



Carbon and nitrogen mineralization and macronutrient recovery in soils amended with sewage sludge

Paula Wellen Barbosa Gonçalves¹ · Paulo Henrique Silveira Cardoso³ · Gustavo de Oliveira Alves² · Reginaldo Arruda Sampaio² · Alcinei Místico Azevedo² · Rodinei Facco Pegoraro²

Received: 25 July 2021 / Accepted: 8 November 2021 / Published online: 22 November 2021
© Saudi Society for Geosciences 2021

Abstract

The recommended dose of sewage sludge is based on the availability of mineral nitrogen and its mineralizable organic fraction, which varies according to sewage sludge, soil type, and several climatic factors. This study evaluated the mineralization and recovery of macronutrients from dried and composted sewage sludge in clayey, medium, and sandy soils textures for 120 days. The behavior of organic carbon (OC), mineral and organic nitrogen (ON), and the available P, K, Ca, and Mg in the soil were investigated. The sewage sludge showed to be an alternative source of P as it presented a mean availability rate higher than 72%. The most severe sewage sludge degradation occurred in the first weeks of monitoring. The highest OC mineralization rate (MR) were equal to or greater than 30% and occurred in the first 30 days of evaluation in all treatments. The MR for OC and ON in the soils that received the residues, at the end of 120 days of monitoring, were greater than 45%, indicating that in tropical soils the mineralization occurs intensely. Soil texture influenced the N mineralization of sewage sludge, with higher MR in the soils with higher sand concentration, for both residues. In field conditions, the application of dried and composted sewage sludge showed ON mineralization rates above 50% for clayey, medium, and sandy soils.

Keywords Biosolid · Decomposition · Mineralization rate · Organic matter · Residue recycling

Introduction

Sewage sludge (SS) is a by-product of the wastewater treatment process and its disposal can generate serious environmental problems if carried out incorrectly. Agricultural, landscaping, and forestry are the most appropriate alternatives for SS disposal due to its potential use as organic fertilizer and soil amendment due to its high concentration of organic matter and nutrients such as nitrogen, phosphorus, and micronutrients (Melo et al. 2018).

However, SS has some restrictions, such as the risk of contamination of the soil with trace metals, organic substances, and pathogens (Murray et al. 2019). Besides, there is the possibility of N leaching, the occurrence of attraction of insect vectors, and the release of odors when the residue is not sufficiently stabilized, compromising directly or indirectly the environment and human health. Sewage sludge post-treatment or stabilization processes reduce the pathogenic load, odor, and attraction of insects, out of which, composting and anaerobic and aerobic digestion are the most common. However, these processes can alter the kinetics of N mineralization and the degradation of organic matter (Corrêa and Silva 2016).

The SS use in Brazil is regulated by the National Council for the Environment (Conselho Nacional de Meio Ambiente—CONAMA) through Resolution no. 498, which regulates the SS dose based on various criteria, one of them is the available N concentration (Brazil 2020). The N criterion considers the estimates of the mineralization rate (MR) of sewage sludge organic nitrogen (ON). In this resolution, the mineralization rate will be provided by located studies in each state of the country, representing

Responsible Editor: Stefan Grab

✉ Paula Wellen Barbosa Gonçalves
paulawellen@hotmail.com

¹ Universidade Estadual Paulista "Júlio de Mesquita Filho", (Unesp), Jaboticabal, SP, Brazil

² Universidade Federal de Minas Gerais (UFMG), Agrarian Science Institute, Montes Claros, MG, Brazil

³ Center of Nuclear Energy in Agriculture, Universidade de São Paulo (USP), Piracicaba, SP, Brazil

the importance of this study, especially in the Cerrado-Caatinga transition area in Brazil.

The SS dose for soil application is calculated based on the N concentration and the ON mineralization rate. To verify the release of mineral N to the soil from the SS, or other sources of organic matter, the material is usually incubated with soil under controlled conditions (Zare and Ronaghi 2019), adjusting to a model that describes the processed as a function of the time. It is then questionable whether the MR of the N in the field would be similar, due to the dynamism of the N. Field incubation ensures closer to real conditions for the mineralization of N, C, and nutrients of the residue. Studies on Brazilian soils with the incubation of SS and their by-products in the field have been reported for MR of C and N above 65% (Backes et al. 2013; Paula et al. 2013). Another critical factor to be elucidated is the contribution and availability of the other macronutrients P, Ca, Mg, and K found in the SS, besides the influence of the soil texture on the waste mineralization process.

Sewage sludge may replace or supplement the use of mineral fertilizers. However, the knowledge of the longevity of SS benefits on soil fertility is still limited (Melo et al. 2018). As a result, further studies are necessary to understand the mineralization process and the availability of macronutrients in the soil in regions of tropical climate, specifically in the Cerrado-Caatinga transition area in Brazil, in order to apply correct SS dose to avoid the excess of nutrients in the soil, beyond the nutritional supply of the plant, minimizing the risks of environmental contamination. Thus, the objective of this study was to evaluate the mineralization rate of C and N and the recovery of macronutrients in soils with different textures treated with sewage sludge in two maturation stages.

Material and methods

Site description

The study was carried out at the Instituto de Ciências Agrárias at Universidade Federal de Minas Gerais-ICA/UFGM, in the municipality of Montes Claros, located in the north of the State of Minas Gerais (MG), latitude 16° 40' 57.70" south, longitude 43° 50' 19.62" west, 650 m above sea level. According to Köppen, the climate in the region is classified as Aw Savannah tropical.

Meteorological data over the study period (August to December 2017) were obtained from Instituto Nacional de Meteorologia (INMET), with rainfall of 13.6 mm in October and 244 mm in December. The average maximum and minimum temperatures observed in the period were 32.8 and 18.2 °C, respectively (INMET 2017).

Soil collection for texture construction and sewage sludge acquirement

Soil was collected from a horizon B layer of a clayey Nitisol (IUSS 2015), with the purpose of reducing the interference of native organic matter of the soil. Then, the soil was mixed with fine sand (< 2 mm) in order to obtain proportions of approximately 25 and 35% of clay, thus composing different textural classes. This was done to guarantee results referring only to the soil granulometry on the mineralization of C and N of the SS and availability of the macronutrients, avoiding the interference of the mineralogical variation of different soils. The soil-sand mixture was homogenized in batches and maintained for an incubation period of 10 cycles of subsequent wetting and drying, followed by stirring the soil at each cycle and moistening to 70% field capacity (Maluf et al. 2015).

