# Content of Heavy Metals in Soil and in Pineapple Fertilized With Sewage Sludge

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Received: February 8, 2019	Accepted: April 21, 2019	Online Published: June 30, 2019
doi:10.5539/jas.v11n9p281	URL: https://doi.org/10.5539	/jas.v11n9p281

#### Abstract

The usage of sewage sludge in agriculture can increase the levels of heavy metals in the soil, compromising their use as fertilizer. The objective of this study was to evaluate the contamination of soil and pineapple by heavy metals after the application of treated sewage sludge by different forms in three orders of soils under greenhouse conditions. The treatments, in a factorial scheme  $7 \times 3$  were distributed in a randomized complete block design with three replications, corresponding to seven fertilization management: soil without fertilization, chemical fertilization with composted sludge sewage, fertilization with vermicompost sewage sludge, fertilization with solarized sewage sludge sewage, combined with three orders of soils: Cambisol, Nitisol and Acrisol. The Zn, Cu, Cr, Pb, Ba, Cd, Ni, As and Se contents were analyzed in the soil, in the leaf of greater length (D leaf) and in the fruit pulp of the pineapple. The Acrisol provided more favorable conditions to the increase in the availability and absorption of Pb and As by D leaf and fruit of the pineapple. Fertilization with composted, vermicompost and solarized sewage sludge provided high levels of Pb, As, Zn in leaf D and in the pineapple fruit, relating the interference of sludge stabilization process with the absorption of metals by pineapple fruits.

Keywords: Ananas comosus var. comosus, biosolid, organic fertilization, solid waste

#### 1. Introduction

The agricultural usage of sewage sludge or products from its stabilization is an important alternative for the reduction in production costs and to the increase of the productivity of several crops, since this residue promotes physical and chemical improvements of the soil due to its high levels of nutrients and organic matter (Nascimento et al., 2004; Lemainski & Silva, 2006; Zuba Junio et al., 2012; Ribeirinho et al., 2012).

Nitrogen is the most important plant nutrient in the sewage sludge. It is the element that determines the dose of sludge to be applied in crop fertilization when there is no surplus of heavy metals (Brasil, 2006). However, the application of sewage sludge based only on the nitrogen concentration can cause nutritional imbalance, since the concentration of potassium in this residue is very low, which may limit the production of the plants. On the other hand, in tropical conditions, the application of sewage sludge based on potassium concentration alone can raise nitrogen levels in the soil to very high levels, increasing the risk of contamination of the water table with this element. So, the application of sewage sludge based on nitrogen concentration, however, complemented with phosphorus and potassium, seems to be the best management for this fertilizer use (Tontti et al., 2017). In this context, the pineapple presents a high demand for N (Caetano et al., 2013; Cardoso et al., 2013) and sludge can replace it, which is an important fertilizer economy for the crop besides providing an adequate destination of the organic residue (Mota et al., 2018).

On the other hand, the sludge may contain higher levels of heavy metals than the soil, particularly Pb, Ni, Cd, Cr, Cu, and Zn, indicating its usage as fertilizer in agriculture (Nogueira et al., 2007; Oliveira et al., 2009;

Nascimento et al., 2014a; Mota et al., 2018). Several studies point to the dependence on soil contamination with heavy metals, the way sewage sludge is managed in agriculture and soil composition. In the long term, with successive applications of this residue, it was observed the rise in the contents of some heavy metals in the soil (Nascimento et al., 2004; Gomes et al., 2006; Silva et al., 2006).

The safe use of sludge in agriculture depends on the adoption of stabilization processes, as they reduce the presence of biological contaminants and can reduce the solubility of heavy metals. Among those processes, the stabilization of sludge by solarization, composted, vermicompost and liming stand out (Nascimento et al., 2014a). According to Nogueira et al. (2007), due to the pH increase, the use of limed sewage sludge promoted an intense reduction in the availability of heavy metals in the soil. In this study, the application of limed sewage sludge provided a soil pH value of 6.63, while the application of solarized sewage sludge with the same amount of nitrogen raised soil pH to 8.33. At this pH increase the reductions in available element contents were 28% Zn, 49% Cu, 80% Pb and 41% Ni.

The objective of this work was to evaluate the contamination of pineapple with heavy metals in three orders of soils after the application of sewage sludge submitted to different stabilization processes.

## 2. Material and Methods

The experiment was carried out in a screened type greenhouse in the Montes Claros county, Minas Gerais state, Brazil, at latitude 16°51′38″ S and longitude 44°55′00″ W, from November 2014 to September 2016, by cultivating pineapple variety Pérola, in three types of soil, whose chemical and physical attributes of the 0-20 cm deep layers are presented in Table 1. The climatic conditions in the growing period are shown in Figure 1.

