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Research Article

Nutrient Availability and Changes on Chemical Attributes of a Paleudult Soil Amended with Liquid Sewage Sludge and Cropped with Surinam Grass

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The liquid sewage sludge (LSS) was applied on a field experiment during four years at successive applications to evaluate the changes in soil attributes and on Surinam grass (*Brachiaria decumbens*) uptake of nutrients. A randomized blocks experimental design, with two treatments (with and without LSS) and three repetitions, was used. Land application of LSS did not alter soil organic matter and exchangeable K until 40 cm depth. However, it increased soil pH, base saturation, labile P, and available Zn and did not change the concentrations of available B (hot water) and Cu, Fe, and Mn (DTPA) at 0–20 cm and 20–40 cm depths and LSS was a source of N, K, P, Ca, Mg, and Zn for the grass, but decreased leaf Mn concentration.

1. Introduction

In the initial steps of water pollution control process there is a great concern about the liquid phase treatment of sewage originated from districts and cities, in order to protect water resources and public health [1]. The treatment of this wastewater generates a solid residue known as biosolid or sewage sludge (SS), which consists mainly of a mass of fungi, bacteria, and protozoa that are responsible for the sewage organic matter degradation. Due to its intrinsic high organic matter content, followed by a variable concentration of other nutrients, heavy metals, human pathogens, and some organic chemicals that may or not be partially degraded during the process, there is a general concern on its environmentally sound disposal. Among the available destination options, the most used still are the SS disposal in sanitary landfills, ponds, or lagoons, and the application to agricultural lands.

As in fact 76% of Brazilian cities do not have adequate sanitary landfills for solid residues disposal, which are usually discarded at open field, the disposal in agricultural lands appears to be an economical and environmentally viable way for this purpose. Nevertheless, the SS use in agriculture may be restricted by the presence of heavy metals, pathogens, and persistent organic compounds, depending on the SS original composition [2, 3].

In general, SS treated soil presents improved soil physical attributes, such as greater aggregate stability [4, 5] and lower soil density, providing better root and shoots development with significant plant biomass increase [6]. Furthermore, the presence of plant macro and micronutrients in SS, mainly N, P, S Ca, Mg, and Zn, may supply, total or in part, the plant's demand for these elements [7]. Approximately one third of N [8] and up to 64% of P [9] content in SS would be available to plants in the first year. When lime is used to cleanse or to improve sewage drainage, the resulting SS increases soil pH and decreases exchangeable Al [7].

However, SS effects listed above are not persistent and last for only one cropping year [10, 11]. Zinc added to soil via SS has been the most absorbed micronutrient by plants, but the increased pH and OM content may induce lower Fe and Mn uptake [12]. These two SS effects (higher soil pH and OM) are also responsible for low heavy metal mobility in the soil

Table 1: Soil chemical attributes before sewage sludge application.

Soil depth	OM	pH	CEC	H+Al	K	Ca	Mg
	$g\,dm^{-3}$				$mmol_c\,dm^{-3}$		
0–20 cm	26	5.3	64	22	4.4	27	11.0
20–40 cm	17	4.8	52	23	2.6	13	6.7
	V	P	B	Cu	Fe	Mn	Zn
	%				$mg\,dm^{-3}$		
0–20 cm	66	3	0.17	2.6	27.0	23	0.7
20–40 cm	42	2	0.18	0.5	16.3	5	0.2

OM: organic matter, pH ($CaCl_2$), CEC: cation exchange capacity, H+Al: potential acidity, V%: base saturation, K, Ca, Mg, P in resin extract, B in hot water extract, and Cu, Fe, Mn, and Zn in DTPA extract.

profile. The heavy metal mobility was found to be inversely proportional to soil Fe and Al oxides concentrations [13].

Usually, the SS disposal in agricultural lands is done after its dehydration and drying mainly to lower transportation costs and to enhance pathogens inactivation. Nevertheless, in areas near the sewage treatment plants, SS might also be applied in the liquid form when nitrogen crops requirement and natural soil water retention capabilities are considered. Such practice may provide lower operational costs since it is easier to handle and enables energy and chemicals products saving, better application homogeneity, and can also supply greater amount of K to plants, which is usually lost with the aqueous phase if solid SS is used. Despite of that, there is some concern that the large quantities of SS applied in the liquid form might lead to heavy metal accumulation in soil and in plant tissues overtime.

Therefore, the aim of this work was to evaluate the changes in soil attributes and on Surinam grass (*Brachiaria decumbens*) uptake of nutrients and heavy metals after successive land applications of liquid SS.

