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Fertilization with biosolids and chemical attributes of soil cultivated with 'Pêra' orange

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Abstract

The study aims to evaluate soil attributes cultivated with orange trees and fertilized with biosolid in their first two years of planting. The experimental design used was completely randomized, with six treatments of eight plants. The treatments were based on the source of fertilization and its dose: T0 - 100% of the recommended dose from mineral fertilization (MF); T1 - 50% MF and 50% biosolid (MF + B); T2 - 100% biosolid (100 B); T3 - 115% biosolid (115 B); T4 - 130% biosolid (130 B); and T5 - 150% biosolid (150 B). The cultivar used was 'Pêra' D6, grafted onto Citrandarim Índio. Each treatment consisted of a row of eight plants, evaluating the four central plants. Soil samples were collected at 3, 9, 15, and 21 months after planting. The data were subjected to analysis of variance and mean tests. Biosolids at doses of 100%, 115%, and 150% increased phosphorus, calcium, and base sum levels. Fertilization with biosolid did not increase the amounts of micronutrients and heavy metals in the soil (such as Cu and Zn), which remained within safe limits for agricultural use. Thus, the use of biosolid can be a viable and sustainable alternative to mineral fertilization, provided that legal regulations are respected and constant monitoring is carried out to prevent soil contamination. Good management of sewage sludge application is crucial to ensure sustainability and maximize the benefits of this agricultural practice.

Keywords: *Citrus sinensis (L.) Osbeck,* soil fertility, trace elements, sewage sludge

Introduction

It must highlight the relevance of the research, including through a literature review, in a maximum of two pages. The last paragraph must present the objective of the research. The use of sewage sludge as an organic fertilizer in agricultural areas has been encouraged in various countries due to the benefits it can bring to the soil (da Silva et al., 2021). Moreover, biosolids have been effective in enhancing a range of agromorphological attributes as well as yields in different crop species (Delibacak et al., 2020). Overall, its application to the soil is a promising agricultural alternative for improving fertility and is a sustainable practice for recycling this byproduct of sewage treatment (Achkir et al., 2023), besides contributing to the adoption of conservation farming practices (Pegoraro et al., 2024).

The use of biosolids is mainly based on their potential to be utilized as organic fertilizers in the soil. Their high fertilization potential is due to the large amount of organic matter and nutrients, such as nitrogen, phosphorus, and micronutrients, which are beneficial for both soil and plants (Delibacak et al., 2020). In addition, the application of biosolids can be a cost-effective and efficient alternative to replace chemical fertilizers and plays an important role in improving the physico-chemical and biological properties of the soil (Achkir et al., 2023; Li et al., 2024).

On the other hand, the agricultural use of biosolids still presents significant limitations, such as the presence of harmful elements that may be present in their composition, as well as their toxicity, persistence in the soil, and long-term exposure (Amorim Junior et al., 2021). Therefore, the use of biosolids for commercial agriculture should be done with caution, as they may often contain residues of toxic metals, and their indiscriminate use can be detrimental to soil productivity and the food chain

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(Delibacak et al., 2020).

The application of biosolids has been studied in some crops, including forestry species like eucalyptus (Bonini et al., 2015; Florentino et al., 2019), agricultural crops like maize (Silva et al., 2024; Yada et al., 2020), and soybeans (Freiberger et al., 2020). However, biosolids also have the potential to be used in fruit crops, including orange trees. According to EMBRAPA (2024), orange cultivation occupies an area of 575 thousand hectares, with a national production of approximately 17,6 million tons. In this context, the use of sewage sludge in citrus farming can represent a sustainable fertilization alternative for the production chain. Therefore, this study aims to evaluate the chemical attributes of soil cultivated with oranges and fertilized with biosolids during the first two years of planting.

Material and methods

The experiment was conducted in the experimental area of the Center for Agricultural Sciences and Engineering at the Federal University of Espírito Santo (CCAE-UFES), located in the municipality of Alegre-ES (20°45'06.9"S and 41°29'14.7"W) (**Figure 1**A) , from December 2021 to November 2023. The climate of the region is classified as "Cwa", that is, tropical hot humid, with a cold and dry winter, an average annual temperature of 23.1 °C, and an average total annual precipitation of 1341 mm (Lima et al., 2008). The climatic conditions during the experimental period are described in Figure 1B.

The experimental design was completely randomized, consisting of six treatments of eight plants each. The treatments were based on the fertilizer source and its dosage: T0 - 100% of the recommended dose from mineral fertilization (MF); T1 - 50% MF and 50% biosolid (MF + B); T2 - 100% biosolid (100 B); T3 - 115% biosolid (115 B); T4 - 130% biosolid (130 B); and T5 - 150% biosolid (150 B). The amount of biosolid applied was based on the nutrient with the highest concentration, namely nitrogen, and calculated according to crop requirements (Prezotti et al., 2007).

The cultivar used was ‘Pêra D6’, grafted onto Citrandarim Índio. Spacing of 4.5 x 2.3 meters was adopted, between rows and between plants, respectively. Each treatment consisted of a row of eight plants per line, with only the four central plants being evaluated.

