

Chloride and sulphate solutions as extractants for soil P

I Effect of ionic species and ionic strength on P desorption

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Abstract. The effect of Cl and SO₄²⁻ anions as well as that of the ionic strength on the desorption of soil P were studied in 102 mineral soil samples by extracting them with KCl and K₂SO₄ solutions at ionic strengths of 0.025 and 0.1. The quantities of salt soluble and water soluble P in the soils were compared.

Both sulphate solutions extracted more P in every single sample than either of the chloride solutions. On the other hand, the material could be divided into three groups according to the position of water in the order of extraction efficiency. In the group W>S>Cl, consisting of 53 samples, water was the most effective extractant, in the group S>W>Cl of 37 samples water was less effective than sulphate solutions but more effective than the chloride solutions, and in 12 samples (S>Cl>W) water was the least effective, less effective than even chloride.

The groups classified according to P extractability did not deviate from each other in terms of soil texture, pH or org. C %, but the salt solutions tended to be the more effective extractants the poorer the P status of the soil was. The salt soluble and water soluble P which are closely related to each other correlated with the same factors. Exclusive of the group of 12 samples (S>Cl>W), the highest values of correlation coefficient were found for NH₄F-P/Al, i.e. the molar ratio of NH₄F soluble P (CHANG and JACKSON's method) to oxalate extractable Al ($r=0.89^{***}-0.93^{***}$). The absolute differences between amounts of P dissolved in KCl and K₂SO₄ solutions of the same initial ionic strength were the greater the more water soluble P the samples contained ($r=0.58^*-0.94^{***}$).

An increase in ionic strength tended to depress the extractability of P in both salt solutions. Therefore the ligand exchange between sulphate and phosphate or hydroxyl was regarded unprobable. A theory of the extraction mechanism of sulphate was presented. The greater extraction efficiency of sulphate was assumed to be caused by the exchange reactions with H₂O groups which affect the ionic strength in a solution and the electric condition near the surface.

Introduction

The ionic composition and concentration of a particular ion in soil solution may vary markedly during the growth season. The phenomenon is well-known in cations, but it holds good also for anions. The competition between anions in soil is maintained by changes in the ionic strength and

anion composition in soil solution which, in turn, are affected e.g. by the fertilization, irrigation and rain as well as water and nutrient uptake of plants.

Thus, external factors may have some effect on the solubility of soil P. The desorption of sorbed anions from soil tends to be depressed by application of so-called indifferent anions (RYDEN and SYERS 1975) and enhanced by application of anions with sorption tendency (DEMOLON and BASTISSE 1934, HINGSTON et al. 1967, etc.). The extent of displacement is dependent on the sorption affinity of the anions involved and their activity ratios in a solution (e.g. HARWARD and REISENAUER 1966) and the anion saturation on the surface of oxides providing sorption sites (e.g. HARTIKAINEN 1979). Also the nature of sorption components may be of importance, but mostly only the hydrated oxides of Al and Fe only are practically responsible for an anion retention observed.

The purpose of the present study was to illustrate the effectiveness of various electrolyte solutions as P extractants. In addition to the action of ionic strength and anion species in a given solution, also the importance of the above factors in different soils was investigated, comparing the extractability of soil P by KCl and K₂SO₄ solutions at two ionic strengths. The results were assumed to give some information about the consequences of the recent development in Finland, where the significance of chloride as a fertilizer component has increased at the expense of sulphate.

Materials and methods

The material consisted of 102 mineral soil samples: 17 heavy clay soils, 51 coarser clay soils and 34 non-clay soils. Exclusive of two heavy clay soil samples no more available, the material was the same as that presented in an earlier paper by HARTIKAINEN (1982).

