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Research article

Agricultural use and pH correction of anaerobic sewage sludge with acid pH

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ABSTRACT

Agricultural use is the main way of recycling sewage sludge. Besides providing nutrients and organic matter to crops and soils, it is an important alternative for recycling this residue. However, problems during the sewage treatment process may generate sludge batches with an acidic pH. Thus, it is essential to understand the consequences of using such sludge on soils and plants, and to explore ways to overcome this limitation. The objective of this study was to evaluate addition rates of anaerobic sewage sludge (ASS) with acidic compositions on the soil fertility and performance of lettuce plants. Additionally, a methodology for pH correction of ASS with acidic pH is proposed. An agronomic experiment was conducted in a greenhouse using seven addition rates of ASS (0.0, 0.25, 0.5, 1, 2, 4 and 8 g kg^{-1} in dry basis), treated with an additional step of disinfection (solarization), and applied in an Albaqualf soil cultivated with lettuce (*Lactuca sativa*). Soil and leaf chemical composition, as well as chlorophyll index and the dry matter of lettuce leaves were evaluated. Failures during the acidogenesis phase of the anaerobic digestion process were probably the cause of ASS acidification. Although this ASS increased soil fertility indicators and plant dry matter, it significantly reduced soil pH, thereby requiring a complementary assay to correct its pH up to 6.0, which was achieved through liming. Anaerobic sewage sludges with an acidic pH can be effectively used in agriculture after being dried and disinfected through solarization, followed by pH correction, avoiding negative impacts on soil chemical attributes and plant response.

1. Introduction

Wastewater treatment has led to the production of large and increasing amounts of sewage sludge (SS) worldwide (Wang et al., 2019). In order to manage SS, several methods have been used including incineration, disposal in landfills, anaerobic digestion and composting (Chen et al., 2014). However, due to the high organic matter, phosphorus, nitrogen and micronutrients content of SS, agricultural use can be seen as an appropriate destination for its management, besides being able to be transformed into value-added products such as fertilizers and soil conditioners (Bravo-Martín-Consuegra et al., 2016; Kacprzak et al., 2017).

The use of SS as a soil conditioner and organic fertilizer improves not only soil macro and micronutrients but also its physical characteristics, e.g. reducing bulk density and increasing macroporosity, favorin2005g water retention and cation exchange capacities and its use in benefitting the soil microbial population (Fia et al., 2005; Hamdi et al., 2019). De Maria et al. (2007) evaluated the use of SS as soil conditioner using addition rates of 5 and 10 g kg^{-1}, and observed an increase in organic matter content and stability of soil aggregates in the 0.0–0.1m layer after two consecutive annual applications.

On the other hand, the use of SS in agriculture holds with it the concern of contamination to soils, water and living organisms, since SS may carry pathogenic contaminants, heavy metals and synthetic organic compounds (Kończak and Oleszczuk, 2018; Hamdi et al., 2019). In Brazil, agricultural use of SS and their derived products is regulated by Resolution No. 375 of August 29, 2006, of the National Environment Council (BRAZIL, 2006), which poses limits for heavy metals, organic

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compounds and pathogenic contaminants. Generally speaking, when pathogenic contaminants are above the limiting levels, agricultural application is allowed only after proper disinfection, albeit under some specific restrictions. In order to reduce pathogens, various procedures can be employed, including lime stabilization, composting, solar drying for long periods of time, drying with heating, in addition to thermal treatment by heating, irradiation with beta rays and pasteurization.

In this context, the solarization of sewage sludge in agricultural greenhouses or in drying beds covered by translucid plastic may be an alternative as an additional disinfection method. Especially in countries with a tropical climate, this method presents a low-cost alternative with little complexity of execution, besides resulting in a reduction of about 80% of the SS volume through dehydration, achieving satisfactory sanitation and allowing for its incorporation into the soil (Vijayavenkataraman et al., 2012; Mathioudakis et al., 2013). In a study by Lima et al. (2009) SS solarization presented promising results, with SS reaching National resolution 375/2006 requirements after 28 days of solar exposure. Authors have recommended the use of this SS in agriculture, however the need for more studies about the agronomic potential of this material remains.

Nonetheless, sewage treatment plants operating with anaerobic digestion may also generate sewage sludge batches with acidic conditions. Acidity and alkalinity conditions may occur during the anaerobic digestion process, and the low pH values are generally associated with high concentrations of volatile fatty acids and the reduction of methanogenic bacteria. Consequently, acidic SS (ASS) indicates problems in the process (Kus and Wiesmann, 1995; van Lier et al., 2008). Thus, when methanogenic capacity is continuously overloaded, volatile fatty acids can cause acidification of the treatment unit, generating an anaerobic sewage sludge which is typically acidic, posing an additional problem for its use in agriculture. To the best of our knowledge, research results considering the effects of such acidic sludge on soil attributes and plant growth have not been documented, nor has an appropriate way to overcome this limitation been disclosed. Considering the current Brazilian legislation, the pH status of SS for agronomic purposes has been only appropriately considered when SS is treated by lime stabilization.

The present work therefore aims to evaluate the consequences of increasing rates of ASS generated in anaerobic treatment plants on soil attributes and the development and composition of lettuce seedlings, as well as to present a simple and fast way to neutralize ASS acidity for safe use in agriculture.

2. Material and methods

The experiment was conducted in a greenhouse at the Lowlands Experimental Station of Embrapa Temperate Climate - Capão do Leão, Rio Grande do Sul State, Brazil (31° 49′ 13″ S and 52° 27′ 50″ W).

The ASS was obtained from a UASB (Upflow Anaerobic Sludge Blanket) reactor, from the Sewage Treatment Station of Passo Fundo, Rio Grande do Sul State, Brazil. The treatment plant capacity is 54 L s^{-1} and serves about 13,448 inhabitants. The sewage is essentially of urban domestic origin, although with some contribution of soil sediments, but without any industrial contribution. One ASS batch (5 Mg, dry basis) was collected from drying beds (50% moisture, 90 days after reactor discharge), then submitted to the solarization process, which consisted of 0.1 m ASS layers distributed in fiber boxes inside an agricultural greenhouse covered by transparent plastic (200 μm). To ensure pathogen reduction, the solarization process lasted 60 days in the summer. The internal agricultural greenhouse temperature and external solar radiation were monitored throughout the solarization period (Fig. 1). Subsequently, the ASS was crushed and sieved in order to reduce and standardize the particle size to <0.84 mm. The ASS was evaluated for agronomic attributes as well as for pathogenic, inorganic and organic contaminants (Table 1).

Treatments consisted of ASS addition rates (0.0, 0.25, 0.5, 1, 2, 4 and 8 g kg^{-1} in dry basis), in addition to a conventional fertilization treatment with 0.088 g kg^{-1} of N (urea), 0.070 g kg^{-1} of P_2O_5 (triple superphosphate) and 0.080 g kg^{-1} of K_2O (potassium chloride).