The sewage sludge was collected from the drying beds of the Sewage Treatment Plant operated by the Autonomous Water and Sewage Service (SAAE), located in Jerônimo Monteiro-ES (20°47'19.3"S and 41°23'50.4"W). At this site, the sludge underwent treatment through drying in sand beds and solarization basins. The analyses were conducted in the laboratory of the experimental area at CCAE-UFES, following the methodology established by the Ministry of Agriculture, Livestock, and Supply (Brazil, 2017). The sludge exhibited a moisture content of 35.42%, a pH in CaCl2 of 6.00, a cation exchange capacity (CEC) of 47.00 cmol.kg^{-1}, an organic matter content of 68.38%, and an organic carbon content of 35.79%. Macronutrient concentrations were determined as follows: nitrogen (N) 3.39%, phosphorus (P) 1.48%, potassium (K) 0.75%, calcium (Ca) 1.33%, magnesium (Mg) 0.33%, and sulfur (S) 0.93%. Additionally, micronutrient levels were analyzed, yielding the following values: iron (Fe) 1.45%, zinc (Zn) 491.7 mg.kg^{-1}, copper (Cu) 174.5 mg.kg^{-1}, and manganese (Mn) 28.4 mg.kg^{-1}. The biosolid was further treated with

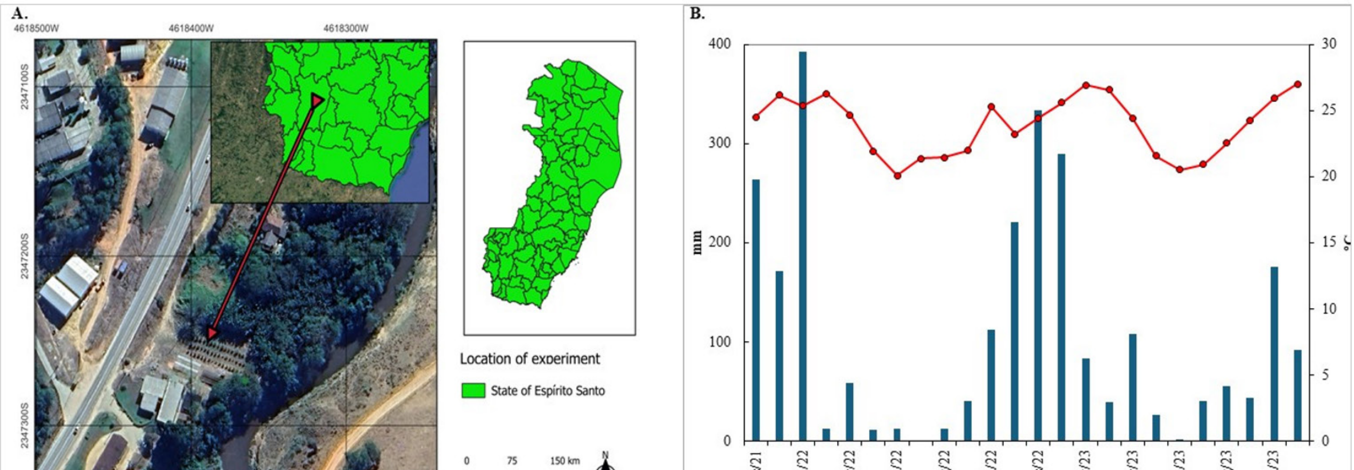

Figure 1. Location of the experiment and monthly averages of temperature and rainfall in Alegre-ES, during the period of December 2021. Source: INMET (2024).

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the application of quicklime at a rate of 72 kg.m⁻³, after which it was rehydrated. The material was air-dried and subsequently stored in raffia bags.

The soil in the study region is classified as Oxisol, exhibiting the following characteristics: pH in water 5.88; available phosphorus (P) 5.80 $mg.dm^{-3}$; sodium (Na) 2.70 $mg.dm^{-3}$; potassium (K) 232.2 $mg.dm^{-3}$; calcium (Ca) 1.83 $cmolc.dm^{-3}$; magnesium (Mg) 0.86 $cmolc.dm^{-3}$; aluminum (Al) 0.00 $cmolc.dm^{-3}$; exchangeable acidity (H + Al) 3.63 $cmolc.dm^{-3}$; base sum 3.30 $cmolc.dm^{-3}$; effective CEC (t) 3.30 $cmolc.dm^{-3}$; potential CEC (T) 6.93 $cmolc.dm^{-3}$; base saturation (V) 47.59%; aluminum saturation 0%; and organic matter content 24.2 $mg.kg^{-1}$, analyzed according to the methodology described by Teixeira et al. (2017). Based on these results, the fertilizer application rates were determined in accordance with the recommendations outlined in the Liming and Fertilization Manual for the State of Espírito Santo (Prezotti et al., 2007) for citrus cultivation.

At the time of planting (December 2021), phosphorus fertilization (MAP) was applied in treatments T0 and T1, in addition to liming to correct acidity and raise the base saturation index to 70%. Furthermore, nitrogen fertilization (urea) was applied as a split topdressing. Treatments composed solely of biosolid (T2, T3, T4, and T5) did not receive liming because the biosolid had undergone the liming process, and only biosolid fertilization was applied at planting. The amount of nutrients applied per treatment, per plant, in the first year was: T0 – 70 g of P2O5 and 100 g of N; T1 – 35 g of P2O5, 50 g of N, and 1.2 kg of biosolid; T2 – 2.5 kg of biosolid; T3 – 2.9 kg of biosolid; T4 – 3.25 kg of biosolid; and T5 – 3.75 kg of biosolid.

