

Remaining phosphorus and sodium fluoride pH in soils with different clay contents and clay mineralogies

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Abstract – The remaining phosphorus (Prem) has been used for estimating the phosphorus buffer capacity (PBC) of soils of some Brazilian regions. Furthermore, the remaining phosphorus can also be used for estimating P, S and Zn soil critical levels determined with PBC-sensible extractants and for defining P and S levels to be used not only in P and S adsorption studies but also for the establishment of P and S response curves. The objective of this work was to evaluate the effects of soil clay content and clay mineralogy on Prem and its relationship with pH values measured in saturated NaF solution (pH NaF). Ammonium-oxalate-extractable aluminum exerts the major impacts on both Prem and pH NaF, which, in turn, are less dependent on soil clay content. Although Prem and pH NaF have consistent correlation, the former has a soil-PBC discriminatory capacity much greater than pH NaF.

Index terms: ammonium-oxalate-extractable Al, phosphate adsorption, phosphorus buffer capacity.

Fósforo remanescente e pH em fluoreto de sódio em solos com diferentes teores e qualidades de argila

Resumo – O fósforo remanescente (Prem) tem sido utilizado para estimar o fator capacidade de P (FCP) de solos de algumas regiões do Brasil. Entre outras finalidades, o P remanescente pode também ser utilizado para estimar níveis críticos de P, S e Zn no solo, determinados com extratores sensíveis ao FCP, e para a definição das doses de P e S a serem usadas, tanto em estudos de adsorção como no estabelecimento de curvas de resposta a esses elementos. O objetivo deste trabalho foi avaliar os efeitos do teor e da composição mineralógica da fração argila do solo sobre o Prem, e sua relação com o pH medido em solução saturada de NaF (pH NaF). Tanto o Prem quanto o pH NaF são mais influenciados pelo teor de Al extraído com oxalato de amônio e menos dependentes do teor de argila. Embora a correlação entre o Prem e o pH NaF seja consistente, o Prem apresenta maior capacidade de estratificar solos quanto ao fator capacidade de fósforo que o pH NaF.

Termos de indexação: alumínio extraível com oxalato, adsorção de fosfato, fator capacidade de fósforo.

Introduction

The remaining phosphorus (Prem) consists of the P that remains in solution after shaking the soil for determined period with a solution containing a known initial P concentration. The reference method adopted in Brazil for the Prem determination consists of shaking the soil for 1 hour with 0.01 M CaCl₂ containing 60 µg mL⁻¹ P at a 1:10 soil/solution ratio and analyzing the centrifuged or filtered solution for P (Alvarez Venegas et al., 2000).

The significance of Prem for soils presenting low P levels and high P sorption is ascribed not only to its simple determination but mainly to its strong correlation with the less easily measurable phosphorus buffer capacity (PBC) of such soils (Novais & Smyth, 1999).

The soil PBC has inverse relationship with the plant efficiency of P utilization and with the effectiveness of PBC-sensible extractants such as Mehlich-1 for extracting elements such as P and Zn (Muniz et al., 1985; Couto et al., 1992). This gives raise, for instance, to different P, S and Zn critical levels in both plant and soil for a same culture when they are determined in different soils using PBC-sensible extractants (Muniz et al., 1985; Alvarez Venegas & Fonseca, 1990; Alvarez Venegas et al., 2000). Therefore, it is possible to establish regression equations where the soil P and Zn critical levels determined with Mehlich-1 and the soil S critical level evaluated with Ca(H₂PO₄)₂ 500 µg mL⁻¹ P in 2 M HOAC figure as Prem dependent variables in order to use such models to a better interpretation of the results

of soil chemical analyses for fertility purposes (Alvarez Venegas et al., 2000). Furthermore, as shown by Novais & Smyth (1999), Prem can be used as an input variable of computational systems comprising mechanistic models that account for the several factors that define the response of a culture to fertilization in order to better recommend fertilizer rates.

