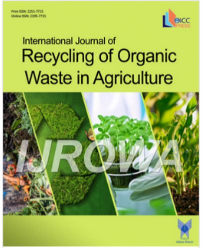

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Nutrient availability in tropical soils fertilized with sewage sludge and natural phosphate

Rodinei Facco Pegoraro*, Jefferson Rodrigo Teixeira Silva, Leidivan Almeida Frazão, Regynaldo Arruda Sampaio, Silvana Ferreira Bicalho, Verônica Aparecida Santos Ferreira Soares

Instituto de Ciências Agrárias, Universidade Federal de Minas Gerais (UFMG), Montes Claros, MG. Brazil.

***Corresponding author:** rodinei@ufmg.br

Original Research

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Abstract:

Purpose: The use of sewage sludge associated with natural rock-based fertilizers can increase nutrient solubility and soil fertility. From this perspective, this study evaluated changes in the chemical soil attributes and the nutrient availability rate after the application of sewage sludge, natural phosphate, and elemental sulfur in soils with different clay contents.
Method: The study was conducted in a controlled environment and was set up in a completely randomized design with four replications in a 3 x 5 x 5 factorial arrangement consisting of soil with three clay contents: 28%, 34%, and 42%; five fertilization managements: unfertilized (Control), fertilized with natural phosphate (NF), fertilized with sewage sludge (SS), fertilized with natural phosphate and elemental sulfur (NF+S), and fertilized with NF+S+SS; and five evaluation times: 0, 30, 60, 90, and 120 days after fertilization. The chemical soil attributes and the nutrient availability rate were evaluated.
Results: The application of sludge, natural phosphate, and sulfur increased the contents of SOM, N, P, K, Ca, Mg, Al, H + Al, SB, and CEC and reduced the soil pH, in addition to favoring the rate and time of availability of P, Ca, and Mg, with means higher than 50% in the soils from 30 to 120 days after application. At 120 days after application of sludge, the average availability rate of P, K, Ca, and Mg corresponded to 37, 13, 144, and 157%, respectively.
Conclusion:These improvements imply savings with mineral fertilizers and contribute to adopting conservationist and sustainable practices. The individual application of sewage sludge or its application with natural phosphate and elemental sulfur increased soil fertility and the nutrient availability rate in soils with different clay contents.

Keywords: Soil chemical attributes; Nutrient recovery rate; Organic compost; Recycling

1. Introduction

Improper solid waste disposal has increased considerably in the last few years due to worldwide pollution (Khan et al., 2022). In 2022, the world population reached 8 billion people (Pison et al., 2022), aggravating the environmental pollution scenario. However, most of this urban waste is still disposed of in landfills (Zat et al., 2021). Therefore, the use of sewage sludge as a source of nutrients for agriculture in weathered tropical soils can be important to mitigate the impacts on natural resources and reduce the consumption of mineral fertilizers.

One such alternative is using sewage sludge in agriculture. In Brazil, several studies used sewage sludge in plant nurseries, floriculture, recovery of degraded areas, and fertilization of forest, annual, and fruit species. Its application in the soil has favored nutrient cycling and physical improvements, especially regarding soil structuring and increased plant production (Breda et al., 2018; Breda et al., 2020; Hamdi et al., 2019; Silva et al., 2019; You et al., 2019; Ahmad et al., 2022). However, in order to enable the use of sewage sludge in fertilization, it is first necessary to determine the nutrient availability rates in tropical soils after its isolated application or associated with mineral nutrient sources. Gonçalves et al. (2021) obtained availability rates of P, Ca, and Mg from sewage sludge of 60, 18, and 26%, respectively, after 120 days of fertilization in Nitisol. This indicates the potential for using of sludge as a source of P and other nutrients for crops in tropical soils.

Tropical soils are considered weathered, have low nutrient availability (Breda et al., 2020), high phosphorus adsorption capacity, in forms not available to plants, and low levels of organic matter (Pegoraro et al., 2020; Gonçalves et al., 2021). The use of sewage sludge, a product rich in organic compounds, associated with rock phosphates and acidic fertilizers (elemental sulfur), can increase phosphorus solubility for plants and other nutrients. The main agents responsible for phosphorus solubilization are attributed to the presence of organic acids, humic compounds, and other organic chelating agents produced during the microbial decomposition of sewage sludge (Korzeniowska et al., 2013; Bustamante et al., 2016; Wollmann et al., 2017), in addition to the development of sulfur-oxidizing bacteria, which react with natural phosphate molecules (calcium phosphate) and generate soluble forms of phosphate in the soil (Stanisławska-Glubiak et al., 2014).

In addition to N, sewage sludge also contains considerable contents of other nutrients that can be considered in managing agricultural fertilization. In that regard, several scientific studies have highlighted the significant contributions of P, K, Ca, Mg, and S for soil fertility and plant nutrition (Alvarenga et al., 2015; Wollmann et al., 2017; Hamdi et al., 2019; Lemming et al., 2019; Wu et al., 2019). However, this information is scarce in tropical soils, leading to environmental contamination and the underestimation of the potential and availability of nutrients for plants.

From this perspective, punctual information related to soil types, sludge, mineralization rate, and soil nutrient availability is necessary for a balanced recommendation of nutrients from sewage sludge. Therefore, this study aimed to evaluate nutrient availability over time after applying sewage sludge in natural phosphate and elemental sulfur mixtures in an Ultisol (Nitossolo) with different textures.

2. Material and methods

Collection and sludge stabilization process

Sewage sludge was provided by the Copasa Sewage Treatment Station (ETE Vieiras) located in the municipality of Montes Claros, Minas Gerais (S 16°68′74″; W 43°85′48″). The material had a granular aspect and was readily stabilized by the supplying company, which dehydrated the product at 350° C in a biogas-powered thermal dryer in an aerobic environment for 30 minutes. The product was categorized as class A, with low concentrations of pathogenic microorganisms per mass unit of total solids (dry basis).

From the material provided, four compound samples formed by three sub-samples of similar volumes were taken randomly from the storage piles. The sludge samples were then homogenized and chemically categorized (Table 1) for use in the study. The determination of organic carbon (OC) was carried out according to the methodology proposed by Alcarde (2009). The total nitrogen (NT), N ammoniacal (NH_4^+), and N nitrate-nitrite (N-NO_3^- + N-NO_2^-) were determined by the Kjeldahl method (Tedesco et al., 1995). Organic nitrogen (ON) was obtained by the difference between NT and IN (N NH_4^++ (N-NO_3^- + N-NO_2^-)). To determine the concentrations of P, K, Ca, Mg, Na, Fe, Mn, Cu, Zn, Ni, As, Cr e Pb, the digestion process with a 3:1 nitric-perchloric solution (nitric acid 70% and perchloric acid 65%) in a digester block was used (Malavolta et al., 1997). The quantification of P was performed by a UV/VIS spectrophotometer (725 nm), while K was determined by a flame photometer. The other elements were analyzed in an AA240FS atomic absorption spectrophotometer, with a precision limit of 0.5%.