Table 1. Chemical and physical attributes of Cambisol (CM), Nitisol (NT) and Acrisol (AC) used in the study

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Soil <sup>1</sup>	$pH_{\rm (H_2O)}$	P-rem	Ν	Р	Κ	Ca	Mg	Al	H+A1	SB	t	Т	m	V
		mg L <sup>-1</sup>	g dm <sup>-3</sup>	mg	dm <sup>-3</sup>				cmol <sub>c</sub> d	m <sup>-3</sup>			%	ó
СМ	6.8	39.00	6.02	4.99	396.50	5.60	1.55	0.0	1.35	8.78	8.78	9.52	0.00	86
NT	5.8	39.80	6.46	3.05	324.00	4.65	1.15	0.0	2.09	6.63	6.63	8.72	0.00	76
AC	6.4	43.32	6.04	6.08	154.00	8.30	2.25	0.0	1.87	10.94	10.94	12.81	0.00	86
Soil	OM	CO	Cr	Cu	Zn	Pb	Ba	Se	As	Sand	Sil	t	Clay	
	dag	kg <sup>-1</sup>			m	ıg dm <sup>-3</sup> -					d	ag kg <sup>-1</sup> -		
СМ	4.06	2.36	45.77	11.60	33.77	21.40	9.89	< 0.1	3.52	25.03	37	.63	37.34	Ļ
NT	5.19	3.81	31.93	7.64	15.88	14.55	1.46	< 0.1	4.93	30.35	27	.13	42.52	2
AC	6.56	3.81	24.83	10.47	21.87	13.20	2.94	< 0.1	3.77	36.42	24	.23	39.35	5

*Note.* <sup>1</sup> Chemical and physical attributes determined according to Embrapa methodologies (Embrapa, 1997), except for the total concentrations of Cr, Cu, Zn, Pb, Ba, Se and As, which were determined according to the methodology of McGrath and Cunliffe (1985).

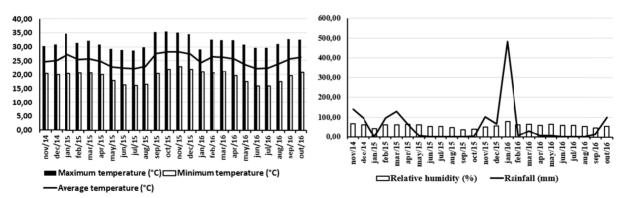


Figure 1. Monthly average values of maximum temperature, minimum temperature, average temperature, relative humidity and precipitation during the period of cultivation of pineapple

The experiment was conducted in a randomized block design with three replicates. The treatments were distributed in a  $7 \times 3$  factorial scheme, as it follows: soil without fertilization, chemical fertilization (NPK), fertilization with composted sewage sludge, fertilization with vermicomposting sewage sludge, fertilization with sewage sludge,

fertilization with sewage sludge dried in dryers and fertilizers with sewage sludge combined with three orders of soils: Cambisol (CM), Nitisol (NT) and Acrisol (AC).

The fertilization corresponding to the NPK treatment was based on the crop requirement and on the chemical analysis of the three orders of soils (Souza et al., 1999), using 20.0 g of urea per plant, 16.7 g of simple superphosphate per plant and 8.6 g of KCl per plant in the Cambisol and the Nitisol, whereas in Acrisol, 20.0 g of urea per plant, 16.7 g of simple superphosphate per plant and 17.2 g KCl per plant, respectively.

The sewage sludge was collected at the Estação de Tratamento de Esgoto-ETE (Sewage Treatment Plant) of Montes Claros-MG, operated by COPASA-MG. The treatment line is composed of preliminary treatment and UASB anaerobic reactor, in which the generated sludge is centrifuged and dried in an oven at 350 °C for 30 minutes and classified as a type A sludge, according to CONAMA Resolution no. 375 (Brasil, 2006). Type A sewage sludge is one containing thermotolerant colliforms with concentration less than 10<sup>3</sup> most probable number (MPN) per gram of total solids (TS), viable helminth eggs with less than 0.25 egg per gram of TS, absence of salmonella in 10 grams of TS and viruses with less than 0.25 plaque forming unit (PFU) per gram of TS.

The limed sewage sludge was obtained by the addition of quicklime in an amount corresponding to 50% of the dry mass of sewage sludge. The residue dose was based on the soil pH increase test described in Brasil (2006), in which the amount applied was that in which the final pH of the soil-limed sludge mixture did not exceed the limit of 7.0, which was: 31.28 g per plant for the Cambisol; 39.85 g per plant for Nitisol and 63.79 g per plant for Acrisol.

For the composting, centrifuged sewage sludge was mixed with grass pruning, whose predominant species was *Paspalum notatum* (grass-batatais), in order to obtain a C:N ratio of 30:1. Periodically, temperature and humidity were monitored, and systematic turning of the piles was performed for material homogenization and humidity control and aeration.

To obtain the vermicompost, a pre-composed of sewage sludge mixed with grass pruning as described for composting. After one month of composting, it was used as a substrate for vermicomposting with California red worms (*Eisenia foetida*).