Soil P was extracted by two KCl and K₂SO₄ solutions of ionic strengths of 0.025 and 0.1, the KCl concentration being 0.025 M and 0.1 M and the K₂SO₄ concentration 0.0083 M and 0.0333 M, respectively. According to MOORE (1974, p. 443), solutions equal in ionic strength involve equal electrostatic forces. KOENIGS et al. (1981) state that the ionic strength in plough layer is about 0.1 immediately after fertilization. The soil - solution ratio was 1:60 (w/v) and desorption time 18 hours. The soil suspensions were centrifuged and filtered through a membrane filter of 0.2 μ m pore size. The pH of the extracts was measured and the solutions were analyzed for P by the molybdenum blue method (ANON. 1969) and for Ca by an atomic absorption spectrophotometer (acetylene - nitrogen oxidule flame).

Results

Table 1 shows the relative amounts of P dissolved from various textural soil classes by KCl and K₂SO₄ solutions of ionic strengths of 0.025 and 0.1. By way of comparison, the quantities of water soluble P (analyzed by

Table 1. Relative extractability of soil P in different solutions. Means with the confidence limits at the 95 per cent level.

Soil class	H ₂ O	KCl		K ₂ SO ₄	
		I = 0.025	I = 0.1	I = 0.025	I = 0.1
Heavy clays	100	68±33	56±31	89±38	80±34
Coarser clays	100	104±41	91±35	134±15	123±42
Non-clay soils	100	102±18	98±21	142±27	140±30

HARTIKAINEN 1982) were denoted by 100. Comparison of means of P quantities extracted by salt solutions and water soluble reserves showed that from heavy clay soils the salt solutions dissolved less P, but from the other soil groups at least as much or even more than did water. A closer examination disclosed, however, coarser clay as well as non-clay soil samples, where P was considerably more extracted by water than by either salt solution.

When comparing only salt solutions as extractants for soil P, both sulphate solutions were found more effective than the chloride solutions. In 88 samples the order of solubility of P in various extractants was:

$K_2SO_4(I = 0.025) > K_2SO_4(I = 0.1) > KCl(I = 0.025) > KCl(I = 0.1)$

This was the average order in the whole soil material, but there were 14 samples from which either of the more concentrated salt solutions seemed to dissolve slightly more P than the less concentrated solution. The results remained, however, statistically uncertain.

Because it was essential in this study to find out the soil properties which control the effectiveness of various salt solutions as extractants for soil P, the soil sample material was divided into three categories according to the solubility of P in different solutions. The criterion of classification was a difference of 0.1 mg/kg between amounts of P dissolved. The extractability sequence was expressed by the following abbreviations: W (= water), S (= sulphate solution) and Cl (= chloride solution). Thus, in the group termed $W > S > Cl$, consisting of 53 soil samples, P was most effectively desorbed by water. In 12 soil samples, termed $S > Cl > W$, water was the most ineffective extractant. In the group of 37 samples, $S > W > Cl$, the extraction ability of water was poorer than that of sulphate solutions but better than that of chloride solutions.

The coarser clay and non-clay soil samples were evenly distributed in the different categories, but there was only one heavy clay soil sample in the groups $S > W > Cl$ and $S > Cl > W$. Most samples in the group $S > Cl > W$ originated from virgin soils or subsoils. The means (with the confidence limits at the 95 per cent level) of some soil characteristics in these soil classes are listed in Table 2 and the means of P amounts dissolved by various solutions in Table 3. By way of comparison, also the data on water soluble P are presented.

The soil classes were equal in means of pH and the differences between the contents of organic carbon were not statistically significant. On the contrary, the soil group $S > Cl > W$ seemed to be poorer in secondary

Table 2. Soil properties in various groups classified according to extractability sequence of P.

	Soil group		
	W > S > Cl	S > W > Cl	S > Cl > W
clay %	47±6	32±4	32±12
pH	5.2±0.1	5.2±0.2	5.0±0.5
org. C %	4.8±0.7	3.6±0.4	3.1±1.1
NH ₄ F-P ppm [*])	131±26	95±24	63±42
NaOH-P ppm [*])	258±30	181±22	152±79
Oxal.extr. Al mmol/kg	70±8	57±7	71±23
Oxal.extr. Fe mmol/kg	80±7	63±7	64±14
NH ₄ F-P/Al ($\cdot 10^2$)	7.1±1.7	5.5±1.3	2.4±0.8
NaOH-P/Fe ($\cdot 10^2$)	10.6±1.2	9.6±1.2	7.9±3.8

^{*}) = analyzed by a modified CHANG and JACKSON's (1957) fractionation method.