Soil texture and analyses

The different soil textures used in the study were constructed based on Ultisol samples with a very clayey texture collected in soil horizon B and near a planting area of eucalyptus at the Experimental Farm Hamilton Abreu Navarro at ICA/UFMG (16°40′19.12″S 43°50′28.18″W), municipality of Montes Claros. After collection, the soil was piled up in three separate windrows, in which different fine sand fractions were added (sieved through a 2-mm mesh) to produce three soil textures. For that purpose, each windrow was subjected to 12 wetting and drying cycles and turned over until the different textures expected were obtained (clayey, medium, and sandy) according to the classification of EMBRAPA (2018). The wetting and drying process was used to segregate between the sand added and the very clayey soil, in addition to promoting greater interaction between the mineral components present (Maluf et al., 2015).

The samples from each soil were subjected to particle size analyses and characterized physically and chemically (Table 1) according to the methodology proposed by EMBRAPA (1997). In brief, the pH (in H_2O) of the soil was determined with a solution ratio of 1:2.5 (m:v). The quantities of Ca^{2+} and Mg^{2+} were extracted with potassium chloride (KCl, 1 mol·L^{-1}) and determined by atomic absorption spectrophotometer. The quantities of P, K^+ were extracted by a Mehlich^{-1} solution, and P was determined by colorimetry, and K^+ by flame photometry. The potential acidity (H + Al) was determined by extraction with a calcium acetate solution ($C_4H_6CaO_4$, 5 mol L^{-1}), and CEC was quantified by the sum of bases (Ca + Mg + K) with the potential acidity (H + Al). Soil organic carbon was quantified according to the method of Yeomans and Bremner (1988), and soil organic matter was obtained by multiplying the organic carbon content by 1.724.

Experimental design and study setup

Table 1. Sewage sludge (SS) and soil characterization used in the study.

Sludge (SS)	**TN** ($g\ kg^{-1}$)	**N-NH_4^+** ($g\ kg^{-1}$)	**N-NO_3^-** ($g\ kg^{-1}$)	**ON** ($g\ kg^{-1}$)	**OC** ($g\ kg^{-1}$)	**Ca** ($g\ kg^{-1}$)	**Mg** ($g\ kg^{-1}$)	**P** ($g\ kg^{-1}$)	**K** ($g\ kg^{-1}$)			
SS	36.50	1.82	0.03	34.64	338.80	9.81	2.02	7.09	3.74			
	Na ($g\ kg^{-1}$)	**Fe** ($mg\ kg^{-1}$)	**Mn** ($mg\ kg^{-1}$)	**Cu** ($mg\ kg^{-1}$)	**Zn** ($mg\ kg^{-1}$)	**Ni** ($mg\ kg^{-1}$)	**As** ($mg\ kg^{-1}$)	**Cr** ($mg\ kg^{-1}$)	**Pb** ($mg\ kg^{-1}$)			
SS	573.28	2.349.16	114.54	115.48	317.75	21.81	0.16	2.87	28.54			
Soil	**pH** -	**SOM** ($g\ kg^{-1}$)	**§P** $mg\ dm^{-3}$	**§K^+** $mg\ dm^{-3}$	**Ca^{2+}** $cmolc\ dm^{-3}$	**Mg^{2+}** $cmolc\ dm^{-3}$	**SB** $cmolc\ dm^{-3}$	**H + Al** $cmolc\ dm^{-3}$	**CEC** $cmolc\ dm^{-3}$	**Sand** ($g\ kg^{-1}$)	**Silt** ($g\ kg^{-1}$)	**Clay** ($g\ kg^{-1}$)
Sandy	6.1	14.10	4.55	56	4.49	0.62	5.25	1.25	6.50	120	460	420
Medium	5.9	16.30	5.12	61	4.89	0.83	5.88	1.54	7.42	200	460	340
Clayey	5.8	21.80	5.91	66	5.21	0.89	6.27	1.92	8.19	400	320	280

TN- total nitrogen. ON- organic nitrogen. OC– organic carbon. SOM: Soil organic matter.
§Extracted with $Mehlich^{-1}$ solution. SB – sum of bases (Ca + Mg + K). H + Al: potential acidity. CEC – cation exchange capacity. Al, Ca, Mg = Extracted with KCl 1 mol L^{-1}.

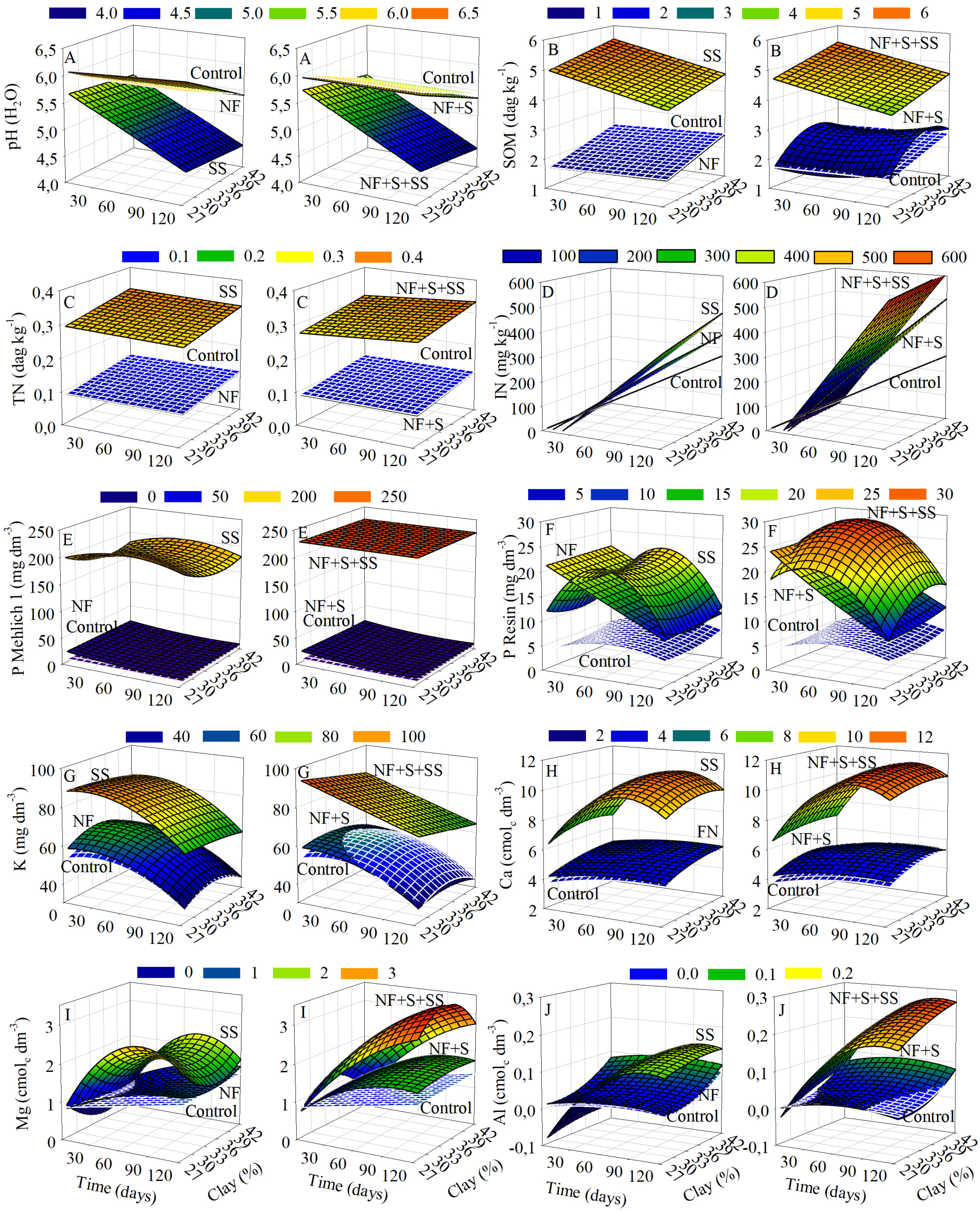


Figure 1. pH in water and contents of organic matter (SOM), total N (TN), mineral N (IN), P extracted by Mehlich 1 and Resin, K, Ca, Mg, and Al in the soils under different fertilization managements

Control: unfertilized control, NF: fertilization with natural phosphate; SS: fertilization with sewage sludge; NF + S: fertilization with natural phosphate and sulfur; and NF + S + SS: fertilization with natural phosphate, sulfur and sewage sludge), clay content (Clay), and incubation time (Time).

