

Research Article

Phosphorus Sorption and Redistribution on Soil Solid Phase in a Brazilian Haplorthox Amended with Biosolids

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Land application of biosolids (SS) can cause a buildup of phosphorus (P) in the top soil. The changes in the soil P characteristics may be assessed by the sorption isotherm and the sequential fractionation techniques. Samples of Haplorthox were collected from a field experiment where maize was cultivated for two years, after two applications of SS originated from two cities of São Paulo State, Brazil. SS applications added a total of 125, 250, 500, 1000 and 2000 kg ha⁻¹ of P in the area. To perform the sorption isotherms and obtain P maximum sorption capacity (Q_{max}) and the binding energy, soil samples were submitted to increasing P concentration solutions until equilibrium was reached. Sequential fractionation was done by a sequential extraction with CaCl₂, NaHCO₃, NaOH, HCl, and HNO₃ + HClO₄ (residual). Addition of biosolids from both cities to the soil decreased Q_{max} and the binding energy obtained by the Langmuir equation. SS additions changed the P fractions distribution in the soil by increasing the labile fractions (P-CaCl₂ and P-NaHCO₃) and the moderately labile fraction (P-NaOH) by 11.2% and 20.3%, respectively, in detriment of the most resistant P fraction.

1. Introduction

A sustainable option to dispose biosolids (SS) originated from sewage treatment plants is the use as soil conditioner or as a source of nutrients to plants. Nevertheless, high SS rates might add to soil excessive amounts of phosphorus (P). One of the consequences is the increase on the risk of environment contamination by P transport to superficial water reservoirs, since P can be weakly sorbed in the superficial soil layer and carried over by rainfall runoff. Furthermore, topsoil P concentrations have been related to P movement below the plough layer and tile flow [1]. Recently, Gebrim et al. [2] observed that P leaching through the profile can be significant in highly weathered soils, where residual P is accumulated by successive crops, particularly when poultry litter is applied as fertilizer.

Among the useful parameters to monitor the increase in soil P availability is the P sorption capacity and P distribution among the main soil fractions. The application of organic residues to soils might implicate in change in soil P sorption capacity. According to Reddy et al. [3], application of organic

wastes increases soluble P and decreases the P sorption capacity of the soil, and these parameters were directly related to the loading rates of animal wastes. The positive effects of manure on the solubility of P are due to the addition of soluble inorganic P and mineralization of organic P during decomposition of waste, which probably saturated the adsorption sites. Or else, some organic acids released during residue decomposition might compete with P for the same sorption sites in the soil solid phase, decreasing the element immobilization or forming stable complexes with Fe and Al and consequently blocking P retention by them [4]. Nevertheless, it is supposed that frequent SS addition can increase the soil organic matter content which has a positive correlation with the capacity of soil in retaining P [5].

The sorption isotherms can be useful to detect such alterations. This technique consists of mixing a P-enriched solution with a soil sample, at constant temperature and ionic strength, and graphically plotting the P quantity sorbed by soil versus the P remained in solution. The curve yielded, which although asymptotic, can be described by equations such as the Freundlich or Langmuir. The Langmuir equation

can better provide estimates of P sorption maximum and the sorption strength of the soil, parameters inherent in determining the rate of P movement through the soil [6]. The Langmuir equation in its simplest form assumes only monolayer sorption and hence tends to greatly underestimate the P sorption maximum but yields an estimate of P sorption strength that represents an average of the total population of sorption sites [6]. Holford et al. [7] proposed that to measure a reasonable P sorption maximum, a “two-surface” Langmuir equation should be used. This equation assumes that the total number of sorption sites can be separated into two populations, one with an affinity much greater than the other. According to these authors, the application of this equation to studies of P sorption in soil has revealed much in terms of the relative effect of soil constituents, including P fertilizer and manure application rates and soil fertility.

After SS addition to the soil, the P species present in the material will react in different ways, depending on the soil chemical, physical, or biological characteristics, until the steady state equilibrium. This dynamics can be evaluated through the element redistribution in the main soil fractions. Therefore, the sequential fractionation is an important tool once it is based on the extractor affinity with a specific-soil P species. The method of Heddley et al. [8], for instance, is able to identify four main P fractions: (a) labile P (P extractable by anionic resin plus P extractable by NaHCO_3); (b) moderately labile P (extractable by NaOH); (c) P bound to Ca (extractable by HCl) and (d) residual P (determined after nitric-perchloric acid digestion).

