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Phosphorus Fractionation in Soil Cultivated with Sugarcane Fertilized by Filter Cake and Phosphate Sources

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Studies of phosphorus (P) forms in high-weathering soils, after long periods of phosphate fertilizer application associated with organic residues, are important to optimize P fertilization. This study aims to evaluate the effect of filter cake application and other distinct phosphate sources on organic and inorganic P fractions in Red Eutrophic Argisoil cultivated with sugarcane. The experiment was established between 2012 and 2014, in Sao Paulo State, Brazil. The treatments were as follows: control (without P), triple superphosphate, natural phosphate from Araxá, and naturally reactive phosphate Bayóvar on the presence and absence of filter cake at 7.5 t ha^{-1} (dry mass). The P rate was 90 kg ha⁻¹ of phosphorus pentoxide (P_2O_5) soluble in citric acid. The experiment was arranged in a randomized block, in a 4×2 factorial with three repetitions. After 22 months of treatments, soil samples were collected on sugarcane planting lines and P fractionation was analyzed. The greatest percentage of P is found as nonlabile P fractions, regardless of filter cake application or not. The use of low solubility sources, such as natural phosphate from Araxá, in association with filter cake has promoted greater solubility of P bound to calcium (Ca), which can enhance the crop uses. Filter cake increases most of labile and moderately labile inorganic P forms in the soil (resin; sodium bicarbonate, NaHCO3; and sodium hydroxide, NaOH, 0.1 mol L^{-1}), which characterizes the importance of this source to sugarcane nutrient supply.

Keywords Organic residue, phosphorus forms, phosphorus sources, residual effect

Introduction

Phosphorus (P) deficiency is a worldwide barrier to plants growth (Khan and Joergensen 2009), holding greater significance in tropical soils, because they have low P availability due to adsorption and precipitation processes. Because P is the second nutrient that most

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limits crop yields (Biswas and Narayanasamy 2006), in case of soil deficiency its availability can be increased by organic and inorganic fertilizer application (Ayaga, Todd, and Brookes 2006). However, 85 to 90% of P added to soil is not available to plants in the first year of application, due to its adsorption and precipitation by iron (Fe), aluminium (Al), and calcium (Ca) bindings (Gichangi, Mnkeni, and Brookes 2009), which creates P storage in the soil called residual (Gikonyo et al. 2008).

The application of distinct phosphate fertilizers results in different soil P fraction accumulations, which mainly depend on rate, crop removal, soil mineralogy, climatic conditions (Schmitt, Buol, and Kamprath 1996), and period of contact. In doing so, the study of P sources with different solubility is important (Meena 2010) to better understand this nutrient behavior on the wide conditions and uses of agricultural soils, especially on tropical regions.

Soil P forms are distributed according to the ligant chemical nature and the binding energy between nutrient and soil. Regarding the ligand nature, P can be found as both organic and inorganic forms bound to Fe, Al, Ca, silicate clays, and Fe and Al oxides. Thereby, the P forms found in the soil have different capacities of desorption and nutrient reposition to soil solution, according to its chemical nature and binding energy (Souza Jr. et al. 2012).

Soil P fractionation is an important technique to identify the dynamic of nutrient's transformation in the soil (Silva and Raij 1996), and it has been a good way to understand total P availability in the soil as well as its solubility. Because of this, the technique has become useful to study soil nutrient dynamics under different management systems (Pavinato, Merlin, and Rosolem 2009) and different phosphate fertilization via varied solubility sources.

After long periods of phosphate fertilization, different residual fractions are accumulated in the soil, with distinct degrees of binding energy (Negassa and Leinweber 2009; Linquist, Ruark, and Hill 2011; Tokura et al. 2011). There is evidence of a synergic effect between phosphate fertilizer and organic compost applications, which increases the content of available P in the soil (Mkhabela and Warman 2005; Ohno et al. 2005; Garg and Bahl 2008), and consequently reduces the mineral fertilizer rate.

The presence of organic compost, such as filter cake, in association with mineral fertilizer might increment labile P fractions of the soil; however, this effect is dependent on fertilizer source, as Almeida Jr. et al. (2011) reported that filter cake corrects acidic soils and, in this condition, natural phosphates would have lower P labile fractions due to its lower solubility on high pH.

