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Long-term sewage sludge application in a tropical Oxisol: Effects on acidity and availability of micronutrients

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ABSTRACT: The objective of this study was to evaluate the effects of acidity and availability of micronutrients on a sandy clay loam Oxisol grown with soybeans (summer) and black oat (autumn/winter), after long-term applications of biodigested sewage sludge (BS) and centrifuged sewage sludge (CS). The experiment was conducted in Botucatu, SP, Brazil, from 2002 to 2014, using a randomized block design, in 2×4 factorial scheme, with four repetitions. Treatments corresponded to six biennial applications of BS and CS at four doses: 0, 2, 4 and 8 Mg ha^{-1} (dry basis). Soil samples up to 0.60 m depth were taken after twelve years, and pH, potential acidity, exchangeable acidity and micronutrient contents were evaluated. The CS applications of 4 and 8 Mg ha^{-1} led to pH values within the recommended range up to depths of 0.10 and 0.20 m, respectively, whereas BS applications did not result in adequate pH values in any layer. The increased soil pH caused by CS applications resulted in a proportional reduction in soil potential acidity, whereas BS applications resulted in high value of potential acidity (70 mmol$_c$ dm^{-3}) in all layers. The CS applications resulted in low Al^{3+} concentrations up to 0.20 m depth, whereas BS applications led to high Al^{3+} concentrations from the 0.05-0.10 m layer. The successive applications of both sludges resulted in Cu^{2+}, Fe^{2+}, Mn^{2+} and Zn^{2+} concentrations that exceeded the maximum values allowed in all soil layers. Low boron concentrations were found in the soil, and pH was the determinant factor for it.

Key words: urban waste, soil acidity, soil fertility

Aplicações de lodos de esgoto em um Oxisol: Efeitos na acidez e disponibilidade de micronutrientes

RESUMO: Objetivou-se avaliar a acidez e a disponibilidade de micronutrientes em um Oxisol, de textura média cultivado com soja (verão) e aveia-preta (outono/inverno), após aplicações de lodo de esgoto biodigerido (LB) e de lodo de esgoto centrifugado (LC). O experimento foi conduzido em Botucatu, SP, Brasil, de 2002 a 2014, em delineamento experimental de blocos ao acaso, em esquema fatorial 2 × 4, com quatro repetições. Os tratamentos corresponderam a seis aplicações bienais de LB e LC em quatro doses: 0, 2, 4 e 8 Mg ha^{-1} (base seca). Após doze anos coletou-se amostras de solo até a profundidade de 0,60 m para avaliação de pH, acidez potencial, acidez trocável e teor de micronutrientes. Aplicações de 4 e 8 Mg ha^{-1} de LC resultaram, respectivamente, em valores de pH dentro da faixa recomendada até a profundidade de 0,10 e 0,20 m, enquanto aplicações de LB não resultaram em valores de pH adequados para nenhuma camada. O aumento do pH do solo pelas aplicações de LC acarretou diminuição proporcional da acidez potencial do solo, enquanto aplicações de LB resultaram em alto valor de acidez potencial (70 mmol$_c$ dm^{-3}) em todas as camadas. Aplicações de LC acarretaram em baixos teores de Al^{3+} até a profundidade de 0,20 m, enquanto as aplicações de LB acarretaram em altos teores de Al^{3+} a partir da camada 0,05-0,10 m. Sucessivas aplicações de ambos os lodos resultaram em teores de Cu^{2+}, Fe^{2+}, Mn^{2+} e Zn^{2+} que ultrapassaram os valores máximos permitidos em todas as camadas do solo. O boro apresentou baixos teores no solo, e o pH foi o fator determinante para tal.

