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Soil fertility, growth and mineral nutrition in *Eucalyptus grandis* plantation fertilized with different kinds of sewage sludge

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Abstract The agricultural use of sewage sludge has increased worldwide, especially in crops and in countries with temperate or subtropical climate. Nevertheless, few studies have been conducted with forest plantations in tropical conditions. This study aimed to evaluate the soil fertility, mineral nutrition and tree growth in *Eucalyptus grandis* plantations fertilized with three types of sewage sludge. The design used was of blocks with randomized plots, four replications and five treatments: control without fertilization (C), conventional mineral fertilizer (MF), fertilization with 15 t ha^{-1} (dry basis) of sewage sludge produced in wastewater treatment plants of Barueri (BS), São Miguel (SS) and Parque Novo Mundo (PS) cities, all located in the metropolitan region of São Paulo, Brazil. It was observed that the application of sewage sludge increased the content of organic matter, nitrogen and phosphorus in the layer of 0–5 cm soil depth, as well as the nitrogen, phosphorus, copper and zinc concentrations in the *Eucalyptus* leaves. In the PS treatment, the sludge (conditioned with quicklime) application also increased the pH and the concentration of calcium in the soil compared to other kinds of sludge (conditioned with polyelectrolyte), resulting in higher Ca:Mg and Ca:K ratios in the leaves. Probably for this reason, wood production in PS was 20 % lower than in the MF treatment at 60 months of age of the trees. Even so, the fertilization with sewage sludge provided a rise of 50–90 % in timber volume compared to C treatment.

Keywords Biosolids · Wood production · Organic fertilization · Forest nutrition · Nutrient imbalance

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Introduction

In Brazil, the expansion of *Eucalyptus* plantation has taken place in areas where soil fertility is generally low (Gonçalves et al. 2013). Therefore, fertilization and crop residue management (e.g. bark and branches) have been adopted with favorable effects on productivity and sustainability of forest plantations (Gonçalves et al. 2004). Consequently, Brazilian and global forestry have become increasingly dependent on fertilizers to maintain or increase forest productivity, which has caused a significant rise in production costs (Laclau et al. 2013).

The production of sewage sludge has increased dramatically in recent years due to the growth of human population, the development of industry and agriculture, and the increased of demand for the adequate treatment of waste produced in large cities (Alleoni et al. 2012; Wang et al. 2012). Generally, sewage sludge contains high levels of organic matter and essential elements for plants, such as nitrogen, phosphorus, calcium, and several micronutrients (Torri and Lavado 2008; Bramryd 2013). The addition of organic matter to agricultural lands often improves the soil's chemical and physical properties and biological activity (Aguilera et al. 2007; Antonious et al. 2010).This is very important in tropical regions because the organic matter plays a fundamental role in soil fertility, since weathered soils of these regions have a low cation exchange capacity and low reserve of nutrients for the plants (Vieira et al. 2005).

The use of sewage sludge in forest land results in economic (Kimberley et al. 2004) and environmental benefits, due to the nutrient recycling from the urban environment to rural ecosystems (Bramryd 2002; Aarab et al. 2006; Bramryd 2013). This residue could replace the expensive inorganic fertilizers used in forest plantations (Ferreiro-Domínguez et al. 2014), particularly on marginal sites where nutrients may be limited (Quaye et al. 2011). In forest soils, the sewage sludge application can improve the water and nutrient holding capacity (Bramryd 2001; Prescott and Blevins 2005), reduce run-off and erosion processes (Ferreiro-Domínguez et al. 2014), increase microbial and N_2-fixing activity (Selivanovskaya et al. 2001), as well as releases nutrients relatively slowly, providing long-term benefits (Horswell et al. 2006).

The risk of sewage sludge application on agricultural land, due to the presence of heavy metal in its composition (Rigueiro-Rodrígues et al. 2012), has favored the use of this residue in forest land (Horswell et al. 2006), mainly because most forest products do not directly enter the human food chain (Denaix et al. 2011). The Brazilian Environmental National Council Resolution 375 establishes limits and criteria for agricultural use of sewage sludge (BRAZIL 2006). This regulation established the limits to heavy metals and N concentrations in sludge, and the N can be readily mineralized within the first year after application to the soil (Berton and Nogueira 2010).

The characteristics of sludge depend on the particularities of the communities where the sewage is collected, on the process and the degree of treatment, and on the sludge type generated in the process (Wang et al. 2008). Because of that, the chemical and physical characteristics of sludge are extremely variable and contrasting than mineral fertilizers, which are manufactured and have a defined and constant chemical composition for a particular nutritional use (Mitchell et al. 2000). Sludge conditioning for dehydration can be carried out with the addition of lime, ferric chloride, or with the addition of polymers (polyelectrolytes), each one resulting in sludges with distinct physicochemical characteristics (Sobrinho 2000). Sewage sludge conditioning with lime and ferric chloride, due to its simplicity and low cost, has been chosen by most sewage treatment plants in Brazil (Paiva et al. 2009). Nevertheless, some wastewater treatment plants (WTPs) of the Basic Sanitation Company of São Paulo State (SABESP) have opted for the use of polymers in the sludge conditioning (Silva et al. 2011).