In Brazil, there is little information about the SS effects on soil P dynamics. Thus, any new available information quantifying and/or identifying some of the soil P dynamic steps will be relevant to improve the efficacy and safety of actual recommendations on SS application in agricultural lands.

The objectives of this work were to evaluate, by means of sorption isotherms, the possible changes in soil P sorption capacity and also to assess P redistribution among the main P fractions in an oxisol that received two successive applications of sewage sludge.

2. Material and Methods

Soil samples were collected from a long-term experiment under field conditions, on an Haplorthox area (dystrophic Red Yellow Oxisol) in the experiment station of Embrapa, at Jaguariúna (22°41'S; 47°06'W·Gr.; altitude 570 m), State of São Paulo, Brazil, after two SS additions. Some of the soil chemical and physical characteristics before SS additions are given in Table 1. Organic matter (OM) was determined by a colorimetric procedure, after digestion of samples with sodium dichromate solution in sulfuric acid and comparing the readings with a standard curve prepared with soil samples previously analyzed by Walkley-Black's method [9]. Soil pH (1:2.5 solid/solution ratio) was determined in a CaCl_2 0.01 mol L⁻¹ solution. In the same suspension, the SMP buffer pH was measured, and with this value, the potential

TABLE 1: Selected chemical and physical characteristics of the studied soil.

Soil attribute	Value
pH (CaCl_2)	4.4
Organic matter (g kg^{-1})	22
P (mg kg^{-1})	9.4
K ($\text{mmol}_c \text{ kg}^{-1}$)	1.2
Ca ($\text{mmol}_c \text{ kg}^{-1}$)	18.9
Mg ($\text{mmol}_c \text{ kg}^{-1}$)	11.8
H + Al ($\text{mmol}_c \text{ kg}^{-1}$)	61.4
S ($\text{mmol}_c \text{ kg}^{-1}$)	31.9
CEC ($\text{mmol}_c \text{ kg}^{-1}$)	93.2
V (%)	34
Fe _{ox} (mmol kg^{-1})	18
Al _{ox} (mmol kg^{-1})	519
Coarse sand (g kg^{-1})	340
Fine sand (g kg^{-1})	240
Silt (g kg^{-1})	100
Clay (g kg^{-1})	320
Bulk density (g dm^{-3})	1.18

acidity was evaluated [10]. Phosphorus (P), calcium (Ca), magnesium (Mg), and potassium (K) were extracted from the soil by an ion-exchange resin procedure, after a 16-hour shaking period. P was determined by colorimetric method, K by flame photometry, and Ca and Mg by atomic absorption spectrophotometry [11]. The values of sum of bases (S), cation exchange capacity (CEC), and percent base saturation (V) were calculated. Oxalate-extractable Fe and Al were obtained by extraction with ammonium oxalate-oxalic acid [12] to determine P associated with amorphous oxides of Fe and Al. Physical analysis was performed according to Camargo et al. [13]. The hydrometer method was used for particle-size analysis after dispersion with a mixture of 0.015 mol L⁻¹ $(\text{NaPO}_3)_6 \cdot \text{Na}_2\text{O}$ and 0.1 mol L⁻¹ NaOH by overnight shaking. Bulk density was determined on 76 mm × 76 mm diameter undisturbed soil cores [14].

The main objective was to evaluate the agroenvironmental effects caused by successive SS applications before maize (*Zea mays* L., CATI AL 30 cultivar) cropping. Hence, in order to evaluate the environmental impacts, such as heavy-metal phytoavailability and accumulation in soil and nitrate leaching, biosolids were also applied in higher amounts than the recommended for optimal crop growth. Biosolids from the cities of Barueri and Franca, both from the State of São Paulo, were used in the experiment. Some SS characteristics are presented in Table 2. Nitrogen was determined in the original SS-samples using the Kjeldahl method (vapor distillation), and the organic-C was determined by titrimetry after dichromate digestion in digester block, according to Raji et al. [15]. Phosphorus, sulfur, calcium, magnesium, and potassium were analyzed according to the recommendations of US-EPA (SW-846) method no. 3051 [16] K by flame photometry, and other elements by ICP-AES. Humidity was

TABLE 2: Partial chemical analysis of biosolids samples originated from Barueri and Franca cities and used for two crops growth.