Palavras-chave: resíduo urbano, acidez do solo, fertilidade do solo

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INTRODUCTION

The high costs of commercial fertilizers and increase in environmental pollution make the use of sewage sludge in agriculture an attractive option, from both the economic and environmental points of view, because of the cycling of nutrients (Santos et al., 2011). Indeed, several studies have demonstrated that, among the alternatives for the final disposal of sewage sludge, the one for agricultural and forest purposes presents itself as one of the most convenient, since this waste is rich in organic matter and nutrients (Abreu et al., 2017; Oliveira et al., 2018) for plants, and its application can be recommended as soil conditioner and/or fertilizer (Camargo et al., 2013).

Corrêa et al. (2009b) reported that the superficial application of centrifuged sewage sludge in an Oxisol resulted in the correction of soil acidity, causing displacement of Ca^{2+}, increase of pH and reduction of Al^{3+} up to 0.40 m depth. Pigozzo et al. (2008) observed that sewage sludge application caused an increase in Fe, Mn, Cu, Zn concentrations and in CEC, besides reduction of pH in an Oxisol (0-0.20 m).

Despite all the benefits already known of the use of sewage sludge in agriculture, the characteristics of the sludge and its inadequate management may cause imbalance in soils (Moretti et al., 2015). Hence, it is important to evaluate how successive applications of sludge types act on soil acidity and micronutrients availability. Therefore, the present study aimed to evaluate micronutrients concentrations, pH, potential acidity and exchangeable acidity after 12 years of biennial applications of centrifuged sewage sludge and biodigested sewage sludge.

MATERIAL AND METHODS

The experiment, which started in August 2002, was carried out at Lageado Experimental Farm, belonging to the Faculdade de Ciências Agronômicas (FCA/UNESP), located in the municipality of Botucatu, SP, Brazil. The soil of the experimental area was classified as Oxisol and its particle-size analysis (Claessen et al., 1997) showed concentrations of 545 g kg^{-1} of sand, 108 g kg^{-1} of silt and 347 g kg^{-1} of clay. The experimental area has a gentle relief and altitude of 740 m. The climate prevailing in the region is Cwa (Alvares et al., 2013), characterized as high-altitude tropical climate, with dry winter and hot rainy summer. The annual volume of precipitation is approximately 1,600 mm, with irregular distribution.

The experimental design used was randomized blocks in 2 × 4 factorial scheme, with four repetitions. Each plot was 6 m wide and 7 m long. Treatments corresponded to biennial applications of two urban waste types: biodigested sludge and centrifuged sludge, at four doses: 0, 2, 4 and 8 Mg ha^{-1} (dry basis). After the first application, in 2002, other five applications were carried out in 2005, 2007, 2009, 2011 and 2013, all manually performed, on surface and without incorporation. All applications used biodigested sludge (BS), obtained in biodigester with addition of polyelectrolytes - and centrifuged sludge (CS), which receives calcium oxide (CaO) in its processing. The results of the basic chemical analysis and the analysis for heavy metals (on dry basis) of the sludges used in the last application are presented in Table 1.

Table 2 shows the accumulated quantities of some elements that were added to the system via application of wastes along the 12 years of experiment. The calculations were carried out based on the quantity applied and chemical analysis of the wastes used in each one of the six applications. Along the years of experiment, the production system used was characterized by the cultivation of soybean [*Glycine max* (L.) Merrill] in the summer season and black oat (*Avena sativa* L.) during the autumn/winter. It is worth pointing out that the plots did not receive limestone at any moment.

The final soil sampling, whose results compose the present study, was carried out in October 2014, 12 years after the beginning of the experiment. Samples were collected with a probe-type auger, in the layers of 0-0.05; 0.05-0.10; 0.10-0.20; 0.20-0.40 and 0.40-0.60 m, obtaining three single samples per plot. The sampled soil was homogenized and placed in plastic bags, dried at ambient temperature, pounded to break up clods, sieved through 2-mm-mesh sieve and then stored in paper bags. Basic chemical analysis was performed in the Laboratory of Soil Chemistry and Fertility of the Departamento de Recursos Naturais of FCA/UNESP. The levels of pH, Al^{3+} and $H^+ + Al^{3+}$ were analyzed according to the methodologies described by Raij et al. (2001). The micronutrients Cu^{2+}, Fe^{2+}, Mn^{2+} and Zn^{2+} were analyzed by extraction with DTPA-TEA (Camargo et al., 1986) and B was determined by the barium chloride-microwave method (Abreu et al., 1998).