We hypothesize that independent of the kind of sewage sludge, either because of its origin (e.g. urban or industrial) or due to the conditioning type (e.g. with lime or polymer), it can be used in forest plantation to improve the organic matter in the soil and the nutritional status of plants, increasing the tree growth. However, the diverse chemical composition of sludge in the space and time can result in different degrees of tree responses. Due to it and to the scarce studies about sludge as soil amendment under tropical condition, the aim of this study was to evaluate soil fertility, mineral nutrition, and growth of *Eucalyptus grandis* trees, when fertilized with three types of sewage sludge produced in different wastewater treatment plants in the metropolitan region of São Paulo–Brazil.

Materials and methods

Study area

The study was carried out in the Experimental Station of Forest Sciences of Itatinga (EECFI) belonging to the University of São Paulo–Brazil (23°02′S, 48°38′W). According to Battie-Laclau et al. (2014), the mean annual precipitation over last the 15 years was 1360 mm, with a dry season from June to September. The mean annual temperature was 20 °C, with mean monthly temperatures ranging from 15 °C (July–September) to 25 °C (October–March).

The experiment was installed on a flat and gently undulating terrain with an altitude of 863 m above sea level. The soil was classified as Typic Hapludox (USDA 2014) (redyellow Latosol—Brazilian Classification System), with a clay content ranging from 15 to 18 % in the layer of 0–40 cm depth. In this layer, the soil is acidic (pH ≈ 3.8) and showed a high aluminum content (12 $mmol_c\ dm^{-3}$), medium inorganic matter (30 $g\ dm^{-3}$), low phosphorus content (2 $mg\ dm^{-3}$), reduced exchangeable bases (K + Ca + Mg = 2.2 to 3.7 $mmol_c\ dm^{-3}$) and low-medium content of micronutrients (0.1 of B, 2.3 of Cu, 153.0 of Fe, 1.0 of Mn and 0.2 of Zn, all in $mg\ dm^{-3}$). These conditions are typical for most *Eucalyptus* plantations in the São Paulo State–Brazil.

Experimental design

The study was installed as a completely randomized block design, with five treatments and four replications. The treatments were: (1) control, without fertilization (C); (2) mineral fertilization (MF), based on the commercial companies recommendation (Gonçalves et al. 2013); (3), (4) and (5) fertilization with 15 $t\ ha^{-1}$ (dry basis) of sewage sludge produced by WTPs from Barueri (BS), São Miguel (SS) and Parque Novo Mundo (PS) cities, respectively.

Although from the same metropolitan area of São Paulo–Brazil, the sewage sludge produced in Barueri and São Miguel were conditioned with polyelectrolyte, while the sludge from Parque Novo Mundo was conditioned with CaO and $FeCl_3$; thus, the different kinds of sludge were chemically distinct from each other (Table 1). It is noteworthy that a

Table 1 Physical and chemical characteristics of the sewage sludge produced in the wastewater treatment plants from three different cities (Parque Novo Mundo, São Miguel and Barueri) of the metropolitan region of São Paulo

Characteristics[a]	Unit	Wastewater treatment plant		
		Parque N. Mundo	São Miguel	Barueri
pH (in $CaCl_2$)	–	8.2	7.6	7.7
Bulk density	g cm^{-3}	0.69	1.08	1.08
Water content	%	67.2	71.3	81.6
Organic matter	g kg^{-1}	506.5	538.4	557.8
Organic carbon	g kg^{-1}	275.7	182.4	302.6
Total nitrogen (N)	g kg^{-1}	17.1	16.4	36.9
Phosphorus (P_2O_5)	g kg^{-1}	28.9	24.8	44.5
Potassium (K_2O)	g kg^{-1}	1.2	1.4	2.2
Calcium (Ca)	g kg^{-1}	112.4	108.4	27.7
Magnesium (Mg)	g kg^{-1}	3.4	1.7	4.3
Sulfur (S) Total	g kg^{-1}	6.1	6.6	6.5
C/N (C organic e N total)	–	16/1	11/1	8/1
Copper (Cu)[b]	mg kg^{-1}	457	66	858
Manganese (Mn)[b]	mg kg^{-1}	289	328	369
Zinc (Zn)[b]	mg kg^{-1}	1130	429	3026
Iron (Fe)[b]	mg kg^{-1}	82,574	34,878	42,401
Boron (B)	mg kg^{-1}	6	3	16
Sodium (Na)[b]	mg kg^{-1}	880	2329	1108
Arsenic (As)[b]	mg kg^{-1}	11	16	nd[c]
Cadmium (Cd)[b]	mg kg^{-1}	5.0	2.0	7.06
Lead (Pb)[b]	mg kg^{-1}	76	36	220
Chrome (Cr)[b]	mg kg^{-1}	526	49	497
Mercury (Hg)[b]	mg kg^{-1}	1.00	3.00	0.18
Molybdenum (Mo)[b]	mg kg^{-1}	20	15	nd[c]
Nickel (Ni)[b]	mg kg^{-1}	141	241	389
Selenium (Se)[b]	mg kg^{-1}	1	1	nd[c]

