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Phosphate Sources and Filter Cake Amendment Affecting Sugarcane Yield and Soil Phosphorus Fractions

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ABSTRACT: The use of mineral phosphate fertilizers associated with organic residues can improve fertilizer use efficiency and consequently decrease their usage costs. Soil application of filter cake (FC) can provide nutrients and enhance physical quality. This study aimed to evaluate the effects of different phosphate fertilizers [rock phosphate (RP) and triple superphosphate (TSP)] applied at different rates (90 and 180 kg ha^{-1} P_2O_5) associated with filter cake (10 Mg ha^{-1} dry matter) on the soil phosphorus (P) fractions after two consecutive seasons of sugarcane in two distinct soils, sandy and clayey. Yield was significantly improved with FC addition in the first year in both soils, while inorganic P sources did not influence crop yield at either location and in both years. Organic and residual P forms were only slightly altered in the sandy soil. The most significant changes occurred in the labile and moderately labile P fractions in both soils. Filter cake was an effective source of nutrients for plant development, increasing the levels of soil available P and keeping it at agronomically adequate levels for up to two years, however it was not able to facilitate the P release from mineral fertilizers, irrespective of the source or rate.

Keywords: reactive phosphate, P solubility, soil P fractionation, organic waste.

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INTRODUCTION

Phosphorus is an essential element for food and biofuel production (Jarvie et al., 2015). Since most Brazilian soils are highly P fixing, agricultural expansion will require large inputs of P fertilizers to overcome the rapid immobilization of inorganic P by clay particles and iron (Fe) and aluminum (Al) (hydr)oxides (Tiessen, 2005; Roy et al., 2016). Generally, only 10-30 % of water soluble P sources applied to P-fixing soils is taken up by plants in the year of application, and the remainder is adsorbed by soil particles, becoming less available for plant uptake (Raghothama, 1999; Savini et al., 2016). This rapid adsorption increases in proportion to the contact time of P sources with soil and their solubility (Novais et al., 1980). Thus, high inputs of P are needed to maintain crop yields at economical levels; for example for a major crop such as sugarcane, P inputs range from 50-80 kg ha^{-1} at establishment with a further annual application of 10-15 kg ha^{-1} after the third year of cultivation (van Raij et al., 1997). A key question is whether this large input of P fertilizer is sustainable when global supplies of rock phosphate (RP) are finite and subjected to considerable price volatility (Cordell and Neset, 2014; Elser et al., 2014). This is especially relevant to Brazil because of the large potential for agricultural expansion into the natural savanna. As example, sugarcane is forecast to expand by 38 % over the next three decades (Withers et al., 2018).

Currently, more than 50 % of mineral P fertilizers used in Brazilian agriculture are imported (ANDA, 2016) and the internal production of RP is estimated to supply the current annual demand for less than 50 years (U.S. Geological Survey, 2016). Alternative strategies are therefore needed for Brazilian farming systems to ensure sustainable crop production in the future (Withers et al., 2018). One strategy is to apply P sources with a low solubility such as RP in sugarcane cultivation instead of soluble P sources, which are more expensive and also more susceptible to rapid adsorption and precipitation. Another strategy could be the use of organic by-products like filter cake (FC) or vinasse, produced by the sugarcane mill industries, very important in case of limitations in soil fertility (Negassa et al., 2010). Filter cake is an industrial waste by-product from the process of sugar clarification, composed of a mixture of ground sediment and crushed sludge. Each ton of sugarcane produces 30-40 kg of FC after processing (Korndörfer and Anderson, 1997). As this compound contains high amounts of nitrogen (N), P, and calcium (Ca), it can be used as a substitute for inorganic fertilizer for many crops (Almeida Junior et al., 2011; Santos et al., 2011; Santana et al., 2012; Ossom et al., 2012; Utami et al., 2012). Nitrogen and P in FC are predominantly in organic forms, and must be mineralized for uptake by plants (Torres et al., 2012). Also, FC contains high levels of organic matter and Ca, and hence can reduce exchangeable Al and acidity in tropical acid soils (Almeida Junior et al., 2011). Besides, it can ameliorate soil physical properties by reducing soil density and increasing soil porosity, leading to the formation of aggregates to avoid soil erosion, especially in sandy soils. Application of solely 15-30 Mg ha^{-1} of wet FC (van Raij et al., 1997) or 2.6-2.7 Mg ha^{-1} in combination with 160-190 kg ha^{-1} of P_2O_5 (Santos et al., 2014) at planting furrow is usually recommended for sugarcane establishment in Brazil.

The effects of organic compounds on P sorption when studied in isolation are well known (Guppy et al., 2005), however it does not diminish the need to examine the interaction of P with dissolved organic carbon compounds derived from organic byproducts addition, like FC. Indeed, the whole suite of compounds produced during decomposition of FC may interact and behave differently with respect to P sorption than the individual components. The benefits of FC for increasing P availability to crops in Brazil's tropical soils is unclear and needs to be demonstrated for more sustainable use of secondary P resources and more efficient use of soluble inorganic P sources. In this research, we hypothesized that organic P presents in FC can be considered available since it is mineralized shortly after application, and that FC organic compounds can reduce the adsorption of inorganic P derived from mineral fertilizers by soils thereby increasing labile P fractions in the soil and consequently the sugarcane yield. We therefore evaluated the effects of mineral phosphate sources and rates in association with FC on the sugarcane yield and on soil P forms after two consecutive cropping seasons in two distinct soil types (sandy and clayey Oxisols).

MATERIALS AND METHODS

Sites descriptions

Two field experiments were conducted in areas under commercial sugarcane cultivation in São Paulo State, Brazil. The locations were selected for their low available (resin) P content and contrasting soil textures, one sandy and one clayey soil. The sandy soil is in Agudos-SP at latitude 22° 33′ 22″ S and longitude 49° 06′ 15″ W at 715 m a.s.l. (Figure 1), classified as *Latossolo Vermelho Amarelo distrófico* (Santos et al., 2013) or Hapludox (Soil Survey Staff, 2014). This site had an annual rainfall of 1,341, 926, and 812 mm in 2013, 2014, and 2015, respectively. The mean annual temperature is approximately 21 °C with the maximum monthly mean temperature of 28 °C in February and the minimum of 11 °C in July. This area was previously degraded pasture for more than 20 years following deforestation. Before sugarcane establishment, the indigenous herbage was desiccated with an application of glyphosate at the rate of 4 L ha^{-1} and the residual straw was incorporated into the soil by deep ploughing (0.00-0.50 m). Then the soil was limed with 3 Mg ha^{-1} of lime ($CaCO_3$ + $MgCO_3$) based on the rates applied to the adjacent area by the farmer and treated with 1.5 Mg ha^{-1} of gypsum ($CaSO_4$). Lime and gypsum were incorporated twice by disc harrow to approximately 0.00-0.20 m depth. The trial was established in August 2013.