2. Material and Methods

The experiment was carried out under field conditions, from 2002 to 2006, at Jaguariúna, State of São Paulo, Brazil, (22°46′25″S; 47°06′99″W; altitude 604 m) in an area already vegetated with Surinam grass (*Braquiaria decumbens*). The soil was classified as medium texture, dystrophic red-yellow Paleudult (Ultisol). The soil characterization is presented in Table 1.

The LSS used in this work was obtained from domiciliary sewage, collected from bathroom and restaurant discharges of an electronic industry. The wastewater treatment was done by means of aerobic reactor during 120 days. Before application, the sludge was treated with 40% (m/m) hydrated lime to raise pH above 12. The objective of this procedure was to guarantee the elimination of pathogenic microorganisms and the control of vectors [14].

The LSS, average humidity of 98.3%, was then applied in the field using a 10,000 L tank coupled to a tractor. LSS applications were split into three times a year to attend the sewage treatment plant necessity to remove the sludge. Hence, the LSS treatments were applied during April, August, and December of each year, and the plots without LSS received the equivalent quantities of water. The chemical composition o LSS sample is shown in Table 2. The test plant used was Surinam grass (*Brachiaria decumbens*). The treatments consisted of: (a) control, without LSS application and (b) LSS application three times a year, during four consecutive years; the LSS rates were calculated to not surpass 40 kg N $ha^{-1}\,year^{-1}$ (available nitrogen) [15]. The experimental design was a randomized complete blocks with three replications. The experimental plot measured 20×50 m (1,000 m^2), and each experimental unit consisted of 10 m × 40 m (400 m^2), spaced at least 10 m between plots.

The available N in the sewage sludge was calculated by the formula

$$\text{SS available } N = \left(\frac{\text{MF}}{100}\right) \times (\text{Kjeldahl-N} - \text{NH}_4{}^{+}\text{-N}) + 0.5\,\text{NH}_4{}^{+}\text{–N} + (\text{NO}_3{}^{-}\text{–N} + \text{NO}_2{}^{-}\text{–N}), \quad (1)$$

where Kjeldahl–N: total nitrogen ($mg\,kg^{-1}$), $NH_4{}^{+}$–N: nitrogen as ammonium ($mg\,kg^{-1}$), $NO_3{}^{-}$–N: nitrogen as nitrate ($mg\,kg^{-1}$), $NO_2{}^{-}$–N–: nitrogen as nitrite ($mg\,kg^{-1}$), and MF: mineralization fraction (47%), determined previously via incubation test in the laboratory [3]. This calculation is used for the SS application onto soil surface without incorporation, according to recommendation of the Company of Environment Sanitation Technology (CETESB) in the 4.230 Directive [3].

The annual LSS rates (based on the available N) applied in the experiment were: 34, 40, 19, and 40 kg ha^{-1} in the cropping years of 2002/2003, 2003/2004, 2004/2005, and 2005/2006, respectively.

One week before each LSS application, from each experimental plot, soil and grass samples were taken. The composite soil samples were collected at 0–20 cm and 20–40 cm depth, as grass samples were obtained by cutting its shoots at 2 cm from the soil surface. Soil samples were dried at 45°C, passed through 2 mm-sieve, and analyzed according to standard procedures adopted by Instituto Agronomico de Campinas [16, 17]. Plant tissue samples were washed with a (0.1% v/v) detergent solution, rinsed in distilled water until removing the detergent and finally rinsed in deionized water. After washing, the samples were put in paper bags and dried at 65°C in a forced air oven until constant weight. Following,

Table 2: Chemical composition of sewage sludge used in the experiment.

