Science of the Total Environment 643 (2018) 1493–1501

Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Ten years of application of sewage sludge on tropical soil. A balance sheet on agricultural crops and environmental quality

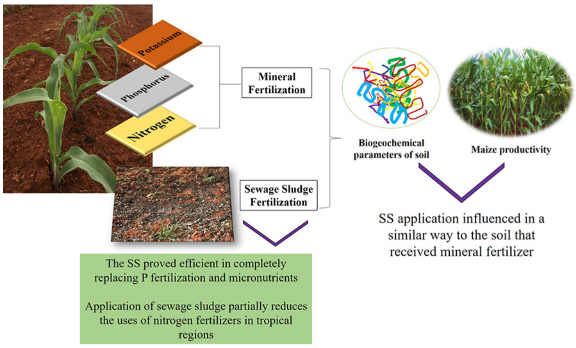
Wanderley Melo [a,b], Denise Delarica [a,*], Ana Guedes [a], Letícia Lavezzo [a], Riviane Donha [a], Ademir de Araújo [c], Gabriel de Melo [b], Fernando Macedo [a]

[a] *São Paulo State University (Unesp), School of Agricultural and Veterinarian Sciences, Path of Access Prof. Paulo Donato Castellane, Km 5, 14884-900 Jaboticabal, SP, Brazil*
[b] *Brazil University, University Campus Descalvado - Hilário da Silva Passos Avenue, 950 - University Park, Descalvado, SP, Brazil*
[c] *Soil Quality Laboratory, Agricultural Science Center, Federal University of Piauí, 64000-000 Teresina, Brazil*

HIGHLIGHTS

- Ten years of application of sewage sludge (SS) on tropical soil was evaluated.
- Performance of maize cultivation and soil quality were the parameters used.
- Ten years of application of SS did not increase soil organic matter.
- The SS proved efficient in completely replacing P fertilization and micronutrientes.
- SS and mineral fertilizer application was similar to biochemical parameters evaluated.

GRAPHICAL ABSTRACT



SS application influenced in a similar way to the soil that received mineral fertilizer

The SS proved efficient in completely replacing P fertilization and micronutrients

Application of sewage sludge partially reduces the uses of nitrogen fertilizers in tropical regions

ARTICLE INFO

Article history:
Received 22 May 2018
Received in revised form 20 June 2018
Accepted 20 June 2018
Available online 4 July 2018

Editor: Frederic Coulon

Keywords:
Soil enzyme
Soil quality
Trace elements
Soil biological activity
Biosolids

ABSTRACT

Sewage sludge (SS) has been used in agriculture on a large scale in Europe. In tropical climate conditions, due to rapid organic matter degradation, SS can provide toxic elements that can cause damage to the environment. We evaluated an area that received doses of SS for 10 years compared to mineral fertilization. The biogeochemical parameters (amylase, cellulase, invertase and dehydrogenase activity, carbon microbial biomass, basal respiration, metabolic quotient, soluble carbohydrates, total carbohydrates and glucose) and maize agronomic performance were evaluated. There was no increase of organic matter in the area due to the SS application. However, the application of SS for 10 consecutive years influenced the biogeochemical parameters evaluated in a similar way to the area that received mineral fertilizer. The SS proved efficient in completely replacing P fertilization and micronutrients. SS can also partially replace N fertilization without reducing maize productivity.

1. Introduction

Sewage sludge (SS) is the final output of the biological treatment of domestic and industrial effluents. The increasing production of urban waste, including SS, raises the need to find alternative and sustainable ways of waste disposal. The use of such waste on agricultural crops

---

* Corresponding author.
E-mail addresses: denise.delarica@gmail.com (D. Delarica), ana.carolina.t.guedes@monsanto.com (A. Guedes), ademir@ufpi.edu.br (A. de Araújo).

https://doi.org/10.1016/j.scitotenv.2018.06.254

has become attractive since it may partially replace industrial fertilizers (Yagmur et al., 2017), thus reducing costs and energy expenditure. An alternative destination for this residue, besides being disposed of in landfills (Eljarrat et al., 2008), ocean and forest areas, or even being incinerated, therefore, is ensuring greater benefits to the environment (Mahapatra et al., 2013).

This residue can enhance soil fertility due to its high content of nutrients for plants (Tontti et al., 2017), in addition to its potential as a soil conditioner and source of organic matter. The application of organic wastes as farming practice can be beneficial to soil quality, and improve the physical, chemical and biological attributes of the soil (Scotti et al., 2015). Nonetheless, the concentration and long-term accumulation of trace elements and toxic substances present in SS may limit its application to the soil (Eljarrat et al., 2008; Sharma et al., 2017). Although the heavy metal concentration in the soil is an indicator of soil contamination, it does not provide sufficient information about the potential environmental impact. Monitoring soil quality by biological and biochemical indices is becoming an essential tool to understand the environmental risks involved in accumulated applications of SS.

Currently there is a large amount of work that has evaluated the agricultural use of SS. However, these works were not developed in tropical climate conditions. In tropical climate regions, the degradation of organic matter occurs more rapidly (Ross, 1993; Castro et al., 2015), with potential mineralization of elements that may cause risks to the environment. This behavior should be monitored, preferably, with long-term field experiments.

The use of soil biochemical properties as indicators of soil quality are sensitive to alterations in soil management (Paz-Ferreiro et al., 2012), offering faster outcomes when compared to the testing of soil chemical and physical properties.

Soil enzymes are key factors in nutrient cycling and can be used to detect changes in the microbial community derived from soil management, e.g. organic waste application. Based on enzyme activity, further significant variations can be checked after applying high levels of organic matter from SS, e.g. toxic trace elements, toxic substances and degree of contamination (Patel and Patra, 2014; Bhattacharyya et al., 2008). Likewise, quantifications of carbon microbial biomass (CMB) and soil basal respiration (SBR) are crucial to detect early symptoms of soil stress, caused by waste application (Santos et al., 2011; Tripathya et al., 2014). Both SMB and enzyme activity have a high sensitivity to waste application, even in conditions of small environmental changes in the soil (Tejada et al., 2014; Zhang et al., 2010).

Our hypothesis is that the use of biogeochemical monitoring tools can assess the environmental impact of the agricultural use of SS in tropical climate conditions. The aim of this study was to support farming policies for SS application to the soil, coming up with alternatives to partially and securely substitute mineral fertilization. In order to reach our aims, we evaluated several biogeochemical parameters and the agronomic performance of maize in an area that had received SS for ten consecutive years.

2. Material and methods

2.1. Background of the experimental area

The field experiment was set in Jaboticabal city, Brazil (21° 15′ 37″ S and 48° 19′ 07″ W). According to Köppen's classification, the weather is classified as Aw type. The local soil is classified as an Oxisol with clayey texture and a moderate kaolinitic horizon (clay: 583; sandy: 210; silt: 207 g kg^{-1}).