Besides the above-mentioned aspects, the Prem is very useful for defining the P and S levels to be added to soils for the establishment of P and S sorption isotherms and for the determination of soil P and S critical levels in greenhouse experiments (Alvarez Venegas & Fonseca, 1990; Alvarez Venegas et al., 2000).

The Prem predictive power seems dependent on the sorption mechanisms. Ferreira et al. (2001) verified that Prem was not a good indicator of the boron buffer capacity of several Brazilian soils and ascribed this to the possible existence in those soils of specific sites for B adsorption or to the extraction of B from organic matter. Therefore, it is possible that the Prem can also be useful in studies dealing with silicon behavior in tropical soils since both Si and P sorption mechanisms are very similar (Hingston et al., 1972).

Although Prem presents adequate correlation with soil PBC, the soil clay content can also be used for soil PBC estimates. However, this later approach can be considered very limited since the soil PBC is dependent on clay mineralogy. Therefore, the use of another soil property presenting similar PBC-dependence on clay mineralogy and cheaper and simpler determination than Prem could be considered for PBC estimates. An option for this purpose could be the soil pH measured in

1 M NaF (pH NaF), which has good correlation with the phosphate adsorption by sesquioxidic soils (Singh & Gilkes, 1991).

The objective of this work was to evaluate the effects of soil clay content and clay mineralogy on Prem and its relationship with pH values measured in saturated NaF solution.

Material and Methods

Subsurface soil samples of representative soils of the São Paulo State, Brazil, were collected, air-dried and passed through a 2-mm sieve (Table 1).

The texture (pipette method), organic carbon (Walkley-Black method) and exchangeable Al (1 M KCl extraction) were determined according to Embrapa (1997). The available P, K, Ca and Mg were evaluated by extraction with ion-exchange resin (Raij et al., 1986). Soil pH values were measured in water (pH H₂O), 0.01 M CaCl₂ (pH CaCl₂) and 1 M KCl (pH KCl) at a 1:2.5 soil/water or solution ratio (Embrapa, 1997). Additionally, the soil pH was also measured after shaking 0.5 g of soil for 1 hour with 20 mL of 1 M NaF (pH NaF) (Bolland et al., 1996).

The Prem was determined according to Alvarez Venegas et al. (2000) by shaking triplicate 2.5 g of soil with 25 mL of 0.01 M CaCl₂ containing 60 µg mL⁻¹ P, filtering the suspensions through Whatman 42 filter and analyzing the solutions for P using the Murphy & Riley (1962) method.

The Fe, Al and Si soil contents associated to secondary minerals were determined after boiling in H₂SO₄ 1:1 at a 1:20 soil/solution ratio (Embrapa, 1997).

Table 1. Classification, parent materials, localization and sampling depth of the soils.

Soil	Brazilian classification ⁽¹⁾	US classification ⁽²⁾	Parent material	Localization	Depth (cm)
1	Latossolo Vermelho acriférrico	Rhodic Acrudox	Basalt	Ribeirão Preto	100-140
2	Latossolo Vermelho eutroférrico	Rhodic Eutrudox	Basalt	Iracemópolis	100-110
3	Latossolo Vermelho distroférrico	Rhodic Hapludox	Basalt	Luís Antonio	80-100
4	Latossolo Vermelho acriférrico	Rhodic Acrudox	Basalt	Luís Antonio	150-170
5	Latossolo Amarelo ácrico	Xanthic Acrustox	Basalt	Guaíra	100-130
6	Latossolo Vermelho distrófico	Typic Hapludox	Schist	Piracicaba	100-110
7	Latossolo Vermelho -Amarelo distrófico	Typic Hapludox	Sandstone	Piracicaba	100-110
8	Latossolo Vermelho -Amarelo distrófico	Typic Hapludox	Sandstone	São Carlos	80-100
9	Nitossolo Vermelho eutroférrico	Typic Hapludalf	Diabase	Piracicaba	30-40
10	Argissolo Vermelho -Amarelo distrófico	Typic Hapludult	Sandstone	Pindorama	100-120
11	Argissolo Vermelho -Amarelo distrófico	Typic Hapludult	Sandstone	Vera Cruz	100-120
12	Argissolo Vermelho eutrófico	Typic Hapludult	Schist	Rio Claro	70-80
13	Argissolo Vermelho distrófico	Typic Hapludult	Basalt	Piracicaba	100-110
14	Neossolo Quartzarênico órtico	Typic Quatzipsamment	Sandstone	São Pedro	80-100

⁽¹⁾Embrapa (1999). ⁽²⁾United States (1998).