The granulometric analysis was performed according to Moniz et al. (2009). The texture of the Nitisol was classified as clayey with 13% of sand, 31% of silt, and 56% of clay. From this soil, a medium-texture soil was obtained, with 42, 21, and 37% of sand, silt, and clay, respectively. And, a sandy soil with 61, 14, and 25% of sand, silt and clay, respectively. Soil chemical attributes were characterized according to Embrapa (1997) (Table 1).

The dried sewage sludge (DS) used was collected from the Copasa Wastewater Treatment Plant in the city of Montes Claros, Minas Gerais, Brazil, where it is treated at 350 °C for 30 min, presenting final humidity around 8%. The composted sewage sludge (CS) was obtained from the DS. Sewage sludge stabilization took place in a furrow, after constant wetting and stirring over the sixty-day composting period. The chemical characterization of the DS and CS was performed according to Alcarde (2009) (Table 1).

Experiment installation

The study was carried out in the field using a complete randomized block design, with four replications, in a factorial scheme with split-plot in time. The plots consisted of 3 × 3 factorial, the subplots were the seven collection times (0, 5, 15, 35, 50, 80, and 120 days after SS application). The factors consisted of three types of soil texture (sandy, medium, and clayey) amended with dried sewage sludge (DS), composted sewage sludge (CS), and without residue (WR).

The experimental units were made up of cylindrical pits open in the soil, with 0.50 m diameter and 0.20 m depth, filled with approximately 40 L the soil-SS mixture, according to each treatment. The SS doses corresponded to the

Table 1 Soil and sewage sludge characterization used in the study

Soil	pH 1:2.5 H ₂ O	P rem [‡] mg L ⁻¹	P [§] mg dm ⁻³	K ⁺	Ca ²⁺ cmol _c dm ⁻³	Mg ²⁺	CEC	H + Al	CO g kg ⁻¹
Clayey	5.5	12.48	1.60	218	3.15	1.39	7.55	2.45	17.80
Medium	6.5	22.84	2.61	147	2.53	0.75	4.91	1.25	11.20
Sandy	6.7	29.03	2.71	117	2.54	0.33	4.58	1.41	9.00
Sludge	N-total g kg ⁻¹	N-NH ₄ ⁺	N-NO ₃ ⁻	ON	OC	Ca	Mg	P	K
Dried at 350 °C	36.50	1.82	0.03	34.64	338.80	9.81	2.02	7.09	3.74
Composted	32.70	0.03	0.43	32.24	269.90	13.89	1.66	10.00	2.80

Methods according to Embrapa (1997)

[‡]Remaining phosphorus

[§]Extracted with Mehlich-1 solution

addition of 1000 kg ha⁻¹ of N, needed for the cultivation of irrigated pineapple with high technological level (Cardoso et al. 2013). According to it, it was applied 1.22 and 1.27 L of DS and CS in 40 L of soil, respectively. So, the amount of organic nitrogen (ON) and organic carbon (OC) applied per kilogram of soil was: 0.51 and 4.28 g for the CS and 0.53 and 5.16 g for the DS, respectively. The experimental plots were irrigated in order to maintain the soil humidity up to 60% of field capacity.

Performed analyses

Soil samples were taken using a PVC probe delimited in 0.20 m at the corresponding period of collection. Soil samples were taken to the laboratory for immediate analysis of mineral nitrogen: ammonium (N-NH₄⁺) and nitrate/nitrite (N-NO_x⁻) using the distiller Kjeldahl (Tedesco et al. 1995). Then, soil samples were air dried, macerated in a porcelain mortar, sieved through a 0.2-mm mesh, and stored until the analyses of the attributes: total organic carbon (OC) (Mendonça and Matos 2005), total nitrogen (TN) (Tedesco et al. 1995), soil pH (1:2.5 H₂O), and available P, K⁺, Ca²⁺, and Mg²⁺. The extractions of P and K⁺ were carried out using Mehlich 1 solution and the extraction of Ca²⁺ and Mg²⁺ were used 1 mol L⁻¹ KCl solution (EMBRAPA 1997).

The concentrations of NT, N-NH₄⁺, and N-NO_x⁻ obtained during the incubation period of the organic residues were used to determine the concentrations of mineral N (Nm) and organic N (ON), according to the Eqs. 1 and 2.

$$Nm = (N - NH_4^+) + (N - NO_x^-) \tag{1}$$

$$ON = NT - Nm \tag{2}$$

Kinetics of sewage sludge organic carbon and nitrogen mineralization in the soil

Concentrations of mineralized N and C of the SS was verified by the difference in soil concentrations between the SS treatments and the control (without the residues). The mathematical model was adjusted by using mineralized and accumulated OC and ON concentrations in time. The kinetics of OC and ON mineralization and availability of inorganic N in the soils were evaluated according to Paula et al. (2013). The data were adjusted by the simple exponential model of first-order chemical kinetics proposed by Stanford and Smith (1972), which was adopted to describe the mineralization process, according to the Eqs. 3 and 4.

$$OC_{(min.)} = OC_{(0)} * (1 - e^{(-kC*t)}) \tag{3}$$

$$ON_{(min.)} = ON_{(0)} * (1 - e^{(-kN*t)}) \tag{4}$$

where OC_(min.), ON_(min.): SS mineralized and accumulated OC (g kg⁻¹) and ON (mg kg⁻¹) per soil mass unit in a specific period; OC₍₀₎, ON₍₀₎: SS potentially mineralizable OC and ON per soil mass unit in a specific time (g kg⁻¹); kC, kN: OC and ON mineralization rate constant in the SS (day⁻¹); t: time referring to the SS incubation period in the soil (days).

Mineralization rate of sewage sludge organic carbon and nitrogen in the soil

The mineralization rates (MR) of the OC and ON of the residues were obtained through the adjustment of the first-order chemical kinetics model, according to Paula et al. (2013). The method uses the adjusted parameters, in the first order exponential equations, which are the values of OC₍₀₎ and ON₍₀₎ and the mineralization coefficients to obtain the values of OC_(min.) and ON_(min.). In this way, Eqs. 3 and 4 are used

to obtain the MR values of OC and ON, obtained by Eqs. 5 and 6.

$$MROC_{(min.)} = 100 * (OC_{(min.)}/OC_{(added)}) \quad (5)$$

$$MRON_{(min.)} = 100 * (ON_{(min.)}/ON_{(added)}) \quad (6)$$

The MR obtained through Eqs. 5 and 6 were calculated with the adjusted parameters, using the amount of OC and ON added per kilogram of soil via SS.