The experimental design was in randomized complete blocks with four treatments and five replications (Fig. 1). Each plot had a total area of 60 m^2 (6 × 10 m). The treatments were constituted by a control treatment and three SS doses. Maize (*Zea mays* L.), sunflower (*Helianthus annuus* L.) and crotalaria (*Crotalaria juncea* L.) were cropped as test plants in the ten years of the experiment. The SS applied to the soil during the 10 years of experiment was obtained in urban/industrial sewage treatment plants in the cities of Barueri and Franca (SP), with activated sludge system with anaerobic reactor. The SS accumulated in each treatment over ten years was 0.0, 50.0, 100.0 and 147.5 Mg ha^{-1} (Supplementary Table1). During the growing season in the tenth year of the experiment, the minimum and maximum temperatures ranged from 18.7 to 21, and 28.9 and 31.7 °C, respectively, and the rainfall was 1.118 mm. The SS used in the experiment was obtained from the Wastewater Treatment Plant (WTP) located in Franca city, Brazil. For the SS chemical characterization (Supplementary Table 2), six single samples were collected at different points in the residue mass, then mixed and combined in a composite sample. The SS was spread on the entire area uniformly at a moisture level similar to when it left the WTP. Next, maize was sown, using a hybrid AG 2060® at a density of seven seeds per linear meter and a spacing of 0.9 m. The control treatment received only, urea (45% N); simple superphosphate (18% P); potassium chloride (58% K) (Table 1).

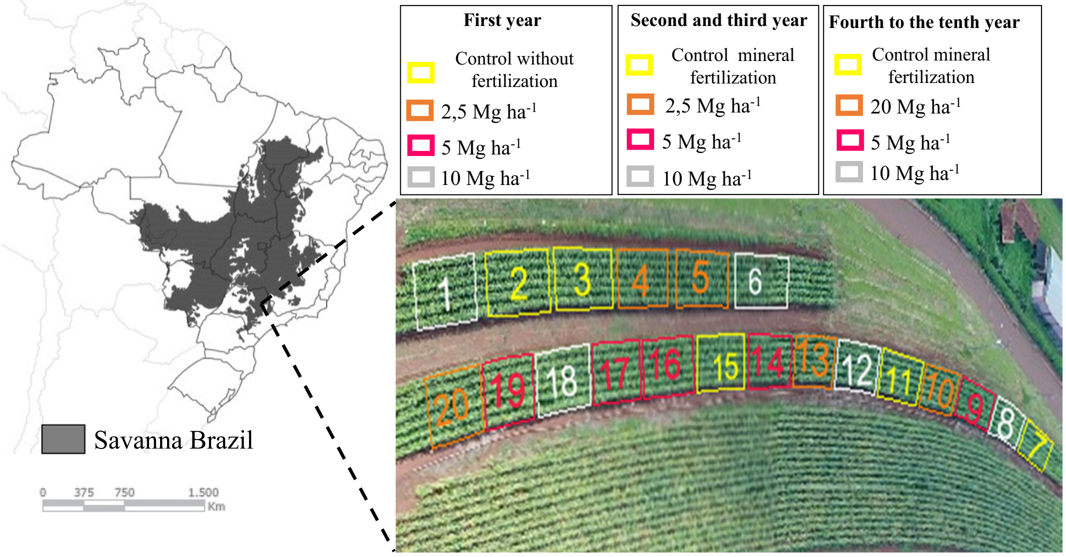


Fig. 1. Treatment history.

Table 1
Mineral fertilization applied to the Oxisol during the ten-year experiment.
Source: Melo et al. (2007) and Nogueira et al. (2010), adapted.

Cropping year	Sewage sludge doses[a]	N			P (P_2O_5)	K(K_2O)
		SS source[c]	SS available[d]	Mineral fertilization[e]	Mineral fertilization[e]	Mineral fertilization[e]
Mg ha^{-1}		kg ha^{-1}				
1	0	0	0	0	0	0
	2.5	80	27	0	0	0
	5	160	53	0	0	0
	10	320	107	0	0	0
2	0	0	0	170	30	50
	5	92	31	139	0	85
	10	185	62	108	0	80
	20	370	123	47	0	70
3	0	0	0	170	50	50
	7.5	73	24	146	0	86
	15	145	48	122	0	81
	30	290	97	73	0	72
4[b]	0	0	0	170	50	50
	27.5	580	193	0	0	47
	20	145	48	92	0	79
	40	290	97	43	0	68
5	0	0	0	170	50	50
	47.5	740	247	0	0	0
	25	185	62	108	0	74
	50	370	123	47	0	58
6	0	0	0	170	50	50
	67.5	680	227	0	0	52
	30	170	57	113	0	81
	60	340	113	57	0	71
7	0	0	0	50	30	30
	87.5	820	273	0	0	28
	35	205	68	0	0	30
	70	410	137	0	0	29
8	0	0	0	0	20	20
	107.5	680	227	0	0	0
	40	170	57	0	0	14
	80	340	113	0	0	7
9	0	0	0	170	50	50
	127.5	680	227	0	0	59
	45	170	57	113	0	82
	90	340	113	57	0	74
10	0	0	0	170	50	50
	147.5	460	153	17	0	64
	50	115	38	132	0	83
	100	230	77	93	0	77

SS: Sewage sludge.
[a] Annual accumulated doses.
[b] For fourth year. It was decided to substitute 2.5 by 20 Mg ha^{-1} of sewage sludge. On a dry basis.
[c] Total nitrogen.
[d] Available nitrogen. Considering that 1/3 was mineralized.
[e] Mineral Fertilization according to Raij et al. (1997).

During the beginning of bloom, leaf sampling was done (Malavolta et al., 1997). The central third of the leaf was used to prepare the sample, and the central rib was detached from the leaf. The samples were rinsed in distilled water and deionized water, oven dried at 65 °C for 72 h, then ground in a Wiley mill with 20 mesh screens opening. The dry matter yields of each plant part were recorded, and the oven-dried plant tissues were ground in a Wiley mill to determine total N by a Kjeldahl method; total P and K by vanado-molybdate spectrophotometry and flame-photometry, respectively, after digestion with nitric-perchloric acids; and the heavy metals by atomic absorption spectrophotometry (AAS) in an acetylene-air flame (Model: AVANTA GBC, Australia) in extracts obtained by digestion with $HNO_3 + H_2O_2 + HCl$ – USEPA3050b- (USEPA, 1996).

For the evaluation of crop production, plants were harvested 131 days after sowing from the two central rows of each plot, dried at 65 °C in a forced air oven, and weighed. The data of grain yield were corrected to 13% moisture.

2.2. Sampling and soil sample preparation

Soil samples were collected at 60 days after emergence of the seedlings. Ten samples were collected per plot at a depth of 0–0.20 m. Then, they were packed in Styrofoam boxes with ice and sent immediately to the laboratory. In the lab, part of each sample was air-dried under shade and passed through a 2-mm mesh sieve; subsequently, these samples were packed into polyethylene plastic bags, properly tagged and stored in a dry chamber until analysis. The other part of each sample was directly packed into labeled plastic bags and kept in a refrigerator at 5 °C until analysis.

2.3. Enzymatic activity evaluations

2.3.1. Amylase

Amylase activity was determined according to the method described in Ross (1965). To evaluate amylase activity, the procedure consists of incubating the soil samples with the enzyme substrate (starch) for a period of 24 h and at a constant temperature of 30 °C; then, glucose evaluations are carried out using a copper reagent.

2.3.2. Cellulase

To determine the cellulase activity in the soil samples, we used the method proposed by Pancholy and Rice (1973), consisting in incubating the samples with carboxymethyl cellulose for a period of 24 h at a constant temperature of 30 °C, then evaluating the amount of glucose produced, as described for amylase.

2.3.3. Invertase

The invertase activity was determined using the method described in Ross (1987). For this enzyme, a 1 g soil sample has to be incubated with 2 ml distilled water, 2 ml sucrose solution (5%, v/v) and 0.2 ml toluene for 24 h at a constant temperature of 30 °C, assessing the amount of glucose produced, as described for amylase.