Table 3. Amounts of P (ppm) dissolved by various solutions from soil groups of different extractability sequence.

Extractant	Soil group		
	W > S > Cl	S > W > Cl	S > Cl > W
H ₂ O	15.6±5.7	9.5±3.6	0.8±0.4
KCl (I = 0.025)	10.9±4.9	8.4±3.3	1.6±0.5
KCl (I = 0.1)	9.5±4.5	7.8±3.1	1.6±0.5
K ₂ SO ₄ (I = 0.025)	13.2±5.4	10.6±3.7	2.3±0.8
K ₂ SO ₄ (I = 0.1)	12.3±5.1	10.0±3.7	2.3±0.8

phosphates with a markedly lower molar ratio NH₄F-P/Al than the other groups. In this group the sulphate solutions seemed to desorb about three times and the chloride solutions about twice as much P as did water. In the class W > S > Cl, on the other hand, the chloride solutions dissolved on an average 60–70 % and the sulphate solutions about 80–85 % of the water soluble P.

The effect of ionic species and ionic strength of the extractant on the desorption of P was studied by the t-test for the means of differences between the paired measurement data (STEEL and TORRIE 1960). As can be seen from the t-values presented in Table 4, the differences between P amounts dissolved in the sulphate and chloride solutions were statistically significant in all soil groups, and so were also the differences between P quantities dissolved by water and all the salt solutions. In addition, exclusive of the soil group S > Cl > W, an increase in ionic strength tended to depress the solubility of P. It should be mentioned that in the group W > S > Cl containing on an average most abundantly water soluble P, the differences between KCl soluble and K₂SO₄ soluble P were significantly more marked at the ionic strength of 0.1 than at 0.025. This was due to an increase in the ionic strength reducing more strongly the extraction ability of the chloride solution than that of sulphate solution.

Table 4. t-values for the means of differences between quantities of P extracted by various solutions.

Difference	Soil group		
	W > S > Cl 53	S > W > Cl 37	S > Cl > W 12
KCl (0.025)–K ₂ SO ₄ (0.025)	–8.49 ^{***}	–8.37 ^{***}	–3.99 ^{***}
KCl (0.1)–K ₂ SO ₄ (0.1)	–7.95 ^{***}	–7.20 ^{***}	–4.24 ^{***}
KCl (0.025)–KCl (0.1)	–6.19 ^{***}	–5.71 ^{***}	–0.72 ^{n.s.}
K ₂ SO ₄ (0.025)–K ₂ SO ₄ (0.1)	–4.79 ^{***}	–5.24 ^{***}	–0.37 ^{n.s.}

n.s. = not significant

In order to get further information about the extraction activity of various salt solutions, the pH of the extracts was measured and the equilibrium solutions were analyzed for Ca exchanged by K from the soil to the extractant. It was observed that the filtrates of higher ionic strength contained more abundantly exchanged Ca, but there were no differences between the Ca concentrations in KCl and K₂SO₄ extracts of the same initial ionic strength.

The pH of the K₂SO₄ extracts were 0.05–0.40 and 0.00–0.35 pH units higher than that of the KCl extracts at ionic strengths of 0.1 and 0.025, respectively. The differences were statistically significant in all soil groups. An increase in the ionic strength lowered the pH level slightly, the decrease of pH being greater in KCl than in K₂SO₄ extracts.