[a] Except for the water content, all the analyses refer to dry matter

[b] Method used for heavy metals: SW 3051, EPA-U.S., as determined by ICP-AES

[c] Not determined

significant concentration of calcium in the sludge produced by the WTP of São Miguel was due to the dumping of industrial wastes, calcium sulphate-based, into the domestic sewage network, while in the WTP of Parque Novo Mundo, the higher concentration of calcium in sludge produced by it was due to its conditioning with quicklime.

In addition to sewage sludge (BS, SS, and PS treatments), 100 kg ha^{-1} of NPK fertilizer (6:30:6) and 80 kg ha^{-1} of *fritted trace elements* (FTE) Br 12 (9 % Zn + 1.8 % B + 0.8 % Cu + 2.0 % Mn + 3.5 % Fe + 0.1 % Mo) were applied at planting, into two side holes close to the seedling. This additional fertilization (reduced quantities) was applied to guarantee the initial survival of the seedlings, because in the first moment most of the nutrients in sludge would be in organic form and, consequently, not available to plants. In these same treatments, we applied 200 kg ha^{-1} of KCl split at 3 and 6 months after planting, spread over the crown projection. The *Eucalyptus* is very potassium dependent (Laclau et al. 2009) and the sewage sludge is normally poor in this element (Wang et al. 2008). The sludge application rate and method (strip of 50 cm on the planting row) was done according to previous studies (Guedes et al. 2006) and criteria established by the standard P 4,230 (CETESB 1999). The sludge was applied 1 month before the seedlings planting in order to minimize possible damages to plants due to ammonia volatilization.

The MF treatment (mineral fertilization) consisted of: 1.5 t ha^{-1} of dolomitic limestone (Effective calcium carbonate = 85 %, CaO = 37 %, MgO = 16 %), applied before planting and spread in all areas of the respective plots; 200 kg ha^{-1} of NPK (6:30:6) and 80 kg ha^{-1} of FTE BR 12, applied at planting into two side holes close to the seedling; and 600 kg ha^{-1} of NPK (16:6:24) split at 3, 6 and 9 months post-planting, spread over the crown projection.

The experiment started in May 2005, with the planting of 3-month-old seedlings produced in tubes of 110 cm^3 by the nursery of *SuzanoPapel e Celulose* Company, from seeds of a single progeny of *Eucalyptus grandis*. The soil was prepared by subsoiling to a depth of 40 cm and the planting spacing was 3 m between lines and 2 m between plants. Each plot had a gross area of 600 m^2 (100 trees) which included an inner plot of 216 m^2 (36 trees) and two border rows (64 trees).

Mineral nutrition of the trees

Nutritional assessment of the eucalypts was performed at 6, 18, and 42 months after planting. Leaves were collected from branches located in the upper third part of the crown of 8 trees per inner plot, at positions corresponding to the four cardinal points. The leaves were combined into a composite sample per plot and subsequently dried in a forced air circulation oven at 60–65 °C, until constant weight. After drying, the samples were ground in a Wiley mill and chemically analyzed: the total-N content was determined by micro Kjeldahl method, after sulfuric acid digestion; the P was determined by colorimetry; the S by turbidimetry, the K by flame photometry and the Ca, Mg, Fe, Cu, Mn and Zn by atomic absorption spectrophotometry, all after nitropercloric digestion (Malavolta et al. 1989).

Tree growth measurements

To estimate the growth and the volume of wood, height and circumference at breast height (CBH) of all trees in the inner plots were measured at 24, 30, 36, 42, 48 and 60 months after planting. Height was measured using an electronic hypsometer, Vertex III model, with an accuracy of 0.1 m. The CBH was measured with tape, with an accuracy of 0.1 cm. To determine the volume (Vol) of individual trees we used the hypsometric equation $Vol = 1.7 \times 10^{-5} \times (CBH/\pi)^{1.9117} \times H^{1.3065}$ adjusted by Guedes (2005).

Sampling and soil analysis

Soil sampling was performed 36 months after the installation of the experiment. Samplings were collected using stainless steel Dutch auger (25 mm of diameter) at depths of 0–5, 5–10, 10–20 and 20–40 cm in the corresponding strip where the sewage sludge and other fertilizers had been applied. In each plot, four single soil samples were collected, and then blended into one composite sample per plot.

The soil samples were dried in a forced-air oven at 40–45 °C until constant weight. After drying, samples were sieved (2 mm mesh), mixed and sent to the ESALQ/USP Soil Sciences Laboratory for chemical analysis. The pH was determined in $CaCl_2$ and total-N quantify by the micro-Kjedhal method; the available phosphorus (P_{resin}) and exchangeable K, Ca, Mg, Cu, Fe, Mn, Zn were displaced by ion-exchange resins; sulfate ($SO_4{}^{2-}$) by turbidimetry; organic matter (OM) by colorimetric determination; aluminum (Al^{3+}) by titration and the content of H + Al in the buffer SMP method (Raij et al. 2001).