Parameter	Unity	Year of application			
		2003	2004	2005	2006
Moisture	%	97.4	98.8	97.8	99.0
pH	—	11.1	11.9	11.8	10.1
Organic carbon	$g\,kg^{-1}$	262	327	282	448
Total N (Kjeldahl)	$g\,kg^{-1}$	33	39	28	45
Ammonium-N	$g\,kg^{-1}$	51.0	4.6	1.3	3.5
Nitrate+nitrite-N	$g\,kg^{-1}$	0.40	0.35	0.55	1.42
Sulfur (S)	$g\,kg^{-1}$	2.9	9.4	5.6	4.5
Magnesium (Mg)	$g\,kg^{-1}$	2.7	3.4	2.9	3.4
Aluminum (Al)	$g\,kg^{-1}$	2.4	1.4	1.7	1.1
Calcium (Ca)	$g\,kg^{-1}$	124	115	69	75
Phosphorus (P)	$g\,kg^{-1}$	7.9	12.3	7.5	5.7
Potassium (K)	$g\,kg^{-1}$	1.9	9.1	16.2	8.6
Sodium (Na)	$g\,kg^{-1}$	2.3	8.5	4.5	13.5
Arsenium (As)	$mg\,kg^{-1}$	<1.0	<1.0	<1.0	<1.0
Cadmium (Cd)	$mg\,kg^{-1}$	1.0	5.4	1.3	<1.0
Lead (Pb)	$mg\,kg^{-1}$	35.7	27.4	8.7	21.0
Cobre (Cu)	$mg\,kg^{-1}$	437	452	419	267
Cromo (Cr)-total	$mg\,kg^{-1}$	14.0	31.3	6.3	5.0
Mercurium (Hg)	$mg\,kg^{-1}$	<1.0	<1.0	<1.0	<1.0
Molibdenum (Mo)	$mg\,kg^{-1}$	<1.0	<1.0	<1.0	<1.0
Nickel (Ni)	$mg\,kg^{-1}$	9.2	47.0	9.0	6.3
Selenium (Se)	$mg\,kg^{-1}$	<1.0	<1.0	<1.0	<1.0
Zinc (Zn)	$mg\,kg^{-1}$	486	845	439	208
Boron (B)	$mg\,kg^{-1}$	8.7	14.8	14.6	12.6

the samples were ground in a Wiley-type grinder (inox chamber and 1 mm sieve), homogenized and submitted to digestion and chemical analysis for the nutrients (N, P, Ca, K, Mg, B, Cu, Fe, Mn, and Zn) [18]. The data from chemical analysis was submitted to analysis of variance (F test, $P < .05$) and to regression analysis.

3. Results and Discussion

The average effects of twelve successive LSS applications on the soil chemical attributes are presented in Table 3 (0–20 cm depth) and Table 4 (20–40 cm depth). The analysis of variance showed there were no significant differences among LSS-treated plots and the control for OM, K, B, Cu, Fe, and Mn concentrations in the superficial soil layer (Table 3). However, an increase on soil pH, Ca, Mg, V%, CEC, P, and Zn, with a decrease in H+Al was observed. Similar results were found at 20–40 cm depth, excluding the CEC (cation exchange capacity) that did not change at this depth (Table 4). Changes in OM content were also not observed in an Abruptic Paleudulf cultivated with sugarcane, which received SS (38% solids) addition rates of 0, 20, and 40 $Mg\,ha^{-1}$ [19]. The increase in soil pH at both depths studied and, also, in the soil exchangeable Ca and Mg concentrations can be attributed to the lime added to LSS during the stabilization process, whichincreased pH above 12 [9]. On the other hand, the increase in soil exchangeable Mg concentrations was due to the high presence of this element in the LSS composition. Among the five micronutrients determined in the soil, only Zn (DTPA extract) showed a significant increase with the SS treatments at both depth layers studied, evidencing higher soil Zn mobility compared to the other metal-nutrients (Fe, Cu, and Mn). Zn mobility was also observed in soils with pH below 5.5 that received SS additions [20].

The annual changes on soil chemical attributes caused by the SS addition can be observed in Figures 1, 2 and 3.

The results showed little OM variation, but significant increases on pH and P concentrations in the first year of LSS application at both soil depths (Figure 1). Such results corroborate reports from the literature [19], when studying the effects of increasing SS rates applied to the soil. Nevertheless, it is interesting to highlight that K concentrations tended to decrease in the LSS-treated plots compared to the control-plot (Figure 1). This fact evidences that the grass cut and removed from the SS-treated plots exported more K than LSS could supply.

As expected, soil exchangeable Ca concentrations tended to increase at both depth layers with the LSS addition. Also Mg concentration increased at 20–40 cm layer (Figure 2), suggesting that this element moved into the soil profile,

