

Article

One Year Residual Effect of Sewage Sludge Biochar as a Soil Amendment for Maize in a Brazilian Oxisol

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Abstract: The thermochemical transformation of sewage sludge (SS) to biochar (SSB) allows exploring the advantages of SS and reduces possible environmental risks associated with its use. Recent studies have shown that SSB is nutrient-rich and may replace mineral fertilizers. However, there are still some questions to be answered about the residual effect of SSB on soil nutrient availability. In addition, most of the previous studies were conducted in pots or soil incubations. Therefore, the residual effect of SSB on soil properties in field conditions remains unclear. This study shows the results of nutrient availability and uptake as well as maize yield the third cropping of a three-year consecutive corn cropping system. The following treatments were compared: (1) control: without mineral fertilizer and biochar; (2) NPK: with mineral fertilizer; (3) SSB300: with biochar produced at 300 °C; (4) SSB300+NPK; (5) SSB500: with biochar produced at 500 °C; and (6) SSB500+NPK. The results show that SSB has one-year residual effects on soil nutrient availability and nutrient uptake by maize, especially phosphorus. Available soil P contents in plots that received SSB were around five times higher than the control and the NPK treatments. Pyrolysis temperature influenced the SSB residual effect on corn yield. One year after suspending the SSB application, SSB300 increased corn yield at the same level as the application of NPK. SSB300 stood out and promoted higher grain yield in the residual period (8524 kg ha⁻¹) than SSB500 (6886 kg ha⁻¹). Regardless of pyrolysis temperature, biochar boosted the mineral fertilizer effect resulting in higher grain yield than the exclusive application of NPK. Additional long-term studies should be focused on SSB as a slow-release phosphate fertilizer.

Keywords: oxisol; phosphorus; pyrolysis; wastewater treatment



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1. Introduction

The sewage sludge (SS) generated from wastewater treatment plants (WWTP) is enriched with nutrients and organic matter and may be useful material as a soil amendment [1,2]. However, various countries' environmental regulations across the world limit the use of SS on agricultural land, mainly as a precautionary measure due to organic and inorganic pollutants and pathogens [3]. Therefore, studies have been carried out to search for new strategies that allow the exploration of the advantages of SS, such as the presence of nutrients for plants, and to reduce possible environmental risks associated with its use [4]. One such strategy would be to transform SS into biochar (SSB), a carbon (C) rich material without pathogens [5]. Biochar is a charcoal-like material that is obtained through a process called pyrolysis, where an organic feedstock is heated in an oxygen-limited or oxygen-free environment at temperatures between 350 and 800 °C [6]. Biochar is getting the attention of both the political and scientific community due to its potential to improve soil productivity, remediate contaminated soils and mitigate climate change [7].

Biochar application improves soil quality by retaining a greater proportion of the soil nutrients flowing through the soil and limiting nutrient loading into aquatic systems [8]. Because of SSB properties, such as its porous and recalcitrant properties and available nutrients, mainly phosphorus (P), its use has received increasing attention in recent years. Applying SSB to soil has been shown to increase soil fertility indicators such as cation exchange capacity (CEC) and nutrient availability [9], nitrogen use efficiency [10], and crop yield [11]. For this reason, it is even possible to replace mineral fertilizers with SS biochar [11–13]. However, increases in soil fertility and crop yield strongly rely on multi factors such as the soil type, biochar-derived feedstock and properties, biochar application rate, mineral fertilizers, and the long and short-term evaluation of application [14]. The yield-stimulating effects of biochar are not universal but may significantly benefit agriculture in low-nutrient, acidic soils in the tropics [15]. For example, applying biochar to acidic soil significantly increased plant-P availability by a factor of 5.1, while there was no significant effect in alkaline soils [16]. Furthermore, the pyrolysis temperature range, which affects the quality and yield of the final product, need to be considered [17].

The combined application of biochar with mineral fertilizers has been a useful strategy to balance the availability of nutrients to crops [9], mainly when the feedstock used to produce the biochar is deficient in certain nutrients. The combination of biochar with *n* fertilizer increased soil P concentration [8]. Compared to other feedstocks, SSB is considered a nutrient-rich feedstock, especially in *n* and P [10,18,19]. Except for potassium (K), SSB can be used as an essential source of nutrients for both short and long-cycle crops with the cropping season of one month and five months, respectively [11,12]. However, due to the variety of the chemical forms of these nutrients in SSB [13,20], there are still some questions to be answered about the availability of these nutrients for plants and their residual effect on soil. Despite the increasing number of biochar studies, approximately two-thirds of 428 data points on biochar use, obtained by meta-analysis, were from studies lasting for up to one year [21]. Additionally, there is a lack of study on nutrients dynamics in the soil–plant system with SSB amendment; and most of the research on SSB has been done in pot experiments rather than at the field scale. Surprisingly, field experiments with SSB are scarce in tropical areas, where the effects of biochar on crop yield are likely to be more notorious [15]. Therefore, long-term field studies help clarify the residual effects of biochar on soil and plant properties [22]. Recently, Figueiredo et al. [23] demonstrated a distinct impact of SSB on soil P fractions under residual phase compared to the SSB direct application. Our previous study [11] performed at the same experimental area demonstrated SSB benefits, applied for two consecutive years, for soil properties and maize yields. In this previous work, under direct effect, SSB was capable of replacing inorganic fertilizers for maize production over two growing seasons in a tropical Oxisol. However, considering the diversity of chemical forms of nutrients and the high recalcitrance of biochars, it is crucial to understand the residual effect of SSB in a tropical Oxisol under field conditions. To the best of our knowledge, the present work is the first to explore the one-year residual effect of SSB on nutrient dynamics in typically tropical soil. The current study was based on the hypothesis that SSB has a residual effect and can replace mineral fertilizers (with NPK) for maize grain production in a weathered tropical soil even one year after cessation of its application.