In the second year, biosolid was applied as topdressing in two installments (October and December 2022), while mineral fertilization was divided into four applications during the rainy season (10/2022 to 02/2024), as shown in Figure 2. The nutrients applied per plant were: T0 – 220 g of N, 160 g of P_2O_5, and 50 g of K_2O; T1 – 110 g of N, 80 g of P_2O_5, 18 g of K_2O, and 3.3 kg of biosolid; T2 – 6.7 kg of biosolid and 15 g of K_2O; T3 – 7.7 kg of biosolid and 12 g of K_2O; T4 – 8.7 kg of biosolid and 10 g of K_2O; and T5 – 10.5 kg of biosolid and 8 g of K_2O. Potassium was used to supplement the low nutrient content in the biosolid, with potassium chloride being used.

The experiment was irrigated according to crop needs and regional evapotranspiration. Weed control was performed through manual weeding and herbicide application when necessary, while pest and disease management was conducted using biological products.

Soil samples were collected from a depth of 0-30 cm around the canopy of the four central plants in each treatment. The first soil sampling was performed three months after planting. The second, third, and fourth samplings were carried out every six months, at 9, 15, and 21 months after planting, respectively (**Figure 2**). The samples were taken to the Routine Soil Analysis Laboratory at UFES, where analyses were conducted according to the methodology of Teixeira et al. (2017).

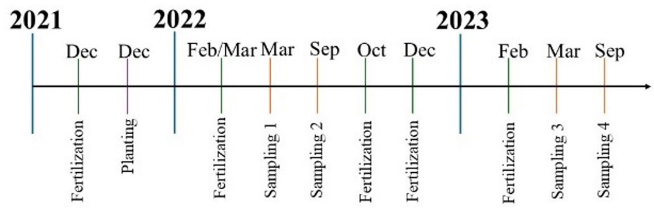

Figure 2. Experiment timeline.

The collected data were subjected to analysis of variance. When the interaction was significant at 5%, or the isolated variable fertilization, the Scott-Knott mean test was performed. When the collection time was significant in isolation, first and second degree equation tests were conducted. Additionally, correlation analysis was performed. All analyses were carried out using the R Studio software (Ferreira et al., 2021).

Results and discussion

Table 1 presents the results of the variance analysis for soil attributes as a function of sampling time, type of fertilization, and their interaction. The values of sodium, potassium, magnesium, aluminum, and aluminum saturation (M) showed a significant interaction at a 5% probability level between sampling and fertilization. For the parameters pH, P, Ca, H + Al, SB, t, V, m, and Zn, the individual factors of sampling and fertilization were significant. For CEC, Fe, Cu, and Mn, only fertilization was significant, while boron showed significance for sampling.

It is possible to observe that mineral fertilization + biosolid resulted in a lower pH value and high levels of H + Al (**Figures 3**A and 3D). When compared to the initial soil pH (5.88), it is possible to observe that only the MF + B treatment reduced the value, while the other treatments showed average values close to or even higher than the initial value. Soil pH is a chemical characteristic related to the availability of soil nutrients. In acidic soils, there is a decrease in the availability of nutrients such as phosphorus, calcium, magnesium, potassium, and molybdenum, and an increase in the solubilization of ions such as zinc, copper, iron, manganese, and aluminum

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Table 1. F values for analysis of variance.

Variable	pH	P	Na	K	Ca	Mg	OM	Al	H + Al	SB
Sampling	9,95*	8,91*	106,09*	1,17	11,00*	4,149 *	12,32*	15,57*	6,38*	9,52*
Fertilization	4,27*	5,23*	8,41*	1,01	3,70*	7,98*	1,90	14,37*	3,23*	4,89*
S*F	1,43	1,61	2,98*	3,69*	1,20	2,06*	1,81	7,17*	1,58	1,35
CV (%)	8,68	76,6	43,01	30,72	39,17	17,82	24,66	120,6	39,85	27,4

	t	CEC	V	m	Fe	Cu	Zn	Mn	B
Sampling	8,69*	2,18	8,43*	13,17 *	1,18	0,82	7,11*	0,34	6,26*
Fertilization	4,49*	9,83*	3,12*	11,90*	3,64*	10,44*	2,65*	22,55*	1,30
S*F	1,46	1,73	1,41	5,61*	0,48	1,39	1,68	0,58	1,70
CV (%)	26,08	10,58	25,07	133,45	31,95	19,73	93,41	31,70	143,45

MO: organic matter; SB: sum of exchangeable bases; t: effective cation exchange capacity, CEC: cation exchange capacity at pH 7.0; V: base saturation index; m: aluminum saturation index. *Significant at 5% probability.

(Veloso et al., 2020). According to Prezotti and Martins (2013), soil pH between 5.5 and 6.5 is ideal for most crops, as it presents an absence of toxic aluminum (Al^{3+}), good availability of boron, and intermediate availability of other micronutrients. Additionally, there is greater availability of macronutrients in this pH range.

The use of quicklime (CaO) for the chemical stabilization of sludge can raise the pH and help neutralize soil acidity and reduce the availability of Al in the soil (Silva, da et al., 2021). However, this pH variation in soils with biosolid application depends on several conditions, including the amount of sludge applied, its quality, soil