Variables	Unity	Barueri		Franca	
		1st crop	2nd crop	1st crop	2nd crop
pH	—	6.6	6.4	6.3	6.4
Organic carbon	g kg ⁻¹	248	271	305	374
C/N ratio	—	11.8	10.3	5.4	7.4
Humidity	g kg ⁻¹	66	533	83	521
Total nitrogen	g kg ⁻¹	21.0	26.4	56.4	50.8
Available nitrogen	g kg ⁻¹	5.7	22.5	15.3	25.7
Total phosphorus	g kg ⁻¹	16	31.2	16	21.3
Total sulfur	g kg ⁻¹	13.4	10.8	16.3	13.3
Total calcium	g kg ⁻¹	40.3	22.8	29.2	16.8
Total magnesium	g kg ⁻¹	3.0	3.7	2.2	2.5
Total potassium	g kg ⁻¹	1.0	2.0	1.0	1.0

determined by mass loss at 60°C and SS sample pH was determined in water extract (1 : 5).

The experiment consisted of twelve treatments in a randomized complete block design with three replications, in a total of 36 plots, each plot measuring 20 m length versus 10 m width. The study treatments were: two control (no N, P and K applied); two mineral fertilization recommended for the crop (46 kg N ha⁻¹ for the first crop and 90 kg N ha⁻¹ for the second crop; 37 kg P ha⁻¹ and 56 kg K ha⁻¹ on both crops) [17]; four treatments with the SS from the city of Franca, four treatments with the SS from the city of Barueri. The sewage sludge treatments were applied based on the N concentration that provides the same amount of N as in the mineral fertilization recommended for corn; two, four, and eight times the N recommended dosage for corn crop. Calculations of sludge rates were performed as a function of the N available for plants and were calculated by the formula

$$\text{SS available N} = \left(\frac{\text{MF}}{100} \right) \times (\text{Kjeldahl-N} - \text{NH}_3\text{-N}) + \text{NH}_3\text{-N} + (\text{NO}_3^- \text{-N} + \text{NO}_2^- \text{-N}), \quad (1)$$

where Kjeldahl-N: total nitrogen (mg kg⁻¹), NH₃-N: nitrogen as ammonia (mg kg⁻¹), NO₃⁻-N: nitrogen as nitrate (mg kg⁻¹), NO₂⁻-N: nitrogen as nitrite (mg kg⁻¹), and MF: mineralization fraction (30%), for aerobically digested sludge.

Supplementary K was applied for treatments with sewage sludge, when necessary. The wet sludge was incorporated on April 1999 and on December 1999. It was toss-distributed in the total area of the experimental plots and incorporated to a depth of 20 cm with a rotary harrow, 3-4 days before sowing.

The P quantity in each SS rate was calculated based on the SS total P content added after two SS applications. Treatments applied were: FC = control without SS (for the plots with SS from Franca); BC = control without SS (for the plots with SS from Barueri); FP1, FP2, FP4, FP8 = 125, 250, 500, and 1000 kg ha⁻¹ of P from Franca-SS applications; BP2, BP4, BP8, and BP16 = 250, 500, 1000, and 2000 kg ha⁻¹ of P from Barueri-SS applications; FNPK and BNPK = mineral fertilized plots.

Soil samples were collected at 0–20 cm depth, after the second maize harvesting, air dried and passed through 2 mm sieve. For the P sorption essay, 3 g of soil samples of each SS- treated plot with 0, 125 (FP1), 250 (BP2), 500 (FP4), 1000 (FP8), and 2000 (BP16) kg ha⁻¹ of P were equilibrated in a 50 mL centrifuge tubes with 30 mL of 0.01 mol L⁻¹ CaCl₂ solution containing 0, 5, 10, 15, 30, 60, 100 and 250 mg L⁻¹ of P. Two drops of toluene were added to preserve solution from microorganism contamination. During six days, solutions were shaken for 30 min thrice a day. Finally, the tubes were centrifuged at 577 ×g for 15 min to separate supernatant from the solid material, and P was determined in the suspension [18]. The difference between the P in the equilibrium solution and P added is the quantity of P adsorbed by the soil. The adsorption isotherms, which represent the linear fit of the relationship between adsorbed P and the equilibrium solution P, were built with the results obtained with all P concentrations. Sorption strengths were calculated with the simple (monolayer) Langmuir equation, which gives an average sorption strength of the P sorbed [6],