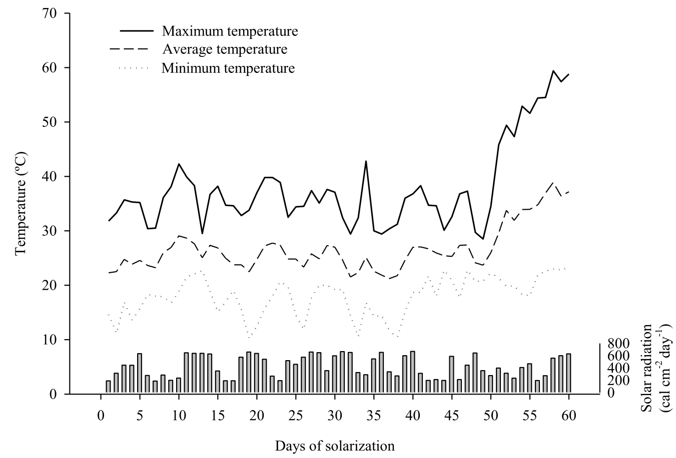

Fig. 1. Maximum, minimum and average internal temperatures (°C) of the greenhouse covered by translucent plastic, and external solar radiation (cal cm^{-2} day^{-1}) during 60 days (from Nov-11 to Jan-01) of anaerobic sewage sludge solarization.

The treatments were applied and homogenized in pots with 2.5 kg of an Albaqualf (IUSS, 2014). Seedlings of lettuce cv. Ceres (Tecnoseed®) were transplanted 15 days after sowing (winter of 2016), conducted under controlled conditions (air temperature and irrigations according to crop requirements).

Lettuce plants were harvested 42 days after their transplant in order to evaluate the dry mass (g $plant^{-1}$) of the aerial portion, determined in four plants of each plot. These were packed in paper bags and placed into a stove with artificial hot air circulation (65 °C). After weighing to obtain the dry mass, a representative aliquot of dry tissue was taken to determine the concentration of nitrogen - N (%), phosphorus - P (%), potassium - K (%), calcium - Ca (%) and magnesium - Mg (%), sulfur - S (%), iron - Fe (mg kg^{-1}) and copper - Cu (mg kg^{-1}) in the leaves. After harvesting the plants, soil samples were taken to determine the pH, soil organic matter – SOM (%) and cation exchange capacity - CEC ($cmol_c$ dm^{-3}), as well as the exchangeable aluminum content - Al^{3+} ($cmol_c$ dm^{-3}) by titration, concentrations of extractable P (mg dm^{-3}) and K ($cmol_c$ dm^{-3}) through Mehlich-1, available Ca ($cmol_c$ dm^{-3}) and Mg ($cmol_c$ dm^{-3}) by titration, the total content of S (mg dm^{-3}) determined through total combustion in CHN–S equipment, while B, Cu, Fe, Zn and Mn (all micronutrients in mg dm^{-3}) were obtained by atomic absorption spectrometry after acid digestion (Teixeira et al., 2017).

The experiment was performed in a randomized block design with eight treatments and four replicates, each replicate consisting of four plants. The results of each response variable were analyzed with respect to the presence of outliers, and if it followed a normal distribution, submitted to analysis of variance – ANOVA (Test F, 5% probability of error). The variables with significant effects were submitted to polynomial regression analysis.

From the results obtained in this experiment, an incubation test was performed in order to increase the pH of ASS with acidic pH. The incubation test was carried out using increasing rates (0.0, 0.5, 1.0, 2.0, 4.0 and 8% of ASS dry mass) of dolomitic limestone (commercial product, 25% CaO, 14% MgO, neutralization power of 80% and all particles with a size less than 0.3 mm), at room temperature. The sewage sludge belonged to the same batch tested in the previous experiment. The limestone was mixed to 400 g of ASS, then homogenized and incubated in 1.0-liter flasks with the moisture content maintained at 80% of the water retention capacity at 10 kPa (WRC = 40.55%).

Measurements of pH using 10 mL of the sample (sludge + liming material, dry weight) and 50 mL of distilled water (1:5) were performed at 7, 14, 28, and 56 days after incubation commenced.

Table 1
Chemical and physical characteristics of anaerobic sewage sludge (ASS) after solarization, maximum contaminant values and minimum guarantees for soil conditioners.

Parameter	Concentration in the ASS	LOQ	Maximum values allowed in SS[a]	Maximum values allowed in soil amendments[b]
Agronomic parameters				
pH	3.80			
Cation exchange capacity - CEC (mmol kg $^{-1}$)	271.00			
Organic carbon content (%)	25.00			
Total nitrogen (%)	3.00			
Total phosphorus (%)	0.60			
Total potassium (%)	0.06			
Total calcium (%)	1.20			
Total magnesium (%)	0.37			
Total sulfur (%)	2.50			
Total iron (%)	3.40			
Total manganese (mg kg^{-1})	377.00			
Total sodium (mg kg^{-1})	423.00			
Total aluminum (%)	5.80			
Density (kg m^{-3})	831.00			
Water retention capacity at 10 kPa - WRC (%)	40.55			
Pathogenic contaminants				
Thermotolerant coliforms (MPN/g of total solids)	45.00	1.1	<1000	<1000
Viable eggs of helminths (eggs/g of total solids)	<0.25	<0.25	<0.25	<0.25
Salmonella (MPN/10g of total solid)	Absent	–	Absent	Absent
Enteric virus (PFU/g of total solids)	Absent	–	Absent	Absent
Inorganic contaminants (mg kg^{-1})				
Arsenic	<0.50	0.5	41.00	20.00
Barium	48.00	6.0	1300.00	
Cadmium	<0.03	0.03	39.00	3.00
Lead	3.80	0.4	300.00	150.00
Copper	6.20	0.2	1500.00	
Chromium	1.10	1.0	1000.00	500.00
Mercury	<0.02	0.02	17.00	1.00
Nickel	<1.00	1.0	420.00	70.00
Selenium	<0.20	0.2	100.00	80.00
Zinc	76.00	2.0	2800.00	
Organic contaminants (mg kg^{1})				
Chlorinated Benzenes				
1,2-Dichlorobenzene	<0.005	0.005	0.73	
1,3- Dichlorobenzene	<0.005	0.005	0.39	
1,4- Dichlorobenzene	<0.005	0.005	0.39	
1,2,3-Trichlorobenzene	<0.005	0.005	0.01	
1,2,4-Trichlorobenzene	<0.005	0.005	0.011	
1,3,5-Trichlorobenzene	<0.005	0.005	0.50	
1,2,3,4-Tetraclorobenzeno	<0.005	0.005	0.16	
1,2,4,5-Tetrachlorobenzene	<0.005	0.005	0.01	
1,2,3,5-Tetrachlorobenzene	<0.005	0.005	0.0065	
Phthalate esters				
Di-n-ButylPhthalate	<0.10	0.10	0.70	
DietilexilFtalato (DEHP)	<0.05	0.05	1.0	
DimethylPhthalate	<0.10	0.10	0.25	
Non-chlorinated phenols				
Total Cresols	<0.01	0.01	0.16	
Chlorinated phenols				
2,4-Dichlorophenol	<0.05	0.05	0.031	
2,4,6-Trichlorophenol	<0.10	0.10	2.40	
Pentachlorophenol	<0.10	0.10	0.16	
Polycyclic aromatic hydrocarbons				
Benzo (a) anthracene	<0.005	0.005	0.025	
Benzo (a) pyrene	<0.005	0.005	0.052	
Benzo [k] Fluoranthene	<0.005	0.005	0.38	
Indene (1,2,3-cd) pyrene	<0.005	0.005	0.031	
Naphthalene	<0.005	0.005	0.12	
Phenanthrene	<0.005	0.005	3.30	
Lindane	<0.0005	0.0005	0.001	