Macronutrient availability rate

The macronutrient concentrations obtained by the routine extractors were used to obtain the availability rate of the P, K⁺, Ca²⁺, and Mg²⁺ in the soils during the period of incubation of the residues. The availability rate of nutrients was calculated using the Eq. 7.

$$AR(\%) = 100 * (tAR - tWR) * tAR^{-1} \quad (7)$$

where AR is the availability rate of a specific nutrient; tAR is the concentration of the nutrient in the soil after SS addition

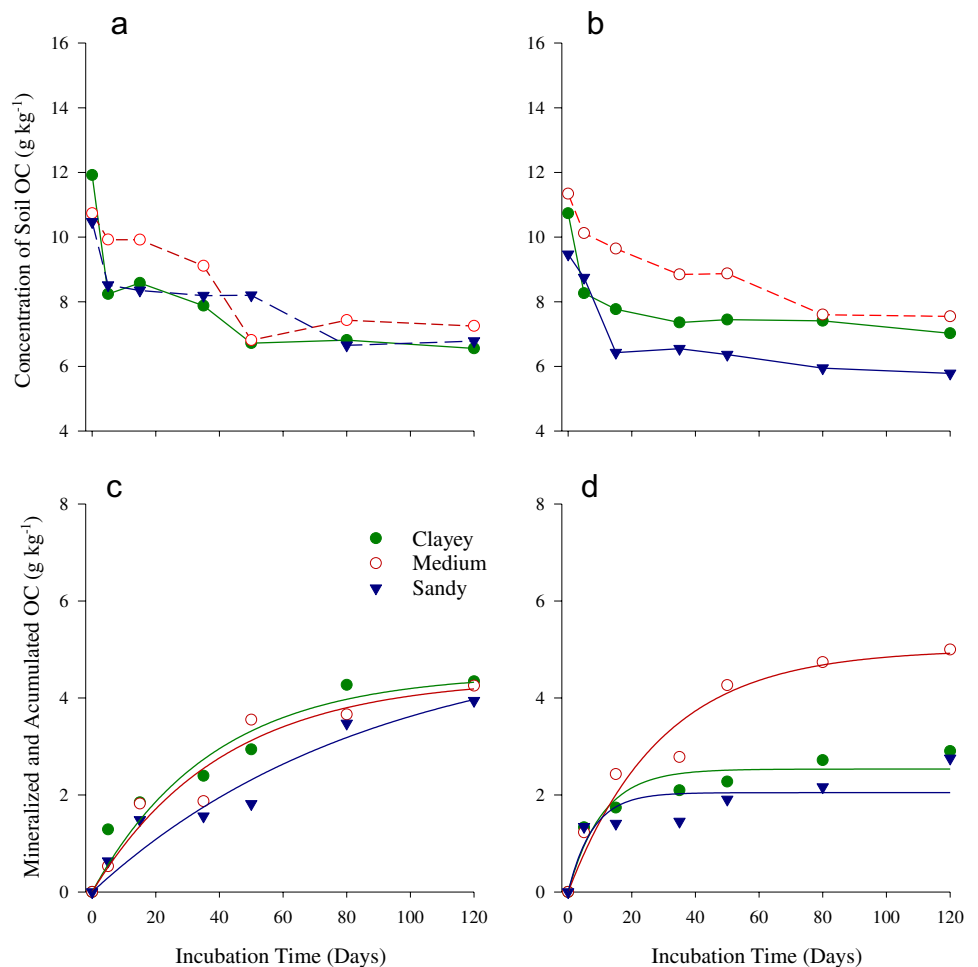
(CS or DS); tWR is the concentration of nutrient available in the control soil (without the residues).

The positive (+) values obtained from AR indicate nutrient availability or mineralization in the soil after SS application; the negative values (–) indicate the non-availability or immobilization of the nutrient; when it is equal to zero (0), it indicates that there was no change in soil nutrient availability.

Statistical analysis

The data obtained in the study were submitted to analysis of variance ($p \leq 0.05$), using a multiple comparison test for the qualitative parameters, texture and residue types, using the Tukey test ($p \leq 0.05$). For the quantitative data, evaluation times, the regression analysis was performed. The analysis was performed using software R (The R Development Core Team 2016). The figures were made in the software SigmaPlot 12.0 (Systat Software, San Jorge, CA, USA).

Fig. 1 Organic carbon concentration and mineralized and accumulated organic carbon concentration in clayey, medium, and sandy soils amended with dried (a, c) and composted (b, d) sewage sludge over the 120-day incubation period



Results

Carbon and nitrogen mineralization

A reduction in the soils OC concentration was found over the evaluation period after DS and CS application (Fig. 1). It was also observed that in the first 20 days, the reduction in soil OC concentration was more intense, with no significant variations after that period (Fig. 1a, b). The lowest OC concentrations were observed in the sandy soil for both SS (Fig. 1c and d). In the sandy and clayey soils, the highest accumulated OC was observed when amended with DS while in the medium soil when it was amended with CS.

The smallest mineralization rate was observed in soils incubated with CS than with DS from the sixtieth day of the study (Table 2). After 30 days of monitoring, MR was already greater than 40% in all treatments, except in sandy soil amended with DS, with almost 30%. At 120 days of monitoring, the highest MR of OC was observed in clayey and medium soils than the sandy soil after DS application, with MR greater than 80%. The same result was observed

with the CS application; however, the MR were 59 and 69% to the clayey and medium soils, respectively.

The highest ammonium (NH₄⁺) concentrations were observed at the first 5 days of DS and CS (Fig. 2a, b) application. The NH₄⁺ and NO_x⁻ concentrations were higher in all soils that received DS (Fig. 2a, c) compared to soils that were amended with CS (Fig. 2b, d). Nitrate forms increased substantially in soils between 45 and 80 days of evaluation, with depletion at 120 days (Fig. 2c, d). Higher mineralized ON levels were observed in both residues in sandy soils after 120 days (Fig. 2e, f). There were no differences between the sewage sludges, except for the sandy soil, that had higher MR with the application of the CS than the DS (Table 2). It was also noticed that the increment of sand increased the ON mineralization rate, regardless the SS, ranging from 50 to 85%.

