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Effect of liming on phosphorus in two soils of different organic matter content

I Changes of native and applied phosphorus in incubation experiment

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Abstract. The effect of increasing lime quantities on reactions of native and applied P was investigated in an incubation experiment performed with two acid mineral soils of pH 4.8 ($CaCl_2$). The soil samples differed considerably in the content of organic matter, which was reflected in their pH buffering power: in the fine sand, rich in organic matter (6.4 % org. C), liming raised the pH less than in the muddy fine sand (3.0 % org. C).

The level of native water-soluble P was markedly lowered in the incubated soil samples treated with nutrient salts. In the muddy fine sand, the decrease tended to be the smaller, whereas in the fine sand the greater, the more intensive liming was. This held true also of added P.

The changes in CHANG and JACKSON's P fractions did not alone satisfactorily explain the dissimilar response of soil P to lime treatments. The fate of P was concluded to be controlled by the quality and quantity of Al species differing in their affinity for P sorption. The changes in the solubility of P are a net result of processes enhancing and of those depressing the sorption tendency. In the fine sand soil of high initial content of water-soluble P, the detrimental effect of liming seemed to be attributed to the abundance of polymerized Al the affinity of which for P retention increased with intensified liming. Further, the high pH buffering power of this soil reduced the efficiency of lime to produce OH^- ions able to compete with phosphate for sorption sites. In the muddy fine sand soil, on the contrary, the formation of sorption-active sites was not equally marked and, owing to the weaker pH buffering, liming raised the OH^- concentration more effectively.

Introduction

Liming affects the reactions of soil P in many ways. For instance, it increases the concentration of OH^- ions able to displace phosphate from oxide surfaces and to alter the distribution of soil P between various fractions used for characterization of different P compounds (CHANG and JACKSON 1957, KAILA 1961 and 1965, HARTIKAINEN 1981 etc.). In acid soils, it seems, these reactions contribute to enhancing the solubility of P in water, even though the promoting effect of increasing pH is concluded to be partly dependent on the P status of the soil (HARTIKAINEN 1981).

On the other hand, the extent of the reactions of P is obviously controlled by the pH buffering power of the soil which relates to the content of organic matter. Further, organic anions also compete with phosphate for sorptive metal oxide surface. Their affinity to react with metal ions and, thus, to compete with phosphate is highly pH-dependent.

The purpose of the present incubation experiment was to elucidate the lime-induced reactions of native and added P in two soils differing markedly in their content of organic carbon. The results were assumed to demonstrate the role of organic matter and other related factors in controlling the efficiency of lime to raise pH and to affect the solubility of native and added P in soils.

Materials and methods

The incubation experiment was carried out with two acid surface soil samples taken from the experimental farm of Helsinki University. Characteristics of the soils are given in Table 1. The samples differed mainly in their content of organic carbon and secondary phosphates. The fine sand soil was sampled from a field used successfully for sugar beet cultivation in 1976–1979. During this period it was supplied with a total of 600 kg P per hectare. Before that, in 1975, the block was manured and limed. The muddy fine sand sample was taken from a field which grew oats, barley and hay in 1976–1979 and at that time had received a total of only 167 kg fertilizer P per hectare. This block was limed in 1969.

The particle size composition of the mineral material in the experimental soils was determined by a pipette method (ELONEN 1971). Soil pH was measured in a 0.01 M $CaCl_2$ suspension in the soil-solution ratio of 1 to 2.5. The content of organic carbon was determined by a modified WALKEY and BLACK wet combustion method (GRAHAM 1948) which was assumed to cover 80 % of the C in the soils. Aluminium, iron and manganese were extracted with 0.05 M NH_4-oxalate (pH 3.3) in the soil-solution ratio of 1:10 (w/v) and determined by an atomic absorption spectrophotometer. Phosphorus was fractionated according to a slightly modified CHANG and JACKSON (1957) procedure.

In the experiment, portions of 450 g of moist soil (corresponding to 390 g of air-dried muddy fine sand and 360 g of air-dried fine sand) were incubated in plastic pots with the following dosages of lime: 0, 0.6, 1.2 and 2.4 g.

Because the incubation experiment was connected with an analogous pot experiment, all the treat-

Table 1. Characteristics of experimental soil samples.