Iron and Al extractable in dithionite (Fe_d and Al_d) and in acid ammonium oxalate (Fe_o and Al_o) were determined in the clay fraction according to Embrapa (1997) and corrected for the whole soil considering the soil clay content. Kaolinite and gibbsite were determined in the deferrified clay fraction by differential thermal analysis (DTA), corrected for the clay fraction and afterwards corrected for the whole soil. The hematite (Hm) and goethite (Gt) clay contents were estimated combining the ratio $Hm/(Hm+Gt)$ and the Al substitutions in these oxides, both determined by X-ray diffraction (XRD) analysis, with the difference $Fe_d - Fe_o$ in the clay as outlined by Netto (1996) with a few modifications. The Hm and Gt clay contents were also corrected for the whole soil as above. Both the selective dissolutions and DTA analyses were performed with three replications.

The experimental results were submitted to regression and correlation analyses using the Statistical Analysis System SAS software, version 6.11.

Results and Discussion

Nine soil samples are clayey (1, 2, 3, 8, 9, 12 and 13) or very clayey (4 and 6), four have medium texture (5, 7, 10 and 11) and one is very sandy (14) (Embrapa, 1999) (Table 2). Most soils are acid or very acid ($pH_{CaCl_2} \leq 5.0$) and the P contents are low or very low in all of them ($P_{resin} < 6 \text{ mg dm}^{-3}$) (Rajj et al., 1996). The positive ΔpH values of soils 1, 4 and 5 agree with their acric characteristics (Embrapa, 1999). The pH_{NaF} values ranged from 8.8 to 10.7 suggesting the displacement of

OH^- by F^- ions from mineral surfaces (Bower & Hatcher, 1967). The remaining phosphorus values showed great amplitude, ranging from $0.1 \mu\text{g mL}^{-1}$ for the Xanthic Acrudox (5) to $43 \mu\text{g mL}^{-1}$ for the Typic Quartzipsamment (14) evidencing expressive PBC variation among the soils.

The Ki index variation (0.49–1.81) reveals that the soils present different weathering status whereas the Kr values separate them into kaolinitic ($Kr > 0.75$) (6, 7, 9, 10, 11, 12, 13 and 14) and oxidic ($Kr < 0.75$) (soils 1, 2, 3, 4, 5 and 8) (Embrapa, 1999), which agrees with the clay mineralogy data (Table 3).

The effects of clay content and clay composition on both Prem and pH_{NaF} were assessed through linear regression analysis (Table 4). The slopes of the significant regression models ($p < 0.05$) indicate that both Prem and pH_{NaF} are influenced very similarly by the soil properties, being the Al_o the variable with greatest impact on them whereas the clay content has the minor influence.

The non-significant influence of kaolinite on Prem is probably the combined effect of its low P adsorption capacity (Muljadi et al., 1966) associated to the presence of Fe and Al oxides, which, in turn, are stronger P adsorbers.

Similarly to the results obtained by Fontes & Weed (1996) for P adsorption by clays of Brazilian Oxisols, the individual contents of hematite and goethite did not have significant effects on the remaining phosphorus. This lack of significance seems to indicate that not only the contents of these oxides but also variations in their intrinsic properties, such as crystallinity degree, Al

Table 2. Electrochemical, chemical and physical properties of the soils.