$$Q = kQ_{\max} \frac{C}{(1 + kC)}, \quad (2)$$

where Q is the P sorbed (mg kg⁻¹), k is the sorption strength (L mg⁻¹ P), Q_{max} is the maximum quantity of P sorbed for monolayer P sorption (mg kg⁻¹), and C is the concentration of P in solution (mg L⁻¹).

The total P sorption maximum (Q_{max}: mg kg⁻¹) was calculated as the sum of P sorption maxima (Q_{max1} and Q_{max2}) derived from the two-surface Langmuir equation [19]. For the two-surface Langmuir equation,

$$Q = \left(k_1 Q_{\max 1} \frac{C}{[1 + k_1 C]} \right) + \left(k_2 Q_{\max 2} \frac{C}{[1 + k_2 C]} \right), \quad (3)$$

where Q is the P sorbed (mg kg⁻¹), k₁ and k₂ are the sorption strengths of the first and the second sorption layers (L mg⁻¹ P), Q_{max1} and Q_{max2} are the maximum quantities of P sorbed for two sorption layers (mg kg⁻¹), and C is the concentration of P in solution (mg L⁻¹).

The sequential fractionation was based on the method of Heddley et al. [8], modified by Sui [20], and also with the following modifications: 0.01 mol L⁻¹ CaCl₂ solution instead of H₂O, centrifugation at 1073 × g at 5°C, and filtering through Whatman 41 paper (20–25 μm) instead of 0.22 μm. Samples were selected only from Barueri SS treatments that added 0, 250 (BP2), 1000 (BP8), and 2000 (BP16) kg P ha⁻¹. An aliquot of 0.5 g of each sample was submitted to nitric-perchloric digestion to determine the total P (Pt) concentration by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). Another equal aliquot sample was transferred to 50 mL centrifuge tubes and submitted to P extraction using a sequence of P extracting solutions: 30 mL of 0.01 mol L⁻¹ CaCl₂ solution; 30 mL of 0.5 mol L⁻¹ NaHCO₃ (pH 8.2); 30 mL 0.1 mol L⁻¹ NaOH; 30 mL of 1.0 mol L⁻¹ HCl. Each soil-solution mixture was shaken for 16 hours, centrifuged at 3,000 g for 15 minutes, and filtered through Whatman 41 filter paper. The inorganic phosphorus (Pi) was determined by colorimetric method described by Murphy and Riley [18], and the organic phosphorus (Po) was determined by the difference between Pt and Pi in the same extract. After the last extracting solution, the soil residue remained in each centrifuge tube was submitted to nitric-perchloric digestion, and P content was determined by ICP-AES.

This procedure of sequential fractionation allowed identifying four types of P bindings to the solid phase: (a) labile or biologically available P, extracted by CaCl₂ solution and by NaHCO₃ solution; (b) chemisorbed or moderately labile Pi and Po (bound to Fe and Al oxides) extracted by NaOH solution; (c) P (bound to Ca) extracted by HCl solution; (d) residual P.

Langmuir monolayer equations were evaluated on the basis of statistical significance of the regression coefficients.

3. Results and Discussion

Sewage sludge addition from both cities (Franca and Barueri) decreased soil P sorption capacity in all rates under investigation (Figures 1(a) and 1(b)). Such behavior was expected since SS usually contains water soluble P [21], and the SS tested here presented more than 0.3% total P [22] and a C:P ratio less than 100 [23] which also favors the decrease in P sorption by releasing more P into soil solution as a result of organic P mineralization. Berton and Pratt [21] also used the isotherms to evaluate the effects of increasing rates of four different organic materials (including SS) on the P adsorption capacity of two soils and concluded that SS decreased P adsorption more effectively compared to the other materials.