2.3.4. Dehydrogenase

For the determination of dehydrogenase activity in the soil samples the method proposed by Alef and Nannipieri (1995) was used. Samples were incubated with triphenyl tetrazolium chloride solution (TTC) for 24 h at a constant temperature of 30 °C, measuring the triphenyl formazan formed.

2.3.5. Soil basal respiration

Estimates of SBR followed the method described by Menyailo et al. (2003), which consists of incubating soil samples without substrate addition for 72 h at room temperature, and assessing the amount of CO_2 produced.

2.3.6. Carbon microbial biomass

CMB was determined by the fumigation-incubation method proposed by Vance et al. (1987), which involves carbon content determination in chloroform fumigated and non-fumigated soil samples. The difference between carbons provides the carbon content of the microbial biomass.

2.3.7. Metabolic quotient (qCO_2)

The qCO_2 followed the method described by Anderson and Domsch (1986), whose formula is as follows:

$$\mathrm{qCO_2}\left(\mathrm{mgC{-}Co_2 \cdot g^{-1}BMS{-}C \cdot h^{-1}}\right) = \frac{\mathrm{SBR}\left(\mathrm{mgC{-}CO_2 \cdot kg^{-1}soil \cdot h^{-1}}\right)}{\mathrm{CMB{-}C}\left(\mathrm{mgC \cdot kg^{-1}soil}\right) \cdot 10^{-3}}$$

2.3.8. Soluble carbohydrates

To determine the soluble carbohydrate content, the method proposed by Melo (1977) was used. The amounts of soluble carbohydrates in soil samples were extracted with 2 mol L^{-1} KCl solution, being stirred for 1 h, then measuring the content of glucose equivalent extracted by reaction with anthrone solution.

2.3.9. Total carbohydrates

The total carbohydrate content was determined according to the method of Brink et al. (1959). The method consists in incubating the soil samples with 3 N H_2SO_4 leaving 24 h in a water bath at 80 °C, evaluating the amount of glucose present in the samples.

2.3.10. Glucose content

The glucose content was determined based on the enzymatic method proposed by Frey et al. (1999). First, soil-extracted glucose is phosphorylated to glucose-6-P, which, in turn, is oxidized to 6-phosphogluconate, reducing NAD^+ to NADH. Absorbance was determined at 340 nm.

2.4. Statistics

The experiment was analyzed in a randomized block design with four treatments and five replications. The data underwent variance analysis (RBD), followed by the Tukey's test at 5% probability to compare average and determine whether the F test was significant.

3. Results

3.1. Biogeochemical parameters

In the tenth year, the application of SS presented only a few and specific differences regarding the biological and enzymatic activities in relation to the treatment that received mineral fertilization (control treatment). Regarding amylase activity in the soil, the only treatment that differed from the control was the 100 Mg ha^{-1} (Fig. 2a). The amylase activity in soils that received a cumulative dose of 100 Mg ha^{-1} of SS was twice as high as in the other treatments. The opposite effect was observed in the evaluation of the CMB, in which its lower concentration was verified in the same treatment – 100 Mg ha^{-1} (Fig. 3a). The soil cellulase activity presented differences only among the treatments that received SS. The treatment that received the lowest dose of SS presented cellulose activity 2.7 times greater than the treatment that received the highest dose SS (Fig. 2b). Invertase activity showed, at the highest dose of SS applied, average activity two times higher than the other treatments (Fig. 4a), and also the soil that received the highest dose of SS presented almost double the (qCO_2) when compared to the control treatment. However, the control treatment and the higher SS dose applied did not differ from the other treatments in terms of qCO_2 (Fig. 3b). Glucose was the only parameter measured in this study that presented a lower concentration in the treatment that did not receive SS (Fig. 4b). For the other biogeochemical parameters (CMB, dehydrogenases activity, SBR, soluble carbohydrates, total carbohydrates) evaluated in this work, there was no highlight for any individual treatment (Figs. 3a, 3c, 5, 6a and b).

3.2. Maize performance

Neither the grain yield nor the dry matter production (Table 3) was influenced by the treatments. Small variations in nutrient concentration in the diagnosed leaves were observed. N, K, S, B and Cu content in the diagnosed leaves presented no difference between treatments. The diagnosed leaves had Mg concentrations below the ideal range in all the evaluated treatments, while B was above the ideal range (Table 4).

4. Discussion

After ten years of SS continuous applications in the area, the biogeochemical parameters evaluated presented few variations when compared to the area that received mineral fertilizer in the same period. In particular, some biogeochemical parameters with high sensitivity to environmental changes such as dehydrogenases activity, soluble carbohydrates, total carbohydrates, and CMB (Machulla, 2003; Duval et al., 2013; Wang et al., 2016) presented similar results between the control treatments and some of the treatments that received SS. These results are important because, although European countries have been using SS in agriculture for several years (Lloret et al., 2016), for tropical climate conditions, there is a lack of research on SS performance in the environment. This has led governments to enact very restrictive laws that have hampered the use of SS in agriculture. In this study, we show that, regarding the environmental impacts, the continuous use of SS, up to the dose of 10 Mg ha^{-1} per year, is similar to mineral fertilizer.

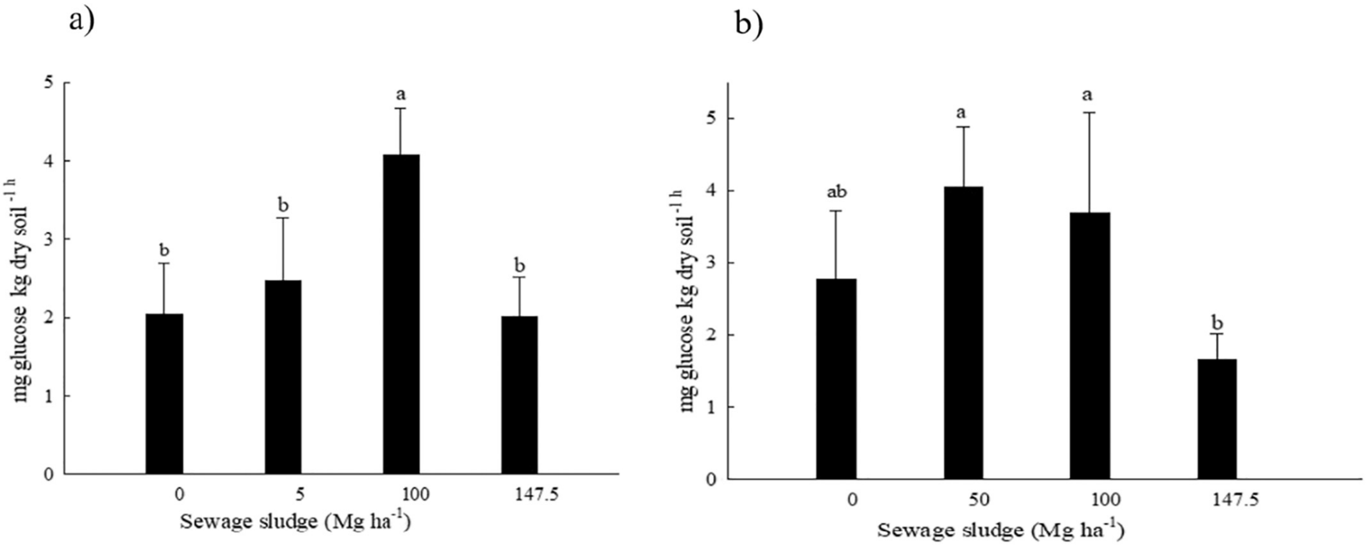


Fig. 2. a) Amylase activity in soils treated with sewage sludge. b) Cellulase activity in soils treated with sewage sludge.