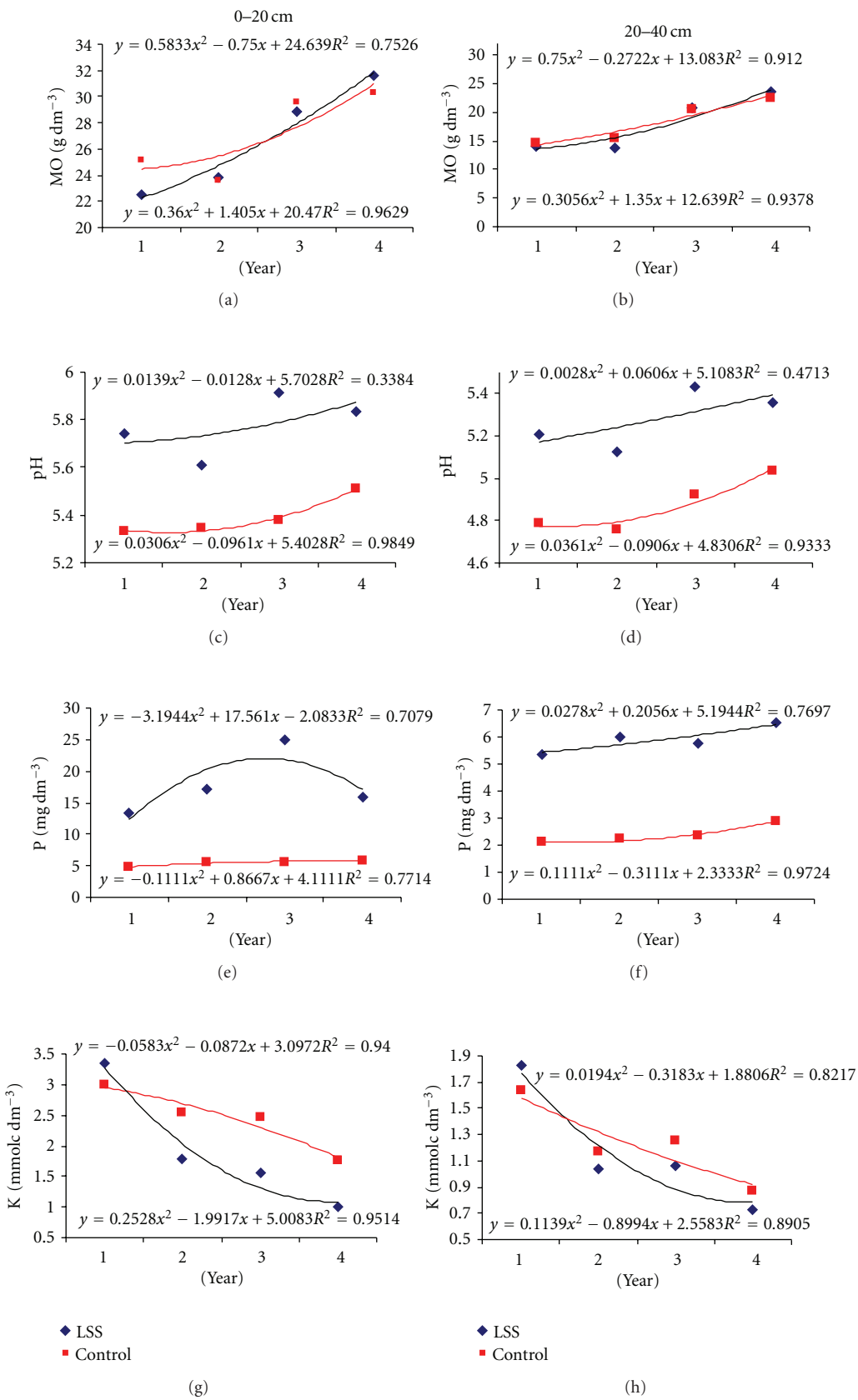

Figure 1: Changes in annual values of pH, soil organic matter (OM), and phosphorus and potassium concentrations, at two soil depth layers (0–20 and 20–40 cm), in experimental plots treated with and without sewage sludge (SS) during four years (2003 to 2006) and cultivated with grass (*Brachiaria decubens*).