MPN: most probable number; TS: total solids; PFU: plaque-forming unit; LOQ: limit of quantification.
[a] Resolution 375 of 2006 (BRAZIL, 2006).
[b] Normative Instruction 7 of 2016 (BRAZIL, 2016).

This experimental design involved a randomized block with four replicates, each repetition consisting of a flask containing the mixture. The results were submitted to ANOVA and regression analysis.

3. Results and discussion

3.1. Physicochemical characteristics of acid anaerobic sewage sludge

The anaerobic sewage sludge (ASS) showed highly desirable agronomic characteristics, such as low density and high levels of organic carbon, N, Ca, S, and micronutrients, besides expressive CEC and WRC, similar to soil conditioners and organic fertilizers (Table 1).

The evaluation of ASS indicated similar values to those found in literature for organic carbon (21–32%), nitrogen (1.09–4.2%), phosphorus (0.56–4.2%), calcium (3.42–3.82%), magnesium (0.14–1.93%), sulfur (0.89–2.68%) and manganese (120–400 mg kg^{-1}), however, the pH (5.4–7.9) and potassium content (0.2–1.5%) were lower than those previously found in the literature, while values for aluminum (0.76–1.64%) and iron (1.03–2.60%) were larger (Bueno et al., 2011; Stemann et al., 2015; Pires et al., 2015; Pesonen et al., 2016; Ren et al., 2015; Herzel et al., 2016; Kodešová et al., 2019). These differences were probably due to particularities in the anaerobic digestion process. This high aluminum total content might be connected to the uncommon pH conditions during the ASS process. Thus, it is possible that some contributions of soil sediments into the sewage collector system may affect ASS aluminum content. Soils from the region are highly weathered and are well known as being rich in aluminum oxides (19–27% of Al_2O_3) (Bortoluzzi et al., 2015).

In relation to inorganic and organic contaminants, results were also well below the maximum limits stated by legislation (Table 1), in accordance with those expected for sewage treatment plants without industrial contributions. Most of the ASS inorganic contaminants were found at concentrations below those presented in the literature, such as barium (235–460 mg kg^{-1}), cadmium (0.3–13.5 mg kg^{-1}), lead (28.6–283 mg kg^{-1}), copper (204–1735 mg kg^{-1}), nickel (17.7–189 mg kg^{-1}) and zinc (300–2110 mg kg^{-1}), while arsenic (0.01–12 mg kg^{-1}), selenium (<0.01), chromium (<0.01–200 mg kg^{-1}) and mercury (<0.01–0.4 mg kg^{-1}) were found in similar concentrations as those previously documented (Bueno et al., 2011; Stemann et al., 2015; Ren et al., 2015; Herzel et al., 2016; Bravo-Martín-Consuegra et al., 2016; Pesonen et al., 2016).

Brazilian legislation (BRAZIL, 2006) limits the amount of SS to be applied in soils by taking into consideration the content of inorganic contaminants. The amount of ASS (used in this study), which could be applied in agricultural areas until this limit is reached can be found in Table 2. From this exercise, if one considers the most restrictive element (i.e. barium), it would be possible to apply up to 5521 ton ha^{-1} of ASS before reaching the accepted limits. Furthermore, for an annual application rate of 16 ton ha^{-1} or 8 g kg^{-1} (the highest rate used in the present study), the same area could receive annual applications of ASS for up to 345 years. These indicators suggest that the present ASS is safe for agricultural use, in terms of inorganic contaminants.

For organic contaminants, none of the evaluated compounds were found to be above the detection limits of the analytical methods. According to Smith (2009), the organic contaminants in SS present minimal risk since most toxic compounds are influenced by a variety of attenuation mechanisms that prevent the transfer to crop tissues, as rapid volatilization and biodegradation, besides minimal or no persistence or strong adsorption.

3.2. Effects of anaerobic sewage sludge on soil chemical attributes

ASS significantly influenced soil chemical characteristics (Fig. 2). In relation to pH, a linear reduction was observed with increasing rates of ASS (Fig. 2a).

According to the classes indicated by CQFS-RS/SC (2016), the pH fell from the "medium" class in the control treatment to "very low" at 4 and 8 g kg^{-1} rates (Fig. 2a). This negative linear relationship demonstrates how the pH of ASS (3.8) strongly affects soil pH. The treatment with NPK also reduced soil pH, dropping from "medium" to "low", comparable to ASS rates of 0.5, 1 and 2 g kg^{-1}.

The low pH of ASS can be related to abnormalities in the digestion process usually subdivided into four phases: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. According to van Lier et al. (2008), hydrolysis is a step where enzymes convert complex and undissolved material into less complex dissolved compounds; in acidogenesis, the dissolved compounds are converted into various compounds that are volatile fatty acids, alcohols, lactic acid, CO_2, H_2, NH_3 and H_2S, as well as new cellular material; in acetogenesis, the products of digestion are converted to acetate, H_2 and CO_2, as well as into new cellular material; and finally, in methanogenesis, acetate, hydrogen plus carbonate, methanol are converted to methane, CO_2 and new cellular material. That being said, when the reactors are overloaded or disturbed in acidogenesis, a sudden drop in pH occurs and when the alkalinity is consumed, pH begins to fall, resulting in a higher concentration of undissociated volatile fatty acids, leading to a more severe inhibition of methanogens and obviously to an even faster increase of volatile fatty acids and a subsequent drop in pH (Kus and Wiesmann, 1995; van Lier et al., 2008).

The ASS effect on soil pH suggests that this material requires a pH correction process before being applied, since pH impacts major soil chemical and biological processes and the overall nutrient availability.

The variables CEC and SOM presented a linear increase in response to ASS rates, with increments of 48 and 23%, respectively (Fig. 2). A significant increase was observed on soil CEC, but the CEC class remained "low" (Fig. 2b). In the case of SOM content, although there was an increase, the contents remained at "low" class according to CQFS-RS/SC (2016) (Fig. 2c).