The relationship between the salt soluble P and soil characteristics was investigated by the correlation analysis. The quantities of P desorbed by both electrolytes were closely related to the amount of water soluble P, the correlation coefficients varying in the groups W > S > Cl and S > W > Cl from $r = 0.98^{***}$ to $r = 0.998^{***}$, in the group S > Cl > W from $r = 0.74^{**}$ to $r = 0.85^{**}$. As anticipated, the quantities of P dissolved by all these extractants were correlated with the same soil properties. In the groups W > S > Cl and S > W > Cl, the highest values of correlation coefficients were for the molar ratio NH₄F-P/Al ($r = 0.89^{***}$ – 0.92^{***}), but in the group S > Cl > W for the ratio NaOH-P/Fe ($r = 0.60^{*}$ – 0.77^{***}). When the effect of NaOH-P/Fe was eliminated in the first two groups, the values of the partial correlation coefficients for relations between salt soluble P and NH₄F-P/Al decreased only somewhat ($r = 0.75^{***}$ – 0.85^{***}). In the group S > Cl > W, on the contrary, the partial correlation analyses showed the extractability of P not to be related to these molar ratios. In this soil category the salt soluble P rather seemed to be related to NaOH-P ($r = 0.59^{*}$ – 0.79^{**}), but when the influence of NH₄F-P was eliminated the values of partial correlation coefficients were lowered to $r = 0.43^{n.s.}$ – 0.66^{*} . The other soil properties studied seemed not to correlate with KCl or K₂SO₄ extractable P in soils.

Comparison of the salt solutions showed that the absolute differences between the amounts of P dissolved by KCl and K₂SO₄ tended to be the greater the more water soluble P the soil contained. The correlation coefficients for the relation between that difference and water soluble P ranged $r = 0.84^{***}$ – 0.94^{***} and $r = 0.58^{*}$ – 0.87^{***} at ionic strengths of 0.1 and 0.025,

respectively, the lowest values of r being found in the soil group $S > Cl > W$. In the soil group $W > S > Cl$, an increase in ionic strength tended, to some extent, to increase the absolute differences but not in the other groups. It should be mentioned also that the differences between the pH values of the extracts seemed not to be associated with the differences in P quantities dissolved by KCl and K_2SO_4 solutions.

Discussion

According to the prevalent notion, water is normally able to desorb more P from soils than solutions of neutral salts (e.g. LEHR and WESEMAEL 1952, CLARK and PEECH 1962, STÅHLBERG 1980). In the present material consisting of 102 ordinary Finnish soils, however, this was the case in 53 samples only. In fact, the soil samples could be divided into three groups differing in the sequence of extractability of soil P in water and salt solutions. As found earlier e.g. by KURTZ et al. (1946) and STÅHLBERG (1980), also the salt solutions differed in their ability to desorb P: both sulphate solutions extracted P more abundantly than either one of the KCl solutions.

KCl and K_2SO_4 soluble P as well as the absolute differences between P quantities dissolved by these extractants seemed to be closely related to the amounts of water soluble P in soils. Because in this soil material the molar ratio NH_4F-P/Al is found to be the first-rate factor controlling the level of water soluble P (HARTIKAINEN 1982), the same factor could be expected to correlate closely also with the amounts of salt soluble P. This suggests that an increase in P coverage on oxide surfaces (a decrease in bonding strength) improves P extractability in electrolyte solutions, too. However, the disparities between the soil samples in the sequence of P extractability in various solutions imply the activity and effectiveness of a particular extractant likely to be dependent on soil characteristics.

An increase in the ionic strength depressed the desorption of P, but enhanced the exchange of cations to the solution. Equal amounts of Ca^{2+} were exchanged from the soils by KCl and K_2SO_4 extractants of the same ionic strength, even if KCl solutions contained 1.5 times more K^+ than the corresponding K_2SO_4 solutions. This shows the ionic strength to be a decisive factor in exchange reactions. Secondly, it demonstrates that the electrostatic forces in the extractants were of the same magnitude and, thus, the difference in the desorption ability was caused by the disparity in the nature of the anions.