	Muddy fine sand	Fine sand
Clay %	22.4	22.5
pH ($CaCl_2$)	4.8	4.8
Org. C % of D.M.	3.0	6.4
P fractions ppm NH_4Cl-P	3.4	10.0
NH_4F-P	107	321
NaOH-P	286	277
H_2SO_4-P	325	285
Oxal. extr. Al ppm	1081	1533
” ” Fe ”	2729	2870
” ” Mn ”	16.3	12.4

ments received plant nutrients as follows: 100 mg N as NH_4NO_3, 20 mg Mg as $MgCl_2 \cdot 6 H_2O$, 0.5 mg B as H_3BO_3, 1.5 mg Cu as $CuSO_4 \cdot 5 H_2O$, 1 mg Mn as $MnSO_4 \cdot 4 H_2O$, 1 mg Zn as $ZnSO_4 \cdot 7 H_2O$ and 0.5 mg Mo as $Na_2MoO_4 \cdot 2 H_2O$. In addition, half of the pots received 40 mg P (corresponding to 104 and 114 mg P per kg of muddy fine sand and fine sand, respectively) as K_2HPO_4 in which they received also 100 mg of K. The pots incubated without P treatment received an equivalent quantity of K as KCl. The test was carried out with four replicates. The soils were incubated for 16 months.

Air-dried subsamples were analyzed after incubation periods of 4 and 16 months. Water-soluble P, extracted by a slightly modified van der PAAUW (1971) and SISSINGH (1971) method, was analyzed by the ascorbic acid method (ANON. 1969). The exchangeable Al was washed with four portions of 1 M KCl in the ratio of soil to solution of 1:5 (w/v) and determined by a modified Aluminon method (YUAN and FISKELL 1959). Soil Al was extracted also with 1 M NH_4OAc solution (according to McLEAN 1965) and determined by an atomic absorption spectrophotometer.

Results

The pH values of the incubated soil samples are reported in Table 2. In the fine sand pots incubated without lime and K_2HPO_4 addition, soil acidity increased by 0.3–0.4 pH units, whereas in the corresponding muddy fine sand pots by 0.5–0.6 pH units. It can be calculated from the titration curve, obtained by plotting the pH value against the lime dosage, that about 0.45 and 0.50 g of $CaCO_3$ per pot, respectively, was required to counterbalance this acidification.

Liming decreased the acidity of the muddy fine sand more effectively than that of the fine sand sample. Further, in the former, the K_2HPO_4 treatment tended somewhat to raise pH. It should be mentioned that immediately after application of nutrient salts the pH of the muddy fine sand and fine sand was 5.4 and 5.2 in the pots treated with P and 4.5 and 4.6 in those without P, respectively.

Table 3 shows the incubation treatment to have depressed markedly the level of native water-soluble P in both soil samples. It was, however,

Table 2. pH of incubated soil samples *

	Incubated for			
Lime	4 months		16 months	
added g	–	P added	–	P added
		Muddy fine sand (pH 4.8)		
0	4.2^{a}	4.3^{ab}	4.3^{ab}	4.4^{b}
0.6	4.9^{cd}	5.1^{e}	4.9^{c}	5.0^{de}
1.2	5.6^{fg}	5.7^{g}	5.5^{f}	5.6^{f}
2.4	6.3^{h}	6.4^{hi}	6.4^{h}	6.5^{i}
		Fine sand (pH 4.8)		
0	4.4^{a}	4.4^{ab}	4.5^{bc}	4.5^{c}
0.6	4.9^{d}	4.9^{d}	4.9^{d}	4.9^{d}
1.2	5.3^{e}	5.3^{e}	5.3^{e}	5.3^{e}
2.4	5.9^{f}	5.9^{f}	5.9^{f}	5.9^{f}

* Both soils are tested separately. The means followed by a common letter do not differ at P=0.05.

interesting to notice that in the muddy fine sand the decrease tended to be the smaller and in the fine sand the greater, the more intensive the liming was. In the most heavily limed pots, prolonged incubation caused some further reduction in the solubility of P in water.

Only a minor portion of applied P was recovered in the water extractable fraction (Table 3): at four months, 7.9–8.5 % in the muddy fine sand and 11.9–14.9 % in the fine sand sample. In the latter soil, liming seemed to depress the solubility of added P, too. Besides, a further conversion of applied P into a less soluble form took place during the long-term incubation.