The data were subjected to analysis of variance. When significant effects were found by the F test, the sewage sludges types were compared by Tukey test at p ≤ 0.05, whereas the doses were subjected to regression analysis, and exponential equations were fitted according to the significance of the regression parameters, F value and adjusted coefficient of

Table 1. Basic chemical composition and heavy metals of the sludges applied in 2013

Sewage sludge	OM[1]	Total-C	N	C/N	P	K	Ca	Mg	S
	(g kg^{-1})				(g kg^{-1})				
Biodigested	190	110	12	9/1	5.2	0.7	7	2	6
Centrifuged	170	90	9	10/1	3.5	0.8	43	2	2
	pH $CaCl_2$	Na	B	Cu	Fe	Mn	Zn	Mo	Ni
		(mg kg^{-1})							
Biodigested	8.4	211	134	239	7769	134	459	3.16	197
Centrifuged	12.6	289	205	90	3120	51	168	1.30	45
	Se	As	Ba	Cd	Pb	Cr	Hg		
	(mg kg^{-1})								
Biodigested	<1.4	5.88	626	7.45	125	364	<0.65		
Centrifuged	<1.4	2.83	295	1.27	24	132	<0.65		

[1] OM - Organic matter

Table 2. Accumulated quantity of elements added to the soil via sewage sludge application along 12 years

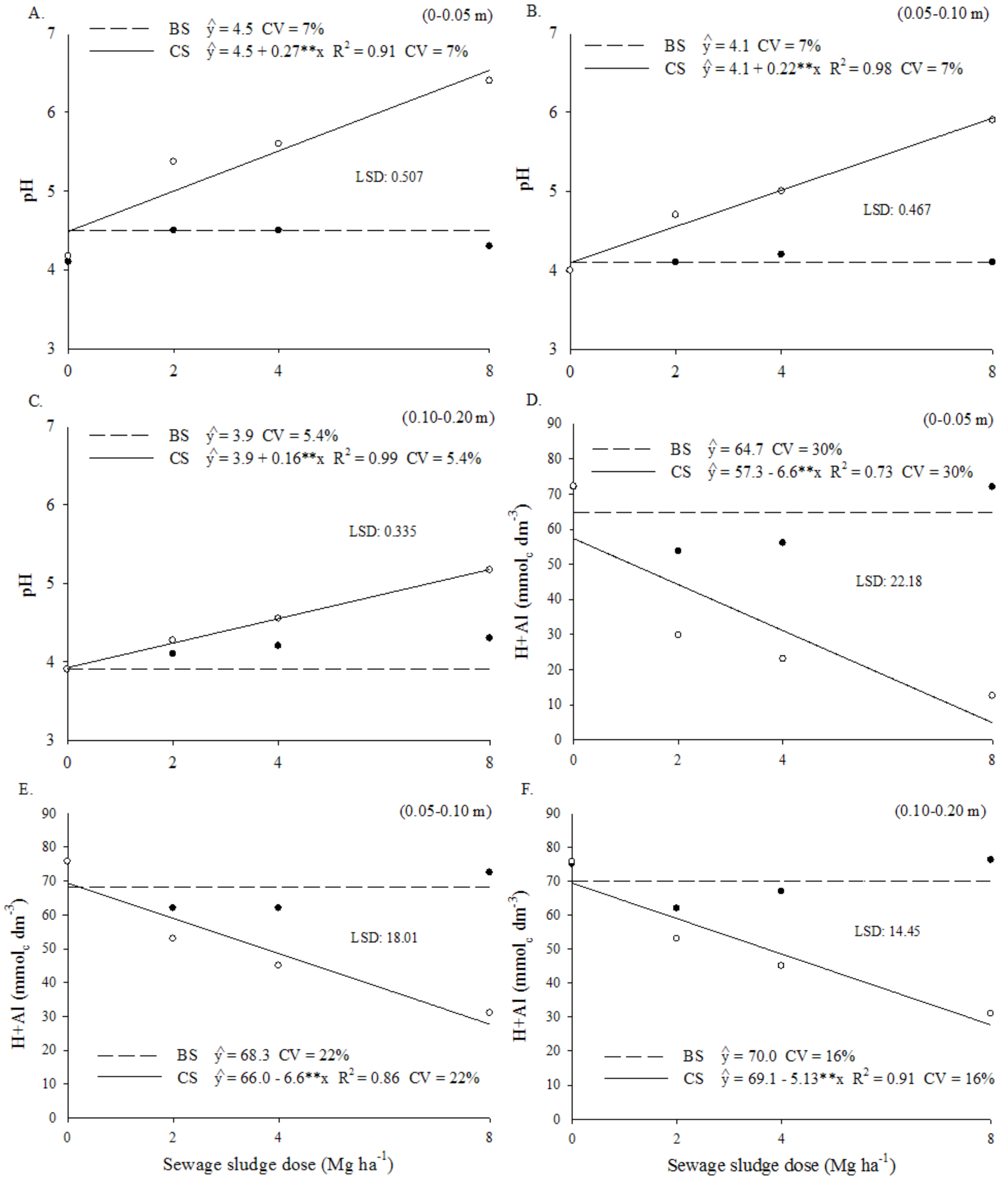
Sewage sludge type	Biennial dose	Accumulated dose	OM¹	Cu	Fe	Mn	Zn	B
	(Mg ha⁻¹)			(kg ha⁻¹)				
Biodigested	2	12	4.85	6.8	286	2.6	30	1.6
	4	24	9.70	14	572	5.2	61	3.2
	8	48	19.4	27	1144	10	121	6.4
Centrifuged	2	12	2.16	2.4	28	1.1	5.5	2.5
	4	24	4.32	4.8	55	2.1	11	4.9
	8	48	8.63	9.5	110	4.2	22	9.8