The Al^{3+} had a 17-fold linear increase from control treatment to the highest rate of 8 g kg^{-1} (Fig. 2d). This result is closely related to the high Al total content in the ASS (5.8%). In addition, the increase in available Al^{3+} content may be caused by the reduction of soil pH, as strongly supported by the negative correlation between these variables ($r = -0.76$, $P < 0.0001$). This relationship is of major relevance and is usually not taken into account during SS soil disposal or even when SS is used for agricultural purposes. The presence of exchangeable Al^{3+} is widely known to reduce root development and nutrient uptake by plants (Jones and Ryan, 2017). The treatment with NPK also showed a higher

Table 2
Estimated maximum load of anaerobic sewage sludge (ASS) that could be applied to agricultural soils, considering the inorganic contaminant levels determined in the sludge.

Inorganic contaminants	Concentration in the ASS (kg ton^{-1})	Maximum permitted quantity of inorganic contaminants in the soil by SS application (kg ha^{-1})[a]	Amount of ASS required to reach maximum permitted concentration of inorganic contaminants permitted (ton ha^{-1})	Number of years required to reach maximum quantity of inorganic contaminants in the soil by SS application, with a rate of 16 ton ha^{-1} or 8g kg^{-1} of ASS per year.
Arsenic	0.0005	30	60,000	3750
Barium	0.048	265	5521	345
Cadmium	0.00003	4	133,333	8333
Lead	0.0038	41	10,789	674
Copper	0.0062	137	22,097	1381
Chromium	0.0011	154	140,000	8750
Mercury	0.00002	1.2	60,000	3750
Nickel	0.001	74	74,000	4625
Selenium	0.0002	13	65,000	4063
Zinc	0.076	445	5855	366

[a] Brazilian Resolution CONAMA N° 375 of 2006 (BRAZIL, 2006).

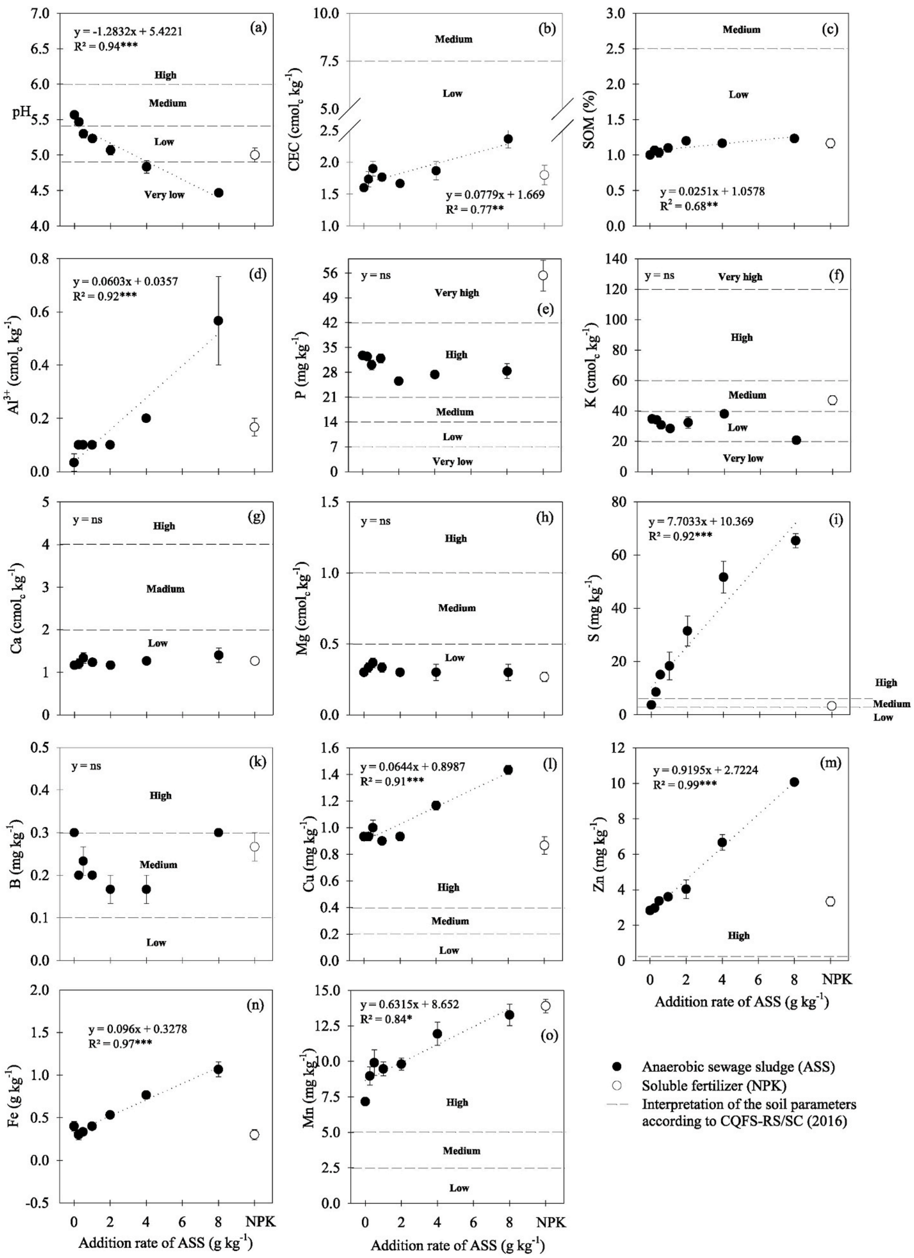

Fig. 2. Polynomial regressions of pH, cation exchange capacity (CEC), soil organic matter (SOM), exchangeable aluminum (Al^{3+}), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sulfur (S), copper (Cu) zinc (Zn), iron (Fe) and manganese (Mn) in an Albaqualf in response to anaerobic sewage sludge (ASS) and additional treatment (reference) with soluble fertilizer composed by nitrogen, phosphorus, and potassium (NPK). *, **, ***, significant at $p < 0.01$, $p < 0.001$ and $p < 0.0001$, respectively.

content of Al^{3+} in relation to the control (Fig. 2d), probably since fertilization with NPK may also cause a reduction to soil pH. This is mainly due to the used source of P (triple superphosphate), which is essentially acid, associated with the acidifying effect of urea, caused either by nitrification of the NH_4^+ ions formed by its hydrolysis or by the leaching of NO_3^-, which is dragged from the system, increasing the concentration of H^+ ions (Helyar, 1976).

There was observed no effect of ASS rates on the P, K, Ca and Mg concentrations in the soil (Fig. 2). In relation to P, NPK provided higher concentrations than ASS, raising the soil content from the “high” to “very high” class (Fig. 2e). The absence of the ASS effect was mainly due to its low total P content (0.60%) (Table 1). Additionally, the sludge was probably forced to precipitate P during sewage treatment and the remaining P was in organic form, therefore microbial mineralization had little effect on phosphorus availability.