Table 3. Extractability of soil P and applied P into water.

	Soil P ppm		Recovery of added P %	
Lime	Incubation time, months			
added g	4	16	4	16
		Muddy fine sand		
	(Original 10.7)			
0	5.4^{a}	5.7^{a}	8.0	5.6
0.6	5.6^{a}	5.8^{a}	8.5	6.4
1.2	6.3^{ab}	6.3^{ab}	7.9	6.0
2.4	7.3^{b}	6.1^{a}	8.3	6.4
		Fine sand		
	(Original 42.1)			
0	28.9^{de}	29.5^{e}	14.9	12.2
0.6	28.2^{de}	26.7^{d}	13.3	11.0
1.2	25.2^{c}	25.0^{bc}	13.2	10.1
2.4	23.6^{b}	20.7^{a}	11.9	8.7

The fractionation analyses gave some intimations of the fate of native as well as applied P in the soils (Table 4). Liming seemed to induce a redistribution of native P between NH_4F, NaOH and H_2SO_4 extractable forms representing P bound by Al, Fe and Ca, respectively, even if the data obtained did not quantitatively account for the changes. In the muddy fine sand sample, the NaOH-P was markedly depleted and H_2SO_4-P increased slightly. In the fine sand, on the other hand, the various fractions were affected to a lesser extent, but some enrichment occured in the NH_4F and NaOH soluble forms.

The difference between a given fraction in the soils treated and not treated with P was assumed to represent recovery of applied P. Table 4 suggests liming not to have affected decisively the distribution of sorbed P. In the fine sand soil, added P was retained mainly in the fluoride soluble fraction, whereas in the muddy fine sand sample, relatively high quantities were sorbed also in the hydroxide extractable one. Further, a surprisingly marked accumulation in the acid soluble form occured in the fine sand soil, also in the unlimed treatment.

The enrichment in native NH_4F-P in the limed fine sand soil did not, however, promote the solubility of P in water (cf. Table 3). This rendered it necessary to investigate the role of some by-reactions induced by the $CaCO_3$ treatment. Because the NH_4F fraction is assumed to represent primarily the Al bound resources, attention was paid to reactions of soil Al.

Table 4. P fractions (ppm) in soils incubated for 16 months with or without P.

	P extracted sequentially by							
Lime	NH_4Cl		NH_4F		NaOH		H_2SO_4	
added g	–	P	–	P	–	P	–	P
				Muddy fine sand				
0	3.8^{b}	6.3^{d}	109^{b}	154^{c}	296^{c}	330^{e}	317^{a}	320^{a}
0.6	2.7^{a}	5.0^{c}	105^{ab}	155^{c}	280^{b}	319de	318^{a}	321^{a}
1.2	2.5^{a}	5.0^{c}	103^{a}	153^{c}	274^{b}	316^{d}	326^{ab}	325^{ab}
2.4	3.8^{b}	7.5^{e}	110^{b}	156^{c}	261^{a}	308^{cd}	333^{b}	335^{b}
				Fine sand				
0	11.3^{c}	16.0^{g}	318^{a}	388^{c}	271^{a}	278^{a}	285^{a}	313^{b}
0.6	8.8^{ab}	13.5^{e}	322^{ab}	403^{d}	273^{a}	283^{ab}	284^{a}	322^{b}
1.2	8.0^{a}	12.5^{d}	332^{b}	407de	277^{a}	277^{a}	283^{a}	305^{b}
2.4	8.9^{b}	14.7^{f}	336^{b}	420^{e}	286^{ab}	298^{b}	273^{a}	306^{b}

Figure 1 shows the relationship between pH and the content of exchangeable Al in the experimental soils incubated for 16 months. As expected, KCl displaceable Al sharply diminished as pH increased. In the fine sand sample, the initial exchangeable Al resources were lower and decreased less than in the muddy fine sand sample. In the latter soil, the Al concentration points for samples treated or not treated with K_2HPO_4 fell on the same graph, whereas in the fine sand soil, application of P caused an additional depression in the exchangeable Al fraction.