Soil fertility and macronutrient recovery

The soil pH was influenced by the SS incubation over time (Table 3). An increase by 0.74 pH units was observed at 93 days of monitoring, with a value of 6.43. The pH of the clayey soil remained lower than those of the medium and

Table 2 Coefficients of the first-order chemical kinetics equation and mineralization rate obtained from the organic carbon and organic nitrogen of clayey, medium, and sandy soils fertilized with dried (DS) and composted sewage sludge (CS)

Residue	Texture	Coefficients		R ²	Y ₍₀₎ [§]				MR [#]			
		a [†]	b [‡]		30 ^{††}	60	90	120	30	60	90	120
Organic carbon												
DS	Clayey	4.51 ***	0.026 *	0.93	2.48	3.60	4.10	4.32	48.5	69.7	79.4	83.8
	Medium	4.42 ***	0.025 *	0.95	2.30	3.41	3.93	4.18	44.6	66.0	76.3	81.2
	Sandy	5.33 *	0.011 ns	0.93	1.54	2.64	3.42	3.97	29.9	51.1	66.2	76.9
CS	Clayey	2.53 ***	0.094 *	0.91	2.38	2.53	2.53	2.53	55.7	59.0	59.2	59.2
	Medium	5.01 ***	0.034 ***	0.96	3.32	4.36	4.77	4.92	45.0	61.0	67.0	69.0
	Sandy	2.03 ***	0.129 ns	0.75	2.01	2.05	2.04	2.04	46.9	47.8	47.9	47.9
Organic nitrogen												
DS	Clayey	319 **	0.015 **	0.94	117	191	238	268	22.1	36.0	45.0	50.5
	Medium	491 *	0.010 *	0.93	122	213	282	334	23.0	40.3	53.3	63.0
	Sandy	447 ***	0.019 ***	0.98	193	302	365	400	36.4	57.1	69.0	75.6
CS	Clayey	316 *	0.016 *	0.86	119	194	240	269	23.4	38.0	47.0	52.7
	Medium	452 ns	0.010 ns	0.78	120	209	273	321	23.6	40.9	53.6	62.9
	Sandy	455 ***	0.026 ns	0.94	244	357	409	434	47.8	70.0	80.3	85.1

^{ns}Not significant until 0.05 probability level

*Significant at the 0.05 probability level

**Significant at the 0.01 probability level

***Significant at the 0.001 probability level

[†]Mineralized organic carbon content (OC_(min.); g kg⁻¹), mineralized organic nitrogen (ON_(min.); mg kg⁻¹), and accumulated

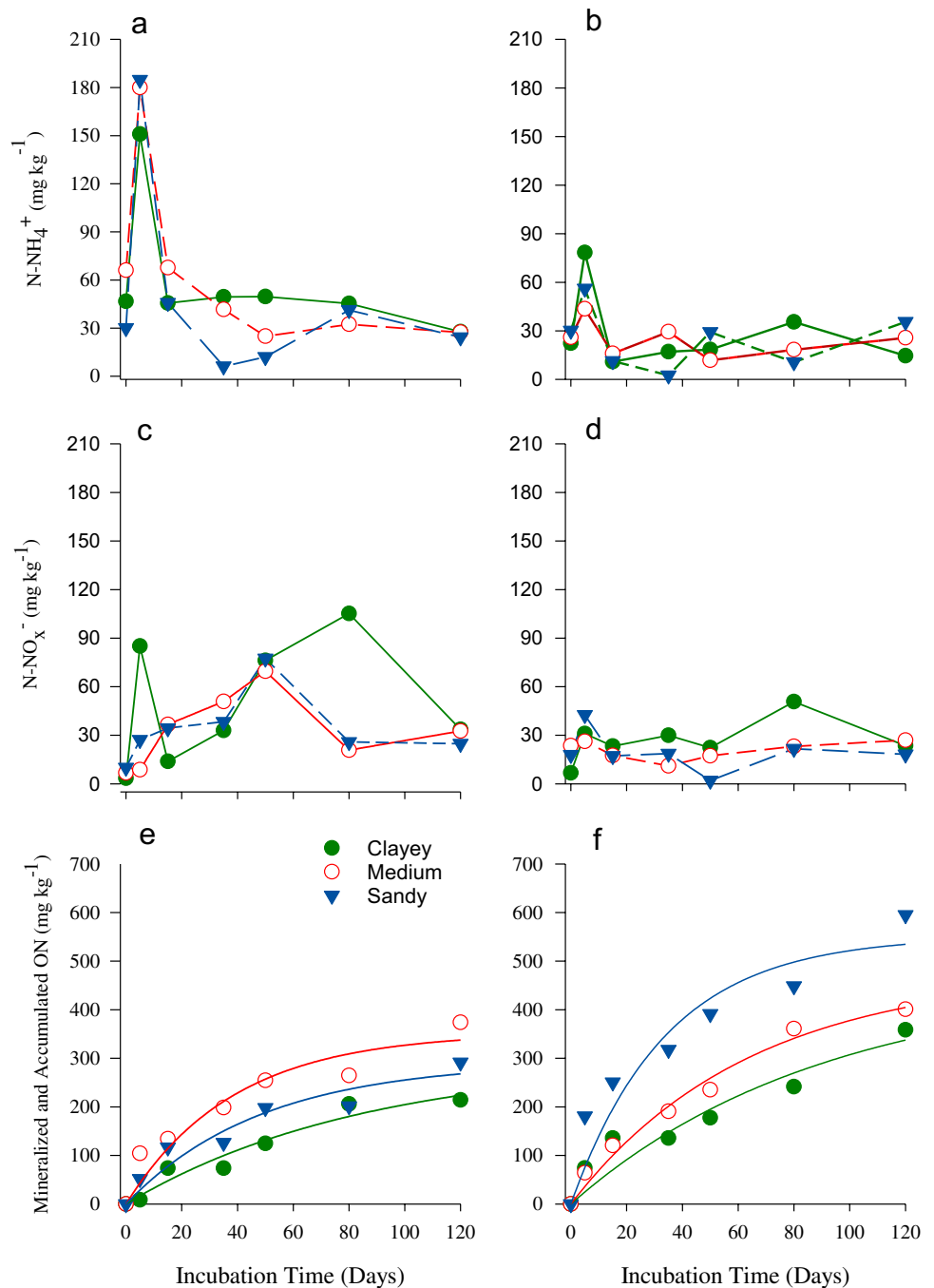
[‡]Mineralization constant rate of OC and ON in the sludge (day⁻¹)

[§]Organic carbon content, in g kg⁻¹, and organic nitrogen, in mg kg⁻¹, potentially mineralizable sludge per soil mass unit over a specific period

[#]OC and ON mineralization rate, in %

^{††}Evaluation days

Fig. 2 NH_4^+ and NO_x^- concentrations and mineralized and accumulated organic nitrogen in clayey, medium, and sandy soils amended with dried (a, c, and e) and composted (b, d, and f) sewage sludge over the 120-day incubation period



sandy soils. In the soils amended with DS, lower values of pH were observed, compared to those with CS and control.

†Values with the same lower-case letters within the texture and residue are not significantly different at $p < 0.05$.