The second site is a clayey soil located in Macatuba-SP with the following coordinates: latitude 22° 29′ 39″ S, longitude 48° 41′ 14″ W at 515 m a.s.l. (Figure 1). This soil is classified as *Latossolo Vermelho Eutrófico chernossólico* (Santos et al., 2013) or Hapludox (Soil Survey Staff, 2014). The total rainfall was 1,161, 1,007, and 1,253 mm in 2014, 2015 and 2016, respectively. The mean annual temperature was 20.8 °C with the maximum monthly mean temperature of 23.7 °C in January and the minimum of 6.5 °C in July. This area has been cultivated with sugarcane for about 50 years and was a tropical forest before that. Before trial establishment, 3.5 Mg ha^{-1} of lime and 2 Mg ha^{-1} of gypsum

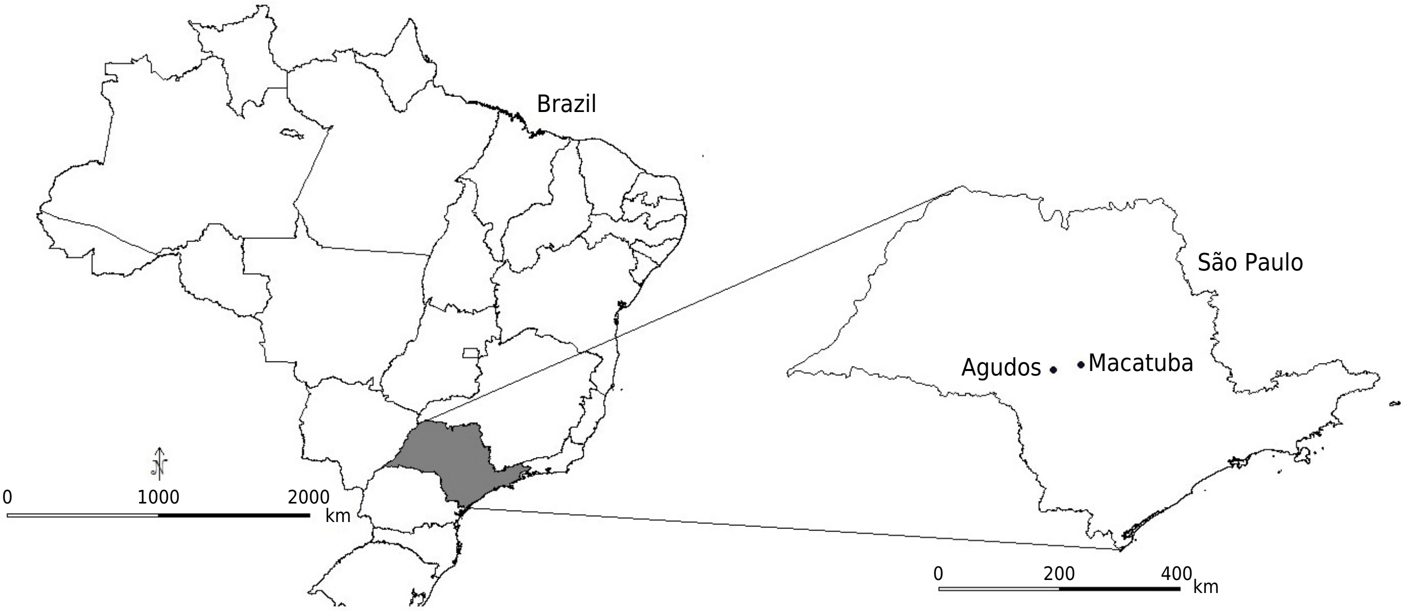


Figure 1. Geographical location where the two sugarcane experiments were set up, in Agudos and Macatuba, São Paulo State.

were applied and incorporated into 0.00-0.20 m of soil. The area was desiccated with 6 L ha^{-1} of glyphosate to eliminate the residues of the last sugarcane cycle. The trial was established in March 2014. Selected properties of both soils are given in table 1.

Treatments and establishment

The FC and inorganic P sources at both locations were arranged in a split-split-plot design with ten treatments and three replicates. The main plots consisted of the absence or presence of 10 Mg ha^{-1} (dry basis) of FC. Chemical properties of FC applied in both locations are shown in table 2. Inorganic P sources were triple superphosphate (TSP - 46 % soluble P_2O_5) at both locations, and Gafsa RP (9.6 % soluble and 28.5 % total P_2O_5) at Agudos, and Bayovar RP (14 % soluble and 29 % total P_2O_5) at Macatuba. These inorganic fertilizers were distributed in subplots at three rates (0, 90, and 180 kg ha^{-1} P_2O_5) based on their soluble P content. Each treated plot (i.e. sub-subplot) consisted of 6 lines of 10 m, with 1.5 m between rows, comprising 90 m^2 in total. Also, at sugarcane establishment, 60 kg ha^{-1} of N and 150 kg ha^{-1} of K_2O were applied in the form of urea and KCl, respectively. Phosphate fertilizer sources and FC were all applied in the bottom of the planting furrow (around 0.20-0.25 m deep from soil surface).

Sugarcane planting was performed by disposing stalks in the planting furrow with 18 to 20 buds per meter, being the buds covered by a 0.10-0.15 m soil layer. The varieties used were RB867515 in Agudos and CTC-16 in Macatuba. The RB867515 is the most cultivated variety in the Central-Southern Brazil, accounting for over 27 % of the cropping area (UFSCAR, 2016), recommended for the environments with medium natural fertility and/or sandy soils. The CTC-16 is a variety recommended for the environments with medium to high potential productivity, in soils of good fertility and high water retention capacity.

Cane harvest was performed in July 2014 and August 2015 for Agudos, and in August 2015 and September 2016 for Macatuba. Sugarcane yield and P uptake were measured at harvest. Tissue sampling and cane yield were performed manually. Yield was based on four central lines of sugarcane in each sub-subplot (total of 60 m^2). Tissue samples

Table 1. Soil physical, mineralogical, and chemical properties before sugarcane trial establishment in Agudos and Macatuba locations