Other soil properties were determined indirectly: the sum of bases (SB) by the equation ($K + Ca^{2+} + Mg^{2+}$); the cation exchange capacity (CEC) by the sum [SB + (H + Al)]; base saturation (V%) by [(SB/CTC)*100] and saturation by aluminum (m%) using [Al/(SB + Al)*100].

Statistical analyses

Statistical basic assumptions (normality and homoscedasticity) and the data's need of transformation were verified. Then, the data were subjected to analysis of variance (ANOVA) and treatments were compared by the Tukey test ($P > 0.05$). Statistical analyses were performed using the Statistical Analysis System (SAS) 9.1 software (SAS Institute 2002–2003).

Results

Even 36 months after planting, the PS sludge treatment increased soil pH ($\approx$2 units) and the concentration of calcium (up to 250 times) more than other treatments, particularly in the layer 0–5 cm depth (Table 2). Additionally, the concentration of aluminum (Al^{3+}), ($Al^{3+} + H^+$) and aluminum saturation (m%) were significantly lower in the PS treatment. Interesting result was observed in the SS treatment, where the sewage sludge application also increased (up to 80 times) the calcium concentration in the soil, although the sludge was conditioned with polyelectrolyte.

In the 0–5 cm layer, the soil organic matter (OM) and total nitrogen (total N) concentration were about 2–4 times higher in SS and PS treatments than in the control and mineral fertilization. In the same layer, the sewage sludge application increased (up to 75 times) the resin phosphorus concentration (P_{resin}), but only in BS and PS treatments was this effect noted in the other layers (5–10 and 10–20 cm). On the other hand, the concentration of sulfur ($S\text{-}SO_4{}^{2-}$) increased from surface to deep layers of the soil, mainly in the BS and SS treatments.

Unlike of most results, the concentration of magnesium (Mg^{2+}) in all soil layers was 2–9 times higher in MF than other treatments. About the micronutrients, the sewage sludge application increased the concentration of copper (2–4 times) and zinc (up to 100 times) in the 0–5 cm soil layer, especially in BS treatment. In deeper soil layers (20–40 cm), these effects of sewage sludge were smaller, but still significant (up to 10 times). However, the manganese concentration in soil was lower (3–5 times) in the SS when compared to MF treatment.

At 6 months after planting, the N and P concentrations in leaves were significantly higher (20–100 %) in the sludge treatments than in C and MF treatments (Table 3). Over time (18 and 42 months), only the phosphorus (P) concentration was higher (20–45 %) in the sewage sludge treatments. Until the 18th month, foliar concentrations of sulfur (S) were

Table 2 Chemical characteristics of soil in *Eucalyptus grandis* stands without fertilization (C), with mineral fertilization (MF) and fertilized with sewage sludge produced in wastewater treatment plants from Barueri (BS), São Miguel (SS) and Parque Novo Mundo (PS) cities—São Paulo/Brazil

Treatment	pH $CaCl_2$	OM g dm^{-3}	P_{resin} mg dm^{-3}	S-SO_4^{2-} mg dm^{-3}	Total N mg kg^{-1}	K^+ $mmol_c$ dm^{-3}	Ca^{2+} $mmol_c$ dm^{-3}	Mg^{2+} $mmol_c$ dm^{-3}	Al^{3+} $mmol_c$ dm^{-3}	$Al^{3+} + H^+$ $mmol_c$ dm^{-3}	Cu mg dm^{-3}	Fe mg dm^{-3}	Mn mg dm^{-3}	Zn mg dm^{-3}
Layer at 0–5 cm														
C	3.5c[a]	29c	2b	6bc	756c	0.4b	1d	1c	14a	96a	4.6c	117b	0.5b	0.3b
MF	4.1bc	37bc	2b	5c	972c	0.4b	10c	9a	8b	62ab	6.3bc	124b	3.8a	0.6b
BS	3.7c	53ab	127a	9ab	1464bc	0.5ab	11c	3ab	11ab	103a	20.0ab	277a	3.4a	61.2a
SS	4.3b	67a	102a	13a	2341ab	0.5b	80b	1c	15a	87a	3.5c	327a	0.8b	30.3a
PS	6.4a	69a	223a	8abc	2999 a	0.7a	249a	3b	0c	27b	22.1a	99b	3.3a	56.3a
Layer at 5–10 cm														
C	3.6bc	23a	1c	6b	na	0.2bc	1c	1b	11c	84ab	0.7c	74b	0.3b	0.2c
MF	3.7b	28a	2bc	5b	na	0.3a	2bc	4a	11c	88ab	1.1bc	94ab	0.7a	0.4c
BS	3.4c	30a	5a	16a	na	0.3ab	2c	1b	16b	119a	2.1ab	166a	0.4b	4.9a
SS	3.7b	32a	2c	28a	na	0.2c	6b	1b	21a	124a	0.6c	116ab	0.2b	2.6a
PS	4.3a	33a	4ab	7b	na	0.3a	27a	1b	6d	64b	2.8a	82 ab	0.5ab	1.7b
Layer at 10–20 cm														
C	3.6ab	21a	1b	9c	na	0.3a	1c	1b	12bc	81ab	0.8ab	78a	0.3ab	0.2c
MF	3.7a	25a	2ab	4d	na	0.2a	1c	2a	10bc	86ab	1.0ab	79a	0.6a	0.3c
BS	3.5b	26a	3a	22ab	na	0.2a	1c	1b	15ab	105a	1.8ab	124a	0.3ab	3.0a
SS	3.6ab	26a	1b	43a	na	0.2a	3b	1b	20a	108a	0.8b	104a	0.2b	1.8ab
PS	3.8a	27a	5a	11bc	na	0.2a	13a	1b	7 c	63b	1.9a	80a	0.3ab	0.8b
Layer at 20–40 cm														
C	3.7ab	18b	1a	7b	600a	0.1a	1c	1b	10bc	67ab	0.8b	58a	0.2ab	0.1b
MF	3.7ab	22ab	1a	7b	697a	0.2a	1bc	2a	10bc	76ab	1.0ab	62a	0.4a	0.2b
BS	3.6b	22a	2a	47a	684a	0.1a	1c	1b	13ab	90ab	1.6a	80a	0.3ab	1.7a