Soil	pH		$\Delta pH^{(1)}$	Prem ⁽²⁾ ($\mu\text{g mL}^{-1}$)	OC ⁽³⁾ (g kg^{-1})	P ($\mu\text{g dm}^{-3}$)	K	Ca	Mg	Al	Sand	Silt	Clay
	CaCl ₂	NaF											
1	5.0	10.4	0.11	1.0	6.0	5	0.5	4	1	0.0	133	338	529
2	5.5	10.3	-0.77	3.1	7.8	5	0.2	43	4	0.0	214	305	481
3	4.3	10.3	-0.08	4.1	4.5	3	0.3	3	1	0.7	292	165	543
4	5.4	10.7	0.36	0.3	4.9	3	1.1	4	1	0.0	44	309	647
5	5.6	10.4	0.87	0.1	2.0	3	2.1	4	1	0.0	519	157	324
6	4.9	10.3	-0.83	3.8	7.4	2	0.5	15	8	0.1	103	123	774
7	4.8	9.3	-0.94	36.1	2.1	2	1.5	8	2	0.0	667	89	244
8	4.8	10.3	-0.42	7.7	2.4	3	0.1	7	3	0.0	522	57	421
9	5.0	9.8	-0.63	5.3	1.8	4	0.3	40	3	0.0	175	228	597
10	4.0	9.5	-1.12	21.8	1.5	2	1.2	10	13	8.2	574	79	347
11	3.7	8.8	-0.76	36.1	2.1	2	0.9	4	2	6.6	747	48	205
12	5.2	9.7	-0.81	2.1	3.0	3	0.5	63	17	0.0	217	308	475
13	3.8	10.0	-1.12	1.5	2.4	2	0.9	7	4	25.4	210	158	632
14	4.0	9.0	-0.47	43.0	0.6	4	0.3	6	1	0.9	914	75	11

⁽¹⁾ $\Delta pH = pH_{KCl} - pH_{H_2O}$ (Mekaru & Uehara, 1972). ⁽²⁾Remaining phosphorus (Alvarez Venegas et al., 2000). ⁽³⁾Organic carbon.

substitution, specific surface area and exposed faces for adsorption, may exert influence on the remaining phosphorus. The significant effect of the sum of hematite and goethite contents on Prem seems to reiterate the above-mentioned supposition. The non-significant effect of the gibbsite content on Prem may also be due to variations in its intrinsic properties as observed by Jones

(1981), who demonstrated the importance of crystallite size of hematite, goethite and gibbsite for the P adsorption by eleven Puerto Rican soils.

Although the higher P adsorptive capacity of Fe-amorphous is not always evidenced in regression analyses (Adams et al., 1987; Fontes & Weed, 1996; Agbenin, 2003), the high slope value found for Fe_o

Table 3. Iron, Al and Si contents from sulfuric acid digestion, weathering indices Ki and Kr, dithionite and oxalate extractable Fe and Al, kaolinite (Ka), gibbsite (Gib), hematite (Hm) and goethite (Gt) soil contents.

Soil	--(H ₂ SO ₄ digestion, g kg ⁻¹)--					------(g kg ⁻¹)-----							
	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	Ki ⁽¹⁾	Kr ⁽²⁾	Fe _d	Fe _o	Al _d	Al _o	Ka	Gib	Hm	Gt
1	310	284	120	0.72	0.42	130.2	8.1	10.3	6.8	123.8	242.7	118.2	17.5
2	235	252	169	1.14	0.71	110.8	6.0	9.0	5.1	194.8	117.7	98.5	17.9
3	209	223	127	0.97	0.61	116.1	6.1	7.4	5.4	221.3	183.0	114.4	4.8
4	324	314	91	0.49	0.30	164.1	9.1	15.0	10.3	97.0	351.3	144.6	25.9
5	122	182	91	0.85	0.60	49.0	1.4	13.9	3.7	96.2	115.6	0.0	67.1
6	101	291	246	1.44	1.18	79.6	4.2	22.8	6.2	404.1	92.3	17.8	85.4
7	39	87	85	1.65	1.29	22.4	1.3	4.6	1.4	154.1	2.5	5.0	20.6
8	69	200	81	0.69	0.56	43.8	1.1	8.5	4.5	107.8	232.6	0.8	67.3
9	202	229	210	1.56	1.00	118.2	10.1	11.3	4.1	318.9	13.9	76.2	50.0
10	33	117	120	1.75	1.48	23.9	1.8	6.5	2.4	208.4	0.8	0.9	29.6
11	18	75	79	1.81	1.56	11.9	0.9	3.5	1.1	158.5	0.0	2.9	11.4
12	162	229	220	1.63	1.12	61.0	6.6	11.4	4.1	251.8	0.0	11.1	59.2
13	86	234	228	1.66	1.34	64.1	5.9	17.5	5.7	324.9	5.4	16.1	56.1
14	8	25	24	1.66	1.39	0.8	0.1	0.3	0.1	7.5	0.2	0.1	0.7