For the treatments with sewage sludge, except for the limed sewage sludge, the amounts applied were based on the nitrogen content available in the residues for subsurface application (Brasil, 2006) and on the requirement of this element by pineapple (Souza et al., 1999), which was 2.43 kg per plant of composted sewage sludge, 2.37 kg per plant of vermicompost sewage sludge, 1.58 kg per plant of solarized sludge plant and 1.33 kg per plant of sludge dried in a dryer.

The chemical characteristics of the fertilizers used in the study and the contribution of heavy metals are presented in Tables 2 and 3.

Treatments	N (available)	Zn	Cu	Cr	Pb	As
	g kg <sup>-1</sup>			mg kg <sup>-1</sup>		
CS	12.70	452.82	8.40	88.68	49.07	0.67
VS	22.64	381.31	7.06	35.41	32.04	0.33
SS	20.12	718.27	24.29	31.35	48.08	0.31
SD	12.56	778.85	26.10	32.83	51.08	0.27
LS	11.44	375.05	5.25	18.91	44.48	0.45
MCA	-	2,800	1,500	1,000	300	41
Urea	440.00	2.50	16.20	0.00	8.00	0.00
SSP	0.00	142.00	16.20	333.00	116.0	0.00
KCl	0.00	2.50	4.62	0.00	83.83	0.00

Table 2. Characteristics<sup>1</sup> of the chemical fertilizers and types of sewage sludge used in the study <sup>1</sup>

*Note.* <sup>1</sup> The content of N available in the sewage sludge was estimated according to the calculation presented in Brazil (2006), which is based on the total and inorganic N contents determined by the Kjeldahl Method (Tedesco et al., 1995); The levels of N and heavy metals in the fertilizers were determined according to the methodologies of Alcarde (2009); The levels of heavy metals in the sewage sludge were determined according to Malavolta's methodologies (2006); The elements Ba, Cd, Ni, and Se were neither detected nor quantified; CS-Composted sewage sludge; VS-Vermicompost sewage sludge; SS-Solarized sewage sludge; SD-Sewage sludge dried in dryer; LS-Lime sewage sludge; MCA-Maximum concentration allowed in sewage sludge or its by-product (Brasil, 2006); SSP-Single superphosphate.

Treatment	Zn	Cu	Cr	Pb	As
			mg/plant		
CS (2.43 kg/plant)	1,100.35	20.41	215.49	119.24	1.63
VS (2.37 kg/plant)	903.71	16.73	83.92	75.94	0.78
SS (1.58 kg/plant)	1,134.87	38.38	49.53	75.97	0.49
SD (1.33 kg/plant)	1,035.87	34.71	43.66	67.94	0.36
LS-CM (31.28 g/plant)	11.73	0.16	0.59	1.39	0.01
LS-NT (39.85 g/plant)	14.95	0.21	0.75	1.77	0.02
LS-AC (63.79 g/plant)	23.92	0.34	1.21	2.84	0.03
CM and NT:					
Urea (20.0 g/plant)	0.05	0.05	0.32	0.00	0.16
SSP (16.7 g/plant)	2.37	2.37	0.27	5.56	1.94
KCl (8.6 g/plant)	0.02	0.02	0.04	0.00	0.72
AC:					
Urea (20.0 g/plant)	0.05	0.05	0.32	0.00	0.16
SSP (16.7 g/plant)	2.37	2.37	0.27	5.56	1.94
KCl (17.2 g/plant)	0.04	0.08	0.00	1.44	0.00

Table 3. Input of heavy metal<sup>1</sup> from chemical fertilizer and types of sewage sludge applied in the study

*Note.* <sup>1</sup>The levels of heavy metals in the fertilizers were determined according to the methodologies of Alcarde (2009); The levels of heavy metals in the sewage sludge were determined according to Malavolta's methodologies (2006); CS-composted sewage sludge; VS-Vermicompost sewage sludge; SS-Solarized sewage sludge; SD-Sewage sludge dried in dryer; LS-Lime sewage sludge; CM-Cambisol; NT-Nitisol; AC-Acrisol; SSP-Simple superphosphate.

The soils used in the experiment were collected at a depth of 0-20 cm, air dried and passed through a 4-mm sieve, mixed to the different treatments and stored in 10 dm<sup>3</sup> pots which were filled up to a volume equivalent to 8 dm<sup>3</sup> where it was planted seedlings of the Pérola cultivar, previously selected by size, approximately 15 cm long and chemically treated with a systemic insecticide of the neonicotinoid chemical group (Evidence 700WG).

Irrigation was carried out in order to keep soil moisture close to field capacity. The control of weeds, phytopathogens, and insects was carried out constantly after planting the seedlings in the pots, when necessary. Floral induction was also done at 12 months after planting the seedlings by applying 1% ethereal solution, applied at the center of the leaf rosette, using about 50 mL of the solution per plant (Oliveira et al., 2002).