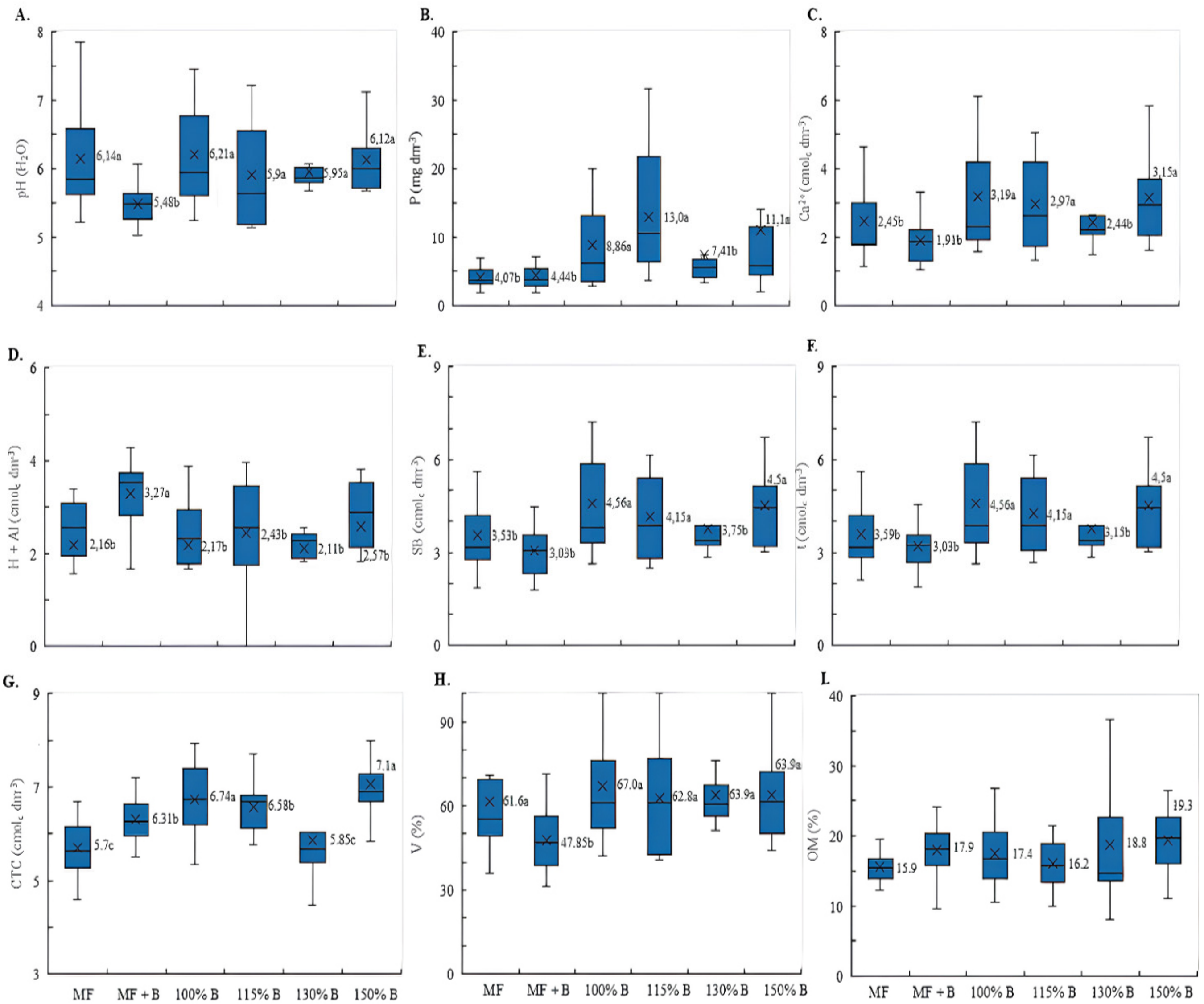

Figure 3. Average of pH, phosphorus, calcium and H+Al values in the soil according to fertilization. MF: mineral fertilization, AM + B: mineral fertilization + biosolids, B: biosolids. X= Average (n=16) ± upper and lower limits. Lowercase letters in each graph represent equal means for the Scott-Knott test at 5% probability.

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characteristics, and mineralization conditions (Achkir et al., 2023).

Phosphorus and calcium levels were statistically higher in treatments with 100%, 115%, and 150% of the recommended biosolid dose, as shown in Figures 3B and 3C. However, all treatments fertilized only with biosolid (including the 115%) have an average higher than the initial soil P value (5.80 cmolc dm^{-3}), which represents an increase of 27.7%, 52.7%, 91.3%, and 124% for treatments T2, T3, T4, and T5, respectively. Biosolid is known for its significant source of P, which, when associated with soil acidity reduction, increases its availability to plants (Silva et al., 2024). In a study by Breda et al. (2020), biosolid promoted an increase in available phosphorus in the soil according to the application rate, while mineral fertilization resulted in low availability of the element in the soil.

Regarding calcium, all treatments showed values higher than the beginning of the experiment (1.83 cmolc dm^{-3}). This can be explained by the fact that the treatment only with mineral fertilization was subjected to liming, whose source is calcium carbonate ($CaCO_3$), and the biosolid was treated with quicklime (CaO). In the soil, these compounds dissociate in the presence of water, forming the Ca^{+2} ion. Achkir et al. (2023) observed that the Ca content increased significantly with the percentages of sludge added, and Bonini et al. (2015) found that a dose of 60 t ha-1 showed a higher calcium content in the soil compared to mineral fertilization.

Fertilizations with biosolid at the doses of 100%, 115%, and 150% provided higher levels in the soil for the sum of bases (SB) and effective CEC (t) (Figures 3E and 3F). For these variables, only the MF + B treatment showed a reduction in the value compared to the initial value of the experiment. The sum of bases represents the sum of exchangeable cations in the soil (Ca^{2+}, K^+, Mg^{2+}, and Na^{2+}), while the effective CEC is the sum of bases plus Al. The CEC at pH 7.0 (CEC) is the sum of bases plus the H + Al content and was higher at the doses of 100% and 150% of biosolid (Figure 3G). However, only the 150% biosolid treatment was superior to the initial soil condition (6.93 cmolc dm^{-3}). According to Moreira et al. (2020), the application of biosolid increased the cation exchange capacity and base saturation of the soil, with treatments having higher application rates of the residue showing higher averages compared to the mineral fertilizer treatment. They also state that since the residue is treated with CaO, this contributes to the base saturation and Ca levels.