Layer	pH($CaCl_2$)	OM	P	K^+	Ca^{2+}	Mg^{2+}	Al^{3+}	H+Al	SB	CEC	BS	m	Texture			CBD		Oxalate		P_{MAX}
													Sand	Silt	Clay	Fe_d	Al_d	Fe_o	Al_o	
m		g kg^{-1}	mg kg^{-1}	mmol$_c$ kg^{-1}							%		g kg^{-1}							mg kg^{-1}
Agudos (sandy soil)																				
0.00-0.10	4.3	14.3	4	1.5	7.0	5	2	25	13.2	38.1	35	11	nd	nd	nd	nd	nd	nd	nd	nd
0.10-0.20	4.2	10.7	3	1.7	5.0	3	3	28	10.3	38.0	27	21	nd	nd	nd	nd	nd	nd	nd	nd
0.00-0.20	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	884	4	112	15.3	42.3	0.6	1.4	342
0.20-0.40	3.9	nd	3	1.9	3.0	2	6	38	6.8	44.8	15	48	835	27	138	nd	nd	nd	nd	nd
0.40-0.60	4.0	nd	2	0.8	2.8	0.8	7	34	4.3	38.5	11	63	832	17	151	nd	nd	nd	nd	nd
0.60-0.80	3.9		1	0.7	2.8	0.9	9	31	3.8	34.6	11	70	825	24	151	nd	nd	nd	nd	nd
Macatuba (clayey soil)																				
0.00-0.10	5.6	31.0	11	1.9	54	25	0.9	22	80.9	103	79	1	nd	nd	nd	nd	nd	nd	nd	nd
0.10-0.20	5.3	28.5	9	1.7	44	22	0.9	28	67.7	96	71	1	nd	nd	nd	nd	nd	nd	nd	nd
0.00-0.20	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	219	152	629	110.9	53.4	4.4	1.1	1557
0.20-0.40	5.2	nd	8	1.2	30	12	0.9	25	43.2	68	63	2	211	122	667	nd	nd	nd	nd	nd
0.40-0.60	5.1	nd	5	0.7	23	9	2	25	32.7	58	57	6	nd	nd	nd	nd	nd	nd	nd	nd
0.60-0.80	4.9	nd	3	0.5	27	13	2	34	40.5	74	54	5	nd	nd	nd	nd	nd	nd	nd	nd

Organic matter determined by Walkley-Black method; P, K^+, Ca^{2+}, and Mg^{2+} extracted by ion exchange resin; Al^{3+} extracted by KCl 1 mol L^{-1}; H+Al extracted by SMP method; SB = sum of basic cations; m = base saturation; m = Al saturation; texture was determined by the Pippet method; Fe_d and Al_d were extracted by citrate-bicarbonate-dithionate; Fe_o and Al_o were extracted by ammonium oxalate; P_{max} = adsorption capacity of P; nd = not determined; pH($CaCl_2$) at a ratio of 1:2.5 m/v.

Table 2. Chemical properties of filter cakes applied by the experimental establishment at Agudos and Macatuba, Brazil

Chemical properties	DM	
	Agudos	Macatuba
pH($CaCl_2$)(1)	6.7	7.4
Total organic matter (%)(2)	28.6	53.3
Organic C (%)(2)	14.3	27.6
Total N (%)(3)	2.3	3.5
Total P_2O_5 (%)(4)	1.3	2.6
Total K_2O (%)(5)	0.14	1.07
Total Ca (%)(5)	2.0	4.1
Total Mg (%)(5)	0.16	0.33
Total S (%)(6)	0.19	0.28
C/N ratio	6.2	7.8
C/P ratio	26.2	24.4

(1) pH in $CaCl_2$ 0.01 mol L^{-1} at a ratio of 1:2.5 (m/v); (2) Loss by ignition; (3) Sulfuric digestion and micro-Kjehldal destilation; (4) Vanadate/molybdate method; (5) Atomic absorption spectrometry; (6) Gravimetry/Barium chloride. DM = mass dried at 65 °C.

of top leaves, dead leaves, and cane stalk were taken and their dry matter (DM) and P content (by acid digestion) determined (Malavolta et al., 1997). After the first season's harvest, 90 kg ha^{-1} of N as urea and 90 kg ha^{-1} of K_2O as KCl were applied as a topdressing in September 2014 at Agudos, and 100 kg ha^{-1} of N as urea and 100 kg ha^{-1} of K_2O as KCl in October 2015 at Macatuba, aiming to supply sufficient amounts of these nutrients for the second season (1st ratoon).

Soil sampling and phosphorus analyses

Soil samples were collected in August 2015 in Agudos and September 2016 in Macatuba, immediately after the harvest of the second season (1st ratoon), to perform P fractionation analysis. Four sub-samples were taken from the central rows of each plot at the top of the planting line at 0.00-0.10, 0.10-0.20, and 0.20-0.40 m layers, bulked, air dried, sieved through 2 mm mesh, packed in polyethylene bags and stored at room temperature prior to laboratory analysis.

The soil P fractionation was performed according to the methodology proposed by Hedley et al. (1982), with modifications made by Condron et al. (1985). Different extractors were added to 0.5 g of soil in sequential order: anion exchange resin (AER); $NaHCO_3$ 0.5 mol L^{-1} (labile inorganic and organic P); NaOH 0.1 mol L^{-1}; HCl 1.0 mol L^{-1} (moderately labile inorganic and organic P), and NaOH 0.5 mol L^{-1} (non-labile inorganic and organic P). In all the five steps, the suspension was stirred for 16 h in an end-over-end shaker (33 rpm). At the end of the sequential extraction, the remaining residual soil was dried at 50 °C, ground to homogenize, and digested by concentrated H_2SO_4, 30 % H_2O_2, and saturated magnesium chloride to extract residual P (Brookes et al., 1982). Phosphorus concentration in the extracts were measured by colorimetric method of Murphy and Riley (1962) for acid extracts and Dick and Tabatabai (1977) for alkaline extracts using spectrophotometer (Femto 600 plus). The inorganic fractions were determined directly in the extract. The organic P fractions were estimated as the difference between total P fractions, determined after digestion of the alkaline extracts with 7.5 % (w/v) ammonium persulfate [$(NH_4)_2S_2O_8$] solution and 50 % H_2SO_4 in an autoclave (103 kPa, 121 °C) for 2 h (Kopp and McKee, 1979), and the respective inorganic fractions.

The maximum P adsorption capacity of those soils (P_{MAX}) was determined in soil samples collected from the depth of 0.00-0.20 m before the experimental establishment, following the methodology proposed by Sui and Thompson (2000). In order to identify the main minerals in the soil, X-ray diffraction (XRD) analysis of the clay fraction was run in the same soil samples from P_{MAX}, following the methodology described by Jackson (2005) for glass slide preparation. The diffractograms were generated using a Miniflex II Desktop X-Ray Diffractometer (Rigaku Corporation, Tokyo, Japan), with CuKα radiation, with intervals from 5 to 30° 2θ and from 10 to 50° 2θ for samples without and with iron, respectively (Figure 2). Accordingly, there was a predominance of kaolinite in Agudos soil with small proportions of iron/aluminum oxides, otherwise in Macatuba a great proportion of hematite/gibbsite besides kaolinite was detected, what explains the high P_{MAX} value observed in this soil.

Statistical analysis

Variance homogeneity and normality of data were tested for each parameter before conducting analysis of variance (ANOVA). Data were transformed using Box-Cox techniques (Box and Cox, 1964) and outliers were removed when needed, and then the data were submitted to ANOVA using PROC GLM to test the effect of FC, phosphate sources, and rates on soil P fractions. When the interaction between factors and/or each isolated factor were significant, means were compared using LSD ($p<0.05$). All the statistical analyses were performed by SAS 9.3 (SAS Institute, Inc., Cary, NC, USA).