It is relevant to emphasize that studies regarding this issue are recent in Brazil, which is a reason to worry, as soil P knowledge is necessary to optimize the use of phosphate fertilization on sustainable bases. Therefore, this study aims to evaluate the effect of filter cake application and other distinct phosphate sources on organic and inorganic P fractions, in Red Eutrophic Argisoil cultivated with sugarcane.

Material and Methods

The experiment took place at Catanduva, São Paulo State, Brazil (21° 05′ 07″ S, 48° 54′ 22″ O, altitude of 550 m), in a Red Eutrophic Argisoil (Santos et al. 2013). Before planting, 20 soil subsamples were taken at 0.0–0.20 cm deep, which were used for fertility chemical analyses, according to the methodology described by Raij et al. (2001). The soil tested showed the following chemical characteristics: pH calcium chloride (CaCl₂) = 5.5; organic matter = 12 g dm⁻³; P (resin) = 5 mg dm⁻³;

 $K^+ = 3.1 \text{ mmol}_c \text{ dm}^{-3}$; $Ca^{2+} = 30 \text{ mmol}_c \text{ dm}^{-3}$; $Mg^{2+} = 13 \text{ mmol}_c \text{ dm}^{-3}$; $H^+ + Al^3$ $^+ = 18 \text{ mmol}_c \text{ dm}^{-3}$; cation exchange capacity = 64 mmol}_c \text{ dm}^{-3}; and base saturation = 72%. Acidic correction was not needed as base saturation was in an accepted range for the crop, according to the regional recommendations (Raij and Cantarella 1997).

Phosphate fertilizers were applied in May 2012 and sugarcane planting was placed afterward. Eight treatments were evaluated: control (without P), triple superphosphate (44% of total P_2O_5 and 41% soluble in 2% citric acid), natural phosphate from Araxá (22% of total P_2O_5 and 4% soluble in 2% citric acid), and naturally reactive phosphate Bayóvar (28% of total P_2O_5 and 14% soluble in 2% citric acid), in the presence and absence of filter cake at 7.5 t ha⁻¹ (dry mass). The experiment was arranged in a randomized block in a 4 × 2 factorial with three repetitions. Each experimental unit was composed of 5 rows, 15 m long and separated by 1.5 m. The evaluations were taken on the three central rows, discarding 1 m of each tip.

Every P application followed the rate of 90 kg ha⁻¹ of P_2O_5 soluble in citric acid. To verify the possibility of reduction on mineral fertilizer rate when filter cake is present, it used 50% of the recommended rate stated by Raij and Cantarella (1997), as greater efficiency of plant P uptake is obtained when organic composts are associated with fertilizers.

Filter cake was submitted to composting and, before its application, chemical analysis following Bataglia et al.'s (1983) methodology was done. The values obtained are expressed below as a dry mass at 60–65 °C: N = 14.0 g kg⁻¹; P = 9.2 g kg⁻¹; K = 3.4 g kg⁻¹; Ca = 25.3 g kg⁻¹; Mg = 9.0 g kg⁻¹; S = 3.3 g kg⁻¹; boron (B) 16 mg kg⁻¹; copper (Cu) = 43 mg kg⁻¹; Fe = 9.374 mg kg⁻¹; manganese (Mn) = 753 mg kg⁻¹; and zinc (Zn) = 70 mg kg⁻¹.

The fertilizer applications were placed at the bottom of planting furrow (about 0.25 to 0.30 m deep). Subsequently, CTC 15 sugarcane seedlings were distributed and covered by a 0.10- to 0.15-m layer of soil. Nitrogen fertilization was also applied 40 days after planting, at the rate of 50 kg ha⁻¹ (ammonium nitrate). In the second year of cultivation (2013), fertilization was done according to sugarcane recommendations (Raij and Cantarella 1997), which provides 120 kg ha⁻¹ of N and 120 kg ha⁻¹ of potassium oxide (K₂O), via ammonium nitrate and potassium chloride, respectively.