Table 2. Summary of the analysis of variance for the attributes of soil.

SV	DF	Mean Square (MQ)						
		pH	SOM	TN	IN	PMeh	PRes	K
Texture(T)	2	5.05*	12.90*	0.0320*	0.00082*	4205.12*	83.77*	1070.58*
Manage-ment(M)	4	8.13*	119.99*	0.7320*	0.00160*	642553.98*	1889.05*	14213.39*
Time (Te)	4	3.60*	0.47*	0.0007*	0.02170*	5428.92*	766.70*	4332.56*
T x M	8	0.48*	0.36*	0.0008*	0.00011*	1738.46*	52.33*	73.70*
T x Te	8	0.10*	0.28*	0.0080*	0.00026*	1887.87*	56.21*	382.46*
M x Te	16	0.89*	1.04*	0.0007*	0.00053*	2702.72*	198.40*	83.08*
T x M x Te	32	0.07*	0.28*	0.0020*	0.00007*	871.31*	45.00*	59.50*
Error	225	0,02	0.13	0.0003	0.00002	75.95	20.63	25.58
CV (%)		2.92	11.32	9.61	23.47	10.18	30.61	8.15
Mean		5.43	3.14	0.18	0,02	85.64	14,84	62.05
SV	DF	Mean Square (MQ)						
		Ca	Mg	Al	H+Al	SB	$CEC_{pH=7}$	BS
Texture (T)	2	14.89*	0.48*	0.0770*	36.57*	12.54*	80.52*	1271.55*
Manage-ment(M)	4	371.36*	14.69*	0.2020*	40.01*	547.04*	881.63*	59.05*
Time (Te)	4	41.88*	17.47*	0.1090*	55.86*	109.49*	308.40*	978.39*
T x M	8	0.41*	1.28*	0.0023*	0.17*	1.52*	1.94*	27.93*
T x Te	8	1.61*	1.38*	0.0068*	2.98*	3.65*	3.30*	119.35*
M x Te	16	6.61*	1.46*	0.0208*	2.94*	12.80*	25.59*	14.91*
T x M x Te	32	0.56*	0.50*	0.0010*	0.35*	0.78*	1.19*	15.82*
Error	225	0.17	0.16	0.0004	0.21	0.28	0.29	7.09
CV (%)		6.09	26.30	35.45	17.40	6.28	6.29	3.47
Mean		6.73	1.54	0.06	2.63	8.43	11.06	76.78

pH in water, mean content of soil organic matter (SOM), total nitrogen (TN), inorganic nitrogen (IN), exchangeable phosphorus by Mehlich (P-Mehlich 1) and resin (P-Resin), exchangeable potassium (K), exchangeable calcium (Ca), exchange-able magnesium (Mg), aluminum (Al), potential acidity (H + Al), sum of bases (SB), total cation exchange capacity ($CEC_{pH=7}$) and base saturation percentage (BS) after the application of the study factors referring to management (Control, NF, SS, NF + S and NF + S + SS), soil clay content /texture (T), and incubation time (Te). SV: source of variation; DF: degree of freedom; NF: soil fertilized with natural phosphate; SS: soil fertilized with dehy-drated sewage sludge; NF+S: soil fertilized with natural phosphate and elemental sulfur; NF + S + SS: soil fertilized with natural phosphate, elemental sulfur and sewage sludge. CV: coefficient of variation in percentage. *Significant at 5% by the F-test.

The study was conducted in the laboratory, with a controlled temperature between $24-27°$ C, following a completely randomized design with four replications and a 3 x 5 x 5 factorial arrangement. The first factor corresponded to the soil clay contents/textures: sandy (28%), medium (34%), and clayey (42%); the second factor consisted of five fertilization managements: control (unfertilized), soil fertilized with natural phosphate (NF); soil fertilized with dehydrated sewage sludge (SS); soil fertilized with natural phosphate and elemental sulfur (NF + S); and soil fertilized with natural phosphate, elemental sulfur, and sewage sludge (NF+S+SS); the third factor corresponded to five evaluation times: 0, 30, 60, 90, and 120 days after applying the fertilizers.

The phosphorus source corresponded to ground natural phosphate with 26% total P_2O_5 (6% soluble P_2O_5 in 20 g L^{-1} citric acid at a ratio of 1:100, and 24% Ca) at the level of 128 kg ha^{-1} of P_2O_5. Elemental sulfur was applied at a ratio of five parts of FN for one part of elemental sulfur (5:1), and the level applied corresponded to 100 kg ha^{-1}. The sewage sludge level mixed with the soil corresponded to 138 t ha^{-1} and was recommended based on the average nitrogen demand for pineapple (Mota et al., 2021), calculated by the sum of the N in the mineral form (N-NH_4^+ + N-NO_3^-) initially contained in the sewage sludge with a 20% mineralized organic N fraction (MF), as proposed by Berton and Nogueira (2010).

The study was set up with 300 experimental units formed by 500-mL plastic containers with airtight lids and adequately identified. According to the treatment, the containers received 200 g of soil or a mixture of soil with sludge and/or mineral fertilizer. The containers were kept at a controlled temperature ($24-27°$ C), with the moisture of their contents being corrected for 70% of field capacity from July 3 to October 3, 2018. The experimental units were opened weekly for approximately 15 minutes to maintain moisture and gas exchange with the environment.

Collection and chemical analysis of soil samples

At the end of each evaluation period (0, 30, 60, 90, and 120 incubation days), the containers were kept open under room conditions for five days for soil drying. Then, a 30-g soil aliquot was ground in a mortar and pestle and passed through a sieve with a mesh size of 65 (0, 212 mm)