Only a few biogeochemical parameters (amylase, cellulase and glucose) showed greater activity in the areas that received intermediate doses of SS (50 and 100 Mg ha^{-1}) and they showed no difference between the control treatment and the highest dose of SS applied (147.5 Mg ha^{-1}). This may have been a response to N supplementation with mineral fertilizer. The control treatment received only mineral

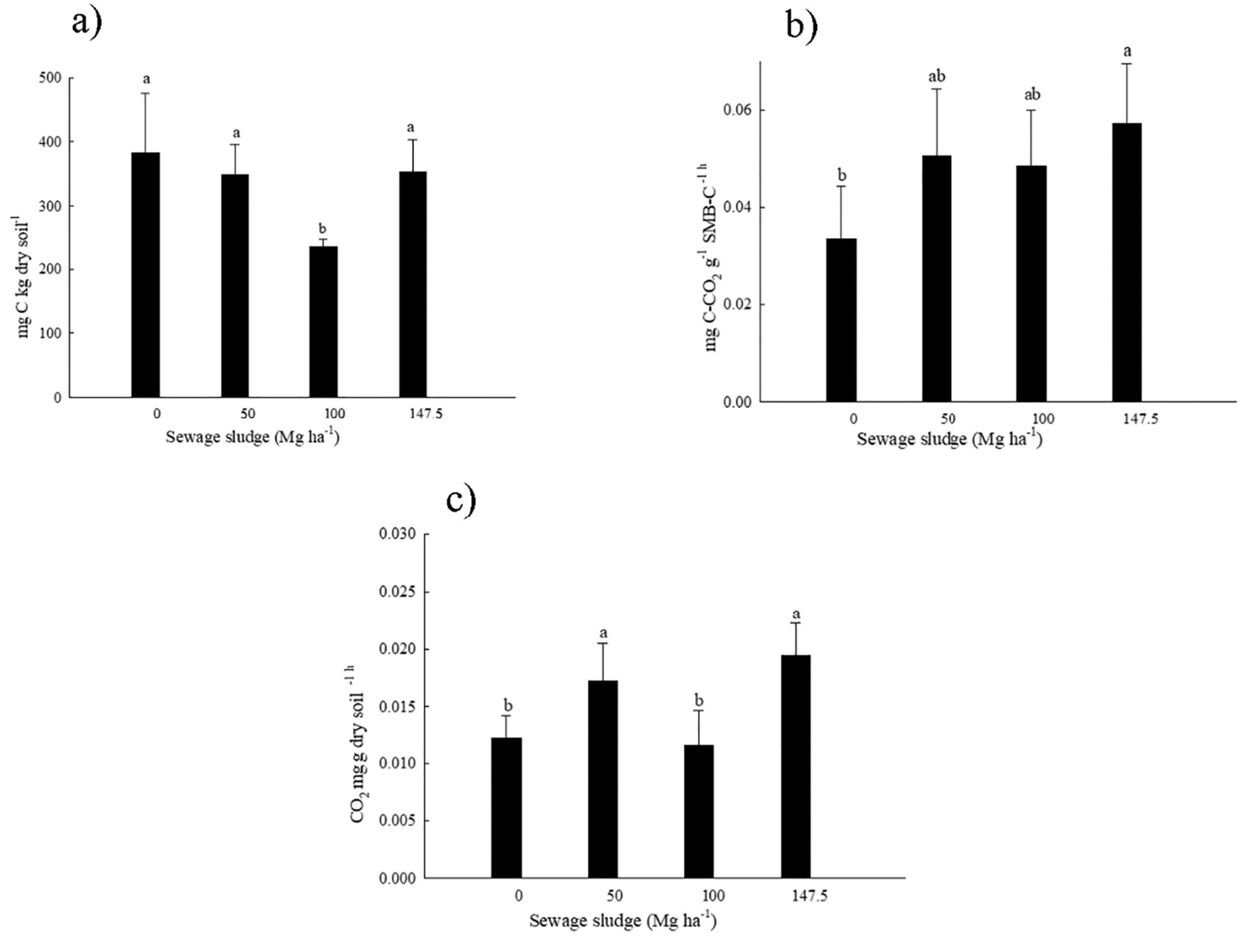


Fig. 3. a) Carbon microbial biomass in soils treated with sewage sludge. b) Metabolic quotient (qCO_2) in soils treated with sewage sludge. c) Soil basal respiration in soils treated with sewage sludge.

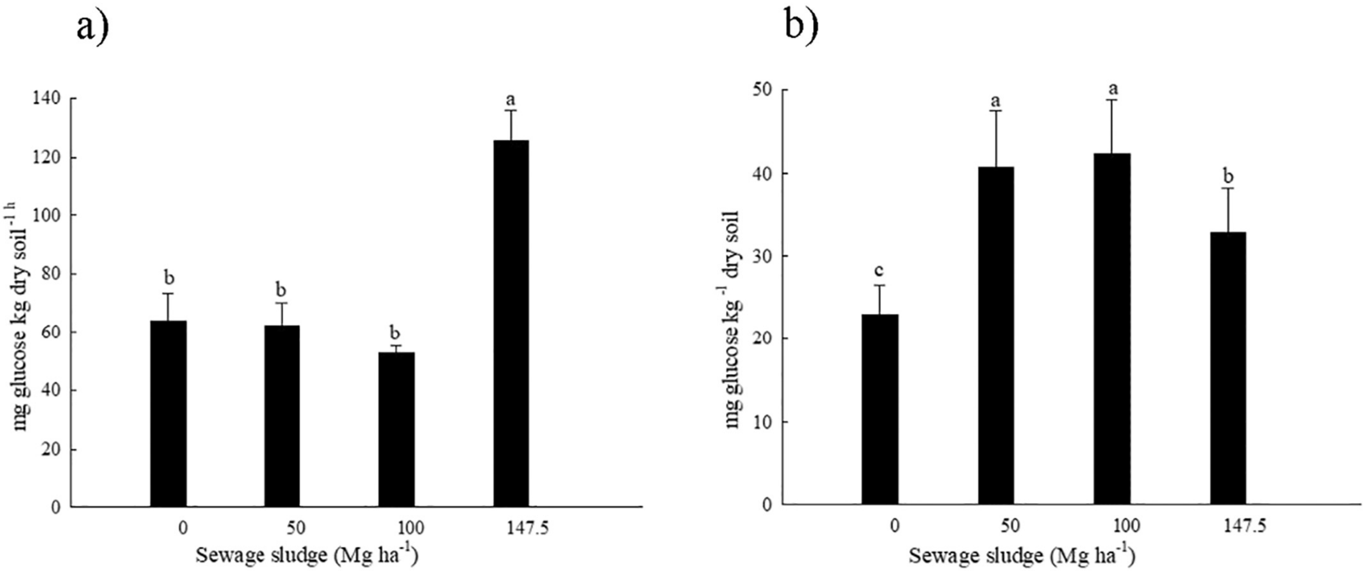


Fig. 4. a) Invertase activity in soils treated with sewage sludge. b) Glucose in soils treated with sewage sludge.