[1] OM - Organic matter

determination (R^2). The analyses were carried out in the statistical program Sisvar (Ferreira, 2014), and the figures were created in the program SigmaPlot (Sigma plot, 2006). The classification of values and citation of threshold values of the soil acidity and micronutrient availability, for discussion purposes, were carried out according to the levels described by Raij et al. (2001).

RESULTS AND DISCUSSION

In the first three layers, the interaction between factors was significant for soil pH (Figures 1A, B and C) and the data fitted to increasing linear equations. As CS doses increased, pH increased from 4.5 to 6.1 (0-0.05 m layer), from 4.1 to 5.8 (0.05-0.1 m layer) and from 3.9 to 5.1 (0.1-0.2 m layer). In these



LSD - Corresponds to the least significant difference (between sludges); ** Significant at $p \leq 0.01$ by F test

Figure 1. Values of soil pH (A, B and C) and potential acidity (H + Al) (D, E and F) in the 0-0.05, 0.05-0.10 and 0.10-0.20 m layers of an Oxisol as a function of the application of doses of biodigested sewage sludge (BS) and centrifuged sewage sludge (CS)

same layers, however, the pH did not vary as a function of the BS doses and remained at 4.4, 4.1 and 3.9, respectively, with differences between the wastes at the doses of 2, 4 and 8 Mg ha^{-1}. The highest dose of CS led to pH values that were 38, 41 and 39% higher than those caused by the same dose of BS in the three layers, respectively. More important than the percentage increase between the wastes is the value of pH obtained. Thus, in the first three soil layers and considering the ideal pH range for the development of most crops (pH between 5.0 and 6.0), it can be observed that CS at dose of 8 Mg ha^{-1} promoted pH within the recommended range and, at the dose of 4 Mg ha^{-1}, adequate values up to the 0.10 m layer. However, BS application did not result in any value of pH within such range. In the other layers, there was no effect on soil pH and its mean values were 4.2 (0.2-0.4 m) and 4.0 (0.4-0.6 m).