The linear form of Langmuir regression equations presented a significant $R^2(P < 0.01)$ for all SS rates applied indicating that the sorption phenomena was adequately described by this isotherm (Table 3). P sorption maxima (Q_{\max}) ranged from 1130 to 1008 mg kg⁻¹ and the binding energy also varied from 0.148 to 0.072 L mg⁻¹ for control and treatment 2000 kg ha⁻¹, respectively (Table 3). Both Q_{\max} and the binding energy values decreased as a result of soil

SS incorporation, but this effect was linear only for the residue derived from Barueri. Although soil Q_{\max} usually presents a positive correlation with soil organic matter content [5], the addition of organic residues to soil, such as manures, causes an inverse effect by decreasing P sorption with increase in P availability to plants [24]. Mechanisms for the reduction of sorption maxima and the affinity constants by organic amendments include the competition of organic acids produced during mineralization for the same sites of P fixation and the complexing of exchangeable Al by organic acids [4, 25, 26]. Furthermore, both sewage sludges contain inorganic ligands such as SO₄²⁻ which can complex exchangeable Al and metals such as Ca and Mg which may form soluble complexes with P in the soil solution, thus preventing P from being fixed by Al or Fe [27].

Only the treatment with the highest P input (2000 kg ha⁻¹) reached the threshold value of 0.07 L mg⁻¹ proposed by McDowell et al. [1], whereby soils less than this value for binding strength are subject to enhance loss of P in subsurface flow. Since the recommended SS rate is eight times less than the highest rate tested (125 kg ha⁻¹ and 250 kg ha⁻¹ for Franca and Barueri cities, resp.), it indicates that the amount of SS recommended as N source for corn crop may be considered safe for potential P losses in subsurface flow, but it should be monitored after successive SS applications.

The amount of P (mg kg⁻¹) in equilibrium with 0.2 mg P L⁻¹ ($P_{0.2}$) calculated from each isotherm equation (Table 3) ranged from 32,49 to 14,31 mg kg⁻¹ for control and treatment 2000 kg ha⁻¹, respectively. The amount of P sorbed at $P_{0.2}$ in the equilibrium solution was decreased by increasing rates of SS from both cities and markedly reduced by the highest rates of these amendments. Sewage sludge from Franca required the double amount of added P to attain $P_{0.2}$ in equilibrium solution compared to the sludge from Barueri (500 to 250 kg ha⁻¹), suggesting that the latter was more efficient in maintaining P in soil solution.

The SS from Barueri city applied to soil changed the P distribution within the studied P fractions (Table 4). Phosphorus extracted by CaCl₂ was markedly increased only at P rate of 2000 kg ha⁻¹. The P rates applied as SS had little effect over P_o -NaHCO₃. In relative values, this P fraction decreased from 10.8 to 4.9% as long as P rates increased from 250 kg ha⁻¹ to 2000 kg ha⁻¹ (Table 5). This apparent little variation of P_o -NaHCO₃ fraction might probably be attributed to an intense organic P mineralization which could have contributed to increase P_i -NaHCO₃ fraction from 9.1% (control plot) to 18.1% (highest P rate-2000 kg ha⁻¹). Similar results were found by Sui et al. [20] who suggested that P_o -NaHCO₃ fraction might be responsible for an extra increase in P_i -NaHCO₃ fraction. Nevertheless, the P_o from the P_o -NaHCO₃ fraction might also be adsorbed to Al and Fe oxides, favoring the increasing P_o -NaOH fraction from 1.5 % to 11.3 % (Table 5).

Compared with any other P fractions, the contribution of P added as SS to the NaOH fraction was most evident and illustrates the P affinity to Fe and Al oxides, a relationship that predominates in oxisols. The increases of chemisorbed P observed on all SS treatments were likely the result of soluble inorganic P added with the organic residue and P

