

Superphosphate Availability as Affected by the CaCO_3 Content of the Soil

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The availability of the various phosphate compounds used as fertilizer has been an essential problem of both the research and practice of plant nutrition for a long time. The study of the effects of superphosphate containing phosphate in water soluble form was begun in the Rothamsted Experimental Station at the middle of the last century. The appropriate application of phosphate fertilizers was one of the first problems studied in the field of agricultural chemistry in Hungary, too. 'SIGMOND himself as a young chemist began to study the availability of several phosphate compounds in pot experiments [20]. As mentioned in his paper, published in 1906, the chemical reactions between the soil and the phosphates resulted in a number of phosphate compounds differing in their chemical properties and physical state (i.e. grain size, specific surface, etc.). These transformations exert both advantageous and disadvantageous influences on the availability of phosphate compounds applied as fertilizer.

On the basis of literary data, as well as of the results of his pot experiments, 'SIGMOND proved that on a calcareous soil dicalcium phosphate, practically insoluble in water, was at least as good nutrient source for plants as superphosphate. Moreover, iron- and mainly aluminium phosphates, commonly considered as unavailable, were taken up by the plants in appreciable amounts.

The interaction between the soil and fertilizer phosphates remained one of the basic problems of fertilizing during the nearly 40 years of 'SIGMOND's scientific career [10] and it is still a problem of topical interest. A number of test methods have been developed for the estimation of the available phosphate resources and there were some attempts to separate the different P-compounds in the soil, as well.

By presenting the data of a long-term field experiment carried out since the fall of 1958 on a chernozem soil variety we wish to report on the present state of our investigation in this field.

Materials and methods

The long-term field experiment was conducted at Martonvásár on a topographically varied area [23]. Each treatment had four replications. Though some properties of brown forest soils could be observed in some horizons of the soil of the experimental area, the typical dynamics of chernozem soils

Table 1
**Lime and humus content of the soil surface layer (0–20 cm) in
 the different blocks (average of 2 replications)**

Number of blocks	Sign	CaCO ₃ %			Humus %		
		$\bar{x} \pm s_{\bar{x}}$	x_{\max}	x_{\min}	$\bar{x} \pm s_{\bar{x}}$	x_{\max}	x_{\min}
I, II	A	10.0 ± 0.8	15.1	5.4	2.74 ± 0.10	3.39	2.25
III, IV	B	1.9 ± 0.5	6.6	0.3	2.85 ± 0.08	3.45	2.41

predominated (Ca-ions represented from 80 to 93% of the CEC) as a result of continuous cultivation. The plough-layer (0–20 cm) varied in its properties owing to the different sediment deposits. The CaCO₃-content of the plough-layer changed systematically (Table 1), so it was possible to evaluate statistically the relations between the lime content and the nutrition of plants. Humus distribution did not show such a systematical variation, though there were considerable differences between the plots. The total P content varied between 154 ± 30 mg P₂O₅ per 100 g soil.

In the sequence of the test plants we generally used hybrid maize twice, followed twice by wheat [11]. As a response to NPK fertilization both grain yield and P-uptake by the above ground part of the plant (= P-uptake) increased considerably.

The yields of the control plots did not decrease; besides N, mostly P-fertilizing had positive effects. The increases obtained with K applied in addition to N + P were generally negligible. P and K with half of the N were mixed into the plough-layer in autumn and the other half of the N dose was broadcasted in spring. (See the average of the measured values in Table 6-Appendix).

Chemical analysis

The sampling of the soil and the plants, as well as the determination of some ingredients were carried out with the standard methods used in our laboratory [12]. The soil extractants CAL and 0.5 M NaHCO₃ were prepared according to SCHÜLLER [19] and OLSEN et al. [17], respectively. For the separation of the inorganic P-compounds in the soil the CHANG–JACKSON method [4] was used (involving only the first four fractions where the influence of the fertilizer treatments could commonly be observed [21]).