The effect of CS doses on pH up to 0.2 m depth occurred fundamentally because this waste is treated with lime (CaO) in the sewage treatment plant. Once in the soil, CaO is solubilized and dissociated into $Ca^{2+} + 2OH^-$, which binds to the H^+ of the soil solution forming water and reducing the active acidity. Oliveira et al. (2002) also observed an increase of pH in an Oxisol after applications of sewage sludge treated with lime. The CaO also makes toxic Al^{3+} insoluble through the formation of $Al(OH)_3$, besides improving the aggregation of soil particles (Corrêa et al., 2009a), because Ca^{2+} makes connections between the carboxylic and phenolic groups of organic matter and soil colloids (Castro Filho et al., 1998). The improvement in the aggregation state of soil particles decreases their density and increases aeration and water retention (Portella et al., 2012), conditions that favor root development and influence soil microbial activity (Iamaguti et al., 2015).

For the concentration of Al^{3+}, there was a difference between the types of sludge up to the 0.2-0.4 m layer, with concentrations always lower under CS application. In surface, the Al^{3+} concentration corresponded to 1.6 mmol$_c$ dm^{-3} when CS was applied (3.0 mmol$_c$ dm^{-3} lower than the concentration found with BS application). In the layers of 0.05-0.1, 0.1-0.2 and 0.2-0.4 m, the Al^{+3} concentrations as a function of the application of CS and BS, in this order, corresponded to 3.0 and 8.0, 5.4 and 10.5, and 8.12 and 11.3 mmol$_c$ dm^{-3}. In the 0.4-0.6 m layer, there was no effect of the treatments and the Al^{3+} concentration remained at 11.3 mmol$_c$ dm^{-3}. Thus, with the application of CS, there were low Al^{3+} concentrations up to the 0.1-0.2 m layer, whereas the application of BS resulted in high Al^{3+} concentrations from the 0.05-0.1 m layer. It is important to remember that the high concentration of this element may lead to inhibition of root growth as a result of the alteration in cell division in the meristematic region and of cell expansion in the root elongation zone (Nichol & Oliveira, 2011).