After 22 months of phosphate treatments applications, soil samples were collected on sugarcane planting lines (expected place to have greater fertilizers concentration) at 0.0–0.30 m deep. Soil samples were formed by mixing 20 subsamples, which were taken on central plant lines of each experimental unit. According to Hedley, Stewart, and Chauhan's (1982) methodology and to the modifications by Condron, Goh, and Newman (1985), soil samples were submitted to fractionation of P forms.

The sequential extraction was as follows: anions exchange resin [0.5 g of soil + 10 mL of water (H₂O) + resin]; 10 mL of sodium bicarbonate [NaHCO₃ (0.5 mol L⁻¹)]; 10 mL of sodium hydroxide [NaOH (0.1 mol L⁻¹)]; 10 mL of hydrochloric acid [HCl (1 mol L⁻¹)]; 10 mL of NaOH (0.5 mol L⁻¹); and residual digestion with concentrated H₂SO₄ ⁺ H₂O₂ (sulfuric acid + hydrogen peroxide). The inorganic P on resin, HCl, and residual digestion extracts were determined by Murphy and Riley (1962) method, whereas P inorganic of alkaline extracts (NaHCO₃ and NaOH, 0.1 and 0.5 mol L⁻¹) were determined by Dick and Tabatabai's (1977) method. The organic fractions on alkaline extracts were obtained by its digestion with ammonium persulfate and diluted H₂SO₄, by autoclaving at 120 °C during 2 h. Then, P contents were determined following Murphy and Riley's (1962) methodology and subtracted by the inorganic P

result found on those fractions, which by calculation provides the organic P content. At the end, the P contents of each fraction were added to residual P in order to obtain the total P in the soil.

The data were submitted to analysis of variance (ANOVA) and means were compared by Scott-Knott (P < 0.05) test, using the statistics program Sisvar (Ferreira 2011).

Results and Discussion

The application of P sources, with and without filter cake, affected all P fractions in the soil (Tables 1 and 2), except residual P (Table 2), which was not influenced by treatments.

The P labile fraction extracted by resin was not affected by P sources (Table 1), and neither differed from control treatment. However, it showed isolated effect of filter cake, which the presence indicated greater contents of that P fraction compared to its absence. Brazilian laboratories conventionally use P determined by resin extractor to diagnose crops' nutrient availability; nevertheless, it corresponds to a small fraction of total P in the soil, as reported by Busato, Canellas, and Velloso (2005) in a study of Cambisoil cultivated with sugarcane. In the present study, triple superphosphate showed only 5% of resin P in the total P; Bayóvar phosphate indicated 3% and phosphate from Araxá 2%. High rates of phosphate fertilizers (greater than crop requirement) increase the content of this P fraction; however, the application of low rates (crop requirement) does not affect this fraction's content (Zamuner, Picone, and Diez 2012). It is important to mention that the cultivated plant species modify P depletion's rate, as observed in Schoninger, Gatiboni,

P sources	P resin	NaHCO ₃		NaOH $(0.1 \text{ mol } \text{L}^{-1})$	
		Pi	Ро	Pi	Ро
Whitout P	8.5	8.1 b	9.5 b	16.0 b	1.6 b
NP	10.7	11.0 b	14.4 a	17.8 b	7.6 a
RNP	10.3	12.9 b	12.7 a	20.5 a	5.1 a
TSP	13.3	17.1 a	11.4 a	24.4 a	4.1 b
Filter cake (FC)					
Presence	15.2 a	15.8 a	11.8	23.7 a	3.9
Absence	6.3 b	8.7 b	12.2	15.6 b	5.3
F-test					
Р	1.11ns	5.39*	3.98**	6.58**	6.45**
FC	22.26**	19.17**	0.15ns	32.88**	2.18ns
$P \times FC$	1.46ns	1.03ns	0.75ns	0.80ns	0.19ns
CV (%)	43.12	32.40	21.35	17.73	52.61

Table 1

Soil phosphorus content (mg kg⁻¹) extracted by anion exchange resin (P resin), sodium bicarbonate (NaHCO₃), and sodium hydroxide (NaOH, 0.1 mol L⁻¹) extractors on soil samples cultivated with sugarcane under phosphorus sources and filter cake fertilizations

Notes. Means following different letters in columns differ from each other by Scott-Knott (P < 0.05). **, *, and ns: significant at 1% and 5% of probability and no significance. CV, coefficient of variance;

NP, natural phosphate from Araxá; RNP, reactive natural phosphate Bayóvar; TSP, triple superphosphate; Pi, inorganic phosphorus; Po, organic phosphorus.