The scheme of our procedure was the following:

Fraction number	Extractant	Shaking time
I.	1.0 N NH ₄ Cl	30 minutes
II.	0.5 N NH ₄ F (pH 8.2)	60 minutes
III.	0.1 N NaOH	17 hours
IV.	0.5 N H ₂ SO ₄	60 minutes

The pH of the NH₄F solution was increased to 8.2 in order to achieve a more selective separation of Al- and Fe-phosphates, and to reduce the dissolution of Ca-phosphates.

The P in the extracts was measured by the MURPHY and RILEY method according to ALEXANDER and ROBERTSON [1] but in the case of NH_4F extract the addition of boric acid was necessary before colour developing. The total P-content was measured after wet digestion with H_2SO_4 (cc) and H_2O_2 (p.a.) [7].

Experimental results

The effects of P-fertilization on grain yield, P-uptake and on the "availability" of P are presented in Tables 2–6. By the factorial design of our field experiments, the so-called "main effects" and "interactions" (see e.g. in COCHRAN-COX [6]) can be determined. In the Tables only the first order interactions (NP, PK) are shown, because the NPK interactions do not furnish any utilizable informations. The main effects and the interactions are calculated for the strongly and moderately calcareous blocks separately, so it is possible to verify the eventual disparities of the effects, too. There are no significant differences in grain yields and P-uptakes; likewise the lime content has not any significant influence on the P-balance (Table 2).

Table 2

The main effects and interactions of P-fertilizer application in several characteristics of the crop yields (averages of 12 years)

Yields		Factorial effects		Level of significance for (A-B)
		A	B	
Grain, q/ha	P	4.7*	3.8*	N.S.
	NP	4.0*	3.4*	N.S.
	PK	0.9	-0.1	N.S.
P_2O_5 -uptake kg/ha	P	8.7***	8.5***	N.S.
	NP	3.7***	3.5**	N.S.
	PK	1.9*	1.3	N.S.
P_2O_5 -balance of 12 years kg/ha	P	553***	543***	N.S.
	NP	-49***	-39***	N.S.
	PK	-14	-18	N.S.

Notes:

Levels of significance: * $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$; $P > 0.1 = \text{N.S.}$
 A and B: state of the lime content (see: Table 1).

Table 3 shows the data obtained by various extractants with several test methods estimating the "available" P and the data of the inorganic fractions. It is evident that the lime content of the soils has not the same influence in case of all methods investigated. The results obtained by OLSEN's bicarbonate method are not affected by it. The acidity of the DL-solution decreases markedly when coming into contact with CaCO_3 , and so does its solvent capacity, too. The removal of phosphates by the CAL- and AL-solutions, having relatively higher buffering capacity and narrower soil: extractant ratio, is practically unaffected by the CaCO_3 %. Even the phosphates with a narrower Ca:P ratio, which are considered as hardly available to plants, get partly

into solution, so the available P-amount of highly calcareous soils might somewhat be overestimated by using the latter method [9, 13, 15, 26].

The changes in the different inorganic P-fractions due to fertilizing, as well as the effects of the lime content are also summarized in Table 3. The percentage distribution of P in the fractions is also considerably affected by the added superphosphate (see Appendix) which increased the amount of P mostly in fractions I and II. The amount of P in fraction III was influenced also by the humus content, so in that case the computations were carried out with the values corrected to an average humus level. In the subsequent stages of the fractionation procedure the influence of P-fertilizing decreases gradually.

Table 3
The main effects and interactions of P-fertilizer application
on the extractability of P as affected by the CaCO_3 content
of the soil (mg $\text{P}_2\text{O}_5/100$ g soil)

Methods		Factorial effects		Level of significance for (A - B)
		A	B	
DL - P_2O_5	P	0.0	3.0***	***
	NP	0.3	-1.1**	***
	PK	0.4	-0.2	+
AL - P_2O_5	P	5.8***	4.0***	***
	NP	-0.6	-1.0*	N.S.
	PK	-0.1	-0.9	N.S.
CAL - P_2O_5	P	3.9***	3.1***	*
	NP	0.5	-0.6	**
	PK	0.3	-0.3	+
"Olsen"- P_2O_5	P	2.3***	2.3***	N.S.
	NP	-0.8*	-0.6*	N.S.
	PK	0.1	-0.1	N.S.
Fraction I	P	0.292***	0.214**	+
	NP	-0.314***	-0.165*	*
	PK	-0.076	-0.106	N.S.
Fraction II	P	1.31 +	2.23**	+
	NP	-0.58	-1.20*	N.S.
	PK	-0.87	-0.29	N.S.
Fraction III (corrected)	P	1.27*	1.43*	N.S.
	NP	0.03	0.61	N.S.
	PK	0.35	-1.06	N.S.
Fraction IV	P	3.65	1.03	N.S.
	NP	-5.22*	-5.20*	N.S.
	PK	-0.36	-3.90	N.S.
Sum of fractions I-IV	P	6.65*	4.57	N.S.
	NP	-5.99*	-6.27*	N.S.
	PK	-0.93	-3.13	N.S.