At 120 days of monitoring, potential acidity ($\text{H} + \text{Al}$) was reduced as soil granulometry increased (Table 4). However, it was higher in the soil with DS application. Both residues increased soil CEC and the soils with higher clay concentration showed higher CEC.

Sewage sludge application increased soil P level in comparison to the treatment without residue and clayey soil had lower P level than the others (Table 4). Soils amended with CS had higher P level than the those amended with DS. The P availability rates (AR) were raised regardless of soil texture (Table 5). The application of CS and DS provided high phosphorus AR extracted by Mehlich-1, with AR above 50%. In addition, the residues consistently

Table 3 Effects of dried and composted sludge after the 120-day incubation period at the pH of the soil solution

Parameters		pH	Tukey [†]
Texture	Clayey	5.61	c
	Medium	6.18	b
	Sandy	6.38	a
Residue	Sewage sludge	5.90	b
	Composted sludge	6.10	a
	Without residue	6.20	a
Time incubation	Equation		R ²
	$\hat{y} = 5.69 + 0.016^{**}x - 0.000086^{*}x^2$		0.94

*Significant at the 0.05 probability level

**Significant at the 0.01 probability level

provided P throughout the incubation period, and the highest AR was observed in soils with CS addition.

The soil K⁺ concentrations were reduced as the soil granulometry was increased and the residues did not contribute to its concentration (Table 4). A low availability rate of K⁺ was found in the soil after the application of the residues, with negative values after the first evaluation. The highest ARs were observed in clayey soils amended with DS (Table 5).

Sewage sludge application increased Ca²⁺ and Mg²⁺ levels in the soil, compared to the control treatment (Table 4). The availability rate of Ca²⁺ and Mg²⁺ in soil was steady over the incubation period (Table 5). However, at 120 days after SS application, negative Ca²⁺ availability rates were obtained in the sandy soil.

Discussion

Carbon and nitrogen mineralization

Reduction in OC concentration was associated with increase in soil microbial activity. Sewage sludge incorporated to the soil causes the increase of microorganism population of both bacteria and fungi (Bai et al. 2019; Hamdi et al. 2019), which contributes to the intense degradation of organic matter in the soil due to the evolution of CO₂ by microbial respiration (Soleimani et al. 2019; Xu et al. 2019). Besides, in the process of microbial degradation of the carbohydrates and proteins of SS, other products, such as ammonium and nitrate (Zare and Ronaghi 2019) are also originated. Sandy soils had lower levels of accumulated OC due to the less physical protection by clay (Six and Paustian 2014). On the other hand, the medium and clay soils amended with DS had higher MR of OC, at the end of 120 days (Table 2). These soils have a higher concentration of native OM, and when less recalcitrant organic compounds are added, microorganisms are stimulated, increasing their activity and degrading OM from the soil (Wei et al. 2014).

The lower MR in CS is likely to have occurred due to the higher degree of OM stabilization after the composting process. In the SS composting process, the formation of humic substances occurs by the reduction in the amount of aliphatic compounds and the increase in aromatic compounds, which are less satisfactory as a source of energy for the microorganisms (Černe et al. 2019; Poggere et al. 2019; Yu et al. 2019). Studies on SS mineralization carried out under field conditions in Brazil showed that in tropical climate soils

Table 4 Chemical attributes in clayey, medium, and sandy soils amended with dried (DS) and composted (CS) sewage sludge after 120-day incubation period

Treatment	Clayey	Medium	Sandy	Clayey	Medium	Sandy
P (mg dm ⁻³) [‡]				K ⁺ (mg dm ⁻³)		
DS	7.19 Bb	9.24 Ab	10.22 Ab	184 Aa	160 Bab	122 Ca
CS	10.15 Ba	13.93 Aa	14.51 Aa	177 Aab	156 Bb	125 Ca
WR	1.89 Ac	2.49 Ac	2.56 Ac	177 Ab	166 Ba	128 Ca
Ca ²⁺ (cmol _c dm ⁻³)				Mg ²⁺ (cmol _c dm ⁻³)		
DS	3.94 Aa	3.96 Aa	3.40 Ba	1.80 Aa	1.67 Aa	1.22 Ba
CS	3.88 Aa	4.05 Aa	3.54 Ba	1.66 Aa	1.53 Aa	1.22 Ba
WR	3.12 Ab	3.33 Ab	2.94 Bb	1.29 Ab	1.31 Ab	0.98 Bb
CEC (cmol _c dm ⁻³)				H + Al (cmol _c dm ⁻³)		
DS	8.32 Aa	7.77 Ba	6.68 Ca	2.10 Aa	1.73 Ba	1.73 Ba
CS	8.15 Aa	7.75 Ba	6.63 Ca	2.15 Aa	1.77 Ba	1.55 Cb
WR	6.75 Ab	6.62 Ab	5.64 Bb	1.89 Ab	1.50 Bb	1.40 Bb

Values with the same lowercase letter in the columns and uppercase letter in the rows, within the soil attribute, are not significantly different by the Tukey's test ($p > 0.05$)

[‡]P extracted with Mehlich-1 solution

CS composted sewage sludge, DS dried sewage sludge, WR without residue, CEC cation exchange capacity, H + Al potential acidity

Table 5 Availability rate (%) of the macronutrients (P, K, Ca, and Mg) in clayey, medium, and sandy soils amended with dried (DS) and composted (CS) sewage sludge over 120-day incubation period

Soil Texture	Incubation Time	Nutrient availability rate (%)							
		DS		CS		DS		CS	
		P [†]	K ⁺	Ca ²⁺	Mg ²⁺				
Clayey	0	77.4	82.3	4.9	3.6	20.0	22.7	26.1	28.3
	5	78.1	85.5	7.3	-1.4	25.9	24.8	24.6	30.9
	15	67.3	78.9	10.5	-4.1	12.5	10.9	46.3	41.6
	35	81.4	87.4	10.0	2.5	21.0	22.6	41.4	33.0
	60	61.7	76.1	-0.67	2.1	26.1	18.3	34.1	28.9
	80	86.5	88.2	3.0	-1.3	23.8	18.0	27.4	26.1
	120	53.3	63.0	-3.8	0.3	19.6	17.4	37.7	21.7
	Average	72.2	80.2	4.5	0.2	21.3	19.2	32.9	19.6
	Medium	0	76.8	82.1	2.2	3.7	23.9	21.4	28.4
5		77.9	86.5	3.4	-4.2	13.7	20.1	25.4	21.8
15		72.8	81.6	1.9	-9.1	9.7	17.6	43.8	29.8
35		75.0	83.2	-9.4	-7.0	19.6	20.9	47.5	19.9
60		60.6	80.3	-3.5	-6.0	5.7	12.7	27.9	16.1
80		81.3	88.2	-4.7	-3.2	22.7	11.2	29.0	26.8
120		60.3	69.6	-10.0	-2.6	18.1	21.3	25.9	10.4
Average		72.1	81.6	-2.9	-4.1	16.2	17.9	25.8	10.5
Sandy		0	68.8	82.7	6.7	6.4	21.2	25.9	31.7
	5	77.7	84.0	1.1	-5.0	16.3	24.2	36.5	33.8
	15	77.5	86.3	-1.0	-2.7	17.8	9.9	40.2	52.0
	35	82.2	85.5	-3.0	-5.0	19.6	15.1	41.9	27.9
	60	70.9	78.7	-7.9	-1.0	15.8	20.8	13.0	-27.8
	80	83.3	86.9	-7.8	0.1	14.1	21.4	30.2	-6.7
	120	57.4	63.7	-17.6	-6.2	-7.6	-0.9	40.3	27.5
	Average	74.0	81.1	-4.2	-1.9	13.9	16.6	18.4	15.4