Table	2
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		NaOH (0.5 mol L^{-1})			
P sources	P (HCl)	Pi	Ро	Residual P	Total P
Whitout P	4.3 c	41.8 b	2.7 b	126.8	219.3 c
NP	283.4 a	56.7 a	37.4 a	131.7	570.8 a
RNP	43.7 b	53.1 a	4.3 b	146.3	309.0 b
TSP	5.3 c	38.6 b	7.1 b	137.9	259.2 c
Filter cake (FC)					
Presence	76.6 a	50.0	12.6	148.7	358.3
Absence	91.8 b	45.1	13.1	122.6	320.8
F-test					
Р	633.5**	4.18*	45.04**	0.22ns	54.99**
FC	8.14*	1.28ns	0.05ns	2.11ns	3.07ns
$P \times FC$	12.1**	0.64ns	0.30ns	0.17ns	0.64ns
CV (%)	15.5	21.95	46.60	32.45	15.41

Soil phosphorus content (mg kg⁻¹) extracted by hydrochloric acid (HCl 1 mol L⁻¹) and sodium hydroxide (NaOH, 0.5 mol L⁻¹); residual phosphorus and total phosphorus of soil samples cultivated with sugarcane under phosphorus sources and filter cake fertilizations

Notes. Means following different letters in columns differ from each other by Scott-Knott (P < 0.05). **, *, and ns: significant at 1% and 5% of probability and no significance. CV, coefficient of variance; NP, natural phosphate from Araxá; RNP, reactive natural phosphate Bayóvar; TSP, triple superphosphate; Pi, inorganic phosphorus; Po, organic phosphorus.

and Ernani's (2012) study, which shows that grasses have greater P uptake capacity. Those results can explain the absence of effect on P labile fraction by the tested sources, as the soluble P rate applied was not high and the crop has high uptake capacity.

There was an isolated effect of P sources on inorganic and organic P fractions determined by sodium bicarbonate (NaHCO₃) extractor. The biggest inorganic P content was observed when triple superphosphate was applied, which was greater than all the other P sources. That P form is also classified as labile P, demonstrating that triple superphosphate increases labile P fraction compared to the other sources. The filter cake also indicated effect on inorganic P fraction, as the fraction presented greater values when the compost is present. For the organic fraction extracted by NaHCO₃, the control treatment presented lower P content than the other P sources, which was already expected. The absence of filter cake effect on organic fractions was not an expected result but might have occurred due to P mineralization on filter cake during the crop cycle.

The inorganic P fraction extracted by resin and the inorganic and organic fractions by NaHCO₃ are considered as labile forms on Hedley fractionation (Table 1), which are the more available forms for plant uptake (Cross and Schlesinger 1995; Gatiboni et al. 2007). The present study did not detect effects of P sources on P labile fractions, in agreement with Santos et al. (2008), who studied different P sources and type of applications during 3 years of corn cultivation. When the total P (P organic + P inorganic) extracted by NaHCO₃ was evaluated, it was observed that only one P fraction represented the lower amount of the total P in the soil, as triple superphosphate showed 11% of this fraction, phosphate Bayóvar 8%, and phosphate from Araxá 4%, resembling the control treatment, which showed 8%.

It is important to point that besides the reactive differences of P sources among the treatments, plant uptake and adsorption processes during the 22 months of applications might have levelled the availability of labile forms in all sources tested, as also stated by Santos et al. (2008) in corn cultivation. Because labile forms represent a small fraction of total P in the soil, isolated analysis of available P, normally used in laboratory routines, might not be a great parameter to accurate interpretations of P content in the soil and its relationship to crop responses.