Notes:
Levels of significance: $P > 0.1 = \text{N.S.}$; $P < 0.1 = +$; $P < 0.05 = *$; $P < 0.01 = **$
 $P < 0.001 = ***$.
Fraction III (corrected) = $\text{Fr. III}_{\text{orig.}} - [-6.95 (\text{humus } \%_{\text{plot}} - 2.81)]$.

Nevertheless, the significance of the negative NP-interaction proves that the acid soluble fraction IV, which predominates among the examined fractions, may also play a role in plant nutrition in case of sufficient N supply.

This fact is also reflected in the data of Table 4 where the parameters of the linear correlation between the values of the soil-P balance and the test methods are presented. With the AL-, CAL- and OLSEN-method the correla-

Table 4
Characteristics of the simple regression between P₂O₅-balance and the amounts of P₂O₅ extracted with various methods

Independent variables	Coefficients of	
	correlation (r)	regression (b ± t _{5%} · s _b) P ₂ O ₅ mg ^o / _o /100 kg P ₂ O ₅
AL-P ₂ O ₅	0.90***	0.92 ± 0.17
CAL-P ₂ O ₅	0.85***	0.65 ± 0.13
"Olsen"-P ₂ O ₅	0.92***	0.43 ± 0.02
DL-P ₂ O _{5₀}	0.37*	0.28 ± 0.26
DL-P ₂ O _{5₀₀}	0.70***	0.52 ± 0.31
DL-P ₂ O ₅	0.26	0.04 ± 0.19
Fraction I	0.55***	0.05 ± 0.03
Fraction II	0.55***	0.34 ± 0.19
Fraction III (corrected)	0.31 +	0.20 ± 0.24
Fraction IV	0.25	0.61 ± 0.89

Notes:

Asterisks indicating the levels of significance have the meaning as in Table 3.

^o = only for plots where the CaCO₃ content is under 5% (15 elements).

⁰⁰ = only for plots where the CaCO₃ content is over 5% (17 elements).

In the other cases all the 32 plots are included.

Table 5
Characteristics of the simple regression between P-amounts extracted with several methods (mg P₂O₅/100 g soil)

Independent variables	Dependent variables					
	"Olsen"-P		AL-P		CAL-P	
	r	b ± t _{5%} · s	r	b ± t _{5%} · s	r	b ± t _{5%} · s
Fraction I	0.61***	2.8 ± 1.4	0.66***	6.7 ± 2.8	0.67***	5.0 ± 2.1
Fraction II	0.77***	0.58 ± 0.18	0.52**	0.86 ± 0.52	0.44*	0.53 ± 0.41
Fraction III (corrected)	0.41*	0.30 ± 0.23	0.20	0.32 ± 0.57	0.17	0.20 ± 0.43
Fraction IV	0.30	0.06 ± 0.07	0.35*	0.15 ± 0.14	0.32	0.10 ± 0.11
CAL-P	0.85***	0.53 ± 0.12	0.95***	1.23 ± 0.16		
AL-P	0.88***	0.41 ± 0.08				

Asterisks as in Table 3.