[†] P extracted with Mehlich-1 solution. DS—Dried sewage sludge; CS—Composted sewage sludge

with high temperatures and satisfactory soil moisture, the degradation of the organic matter of the SS occurs rapidly, and likely to obtain MR up to 100% for OC (Paula et al. 2013; Diniz et al. 2016). Such high MR may be related to the priming effect, which results from the intensification of the activity of the microorganisms that consume the readily biodegradable compounds of the OM of the residues. In addition, after their consumption, they induce the degradation of the readily oxidizable OC present in the soil (Paula et al. 2013; Silva et al. 2019).

The degradation of SS was more intense in the first days after its incorporation (Fig. 1) by the decomposition of readily degradable compounds. Another important point to highlight is the composition and characteristics of the SS itself. In its constitution, dried sewage sludge has 42.2% of proteins, 13.2% of lipids, 12.6% of hemicellulose, 9.3% of cellulose, and 21.3% of lignin (Carvalho et al. 2015). Thus, most of the sewage sludge is readily degraded by microorganisms. It is reported that in tropical soils, the degradation of the organic matter of the SS occurs rapidly, which hinders the increase of the soil organic matter.

Concerning the ON mineralization, after the first weeks of the SS incubation, N predominated as nitrate (Carneiro et al. 2013). However, both NH_4^+ and NO_x^- levels were higher in the soils treated with DS (Fig. 2). This is because, at the beginning of the study, 95% of the total N in the DS was in the organic form and 5% in the inorganic form. In the CS, only 1.4% of the total N was in the inorganic form (Table 1). Also, it can be inferred that during the composting process of the dried sewage sludge, inorganic N was lost. Hence, the different stabilization processes guarantee different N concentration. In addition, the higher degree of stabilization of the CS decreased the mineral N release to the soil, in relation to the DS, which gave rise to it (Corrêa et al. 2012), due to the higher humification degree of the residue submitted to composting process (Kulikowska 2016).

The sandy soils showed lower N-mineral concentration after 120 days of incubation, for both residues, which was justified by the lower concentration of clay, original organic matter in the soil itself, and lower CEC (Carneiro et al. 2013). Moreover, the precipitation that occurred at the end of the experimental period is another factor that may

have influenced the lower levels of N-mineral in sandy soils (Fig. 1). This is because nitrate is mobile in the soil, and it can be easily lost by leaching after intense precipitation (Paula et al. 2013).

The lowest C/N ratio in CS (8:1), when compared to the DS (9:1), may have contributed to the higher CS mineralization. High-quality organic matter is the one with the lowest C/N ratio and with enough N to support microbial growth (Masunga et al. 2016), but MR was not significantly different between the residues. The mineralization rates in the sewage sludge submitted to stabilization processes are low because they are constituted of stable organic structures that slow down the mineralization (Corrêa et al. 2012).

Soil attributes and nutrient recovery

The CS has a greater stabilization than the DS (Corrêa et al. 2012). Thus, when DS was incorporated to the soil, an intense process of organic matter decomposition occurred. The degradation of the organic matter of the SS favors the acidification of the soil solution because H^+ is released in this process as result of the formation of organic acids and through the oxidation process of nitrate to nitrite (Borba et al. 2018). The highest values of pH found in sandy and medium soils are due to the use of water with high concentrations of calcium carbonate, about 222 mg L^{-1} , used in the construction of the textures of these soils. The higher pH value in the sandy soil, in relation to the others, is related to its lower buffering power (Table 4), due to the lower clay concentration.

The high availability rates of P show that SS can be used as a secondary source for plant nutrition (Gorazda et al. 2016; Houben et al. 2019). The use of SS would contribute to the reduction in the consumption of natural phosphate deposits since the source of phosphate fertilizers currently mostly processed from phosphate rocks, a practice considered unsustainable because it is a non-renewable natural resource (Gorazda et al. 2016; Wollmann et al. 2018). Hence, the importance of organic sources of P, as an alternative to mineral sources (Blöcher et al. 2012; Gorazda et al. 2016), is highlighted, especially in tropical soils with a high degree of weathering, poor in organic matter and high phosphate specific adsorption capacity. For P, there is no natural recovery process in tropical soils, as occurs with the N cycle, that is, when it is applied, it cannot be recycled naturally in the short term (Gorazda et al. 2016).

The highest P availability rates (AR) in this study occurred with the application of CS (Table 5), justified by the higher P concentration in the CS than in the DS (Table 1). This occurs due to the decomposition of organic matter by the action of microorganisms in the composting process, which releases carbon in the form of CO_2 , promoting the mineralization of nutrients such as P. The AR of P

was raised regardless of the texture (Table 5). This result was attributed to the high levels of P in the residues. Adequate pH conditions of the soil and SS mixture (pH within the range of 5.5 to 6.5) also contributed to the greater availability of P in comparison to other nutrients (Raymond et al. 2018; Shi and Xu 2019). Cherubin et al. (2016) report that although organic matter does not prevent phosphate adsorption process, it does play a fundamental role in the Cerrado soils as it slows down the process, therefore, contributing to the P availability.

Sewage sludge contributes substantially to the increase of Ca^{2+} and Mg^{2+} in the soil and its CEC by the addition of organic matter to the soil (Table 4), with higher charge density, compared to mineral constituents of weathered tropic soils, such as kaolinite and oxides, favoring nutrient retention such as Ca^{2+} and Mg^{2+} .

The low levels and AR of K^+ in the soils are justifiable, since SS has low K^+ concentration (Table 1). This is because K^+ does not take part in any stable organic compound (Antoniadis et al. 2015) and is carried with the water at the end of the sewage treatment. In the soil with higher clay concentration, K^+ availability rates were higher due to its higher CEC (Table 4), providing a lower K^+ loss potential caused by leaching (Werle et al. 2008).