The desorption ability of an anion can be decided from its sorption tendency. The retention of phosphate by hydrated oxides of Al and Fe is considered to occur by the so-called ligand exchange. The theory of acids and bases presented by PEARSON (1966) is applied when explaining this reaction in soils (AURA 1980): the central ions Al^{3+} and Fe^{3+} are hard Lewis acids preferring to associate with hard bases with low polarizability and high electronegativity (e.g. F^- and O^{2-}). The pK_a -value of an acid corresponding to the oxyanion shows the ability of anion oxygen to bind a proton (a hard

Lewis acid) and also the nucleophilic strength on which the binding of oxygen by the central ion primarily is dependent. Thus, on the basis of the greater basicity, the SO_4^{2-} ion can be concluded to have a greater sorption tendency than the Cl^- ion.

Also the pH values of the extracts suggest some disparity in the action of the salt solutions. On the basis of Ca^{2+} exchange, it can be concluded that equal amounts of H^+ ions as well would have been exchanged from the soil by both electrolyte solutions at the same ionic strength. The pH in the K_2SO_4 extracts was, however, always somewhat higher than that in KCl extracts and an increase in ionic strength lowered pH more in KCl than in K_2SO_4 extracts. Also CHAO et al. (1965) found similar differences in pH in KCl and K_2SO_4 soil filtrates in spite of the salt solutions compared being of the same normality, not of the same ionic strength. They assumed the differences in pH values to be due to the greater ability of sulphate to replace OH^- ions from the soil. If this occurs, the OH^- exchanged does not originate from the oxide surface. According to HINGSTON et al. (1972) and RAJAN (1979), sulphate belongs to anions which are more strongly bound to the oxides than is water, but cannot change the structure of oxides, i.e. they are not firmly bound by oxides above the point of zero charge (pzc). Also the pK_a value corresponding to the OH^- ion is so high, as compared to that corresponding to SO_4^{2-} , that this specific ligand exchange is questionable.

P desorption can further be assumed to improve when the concentration or activity of an ion, capable of specific ligand exchange, in the extract increases. Because an increase in ionic strength (i.e. sulphate concentration) depressed the P desorption, a direct ligand exchange with phosphate hardly explains the greater desorptive power of sulphate as compared to that of chloride.

On the basis of the facts stated above, some contributory factors improving P desorption seem to be active in the K_2SO_4 extraction. Further investigations are needed, but some theoretical aspects can be presented. The pH in the extracts being a result of reactions occurring in the treatment, it gives reason to suppose that the higher pH in the K_2SO_4 extracts indicates an enhanced H^+ adsorption rather than the desorption of OH^- . This would mean that part of H^+ ions exchanged from the soil would have reacted with SO_4^{2-} , forming HSO_4^- . If this occurs, it can be expected to have an additional consequence. Provided the quantities of cations exchanged by KCl and K_2SO_4 solutions are equal, the formation of some HSO_4^- decreases the ionic strength of sulphate solutions as compared to that of chloride solutions which, in turn, may favour the desorption of P from soil. This process is not possible in the chloride solution.

H_2SO_4 is, however, a strong acid likely to be completely dissociated at the pH range prevailing in the soil. Thus, it seems more obvious that the higher pH of K_2SO_4 extracts would result from an enhanced adsorption of H^+ ions by HPO_4^{2-} ions, these being more abundantly dissolved in the sulphate solutions. The greater desorption power of sulphate, on the other hand, may be caused by its ability to replace H_2O groups from the surface of oxides (HINGSTON et al. 1972, RAJAN 1979). This exchange reaction can

favour P desorption by lowering the ionic strength in the solution or/ and by weakening the bonding strength of P due to the increasing negative charge near the surface. Consequently, it can be concluded that the replacement power of sulphate increases with a decrease in the P coverage and in the soil pH because both these factors contribute to an increased portion of H₂O groups on the oxide surface.

If this hypothesis presented above is valid, it could explain, at least to some extent, the different extractability sequence in the soils. The P replacement power of sulphate tended to increase as compared to that of water and chloride solutions when the content of secondary P as well as the P coverage decreased. Nevertheless, further studies are needed to elucidate the significance of cation exchange reactions as well as that of the ligand exchange between sulphate ions in a solution and H₂O groups on the oxide surface in P desorption.