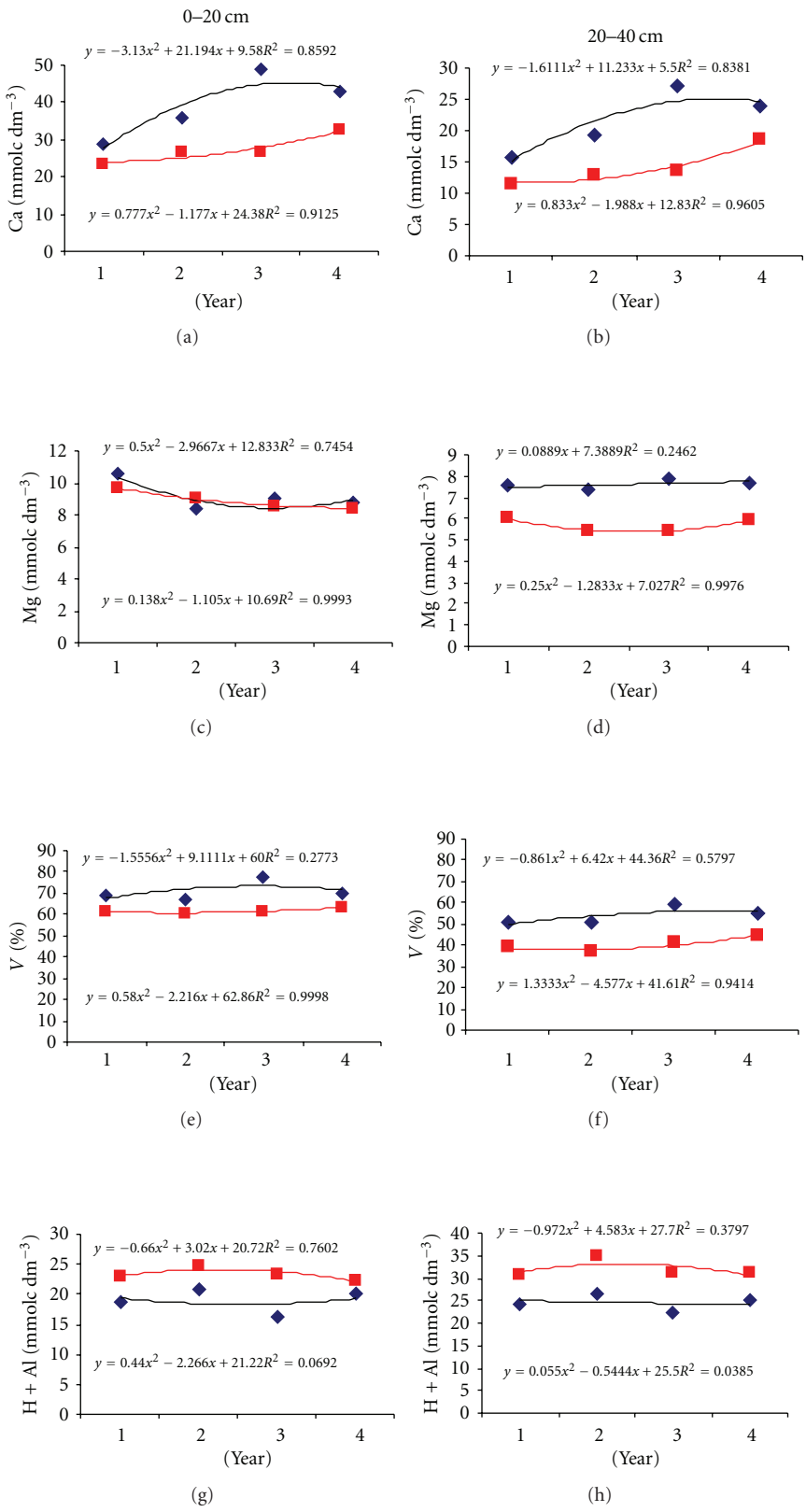

Figure 2: Continued.

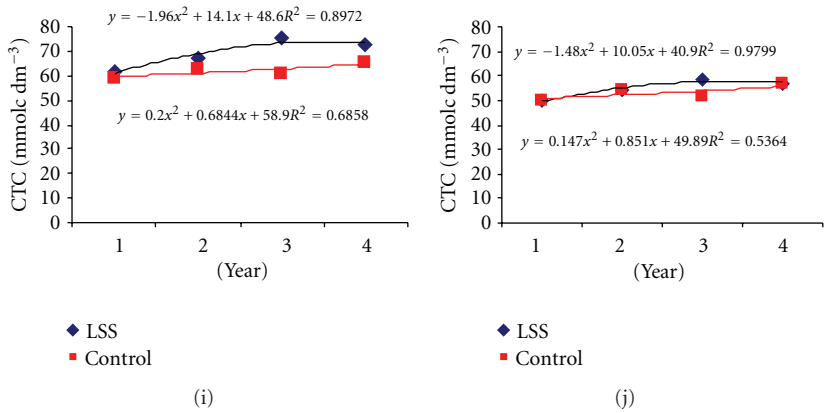

Figure 2: Changes in annual values of soil calcium (Ca), magnesium (Mg) concentrations, base saturation (V%), potential acidity (H+Al), and cation exchange capacity (CEC), at two soil depth layers (0–20 and 20–40 cm), in experimental plots treated with and without sewage sludge (SS) during four years (2003 to 2006).