Regarding the base saturation index (Figure 4G), it was lower in the treatment with mineral fertilization associated with biosolid, while the others showed averages above 60%. However, all were superior to the beginning of the experiment, when V was 47.59%. Considering that SB and V% are directly related to the presence of bases in the soil, it is observed that the higher addition of Ca, Mg, and K in the treatment with limed sewage sludge resulted in higher SB and V% values in the two evaluated depths (Nascimento, Sampaio, Junio, et al., 2014). The soil organic matter content did not differ significantly (5% probability) between the tested treatments (Figure 4I), although mineral fertilization showed a lower average. Breda et al. (2020) also observed that the application of the lowest dose of biosolid did not result in a significant difference in soil properties compared to the control and chemical treatment. However, most studies find an increase in organic matter content with the application of biosolid, as found by Li et al. (2024), Achkir et al. (2023), and Pegoraro et al. (2024).

Regarding the sampling time, it is possible to observe that pH and potential CEC (pH 7.0) showed a reduction in their values over the months (Figures 4A and 4B). Hamdi et al. (2019) observed that after three successive annual sludge applications, there was a slight reduction in soil pH with the increase in the applied biosolid dose. According to Viçosi and Garcia (2024), limed sewage sludge initially presents high reactivity in the soil, with a subsequent reduction in the alkalization reaction over time, which makes the residue usable in agriculture as a soil amendment. However, the subsequent soil acidification is the result of decomposing organic matter, which produces H^+ and CO_2, forming weak organic acids, resulting in a gradual decrease in pH (Hamdi et al., 2019). Additionally, the same authors attribute that the variation of pH in modified soils depends on several factors, including sludge quality, dose, soil characteristics, and mineralization conditions.

The levels of calcium, sum of bases, effective CEC, and base saturation (V) have a quadratic behavior (**Figures 4**B, 4C, 4D, and 4F), with the maximum value close to nine months after planting, while H + Al had its minimum point during this period (Figure 4E). Pegoraro et al. (2024) observed quadratic regressions for the increase in P, Ca, Mg, and SB levels in sludge-treated soil, which they claim indicates that soil biological activity provided higher nutrient mineralization rates between 30 and 90 days.

Phosphorus released by biosolid occurs gradually in the soil solution, with most being in unavailable forms or immobilized through physical-chemical sorption and interaction with microorganisms (Silva et al., 2021;

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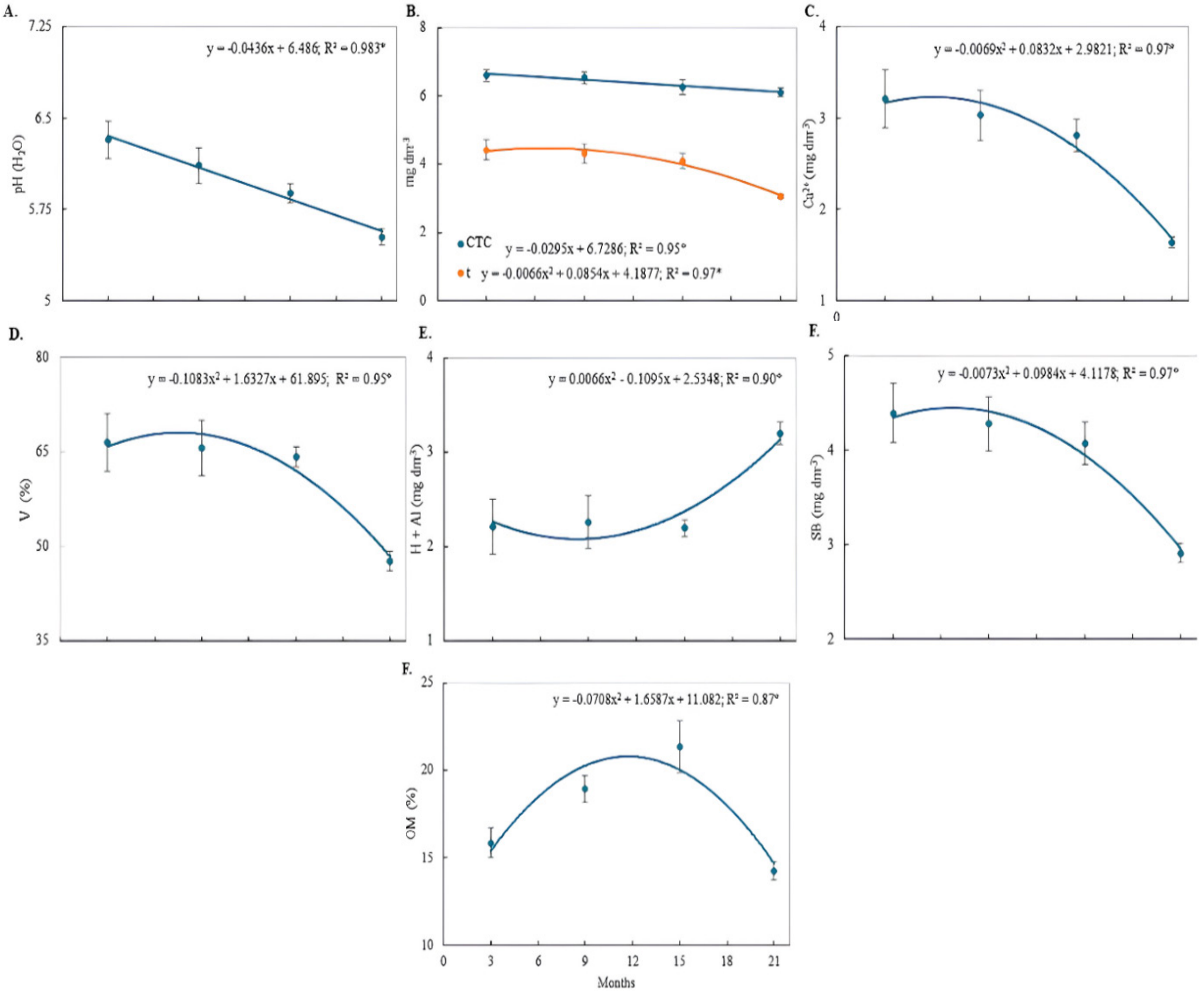