The NPK also provided a K increase in the soil, superior to ASS treatments, raising soil content from “low” to “medium” levels (Fig. 2f). Despite having a high K releasing rate, the lack of ASS influence occurs by its very low natural K concentration (0.06%) (Table 1). Thus, when ASS is considered for agricultural purposes, a K supplement must be applied in order to fully provide this nutrient to the crops.

The lettuce crop cycle was probably too short for ASS to demonstrate the significant release of Ca and Mg in the soil solution and exchangeable complex (Fig. 2g and h). Especially for Ca, the availability seems to be low since an amount of 0.096 g kg^{-1} of Ca would be expected to be provided by the highest applied dose (8 g kg^{-1}) considering the ASS content (1.2%), yet no differences were observed in the soil. For both Ca and Mg, soil levels were interpreted as “low” (CQFS-RS/SC, 2016). These results address the necessity of a highly available source of Ca and Mg to be supplemented in ASS compositions in order to accomplish fast growth crop requirements.

The absence of the ASS rate effect on K, Ca and Mg soil levels suggests that the increase in CEC is essentially a reflex of the increase in the availability of Al^{3+}, as supported by the positive correlation between Al^{3+} and CEC ($r = 0.72$, $P = 0.0002$). Higher soil CEC is desirable, but not when Al^{3+} plays a major role in this increment, since this element is generally toxic to plants. This result strongly indicates the need for liming additives in ASS to decrease the availability of Al^{3+}.

Soil treated with ASS presented a linear increase of S total content (Fig. 2i). Even from the addition rate of 0.25 g kg^{-1}, S had already reached levels considered "high" according to regional recommendations. With NPK application, S was classified as "medium", similar to the control (Fig. 2i) and therefore did not change significantly.

Concentrations of evaluated micronutrients linearly increased with the ASS rates (Fig. 2). The concentrations of Cu, Zn and Mn (Fig. 2), besides increasing linearly in response to ASS rates, were classified as "high", similar to the NPK treatment (CQFS-RS/SC, 2016). On the other hand, although Fe presented a linear increase with ASS rates, levels were always lower than the "high" class (CQFS-RS/SC, 2004) (Fig. 2n). In regards to Mn, soil content with NPK was similar to the rate of 8 g kg^{-1} of ASS (Fig. 2o).

The expressive concentrations in ASS, as well as the reduction of soil pH induced by high sludge rates probably promoted the greater availability of these elements. When these elements are in very high soil concentrations, plants may show symptoms of phytotoxicity. This fact indicates the need for ASS or soil pH correction to bring the values closer to neutrality. The availability of heavy metals is relatively low at pH values higher to 6.0 (Kabata-Pendias and Pendias, 1987), which is the

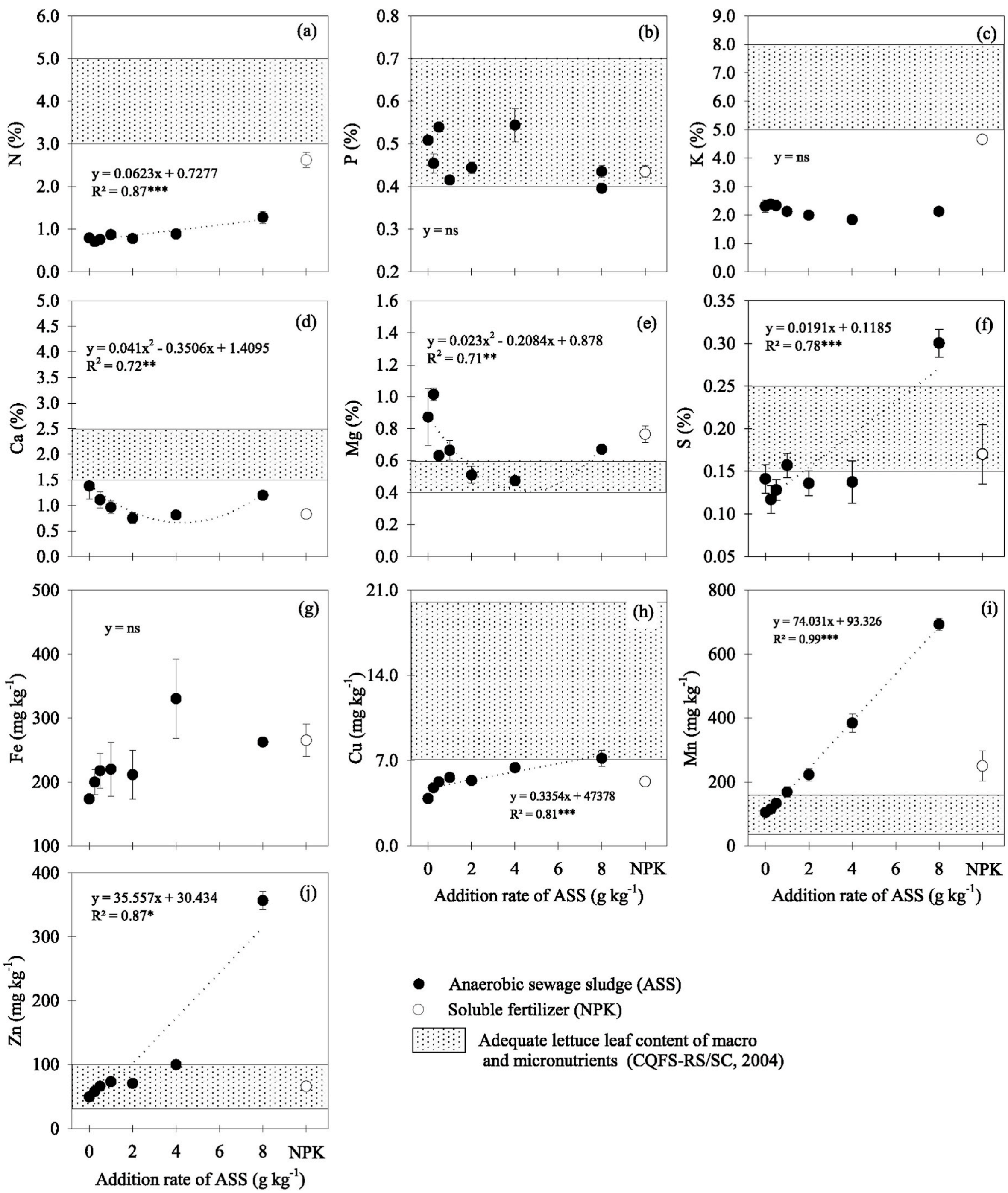

Fig. 3. The concentration of nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mn), sulfur (S), iron (Fe), copper (Cu), manganese and zinc (Zn) in lettuce leaves, in response to solarized anaerobic sewage sludge (ASS) and additional treatment (reference) with soluble fertilizer composed by nitrogen, phosphorus, and potassium (NPK). *, **, ***, significant at $p < 0.01$, $p < 0.001$ and $p < 0.0001$, respectively.

appropriate pH for most cultures.