Conclusions

The contribution of sewage sludge to the availability of macronutrients is important, and the availability of P during the entire monitoring period is highlighted, and sewage sludge should be considered as a potential alternative source of nutrients, especially P. The mineralization of C in the sewage sludge dried at $350 \text{ }^\circ\text{C}$ is intense and greater than that with composted sewage sludge. Meantime, both sewage sludges, in the study under field conditions in a Cerrado-Caatinga transition area in Brazil, showed organic N mineralization rate higher than 50, 60, and 75% in clayey, medium, and sandy soils, respectively. Thereby, soil granulometry had a significant influence on the dynamics of N mineralization of the residues.

Author contribution Conception and design of the work: PG, RP, and RS. Acquisition of data: PG, PC, and GA. Analysis and interpretation of data: PG, PC, and AA. Drafting and revising the work: PG, RP, and PC. All authors contributed to the article and approved the submitted version.

Funding This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior—Brasil (CAPES)—Finance Code 001; by the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), and by the Fundação de Amparo à Pesquisa do Estado de Minas Gerais (FAPEMIG).

Availability of Data and Material Not applicable.

Code availability Not applicable.

Declarations

Conflict of interest The authors have no conflicts of interest to declare that are relevant to the content of this article.

References

- Alcarde JC (2009) Manual de Análise de Fertilizantes, FEALQ, Piracicaba
- Antoniadis V, Koutroubas SD, Fotiadis S (2015) Nitrogen, phosphorus, and potassium availability in manure-and sewage sludge-applied soil. *Commun Soil Sci Plant Anal* 46:393–404. <https://doi.org/10.1080/00103624.2014.983241>
- Backes C, Santos AJM, Godoy LJGD, Villas Boas RL, Oliveira MRD, Oliveira FCD (2013) Doses de lodo de esgoto compostado em produção de tapete de grama esmeralda imperial. *Rev Bras Ciênc Solo* 37:1402–1414
- Bai Y, Mei L, Zuo W, Zhang Y, Gu C, Shan Y, Dai Q (2019) Response of bacterial communities in coastal mudflat saline soil to sewage sludge amendment. *Appl Soil Ecol* 144:107–111. <https://doi.org/10.1016/j.apsoil.2019.07.007>
- Blöcher C, Niewersch C, Melin T (2012) Phosphorus recovery from sewage sludge with a hybrid process of low-pressure wet oxidation and nanofiltration. *Water Res* 46:2009–2019. <https://doi.org/10.1016/j.watres.2012.01.022>. <https://ainfo.cnptia.embrapa.br/digital/bitstream/CNPMA-2010/8585/1/circular-20.pdf>. Accessed in 15 June 2019
- Borba RP, Ribeirinho VS, Camargo OA, Andrade CA, Kira CS, Coscione AR (2018) Ion leaching and soil solution acidification in a vadose zone under soil treated with sewage sludge for agriculture. *Chemosphere* 192:81–89. <https://doi.org/10.1016/j.chemosphere.2017.10.112>
- Brazil (2020) Brazilian National Environment Council. Resolução CONAMA N° 498/2020, August 19, 2020. <http://www2.mma.gov.br/port/conama/legiabre.cfm?codlegi=749>
- Cardoso MM, Pegoraro RF, Maia VM, Kondo MK, Fernandes LA (2013) Crescimento do abacaxizeiro Vitória irrigado sob diferentes densidades populacionais, fontes e doses de nitrogênio. *Rev Bras Frutic* 35:769–781
- Carneiro OWJ, Silva CA, Muniz JA, Villela ST (2013) Mineralização de nitrogênio em Latossolos adubados com resíduos orgânicos. *Rev Bras Ciênc Solo* 37:715–725. <https://doi.org/10.1590/S0100-068320130003000018>
- Carvalho CS, Ribeirinho VS, Andrade CA, Grutmacher P, Pires AMM (2015) Composição química da matéria orgânica de lodos de esgoto. *Ver Bras Ciênc Agr* 10:413–419
- Černe M, Palčić I, Pasković I, Major N, Romić M, Filipović V, Ban D (2019) The effect of stabilization on the utilization of municipal sewage sludge as a soil amendment. *Waste Manag* 94:27–38. <https://doi.org/10.1016/j.wasman.2019.05.032>
- Cherubin MR, Franco AL, Cerri CE, Karlen DL, Pavinato PS, Rodrigues M, Cerri CC (2016) Phosphorus pools responses to land-use change for sugarcane expansion in weathered Brazilian soils. *Geoderma* 265:27–38. <https://doi.org/10.1016/j.geoderma.2015.11.017>
- Corrêa RS, Silva DJ (2016) Effectiveness of five biosolids as nitrogen sources to produce single and cumulative ryegrass harvests in two Australian soils. *Rev Bras Ciênc Solo* 40:e0150216. <https://doi.org/10.1590/18069657rbcs20150216>
- Corrêa RS, White RE, Weatherley AJ (2012) Effects of sewage sludge stabilization on organic-N mineralization in two soils. *Soil Use Manage* 28:12–18. <https://doi.org/10.1111/j.1475-2743.2012.00387.x>
- Diniz IC, Matos AT, Borges AC, Aquino JM, Matos MP (2016) Degradation of sewage sludge compost disposed on the soil. *Eng Agríc* 36:822–829. <https://doi.org/10.1590/1809-4430-EngAgric.v36n5p822-829/2016>
- EMBRAPA – Empresa Brasileira de Pesquisa Agropecuária (1997) Manual de Métodos de Análise de Solo, Rio de Janeiro
- Gorazda K, Tarko B, Wzorek Z, Nowak AK, Kulczycka J, Henclik A (2016) Characteristic of wet method of phosphorus recovery from polish sewage sludge ash with nitric acid. *Open Chem* 14:37–45. <https://doi.org/10.1515/chem-2016-0006>
- Hamdi H, Hechmi S, Khelil MN, Zoghliani IR, Benzarti S, Mokni-Tlili S, Jedidi N (2019) Repetitive land application of urban sewage sludge: Effect of amendment rates and soil texture on fertility and degradation parameters. *CATENA* 172:11–20. <https://doi.org/10.1016/j.catena.2018.08.015>
- Houben D, Michel E, Nobile C, Lambers H, Kandeler E, Faucon MP (2019) Response of phosphorus dynamics to sewage sludge application in an agroecosystem in northern France. *Appl Soil Ecol* 137:178–186. <https://doi.org/10.1016/j.apsoil.2019.02.017>
- INMET – Instituto Nacional de Meteorologia (2017) BDMEP – Banco de dados históricos. <http://www.inmet.gov.br/portal/>. Accessed 20 Aug 2018
- IUSS – International Union of Soil Science Working (2015) World Reference Base for Soil Resources (WRB), <http://www.fao.org/3/a-i3794e.pdf>. Accessed 12 Oct 2019
- Kulikowska D (2016) Kinetics of organic matter removal and humification progress during sewage sludge composting. *Waste Manag* 49:196–203. <https://doi.org/10.1016/j.wasman.2016.01.005>
- Maluf HJGM, Soares BEM, Silva IR, Neves JCL, Silva MFO (2015) Disponibilidade e recuperação de nutrientes de resíduos culturais em solo com diferentes texturas. *Rev Bras Ciênc Solo* 39:1690–1702. <https://doi.org/10.1590/01000683rbcs20140657>
- Masunga RH, Uzokwe VN, Mlay PD, Odeh I, Singh A, Buchan D, Neve S (2016) Nitrogen mineralization dynamics of different valuable organic amendments commonly used in agriculture. *Appl Soil Ecol* 101:185–193. <https://doi.org/10.1016/j.apsoil.2016.01.006>
- Melo WJ, Delarica D, Guedes A, Lavezzo L, Donha R, Araújo A, Melo G, Macedo F (2018) Ten years of application of sewage sludge on tropical soil. A balance sheet on agricultural crops and environmental quality. *Sci Total Environ* 643:1493–1501. <https://doi.org/10.1016/j.scitotenv.2018.06.254>
- Mendonça ES, Matos ES (2005) Matéria orgânica do solo: métodos de análise, UFV, Viçosa
- Moniz AC, Jorge JA, Valadares JMAS (2009) Métodos de Análise Química, Mineralógica e Física de Solos do Instituto Agrônomo de Campinas. Instituto Agrônomo, Campinas
- Murray R, Tien YC, Scott A, Topp E (2019) The impact of municipal sewage sludge stabilization processes on the abundance, field persistence, and transmission of antibiotic resistant bacteria and antibiotic resistance genes to vegetables at harvest. *Sci Total Environ* 651:1680–1687. <https://doi.org/10.1016/j.scitotenv.2018.10.030>
- Paula JR, Matos AT, Matos MP, Santos PM, Andrade CA (2013) Mineralização do carbono e nitrogênio de resíduos aplicados ao solo em campo. *Rev Bras Ciênc Solo* 37:1729–1741. <https://doi.org/10.1590/S0100-06832013000600029>
- Poggere GC, Melo VF, Serrat BM, Mangrich AS, França AA, Corrêa RS, Barbosa JZ (2019) Clay mineralogy affects the efficiency of sewage sludge in reducing lead retention of soils. *J Environ Sci* 80:45–57. <https://doi.org/10.1016/j.jes.2018.07.017>
- R Core Team (2016) R: A Language and Environment for Statistical Computing. R Foundation for Statistical Computing, Vienna, Austria. <https://www.R-project.org/>