Figure 4. Regression for soil chemical attributes according to the time of collection after planting.
SB: sum of bases, V: base saturation index. *Significant at 5% probability. Average (n=24) ± SE.

Silva et al., 2024). However, over the incubation period, the availability of P decreased over time, indicating a strong interaction of the released phosphorus with soil clay minerals (Pegoraro et al., 2024). Phosphorus released by biosolid occurs gradually in the soil solution, with most being in unavailable forms or immobilized through physical-chemical sorption and interaction with microorganisms (Silva et al., 2021; Silva et al., 2024). However, over the incubation period, the availability of P decreased over time, indicating a strong interaction of the released phosphorus with soil clay minerals (Pegoraro et al., 2024).

The organic matter content reached its maximum (20.6%) at 11.7 months, as shown in Figure 5F. The organic matter in biosolids consists predominantly of recalcitrant compounds, and after application to the soil, the degradation rate depends on the intensity of biological processes (Andrade et al., 2006). Silva, da et al. (2022) observed that the highest mineralization rates of organic matter occurred in the first 20 days of sample incubation, a period during which 20 to 35% of quantified organic carbon was degraded.

The degradation process occurs in two stages: the first, intense and short (average of 8 days), where more labile carbon compounds are exhausted; and the second stage, less intense and responsible for more than 65% of the total organic carbon degraded over the 70-day evaluation period (Andrade et al., 2006). Additionally, the low C/N ratio can also promote rapid mineralization of organic matter in sewage sludge (Balík et al., 2022).

The soil parameters that showed interaction between the fertilization source and sampling time are in **Table 2**. The collection performed nine months after planting showed higher sodium content in the soil,

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Table 2. Values of sodium, potassium, magnesium, aluminum and aluminum saturation (m) in soil cultivated with orange and different fertilizer sources over time

	Na (mg dm^{-3})				K (mg dm^{-3})			
	3 months	**9 months**	**15 months**	**21 months**	**3 months**	**9 months**	**15 months**	**21 months**
MF	8,07 Ba	21,55 Ab	2,40 Ba	8,00 Bb	185,60 Aa	112,35 Aa	125,47 Ab	146,90 Aa
MF + B	2,17 Ba	14,37 Ac	0,47 Ba	4,52 Bb	149,20 Aa	155,37 Aa	84,50 Bb	82,12 Bb
100% B	5,10 Ba	33,17 Aa	6,60 Ba	11,16 Bb	119,30 Aa	146,97 Aa	100,45 Ab	169,06 Ab
115% B	3,90 Ba	19,12 Ab	2,42 Ba	6,63 Bb	115,95 Ba	98,65 Ba	181,82 Aa	67,00 Ba
130% B	1,90 Ba	23,12 Ab	2,42 Ba	19,43 Aa	109,65 Aa	112,57 Aa	117,92 Ab	169,96 Aa
150% B	4,47 Ca	29,65 Aa	4,30 Ca	13,22 Ba	140,45 Aa	108,87 Aa	102,40 Ab	165,20 Aa
	Mg (mg dm^{-3})				**Al (cmol$_c$ dm^{-3})**			
MF	0,74 Aa	0,57 Ab	0,58 Ac	0,80 Aa	0,00 Aa	0,05 Ab	0,07 Aa	0,10 Ac
MF + B	0,79 Aa	0,78 Aa	0,90 Ab	0,74 Aa	0,06 Ca	0,13 Ba	0,00 Ca	0,48 Aa
100% B	0,85 Ba	0,86 Ba	1,21 Aa	0,90 Ba	0,06 Aa	0,00 Ab	0,03 Aa	0,00 Ac
115% B	0,80 Aa	0,84 Aa	0,84 Ab	0,85 Aa	0,05 Ba	0,02 Bb	0,03 Ba	0,27 Ab
130% B	0,81 Aa	0,84 Aa	0,89 Ab	0,88 Aa	0,00 Aa	0,00 Ab	0,00 Aa	0,00 Ac
150% B	0,75 Ba	0,99 Ba	1,20 Aa	0,90 Ba	0,00 Aa	0,00 Ab	0,00 Aa	0,00 Ac

	m (%)			
	3 months	9 months	15 months	21 months
MF	0,00 Aa	2,40 Aa	2,59 Aa	4,65 Ac
MF + B	2,42 Ba	4,99 Ba	0,00 Ba	17,80 Aa
100% B	2,02 Aa	0,00 Aa	0,98 Aa	0,00 Ad
115% B	1,87 Ba	0,79 Ba	1,29 Ba	8,97 Ab
130% B	0,00 Aa	0,00 Aa	0,00 Aa	0,00 Ad
150% B	0,00 Aa	0,00 Aa	0,00 Aa	0,00 Ad

especially when associated with the treatment with 100% and 150% biosolid. Additionally, the treatment with 130% biosolid at 21 months showed higher levels, while the lowest levels were found at 3 and 15 months, a period closer to fertilization application and shortly after the rainy season. According to Jesus & Borges (2020), periods of lower rainfall do not allow adequate leaching of salt in the soil, leading to higher accumulation. This may have occurred because the collection at nine months was conducted in a dry period, and moreover, the drip system forms a wet bulb that promotes leaching of salts only within the irrigated area.