Table 6
Appendix

The data of the 2³-factorial field experiment (in the average of years 1959—1970) and the results of the soil investigations (soil sampling in Aug. 1970) (All the values are the averages of two replications)

Measured values	Blocks	Treatments (N, P ₂ O ₅ , K ₂ O kg/ha/years)									
		∅	N ₉₁	P ₅₄	K ₆₂	N ₉₁ P ₅₄	N ₉₁ K ₆₂	P ₅₄ K ₆₂	N ₉₁ P ₅₄ K ₆₂		
Grain q/ha/year	A	30.2	37.0	28.9	27.0	43.8	34.8	30.0	45.2		
80% dry matter	B	30.6	37.4	31.0	30.0	43.2	37.4	30.4	46.6		
P ₂ O ₅ -uptake	A	19.0	20.6	22.2	17.0	31.0	18.8	23.8	33.4		
kg/ha/year	B	19.3	20.8	23.8	19.0	30.6	20.3	24.2	34.5		
P ₂ O ₅ -balances of 12 yrs	A	-230	-248	383	-205	278	-220	364	250		
kg/ha	B	-232	-250	365	-227	283	-244	360	236		
CaCO ₃ %	A	10.4	8.8	8.4	14.2	11.8	8.2	8.4	10.2		
	B	0.8	0.9	1.8	0.6	2.6	5.3	0.6	0.6		
Humus %	A	2.46	2.48	2.40	2.84	3.28	3.24	2.36	2.84		
	B	2.64	2.74	2.78	3.28	3.04	2.74	3.10	2.62		
DL-P ₂ O ₅ mg %	A	1.3	1.0	1.6	0.8	0.9	0.6	1.2	1.2		
	B	2.4	3.0	6.6	2.4	3.6	2.6	6.2	5.6		
AL-P ₂ O ₅ mg %	A	3.3	3.5	10.4	3.7	8.3	3.1	9.6	8.8		
	B	2.2	3.3	9.5	3.6	6.6	4.3	7.1	6.3		
CAL-P ₂ O ₅ mg %	A	2.5	2.7	7.6	2.6	5.8	2.2	6.5	5.4		
	B	1.5	1.1	6.6	1.4	4.0	2.4	4.1	4.1		
"Olsen"-P ₂ O ₅ mg %	A	1.0	1.2	4.1	1.0	2.2	0.8	3.9	2.6		
	B	1.1	0.8	4.2	0.8	2.4	1.0	3.6	2.8		
Fraction I	A	0.18	0.21	0.96	0.60	0.18	0.34	1.04	0.34		
P ₂ O ₅ mg %	B	0.12	0.28	0.68	0.30	0.36	0.34	0.50	0.37		
Fraction II	A	4.12	4.14	7.23	5.86	5.39	5.74	6.54	5.96		
P ₂ O ₅ mg %	B	3.67	4.59	8.25	6.64	5.04	5.05	8.92	6.65		
Fraction III	A	0.86	0.86	0.74	1.66	7.92	6.77	0.46	4.64		
P ₂ O ₅ mg %	B	4.24	2.58	5.72	6.65	4.24	0.80	6.54	4.42		
Fraction III (corrected)	A	3.22	3.08	3.56	1.38	4.58	3.30	3.52	4.40		
P ₂ O ₅ mg %	B	5.36	3.02	3.32	3.32	2.58	2.16	4.46	5.70		
Fraction IV	A	62.6	62.8	72.5	70.2	61.0	65.3	78.0	64.2		
P ₂ O ₅ mg %	B	58.0	63.4	66.3	65.6	64.8	71.2	69.8	61.0		
Fractions I-IV	A	70.1	70.2	84.2	78.0	71.2	74.7	89.1	74.9		
P ₂ O ₅ mg %	B	67.2	71.3	81.0	75.9	72.8	78.8	83.7	73.7		

tion was close for each plot but with the DL-method this could be observed only in plots in which the lime content was below 5%. Similar close linear correlations could be shown for the P-balance versus fractions I and II; and a loose one for fraction III.

In Table 5 the parameters of the simple regression between the data obtained with the test methods and the CHANG-JACKSON's fractions are shown.

Discussion

The nutrients are present in the soil in several compounds differing in solubility, active surface and in the degree of crystallization. When superphosphate is added into a calcareous soil, its water solubility becomes significantly reduced due to the free CaCO_3 . As proved by the data of soil chemical analyses [13, 14, 16, 27], superphosphate is transformed into compounds with narrower Ca : P ratios and thus fertilizer phosphate becomes more and more "aged". The results of numerous experiments prove that this aging is a slow process, so the test plants are able to take up the fertilizer-P. The lime content did not cause any differences in the grain yield nor in the rate of P-uptake during the 12 years of our experiment.