RESULTS

There was no significant interaction between FC and either P sources or rates at both locations. Hence we investigated the simple effect of FC addition on sugarcane yield and soil P fractions. Results for the soil P fractionation analysis are averaged over 0.00-0.40 m to take full account of the distribution of P placed at 0.20-0.25 m, it was impossible to get a good picture of the treatment effect when evaluating isolated each layer (0.00-0.10, 0.10-0.20, and 0.20-0.40 m). There were however significant interactions between inorganic P sources and rates and their effects on yield and soil P.

Cane yield and P uptake

At the sandy soil site (Agudos), FC addition increased significantly the yield of sugarcane in the first year across all P sources and rates, ranging from a 6.0 % increase with TSP_{180}

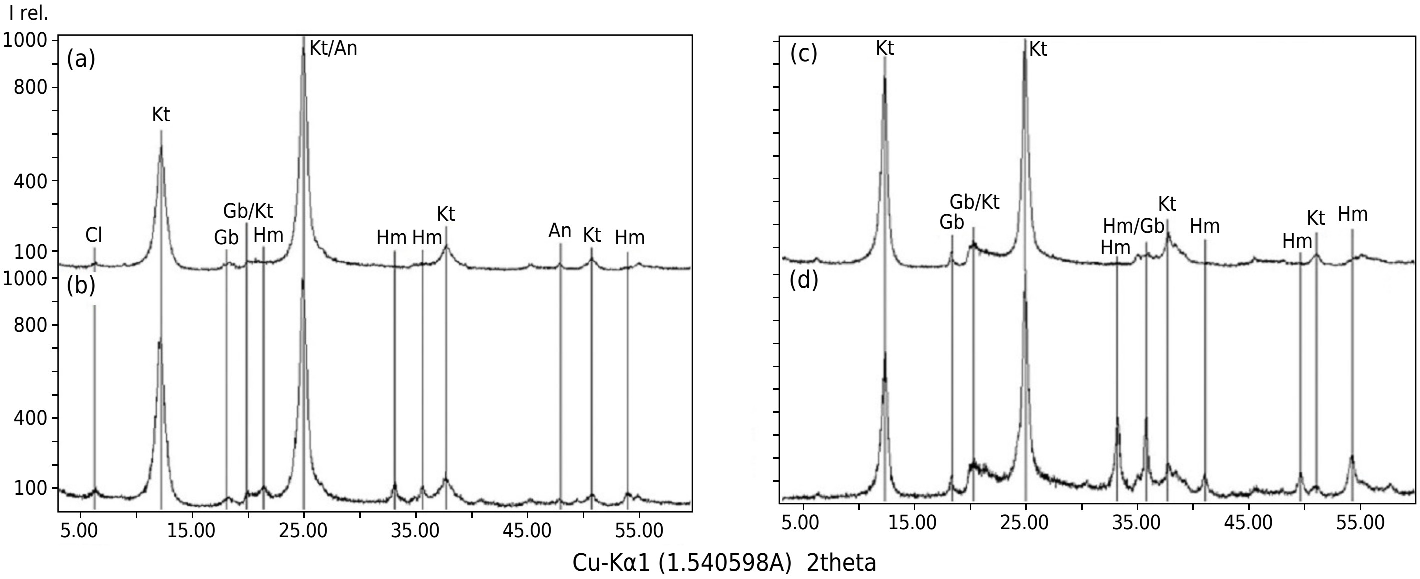


Figure 2. X-ray diffraction of the clay fraction from the layer of 0.00-0.20 m treated with (a and c) and without (b and d) citrate-bicarbonate-dithionate (CBD). Figures (a) and (b) are related to Agudos and figures (c) and (d) are related to Macatuba. An = anatase; Cl = chlorite; Gb = gibbsite; Hm = hematite; Kt = kaolinite.

to a 20.1 % increase with RP_{180} (Figure 3a). However in the second year, the effect of FC on cane yield was negligible, being significant only for nil-P (Figure 3b). The P uptake by sugarcane at Agudos was also not changed significantly in either year with FC application (Table 3). When different sources and rates of mineral phosphate were used, the P uptake increased by 19 % in the first year (from 12.0 to 14.3 kg ha^{-1} under nil-P and averaged across fertilized treatments, respectively) and by 29 % in the second year (from 15 to 19.4 kg ha^{-1} under nil-P and averaged across fertilized treatments, respectively) without considering FC application. Although the yield was not changed substantially, P uptake on average increased around 34 % from the first to the second year.

At the clayey soil site (Macatuba), with previous sugarcane cultivation and greater soil P fertility levels, FC application enhanced yield significantly only under TSP application, in both rates, without any response under nil-P and RP (Figure 3c), while in the second year no influence of FC addition was detected (Figure 3d). The P uptake at Macatuba was not affected by FC application in either years. When FC was applied, the highest yield was recorded under nil-P and RP_{90}, while in NFC treatments, P sources and rates did not influence the yield of sugarcane in the first year (Figure 3c). In the second