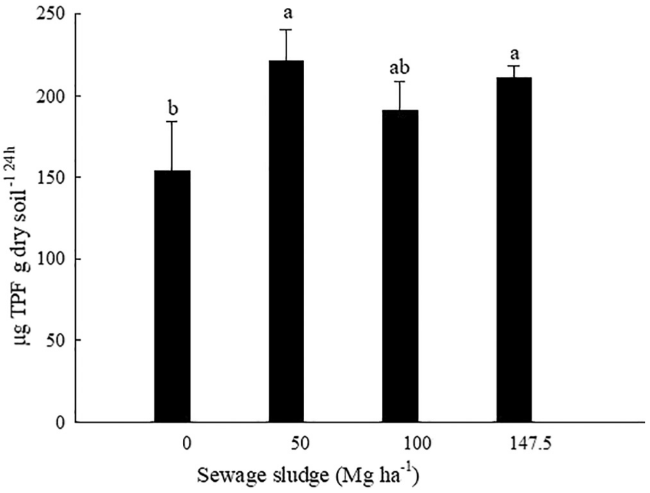


Fig. 5. Dehydrogenases activity in soils treated with sewage sludge.

fertilizer, while the larger SS dose required a small amount of N supplementation. The intermediate treatments received SS and a considerable amount of readily soluble N from mineral fertilizer (Supplementary Table 3). This may have contributed to the activities of the amylase and cellulase enzymes, and the glucose rates were higher in these treatments. In addition, there is a direct relationship between soil C—N availability and soil enzymatic activities (Fernandes et al., 2005; Ferreira et al., 2016; Masunga et al., 2016; Harantová et al., 2018). SBR and $q\text{CO}_2$ presented great variation between the control treatment and the highest SS dose applied. However, it should be noted that our soil collection for enzyme analysis occurred at 60 DAE. The temporary increment of organic matter from SS explains these values (Santos et al., 2012; Silva et al., 2014). All values referring to SBR and $q\text{CO}_2$ suggested that microbial populations operate in the absence of metabolic stress (Silva et al., 2014). We know that organic matter from SS when applied to soils of tropical regions is rapidly degraded (Busato et al., 2012; Vieira et al., 2014 and the increase of organic matter in the soil over time is very low or zero (Yada et al., 2015, Kirchmann et al., 2017). In the present study, no increase of soil organic matter was observed due to the

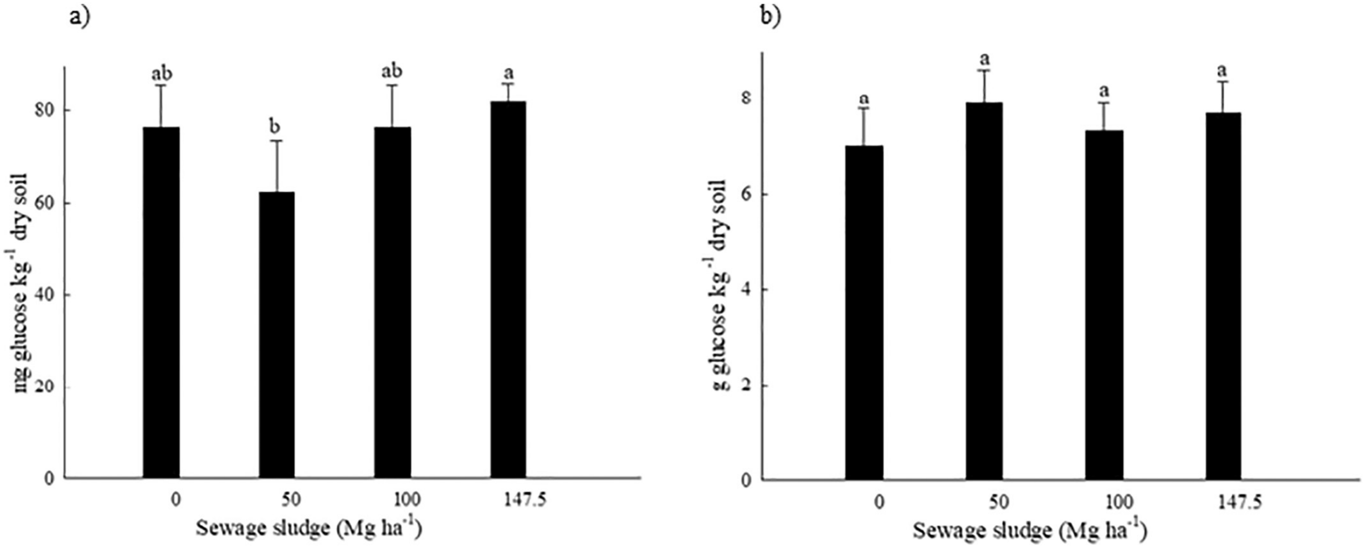


Fig. 6. a) Soluble carbohydrate in soils treated with sewage sludge. b) Total carbohydrate in soils treated with sewage sludge.

Table 2
Chemical characterization of the soil before the start of the experiment. and after 10 years applications of sewage sludge.

	pH ($CaCl_2$)	OM[a]	P[b]	K[b]	Ca[b]	Mg[b]	H + Al	BS[c]	CEC[d]	BCS[e]d
		g dm^{-3}	mg dm^{-3}	$mmol_c$ dm^{-3}						%
	Oxisoil									
	First year[f]									
	5.7	34	67	4.9	42	19	22	66	88	75
Sewage sludge (Mg ha^{-1})	Tenth year									
0.0	5.3	28	57	6.1	41	12	31	59	90	66
50.0	5.5	28	104	3.5	51	11	31	65	96	68
100.0	5.6	28	142	4.6	54	12	31	70	101	69
147.5	5.1	28	152	3.6	37	8	42	48	90	54

[a] OM: Organic matter.
[b] P. K. Ca. and Mg extracted by resin: mixed ion exchange resin. Extracted from a soil: solution ratio of 1:20. With 16 h of stirring time.
[c] BS: base-cation sum (K ++, Mg + 2, Ca + 2).
[d] CEC: cation exchange capacity.
[e] BCS: base-cation saturation.
[f] Before the start of the experiment.

addition of SS in the first ten years of the experiment (Table 2). We has been already verified that the OM of the area presents great capacity of decomposition and has decreased the degree of humification of OM over the years (Santos et al., 2010). The SS obtained from urban and industrial waste presents low levels of lignin and cellulose, that contribute to the increase of OM in the soil, and high protein content (Carvalho et al., 2015). Other works that have verified increase of OM in tropical soils have used high doses of SS, in a short time, and carried out soil collections very close to the SS application (Canellas et al., 2001; Wang et al., 2008). Perhaps these factors may have interfered with the outcome It is noteworthy that, in our area after application, the SS has been incorporated by means of harrowing. This may have contributed to the fact that the SS application in soil did not presented additions of OM. Adicionalmente, tem se verificado, sob condiçoes tropicais, aumento de ácido fulvico em solos que receram SS (Canellas et al., 2001). For a long time, it was thought that the decomposition of organic matter from the SS would provide the availability of the heavy metals contained in it - the time-bomb hypothesis (McBride, 1995). This could have a major impact on the environment. In our work, even with the rapid decomposition of organic matter, there were no changes in the main biogeochemical parameters evaluated in relation to the control treatment. In the case of Oxisol in tropical areas, due to the low concentration of organic matter, there is a greater interaction of the heavy metals with the mineral fraction of the soil than with the organic matter (Silveira et al., 2003; Nogueira et al., 2010; Soriano-Disla et al., 2014), so that only a small amount of heavy metals is bioavailable. A number of studies have shown that there is no relation between the increase of heavy metals in the soil due to SS application and the absorption of these metals by plants (Macedo et al., 2014; Soriano-Disla et al., 2014; Alvarenga et al., 2016; Yagmur et al., 2017).