In the subsequent stages of the fractionation procedure, when we used more and more vigorous extractants, the quantities dissolved were less and less dependent on P-fertilizing and on the lime content of the soil. As a matter of fact, the addition of P-fertilizer increased directly the amount of P in the soil solution, that of the adsorbed P on the soil particles and, obviously, on the CaCO_3 surface, as well as the slightly bound forms of Al- and Ca-phosphates [see also 18]. In the CHANG-JACKSON fraction I the highly significant main effect of P was connected with a similarly strong but negative NP interaction. This meant that in the case of N- and P-fertilizing this fraction, present in very low concentration, was exhausted, i.e. it could not build up reserves. During the vegetation period, fraction I must have been continuously supplied from the other three fractions, primarily from the II. This assumption is proved by the significant negative NP interactions with fractions II and IV, as well as by the significant main effects with fractions II and III as a sign of P-accumulation due to systematic P-fertilizing.

The CaCO_3 content had significant positive effect on P-accumulation mostly in fraction I and a slight one in fractions II and III. These facts show that there is some role of CaCO_3 in the immobilization of phosphates practically in all fractions. This, however, does not mean the fixation of phosphate as it has been clearly shown by the grain yields and the P-uptakes (Table 2). In other words, a positive rather than a negative role could be attributed to CaCO_3 in this respect on this level of superphosphate application.

On the basis of the results obtained with extractants used commonly for the estimation of phosphorus availability the following conclusions were drawn:

The closest correlation was observed in case of using either acidic or bicarbonate extractants with fractions I and II; for fraction III the bicarbonate- and for fraction IV the acidic ammonium lactate solutions were preferred. This means that the continuous and actual nutrient supply of the

plants was primarily reflected in fractions I and II. It was only the DL-method with its lower buffering capacity that could not indicate appropriately the mobile P reserves of soils in which the CaCO_3 content was above 5%.

It was also proved that on chernozems with a CaCO_3 content below 15%, the AL-, CAL- and OLSEN-extractants are equally suitable for the estimation of the availability of soil-P.

Similar observations have been made by several authors [22, 24, 25, 27], i.e. that there is not necessarily a relationship — especially in relatively short duration — between the CaCO_3 content of the soil and the availability of phosphates. The residues of superphosphate remain available even in highly calcareous soils. BROMFIELD [3] and other authors [2, 8, 24] proved that the NH_4F solution (pH 8.2 or 8.5) extracted not only Al-phosphates but the relatively easily soluble Ca-phosphates, i.e. CaHPO_4 and its monohydrate, too. Therefore, this P-fraction can certainly be considered as the main reserve of available phosphorus both on acid and on calcareous soils [5, 28, 29].

Summary

The relationships between the CaCO_3 content of the plough-layer (0 to 20 cm) and the availability and/or extractability of superphosphate residues were investigated in a factorial (2^3) longterm NPK field experiment (1959—1970) on a chernozem soil variety.

It was OLSEN's bicarbonate method (pH 8.5) that gave the closest relation with the P-balance and with the several inorganic P-fractions. The linear regression between the values of the balance and the values obtained with AL (pH 3.7) and CAL (pH 4.2) extractants was also excellent. Only the DL-extractant gave unrealistic results on soils of high (>5%) CaCO_3 -content.

There was a decreasing tendency in the closeness of the correlation between the inorganic P-fractions and the P-residues in the soil from fraction I towards fraction IV, but all the four fractions reflected more or less the changes due to P fertilizing and/or to P-uptake. This change was significant in the case of fraction IV only with an appropriate N fertilizing. Though fraction IV contained 82—90% of the extracted inorganic P, the effect of fertilizing and P-uptake provoked the most distinguished changes mainly in fractions I and II, which contained at most a few per cent of the total P-content of the soil.

The slight positive correlation between fraction III and P-fertilizing could only be demonstrated if these values were corrected for an average humus level.

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