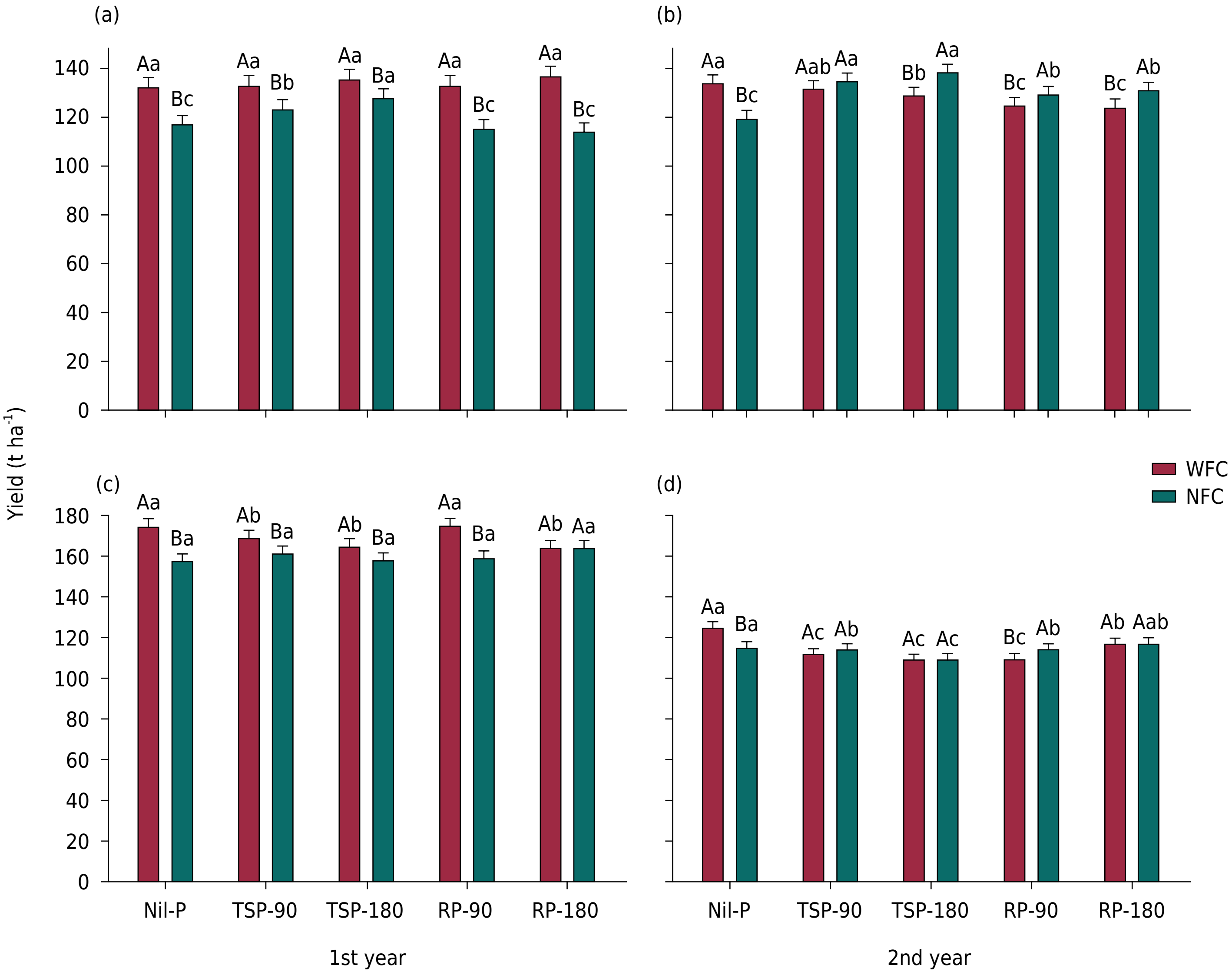


Figure 3. Yield of sugarcane at Agudos in 2014 (a) and 2015 (b) and at Macatuba in 2015 (c) and 2016 (d) affected by filter cake addition and P sources and rates. NFC = no filter cake; WFC = with filter cake; Nil-P = no phosphate sources; TSP = triple superphosphate; RP = rock phosphate; 1st and 2nd years are 2014 and 2015 at Agudos and 2015 and 2016 at Macatuba, respectively. Within each fertilizer treatment, means followed by the same capital letter were not significantly different, and within each filter cake treatment, means followed by the same small letter were not significantly different at $p<0.05$ by LSD test, error bars represent standard errors.

year, the highest yield was obtained under nil-P followed by RP_{180} when FC applied. In treatments without FC application, the highest yield was recorded under nil-P, while TSP_{180} resulted in the lowest yield compared to the other treatments (Figure 3d). A reduction of 31 % in the average yield and P uptake was detected from the first to the second year in Macatuba.

Effects of filter cake on soil P fractions

Among labile P fractions, FC addition enhanced only Pi_{BIC} by 45.7 % (averaged among P sources and rates) at Agudos (Figure 4a), while at Macatuba it increased both inorganic labile P fractions (P_{AER} and Pi_{BIC}) by 75 and 71.4 % (averaged among P sources and rates) for P_{AER} and Pi_{BIC}, respectively (Figure 4b). The organic labile P (Po_{BIC}) was not affected by FC application at either site (Figures 4a and 4b). Mod-labile P fractions were not drastically affected by FC addition, with only a slight increase in $Pi_{HID\text{-}0.1}$ at Agudos (Figure 4c) and $P_{HID\text{-}0.1}$ (inorganic and organic) at Macatuba (Figure 4d), and with no changes in P_{HCl} fraction. Non-labile P fractions were not affected by FC application at either location (Figures 4e and 4f).

Filter cake addition (WFC) did not change the proportion of each P fraction (i.e. % of total P) and, on average, non-labile P was 70.9 % of total P at Macatuba and 43.2 % at Agudos. Labile P represented 12.2 and 4.8 % at Agudos and Macatuba, respectively, but it is noteworthy that total soil P at Macatuba was much higher than at Agudos, mostly accumulated as the non-labile P fraction, as a consequence of the much greater soil P adsorption capacity (Table 1) and mineralogical constitution predominantly constituted of Fe/Al (hydr)oxides in the clay fraction (Figure 2).

Effects of P sources and rates on soil P fractions

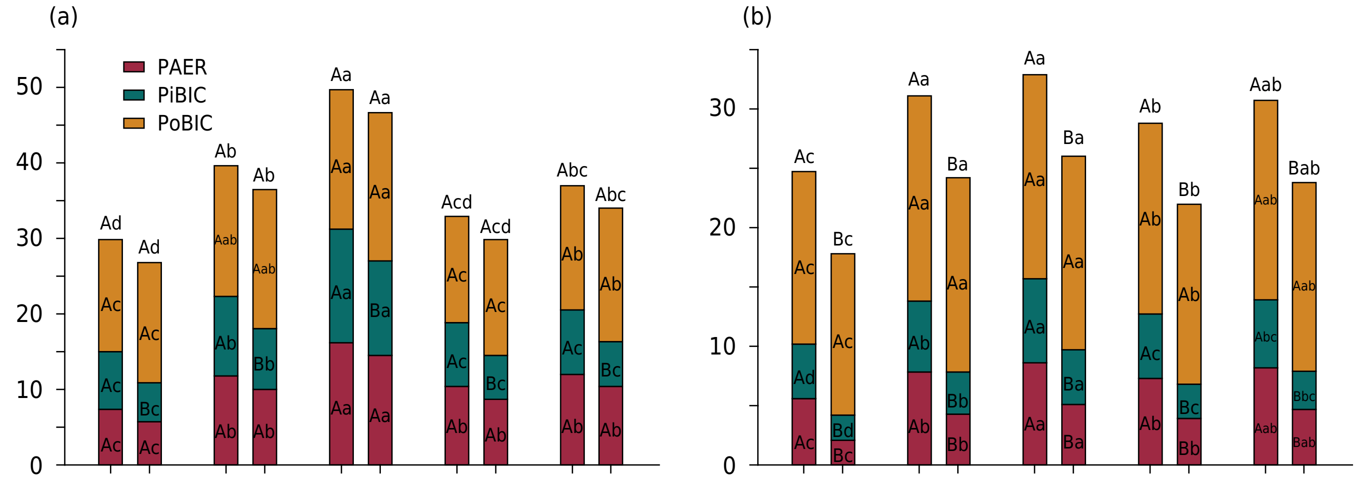
Triple superphosphate was the most effective P source to increase the labile P fractions in the soil. The treatment TSP_{180} enhanced inorganic labile P substantially compared to the other treatments, especially at Agudos (Figure 4a). The TSP_{90} and RP at both rates (RP_{90} and RP_{180}) also increased P_{AER} compared to nil-P, but these treatments did not differ from each other. The TSP_{90} raised Pi_{BIC} in comparison with nil-P, while RP did not influence this P fraction. The TSP_{180} was the only treatment which enhanced Po_{BIC} relative to nil-P.