Table 3
Grain yield and dry matter plants grown in soil treated with sewage sludge for ten consecutive years.

Sewage sludge doses	Grain yield	Dry matter productivity
Mg ha^{-1}		
Third year		
0	6.93a	14.26a
7.5	8.26a	14.96a
15	7.63a	15.55a
30	8.07a	15.72a
Sixth year		
0	3.89b	14.21a
67.5	6.09a	15.69a
30	6.03a	15.86a
60	5.72a	14.04a
Seventh year		
0	1.6a	nd[(b)]
87.5	1.8a	nd
35	1.8a	nd
70	2.1a	nd
Eighth year		
Crotalaria incorporated in soil		
Ninth year		
0	5.84a	15.05ab
127.5	5.51a	15.02ab
45	6.29a	12.07a
90	6.06a	16.03b
Tenth year		
0.0	8.14a	21.51a
50.0	7.30a	21.30a
100.0	7.68a	19.96a
147.5	7.79a	21.91a

a) Means followed by different letters differ by Tukey test ($p < 0.05$). b) not determined.

Table 4
Nutrients content in maize diagnosis leaves treated with sewage sludge for ten consecutive years.

Element	0	50	100	147.5	Ideal range[(b)]
N (g kg^{-1})	27.4 a	27.8a	28.0a	27.6a	27.5–32.5
P (g kg^{-1})	2.7 b	2.6b	2.9ab	3.1a	2.5–3.5
K (g kg^{-1})	19.0 a	19.2a	20.5a	19.9a	17.5–22.5
Ca (g kg^{-1})	2.9 b	3.7ab	3.8ab	4.1a	2.5–4.0
Mg (g kg^{-1})	1.3 b	1.6ab	1.6ab	1.8a	2.5–4.0
S (g kg^{-1})	1.2 a	1.3a	1.2a	1.3a	1.5–2.0
B (mg kg^{-1})	21.3 a	23.2a	22.1a	23.1a	15–20
Cu (mg kg^{-1})	11.2 a	11.3a	11.0a	12.0a	0–20
Fe (mg kg^{-1})	63.5 b	66.4ab	89.0ab	91.0a	50–250
Mn (mg kg^{-1})	33.4 ab	31.4b	31.9b	42.9a	50–150
Zn (mg kg^{-1})	56.5 a	22.2b	20.8b	72.0a	15–50
Ni (mg kg^{-1})	0.92 ab	0.76b	1.5a	1.2ab	–
Mo (mg kg^{-1})	0.09 b	0.10ab	0.14a	0.09b	–

a) Means followed by different letters differ by Tukey test ($p < 0.05$).
b) Ideal range for Malavolta et al. (1997).

Continuous SS applications provided an increased concentration of available P in the soil. It was verified that, even in the soil that received a lower SS dose (50 Mg ha^{-1}), there was an increase of around 100% in relation to the soil that received only mineral fertilization. Soils from tropical climates generally present low P availability to plants (Rodrigues et al., 2016; Teles et al., 2017). All treatments were efficient in providing an adequate amount of P for maize plants. This indicates that SS is efficient in providing P to the plants (Lee et al., 2018) without the need for mineral fertilizer supplementation (Tontti et al., 2017). In other years, in the same experiment, similar results were observed for other crops (Ribeirinho et al., 2012; Macedo et al., 2012) in relation to P uptake.

The treatments that received SS needed to be supplemented with N from the mineral fertilizer, since not all the N contained in SS is bioavailable. However, our results showed that the N application from mineral fertilizers can be partially replaced by SS and reach large yields (Macedo et al., 2012; Motta and Maggiore, 2013; Yagmur et al., 2017). Our study also showed that even with the large imbalance between the amount of micronutrients contained in SS and the amount required by maize plants, SS proved to be efficient in providing micronutrients to maize plants (Pigozzo et al., 2000; Macedo et al., 2014; Nikzad et al., 2015).

5. Conclusions

Our results showed that for the evaluated biogeochemical variables, even after ten years of continuous SS application, agricultural Oxisol under a tropical climate presented similar responses to the cropped areas with mineral fertilizers. In this study, we verified that for the evaluation of the environmental impact of agricultural areas it is necessary to use a set of biogeochemical parameters, since individually they can present results that are difficult to interpret. The biogeochemical parameters used in this work were efficient to evaluate the variations caused by the use of SS in agriculture. Regarding the agronomic evaluation, the SS was efficient in providing total P, partial N and micronutrients for the maize crop.

Author contribution

All authors listed have made substantial, direct, and intellectual contribution to the work, and approved it for publication.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2018.06.254.

References

Alef, K., Nannipieri, P., 1995. Methods in Applied Soil Microbiology and Biochemistry. Academic Press, New York.

Alvarenga, P., Farto, M., Mourinha, C., Palma, P., 2016. Beneficial use of dewatered and composted sewage sludge as soil amendments: behaviour of metals in soils and their uptake by plants. Waste Biomass Valoriz. 7. https://doi.org/10.1007/s12649-016-9519-z.

Anderson, T.H., Domsch, K.H., 1986. Carbon assimilation and microbial activity in soil. Z. Pflanzenernähr. Bodenkd. J. Plant Nutr. Soil Sci. 149:457–468. https://doi.org/10.1002/jpln.19861490409.

Bhattacharyya, P., Tripathy, S., Chakrabarti, K., Chakraborty, A., Banik, P., 2008. Fractionation and bioavailability of metals and their impacts on microbial properties in sewage irrigated soil. Chemosphere 72:543–550. https://doi.org/10.1016/j.chemosphere.2008.03.035.

Brink, R.H., Dubach, P., Lynch, D.L., 1959. Measuremnt of carbohydrates in soils hydrolizates with antrone. Soil Sci., Baltimore 89, 157–166.

Busato, J.G., Leão, T.P., Baldotto, M.A., Canellas, L.P., 2012. Organic matter quality and dynamics in tropical soils amended with sugar industry residue. Rev. Bras. Ciênc. Solo 36:1179–1188. https://doi.org/10.1590/S0100-06832012000400012.

Canellas, L.P., Santos, G.D.E.A., Rumjanek, V.M., Moraes, A.A., Guridi, F., 2001. Distribution of the organic matter and humic acid characteristic in soils with addition of residues of urban origin. P. Agropec. Bras. 36:1529–1538. https://doi.org/10.1590/S0100-204X2001001200010.

Carvalho, C.S., Ribeirinho, V.S., Andrade, C.A., Grutzmacher, P., Pires, A.M.M., 2015. Chemical composition of sewage sludge organic matter. Braz. J. Agric. Sci. 10:413–419. https://doi.org/10.5039/agraria.v10i3a5174.

Castro, G.S.A., Crusciol, C.A.C., Calonego, J.C., Rosolem, C.A., 2015. Management impacts on soil organic matter of tropical soils. Vadose Zone J. 14:1–8. https://doi.org/10.2136/vzj2014.07.0093.