Table 2 continued

Treatment	pH $CaCl_2$	OM g dm^{-3}	P_{resin} mg dm^{-3}	$S\text{-}SO_4^{2-}$ mg dm^{-3}	Total N mg kg^{-1}	K^+ $mmol_c$ dm^{-3}	Ca^{2+} $mmol_c$ dm^{-3}	Mg^{2+} $mmol_c$ dm^{-3}	Al^{3+} $mmol_c$ dm^{-3}	$Al^{3+} + H^+$ $mmol_c$ dm^{-3}	Cu mg dm^{-3}	Fe mg dm^{-3}	Mn mg dm^{-3}	Zn mg dm^{-3}
SS	3.7ab	24a	2a	101a	731a	0.1a	4ab	1b	17a	98a	0.9ab	75a	0.1b	1.4a
PS	3.9a	23a	1a	14b	815a	0.2a	9a	1b	7c	54b	1.4ab	62a	0.2ab	0.5a

[a] For each layer and chemical characteristics of soil (column), means followed by same lower letter not differ by Tukey's test ($P > 0.05$)

Table 3 Concentration and relationships of nutrients in the leaves of *Eucalyptus grandis* stands without fertilization (C), with mineral fertilization (MF) and fertilized with sewage sludges from waste water treatment plants of Barueri (BS), São Miguel (SS) and Parque Novo Mundo (PS)—São Paulo State/Brazil

Treatment	N $g\ kg^{-1}$	P $g\ kg^{-1}$	K $g\ kg^{-1}$	Ca $g\ kg^{-1}$	Mg $g\ kg^{-1}$	S $g\ kg^{-1}$	Cu $mg\ kg^{-1}$	Fe $mg\ kg^{-1}$	Mn $mg\ kg^{-1}$	Zn $mg\ kg^{-1}$	Ca:Mg unit	Ca:K unit	N:P unit	K:P unit
(6) Six months														
C	23.6b[a]	0.8c	6.0a	3.9d	1.8c	0.9c	15b	82b	788ab	20b	2.1c	0.6d	30.3a	7.7a
MF	23.1b	1.0c	6.1a	5.1c	3.1a	1.0bc	13b	90b	678ab	20b	1.7d	0.8c	24.1b	6.4a
BS	34.9a	1.6a	5.4a	6.1b	2.3b	1.6ab	25a	110a	804ab	31a	2.6b	1.1bc	22.3b	3.5b
SS	33.2a	1.5a	4.6a	5.8bc	2.1bc	1.7a	23a	110a	866a	29a	2.8b	1.3ab	21.8b	3.0b
PS	31.9a	1.3b	5.4a	9.5a	2.3bc	1.2abc	29a	102a	625b	29a	4.2a	1.7a	25.3b	4.4b
(18) Eighteen months														
C	20.4a	1.1b	3.7a	5.0b	2.2b	0.6b	15a	84b	707a	27b	2.2c	1.4c	18.6a	3.3a
MF	21.2a	1.1b	3.3ab	4.7b	3.2a	0.4c	8c	89ab	287c	20c	1.5d	1.4bc	18.9a	2.9ab
BS	20.5a	1.2ab	2.9b	5.5b	2.2bc	0.4c	10bc	111ab	459b	34a	2.5c	1.9ab	16.8ab	2.4b
SS	21.8a	1.6a	3.4ab	6.7a	2.1bc	0.8a	16a	115a	662a	34a	3.3b	2.0a	14.1b	2.2b
PS	21.9a	1.2ab	3.3ab	7.8a	2.0c	0.6b	14ab	106ab	444b	30ab	3.9a	2.4a	17.8a	2.7ab
(42) Forty-two months														
C	18.2ab	1.0bc	3.3a	2.6d	1.3b	0.7a	10a	71ab	213b	12bc	2.0c	0.8d	19.1a	3.4a
MF	16.2b	0.9c	2.2b	3.2cd	2.5a	0.6a	8b	73ab	233ab	10c	1.3d	1.5bc	17.3b	2.3b
BS	17.7ab	1.1ab	2.8ab	3.5bc	1.6b	0.6a	10a	88a	341a	16ab	2.2c	1.3c	16.1b	2.5b
SS	16.5b	0.9bc	2.4b	4.3b	1.2b	0.7a	9ab	87ab	340a	12bc	3.5b	1.8b	17.7ab	2.5b
PS	19.0a	1.1a	2.7ab	6.8a	1.5b	0.6a	11a	63b	276ab	18a	4.5a	2.6a	17.2b	2.4b