Because the fine sand soil, poorer in the exchangeable Al, contained more abundantly oxalate soluble Al than the muddy fine sand, the experimental soils can be concluded to differ in the distribution of their Al resources into various forms. For more accurate information about active Al reserves, the incubated samples as well as the original ones were extracted with NH_4-acetate which, according to McLEAN (1965), attacks in addition to exchangeable reserves also polymerized Al hydroxides and organic Al complexes. The differences between the NH_4-acetate and KCl extractable forms, presented in Table 5, indicate the initial content of the polymerized or complexed Al

Table 5. Differences (ppm) between NH_4OAc and KCl soluble Al in the various treatments.*

	Muddy fine sand		Fine sand	
Treatment	–	P added	–	P added
Original soil	85^{cd}		147^{d}	
$CaCO_3$ 0 g/pot	67^{ab}	64^{a}	122^{b}	121^{b}
” 0.6 ”	91^{d}	97^{e}	138^{cd}	135^{c}
” 1.2 ”	83^{c}	81^{c}	126^{b}	119^{b}
” 2.4 ”	70^{b}	66^{ab}	110^{a}	103^{a}

* Both soil samples are tested separately.

forms to be markedly higher in the fine sand sample rich in organic matter. Further, the data reveal the incubation treatments without lime as well as with moderate and high lime dosages to diminish the difference between these forms.

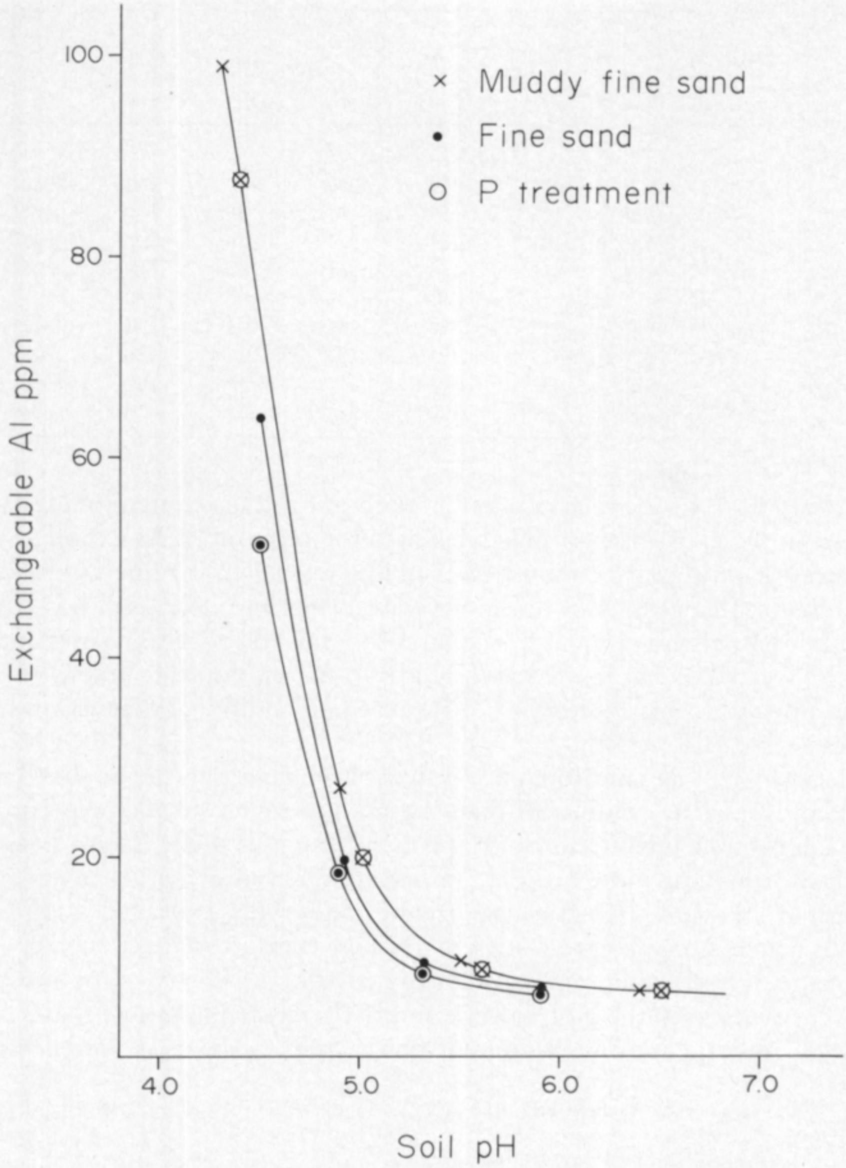

Fig. 1. Content of exchangeable Al in incubated soils as a function of soil acidity.