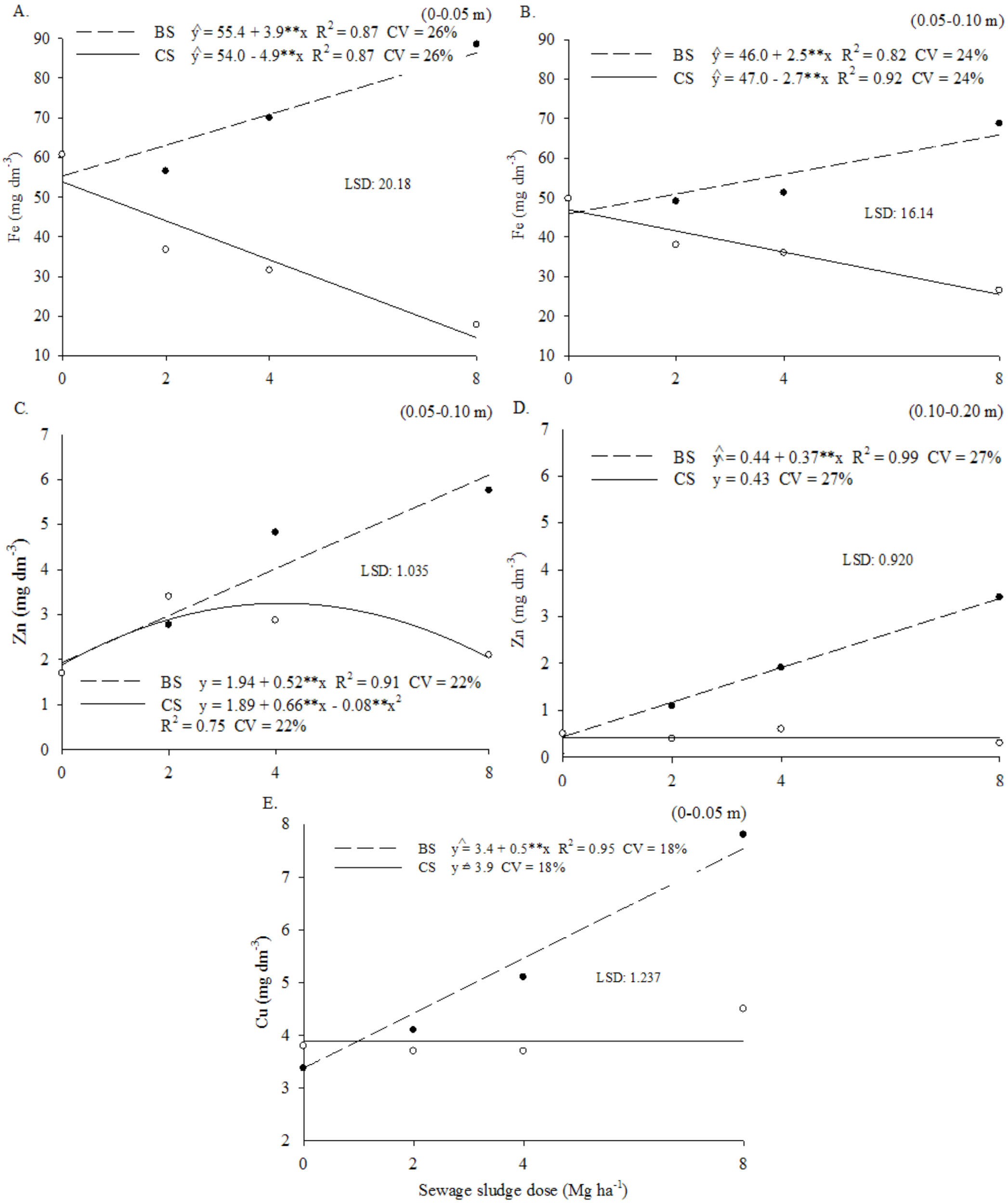
Corrêa et al. (2007) reported increase in soil pH and base saturation up to 0.4 m depth after only three months of application of centrifuged sludge. According to the authors, this result occurred because of the higher concentration of compounds resulting from the neutralization reaction, which allow the negative charges of the soil originating from the pH increase to be neutralized, which makes the excess of cations available in the solution of the soil in a shorter time, making possible the displacement of the alkalinizing front in subsurface.

Due to the previously described effects, the $H^+ + Al^{3+}$ concentrations also decreased when CS was applied. Significant interaction was observed between factors for potential acidity in the first three layers (Figures 1D, E and F), with fit to decreasing linear equations for CS, while the application of BS resulted in $H^+ + Al^{3+}$ concentrations that remained at 64.7, 68.3 and 70.0 mmol$_c$ dm^{-3}, which were significantly higher than those observed with the application of any CS dose. Thus, with the dose of 2 Mg ha^{-1}, the $H^+ + Al^{3+}$ concentration was 1.3 and 1.2 times lower in the first two layers, respectively, under CS application. With the dose of 4 Mg ha^{-1}, this difference was larger, 2.2 times lower in the superficial layer and 1.4 times lower in the two subsequent layers. The greatest difference between the wastes, however, occurred with the application of the dose of 8 Mg ha^{-1}, when the value of $H^+ + Al^{3+}$ was 9.3 times lower in the superficial layer with the use of CS compared to BS. In the following two layers, this value was 2.3 times lower for this dose of CS. In the 0.2-0.4 m layer, the type of sludge had an effect on the levels of potential acidity, which were equal to 68.4 and 59.5 mmol$_c$ dm^{-3} for the application of BS and CS, respectively. In the 0.4-0.6 m layer, as observed for the Al^{3+} concentration, there was no effect of the treatments, and the $H^+ + Al^{+3}$ concentration corresponded to 86.0 mmol$_c$ dm^{-3}. It is important to note that, with the increase of soil pH (due to CS application), there was a proportional reduction in the potential acidity, a relationship already reported in the literature (Nicolodi et al., 2008).