3.3. Effects of anaerobic sewage sludge on lettuce macro and micronutrients

Analysis of leaf nutrient content indicated a significant effect of sludge treatments (Fig. 3). Leaf N content linearly increased in response to ASS rates (Fig. 3a), although was consistently within the 'low' level, even at the highest dose. Even with the highest ASS rate (8 g kg^{-1}), adding 480 kg ha^{-1} N, the foliar N content in NPK treatment was twice as high as those that received ASS rates (Fig. 3a). It was also observed that the foliar levels of N were lower than those considered "normal" for lettuce tissues (3.0–5.0%, CQFS-RS/SC, 2004), even with the highest rate of ASS. These results indicate that only a small part of the N present in ASS was released during the experiment, agreeing with previous findings stated in CQFS-RS/SC (2016), which suggest a 20% efficiency of sewage sludge (SS) for the supply of available N for the first crop cycle.

No significant effects of ASS rates were observed in regards to P and K content (Fig. 3). In the case of P, all treatments presented leaf content considered normal for the crop (0.4–0.7%, CQFS-RS/SC, 2004), possibly since soil concentrations were high regardless of the treatment (Fig. 3b). In relation to K, the NPK treatment presented concentrations twice as high as the treatments with ASS (Fig. 3c). However, all treatments showed K levels lower than that considered suitable for the crop (5–8%, CQFS-RS/SC, 2004).

Foliar concentrations of Ca and Mg followed a quadratic response to ASS rates, with a reduction of their content up to the rate of 4 g kg^{-1}, with a subsequent increase up to the highest rate (Fig. 3d and e). The reduction of Ca and Mg until 4 g kg^{-1} can be explained by the decrease in pH imposed by the increasing rates of ASS, while the large S-supply in the rate of 8 g kg^{-1} may provide increased Ca and Mg concentrations in plants (Klikocka and Gáowacka, 2013; Moda et al., 2013).

In general, Ca leaf content was lower than that considered adequate for lettuce (1.5–2.5%, CQFS-RS/SC, 2004) (Fig. 3d). For Mg, the ASS addition rates of 2 and 4 g kg^{-1} provided adequate levels whereas in the other treatments, the concentrations were higher than the adequate range (0.4–0.6%; CQFS-RS/SC, 2004) (Fig. 3e).

Although S content in the soil presented a strong linear and rate-dependent increase, in lettuce leaves the S content was clearly dependent of the highest dose of 8 g kg^{-1} (Fig. 3f). In this case, the content was higher than that considered adequate for the crop (0.15–0.25%, CQFS-RS/SC, 2004). This result was probably due to the high content of S in ASS (2.5%), corresponding to the incorporation of 400 kg ha^{-1} in the highest rate.

Contrary to what was observed in the soil, ASS rates did not influence Fe leaf contents, which were similar to the control treatment (Fig. 3g). Higher Fe content was observed in the 4 and 8 g kg^{-1} rates of ASS and should be associated with the large amount of this element in ASS (3.4%).

Conversely, tissue concentrations of Cu, Mn and Zn increased linearly in response to ASS rates (Fig. 3). Cu reached the appropriate concentration (7–20 mg kg^{-1}; CQFS-RS/SC, 2004) only at the rate of 8 g kg^{-1} (Fig. 3h), while Mn was observed in adequate concentrations (30–150 mg kg^{-1}; CQFS-RS/SC, 2004) up to the rate of 1 g kg^{-1}, after which the contents were higher than recommended (Fig. 3i). In relation to Zn, except for the treatment with 8 g kg^{-1} of ASS, with concentrations three times higher than that considered suitable (30–100 mg kg^{-1}, CQFS-RS/SC, 2004), all other rates had adequate concentrations (Fig. 3j).

The increase of Cu, Mn and Zn in lettuce leaves was mainly due to ASS composition, as well as due to the acidic soil pH provided by the incorporation of ASS (Table 1). In this sense, there were significant linear correlations between the decrease of soil pH and the increase of leaf contents of Cu ($r = -0.89$, $P < 0.0001$), Mn ($r = -0.92$, $P < 0.0001$) and Zn ($r = -0.82$, $P < 0.0001$).

3.4. Effects of anaerobic sewage sludge on lettuce growth

Changes in the chemical composition of soil and leaves, provided by treatments, reflected significant changes in chlorophyll content and plant growth (Fig. 4).

The chlorophyll index increased linearly as a function of ASS rates (13% at 8 g kg^{-1} compared to control, Fig. 4a). Even so, ASS was lower than NPK, which increased by 48% compared to the treatment without the addition of ASS (Fig. 4a).

Chlorophyll content at the end of the vegetative phase has been closely related to N foliar concentration in many crops (Argenta et al., 2001), since 50–70% of the N content in leaves is part of the enzymes associated with chloroplasts (Chapman and Barreto, 1997). However, in the present study, leaf N content from ASS treatments did not correlate with the chlorophyll index ($r = 0.38$; $P = 0.09$), probably due to the non-pronounced effect of ASS on N leaf content. Although significant, it barely raised N levels, and was lower than the range considered adequate.

On the other hand, the micronutrients Cu ($r = 0.65$, $P = 0.0015$), Mn ($r = 0.76$, $P = 0.00008$) and Zn ($r = 0.77$, $P = 0.00005$) presented a positive correlation with the chlorophyll index, agreeing with Zabini et al. (2007), who observed an analogous correlation when studying coffee leaves. In the same sense, Santos et al. (2013), studying the omission of micronutrients in *Jatropha curcas*, concluded that the Cu, Mn and Zn deficiency had caused a reduction in the chlorophyll index, with negative reflections in the photosynthetic rate.

The dry mass of plants increased linearly in response to ASS rates (32% more at 8 g kg^{-1} in relation to the control, Fig. 4b). On the other hand, plants with NPK produced a 137% increase in dry mass when compared to the control (Fig. 4b), indicating that soluble fertilization was more efficient in providing nutrients for lettuce, a very fast-growing crop.

Although many soil and plant parameters had a similar response to the dry mass of plants, none showed a significant correlation except for chlorophyll index ($r = 0.48$, $P = 0.0281$), which was probably influenced by the micronutrient concentration in leaves (Cu, Mn, and Zn), as previously mentioned.

Despite all these findings, the growth performance of lettuce plants was determined by more complex interactions than only the chemical characteristics of ASS analyzed in the present study. Some studies indicate important conditioning effects on soil physical characteristics caused by sewage sludge (Campos and Alves, 2008; Hamdi et al., 2019). According to De Maria et al. (2010), applications of sewage sludge as a soil conditioner decrease bulk density and microporosity and increase macroporosity. The same authors observed a recovery of soil quality in comparison to soils with mineral fertilizers, particularly in degraded areas, besides an increase in the dry mass of *Brachiaria decumbens* and *Eucalyptus citriodora*. The results obtained by these authors reinforce the hypothesis that the increase of the dry mass of lettuce plants may not only be related to the beneficial effect of sewage sludge with respect to the chemical composition, but also to the soil physical characteristics.