At the beginning of the flowering of the plants, which varied from 10 to 13 months after the planting of the seedlings, D leaf of pineapple and soil samples were collected to determine the heavy metal contents. D leaf is the youngest among the adult leaves and the longest of the leaves, with convergent edges at the base (Figure 2). It is the metabolically active leaf of the pineapple, being used in the analysis of the nutritional state of the plant and to evaluate the moment of the floral induction. The D leaf was cut into 1 cm wide pieces, eliminating the basal portion without chlorophyll, being homogenized and analyzed (Cunha et al., 1999; Silva, 1999). After the fruit harvest, samples were taken from the pulp to determine the heavy metal contents.

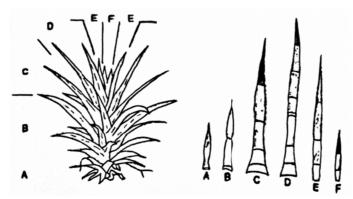


Figure 2. Position of pineapple leaves according to age. A-Older plant leaf, F-Younger plant leaf. Fonte: Catunda et al. (2006)

The content of N available in the sewage sludge was estimated according to the calculation presented in Brasil (2006), which is based on the total and inorganic N contents determined by the Kjeldahl Method (Tedesco et al., 1995). On the other hand, the determination of total N in chemical fertilizers followed the niquel Raney methodology, as described in Alcarde (2009).

The extractions of the Se, As, Ni, Cd, Cu, Zn, Pb, Ba e Cr in the soil were done with aqua regia (HCl:HNO<sub>3</sub>, 3:1, with 37% HCl and 65% HNO<sub>3</sub>), according to the methodology described in McGrath and Cunliffe (1985), in the sewage sludge and plant tissues were done by nitric-perchloric digestion (65% HNO<sub>3</sub> + 72% HClO<sub>4</sub>), according to the methodology described in Malavolta (2006), while the extractions of heavy metals in mineral fertilizers were done with 37% concentrated HCl and heated to near dryness and diluted with 2M HCl according to Alcarde (2009).

Readings of the soil heavy metals were performed using a Varian atomic absorption spectrophotometer, model AA 240, with the following quantification limits: Se (4.25  $\mu$ g dm<sup>-3</sup>), As (2  $\mu$ g dm<sup>-3</sup>), Ni (5 mg dm<sup>-3</sup>), Cd (0.3 mg dm<sup>-3</sup>), Cu (0.1 mg dm<sup>-3</sup>), Zn (0.15 mg dm<sup>-3</sup>), Pb (2.5 mg dm<sup>-3</sup>), Ba (5 mg dm<sup>-3</sup>) and Cr (1.25 mg dm<sup>-3</sup>). On the other hand, the metal readings for 'D' leaf and fruit pulp were made by mass spectrometer with inductively coupled plasma source (ICP-MS), with the following quantification limits: As, Ba, Pb (1  $\mu$ g L<sup>-1</sup>), Cr, Cu (0.5  $\mu$ g L<sup>-1</sup>) and Se, Zn (2.5  $\mu$ g L<sup>-1</sup>).

The data were submitted to analysis of variance and the means of the treatments were compared at 5% probability by the Scott-Knott test.

## 3. Results and Discussion

Concentrations of Ba, Cd, Ni and Se in the sewage sludge were below the levels of detection or quantification by the analytical methods used. On the other hand, Zn, Cu, Cr, Pb and As were quantified (Table 2), however, they were below the maximum concentrations allowed in sewage sludge or sludge products established by CONAMA Resolution no. 375 (Brasil, 2006).

Regarding contents of As in the soil, there was no interaction between the types of fertilizers and soil orders, whereas, for contents of Pb, Cr, Cu, and Zn, there was an interaction between the evaluated factors (Table 4). The Acrisol was the order of soil with the smallest contents of Pb, Cr and As, which are considered to be those with the greatest impact on the environment, among the elements considered in this study. In relation to Pb and Cr, the total contents found after 10 months of cultivation, including that of the control, were greater than the levels obtained in the soil characterization (Table 1), pointing to some additional contribution of these elements by the irrigation water. However, in the case of As, a substantial decrease of this element was observed after the mentioned cultivation period. The levels of Pb, Cr and As in the different soils presented variation close to those observed in the characterization (Table 1). As for Cu and Zn, the contents were, in general, very close among the orders of soils, with a few variations, possibly related to the absorption of the elements by the plant.

As described in Embrapa (2018), the three orders of soils used in this study are mineral and non-hydromorphic, with high redox potential. The Cambisol presents a little advanced pedogenesis, with the highest silt content in its granulometric composition, and the predominant presence of ilite and kaolinite in its clay fraction, the pH of this soil being close to neutrality (Table 1). On the other hand, Nitisol has advanced pedogeneic evolution by the performance of ferralitization with intense hydrolysis, causing kaolinite-oxidic or even kaolinite composition, or with hydroxyl Al between layers in its clay fraction. It always presents a clayey or very clayey texture and is, in general, moderately acidic to acidic. The Acrisol also presents advanced pedogenetic evolution, however, with incomplete performance of the ferralitization process, with predominance of kaolinite in its clay fraction. In this soil, the upper horizon tends to be more sandy and the acidity varies from strong to moderate. Apparently, the higher organic matter content observed in Acrisol (Table 1) may have caused a greater reduction of the redox potential (Asghar and Kanehiro, 1988), with consequent reduction of iron oxides and increased availability of the mentioned metals.