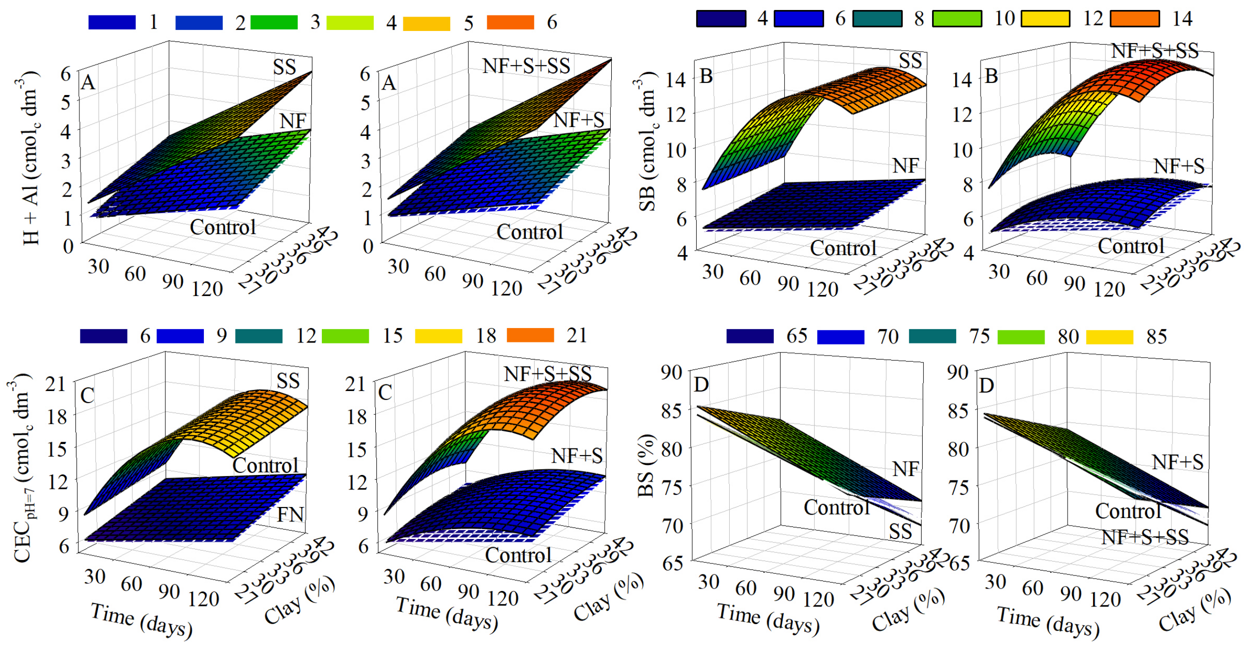


Figure 2. Potential acidity (H + Al), sum of bases (SB), cation exchange capacity ($CEC_{pH=7}$), and base saturation percentage (BS) in soils under different fertilization managements
Control: unfertilized, NF: fertilization with natural phosphate; SS: fertilization with sewage sludge; NF + S: fertilization with natural phosphate and sulfur; and NF + S + SS: fertilization with natural phosphate, sulfur and sewage sludge), clay content (Clay), and incubation.

for chemical characterization (EMBRAPA, 1997).
The soil samples were used to characterize the contents of total organic carbon (TOC) by the wet oxidation method with external heating (Yeomans and Bremner, 1988). Total nitrogen (TN) was determined by distillation after sulfuric acid digestion (Bremner, 1996). The analyses also included the determination of the contents of inorganic nitrogen (IN), obtained by the sum between N NH_4^+ and N-NO_3^- + N-NO_2^- (Equation 1).

$$IN = (N - NH_4^+) + (N - NO_3^- + N - NO_2^-) \qquad (1)$$

The nutrient availability rate (AR) in the soil was calculated based on the contents obtained by the soil nutrient extractors in the treatments with fertilization compared to the mean contents in the soil without fertilization. The results were expressed as percentages, as follows (Equation 2):

$$AR = \frac{(NF - NU)}{NF} \times 100 \qquad (2)$$

AR= nutrient availability rate (%)
NF= nutrient content in the fertilized soil (mg dm^{-3})
NU= nutrient content in the unfertilized soil (mg dm^{-3}) – Control, soil unfertilized with SS, NF and S.

Statistical analyses

The data referring to the different attributes analyzed were subjected to the test of normality (Shapiro-Wilk test) and later to the analysis of variance at 5% of probability ($p < 0.05$). The multiple comparison test was performed for the quantitative factors, texture, and types of residues using Tukey's test ($p < 0.05$). Regression analysis was performed for the quantitative factor referring to the evaluation time. The statistical analyses were performed using the software Sisvar 5.6. Also, multivariate analysis was performed with the principal components using the software R (R Studio version 4.0.3).

3. Results and discussion

Soil chemical attributes after the application of sludge and mineral fertilizers

The soil chemical attributes were influenced ($p < 0.05$) by the texture, management (Control, NF, SS, NF + S and NF + S + SS), and incubation time (Table 2). From this perspective, this study followed the unfolding of the triple interaction to present and discuss the results obtained (Figs 1 and 2, Tables 3 and 4).
Fertilization with sewage sludge (SS) and sewage sludge with natural phosphate and elemental sulfur (NF + S + SS) promoted the highest contents of TN, P, K, Ca, Mg, Al, H + Al, SB, and CEC and reduced the pH of soils with different textures and over the incubation time (Figs 1 and 2, Tables 3 and 4).
When added to the soil, sewage sludge (SS and NF + S + SS) reduced the soil pH (Fig. 1A, Table 3) due to the higher release of organic compounds. According to Boeira (2009), the acidification of the soil solution occurs due

Table 3. Mathematical equations adjusted for the soil chemical attributes described in Fig. 1.