The highest dry mass with NPK occurred as expected, and served as a reference to compare with the performance of ASS. In the case of NPK, the dry mass of lettuce was mainly affected by available N and K levels in the soil (Fig. 2c and f), easily translocated to plant (Fig. 3a and c), since these were the elements present in higher concentrations in lettuce tissues (Kapoulas et al., 2017).

The results of ASS in this study indicate improvements of several soil parameters, such as CEC, SOM content and micronutrient concentration, as well as the unmeasured positive effects on soil physical characteristics, resulting in benefits for leaf composition and dry mass yield.

On the other hand, ASS without pH adjustment has limited conditioning and fertilizing potential. Low pH clearly increased the free Al^{3+} content, restricted the availability of some nutrients, as well as did not provide sufficient amounts of major macronutrients (N, K, Ca, and Mg).

Thus, the pH correction of ASS can be an improvement for

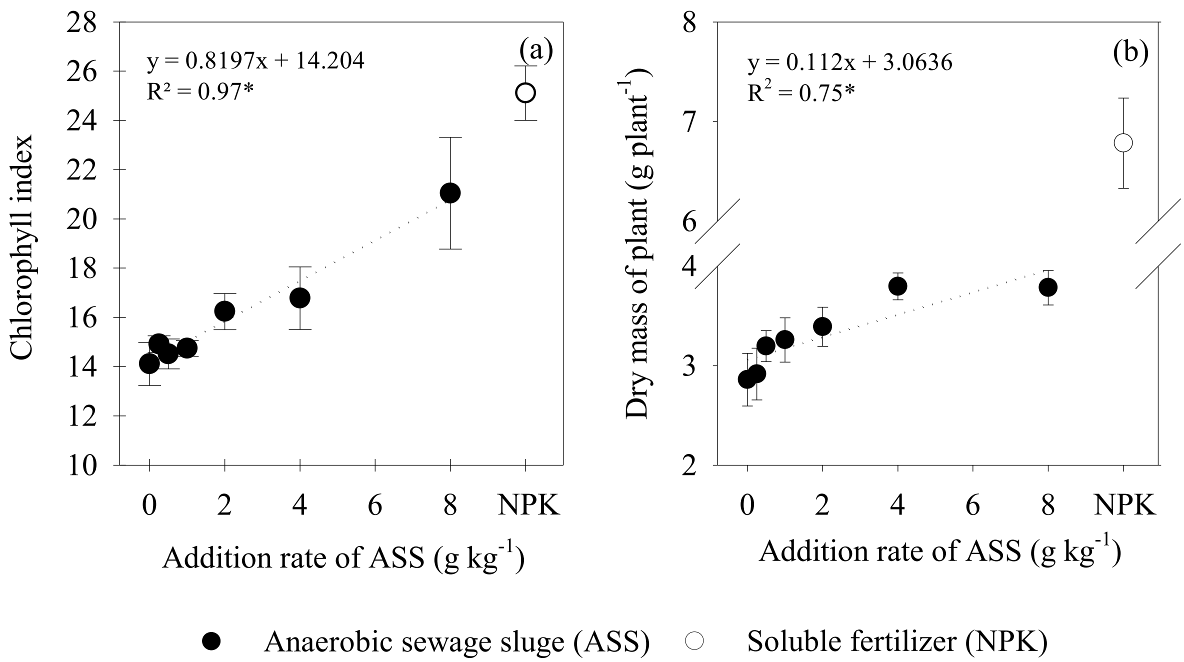

Fig. 4. Chlorophyll index and dry mass of lettuce leaves (g plant^{-1}) in response to anaerobic sewage sludge (ASS) and additional treatment (reference) with soluble fertilizer composed by nitrogen, phosphorus, and potassium (NPK). *, **, ***, significant at $p < 0.01$, $p < 0.001$ and $p < 0.0001$, respectively.

agricultural use, besides providing greater amounts of Ca and Mg. Therefore, one empirical study with a liming material was performed in order to determine the most appropriate corrective rate for ASS as follows.

3.5. *Correction of anaerobic sewage sludge with acidic pH for agricultural use*

A significant effect of limestone on ASS pH was observed from 7 days of incubation (Fig. 5a). However, the ASS pH reached 6.0 only after 28 days using the highest limestone rate (Fig. 5c and d). These results indicate that the ASS/liming mixture must be rested at least 28 days to ensure pH correction for agricultural purposes.

Current Brazilian law allows the hygienisation of anaerobic sewage sludge by lime stabilization, in which the sludge pH is raised up to 12 using large amounts of alkaline material, generally hydrated lime (~50% dry mass), and aims to reduce the load of pathogens to suitable levels (Bina et al., 2004). In the end, the sewage sludge becomes highly alkaline and, when applied to agricultural soil, acts essentially as a liming material and organic matter supplier, and is therefore mostly indicated for acidic soils. However, lime stabilization reduces SS fertilizer potential mainly since most of the nitrogen content is lost during the process. Nutrient imbalance and alkaline sites may also form inside the soil since hydrated lime has a strong neutralizing power.

However, the pH correction of ASS up to 6.0 with dolomitic limestone, as carried out in the present essay, provided a gentle and cheap correction of sewage sludge pH, making it more suitable for agricultural purposes. In this case, the sewage sludge should not present limitations related to pathogens, as obtained after the solarization process used in this work. The pH correction of ASS with acidic pH can preserve important agronomic properties, such as N, P, S and micronutrient contents, or even increase the supply of Ca and Mg. Significant change is expected in the nature of CEC of ASS with acidic pH (CEC = $Ca^{2+} + Mg^{+} + K^{+} + Na^{+} + Al^{3+}$). As observed in this study, the high Al content in ASS and its low pH contributed to a significant proportion of soil CEC that was increased by the Al^{3+} supplied by ASS. However, when the pH of ASS is corrected with dolomitic limestone (39% of CaO + MgO), it is likely that the increase in CEC will be due to the increase in available Ca and Mg and not by Al^{3+}, which tends to be unavailable at a pH of 6.0. This correction will also help avoid increasing Cu, Zn, Fe and Mn concentrations to phytotoxic levels for major crops. These effects can be evaluated in future studies.

After 56 days of incubation, when the correction reaction was considered stable, Table 3 was developed, aiming to indicate the right dolomitic limestone rate necessary for pH correction of acidic ASS. Note that the amount of liming material required varies according to the quadratic shape model (Fig. 5d), from 0.13 to 5.38% (mass/mass), when the initial pH of the ASS was 5.9 and 3.8, respectively. A pH of 6.0 was considered ideal as it is the recommended value for most agricultural species, including lettuce.

4. Conclusion

The application of sewage sludge in agriculture is a matter of huge debate around the world. This study presents relevant information that can help to improve the sustainable use of SS in agriculture.