Table 3: Soil chemical attributes at the 0–20 cm depth layer of soil samples from plots with and without sewage sludge (SS) addition. Means over four years of application (*).

Treatment	OM	pH	CEC	H+Al	K	Ca	Mg
	$g \cdot dm^{-3}$				$mmol_c\ dm^{-3}$		
+SS	26.7a	5.77a	69a	19a	1.93a	39a	9.17a
−SS (Control)	27.1a	5.39b	62b	23b	2.44a	27b	8.92b
	V%	P	B	Cu	Fe	Mn	Zn
				$mg\ dm^{-3}$			
+SS	71a	17.9a	0.14a	2.77a	23.3a	17.4a	1.3a
−SS (Control)	62b	5.44b	0.16a	2.41a	26.6a	20.2a	0.9b

(*) Means followed by the same letters in the column do not differ by F test ($P < .05$).

Table 4: Soil chemical attributes at 20–40 cm depth layer, in plots treated with and without sewage sludge (SS). Means of four years of application(*).

Treatment	OM	pH	CEC	H+Al	K	Ca	Mg
	$g \cdot dm^{-3}$				$mmol_c\ dm^{-3}$		
+SS	18.02a	5.28a	55a	25a	1.16a	21a	7.6a
−SS (Control)	18.31a	4.87b	53a	32b	1.23a	14b	5.7b
	V%	P	B	Cu	Fe	Mn	Zn
				$mg\ dm^{-3}$			
+SS	54a	6.81a	0.14a	0.72a	15.2a	6.5a	0.5a
−SS (Control)	40b	2.38b	0.16a	0.52a	15.7a	5.5a	0.3b

(*) Means followed by the same letters in the column do not differ by F test ($P < .05$).

probably due to its displacement caused by the increase in soil Ca at the LSS-treated plots. The effects of SS-Ca addition on the Mg and K lixiviation in SS-treated soils have already been reported [21].

In the present work, the increase observed on soil pH and exchangeable Ca and Mg concentrations contributed to the increase in base saturation values and to the decrease in the potential acidity (H+Al), just after the first year of LSS application (Figure 2).

The CEC increase was observed after the third year of LSS application, and this effect was restricted to the superficial soil layer (0–20 cm). Once there was no soil OM increase, it is supposed that the CEC increase was a consequence of the method used for its calculation, which is based on the sum of the exchangeable cations (Ca, Mg, and K) and the potential acidity (H+Al) (Figure 2).

Although without statistical significance, there was observed a slight increase on available Cu and decrease on