Manag.	**Equation**	R^2	**Equation**	R^2
	pH		OC	
Control	$\hat{z} = 7.07 - 0.04^{*}x + 5.3e^{-15ns}y$	0.81	$\hat{z} = 0.23 + 0.05^{*}x + 0.001^{ns}y$	0.78
NF	$\hat{z} = 7.51 - 0.05^{*}x + 0.001^{ns}y$	0.85	$\hat{z} = 0.27 + 0.05^{*}x + 0.001^{ns}y$	0.94
SS	$\hat{z} = 5.89 - 0.009^{ns}x - 0.009^{*}y$	0.80	$\hat{z} = 3.81 + 0.04^{*}x - 0.008^{*}y$	0.77
NF+S	$\hat{z} = 7.07 - 0.04^{*}x - 0.0001^{ns}y$	0.74	$\hat{z} = -14.1 + 0.90^{*}x - 0.004^{ns}y - 0.01^{**}x^2 + 5.4e^{-5ns}y^2$	0.85
NF+S+SS	$\hat{z} = 6.10 - 0.01^{ns}x - 0.010^{*}y$	0.86	$\hat{z} = 3.20 + 0.05^{*}x - 0.007^{*}y$	0.90
	TN		IN	
Control	$\hat{z} = 0.02 + 0.002^{*}x + 1.4e^{-5ns}y$	0.94	$\hat{z} = -1.4 + 0.15^{ns}x + 2.17^{*}y$	0.88
NF	$\hat{z} = 0.02 + 0.002^{*}x + 1.4e^{-5ns}y$	0.95	$\hat{z} = -87.2 + 2.52^{ns}x + 2.80^{*}y$	0.87
SS	$\hat{z} = 0.21 + 0.003^{*}x - 8.2e^{-5ns}y$	0.84	$\hat{z} = -215.9 + 5.62^{ns}x + 3.55^{*}y$	0.87
NF+S	$\hat{z} = 0.02 + 0.002^{*}x + 1.2e^{-5ns}y$	0.98	$\hat{z} = -276.6 + 7.39^{ns}x + 3.93^{*}y$	0.84
NF+S+SS	$\hat{z} = 0.19 + 0.003^{*}x + 0.2e^{-3ns}y$	0.81	$\hat{z} = -168.76 + 3.57^{ns}x + 5.27^{*}y$	0.87
	P-Mehlich 1		P-Resin	
Control	$\hat{z} = \bar{z} = 5.18$		$\hat{z} = \bar{z} = 5.44$	
NF	$\hat{z} = 13.6 + 0.42^{ns}x - 0.20^{*}y - 0.01^{ns}x^2 + 0.001^{*}y^2$	0.95	$\hat{z} = 19.90 + 0.04^{ns}x - 0.10^{*}y$	0.80
SS	$\hat{z} = 620.3 - 24.43^{*}x + 0.63^{*}y + 0.33^{*}x^2 - 0.005^{*}y^2$	0.84	$\hat{z} = 53.15 - 2.49^{ns}x + 0.34^{*}y + 0.07^{ns}x^2 - 0.002^{*}y^2$	0.81
NF+S	$\hat{z} = 38.9 - 1.00^{ns}x - 0.21^{*}y + 0.01^{ns}x^2 + 0.001^{*}y^2$	0.96	$\hat{z} = 30.97 - 0.51^{ns}x - 0.15^{*}y + 0.009^{ns}x^2 + 0.0002^{ns}y^2$	0.89
NF+S+SS	$\hat{z} = \bar{z} = 226.32$		$\hat{z} = -78.28 + 5.43^{*}x + 0.15^{*}y - 0.07^{ns}x^2 - 0.002^{*}y^2$	0.81
	K		Ca	
Control	$\hat{z} = -94.9 + 8.83^{*}x + 0.14^{ns}y - 0.13^{*}x^2 - 0.002^{*}y^2$	0.90	$\hat{z} = -7.04 + 0.59^{*}x + 0.02^{*}y - 0.008^{ns}x^2 - 8.6e^{-5ns}y^2$	0.85
NF	$\hat{z} = -114.9 + 10.36^{*}x + 0.08^{ns}y - 0.15^{*}x^2 - 0.002^{*}y^2$	0.92	$\hat{z} = -3.20 + 0.39^{*}x + 0.01^{*}y - 0.005^{ns}x^2 - 6.9e^{-5ns}y^2$	0.78
SS	$\hat{z} = 72.75 + 1.08^{ns}x + 0.08^{ns}y - 0.02^{ns}x^2 - 0.002^{*}y^2$	0.89	$\hat{z} = -6.36 + 0.72^{*}x + 0.09^{*}y - 0.01^{ns}x^2 - 0.7e^{-3*}y^2$	095
NF+S	$\hat{z} = -13.30 + 4.36^{ns}x + 0.08^{ns}y - 0.06^{ns}x^2 - 0.002^{*}y^2$	0.91	$\hat{z} = -9.67 + 0.80^{*}x + 0.02^{*}y - 0.01^{*}x^2 - 9.6e^{-5ns}y^2$	0.78
NF+S+SS	$\hat{z} = 109.43 - 0.59^{ns}x - 0.17^{*}y$	0.77	$\hat{z} = -0.89 + 0.42^{ns}x + 0.10^{*}y - 0.006^{ns}x^2 - 0.6e^{-3*}y^2$	0.97
	Mg		Al	
Control	$\hat{z} = 0.77 + 0.001^{ns}x + 0.005^{*}y$	0.78	$\hat{z} = 0.35 - 0.02^{ns}x + 0.0006^{ns}y + 0.0004^{*}x^2 - 3.8e^{-6ns}y^2$	0.86
NF	$\hat{z} = 5.1 - 0.26^{ns}x + 0.01^{*}y + 0.004^{ns}x^2 - 5.4e^{-5ns}y^2$	0.88	$\hat{z} = 0.45 - 0.03^{*}x + 0.0006^{ns}y + 0.0005^{*}x^2 - 4.1e^{-6ns}y^2$	0.90
SS	$\hat{z} = 12.9 + 0.71^{ns}x - 0.05^{*}y + 0.01^{ns}x^2 - 0.3e^{-3*}y^2$	0.88	$\hat{z} = -0.37 - 0.02^{ns}x + 0.004^{*}y - 0.0002^{ns}x^2 - 2.0e^{-5*}y^2$	0.92
NF+S	$\hat{z} = -1.6 + 0.1^{ns}x + 0.02^{*}y - 0.002^{ns}x^2 - 0.1e^{-3*}y^2$	0.91	$\hat{z} = -1.6 + 0.1^{ns}x + 0.02^{*}y - 0.002^{ns}x^2 - 0.1e^{-3*}y^2$	0.89
NF+S+SS	$\hat{z} = -15.1 + 0.95^{ns}x + 0.03^{*}y - 0.01^{*}x^2 - 0.1e^{-3*}y^2$	0.87	$\hat{z} = -0.43 + 0.02^{ns}x + 0.004^{*}y - 0.0002^{ns}x^2 - 2.1e^{-5ns}y^2$	0.92

*, ^{ns}: significant at 5% and non-significant by the t-test.

Table 4. Mathematical equations adjusted for the soil chemical attributes described in Fig. 2.

Manag.	**Equation**	$\mathbf{R^2}$	**Equation**	$\mathbf{R^2}$
	H+Al		SB	
Control	$\hat{z} = -1.59 + 0.08^{*}x + 0.010^{*}y$	0.83	$\hat{z} = 3.14 + 0.06^{*}x + 0.011^{*}y$	0.81
NF	$\hat{z} = -1.79 + 0.09^{*}x + 0.012^{*}y$	0.85	$\hat{z} = 3.41 + 0.06^{*}x + 0.011^{*}y$	0.80
SS	$\hat{z} = -0.91 + 0.08^{*}x + 0.026^{*}y$	0.86	$\hat{z} = 7.15 - 0.002^{ns}x + 0.14^{*}y + 0.0004^{ns}x^2 - 0.001^{*}y^2$	0.96
NF+S	$\hat{z} = -1.58 + 0.09^{*}x + 0.012^{*}y$	0.82	$\hat{z} = -11.77 + 0.97^{*}x + 0.04^{*}y - 0.013^{*}x^2 - 0.0002^{*}y^2$	0.88
NF+S+SS	$\hat{z} = -1.04 + 0.09^{*}x + 0.027^{*}y$	0.81	$\hat{z} = -15.61 + 1.36^{*}x + 0.13^{*}y - 0.019^{*}x^2 - 0.0007^{*}y^2$	0.97
	CEC		BS	
Control	$\hat{z} = 1.56 + 0.147^{*}x + 0.022^{*}y$	0.88	$\hat{z} = 98.95 + 0.55^{*}x - 0.062^{*}y$	0.79
NF	$\hat{z} = 1.61 + 0.154^{*}x + 0.023^{*}y$	0.88	$\hat{z} = 100,99 - 0.61^{*}x - 0.065^{*}y$	0.83
SS	$\hat{z} = 6.04 + 0.06^{ns}x + 0.19^{*}y + 0.0006^{ns}x^2 - 0.001^{*}y^2$	0.97	$\hat{z} = 94.91 - 0.35^{*}x - 0.077^{*}y$	0.80
NF+S	$\hat{z} = -7.74 + 0.71^{ns}x + 0.06^{*}y - 0.008^{ns}x^2 - 0.0003^{*}y^2$	0.92	$\hat{z} = 100.69 - 0.61^{*}x - 0.063^{*}y$	0.76
NF+S+SS	$\hat{z} = -21.26 + 1.69^{ns}x + 0.20^{*}y - 0.023^{ns}x^2 - 0.001^{*}y^2$	0.97	$\hat{z} = 94.83 - 0.38^{*}x - 0.073^{*}y$	0.71

*, ^{ns}: significant at 5% and non-significant by the t-test.