Element (mg dm <sup>-3</sup> )	S all	Fertilizations							A		MC
	Soil	T1	T2	T3	T4	T5	T6	T7	- Aver	CV (%)	MC
	СМ	31.73Aa	32.94Aa	36.13Aa	35.03Aa	33.68Aa	34.90Aa	31.50Aa	-		
Pb	NT	33.11Aa	30.77Ab	37.47Aa	31.42Ab	35.71Aa	34.41Aa	26.90Bc	-	7.51	180
	AC	21.60Bb	22.48Bb	24.15Ba	27.47Ba	19.92Bb	25.34Ba	22.14Cb	-		
	СМ	73.47Bb	61.82Ac	92.51Ba	81.43Ab	52.58Bc	77.08Ab	51.76Ac	-		
Cr	NT	90.54Ab	67.81Ac	174.71Aa	56.72Bd	88.59Ab	85.75Ab	50.37Ad	-	12.02	150
	AC	40.17Cb	26.03Bb	33.63Cb	73.21Aa	36.32Cb	61.00Ba	37.59Ab	-		
	СМ	15.95Ad	15.95Ad	30.86Ac	35.64Ab	39.87Aa	30.52Bc	15.19Ad	-		
Cu	NT	15.24Ad	14.97Ad	33.23Ab	31.64Ab	41.22Aa	28.43Bc	15.28Ad	-	9.27	200
	AC	14.01Ac	13.51Ac	28.02Bb	34.82Aa	33.22Ba	34.09Aa	14.39Ac	-		
	СМ	18.55Ad	74.29Ac	115.46Ab	98.97Bb	184.51Aa	131.90Ab	39.79Ad	-		
Zn	NT	34.95Ab	33.97Bb	133.00Aa	141.84Aa	161.33Aa	147.00Aa	39.23Ab	-	20.00	450
	AC	36.17Ad	44.33Bd	82.15Bc	153.44Aa	149.98Aa	115.30Ab	36.69Ad	-		
	СМ	-	-	-	-	-	-	-	0.70B		
As	NT	-	-	-	-	-	-	-	0.82A	15.95	35
r13	AC	-	-	-	-	-	-	-	0.45C	13.73	55
	Aver	0.72a	0.65a	0.70a	0.60b	0.56b	0.68a	0.67a	-		

Table 4. Contents of heav	v metals in the soi	1 fertilized with ch	nemical fertilizer a	nd types of se	wage sludge
	J				

*Note.* Score: T1-Control; T2-Chemical fertilization; T3-Composted sewage sludge; T4-Vermicompost sewage sludge; T5-Solarized sewage sludge; T6-Sewage sludge dried in dryer; T7-Lime sewage sludge; MC-Maximum contented limit for farming soils in a dry basis; (Minas Gerais, 2011); CM-Cambisol; NT-Nitisol; AC-Acrisol; Aver-Average.

For each variable, means followed by the same upper-case letter in the vertical or lower-case letter in the horizontal are not statistically different from each other by the Scott-Knott test at 5% of probability.

The contents of Pb, Cr, Cu, and Zn were considered low in the soils of the control treatments, chemical fertilization and fertilization with sewage sludge (Table 4). This fact can be attributed to the lower contribution of these elements in the fertilization process (Table 3). For As, lower contents were obtained in soils fertilized with vermicompost sludge and solarized sludge, indicating that the addition of these composts may have contributed to the addition of fulvic acids, which compete with As for adsorption sites of soil minerals, mainly iron, making it possible to obtain losses by volatilization or absorption by plants at moderately acidic pH (Grafe et al., 2002), as observed in these soils (pH  $5.5\pm0.5$ , data not shown).

Based on the results achieved in the study, it can be stated that after the application of chemical or sludge fertilizers, the levels of heavy metals in the soil were below the critical limits established by COPAM Normative Resolution No. 166 of Minas Gerais, published on June 29, 2011 (Minas Gerais, 2011), suggesting the safe use of sludge or sludge compounds in agriculture due to the smallest content of heavy metals in these soils. However, there are many discrepancies in the literature regarding levels of soil contamination with heavy metals from the sewage sludge, which vary greatly depending on the soil, the quality of the sewage sludge and its by-products, the applied dose and the frequency of fertilization (Nogueira et al., 2007, 2008; Zuba Junio et al., 2011; Nascimento et al., 2014a, 2014b, 2015).