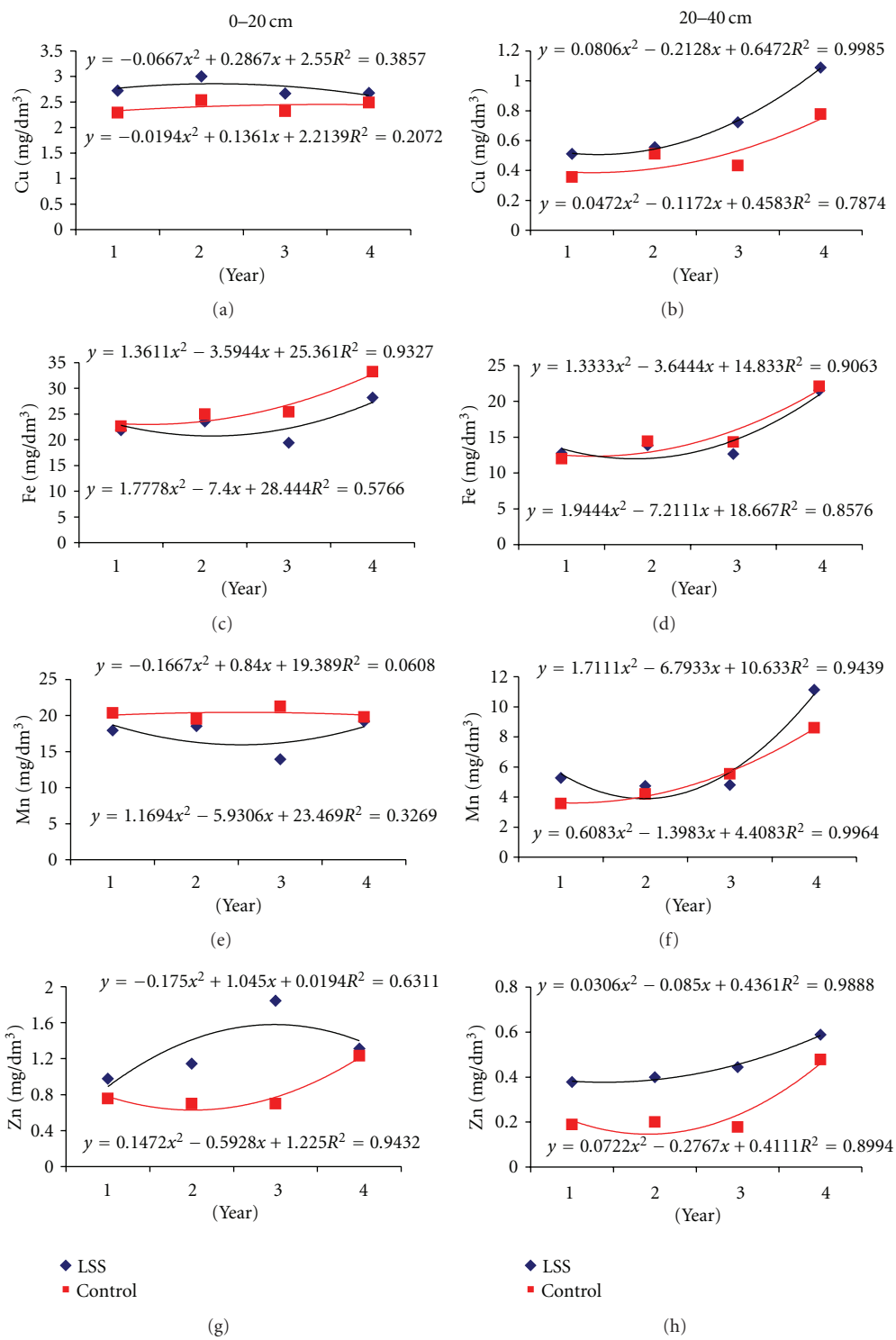

Figure 3: Soil micronutrient's concentration, at two soil depth layers (0–20 and 20–40 cm), for treatments with and without sewage sludge (SS) during four years (2003 to 2006).

available Fe and Mn in LSS-treated plots compared with the control plots at both depth layers studied (Figure 3). Since the liquid LSS content of such metals was quite low, at this point, even after twelve applications, no clear effect of accumulation or in the mobility of such metals in soil was observed. Despite of that, reports in the literature did observe significant decrease on exchangeable Mn concentrations in an amended clayey soil, with consequent increase in the organic fraction and noncrystal Fe and Al oxides [22].