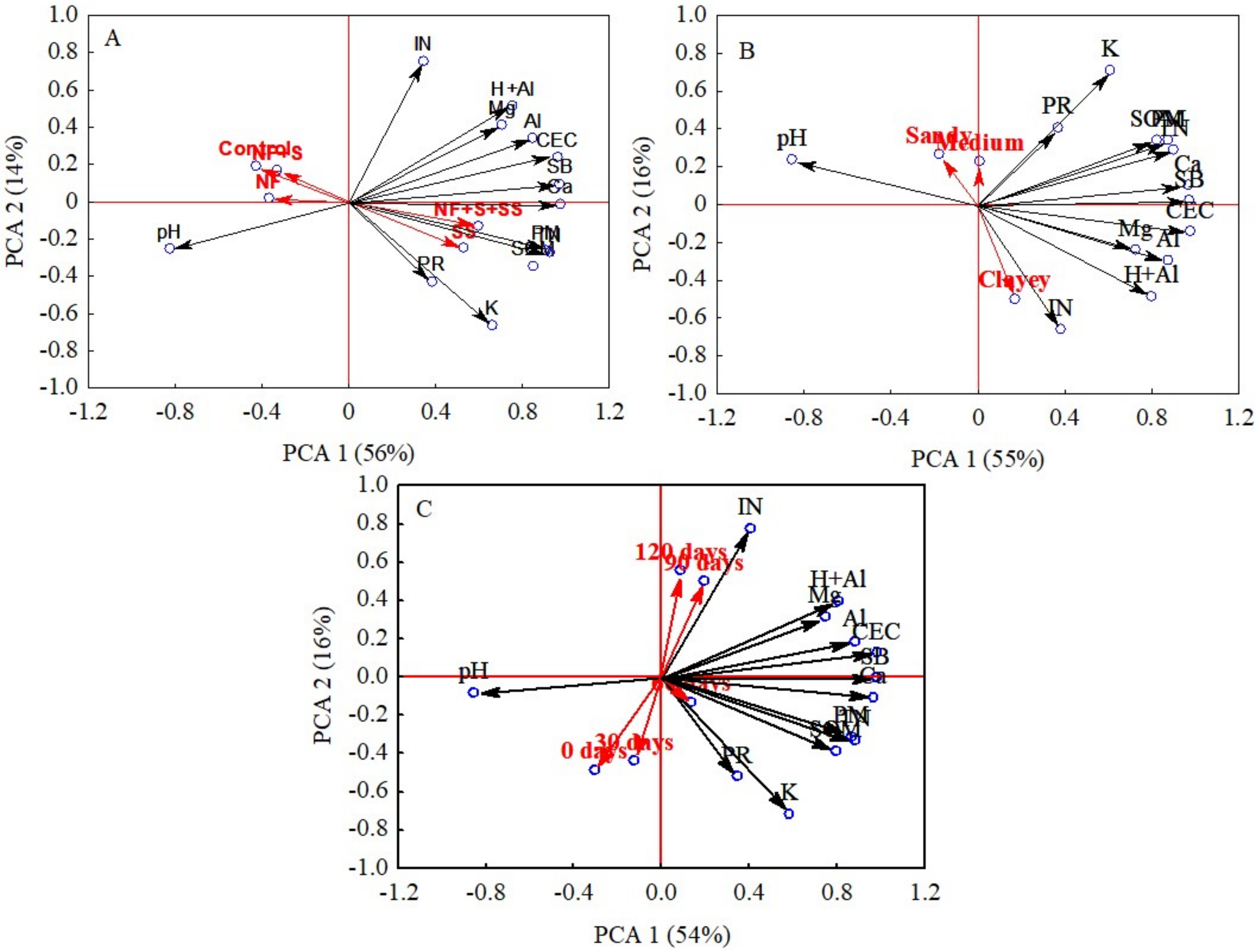


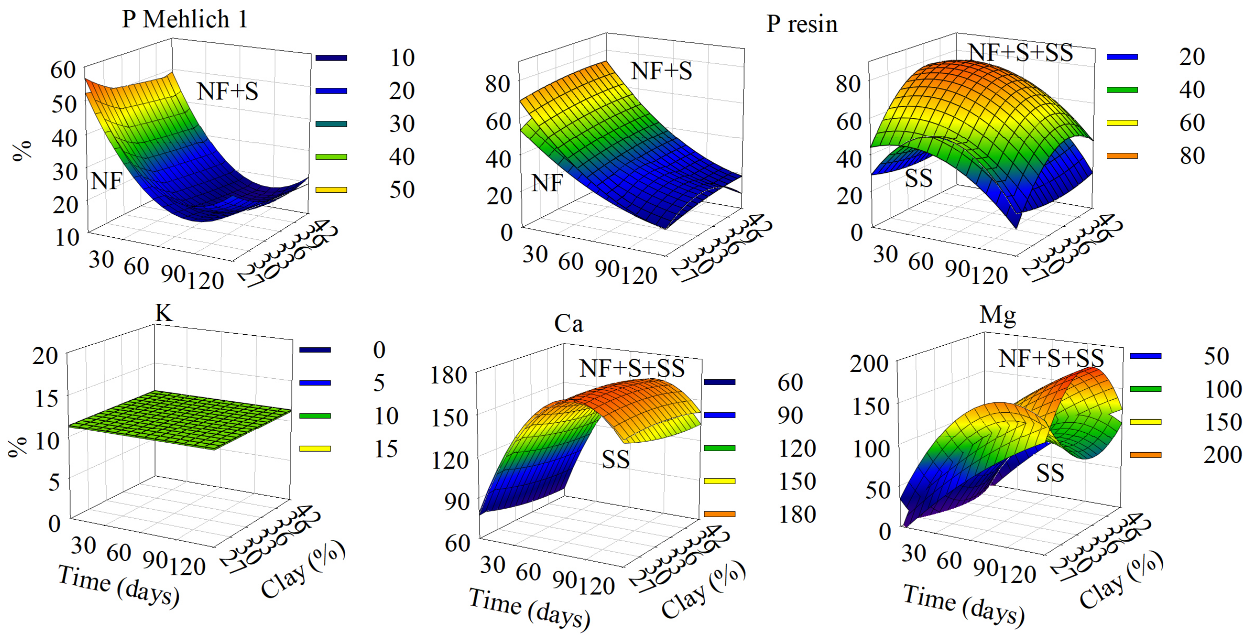
Figure 3. Principal component analysis (PCA) biplot expressing the relationship of the first two principal components with soil characteristics and fertilization management -A; soil texture (Sandy, Medium and Clayey) – B; or evaluation time - C.

to the formation of organic acids and the process of waste nitrification. On the other hand, Borba et al. (2018) monitored a dystrophic dark-red Oxisol for ten years and observed higher acidification of the solution in the first ten years after the application of sewage sludge. This pH reduction can be attributed to the oxidation of nitrite into nitrate (Borba et al., 2018) due to the microbial decomposition of soil organic matter (SOM).