For the concentration of Fe^{2+}, the interaction between factors was significant in the first two layers (Figures 2A and B) and, in the superficial layer, the Fe^{2+} concentration (69 mg dm^{-3}) was 2 times higher with the dose of 4 Mg ha^{-1} and 5 times higher (81 mg dm^{-3}) with the dose of 8 Mg ha^{-1} for BS application. In the following layer, the Fe^{2+} concentration was also significantly higher with these two doses of BS and the values (52 and 62 mg dm^{-3}) were 1.5 and 2 times higher, respectively. In 0.1-0.2, 0.2-0.4 and 0.4-0.6 m layers, the Fe^{2+} concentration showed no difference as a function of the treatments and remained, respectively, at 34.3, 24.4 and 23.2 mg dm^{-3}. In all layers, the Fe^{2+} concentrations were higher than 12 mg dm^{-3}, which indicates a high concentration of the element.

The Mn^{2+} concentration varied according to the type of waste and only in the first three layers of the soil. The concentrations of this micronutrient were higher with BS application and corresponded to 19.2, 12.5 and 11.5 mg kg^{-1}, respectively, in layers of 0-0.05, 0.05-0.1 and 0.1-0.2 m, whereas for CS, the concentrations in these layers were 17.2, 10.8 and 7.9 mg kg^{-1}, respectively. In the 0.2-0.4 and 0.4-0.6 m layers, the mean Mn^{2+} concentrations corresponded to 6.8 and 5.5 mg kg^{-1}, respectively.

The Zn^{2+} concentration in the soil was also higher under BS application. Thus, in the 0-0.05 m layer, there was an effect of the type of waste and, for BS and CS, the Zn^{2+} concentrations corresponded to 6.08 and 5.07 mg kg^{-1}, respectively. In the 0.05-0.1 m layer, there was a significant interaction between factors, with increased Zn^{+2} concentrations from 1.94 to 6.10 mg kg^{-1} as a function of the increase in BS doses, and quadratic effect of CS application, whose Zn^{2+} concentration increased up to the estimated dose of 4.1 Mg ha^{-1}, when it corresponded to 3.24 mg kg^{-1} (Figure 2C), differing from BS for the doses of 4 and 8 Mg ha^{-1}. In the 0.1-0.2 m layer, there was also significant interaction between factors, and the Zn^{2+} concentration remained at mean value of 0.43



LSD: corresponds to the least significant difference (between sludges); **Significant at $p \leq 0.01$ by F test

Figure 2. Iron (Fe) concentrations (mg dm^{-3}) in the 0-0.05 (A) and 0.05-0.10 m layers (B), zinc (Zn) concentrations (mg dm^{-3}) in the 0.05-0.10 (C) and 0.10-0.20 m layers (D), and copper (Cu) concentrations (mg dm^{-3}) in the 0-0.05 m layer (E) of an Oxisol as a function of the application of doses of biodigested sewage sludge (BS) and centrifuged sewage sludge (CS)

mg kg^{-1} for the CS application, differing from BS at the doses of 4 and 8 Mg ha^{-1}, at which the concentrations of the element were 4.6 and 7.4 times higher (Figure 2D). In the 0.2-0.4 m layer, there was still an effect of the type of sludge on Zn^{2+} concentration, which was 2.2 times higher with BS application (0.77 mg kg^{-1}). In the last layer, there was no effect of the treatments and Zn^{+2} concentration remained at 0.62 mg kg^{-1}. For the Cu^{2+} concentration, however, there was significant interaction between factors in the superficial layer, so with the increase of BS doses, its concentration increased from 3.4 to 7.4 mg kg^{-1}, while for CS its concentration remained at 3.9 mg kg^{-1}, differing from BS at the doses 4 and 8 Mg ha^{-1} (Figure 2E). In the layers of 0.05-0.1, 0.1-0.2, 0.2-0.4 and 0.4-0.6 m, the Cu^{+2} concentration did not vary, remaining at 3.6, 3.2, 3.3 and 3.4 mg kg^{-1}, respectively.