[a] For each sample date and nutrient (column), means followed by same lower letter do not differ by Tukey's test ($P > 0.05$)

higher (30–70 %) only in the SS treatment, when compared to the control as well as to the other sewage sludge treatments. In this same period, foliar concentration of potassium (K) in the C treatment was higher (30–40 %) than MF and some sludge treatments.

On all sample dates, a higher foliar concentration of calcium (up to 140 %) and magnesium (up to 80 %) was detected in PS and MF treatment, respectively. Because of that, the foliar Ca:Mg and Ca:K ratios were significantly higher in PS treatment (20–125 %) and lowest in MF treatment (20–60 %), both compared to the other treatments. Similar results were observed in SS treatment, although the effects were less pronounced than PS treatment. Nevertheless, the N:P and K:P ratios in the leaves were 10–30 and 40–100 % higher, respectively, in the control compared to other treatments.

Foliar concentration of copper, iron and zinc were also significantly higher (25–80 %) in the sewage sludge treatments at 6 months after planting; however, at 18 and 42 months, these effects were limited only to zinc, mainly in BS treatment. On the other hand, the foliar concentration of manganese (at 6 and 18 months) was approximately 30 % lower in the SS treatment than in MF treatment.

On all sample dates, sewage sludge (BS, SS and PS) treatments and the mineral fertilizer (MF) treatment stimulated tree growth, which had a CBH 15–50 % and a height up to 4.5 m higher than trees in the control treatment (C) (Fig. 1). In relation to MF treatment, the tree growth was up to 10 % higher in BS and 10 % lower in PS treatment.

The volume of wood in the BS treatment was 20–75 % more than in the control and PS treatments, but was similar to the MF and SS treatment (Fig. 1).Consequently, the mean annual increment (MAI) was significantly lower in the control compared to the sewage sludge and mineral fertilizer treatments (Fig. 1); however, it is worth noting that these differences reduced over time (400–50 %).

Discussion

In our study, the sewage sludge increased the foliar concentration of nitrogen (N) and phosphorus (P), but these effects were more or less pronounced according to the type of sludge and the development stage of eucalypts plantation. Generally, sewage sludge is a heterogeneous mixture of different organic and inorganic fractions resulting from the wastewater as well as sewage treatment process (Bergkvist et al. 2003). Due to its heterogeneity, decomposition of sewage sludge may vary considerably in time and space (Fernández et al. 2007), which may affect the rate of mineralization and availability of the nutrients.

The N concentrations in *Eucalyptus grandis* leaves are often related to the expansion of the crown, with the photosynthetic rate of plants and, consequently, with the growth rate of trees (Kriedemann and Cromer 1996). These were evident in our study; because the N concentration in leaves and the tree growth rate were improved by sewage sludge application, mainly in initial phase of eucalypts development. According to Gonçalves et al. (2004), the higher initial growth of trees fertilized with sewage sludge or mineral fertilizer, apart from enabling significant gains in timber volume, increased the trees' ability to obtain water and nutrients when in competition with invasive plants, and promoted the rapid ground cover and its protection against erosion.

Normally, tropical soils are very weathered and the phosphorus is quickly fixed to the iron and aluminum oxides (Fontes and Alleoni 2006). In our study, the sewage sludge treatments increased the available phosphorous (P-Sol) in the soil, even after 36 months of