The SOM content increased with the application of sewage sludge compared to the control treatment. However, with the increased incubation time and reduced soil clay contents, part of these compounds was decomposed, thus decreasing the initial SOM content (Fig. 1B, Table 3). Similar results were found by Pegoraro et al. (2020), Mota et al. (2021), and Gonçalves et al. (2021), whose studies showed that the application of sewage sludge increased the SOM contents of tropical soils. The lower buffering capacity of sewage sludge increased the decomposition rate of SOM from sewage sludge in tropical soils. According to Gonçalves et al. (2021), the C mineralization rate, the main constituent of SOM, can be higher than 45% in sandy soils after 120 days of soil incorporation.

In soil incorporated with sewage sludge for six years, the added material positively influenced the soil's chemical features, including pH, SOM, and the CEC and phosphorus contents (Costa et al., 2014). The increase in the nutrient contents after fertilization with sludge or with sludge associated with natural phosphorus and elemental sulfur can also be attributed to the mineralization of nutrients present in its organic fraction, the solubilization of acid compounds, and physicochemical changes in the soil colloid fraction.

Adding organic compounds increases the formation of functional groups with negative surface charges in the organic matter of tropical soils (Muraishi et al., 2011). This phenomenon facilitates the formation of organic-mineral bounds with the solid phase of the soil (oxides) and blocks adsorption sites, thus increasing the solubility of the phosphate anion and the CEC of the soil (Andrade et al., 2013). In that regard, Mota et al. (2021) observed that fertilization with SS resulted in an increase of 318 and 158% in the soil P content in the 0 – 0.20 and 0.20 – 0.40 m depth layers, respectively. Furthermore, Rehman and Qayyum (2020) also reported an increase in the soil phosphorus content in response to the addition of sludge in view of the high concentration of this element in the material, the reduction in the specific adsorption of phosphates present in the soil, and the increased bioavailability.



P Mehlich 1

$NF=66.75-0.698^{ns}x-0.741*y+0.006^{ns}x^2+0.0042*y^2$ $R^2=0.87$

$SS=636.20$

$NF+S=156.99-5.789^{ns}x-0.7693*y+0.077^{ns}x^2+0.0045*y^2$ $R^2=0.88$

$NF+S+SS=745.87$

P resin

$NF=-157.25+13.07^{ns}x-0.543*y-0.195*x^2+0.0018^{ns}y^2$ $R^2=0.95$

$NF+S=25.03+2.49*x-0.785*y-0.032*x^2+0.0027*y^2$ $R^2=0.83$

$SS=111.93-4.97^{ns}x+0.929*y+0.070^{ns}x^2-0.0082*y^2$ $R^2=0.87$

$NF+S+SS=-389+25.30*x+0.24^{ns}y-0.34*x^2-0.004^{ns}y^2$ $R^2=0.84$

K

$NF=0.00$

$SS=11.21$

$NF+S=0.00$

$NF+S+SS=11.39$

Ca

$NF=87.01$

$SS=19.93+3.757^{ns}x+2.244*y-0.0608^{ns}x^2-0.0138*y^2$ $R^2=0.88$

$NF+S=123.31$

$NF+S+SS=167.3-4.80^{ns}x+2.29*y+0.06^{ns}x^2-0.015*y^2$ $R^2=0.84$

Mg

$NF=0.00$

$SS=1023.94-58.24^{ns}x+2.984*y+0.799^{ns}x^2-0.017*y^2$ $R^2=0.70$

$NF+S=0.00$

$NF+S+SS=-1360.8+82.54^{ns}x+1.57*y-1.20^{ns}x^2-0.004*y^2$ $R^2=0.71$

Figure 4. Percentage of P, K, Ca, and Mg recovered via extractors from soils with different clay contents and incubation times (120 days) after the application of natural phosphate (NF), sewage sludge (SS), natural phosphate with elemental sulfur (NF + S), and sewage sludge with natural phosphate and elemental sulfur (NF + S + SS).

Applying elemental sulfur in association with organic compounds of sludge and natural phosphate increases the biological availability, soil acidification, and nutrient mineralization rate, especially nitrogen, phosphorus, calcium, and magnesium (Fig. 1). Nadeem et al. (2022) observed reduced soil pH and higher availability of P and micronutrients after applying elemental sulfur with cattle manure rich in oxidizing bacteria in calcareous soil. In the presence of sulfur-oxidizing bacteria, elemental sulfur is converted into sulfuric acid, which reduces the soil pH and increases the availability of phosphorus, manganese, calcium, magnesium, and sulfate for plants (Khan et al., 2020; Serri et al., 2021; Nadeem et al., 2022).

Quadratic responses for the increase in the P, Ca, and Mg contents (Fig. 1F, H and I) and the sum of bases (Fig. 2B, Table 4) in the soil treated with sludge (SS) and with sludge plus natural phosphate and sulfur-(NF + S + SS), indicate that the biological activity of the soil provided higher nutrient mineralization rates in the incubation period, which ranged from 30 to 90 days. According to Pradhan et al. (2021), longer incubation times (minimum of 42 days) were necessary to increase the solubilization of phosphorus from sewage sludge, supplemented with sulfur and acidifying bacteria (*Acidithiobacillus thiooxidans*). Under this management condition, the presence of sulfur and bacteria increases the nutrient mineralization efficiency in the medium for up to 63 days after incubation.

For the principal component analysis, the first components (PC1) for the three factors explained 56, 55, and 54% of data variability, positively influencing most chemical

Table 5. Percentage of P, K, Ca, and Mg made available via extractors 120 days after fertilization with natural phosphate (NF), elemental sulfur (S), and sewage sludge (SS) in soils with a sandy (28% clay), medium (34% clay), and clayey texture (42% clay).

	P-Resin			**K**		
	Sandy (28)	Medium (34)	Clayey (42)	Sandy (28)	Medium (34)	Clayey (42)
	Percentage (%)					
NF	19.69	20.53	12.66			
SS	33.02	19.91	22.56	9.86	11.34	10.35
NF+S	19.24	18.39	18.28			
NF+S+SS	34.76	37.11	39.42	11.34	15.78	10.85
Mean	26.68	23.99	23.23	4.24	5.42	4.24
	Ca			**Mg**		
	Sandy (28)	Medium (34)	Clayey (42)	Sandy (28)	Medium (34)	Clayey (42)
	Percentage (%)					
NF	110.34	39.66	80.17			
SS	148.01	153.91	150.81	215.13	30.17	49.85
NF+S	141.38	60.34	74.14			
NF+S+SS	154.69	136.80	139.46	227.38	190.65	44.60
Mean	110.88	78.14	88.92	88.50	44.16	18.89

NF: soil fertilized with natural phosphate; SS: soil fertilized with dehydrated sewage sludge; NF + S: soil fertilized with natural phosphate and elemental sulfur; NF + S + SS: soil fertilized with natural phosphate, sewage sludge, and elemental sulfur.

attributes evaluated, except soil pH (Fig. 3). For the factor referring to the different fertilization methods, the control, natural phosphate, and natural phosphate plus elemental sulfur treatments did not influence any soil chemical attributes (Fig. 3A). In contrast, the treatment with sewage sludge alone and with natural phosphate and elemental sulfur affected all chemical attributes evaluated except pH, which was not influenced by any treatment (Fig. 3A).