Discussion

The addition of nutrient salts immediately increased the ionic strength in soil solution and, thus, decreased the pH values. Obviously, the nitrification of added NH_4-N during the incubation contributed to a further decrease in soil pH. Also the decrease in the level of water-soluble P in all the incubation treatments can be ascribed to the increased ionic strength, shown in most soils to depress desorption of P (RYDEN and SYERS 1975, HARTIKAINEN and YLI-HALLA 1982).

In the muddy fine sand soil, water- soluble P was somewhat increased as a result of liming, but remained far below the level in the original sample. In the fine sand soil, on the contrary, liming seemed to cause a considerable additional reduction in this P fraction. COLEMAN et al. (1960) and MOKWUNYE (1975) have reported similar exceptions to the general rule that liming improves the solubility of soil P. They ascribed these to the hydrolysis and polymerization of exchangeable Al^{3+} ions upon pH increase, because freshly polymerized Al compounds more effectively retain phosphate than do Al^{3+} ions.

The hydrolysis of exchangeable monomeric hexaquo-aluminium ion can be presented schematically as follows:

$$Al(H_2O)_6^{3+} + H_2O \rightleftharpoons Al(OH)\,(H_2O)_5^{2+} + H_3O^+$$

The reaction is driven to the right by consumption of H_3O^+ ions, gradually leading to the complete neutralization of Al ions and to the formation of $Al(OH)_3(H_2O)_3^0$. The hydrolysis products of trivalent Al ions are known, however, to polymerize rapidly and to form large multicharged units.

The results of the present study, too, imply that the lime-induced redistribution of P between various fractions was, at least to some extent, accompanied by simultaneous changes in the quantities of sorptive compounds. Even if an enrichment in the Al-bound fraction is considered to enhance the solubility of P in water (HARTIKAINEN 1981 and 1982), this did not hold true of the fine sand soil. The exchangeable Al did not, however, explain the dissimilar responses of the experimental soils to liming. In the muddy fine sand, where the reduction in exchangeable Al was more considerable than in the fine sand, P retention did not increase.

On the other hand, organic matter contributes to the relationship between pH and exchangeable Al. According to EVANS and KAMPRATH (1970), soils of high organic matter content involve less exchangeable Al at a given pH than soils poorer in this material, which indicates complexation reactions. Also in the present study, the content of polymerized or organically bound (and more sorption active) Al species was higher in the fine sand soil rich in organic matter. This gives reason to suppose that they were responsible for a decrease in H_2O-P as liming was intensified.

Figure 1 and the data in Table 5 give some evidence of the pH-dependent equilibrium between various Al species and give some indication of the fate of P. The increased acidity in the unlimed soils transferred the equilibrium between polymerized and exchangeable forms to the direction of the latter species. In the fine sand soils incubated for 16 months, this was reflected as a higher solubility of P in water (see Table 3). Further, the level of hydroxy-Al in the soils supplied with 0.6 g of $CaCO_3$ was about equal to that in the original ones. This may be due to this dosage being required to counterbalance the incubation-induced acidification. The decreasing contents at higher liming intensities may, in turn, indicate a more complete neutralization of hydroxy-Al to compounds no more extractable in NH_4-acetate.

If the latter assumption is valid, Table 5 justifies to suppose that a higher quantity of hydroxy-Al species reached this neutralization stage in the fine sand soil richer in organic matter than in the muddy fine sand sample. The conclusion is supported by the view presented by HARGROVE and THOMAS (1982) according to which organically bound Al is apparently hydrolyzed to a greater extent at a given low pH. Thus, it is theoretically possible that owing to the greater polymerization degree the affinity of Al for P retention is higher in soils of high organic matter content.