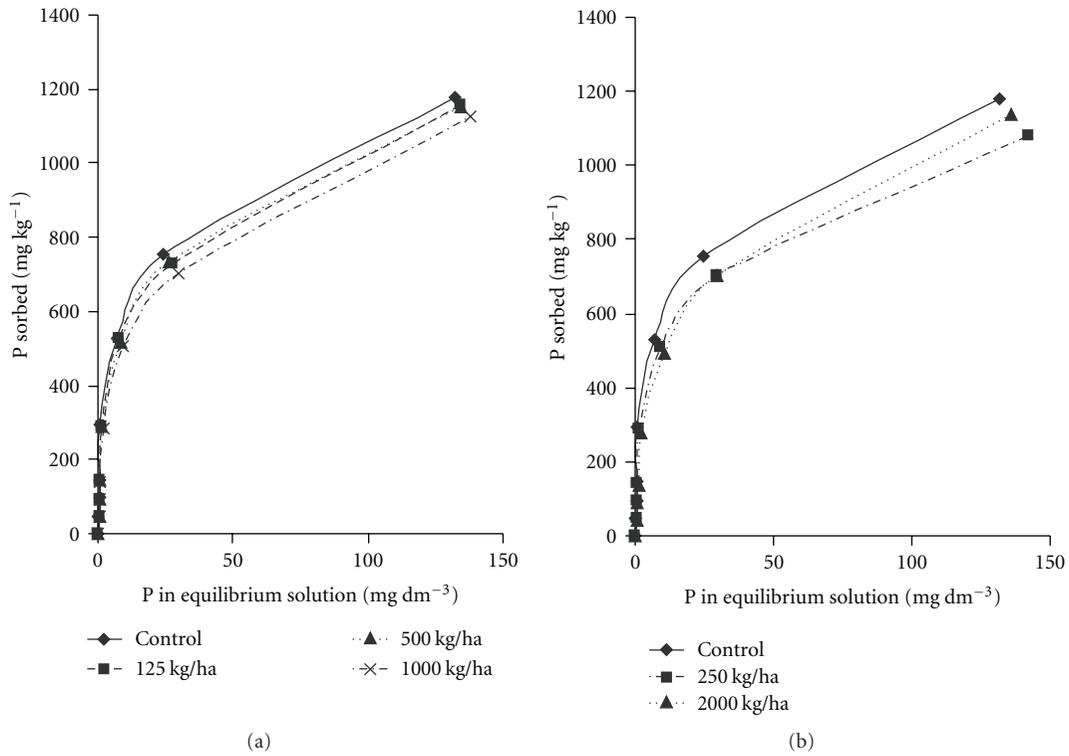


FIGURE 1: Phosphorus sorption in a Haplorthox treated with increasing amounts of P applied through biosolids addition from cities of Franca (a) and Barueri (b).

TABLE 3: Linear Langmuir equations and parameters in the soil samples which received phosphorus addition as biosolids.

Phosphorus inputs kg ha ⁻¹	Regression Equations ⁽¹⁾	R ²	Q _{max} ⁽²⁾ mg kg ⁻¹	Binding energy L ⁻¹ mg	P _{0.2} ⁽³⁾ mg kg ⁻¹
0	Y = 5.51 + 0.82X SS from Franca	0.99**	1130	0.148	32,5
125	Y = 7.08 + 0.82X	0.99**	1074	0.116	24.4
500	Y = 7,79 + 0.82X	0.99**	1086	0.105	22.3
1000	Y = 9.91 + 0.83X SS from Barueri	0.99**	1050	0.084	17.4
250	Y = 8.21 + 0.88X	0.99**	1082	0.107	22.7
2000	Y = 11.22 + 0.81X	0.99**	1008	0.072	14.3

⁽¹⁾For single-surface Langmuir equation. Y = C/Q, in g L⁻¹; X = equilibrium concentration, in mg L⁻¹.

⁽²⁾Sum of P sorption maximum of low-and high-energy sorbing sites derived from the two-surface Langmuir equation.

⁽³⁾P sorbed at equilibrium concentration of 0.2 mg L⁻¹.

**Significant at P < 0.01 by F-test.

Q_{max}: maximum P sorption capacity.

TABLE 4: Phosphorus fractions in a soil treated with increasing rates of biosolids.

Phosphorus rate kg ha ⁻¹	CaCl ₂	NaHCO ₃			NaOH			HCl	Residual	Total (sum)	Total
		P _o	P _i	P _t	P _o	P _i	P _t				
0	5	11	36	47	6f	32	38	15	289	394	343
250	6	51	47	98	14	54	68	36	260	468	367
1000	8	41	80	121	32	103	135	33	322	618	507
2000	14	44	165	209	103	170	273	39	378	913	787

P_i: inorganic phosphorus; P_o: organic phosphorus; P_t: total phosphorus.