The soil with the highest clay content (clayey texture = 42%) influenced the mineral N parameter and had a lower effect on the H + Al, Mg, and CEC variables (Fig. 3B). The soils with lower clay contents (sandy - 28% and average textures - 34%) did not influence the chemical attributes evaluated (Fig. 3B). Soil pH was not influenced (Fig. 3B). Moreover, for the factor referring to the days after incubation, the periods after 0 and 30 days of incubation did not influence any of the soil chemical attributes (Fig. 3C). However, after 60 days of incubation, there was an influence on the chemical attributes of PR, K, SOM, and Ca (Fig. 3C). After 90 and 120 days of incubation, the mineral N content in the soil was also influenced, probably due to the longer decomposition time and the resulting N release into the soil solution.

In summary, sewage sludge showed a potential for increasing soil fertility due to the considerable organic matter and nutrient contents for plants (Usman et al., 2012; Gonçalves et al., 2021). The increase in N and P and the improvement in the physical, chemical, and biological soil properties are detectable in the soil that received sewage sludge (Saha et al., 2017; Pegoraro et al., 2020). The use of biosolids certainly contributes to improving the general soil conditions, which is essential for sustaining soil productivity and giving a useful destination for sewage sludge (Usman et al., 2012; Saha et al., 2017; Mota et al., 2021).

Nutrient availability rate (AR) in the soil

Sewage sludge and natural phosphate increased the nutrient availability rate of P, K, Ca, and Mg, indicating considerable contributions to plant nutrition over the incubation time and at the end of the evaluation period (Fig. 4, Table 4). The soil clay content was less influenced by the nutrient availability rate.

The use of elemental sulfur associated with natural phosphate (NF + S) increased the P availability rate in the soil compared to the isolated use of natural phosphate (Fig. 4), a behavior observed for both extractors (Mehlich 1 and Resin) and attributed to soil acidification and the solubilization of calcium phosphate present in NF. However, over the incubation time, P availability decreased by 74% in the initial period for sludge percentages under 30% after 40 days of application, indicating a strong interaction of phosphorus made available with soil clay minerals.

In the soils fertilized with SS and NF + S + SS, the anion-exchange resin was more sensitive to changes in the available form of phosphorus over the incubation time and clay content than the Mehlich 1 extractor (Fig. 4). This behavior is possible due to its extraction capacity only with labile forms of phosphorus in an alkaline medium (P-solution, P-Fe, P-Al). In contrast, the Mehlich 1 extractor also solubilizes P forms bound to Ca in an acid medium and is present in natural phosphate and sewage sludge (Mumbach et al., 2018).

Fertilization with sludge, especially in the presence of elemental sulfur (NF + S), reduced phosphorus adsorption and increased its availability in soils with different clay contents and over the incubation time by the Resin extractor (Fig. 4). From this perspective, the highest phosphorus availability rates were obtained from 32 to 56 days after fertilization with NF + S + SS and NF + S, corresponding to 81 and 55% in soils with 37 and 27% of clay, respectively. These results confirm the positive effect of the associated or individual use of organic fertilizer sources and elemental sulfur as alternatives to increasing the contents and the availability time of phosphorus in weathered tropical soils. The incubation times and clay contents did not interfere with the K availability rate in the soil (Fig. 4). However, only an average rate of 11% of availability was obtained for the treatments with SS and NF + S + SS, whereas the treatments with NF andNF + S showed no K availability (Fig. 4) due to the absence of this element in the composition of natural phosphate and the lower K contents in sewage sludge (Table 1).

The fertilization treatments used in this study increased the Ca and Mg availability rates in the soils (Fig. 4). The highest increases occurred in the NF + S + SS treatment, corresponding to 169% for Ca 80 days after incubation in soil with 31% clay and 194% for Mg 120 days after incubation in soil with 34% clay. This scenario suggests that the application of sludge alone or sludge with elemental sulfur increased Ca and Mg availability in the soils over time due to the increase in the mineralization rates resulting from microbial decomposition, the solubilization of native Ca and Mg in the soil, and the high contents of these nutrients in the fertilizers used (Table 1).

At the end of the evaluation period, 120 days after the application of fertilizers, the following descending order was observed for the mean rate of soil nutrient availability: Ca>Mg>P>K (Table 5), with the highest means corresponding to the soils with lower clay contents and after fertilization with NF + S + SS or SS. Other studies also described the contribution of sludge compounds to nutrient availability. Gonçalves et al. (2021) did so when using dry sewage sludge with variable textures, finding P, Ca, and Mg availability rates higher than 50, 20, and 40%, respectively, during the 120-day evaluation period. Backes et al. (2013) observed that the application of an organic compost produced with sewage sludge after 120 days resulted in mineralization rates of 100, 90, 57, 40, and 31% of Mg, K, S, P, and Ca, respectively, contributing to the nutrition of zoysiagrass sod cultivated in Itapetinga, SP. The application of solid waste from municipal sewage sludge can improve the yield of agricultural crops, support macronutrient cycling, increase sustainability by reducing the disposal of ash and sludge, and reduce the dependency on mineral fertilizers (Antonkiewicz et al., 2020). However, disregarding these nutrients in the recommendation system for fertilization with sewage sludge can lead to a nutrient imbalance in crops and the excessive accumulation of nutrients in the soil. Therefore, one alternative to prevent this scenario would be to use slow-release fertilizers with biosolid coating with urea (Antille et al., 2013).

The use of sewage sludge biochar also increased the N and P contents in the soil and maize plants after the application of 60 t ha^{-1} (Gonzaga et al., 2017). However, despite the indication of an increase in the soil availability of these nutrients, the characterization of the mineralization rate and the recovery of macronutrients from the soil by extractors after fertilization with organic compounds still needs to be a management practice recommended in agriculture. From this perspective, using organic compounds in typically acid tropical soils, poor in SOM, N, P, and exchangeable bases, greatly improves the chemical, physical, and biological soil conditions. As a result, these improvements promote savings with mineral fertilizers and contribute to adopting conservationist and sustainable cultivation practices.

4. Conclusion

The individual application of sewage sludge or its use with natural phosphate and elemental sulfur increases the contents of SOM, N, P, K, Ca, Mg, Al, H+Al, SB and CEC and reduces the soil pH.

After 120 days of incubation, the K and BS contents decreased in all soil textures, whereas the SOM decreased in soils with sandy and medium textures. The availability rate of P, K, Ca, and Mg by the extractors was positive in sewage sludge and natural phosphate.

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Ethical approval

This manuscript does not report on or involve the use of any animal or human data or tissue. So the ethical approval is not applicable.

Author contribution

The authors confirm the study conception and design: Rodinei Facco Pegoraro, Jefferson Rodrigo Teixeira Silva; data collection: Rodinei Facco Pegoraro, Jefferson Rodrigo Teixeira Silva, Verônica Aparecida Santos Ferreira Soares; analysis and interpretation of results: Jefferson Rodrigo Teixeira Silva, Rodinei Facco Pegoraro, Leidivan Almeida Frazão, Regynaldo Arruda Sampaio, Silvana Ferreira Bicalho; draft manuscript preparation: Jefferson Rodrigo Teixeira Silva, Rodinei Facco Pegoraro, Silvana Ferreira Bicalho. The results were evaluated by all authors, and the final version of the manuscript was approved.

Availability of data and materials

Data presented in the manuscript are available via request.

Conflict of interest statement

The authors declare that there are no conflicts of interest associated with this study.

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