13$  and  $4.81 \text{ g}\cdot\text{pot}^{-1}$ ) gave the highest spring barley shoot yields (Table 4).

#### Relationship between phosphate rock solubility and spring barley shoot yields

In the case of both soils only a weak correlation ( $r^2 = 0.47\text{--}0.53$ ) was established between the total added P of the six phosphate rock treatments and spring barley

Table 5

Correlation coefficients ( $r^2$ ) between the total, and "soluble" P amounts added and tillering stage shoot yield of spring barley grown in a pot experiment set up with acidic sandy (Nyírlugos) and acidic clay loam (Ragály) soils and six phosphate rock treatments ( $y = a \ln(x) + b$ ;  $n = 18$ , excluding the NK treatments)

Solvent	Acidic sandy soil (Nyírlugos)	Acidic clay loam soil (Ragály)
65ww% HNO <sub>3</sub> +30 ww%H <sub>2</sub> O <sub>2</sub> (total)	0.5259	0.4727
2% formic acid	0.7277	0.7209
2% citric acid	0.6556	0.6199
Neutral NH <sub>4</sub> <sup>+</sup> -citrate	0.7421	0.7510

P responses. If the P solubility of the phosphate rocks was also taken into consideration, the correlation between the added formic acid, citric acid or neutral ammonium citrate soluble P amounts and spring barley responses became much stronger (Table 5). The strongest correlation was obtained when the amount of P added was based on the neutral ammonium citrate soluble P ( $r^2 = 0.74\text{--}0.75$ ) (Table 5). Similar results were also obtained by CHIEN and HAMMOND (1978), LÉON et al. (1986), and ZAHARAH and SHARIFUDDIN (2002).

Table 6

Relative agronomic effectiveness (RAE, %) of the phosphate rocks applied to the acidic sandy soil (Nyírlugos) and acidic clay loam soil (Ragály) in the spring barley pot experiment

P source	Acidic sandy soil (Nyírlugos)			Acidic clay loam soil (Ragály)		
	P applied, mg total $P_2O_5 \cdot kg^{-1}$			P applied, mg total $P_2O_5 \cdot kg^{-1}$		
	100	400	1600	100	400	1600
ALG	289	160	117	52	87	98
FLO	73	30	35	13	34	69
NCA	183	76	74	24	79	102
SEN	61	18	37	0	14	41
MOR	79	129	102	25	82	95
HYP	241	156	121	31	69	79
SSP+Ca	465	111	47	57	112	99

Note: P sources: ALG: Algeria; FLO: North Florida; NCA: North Carolina; SEN: Senegal; MOR: Morocco; HYP: Hyperphosphate; SSP: single superphosphate (Linz, Austria)

The relative agronomic effectiveness (RAE) of phosphate rocks was also evaluated, in which the spring barley shoot yields of phosphate rock treatments was compared to yields obtained with single superphosphate treatment (SSP) (Table 6):

$$RAE\% = \left( \frac{X_1 - X_0}{X_2 - X_0} \right) \cdot 100$$

where:  $X_1$  = spring barley shoot weight at a given P level with phosphate rock application;  $X_2$  = spring barley shoot weight at the same P level with single superphosphate (SSP) application;  $X_0$  = spring barley shoot weight in the NK plot without P. The phosphate rock from Senegal (SEN) had the lowest effectiveness among the studied phosphate rocks. The relative agronomic effectiveness of the Algerian (ALG) phosphate rock was almost comparable to that of the SSP+CaCO<sub>3</sub> treatment.

#### Correlation between the soil P test values and spring barley shoot yields

When phosphate rocks were applied as P source, the comparison of soil test P methods showed a different picture on the two soils. In the case of the acidic sandy soil (Nyírlugos) the highest correlation coefficients with spring barley P response were given by the strongly acid LE-P ( $r^2 = 0.83$ ) and AL-P ( $r^2 = 0.74$ ) tests, while

Table 7

Correlation coefficients ( $r^2$ ) between the soil P test values and air-dry shoot weights of spring barley grown in a pot experiment set up with acidic sandy soil (Nyírlugos) and acidic clay loam soil (Ragály) and six phosphate rock treatments ( $y = a \ln(x) + b$ ;  $n = 24$ , including the NK treatments)

Correlation between	Acidic sandy soil (Nyírlugos)	Acidic clay loam soil (Ragály)
AL-P and shoot weight	0.7364	0.8092
LE-P and shoot weight	0.8267	0.8372
Olsen-P and shoot weight	0.4878	0.8819
Water-P and shoot weight	0.6860	0.8332
Bray1-P and shoot weight	0.6459	0.8799

for the acidic clay loam soil (Ragály) the highest correlation coefficients were obtained with the Olsen-P ( $r^2 = 0.88$ ) and Bray1-P ( $r^2 = 0.88$ ) methods (Table 7).

On the acidic clay loam soil the revealed results also confirmed that – similarly to the Bray-2 and double acid methods – the AL-method is not or is less applicable for the soil testing of available P from phosphate rock treated soils than the Olsen or Bray1 method (HAMMOND et al., 1986).

### Summary

Depending on their origin, sedimentary phosphate rocks (PRs) may differ in their P solubility, and, as a consequence, in their agronomic effectiveness. The effect of six phosphate rocks (PR) – originating from Algeria (ALG), North Florida (FLO), North Carolina (NCA), Senegal (SEN) Morocco (MOR) and Hyperphosphate (HYP) with various P solubility (evaluated by 2% formic acid, 2% citric acid, and neutral ammonium citrate) – as well as single superphosphate (SSP) and superphosphate + lime (SSP + Ca) (each P source on 4 P levels, with doses of 0, 100, 400 and 1600 mg  $P_2O_5 \cdot kg^{-1}$  soil) on the shoot yield of tillering stage spring barley, soil available P (i.e.  $H_2O$ , Olsen, Bray1, Lakanen-Erviö (LE) and ammonium lactate (AL) extractable P contents) were studied in pot experiments set up with acidic sandy soil (Nyírlugos, Hungary) and acidic clay loam soil (Ragály, Hungary), both with low P supplies.

The average spring barley shoot yield at the beginning of shooting was 95% higher on the colloid-rich acidic ( $pH_{KCl}$ : 4.5) clay loam soil than on the colloid-poor acidic ( $pH_{KCl}$ : 3.8) sandy soil. The differences in the solubility of phosphate rocks showed close correlation to the differences in P responses. On both soils, the correlation between total PR-P added and P responses in spring barley shoot yield was much weaker than that between neutral ammonium citrate soluble PR-P added and P responses in spring barley shoot yield. When phosphate rocks were applied as P sources, the comparison of soil test P methods showed a different picture on the two soils. In the case of the acidic sandy soil (Nyírlugos), the strongly acid LE-P ( $r^2 =$

0.83) and AL-P ( $r^2 = 0.74$ ) tests gave the highest correlation coefficients with spring barley responses to P, while on the acidic clay loam soil (Ragály) these were achieved by the Olsen-P ( $r^2 = 0.88$ ) and Bray1-P ( $r^2 = 0.88$ ) methods.

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**Key words:** sedimentary phosphate rocks, soil P tests, P solubility of phosphate rocks, relative agronomic effectiveness

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