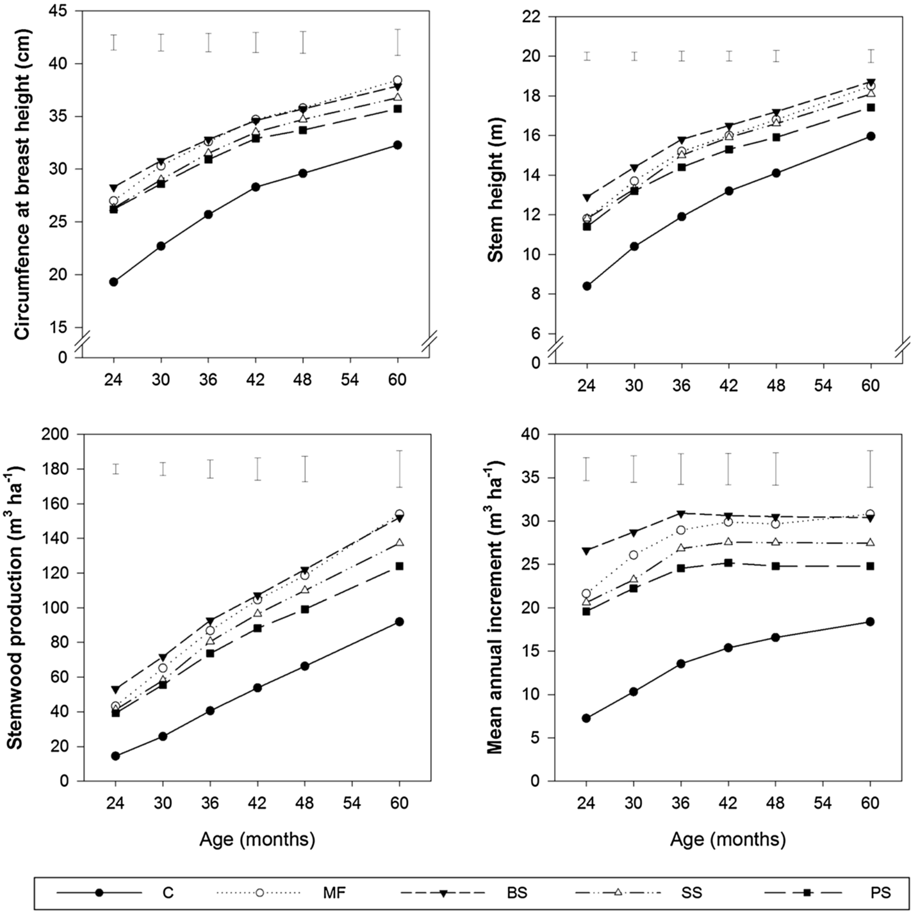

Fig. 1 Circumference at breast height, stem height, stem wood production and mean annual increment in *Eucalyptus grandis* plantation, in the treatments: control (C); mineral fertilization (MF), sewage sludges from Barueri (BS), São Miguel (SS) and Parque Novo Mundo (PS), all wastewater stations of São Paulo–Brazil. For each age, the *vertical bars* indicate the least significant difference (LSD) by the Tukey test ($P > 0.05$)

application. This result can be related to the slow P organic mineralization contained in the sludge (Egiarte et al. 2005), avoiding the nutrients leaching and favoring the P uptake by the plants over time. In Brazil, the phosphorous is also the main limiting nutritional factor in the development of *Eucalyptus* cultivation (Costa et al. 2016), and its demands is greater in the early growth stages (Melo et al. 2016). This was verified in our study; due the higher foliar concentration of phosphorus at 6 months after planting caused by the use of sewage sludge, which supported the fast initial growth of eucalyptus (Fig. 1).

According to Berton and Nogueira (2010), stabilized sludge with hydrated lime tends to increase the pH and calcium content in the soil, which can cause potassium and magnesium leaching to the sub-surface layers. This increase of the soil pH may have reduced the solubility of aluminum (Al^{3+}), causing its precipitation in the form of $Al(OH)^{2+}$ (Meriño-Gergichevich et al. 2010), leading the potential acidity to relatively low levels. These effects were observed in the PS treatment (conditioned with lime), which may have contributed to root system development (Ferraz and Poggiani 2014) rather than aerial growth of trees. Similar results were verified in SS treatment (conditioned with polyelectrolyte), but the intensity was lower than PS treatment. Despite of the high amount of calcium applied ($\approx$1650 kg ha^{-1}) in both treatments (PS and SS); the calcium source in the two kinds of sludge was a determining factor in the availability of this element in the soil. As described previously, the PS sludge was conditioned with quicklime (CaO) and the SS sludge was enriched with calcium sulfate ($CaSO_4$), coming from industrial effluents. In contact with water, the CaO becomes a strong base [$Ca(OH)_2$] which quickly reacts with the soil (Havlin et al. 2005); in turn, the $CaSO_4$ is a salt that has low solubility in water (2.5 g L^{-1}), but may act by increasing the ionic strength of the soil, so there is a continuous release of salt ions into the solution for a long period of time (Sousa et al. 2007).

The foliar concentration of calcium was very high in PS treatment, which may have brought nutritional imbalance to the plants. In our study, the ratios of Ca:Mg and Ca:K in leaves were higher in PS and lower in MF treatment. Silveira et al. (2005), evaluating *E. grandis* plantations fertilized with lime mud doses, found that the productivity at 2 years of age was inversely proportional to the leaves Ca:Mg ratio (2.5–5.0). Perhaps, for this reason, the tree growth in the PS treatment was significantly lower than MF treatment (Fig. 1). Moreover, we observed K-deficiency symptoms (e.g. marginal necrosis) in the *Eucalyptus* leaves of the PS treatment, mainly in dry season (between June and September). It is known that K uptake by plants is influenced by antagonistic interaction with calcium (Havlin et al. 2005) and the excess of this element can induce K deficiency in plants.