Table 3. Phosphorus uptake by sugarcane in two consecutive crop seasons as affected by filter cake addition and P sources and rates at crop establishment

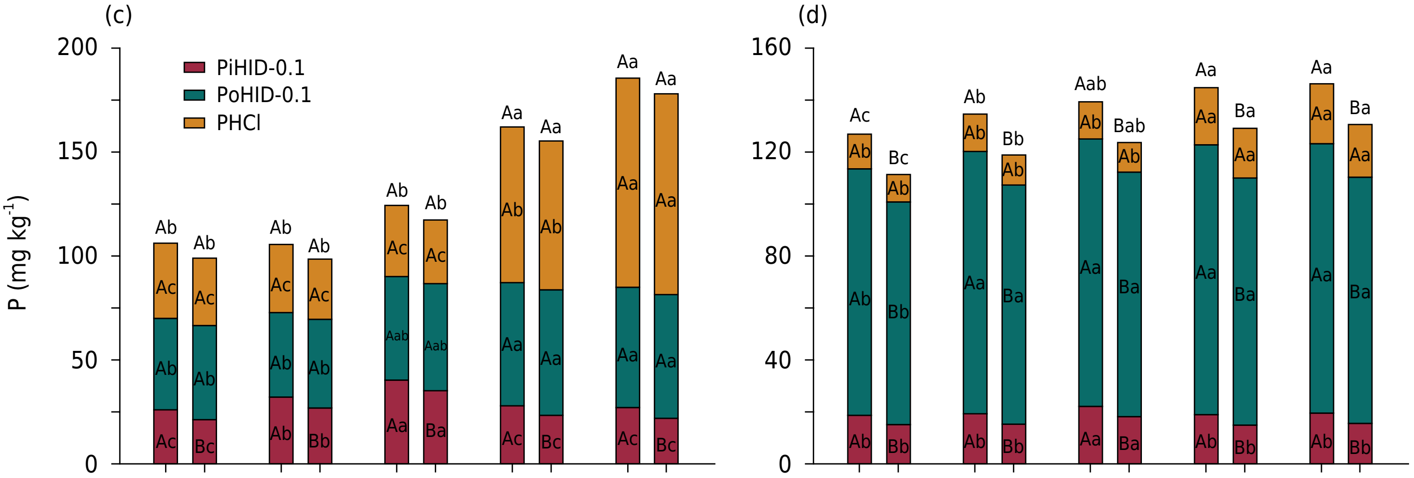
Filter cake	Mineral phosphate source/rate	Agudos 1st year	Agudos 2nd year	Macatuba 1st year	Macatuba 2nd year
		P uptake[1]			
		kg ha^{-1}			
WFC	Nil-P	13.1^{ns}	17.0^{ns}	26.8^{ns}	19.9^{ns}
	TSP-90	15.2	20.1	24.3	19.0
	TSP-180	14.9	19.9	24.2	18.8
	RP-90	15.0	19.6	25.0	19.2
	RP-180	14.7	19.5	24.8	19.6
NFC	Nil-P	10.9	13.0	21.5	18.9
	TSP-90	13.8	19.3	22.0	18.0
	TSP-180	13.6	19.1	21.8	17.8
	RP-90	13.6	18.8	22.7	18.2
	RP-180	13.4	18.6	22.5	18.6

[1] Nitro-perchloric digestion and vanadate/molybdate determination. NFC = no filter cake; WFC = with filter cake; Nil-P = no phosphate sources; TSP = triple superphosphate; RP = rock phosphate; 1st and 2nd years are 2014 and 2015 in Agudos and 2015 and 2016 in Macatuba, respectively. ^{ns} = non-significant at p<0.05 error probability.

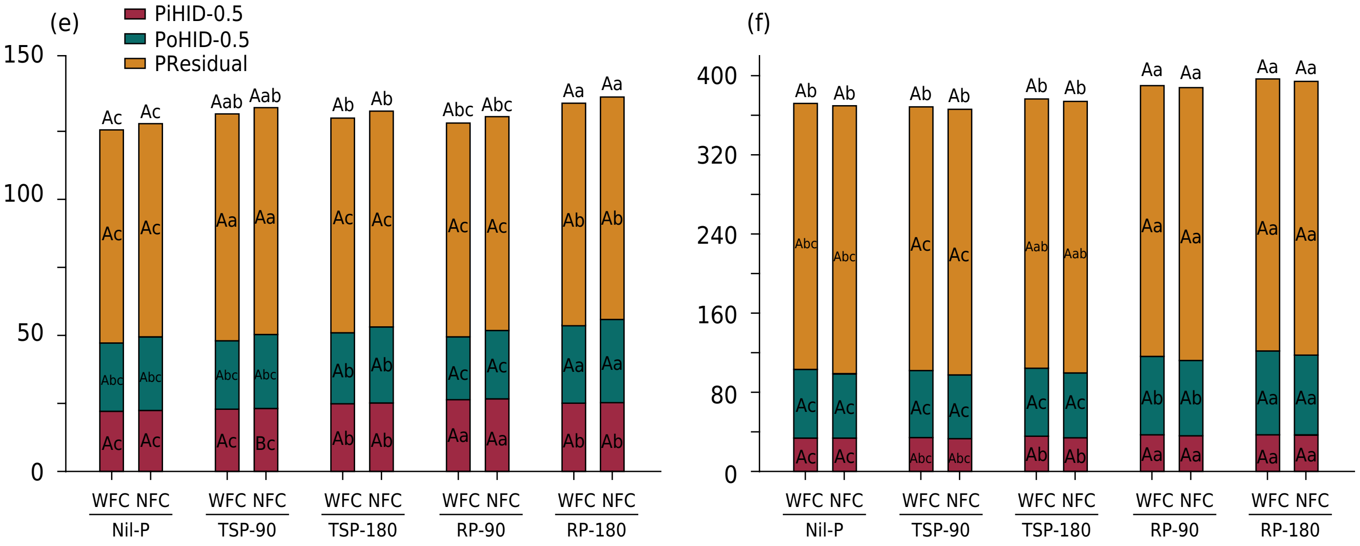
In general, labile P was increased by all the treatments except RP_{90} relative to nil-P, and the greatest increase was observed with TSP_{180} (194 %) followed by TSP_{90} (95 %).



Labile P fractions



Mod-labile P fractions



Non-labile P fractions

Figure 4. Labile (a), mod-labile (c), and non-labile (e) P fractions at Agudos and labile (b), mod-labile (d), and non-labile (f) P fractions at Macatuba affected by filter cake addition and P sources and rates after two years. Within each fertilizer treatment, means followed by the same capital letter were not significantly different, and within each filter cake treatment, means followed by the same small letter were not significantly different at $p<0.05$ by LSD test.