The absorption of heavy metals by the pineapple tissues was influenced by the soil classes and fertilization types, obtaining the interaction of these factors for Cu contents, and simple effects for the other elements (Table 5). The highest leaf contents of Cu were observed in the Argissolo fertilized with composted sludge and in the Nitossolo fertilized with vermicompost. However, these levels of Cu in the D leaf were considered below the excessive limits described by Kabata-Pendias and Pendias (2001).

Element (mg kg <sup>-1</sup> )	Soil		Treatments								Exc
	3011	T1	T2	T3	T4	T5	T6	T7	- Aver	CV (%)	EXC
Cu	СМ	3.10Aa	3.60Aa	2.94Ba	2.52Ba	2.77Aa	4.39Aa	4.48Aa	-		
	NT	2.43Ab	4.66Ab	3.05Bb	10.54Aa	2.98Ab	3.73Ab	3.35Ab	-	54 27	20 -100
	AC	2.15Aa	2.90Aa	6.84Aa	2.54Ba	3.71Aa	4.47Aa	3.33Aa	-	54.27	20-100
	Aver	-	-	-	-	-	-	-			
	СМ	-	-	-	-	-	-	-	0.71B		
Pb	NT	-	-	-	-	-	-	-	0.96B	72 71	30-300
PD	AC	-	-	-	-	-	-	-	1.51A	72.71	30-300
	Aver	1.08a	0.83a	0.92a	0.80a	1.05a	1.03a	1.69a			
	СМ	-	-	-	-	-	-	-	1.73A		
Ca	NT	-	-	-	-	-	-	-	1.56A	20.00	5 20
Cr	AC	-	-	-	-	-	-	-	1.42A	29.66	5-30
	Aver	1.47a	1.55a	1.61a	1.77a	1.66a	1.59a	1.33a			
	СМ	-	-	-	-	-	-	-	14.59A	40.00	100-400
7	NT	-	-	-	-	-	-	-	13.59A		
Zn	AC	-	-	-	-	-	-	-	10.91A	48.23	
	Aver	9.41b	12.05b	17.95a	18.89a	12.89b	9.10b	10.91b			
	СМ	-	-	-	-	-	-	-	1.91A		
D	NT	-	-	-	-	-	-	-	1.97A	27.00	500
Ba	AC	-	-	-	-	-	-	-	2.18A	37.90	500
	Aver	2.16b	3.03a	1.43b	1.79b	1.45b	1.55b	2.73a			
	СМ	-	-	-	-	-	-	-	0.08A		
	NT	-	-	-	-	-	-	-	0.04A	116.44	5.00
As	AC	-	-	-	-	-	-	-	0.04A	116.44	5-20
	Aver	0.05a	0.08a	0.06a	0.05a	0.04a	0.05a	0.05a			
	СМ	-	-	-					0.57A		
G	NT	-	-	-	-	-	-	-	0.12A	202.24	5.20
Se	AC	-	-	-	-	-	-	-	0.50A	393.24	5-30
	Aver	0.07a	0.28a	0.10a	0.13a	0.08a	0.10a	1.13a			

Table 5. Contents of heavy metal in the D leaf of the pineapple fertilized with chemical fertilizer and types of sewage sludge

*Note.* Score: T1-Control; T2-Chemical fertilization; T3-Composted sewage sludge; T4-Vermicompost sewage sludge; T5-Solarized sewage sludge; T6-Sewage sludge dried in dryer; T7-Lime sewage sludge; CM-Cambisol; NT-Nitisol; AC-Acrisol; Exc-excess of the element according to Kabata-Pendias and Pendias (2001); Aver-Average.

For each variable, means followed by the same upper case in the vertical or lower-case letter in the horizontal are not statistically different from each other by the Scott-Knott test at 5% of probability.

The average content of Pb in pineapple tissues was greater in the treatment with Acrisol (Table 5), even with the lowest levels in this soil compared to the others (Table 4), showing that other chemical and physical characteristics of Acrisol have interfered in the absorption of Pb by plants such as the greater presence of organic compounds of low molecular weight responsible for the greater mobility of metals in the soil, the largest native organic matter contents of the soil were achieved in the Acrisol. However, for the elements Cr, Zn, Ba, As and Se, no difference was found in the contents in the D leaf among the orders of soils.

The application of composted and vermicompost sludge also increased the Zn content in the pineapple D leaf, with no differences among the other types of fertilization (Table 5). Besides the greater application of the compost and vermicompost sludge, it is inferred that these composts contributed to a higher formation of soluble organo-mineral complexes between Zn, Cu, and humic substances, making these elements less susceptible to soil interactions and more available for absorption by the plants (García-Mina et al., 2004). The Zn content presented lower values with the addition of dry and solarized sludge, even with greater application of this element by these residues. This may imply that the less stabilized organic matter of these residues may contribute to the decrease in the availability of the Zn content in the soil and consequently in the various parts of the plants.