Plants need plenty of P during the early growth season. However, the ionic strength of the soil solution is then highest and the P concentration therefore lowest (WIKLANDER and ANDERSSON 1972, KOENIGS et al. 1981). According to ERIKSSON (1940), yields may even decrease because of the depressing effect of KCl fertilization on the solubility of soil P. If sulphate enhances P concentration also in soil solution, where the solution – soil ratio is much below one, it would be advantageous to give potassium fertilization using sulphate salt instead of chloride salt. The weight and cost of fertilizer containing a certain mass of potassium would, however, increase. Further studies are needed to confirm the advantages or disadvantages of these contradicting points of view.

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SELOSTUS

Kloridi- ja sulfaattiliuokset maan fosforin uuttajina

I Ionilajin ja ionivahvuuden vaikutus P:n desorptioon

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Tutkimuksessa selvitettiin liuoksen anionien vaikutusta fosforin desorptioon 102 kivennäismaanäytteessä. Vertailtavien KCl- ja K₂SO₄-liuosten ionivahvuus säädettiin yhtä suureksi (0.025 ja 0.1), jotta liuoksissa vaikuttavat elektrostaattiset voimat olisivat yhtä suuret. Fosforin lisäksi utteista määritettiin Ca:n pitoisuus ja pH. Suolaliuoksiin uuttuneita P:n määriä verrattiin vesiliukoisen P:n määriin.

Sulfaattiliuokset olivat aina kloridiliuoksia tehokkaampia P:n uuttajia. Sen sijaan veden sijoittuminen tehokkuusjärjestyksessä vaihteli: 53 näytteessä (ryhmä W > S > Cl) se uutti eniten, 12 näytteessä (ryhmä S > Cl > W) vähiten ja 37 näytteessä (ryhmä S > W > Cl) se oli heikompi uuttaja kuin sulfaattiliuokset mutta tehokkaampi kuin kloridiliuokset. Uuttumisjärjestyksen mukaan muodostetut ryhmät eivät poikenneet toisistaan lajitekoostumuksen, pH:n ja orgaanisen aineksen pitoisuuden suhteen.

Suolaliuoksiin ja veteen desorboituneet P:n määrät korreloivat voimakkaasti keskenään ($r = 0.74^{**} - 0.998^{***}$). Kloridi- ja sulfaattiliukoisen P:n määrä näytti kytkeytyvän kiinteimmin NH₄F-liukoisen P:n (määritetty CHANGin ja JACKSONin menetelmällä) ja oksalaattiuuttoisen aluminiumin suhteeseen ($r = 0.60^* - 0.92^{***}$). K₂SO₄-liuos vaihtoi maasta yhtä paljon kalsiumia kuin ionivahvuudeltaan vastaava KCl-liuos, mutta sen pH pyrki olemaan jonkin

verran korkeampi. Nämä tekijät eivät kuitenkaan näyttäneet selittävän suolaliuosten uuttotehokkuuden eroja.

Ionivahvuuden kasvaessa fosfaarin desorptio molempiin suolaliuoksiin väheni, minkä vuoksi suora ligandinvaihto sulfaatin ja fosfaatin välillä näyttää olevan epätodennäköistä. Esitetyn teorian mukaan sulfaatin suurempi uuttokyky perustuu vaihtoreaktioihin oksidipintojen H_2O -ryhmien kanssa, jolloin liuoksen ionivahvuus pienenee ja P:n desorptio kasvaa. Samaan suuntaan saattaa vaikuttaa myös negatiivisen varauksen kasvaminen oksidipinnan läheisyydessä. Vastaavat reaktiot eivät ole todennäköisiä kloridiliuoksissa.

Tulokset viittaavat siihen, että maanesteen P:n konsentraatiota voidaan kohottaa ja kasvien P:n saantia edistää käyttämällä moniravinteisten lannoitteiden K:n lähteenä K_2SO_4 :a KCl:n asemesta. Lisäksi ne antavat aiheutta olettaa, että $CaSO_4$:n syntyminen happamien sulfaattimaiden kalkituksessa voi edistää maan P:n desorptiota pienentämällä maanesteen ionivahvuutta.