⁽¹⁾Ki = 1.7 (SiO₂/Al₂O₃) (Embrapa, 1997). ⁽²⁾Kr = 1.7 (SiO₂)/(Al₂O₃ + 0.64Fe₂O₃) (Embrapa, 1997).

Table 4. Results of linear regression analyses relating remaining phosphorus (Prem) and sodium fluoride pH (pH NaF) to soil contents of clay and clay components (g kg⁻¹ whole soil) (n = 14).

Regression equation	R ²	F value	p>F
Prem = 39.9 - 0.0630clay	0.689	26.553	0.0002*
Prem = 23.0 - 0.0584kaolinite	0.165	2.368	0.1498 ^{ns}
Prem = 18.7 - 0.0706gibbsite	0.220	4.662	0.0518 ^{ns}
Prem = 18.1 - 0.1430hematite	0.250	4.004	0.0685 ^{ns}
Prem = 22.9 - 0.3018goethite	0.276	4.577	0.0537 ^{ns}
Prem = 31.7 - 0.2480(hematite + goethite)	0.661	23.401	0.0004*
Prem = 28.3 - 0.2317Fe _d ⁽¹⁾	0.562	15.416	0.0020*
Prem = 26.0 - 3.1485Fe _o ⁽²⁾	0.473	10.760	0.0066*
Prem = 28.3 - 0.2461(Fe _d - Fe _o)	0.561	15.311	0.0021*
Prem = 32.6 - 4.7760Al _o ⁽³⁾	0.663	23.578	0.0004*
pH NaF = 9.0 + 0.0021clay	0.567	15.683	0.0019*
pH NaF = 9.7 + 0.0008kaolinite	0.025	0.301	0.5933 ^{ns}
pH NaF = 9.5 + 0.0039gibbsite	0.629	20.068	0.0008*
pH NaF = 9.6 + 0.0064hematite	0.366	6.931	0.0219*
pH NaF = 9.6 + 0.0082goethite	0.149	2.099	0.1730 ^{ns}
pH NaF = 9.1 + 0.0096(hematite + goethite)	0.724	31.467	0.0001*
pH NaF = 9.3 + 0.0090Fe _d	0.616	19.260	0.0009*
pH NaF = 9.5 + 0.0941Fe _o	0.307	5.305	0.0400*
pH NaF = 9.3 + 0.0097(Fe _d - Fe _o)	0.633	20.699	0.0007*
pH NaF = 9.1 + 0.1924Al _o	0.781	42.744	0.0001*

⁽¹⁾Fe_d: dithionite-extractable iron. ⁽²⁾Fe_o: ammonium-oxalate-extractable iron. ⁽³⁾Al_o: ammonium-oxalate-extractable aluminum. ^{ns}Not-significant. *Significant at p<0.05 (t test).

suggests its expressive influence on Prem (Table 4). However, the correlation verified between Fe_o and Al_o ($r = 0.72$; $p < 0.01$) makes difficult to assess the real impacts of both Fe_o and Al_o on the Prem magnitude. In spite of this, not only the greatest slope but mainly the greater F and R^2 values found when Prem was regressed against Al_o strongly suggest the greatest Al_o influence on Prem.