Duval, M.E., Galantini, J.A., Iglesias, J.O., Canelo, S., Martinez, J.M., Wall, L., 2013. Analysis of organic fractions as indicators of soil quality under natural and cultivated systems. Soil Tillage Res. 131:11–19. https://doi.org/10.1016/j.still.2013.03.001.

Eljarrat, E., Marsh, G., Labandeira, A., Barceló, D., 2008. Effect of sewage sludges contaminated with polybrominated diphenylethers on agricultural soils. Chemosphere 71: 1079–1086. https://doi.org/10.1016/j.chemosphere.2007.10.047.

Fernandes, S.A.P., Bettiol, W., Cerri, C., 2005. Effect of sewage sludge on microbial biomass, basal respiration, metabolic quotient and soil enzymatic activity. Appl. Soil Ecol. 30: 65–77. https://doi.org/10.1016/j.apsoil.2004.03.008.

Ferreira, A.C.C., Letie, L.F.C., Araújo, A.S.F., Eisenhauer, N., 2016. Land-use type effects on soil organic carbon and microbial properties in a semi-arid region of northeast Brazil. Land Degrad. Dev. 27:171–178. https://doi.org/10.1002/ldr.2282.

Frey, S.D., Elliott, E.T., Paustian, K., 1999. Application of the hexokinase–glucose-6-phosphate dehydrogenase enzymatic assay for measurement of glucose in amended soil. Soil Biol. Biochem. 31:933–935. https://doi.org/10.1016/S0038-0717(98)00176-X.

Harantová, L., Mudrák, O., Kohout, P., Elhottová, D., Frouz, J., Baldrian, P., 2018. Developtment of microbial community during primary succession in areas degraded by mining activities. Land Degrad. Dev.:1–11 https://doi.org/10.1002/ldr.2817.

Kirchmann, H., Börjesson, G., Kätterer, T., Cohen, Y., 2017. From agricultural use of sewage sludge to nutrient extraction: a soil science outlook. Ambio 46:143. https://doi.org/10.1007/s13280-016-0816-3.

Lee, C.G., Alvarez, P.J.J., Kim, H.G., Jeong, S., Lee, S., Lee, K.B., Lee, S.H., Choi, J.W., 2018. Phosphorous recovery from sewage sludge using calcium silicate hydrates. Chemosphere 193:1087–1093. https://doi.org/10.1016/j.chemosphere.2017.11.129.

Lloret, E., Pascual, J.A., Brodie, E.L., Bouskill, N.J., Insam, H., Juárez, M.F.D., Goberna, M., 2016. Sewage sludge addition modifies soil microbial communities and plant performance depending on the sludge stabilization process. Appl. Soil Ecol. 101:37–46. https://doi.org/10.1016/j.apsoil.2016.01.002.

Macedo, F.G., Melo, W.J., Merlino, L.C.S., Ribeiro, M.H., Camacho, M.A., Melo, G.M.P., 2012. Agronomic traits of corn fertilized with sewage sludge. Commun. Soil Sci. Plant Anal. 43:1790–1799. https://doi.org/10.1080/00103624.2012.684987.

Macedo, F.G., Melo, W.J., Merlino, L.C.S., Donha, R.M.A., Melo, G.M.P., Lavres Junior, J., 2014. Dynamics of zinc (Zn) and nickel (Ni) in a Cerrado Oxisol treated with sewage sludge for a long period. Aust. J. Crop Sci. 8, 1487–1494.

Machulla, G., 2003. Soil microbial indicators and their environmental significance. J. Soils Sediments 3:229. https://doi.org/10.1007/BF02988663.

Mahapatra, K., Ramteke, D.S., Paliwal, L.J., Narendra, K.N., 2013. Agronomic application of food processing industrial sludge to improve soil quality and crop productivity. Geoderma 207:205–211. https://doi.org/10.1016/j.geoderma.2013.05.014.

Evaluation of the nutritional status of plants: principles and applications. In: Malavolta, E., Vitti, G.C., Oliveira, S.A. (Eds.), Potafos, Piracicaba, 2° ed..

Masunga, R.H., Uzokwe, V.N., Mlay, P.D., Odeh, I., Singh, A., Buchan, D., Neve, S., 2016. Nitrogen mineralization dynamics of different valuable organic amendments commonly used in agriculture. Appl. Soil Ecol. 101:185–193. https://doi.org/10.1016/j.apsoil.2016.01.006.

McBride, M.B., 1995. Toxic metal accumulation from agricultural use of sludge: are USEPA regulations protective? J. Environ. Qual. 24:5–18. https://doi.org/10.2134/jeq1995.00472425002400010002x.

Melo, W.J., 1977. Dynamics of Carbon and Nitrogen Forms in a Purple Latosol Cultivated With *Sorghum bicolor* (L) Moench and with *Dolichos lablab* L. Isolated or in Intercropped Culture. Free teaching thesis. São Paulo State University (Unesp), School of Agricultural and Veterinarian Sciences, Jaboticabal, Brazil.

Melo, W.J., Aguiar, P.S., de Melo, G.M.P., de Melo, V.P, 2007. Nickel in a tropical soil treated with sewage sludge and cropped with maize in a long-term field study. Soil Biol. Biochem. 39 (6):1341–1347. https://doi.org/10.1016/j.soilbio.2006.12.010.

Menyailo, O.V., Lehmann, J., Cravo, M.S., Zech, W., 2003. Soil microbial activities in tree-based cropping systems and natural forests of the Central Amazon. Biol. Fertil. Soils 38:1–9. https://doi.org/10.1007/s00374-003-0631-4.

Motta, S.R., Maggiore, T., 2013. Evaluation of nitrogen management in maize cultivation grows on soil amended with sewage sludge and urea. Eur. J. Agron. 45:59–67. https://doi.org/10.1016/j.eja.2012.10.007.

Nikzad, E., Kalbasi, M., Hoodaji, M., Fallahzade, J., 2015. Effect of sewage sludge urgan application on concentration of Fe, Mn and some nutrient elemento in Parsley. Res. J. Soil Biol. 7:46–55. https://doi.org/10.3923/rjsb.2015.46.55.

Nogueira, T.A.R., Melo, W.J., Fonseca, I.M., Marques, M.O., He, Z.L., 2010. Barium uptake by maize plants as affected by sewage sludge in a long-term field study. J. Hazard. Mater. 181:1148–1157. https://doi.org/10.1016/j.jhazmat.2010.05.138.

Pancholy, S.K., Rice, E.L., 1973. Soil enzymes in relation to old filed sucession: amylase, celulose, invertase, dehydrogenase and urease. Soil Sci. Soc. Am. J. 37:47–50. https://doi.org/10.2136/sssaj1973.03615995003700010018x.

Patel, A., Patra, D.D., 2014. Influence of heavy metal rich tannery sludge on soil enzymes vis-à-vis growth of *Tagetes minuta*, an essential oil bearing crop. Chemosphere 112: 323–332. https://doi.org/10.1016/j.chemosphere.2014.04.063.

Paz-Ferreiro, J., Gascó, G., Gutiérrez, B., Méndez, A., 2012. Soil biochemical activities and the geometric mean of enzyme activities after application of sewage sludge and sewage sludge biochar to soil. Biol. Fertil. Soils 48:511. https://doi.org/10.1007/s00374-011-0644-3.