Regarding potassium, the treatment with 115% of the biosolid dose was statistically inferior in the collections at 3, 9, and 21 months, while treatment 2 showed lower values in the collections at 15 and 21 months. Treatments 0, 2, 4, and 5 maintained statistically equal averages throughout the collections. This follows the trend observed by Rodrigues et al. (2024), who noted differences in K between treatments but with little consistency regarding the nature of the treatments included in the study. Despite this, all treatments showed K content lower than that obtained before the experiment (232 mg dm^{-3}). According to Amorim Júnior et al. (2021), potassium is the nutrient that is not adequately present in biosolid to meet, partially or totally, the needs of agricultural crops. Being highly soluble, and as sewage arriving for treatment is composed of 99.9% water, a large part of K and Na remains solubilized in the wastewater (Abreu et al., 2019).

For magnesium levels, values above 1 cmolc dm^{-3} are present in the collection at 15 months, in the treatments with 100% B and 150% of the recommended biosolid dose. Breda et al. (2020) noted differences in Mg concentration only in the first and second sludge applications. For Rodrigues et al. (2024), the levels of S, K, and Mg were minimally influenced by the characteristics and quantities of sewage sludge, with few significant differences compared to mineral fertilization. The highest aluminum levels in the soil are in treatments 1 and 3, and in the collection carried out at 21 months, as well as the aluminum saturation (m) values. The aluminum content indicates the element's level in the ionic form Al^{3+} (also known as exchangeable acidity), which is toxic to plants. With the increase in pH, Al binds to OH- in the soil, forming the inert precipitate $Al(OH)_3$ (Prezotti & Martins, 2013).

The levels of micronutrients present in the soil are shown in Figure 5. For Fe content in the soil, higher values were found in treatments with mineral fertilization and mineral fertilization with biosolid, while the lowest averages are in treatments with biosolid (**Figure 5**A). Lower Cu levels were found in the treatment with 130% biosolid (Figure 5B). The zinc values were equal in all treatments but showed considerable variation within them (Figure 5C). According to Feng et al. (2023), Zn is distributed in exchangeable, reducible, oxidizable, and residual fractions of sewage sludge and, therefore, is relatively mobile, while Cu is predominantly distributed in the oxidizable fraction.

Regarding manganese (Figure 5D), the highest content was found with 150% biosolid, intermediate levels where there is mineral fertilization, and lower levels with 100%, 115%, and 130% biosolid. The boron content was