The adoption of simple and inexpensive technology such as solarization, effectively reduces the volume of sludge that needs to be recycled while preserving its main agronomic characteristics. By doing so, this additional step for the treatment of ASS might also contribute towards reducing levels of pathogenic contaminants.

The ASS applied in this study acted as a soil conditioner/organic fertilizer. Increasing soil organic matter concentrations and macro and micronutrient content had a significant impact on lettuce plant nutrition, mainly in leaf concentration of nitrogen, sulfur, zinc, iron, and manganese, reflected in a larger dry mass of plants.

In several cases, the agronomic potential of ASS is strongly limited by its acidic pH, due to failures in the acidogenesis phase of the anaerobic digestion process. Specific management of ASS is essential for proper recycling of this waste, such as pH correction of ASS using dolomitic limestone. The pH correction of ASS proved to be an efficient alternative way to overcome major deficiencies, as it employs relatively small quantities (0.13–5.38%) of an inexpensive input that is already commonly utilized in agriculture worldwide.

Finally, from this study it can be concluded that the ASS management process based on solarization, pH correction and agricultural use is

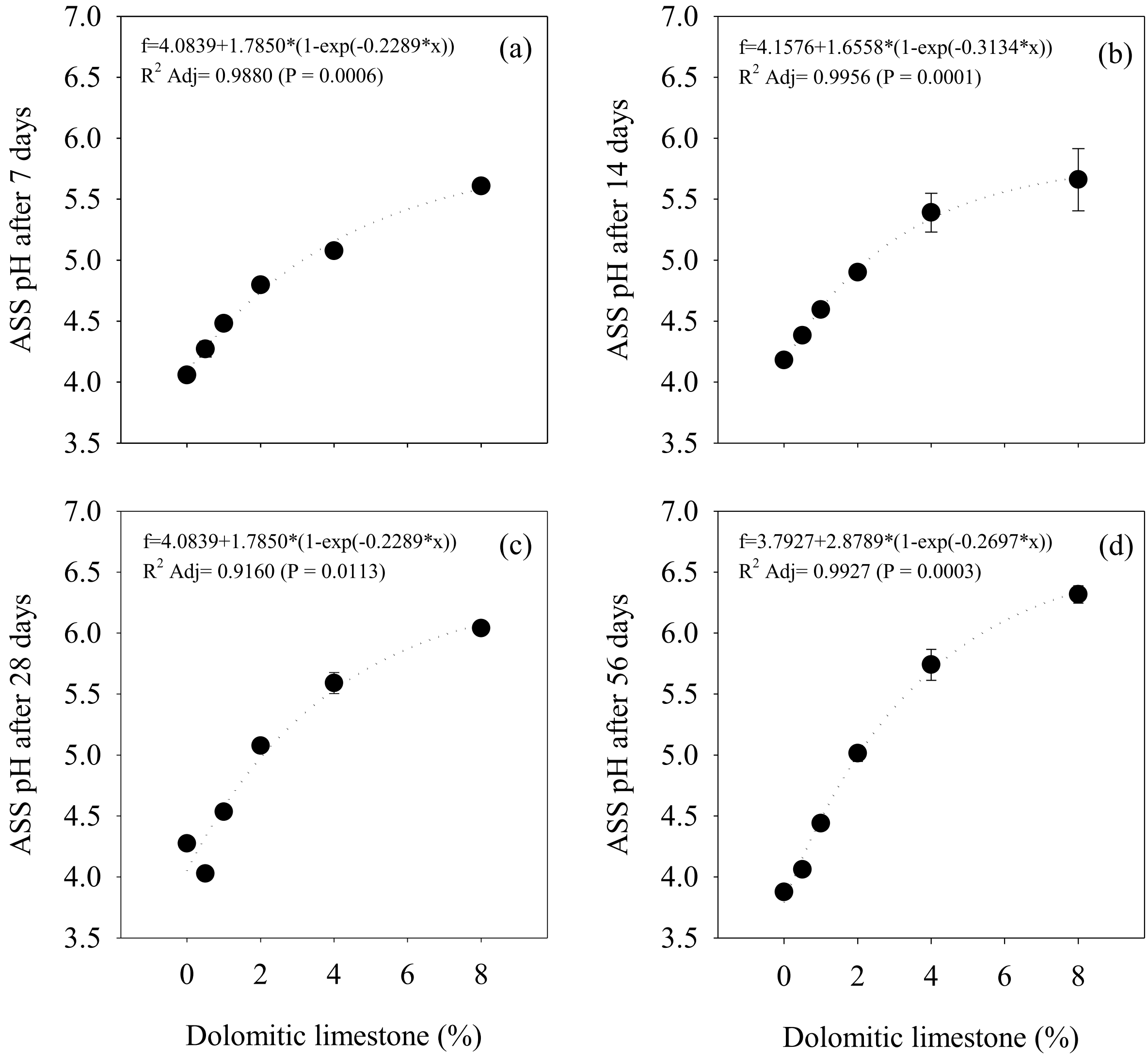

Fig. 5. Values of pH of anaerobic sewage sludge (ASS), after 7, 14, 28 and 56 days of incubation with increasing rates of dolomitic limestone.

Table 3
Recommended rate of dolomitic limestone for pH correction of anaerobic sewage sludge up to a desired pH = 6.0, based on the initial pH of the sludge.

Initial pH of ASS	Recommended rate of dolomitic limestone (%)	Initial pH of ASS	Recommended rate of dolomitic limestone (%)
3.8	**5.38**	5.0	**1.58**
3.9	**4.85**	5.1	**1.39**
4.0	**4.40**	5.2	**1.20**
4.1	**4.00**	5.3	**1.03**
4.2	**3.65**	5.4	**0.86**
4.3	**3.30**	5.5	**0.71**
4.4	**3.01**	5.6	**0.55**
4.5	**2.73**	5.7	**0.41**
4.6	**2.48**	5.8	**0.27**
4.7	**2.23**	5.9	**0.13**
4.8	**2.00**	6.0	–
4.9	**1.78**		

technically viable, relatively easy and inexpensive to perform, and can be replicated in various regions of the world, especially in developing countries.

CRediT authorship contribution statement

Ivan dos Santos Pereira: Project administration, Conceptualization, Methodology, Visualization, Writing - review & editing, Writing - original draft. **Adilson Luís Bamberg:** Writing - original draft, Project administration, Funding acquisition, Conceptualization, Writing - review & editing. **Rogério Oliveira de Sousa:** Conceptualization, Formal analysis, Visualization, Resources, Writing - review & editing. **Alex Becker Monteiro:** Formal analysis, Visualization, Investigation. **Rosane Martinazzo:** Formal analysis, Project administration, Investigation, Writing - review & editing. **Carlos Augusto Posser Silveira:** Project administration, Formal analysis, Investigation, Writing - review & editing. **Andressa de Oliveira Silveira:** Methodology, Visualization, Writing - review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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