Compared to the control, the mean annual increment (MAI) in sewage sludge and mineral fertilization treatments were bigger, but these MAIs stagnated at 36 months after planting (Fig. 1). Probably, the faster growth of trees in MF and sewage sludge treatments stimulated other forms of intraspecific competition (e.g. for space, light and water) (Binkley et al. 2013), which may have caused this MAI stagnation before in the C treatment. Moreover, the canopy closure (at 36 months after planting) culminates in a greater participation of nutrient cycling in the trees nutrition and growth (Laclau et al. 2010), making them less dependent on the soil fertility. In our study, the MF treatment increased the magnesium concentration in soil and plant, offering better conditions to tree growth than other treatments. Silva et al. (2011) studying nutrient cycling in *E. grandis* plantations fertilized with wet and dry sewage sludge, found similar results, and they concluded that this was due to addition of dolomitic limestone applied only in mineral fertilizer treatment.

The occasional application of sewage sludge in plantations, for example, every beginning of the crop cycle (which can range from 5 to 100 years), obviously has little impact on the total amount of heavy metal in soil and on plants; but the addition of metal via sludge application may produce positive effects on the stock of micronutrients in the soil, offsetting the amounts of these elements exported with harvest timber (Bramryd 2013). In our study, the sewage sludge treatments increased soil concentration of copper and zinc, as well as the foliar concentration in eucalypts leaves. These results may be related to the amount of Cu and Zn added to the soil (Martins et al. 2003) and probably to the decomposition rate of organic matter and the mineralization of these nutrients contained in each type of sewage sludge. Bramryd (2001) studied the effect of the dry and wet sewage sludge application in a *Pinus sylvestris* L. forest and found in the organic soil layer about 140 mg kg^{-1} of Zn, corresponding to a concentration 3 times higher than the control treatment. Although zinc is considered an essential element for plants and animals, it can become toxic when in high concentration (Ngole and Ekosse 2009). However, in this work we don't observed symptoms of zinc toxicity, such as chlorosis or red pigmentation in the petiole and in the leaf veins (Dechen and Nachtigall 2006). The soil concentration of zinc was bigger in sewage sludge treatments and this effect decreased from superficial to deeper layers (0–40 cm). According to Schwab et al. (2008), Zn may have high mobility in the soil profile, especially after the application of sewage sludge, due to its complexation by soluble organic substances such as citric, malic and tartaric acids. Nevertheless, the tree roots reach deeper layers of soil, take up the heavy metals and reduce the risk of leaching (Rigueiro-Rodríguez et al. 2008).

According to the criteria established by Knecht and Göransson (2004), all treatments in our study provided N:P and K:P ratios up to the common values (10:1 and 1.5:1, respectively) for most deciduous tree species. However, these authors emphasize that the N:P and K: P ratios may vary with the fertilizer practices over time; therefore, the different characteristics of each forest ecosystem should be considered. Despite of the differences among the types of sewage sludge, our results show the benefits that the nutrient recycling could bring to *Eucalyptus* plantations, permitting the partial or even the total replacement of mineral fertilization by sewage sludge.

Conclusions

The application of different types of sewage sludge improved soil fertility, especially in the 0–5 cm depth of layer of soils poor in nutrients. Regardless of its genesis and conditioning, sewage sludge increased the content of organic matter, phosphorus, nitrogen, sulfur, copper and zinc in the soil. However, the fertilizer treated with quicklime sludge also increased the pH and calcium concentration, and reduced aluminum in the soil.

In general, the fertilization of *Eucalyptus grandis* plantations with the three types of sewage sludge increased the nutrients concentration in the leaves, especially in the early stages of plant development. The use of sludge enriched with calcium (Ca), either due to its conditioning with lime or due to its industrial effluents, favors the absorption and increases Ca leaf concentration and can induce deficiency of potassium or magnesium; in those leaves that present high Ca:Mg and Ca:K ratios.

Fertilization of *E. grandis* plantations with sewage sludge provided a satisfactory tree growth compared to crops without fertilizer; however, only the use of sludge conditioned with polyelectrolyte yielded a similar productivity (m^3 of wood/ha/year) to mineral fertilization. The agricultural use of sewage sludge in forest stands seems promising because it reconciles the recycling of nutrients with good timber production levels. However, the heterogeneity of sludge generated among wastewater treatment plants may lead nutritional imbalances (e.g. excess of calcium inducing potassium deficiency), that preclude an adequate response of trees to fertilization with sewage sludge. Therefore, the combination of sewage sludge with other sources of nutrients should be studied in order to achieve a nutritional balance and adequate productivity in forest stands.

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