In the fine sand soil, the addition of K_2HPO_4 decreased the exchangeable Al, which indicates some interaction between Al and P. Because this sample was relatively poor in exchangeable Al^3+ and rich in native water-soluble P, the application of P possibly led to the solubility product of an aluminium phosphate to be exceeded and to its precipitation. ROBARGE and COREY (1979) observed in their cation resin study a similar decrease in the percentage of Al^{3+} ions at higher P additions but not at low ones. The quite marked accumulation of added P seen in the H_2SO_4 soluble fraction may be a result of the inaccuracy of the fractionation method and the formation of some discrete compounds. Therefore, further studies are needed to elucidate the interaction between P and exchangeable Al^{3+} ions in soils.

The results discussed above illustrate the pH-dependent relationship between P retention tendency, soil Al and organic matter. In addition, organic matter and Al may also indirectly affect the fate of P. Al-organic matter complexes are considered responsible for the weak-acid behaviour of the organic matter in acid soils and an important source of pH buffering (COLEMAN and THOMAS 1967, KAILA 1971, BLOOM et al. 1979). Recently, HARGROVE and THOMAS (1982) have proved the pK_a values for the organic matter samples to increase considerably (the acid strength to decrease) as the Al content increases. Thus, in the fine sand soil of high content of hydroxy-Al species and high pH buffering power, liming did not effectively raise the pH nor produce OH^- ions enough to compete successfully with phosphate ions for sorption sites. On the other hand, in the muddy fine sand, poorer in the hydroxy-Al and of lower pH buffering power, liming increased the OH^- ion concentration sufficiently to cause a displacement of sorbed phosphate.

Even if the comparison of two soil samples does not justify categorical conclusions, the results suggest the quantity and quality of Al species and the factors involved to be of importance in controlling the efficiency of liming to regulate the changes in native and added P in soils.

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Kalkituksen vaikutus fosforiin kahdessa erityyppisessä hietamaassa

I Luontaisen ja maahan lisätyn fosforin muutokset muhituskokeessa

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Kahdella happamalla maanäytteellä ($pHCaCl_2$ 4.8) tehdyssä muhituskokeessa selvitettiin nousevien kalsiittikalkkimäärien vaikutusta maan luontaisen sekä maahan lisätyn fosforin reaktioihin. Maanäytteet poikkesivat selvästi toisistaan orgaanisen aineksen suhteen, mikä heijastui niiden pH-puskurikyvyssä: kalkitus nosti runsaasti orgaanista ainesta (6.4 % org. C) sisältäneen karkean hiedan pH:ta vähemmän kuin liejuisen hiedan (3.0 % org. C).

Ravinnesuolojen lisäys pienensi merkittävästi vesiliukoisen fosforin määrää molemmissa maanäytteissä. Liejuisen hiedan kalkituissa koejäsenissä väheneminen ei kuitenkaan näyttänyt olevan yhtä voimakasta kuin kalkitsemattomassa. Karkeassa hiedassa kalkitus sen sijaan edelleen selvästi vähensi sekä luontaisen että maahan lisätyn fosforin uuttumista.

Fosforin fraktioissa tapahtuneet muutokset eivät selittäneet riittävästi kalkituksen erilaista vaikutusta maanäytteiden fosforin uuttuvuuteen, joka näytti pikemminkin riippuvan kalkituksen vaikutuksesta maan sorptioaktiivisen aluminiumin määrään. Alunperin runsaasti vesiliukoista fosforia sisältäneessä karkeassa hiedassa kalkituksen fosforin liukoisuutta vähentävä vaikutus näytti aiheutuvan siitä, että tässä näytteessä oli runsaasti orgaaniseen ainekseen sitoutunutta tai polymeroitunutta aluminiumia, jonka fosforinsitomistaipumus lisääntyi kalkittaessa. Lisäksi tehokkaan pH-puskuroinnin vuoksi OH-ionien pitoisuus ei ilmeisesti noussut riittävästi, jotta ne olisivat pystyneet merkittävästi kilpailemaan fosfaatin kanssa sorptiopaikoista. Liejuisessa hiedassa aluminiumin muodostaman uuden sorptiopinnan syntyminen ei ollut yhtä merkittävää. Lisäksi heikomman pH:n puskuroinnin vuoksi kalkitus pystyi tässä näytteessä nostamaan tehokkaammin OH-ionien pitoisuutta ja siten lisäämään fosforin liukoisuutta.