The application of chemical fertilization and limed sludge increased the content of Ba in the leaves in comparison to the other treatments (Table 5). In this case, the increase of Ba in the leaves is not related to the direct input of fertilizers to the soil (Table 3), but due to the solubilization of native Ba in the soil, as a consequence of chemical

and biological reactions caused by the application of fertilizers. As a result, there seems to have been an effect of the organic matter of the sewage sludge in the reduction of the absorption of this element by the plant. Although for some heavy metals there may be increased availability in the presence of organic matter, for elements such as Ba, sulfur bonds and precipitation in the form of barium sulphates or carbonates may occur, reducing their availability and absorption by the plant (Ippolito & Barbarick, 2006), mainly in the range of pH close to neutrality or alkaline conditions (Abreu et al., 2012). On the other hand, the contents of Cr, As and Se in leaf D were not influenced by the application of the different types of fertilizers.

The heavy metals levels obtained in pineapple tissues were below those considered excessive or that could cause toxicity to the plant (Kabata-Pendias & Pendias, 2001). In addition, no substantial differences occurred among the contents of heavy metals in the leaf when the fertilization was carried out with sewage sludge or with mineral fertilizer. Similar behavior was observed by Mota et al. (1990), when studying the contents of As, Ba, Pb, Cu, Cd, Cr, Ni and Zn in leaves of varieties of pineapples fertilized with sewage sludge and mineral fertilizer. According to the authors, Zn was the only element that presented higher levels in the leaves of the pineapples in fertilization with sewage sludge, compared to mineral fertilization.

In the evaluation of the contents of metals present in the pineapple fruit (Table 6), an interaction between the orders of soils and the types of fertilization was observed for Pb and As. The Zn content in the fruit was influenced by the types of fertilization and the other elements, Cr, Cu, Ba, and Se were not altered by the treatments.

Element (mg lrg <sup>-1</sup> )	Soil			Arron	CV(0/)	м					
Element (mg kg <sup>-1</sup> )	5011	T1	T2	T3	T4	T5	T6	T7	- Aver	CV (%)	ML
	СМ	0.13Aa	0.11Aa	0.12Aa	0.08Ba	0.09Aa	0.15Aa	0.06Ba	-		
Pb	NT	0.08Aa	0.07Aa	0.09Aa	0.07Ba	0.06Aa	0.10Aa	0.15Aa	-	57 50	*0.10
PO	AC	0.18Ab	0.12Ab	0.14Ab	0.40Aa	0.10Ab	0.13Ab	0.23Ab	-	57.58	
	Aver	-	-	-	-	-	-	-			
	СМ	0.003Aa	0.003Aa	0.002Ba	0.003Ba	0.012Aa	0.002Aa	0.002Aa	-		
As	NT	0.003Aa	0.003Aa	0.002Ba	0.002Ba	0.002Ba	0.002Aa	0.002Aa	-	111.76	*0.30
AS	AC	0.002Ab	0.002Ab	0.015Aa	0.018Aa	0.014Aa	0.009Ab	0.006Ab	-	111.70	0.30
	Aver	-	-	-	-	-	-	-			
	СМ	-	-	-	-	-	-	-	0.14A		
C-	NT	-	-	-	-	-	-	-	0.14A	26.26	**0.10
Cr	AC	-	-	-	-	-	-	-	0.17A	36.26	
	Aver	0.18a	0.14a	0.16a	0.14a	0.14a	0.15a	0.14a			
	СМ	-	-	-	-	-	-	-	0.43A	56.4	***10.0
Cu	NT	-	-	-	-	-	-	-	0.42A		
Cu	AC	-	-	-	-	-	-	-	0.39A	30.4	
	Aver	0.52a	0.45a	0.38a	0.35a	0.46a	0.41a	0.32a			
	СМ	-	-	-	-	-	-	-	1.37A		*** 50.0
Zn	NT	-	-	-	-	-	-	-	1.24A	41.31	
ZII	AC	-	-	-	-	-	-	-	1.11A	41.51	**50.0
	Aver	0.50c	0.66c	1.58b	1.54b	2.39a	1.10c	0.90c			
	СМ	-	-	-	-	-	-	-	0.26A		
Ва	NT	-	-	-	-	-	-	-	0.27A	79.49	
Da	AC	-	-	-	-	-	-	-	0.25A	/9.49	-
	Aver	0.28a	0.34a	0.40a	0.17a	0.19a	0.21a	0.24a			
	СМ	-	-	-	-	-	-	-	0.002A		
S	NT	-	-	-	-	-	-	-	0.002A	202.6	**0.20
Se	AC	-	-	-	-	-	-	-	0.003A		**0.30
	Aver	0.002a	0.003a	0.002a	0.003a	0.002a	0.003a	0.002a			

Table 6. Contents of heavy metals in pineapple fruit fresh mass fertilized with chemical fertilizer and types of sewage sludge

*Note.* Scores: T1-Control; T2-Chemical fertilization; T3-Composted sewage sludge; T4-Vermicompost sewage sludge; T5-Solarized sewage sludge; T6-Sewage sludge dried in dryer; T7-Lime sewage sludge; CM-Cambisol; NT-Nitisol; AC-Acrisol; LM\*-Maximum limits of Inorganic contaminants allowed in food, on a fresh basis (Brasil, 2013); ML\*-Maximum limits of Inorganic contaminants allowed in food in a fresh basis (Brasil, 1965); ML\*\*\*-Maximum limits of inorganic contaminants allowed in food in a fresh basis (Brasil, 1998); Aver-Average.