The determination of P fractions by the extractor sodium hydroxide (NaOH, $0.1 \text{ mol } \text{L}^{-1}$) were affected by P sources, as greater contents of inorganic P were detected on phosphate Bayóvar and triple superphosphate compared to phosphate from Araxá and absence of P (control). Filter cake increased inorganic P fractions in relation to its absence. The organic fraction showed greater contents in the presence of phosphates from Araxá and Bayóvar than in the presence of triple superphosphate and absence of P, while filter cake did not demonstrate any effect. Conversely, absolutes values of organic P were pretty low in comparison to previous studies, which represented small amount on total P. Gatiboni et al. (2007), studying soils grown with different plant species in the south of Brazil, found greater values of that P fraction, which ranged from 46.6 to 115.8 mg kg⁻¹ after applications of different rates of P. In addition, Pavinato, Merlin, and Rosolem (2009) found values ranging from 20 to 105 mg kg⁻¹.

The inorganic and organic forms of P extracted by NaOH 0.1 mol L^{-1} are considered moderately labile and, in general, represent inorganic P bound to oxides and to silicate clays with intermediate binding energy, and organic P is considered as averagely labile form (Cross and Schlesinger 1995; Gatiboni et al. 2008). Because the fractions are classified as averagely labile, it will contribute to P availability to future plant uptake (Pavinato, Merlin, and Rosolem 2009). This research indicated 11% of total P, extracted by NaOH 0.1 mol L^{-1} , when triple superphosphate was applied, 8% under phosphate Bayóvar and control treatments, and 4% under phosphate from Araxá fertilization.

Different behavior was observed when hydrochloric acid (HCl) was used as the extractor, which showed significant interaction among the factors (Table 2). In either presence or absence of the filter cake, phosphate from Araxá demonstrated greater content of P, followed by phosphate Bayóvar, which were greater than triple superphosphate and control treatments (Table 3). This result was expected, as nonreactive natural phosphates have great amounts of stable calcium phosphate (apatite), which is solubilized on the extraction due to its medium acidification. It is important to state that the rate used was based on soluble P; therefore, it was applied high amounts of nonsoluble P under natural

cultivated with sugarcane under phosphorus sources and inter cake returzations						
Parameter	Without P	Araxá	Bayóvar	TSP	F-test	
With filter cake Without filter cake F-test	5.9 aC 2.7 aC 0.10ns	248.1 bA 318.8 aA 44.01**	47.1 aB 40.3 aB 0.41ns	5.2 aC 5.3 aC 0.00ns	237.1** 408.6	

 Table 3

 Soil phosphorus content (mg kg⁻¹) extracted by hydrochloric acid (HCl) of soil samples cultivated with sugarcane under phosphorus sources and filter cake fertilizations

Notes. Means following different lowercase letters on columns and uppercase letters on lines differ from each other by Scott-Knott (P < 0.05). **, *, and ns: significant at 1% and 5% of probability and no significance.

phosphate treatments, especially on phosphate from Araxá but also with Bayóvar. According to the sources' data within filter cake treatment, only the phosphate from Araxá promoted a significant effect, which presented greater P content in filter cake absence.

Phosphorus extractions using HCl are related to P forms bound to Ca (Hedley, Stewart, and Chauhan 1982) and, according to Pavinato, Merlin, and Rosolem (2009), the results of this fraction observed in their study were low in relation to soil total P, which allows the assumption of low compost levels in the soil, similar to the results presented in this study. On the other hand, Yu et al. (2006) stated that P fraction was responsible for 45–60% of total P, in neutral and alkaline soils from Florida.

In the present work we found 2% of this fraction under triple superphosphate, 14% under phosphate Bayóvar, 50% under phosphate from Araxá, and 2% in the control treatment. This result demonstrates that the P found on triple superphosphate was almost not found in this fraction, as the result was similar to the control. The high percent of this fraction on phosphate from Araxá is explained by the predominance of apatite on the source, as previous mentioned. It is relevant to cite the effect of filter cake on phosphate from Araxá, which solubilizes part of the nonsoluble P, as it had less P bound to Ca in comparison to the absence of filter cake treatment.