The availability of micronutrients (Cu^{2+}, Fe^{2+}, Mn^{2+} and Zn^{2+}) was higher after application of BS, probably because with this waste the soil pH remained close to 4.0 in all layers, a condition in which there is greater availability of these micronutrients. When the pH exceeds this value, cationic micronutrients become unavailable (Abreu et al., 2007). Borges & Coutinho (2004) observed that the addition of limestone in sewage sludge also promoted the elevation of soil pH, and that this resulted in the redistribution of Cu^{2+}, Fe^{2+}, Mn^{2+} and Zn^{2+} from the exchangeable fraction to less available forms (organic and/or of oxides). In all layers, the concentration of Fe^{2+} exceeded the value of 12 mg dm^{-3}, which indicates high concentration of the element in soil. The high concentrations of Fe^{2+} can be justified by the fact that the sludge showed high concentration of this element, especially BS, which had 2.5 times more Fe^{2+} compared to CS (Table 2). Thus, with the highest dose of BS and CS, the Fe^{2+} concentration exceeded the threshold of 12 mg dm^{-3} respectively by 7.0 and 1.5 times in the 0-0.05 m layer. Hence, regardless of the type or dose of sludge, the concentrations of Mn^{2+}, Zn^{2+} and Cu^{2+} in the soil are also considered high, because they exceed the threshold values of 5.0, 1.2 and 0.8 mg kg^{-1}, respectively. Both types of sludge also had a high quantity of these micronutrients, but BS has 2.6 times more Mn^{2+}, Zn^{2+} and Cu^{2+} when compared to CS (Table 2). Thus, with the highest dose of BS, the concentrations of Cu^{2+}, Fe^{2+}, Mn^{2+} and Zn^{2+} exceeded the values established as high respectively by 9, 7, 2 and 5 times.

The B concentration in the soil was not altered by any of the factors and corresponded to 0.19, 0.14, 0.14, 0.13 and 0.21 mg kg^{-1} in the layers of 0.05-0.1, 0.1-0.2, 0.2-0.4 and 0.4-0.6 m, respectively. Up to the 0.4 m layer of the soil, B concentration is considered low, differently from the results observed for the other micronutrients. Although B is bound to soil OM (Silva & Mendonça, 2007) and the sludges had high OM concentrations, especially BS (Table 2), the pH was the most determinant factor in the concentration of this element. The adsorption of B by Fe^{2+} and Al^{3+} oxides is pH-dependent and is higher within the pH range from 6 to 9 (Abreu et al., 2007). Thus, as the pH increased when the CS was applied, although this waste contained 1.5 times more B than BS, there was probably an effect of adsorption of the element, which led to the absence of change in its concentration in the soil, thus requiring supplementation of this element when CS or BS is applied.

Conclusions

1. Successive applications of 4 and 8 Mg ha^{-1} of centrifuged sewage sludge resulted, respectively, in pH values within the recommended range up to 0.10 (pH of 5.0) and 0.20 m (pH of 5.1) depths.

2. Biodigested sewage sludge applications did not result in adequate pH values in any soil layer (up to 0.60 m), but resulted in potential acidity close to the value of 70 mmol$_c$ dm^{-3}.

3. Centrifuged sewage sludge applications resulted in low exchangeable acidity up to the 0.20 m depth, whereas biodigested sewage sludge applications resulted in high Al^{+3} concentrations from the 0.05 to 0.60 m depth.

4. Successive applications of centrifuged and biodigested sewage sludge resulted in Cu^{2+}, Fe^{2+}, Mn^{2+} and Zn^{2+} concentrations above the maximum values allowed in all soil layers. On the other hand, boron concentrations were low in the soil and pH was the determinant factor for it.

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