At Macatuba, all the inorganic P treatments (sources and rates) enhanced P_{AER} and Pi_{BIC} compared to nil-P (Figure 4b). The highest P_{AER} and Pi_{BIC} values were recorded under TSP_{180}, significantly higher than nil-P and RP_{90} for P_{AER} and significantly higher than all the treatments for Pi_{BIC}. The organic labile P (Po_{BIC}) was enhanced by P sources and rates compared to nil-P, and this fraction constituted the highest proportion of labile P in Macatuba, varying from 53.7 % (averaged among WFC and NFC) under TSP_{180} to 75.2 % (averaged among WFC and NFC) under nil-P, reducing accordingly with the increase in the rate of P sources. In general, labile P was increased by P addition with the greatest increase observed under TSP_{180} (+103 %).

Investigating the mod-labile P fractions at Agudos, the highest $Pi_{HID-0.1}$ was recorded under TSP_{180}, significantly different from all the other treatments (Figure 4c). Among other treatments, only TSP_{90} succeeded to enhance this P fraction while RP addition did not affect it related to nil-P. The $Po_{HID-0.1}$ levels were significantly higher than nil-P when RP was applied, irrespective of the rate. As expected, P_{HCl} levels under RP application were significantly higher than the other treatments, while TSP did not change this P fraction compared to nil-P. The P_{HCl} fraction constituted 50.2 and 60.2 % (averaged among WFC and NFC) of mod-labile P under RP_{90} and RP_{180}, respectively. In general, mod-labile P under TSP application at both rates was similar to nil-P, while RP addition enhanced mod-labile P compared to nil-P at both rates, 156 and 222 % (averaged among WFC and NFC) under RP_{90} and RP_{180}, respectively, mostly due to the increment of P_{HCl}.

At Macatuba, $Pi_{HID-0.1}$ was raised by TSP_{180} compared to nil-P, while the other treatments did not influence this P fraction (Figure 4d). Otherwise, $Po_{HID-0.1}$ under all P sources and rates was higher than nil-P, while no differences among P sources and rates. This fraction constituted the highest proportion of mod-labile P, even under RP application, from 69.5 % (averaged among WFC and NFC) under RP_{180} to 78.2 % (averaged among WFC and NFC) under TSP_{90}. The P_{HCl} increased similarly by RP addition at both rates compared to the other treatments (TSP and nil-P). In general, mod-labile P was enhanced by all the treatments related to nil-P, but the increment was not as substantial as observed in Agudos.

Considering the non-labile P fractions, $Pi_{HID-0.5}$ was increased by all the treatments other than TSP_{90} in comparison with nil-P at Agudos (Figure 4e). The highest $Po_{HID-0.5}$ and $P_{Residual}$ levels were obtained under RP_{180} and TSP_{90}, respectively. The RP_{180} resulted in the highest accumulation of P in non-labile fraction, significantly higher than all the treatments except TSP_{90}. At Macatuba, RP application at both rates resulted in the highest $Pi_{HID-0.5}$ and $P_{Residual}$ levels (Figure 4f). Rock phosphate addition also enhanced $Po_{HID-0.5}$ and this enhancement intensified with increasing the rate. In general, RP addition at both rates enhanced non-labile P compared to the other treatments, while TSP did not show any difference with nil-P.

DISCUSSION

Filter cake as an organic residue rich in nutrients is able to increase exchangeable basic cations like Ca and reduce exchangeable Al and acidity (Almeida Junior et al., 2011), potentially reducing P fixation capacity leading to more available P in the soil. The combination of these positive effects of FC can result in yield improvement, as was observed at both Agudos and Macatuba soils in the first year when comparing absence and presence of FC. Filter cake can also enhance water retention which is important in sugarcane cultivation, especially during months with low rainfall and without irrigation. The clayey soil at Macatuba had originally a better water retention capacity compared to the sandy soil in Agudos (data not shown). The yield enhancement from FC was also observed not just on the nil-P plots but also on treated plots and the addition of FC did not significantly increase crop P uptake at either sites. Hence it is more likely that the greater positive effect of FC addition on yield at Agudos compared to

Macatuba was due to improved water availability and soil general improvement rather than a lack of available P, even though soil P fertility at Agudos was classified as low (van Raij et al., 1997).

Our results are in accordance with those reported by Sousa et al. (2015) investigating the effect of FC addition in sugarcane in a clayey soil, where they observed the same trend with response to FC only in the first year of cultivation. As the levels of P in non-fertilized treatments were low in both trials, yield response should be expected to P addition. However, cane yield and P uptake were not different between TSP and RP at both locations and in both years, in this way, we cannot infer that a slow-release P source such as RP is as efficient as soluble source. Similarly, Lima et al. (2006) and Korndörfer and Melo (2009) observed that both cane yield and sugar content were not enhanced with P fertilizer.

The proportion of P and other nutrients in organic residues is affected by the rate of mineralization of the material and release of nutrients to the soil. Total P in residues higher than 0.25 % and C:P ratio less than 200:1 are prerequisites for the quick release of P from organic material by mineralization (Utami et al., 2012). Both FCs used at Agudos and Macatuba are well fitted in these characteristics (Table 2). The amount of P in FC applied at Macatuba (2.59 % P_2O_5) was higher than Agudos (1.25 % P_2O_5) which influenced the total P added to the soil. Moreover, FC as an organic product presents compounds able to compete with phosphate for adsorption sites in the soil, and this competition should be more pronounced at Macatuba in which high levels of hematite/gibbsite are present, promoting high P fixing (P_{MAX}). For example, at Macatuba, FC addition enhanced P_{AER} and Pi_{BIC} by 75 and 71.4 %, respectively, compared to NFC, while in Agudos it increased only Pi_{BIC} by 45.7 %. Similarly, increasing soil available P due to FC application has been reported in other studies (Shankaraiah and Murthy, 2005; Elsayed et al., 2008; Lima, 2011; Caione et al., 2015).

Phosphorus in FC is mostly organic (Negassa et al., 2010) and its release happens gradually (maybe in two or more years depending on the climate and location) with mineralization by soil microorganisms (Torres et al., 2012). Organic amendments like FC and vinasse can increase organic labile P pool (Po_{BIC}) in sugarcane fields (Cherubin et al., 2016), although this enhancement was not observed at our study locations. Iyamuremye et al. (1996) and Li et al. (2015) reported an increase in NaOH-Pi (Pi_{HID}) and to a lesser extent NaOH-Po (Po_{HID}) when P-rich organic amendments were applied to the soil. Confirming their results, we can state that the addition of these kinds of organic amendments can enhance $Pi_{HID-0.1}$ and to a lesser extent $Po_{HID-0.1}$ (only in Macatuba) without any changes in $P_{HID-0.5}$ fractions. Nunes Junior (2008) reported that FC can be useful up to three years depending on the climate, in tropical regions it remains for two years, while in subtropical regions like São Paulo and Paraná States, it can be effective for up to three years. In our trials, we can state that this effectiveness is soil specific and depends on the amount of nutrients in FC. Higher amounts of nutrients in FC and finer-textured soil normally result in longer effectiveness time for sugarcane nutrition.