TABLE 5: Average proportions of P fractions in soil samples amended with increasing amounts of biosolids.

P rate kg ha ⁻¹	CaCl ₂	NaHCO ₃		NaOH		HCl	Residual
		P _o	P _i	P _o	P _i		
0	1.3	2.8	9.1	1.5	8.1	3.8	73.4
250	1.3	10.9	10.0	3.0	11.5	7.7	55.6
1000	1.3	6.6	12.9	5.2	16.7	5.3	52.1
2000	1.5	4.8	18.1	11.3	18.6	4.3	41.4

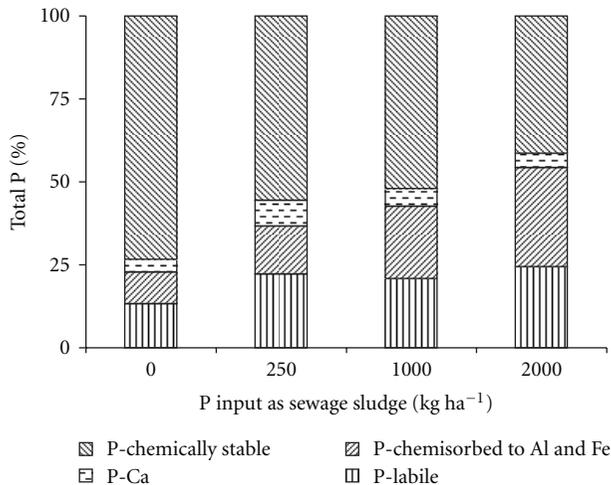


FIGURE 2: Percent distribution of P fractions in soil samples amended with increasing amounts of biosolids from Barueri city.

mineralized from added organic P [28]. Since the NaOH extractant removes a P fraction which is associated with amorphous and crystalline Al and Fe phosphates [29, 30], the addition of SS appears to have a significant impact on soil P sorption, as showed by the sorption isotherms (Figure 1(b)).

Addition of SS increased P-HCl fraction, but it was not proportional to the rates of SS applied. Novais and Smyth [31] suggested that the low variation of P-HCl fraction with the increasing P rates applied to soil evidenced that P bound to Ca did not have any important role in P dynamics of more weathered acid soils and not treated with natural phosphates as P source. In these soils, the poor calcium phosphate content was probably the main factor determining the HCl-extractable P quantity. In a similar way, Rheinheimer [32] observed in two oxisols that P extracted with 1.0 mol L⁻¹ HCl (after resin, 0.5 mol L⁻¹ NaHCO₃ and 0.1 mol L⁻¹ NaOH extractions) did not change with the successive extractions.

The effect of SS on soil P species distribution, increasing the element availability, can be observed by the variation occurred in three P fractions: (a) the labile fraction represented by P-CaCl₂ plus P-NaHCO₃ increased from 13.2% (control) to 24.4% of total P; (b) the chemisorbed P or moderately labile fraction suggested by Bowman and Cole [30], represented by P forms extracted by 0.1 mol L⁻¹ NaOH increased from 9.6 % (control) to 29.9 % of total P; consequently, (c) the most resistant residual P fraction decreased

from 73.4 % (control) to 41.4 % (Figure 2). The redistribution of residual P to more labile forms was also observed by Barcellos [33] on an eutroferic red latosol amended with successive applications of SS.

4. Conclusions

(1) The biosolids application to soil decreased soil P sorption which resulted in an increase of P in equilibrium solution.

(2) The positive correlation of P extracted by NaOH with total P added as SS suggested that the decrease in soil P sorption can be mainly attributed to the P chemisorbed to amorphous and crystalline Al and Fe.

(3) The addition of biosolids changed the P fractions distribution in the soil, favoring P availability to plants and its geoavailability by increasing the labile fractions (P-CaCl₂ and P-NaHCO₃) and the moderately labile fraction (P-NaOH) by 11.2 % and 20.3 %, respectively, in detriment of the most resistant P fraction.

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