Among the P fractions determined by NaOH 0.5 mol L^{-1} extractor (Table 2), inorganic P was greater in phosphate from Araxá and Bayóvar phosphate applications, overcoming triple superphosphate and control treatment. The smallest value of inorganic P on this fraction under triple superphosphate might have occurred because that source keeps its P as a soluble forms of soil fraction, which were determined on previous extractions. Upon the organic P with this extractor, greater value was observed on phosphate from Araxá application, which was greater than all others. Possibly, the phosphates present on phosphate from Araxá as low solubility forms were solubilized after the acidification promoted by HCl extraction, as well as after the digestion with persulfate and diluted H₂SO₄, which indicates a possible error in the classification of that fraction as an organic one, as it would be stable calcium phosphates that were broken after acidification and not measured on the previous extraction.

The filter cake did not show any effect on P fractions using NaOH 0.5 mol L^{-1} , similar to previous results that studied organic residue (pig slurry) application (Gatiboni et al. 2008). Regarding the percentage of those fractions on total soil P, organic P + inorganic P extracted by NaOH 0.5 mol L^{-1} presented 18% under triple superphosphate, 19% under phosphate Bayóvar, and 16% under phosphate from Araxá, which are lower values than the 20% observed on control treatment.

After soil P application, there is an increasing trend in inorganic labile and moderately labile fractions (Zhang et al. 2004; Gatiboni et al. 2007), which are responsible to replace P in the soil solution. This fact was also observed in the present study, as the control treatment presented lower content of these fractions, excepted on resin, which did not differ from it.

The studied sources did not showed effects on residual P, as it indicated 53% of this fraction when triple superphosphate was used, 47% with Bayóvar phosphate, and 23% on phosphate from Araxá application, compared to 58% of the control treatment. Therefore, it can be stated that, in general, residual P represents the main percentage of total P in the soil, except on phosphate from Araxá treatment, in which the total P applied was too high. It indicates that the main part of P is found as forms with high binding energy to soil colloids, as observed by Conte (2001) in soil samples from the south of Brazil. The results is also in agreement with Gonçalves and Meurer (2009) and Schmitt et al. (2013), who related that many soils from the south of Brazil, including some that had phosphate fertilization historic, present the P residual fraction as the main fraction of soil total P.

The residual fraction of P fractionation, obtained by acidic soil digestion, is constituted by the P that was not extracted by the selected extractors of Hedley fractionation, and showed as organic and inorganic recalcitrant P forms (Gatiboni et al. 2007; Santos et al. 2008). Gatiboni et al. (2007), in a 6-year phosphate fertilization study, did not observed increase on this fraction of soil, which indicates that the P added is preferably accumulated as greater labile fractions. These authors stated that even on P deficiency systems, plant uptake causes a riffle effect of P labile replacement, first by intermediate labile forms and then by low labile forms.

The total P in the soil, obtained by the sum of fractions of each extractor, was greater on phosphate from Araxá, followed by Bayóvar phosphate, and both values were greater than triple superphosphate and control treatment (Table 2). In addition, the P rate was calculated in a soluble P base, as already mentioned on P extracted by HCl. In this way, lower solubility forms receive greater total P rates of fertilizer. The filter cake did not demonstrate an effect on this fraction.

In general, there were no observed significant interactions between P sources and filter cake; however, the filter cake use (isolated factor) increases most of labile fraction of the soil, which can be explained by the content of P in this residue (9.2 g kg⁻¹ of P). Several pieces of information about the increment on phosphate fertilization's efficiency when it is associated with organic composts are present in the literature (Mkhabela and Warman 2005; Ohno et al. 2005; Garg and Bahl 2008), but synergic effects can also occur, as organic composts might act as a maintenance factor on the increment of labile forms in the soil, via competition (Pavinato, Merlin, and Rosolem 2009).

Conclusions

After 22 months of phosphate sources application, the greatest percentage of P is found as nonlabile P fractions, regardless of filter cake application.

The use of low solubility sources, such as natural phosphate from Araxá, in association with filter cake has promoted greater solubility of P bound to Ca, which can enhance the crop uses.

Filter cake increases most of labile and moderately labile inorganic P forms in the soil (resin, NaHCO₃, and NaOH, 0.1 mol L^{-1}), which characterizes the importance of this source to sugarcane nutrient supply.

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