For each variable, means followed by the same upper case in the vertical or lower case in the horizontal are not different from each other by the Scott-Knott test at 5% of probability.

Acrisol combined with the application of vermicompost and limed sludge provided a greater content of Pb in the pineapple fruit, and in association with composted, vermicompost and solarized sludge increased As content in the fruit (Table 6). As already mentioned, the higher organic matter content observed in Acrisol (Table 1) may have caused a greater reduction of the redox potential and consequent reduction of iron oxides, and increased availability of the mentioned metals (Asghar & Kanehiro, 1988). In addition, there may have been influence of increased addition of fulvic acids by some types of sewage sludge on increasing availability and uptake of As by the plant (Grafe et al., 2002).

It is worth mentioning that Acrisol presented lower buffer capacity (greater content of reminiscent P) than the other soils, which may contribute to the smaller adsorption capacity of ions to soil clay minerals, making them more available for the plants. The remaining P is used to indirectly measure soil buffer capacity in relation to P (Teixeira et al., 2017) and correlates with soil specific surface (Fontana et al., 2013). In studies on the immobilization of Pb and As in soils, Pierangeli et al. (2001) and Ladeira et al. (2002) state that the greater adsorption of these elements by the soils, among other factors, is associated with a larger specific surface area, as well as to the high levels of aluminum and iron oxides. Another factor that may have contributed to the higher absorption of these metals in the fruit is the higher content of organic matter of soil, implying a higher availability of low molecular weight chelating compounds.

Overall, the levels of Pb and As in pineapple fruits in the Acrisol adjust with the observations made for the contents of these elements in the soil and the D leaf, evidencing that the chemical properties of this soil were, in fact, more favorable to the increase of the availability of these elements. Regarding Pb, the greatest content observed in the pineapple fruit with the application of the limed sludge in the Acrisol (Table 6) may also be attributed to the larger amount of fertilizer applied to this soil, with a greater input of this element (Table 3).

The contents of Pb and As in the pineapple fruit did not vary in Cambisol either in Nitisol when the different types of fertilization were applied (Table 6). However, in Acrisol, the fertilization with vermicompost sewage sludge, in the case of Pb, and fertilization with composted, vermicompost and solarized sewage sludge, in the case of the As promoted the highest levels of these elements. Moreover, in relation to Zn, regardless of the soil type, the fertilization with composted, vermicompost and solarized sewage sludge also provided the greatest levels of this element in the fruit, particularly fertilization with solarized sludge. In general, this fact can be attributed to the greater contribution of these elements in the application of sludge-containing fertilizers, except for the sludge dried in a dryer, since because at the high drying temperature, carbonization process occurs, making sewage sludge more recalcitrant to the decomposition and release of the elements.

In the case of Pb and Cr, the contents of the pineapple fruit were close to or exceeded the maximum limits allowed for food in Brazil (Brasil, 2013) (Table 6). The maximum limits of tolerable heavy metals in Brazil were established by Decree-Law 55871-65 of March 26, 1965 (Brasil, 1965), and maintained and updated by Ministry of Health Ordinance No. 685-980 of 27 of August 27, 1998 (Brasil, 1998) and by the Resolution of the Anvisa-Mercosul Collegiate Board No. 42 of August 29, 2013 (Brasil, 2013).

However, it is noteworthy that the above-mentioned standards refer to fruits in general, without specifying them, and it is important to carry out a more specific estimate for pineapple. Thus, considering the highest levels of Pb, Cr and Ba observed in the fresh fruit of the pineapple, which were 0.40; 0.18 and 0.40 mg kg<sup>-1</sup>, respectively (Table 6), and the daily intake of two slices of pineapple with a total fresh mass estimated at 180 g for a person with 60 kg body weight, the intake rates of mentioned heavy metals, in  $\mu$ g day<sup>-1</sup>, would be 72.0 for Pb, 32.4 for Cr and 72.0 for Ba. Since the provisional limits of daily intake rates established by international legislation for Pb, Cr and Ba are 210; 1,500; and 1,200  $\mu$ g day<sup>-1</sup>, respectively (WHO, 2001; JECFA, 2003; USEPA, 2003), it can be said that the values found in the study were within the safety limits for the consumption of this food.

# 4. Conclusions

Cambisol and Nitisol provide less favorable conditions to the rise in the availability and absorption of Pb e As by the pineapple fruit.

Fertilization with sewage sludge dried in a dryer and lime sewage sludge provide the lowest concentrations of Pb, As and Zn in the pineapple fruit, the most suitable types being for fertilization with this residue.

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