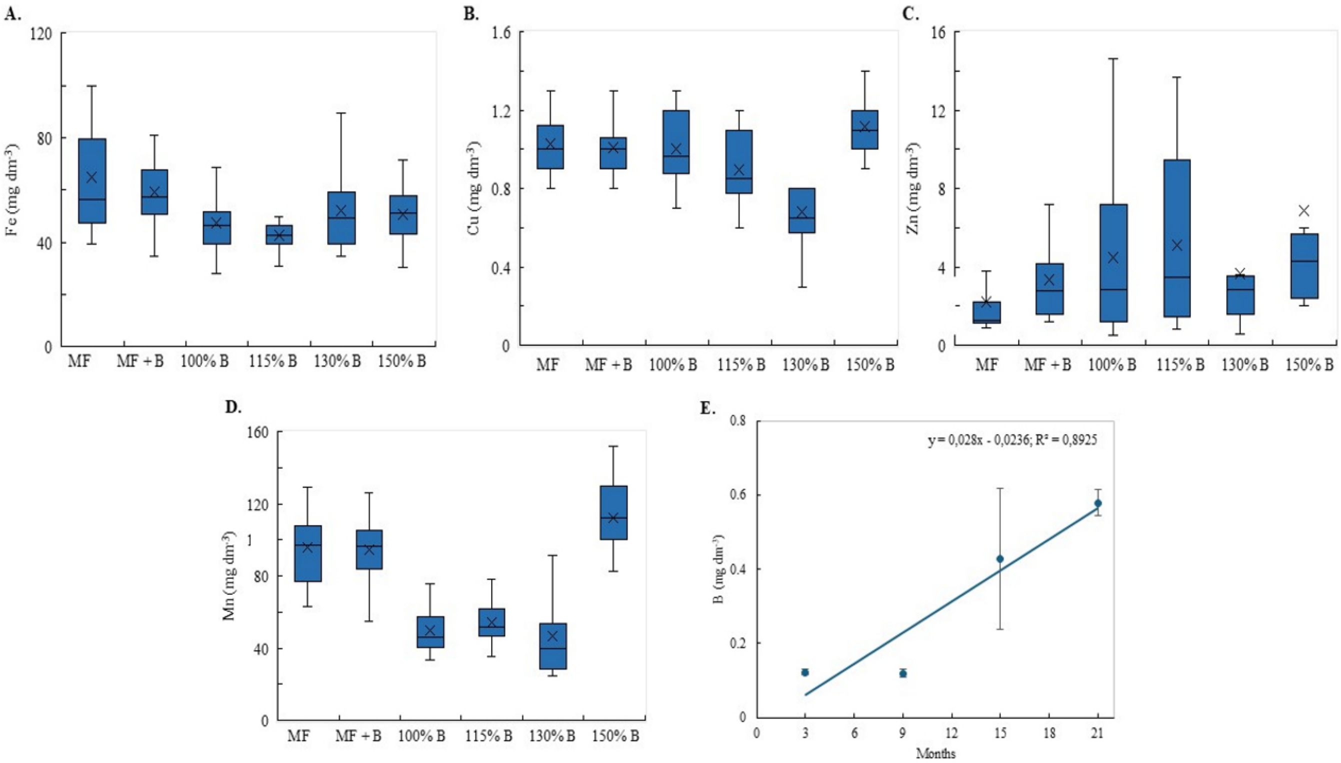

Figure 5 - Levels of micronutrients in soil cultivated with orange trees and fertilized with biosolid. FM: mineral fertilization, FM + B: mineral fertilization + biosolid, B: biosolid.
MF: mineral fertilization, AM + B: mineral fertilization + biosolids, B: biosolids. X= Average (n=16) ± upper and lower limits. Lowercase letters in each graph represent equal means for the Scott-Knott test at 5% probability.
For regression: *Significant at 5% probability. Average (n=24) ± SE.

not significant. Freiberger et al. (2020) observed that the concentration of B in the soil was not altered by the addition of biosolid and attributed this to the increase in pH, which promoted an adsorption effect of the element, leading to the absence of alteration in its concentration in the soil. According to Hamdi et al. (2019), the addition of sewage sludge caused a substantial increase in the content of heavy metals (Pb, Ni, Zn, and Cu) in a dose-dependent manner. AL-Huqail et al. (2023) state that despite the increase in Cd, Cr, Cu, Fe, Zn, Pb, and Ni levels in the soil, all were within safe soil quality limits for agricultural crops. Soil application leads to a low increase in available heavy metals in the soil, similar to what was observed with mineral fertilizer, because these elements were also present at low levels in the sludge (Nascimento et al., 2023). The same was observed for Cu and Zn, as the amount of metals added with a typical agricultural application of biosolid is relatively small compared to background values in agricultural soil (Feng et al., 2023). Organic matter content is another important factor that controls the bioavailability of heavy metals, being recognized as an important adsorptive agent for trace metals in the soil (Hamdi et al., 2019). Most metals are bound in an unavailable fraction and, in some cases, were bound to the organic matter fraction (Wydro et al., 2021).

Regarding time, only zinc and boron showed significant regression. Zn tended to reduce its content over time, but the regression showed low data adjustment ($y = -0.146x + 6.0315$; $R^2 = 0.27$). Regarding boron, the behavior was positive, with an increase in micronutrient content over time (Figure 5E).

The reduction in Zn content may be due to greater losses of the element due to leaching and plant absorption, as well as changes to unavailable forms (Nascimento et al., 2023). The phytoavailability of nutrients in the soil depends on the presence of organic and inorganic constituents, soil properties, biological activity, among other factors. Additionally, due to the buffering capacity of soils rich in Fe and Al oxy-hydroxides and organic matter, chelate formation may occur, reducing availability (Yada et al., 2020). According to Florentino et al. (2019), a tropical weathering climate with high average precipitation and temperatures promotes leaching phenomena that prevent the accumulation of toxic elements over time.

The application of biosolid to soils can also affect soil microbiology. Adding sewage sludge can increase microbial activities, population, and biomass production (Kominko et al., 2017). Additionally, it also increases enzymatic activity and soil enzyme reaction rates during its decomposition (Hamdi et al., 2019). Among the various enzymes present in the soil, acid and alkaline phosphatases and dehydrogenases responded positively to the residue application, and even repeated high-dose applications do not limit the overall biochemical activity of the soil (Siebielec et al., 2018).

Although our work does not show an increase beyond the permitted limits of metals in the soil through fertilization with biosolid in 21 months of study, it is essential to monitor the elements in the long term. Implementing rigorous monitoring and regulation practices, as well as regular tests for heavy metals and other soil contaminants, can help mitigate the risks associated with sludge use (Hernández et al., 2024).

When the application of biosolids to the soil respects the legal feasibility aspects, it can be continued without negative impact on the ecosystem over several years (Amorim Júnior et al., 2021). Thus, good management of sewage sludge application is recommended to ensure sustainability and benefits while controlling soil contamination risks (Achkir et al., 2023). In this way, biosolid can be used instead of chemical fertilizer to reduce the sludge disposal burden and maintain environmental quality (Nahar & Shahadat Hossen, 2021).

Conclusions

Biosolids at doses of 100%, 115%, and 150% significantly increased the levels of phosphorus, calcium, and the sum of bases in the soil. This demonstrates that the application of biosolids can improve soil fertility compared to exclusively mineral fertilization. Fertilization with biosolids did not increase the amounts of micronutrients and heavy metals in the soil (such as Cu and Zn), which remained within safe limits for agricultural use.

Thus, the use of biosolids can be a viable and sustainable alternative to mineral fertilization, provided that legal standards are respected and constant monitoring is carried out to prevent soil contamination. Proper management of sewage sludge application is crucial to ensure sustainability and maximize the benefits of this agricultural practice.

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