Sakurai et al. (1989) verified that the Al_o exerted the greatest impact on the zero point of charge (ZPC) values of Japanese and Thai soils, whereas Parks (1965) reported values between 7.5 and 8.0 and Perrott (1977) found a value of 9.3 for the ZPC of synthetic amorphous aluminum oxides. Considering that the phosphate adsorption occurs through exchange with single coordinated surface hydroxyls (Parfitt, 1978) and that these hydroxyls are probably the easiest ionizable ones, the minerals presenting higher ZPC values would have a great number of these surface hydroxyls and therefore a greater impact on P adsorption (Fontes et al., 2001). Therefore, both the presence of a great number of single coordinated surface hydroxyls and the greater specific surface areas of the forms that contribute for Al_o probably explain their greatest lowering effect on Prem, as verified in the present study, and their greatest impact on P adsorption, as verified by Adams et al. (1987) for soils of Wales, Gilkes & Hughes (1994) for soils of Australia, and Fontes & Weed (1996) for clay fractions of Brazilian Oxisols.

The greatest slope, R^2 and F values verified when pH NaF was regressed against Al_o indicate the greatest impact of oxalate-extractable Al forms on pH NaF whereas the clay content has the minor effect (Table 4). The lack of significance for the kaolinite and goethite effects and the smaller slopes of gibbsite and hematite agree with the low capacities of hydroxyl release of these minerals to the NaF solution as demonstrated by Perrott et al. (1976). In the same way, the major effect of Al_o is also in agreement with the greater reactivity of the Al-amorphous oxides towards NaF solution (Perrott et al., 1976). Finally, it is probable that the low significance found for the regression between pH NaF and Fe_o may be due to the above-mentioned correlation between Fe_o and Al_o . Adams et al. (1987) verified no effect of Fe_o on the soil hydroxyl release to the NaF solution. Furthermore, most studies show that Fe_o seems less or even uncorrelated to pH NaF (Singh & Gilkes, 1991; Gilkes & Hughes, 1994; Bolland et al., 1996).

The similar effects of soil properties on both Prem and pH NaF demonstrate that the correlation between these two variables ($r = -0.89$; $p < 0.01$) is very consistent. Furthermore, these observations show that the pH NaF is a good indicator of the amount of surface hydroxyls that can be exchanged by phosphate ions during the P adsorption and that it can substitute, with great advantage, the clay content as PBC estimator in weathered acid soils presenting low P contents. This point is reiterated by the fact that although the use of the soil clay content for PBC estimates is recommended only for soils presenting similar clay mineralogies (Novais & Smyth, 1999), the P adsorption can vary greatly with small variations of Al_o , which, in turn, is extracted from clay components whose minor contents are not accounted for discriminating soils as their clay mineralogies.

Although the pH NaF presents cheaper and less time consuming determination than the soil clay content and is dependent mainly on the contents of the most effective P-adsorbers (Al_o compounds), its low variability among the studied soils, characterized by a coefficient of variation (CV) of 5.8%, indicates that its capacity of stratifying soils as PBC values is much lower than that of Prem, whose greater sensibility to Al_o resulted in a CV of 130% within the soil set evaluated in this study. Therefore, although pH NaF allows better soil PBC estimates than those given by the soil clay content, probably it would be more useful for the development of multiple regression models (i.e., pedotransfer functions) for Prem (or even PBC) estimates from easily determinable or routinely determined soil properties. The inclusion of pH NaF in such models could determine the need of less soil properties for reliable Prem or PBC estimates. This aspect should be evaluated in future research.

Conclusions

1. The remaining phosphorus (Prem) and pH NaF values are dependent mainly on Al_o soil contents and less dependent on soil clay content.
2. The Prem and pH NaF present consistent correlation.
3. The Prem has a soil-PBC discriminatory capacity greater than pH NaF.

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