Pigozzo, A.T.J., Gobbi, M.A., Lenzi, E., Luchese, E.B., 2000. Effects of the application of sewage sludge and petrochemical residue in maize culture as source of micronutrients on soils of Paraná state. Braz. Arch. Biol. Technol. 43:143–149. https://doi.org/10.1590/S1516-89132000000200002.

Raij, B.V., Cantarella, H., Quaggio, J.A., Furlani, A.M.C, 1997. Recommendations for Fertilizer and Lime to the State of Sao Paulo. 2nd ed. Agronomic Institute, Campinas, pp. 56–59.

Ribeirinho, V.S., Melo, W.J., Silva, D.H., Figueiredo, L.A., Melo, G.M.P., 2012. Soil fertility, nutritional status, and yield of sunflower fertilized with sewage sludge. Pesqui. Agropec. Trop. 42:166–173. https://doi.org/10.1590/S1983-40632012000200002.

Rodrigues, M., Pavinato, P.S., Withers, P.J.A., Teles, A.P.B., Herrera, W.F.B., 2016. Legacy phosphorus and no tillage agriculture in tropical oxisols of the Brazilian savanna. Sci. Total Environ. 542:1050–1061. https://doi.org/10.1016/j.scitotenv.2015.08.118.

Ross, D.J., 1965. Seasonal studies of oxygen uptake of some pasture soils and activities of enzymes hydrolyzing sucrose and starch. J. Soil Sci. 16:73–85. https://doi.org/10.1111/j.1365-2389.1965.tb01421.x.

Ross, D.J., 1987. Assays of invertase activity in acid soil. Influence of buffers. Plant Soil 97: 285–289. https://doi.org/10.1007/BF02374952.

Ross, S.M., 1993. Organic matter in tropical soils: current conditions, concerns and prospects for conservation. Prog. Phys. Geogr. 17:265–305. https://doi.org/10.1177/030913339301700301.

Santos, L.M., Simoes, M.L., Melo, W.J., Martin-Neto, L., Pereira-Filho, E.R., 2010. Application of chemometric methods in the evaluation of chemical and spectroscopic data on organic matter from Oxisols in sewage sludge applications. Geoderma 155, 121–127.

Santos, J.A., Nunes, L.A.P.L., Melo, W.J., Araújo, A.S.F., 2011. Tannery sludge compost amendment rates on soil microbial biomass in two different soils. Eur. J. Soil Biol. 47:146–151. https://doi.org/10.1016/j.ejsobi.2011.01.002.

Santos, V.B., Leite, L.F.C., Nunes, L.A.P.L., Melo, W.J., 2012. Soil microbial biomass and organic matter fractions during transition from conventional to organic farming systems. Geoderma 170:227–231. https://doi.org/10.1016/j.geoderma.2011.11.007.

Scotti, R., Bonanomi, G., Scelza, R., Zoina, A., Rao, M.A., 2015. Organic amendments as sustainable tool to recovery fertility in intensive agricultural systems. J. Soil Sci. Plant Nutr. 15:333–352. https://doi.org/10.4067/S0718-95162015005000031.

Sharma, B., Sarkar, A., Singh, P., Singh, R.P., 2017. Agricultural utilization of biosolids: a review on potential effects on soil and plant grown. Waste Manag. 64:117–132. https://doi.org/10.1016/j.wasman.2017.03.002.

Silva, M.D.M., Barajas-Aceves, M., Araújo, A.S.F., Araújo, F.F., Melo, W.J., 2014. Soil microbial biomass after three-year consecutive composted tannery sludge amendment. Pedosphere 24:469–475. https://doi.org/10.1016/S1002-0160(14)60033-3.

Silveira, M.L.A., Alleoni, L.R.F., Guilherme, L.R.G., 2003. Biosolids and heavy metals in soils. Sci. Agric. 60:793–806. https://doi.org/10.1590/S0103-90162003000400029.

Soriano-Disla, J.M., Gómez, I., Navarro-Pedreño, J., Jordán, M.M., 2014. The transfer of heavy metals to barley plants from soils amended with sewage slduge with diferente heavy metal burdens. J. Soils Sediments 14:687. https://doi.org/10.1007/s11368-013-0773-4.

Tejada, M., Gómez, I., Fernández-Boy, E., Díaz, M.J., 2014. Effects of sewage sludge and *Acacia dealbata* composts on soil biochemical and chemical properties. Commun. Soil Sci. Plant Anal. 45:570–580. https://doi.org/10.1080/00103624.2013.874017.

Teles, A.P.B., Rodrigues, M., Herrera, W.F.B., Solthangheisi, A., Sartor, L.R., Withers, P.J.A., Pavinato, P.S., 2017. Do cover crops change the lability of phosphorus in a clayey subtropical soil under different phosphate fertilizers? Soil Use Manag. 33:34–44. https://doi.org/10.1111/sum.12327.

Tontti, T., Poutiainen, H., Heinonen-Tanski, H., 2017. Efficiently treated sewage sludge supplemented with nitrogen and potassium is a good fertilizer for cereals. Land Degrad. Dev. 28:742–751. https://doi.org/10.1002/ldr.2528.

Tripathya, S., Bhattacharyya, P., Mohapatra, R., Som, A., Doyel, C., 2014. Influence of different fractions of heavy metals on microbial ecophysiological indicators and enzyme activities in century old municipal solid waste amended soil. Ecol. Eng. 70:25–34. https://doi.org/10.1016/j.ecoleng.2014.04.013.

USEPA - United States Environmental Protection Agency, 1996. Acid digestion of sediments, sludges and soils. Metod 3050b. EPA, Washington 12p.

Vance, E.D., Brookes, P.C., Jenkinson, D.S., 1987. An extraction method for measuring soil microbial biomass C. Soil Biol. Biochem. 19:703–707. https://doi.org/10.1016/0038-0717(87)90052-6.

Vieira, R.F., Moriconi, W., Pazianotto, R.A.A., 2014. Residual and cumulative effects of soil application of sewage sludge on corn productivity. Environ. Sci. Pollut. Res. 21: 6472–6481. https://doi.org/10.1007/s11356-014-2492-9.

Wang, X., Chen, T., Ge, Y., Jia, Y., 2008. Studies on land application of sewage sludge and its limiting factors. J. Hazard. Mater. 160:554–558. https://doi.org/10.1016/j.jhazmat.2008.03.046.

Wang, L., Yang, F.E.Y., Yuan, J., Raza, W., Huang, Q., Shen, Q., 2016. Long-term application of bioorganic fertilizers improved soil biochemical properties and microbial communities of an apple orchard soil. Front. Microbiol. 7:1893. https://doi.org/10.3389/fmicb.2016.01893.

Yada, M.M., Melo, W.J., Mingotte, F.L.C., Melo, V.P., Melo, G.M.P., 2015. Chemical and biochemical properties of oxisols after sewage sludge application for 16 years. Rev. Bras. Ciênc. Solo 39:1303–1310. https://doi.org/10.1590/01000683rbcs20140728.

Yagmur, M., Arpalı, D., Gulser, F., 2017. The effects of sewage sludge treatment on triticale straw yield and its chemical contents in rainfed condition, J. Anim. Plant Sci. 27, 971–977.

Zhang, F.P., Li, C.F., Tong, L.G., Yue, L.X., Li, P., Ciren, Y.J., Cao, C.G., 2010. Response of microbial characteristics to heavy metal pollution of mining soils in central Tibet, China. Appl. Soil Ecol. 45:144–151. https://doi.org/10.1016/j.apsoil.2010.03.006.