- Raymond NS, Stöver DM, Peltre C, Nielsen HH, Jensen LS (2018) Use of *Penicillium bilaiae* to improve phosphorus bioavailability of thermally treated sewage sludge—A potential novel type biofertiliser. *Process Biochem* 69:169–177. <https://doi.org/10.1016/j.procbio.2018.03.021>
- Shi S, Xu G (2019) Identification of phosphorus fractions of biofilm sludge and phosphorus release, transformation and modeling in biofilm sludge treatment related to pH. *Chem Eng J* 369:694–704. <https://doi.org/10.1016/j.cej.2019.03.120>
- Silva DAP, Matos AT, Matos MP (2019) Mineralization of organic matter and productivity of tifton 85 grass (*Cynodon* spp.) in soil incorporated with stabilized sludge from a vertical flow constructed wetland. *J Water Sanit Hyg De* 9:309–318. <https://doi.org/10.2166/washdev.2019.133>
- Six J, Paustian K (2014) Aggregate-associated soil organic matter as an ecosystem property and a measurement tool. *Soil Biol Biochem* 68:A4–A9. <https://doi.org/10.1016/j.soilbio.2013.06.014>
- Soleimani A, Hosseini SM, Bavani ARM, Jafari M, Francaviglia R (2019) Influence of land use and land cover change on soil organic carbon and microbial activity in the forests of northern Iran. *CATENA* 177:227–237. <https://doi.org/10.1016/j.catena.2019.02.018>
- Stanford G, Smith SJ (1972) Nitrogen mineralization potentials of soils. *Soil Sci Soc Am J* 36:465–471
- Tedesco MJ, Gianello C, Bissani CA, Bohnen H, Volkweiss SJ (1995) *Análise de solo, plantas e outros materiais*, second ed. UFRGS, Porto Alegre
- Wei H, Guenet B, Vicca S, Nunan N, Asard H, AbdElgawad H, Janssens IA (2014) High clay content accelerates the decomposition of fresh organic matter in artificial soils. *Soil Biol Biochem* 77:100–108. <https://doi.org/10.1016/j.soilbio.2014.06.006>
- Werle R, Garcia RA, Rosolem CA (2008) Lixiviação de potássio em função da textura e da disponibilidade do nutriente no solo. *Rev Bras Cienc Solo* 32:2297–2305. <https://doi.org/10.1590/S0100-06832008000600009>
- Wollmann I, Gauro A, Müller T, Möller K (2018) Phosphorus bioavailability of sewage sludge-based recycled fertilizers. *J Soil Sci Plant Nutr* 181:158–166. <https://doi.org/10.1002/jpln.201700111>
- Xu W, Yuan W, Cui L, Ma M, Zhang F (2019) Responses of soil organic carbon decomposition to warming depend on the natural warming gradient. *Geoderma* 343:10–18. <https://doi.org/10.1016/j.geoderma.2019.02.017>
- Yu Z, Liu X, Zhao M, Zhao W, Liu J, Tang J, Liou H, Chen Z, Zhou S (2019) Hyperthermophilic composting accelerates the humification process of sewage sludge: molecular characterization of dissolved organic matter using EEM–PARAFAC and two-dimensional correlation spectroscopy. *Bioresour Technol* 274:198–206. <https://doi.org/10.1016/j.biortech.2018.11.084>
- Zare L, Ronaghi A (2019) Comparison of N mineralization rate and pattern in different manure-and sewage sludge-amended calcareous soil. *Commun Soil Sci Plant Anal* 50:559–569. <https://doi.org/10.1080/00103624.2019.1573247>