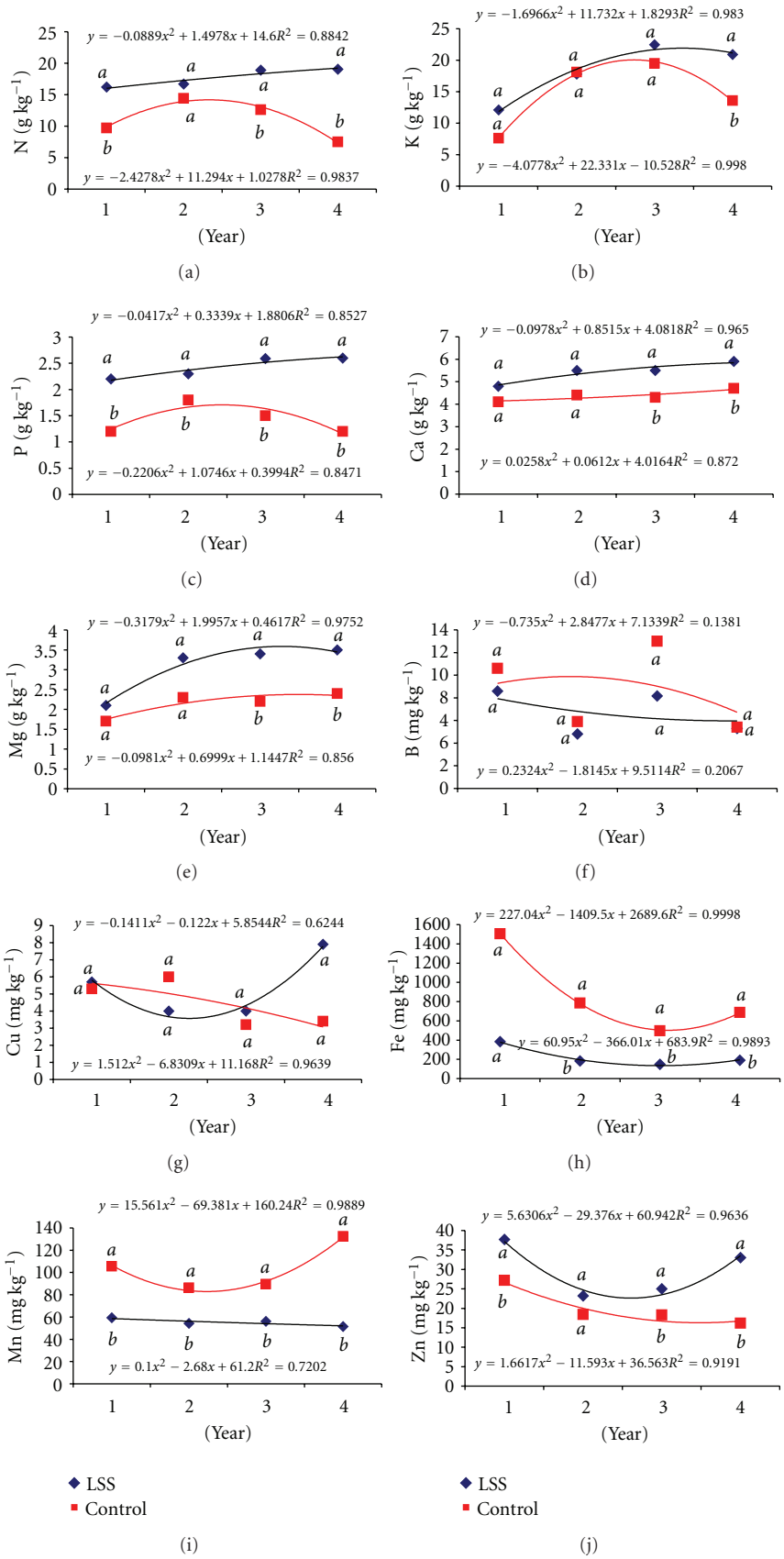

Figure 4: Grass nutrient's concentration for treatments with and without sewage sludge (SS), during four years (2003 to 2006). Means followed by the same letters between treatments in each year do not differ by F test ($P < .05$).

The soil analysis results for Zn indicated higher concentrations at both depth layers during the four years of LSS application (Figure 3). Positive correlations between soil Zn and Zn absorbed by corn plants grown in three SS-treated soils with or without $CaCO_3$ have been reported [23].

LSS treatments significantly increased leaf N and P concentrations in the first year of application (Figure 4), as it has been reported in the literature for maize plants [9, 24]. Leaf K, Ca, and Mg concentrations increased with LSS treatments since the first year, but were significantly higher after the third (Ca and Mg) and fourth years (K). No significant differences among treatments for the leaf B and Cu were found, probably due to the low LSS content of these elements and, also, to the low element uptake and translocation rate from roots to shoots. However, significant leaf Mn decrease was observed along the four years and, also, leaf Fe decrease after the second year of application. Increased leaf Zn was also observed during three years (Figure 4). These results agree with the reported in the literature that observed significant increase on leaf and shoot Zn concentrations, decrease on Fe and Mn, and no changes on Cu of maize plants grown in an SS-treated red Oxisol [12]. In the present work, the grass leaf nutrient levels were within the adequate concentration ranges for *Brachiaria decumbens*, except for leaf B ($<10\,\mathrm{mg\,kg^{-1}}$) [25].

4. Conclusions

The application of liquid sewage sludge (stabilized with hydrated lime) to the field cropped with Surinam grass did not change the soil organic matter and exchangeable K contents, increased soil pH and base saturation, and decreased the potential acidity at 0–20 and 20–40 cm depth layers.

The sewage sludge applied increased soil labile P and available Zn concentrations but did not change available B, Cu, Fe, and Mn concentrations until 40 cm depth.

The sewage sludge produced in an aerobic reactor and applied in liquid form behaved as a source of nitrogen, phosphorus, calcium, magnesium, and zinc for the Surinam grass (*Brachiaria decumbens*), mainly after two years of residue application to the soil.

The sewage sludge used, although containing more K than most residues, did not supply enough K to plants, and, therefore, it required complementation with mineral forms of this nutrient. It is recommended to monitor soil K concentration using a reference critical level (Boletim 100, IAC) to prevent K deficiency in plants.

The grass harvested shoots reflected the effects of sewage sludge addition to the soil. Most nutrients were not affected, but lower leaf Mn concentrations were observed. However, it is recommended to monitor leaf B concentration in order to prevent B deficiency in plants.

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