The association of phosphate fertilizer sources with FC is an option to increase the fertilizer efficiency by adding more compounds into the soil to compete for adsorption sites. Penso et al. (1982) recommended mixed application of FC and mineral phosphate, expecting that FC facilitates the solubility of P sources compared to the use of mineral P alone, but this effect was not observed in our research as FC did not interact with P sources and rates. On the other hand, FC did not act as a carrier for P, however, it was an important source of P and other nutrients for sugarcane. Moreover, FC may also enhance the release of phosphatase enzymes by sugarcane roots, which needs more investigation.

Application of TSP at the rate of 180 kg ha^{-1} P_2O_5 in combination with FC addition in Agudos was the only treatment at either location that maintained P_{AER} at agronomically sufficient levels (≥15 mg kg^{-1}, van Raij et al., 1997) after two years of cultivation. Under RP application, we expected to record higher (Ruaysoongnern and Keerati-Kasikorn, 1998) amounts of P_{BIC} (sum of Pi_{BIC} and Po_{BIC}) compared to TSP application, while in both locations the reverse trend was observed. However, considering the P_{AER} + Pi_{BIC} as the inorganic labile P, it suggests that TSP applied at both rates and RP_{180} kept available P at adequate levels for sugarcane development (≥15 mg kg^{-1}) at Agudos. At Macatuba, the levels of P_{AER} were considered as the inorganic labile P, the values were adequate under TSP_{180} but not under other treatments. Otherwise, considering the levels of Po_{BIC}, its amount at Macatuba was much higher than Agudos. This P fraction is mineralized by microorganisms, supposedly buffering inorganic P in solution when depleted and, consequently, keeping the soil capacity to supply P for the plants.

Triple superphosphate applied at the rate of 90 kg ha^{-1} P_2O_5 (TSP_{90}) did not enhance Pi_{HID} in either soils, but this fraction was increased by TSP_{180}. This is in contrast with other results (Sample et al., 1980; Zoysa et al., 2001; Savini et al., 2006) whose authors reported that the P release from TSP may not be synchronized with plant P uptake and the excessive amounts of P released into the soil solution would be transformed into Pi_{HID} fraction, especially in high P fixing soils. The fraction P_{HCl}, the primary P mineral fraction of the soil when soluble sources are applied (Stewart et al., 1987), was not influenced by TSP application at both rates in both soil textures. This is in agreement with Wagar et al. (1986) who showed that broadcast application of soluble P sources at the rate of 160 kg ha^{-1} did not change this P pool in Chernozomic soils.

Considering organic P fractions (Po_{BIC}, $Po_{HID-0.1}$, and $Po_{HID-0.5}$), labile organic P (Po_{BIC}) was enhanced by TSP application at the rate of 180 kg ha^{-1} P_2O_5 (TSP_{180}) in the sandy soil, while in the clayey soil this P fraction was not affected by P addition. Mod-labile organic P fraction ($Po_{HID-0.1}$) was increased by RP addition at both rates again only in sandy soil with no significant changes in clayey soil. Non-labile organic P fraction ($Po_{HID-0.5}$) was only affected by RP addition at the rate of 180 kg ha^{-1} P_2O_5 (RP_{180}) in the clayey soil but with no changes in the sandy soil. Averaged across the treatments, total organic P at Macatuba was two-fold greater than at Agudos (185.2 and 94.1 mg kg^{-1} in Macatuba and Agudos, respectively). Higher soil moisture and fertility at Macatuba due to the higher clay content can result in more plant growth and plant inputs to the soil and, consequently, higher microbial activity. Organic P in root exudates and in microbial products stabilized more intensely in the clayey soil compared to the sandy soil (Tiessen et al., 1984) resulting in higher organic P in the soil with higher clay content. The increment of organic P fractions with decreasing the particle size of the soil was also observed in forest and croplands in Denmark and Germany (Rubæk et al., 1999), Tanzania (Solomon and Lehmann, 2000), and Caucasus (Makarov et al., 2004).

The proportion of labile P fractions in Macatuba was less than Agudos and a very high proportion of P accumulated as non-labile P at Macatuba due to the higher P adsorption capacity of the soil (1,557 mg kg^{-1}) compared to Agudos (342 mg kg^{-1}) (Table 1). Prochnow et al. (2006) showed that when soluble P sources were applied to the soils with high P fixing capacity, the available P for plant uptake (Bray P_1) was reduced, the same trend was observed here for RP (slow release) in sugarcane field after two years from application. In both places, the proportion of labile P increased with increasing the rate of P applied at both sources which contradicts the results obtained by Castillo and Wright (2008), who reported the reverse trend in sugarcane fields of Everglades, USA, probably due to the short time of investigation in their study (21 days after P application). Stewart et al. (1987) stated that the proportion of residual P fractions did not change with soil texture, however, the proportion of residual P was not changed with P sources and P rates in clayey soil, while in sandy soil this proportion reduced with increasing P application rates and it was smaller under RP compared to TSP due to greater P_{HCl} fraction under this source. Averaged across P sources and P rates, a big proportion of P (70.8 %) was accumulated in non-labile fraction in Macatuba, while this proportion was 45.1 % in Agudos, what is partially explained by the total Fe content in Macatuba (115.3 g kg^{-1}), 7.3 times higher than Agudos (15.9 g kg^{-1}). In acidic soils, P precipitation by Fe and Al is considered as the primary mechanism of P retention (Sanchez and Porter, 1994) which resulted in this difference in non-labile P fractions among our locations. When RP was applied at Agudos, the proportion of P accumulated in non-labile pool decreased from 51.4 % (averaged across nil-P and TSP) to 35.8 %, while at Macatuba it was not changed. In addition to which was stated above about the use of RP at the rate of 180 kg ha^{-1} P_2O_5 to keep the sufficient amount of labile P for sugarcane development in sandy soil, this lower amount of non-labile P when RP was applied in Agudos can strengthen our recommendation.

CONCLUSIONS

Filter cake application at sugarcane establishment increased crop yields in the first year on both sandy and clayey soils, and also the levels of available P in the soil for up to two years, being more effective in sandy soil, however FC addition was not able to enhance soil organic P fractions. Moreover, FC efficiency can be improved when enriched in nutrient content, especially when applied in sandy soils.

Inorganic phosphate sources were effective to maintain adequate soil P levels after two years, especially TSP, irrespective of the rate. Rock phosphate was not as effective as expected since a big proportion of it was accumulated in mod-labile P fraction extracted with HCl 1.0 mol L^{-1}, and not solubilized in a reasonable time. However, none of the phosphate sources were capable to improve sugarcane yield in both years and locals evaluated here.

Our hypothesis that filter cake should have a great interaction with mineral P sources and keep more P available in the soil was not proven here, since we did not get any substantial effect of filter cake in the solubilization of rock phosphate nor on the availability of P from soluble sources (triple superphosphate).

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