2. Materials and Methods

2.1. Biochar Production

Biochars were produced from SS samples collected at the Gama wastewater treatment plant (WWTP), belonging to the Environmental Sanitation Company of the Federal District, in Brasilia, Distrito Federal, Brazil. This facility uses a tertiary treatment system in which, after wastewater treatment, nutrients such as P and N remain in the final SS mass.

The SS was air-dried (approximately 20% moisture content), passed through an 8 mm sieve, and then pyrolyzed at 300 °C and 500 °C. Pyrolysis was performed in a muffle furnace (model KK-220, Linn Elektro Therm, Eschenfelden, Germany) at a mean temperature

increase rate of $2.5\text{ }^{\circ}\text{C min}^{-1}$, totaling 120 and 200 min to reach the respective temperatures and residence time of 30 min. The samples were placed in a metal container adapted to the internal space of the furnace containing a gas and bio-oil exit system, with a mechanism to prevent oxygen flow, as well as a digital thermostat for temperature control. The samples were degassed at 300 and 500 $^{\circ}\text{C}$ for SSB300 and SSB500, respectively.

Table 1 presents the chemical and physical characteristics of the SS and SSBs used in the present study. The SSB surface areas and pore volumes were determined by N_2 adsorption isotherms at $-196.2\text{ }^{\circ}\text{C}$ in a surface area analyzer (NOVA 2200, Quantachrome, FL, USA). Total C and N contents in the SS and SSB were determined using a CHN Elemental analyzer (model PE 2400, series II CHNS/O, PerkinElmer, Norwalk, CT, USA). The pH was determined in a CaCl_2 0.01 M solution, using a 1:5 (*w/v*) biochar:solution ratio suspension. Electrical conductivity (EC) was measured in a 1:10 (*w/v*; g mL^{-1}) ratio using a conductivity meter. Dried and ground samples were subjected to acid digestion with concentrated HCl/HNO_3 according to the method 3050B of the United States Environmental Protection Agency (USEPA) [24]. Macro and micronutrient contents were determined after nitric-perchloric acid digestion [25]. Nitrate and ammonium were determined by the Kjeldahl method [26]. Humic substances (HS) were determined by fractionation based on their solubility in alkali and acid [27]. Humic substances were extracted by NaOH 0.1 M solution, and the carbon contents were determined according to Yeomans and Bremner [28].

Table 1. Characteristics of the sewage sludge (SS), biochars (SSB300 and SSB500), and soil.

Property ^a	Unity	SS	SSB300	SSB500	Soil
pH (CaCl_2)	—	4.8 ± 0.4	5.8 ± 0.2	6.5 ± 0.3	4.9
C	%	21.0 ± 0.4	23.4 ± 0.4	19.0 ± 0.2	1.98^b
H	%	4.2 ± 0.1	3.6 ± 0.1	1.7 ± 0.1	nd
N	%	3.0 ± 0.1	3.3 ± 0.1	2.3 ± 0.1	nd
C/N	—	7.0 ± 0.1	7.0 ± 0.1	8.3 ± 0.1	nd
P	g kg^{-1}	35.7 ± 2.8	41.1 ± 3.2	61.3 ± 5.6	0.0023^c
K	g kg^{-1}	0.8 ± 0.1	1.1 ± 0.1	1.3 ± 0.1	0.061
Ca	g kg^{-1}	6.6 ± 0.2	6.7 ± 0.2	8.2 ± 0.3	1.908
Mg	g kg^{-1}	0.8 ± 0.1	1.8 ± 0.1	1.7 ± 0.1	0.418
S	g kg^{-1}	6.7 ± 0.2	15.1 ± 1.0	7.4 ± 0.4	nd
PV	mL g^{-1}	0.022 ± 0.001	0.027 ± 0.001	0.053 ± 0.002	nd
SSA	$\text{m}^2 \text{g}^{-1}$	18.2 ± 1.2	20.2 ± 1.8	52.5 ± 4.3	nd
Volatile material	% (db)	55.3 ± 3.4	36.8 ± 4.4	17.8 ± 0.6	nd
Ash	% (db)	41.0 ± 3.7	56.6 ± 2.6	77.6 ± 0.6	nd
Fixed carbon	% (db)	3.6 ± 1.3	6.5 ± 1.8	4.7 ± 0.1	nd
Yield	%	-	86 ± 8	65 ± 4	-
Total Heavy Metal Content					
Cd	mg kg^{-1}	21 ± 8	24 ± 1	32 ± 1	19 ± 1
Co	mg kg^{-1}	14 ± 1	16 ± 1	19 ± 2	22 ± 1
Cr	mg kg^{-1}	86 ± 2	79 ± 3	121 ± 4	116 ± 3
Cu	mg kg^{-1}	110 ± 2	152 ± 2	138 ± 3	6 ± 1
Mn	mg kg^{-1}	82 ± 2	102 ± 1	128 ± 4	70 ± 4
Ni	mg kg^{-1}	59 ± 5	50 ± 4	76 ± 2	23 ± 2
Pb	mg kg^{-1}	142 ± 19	198 ± 2	193 ± 14	0.6 ± 1.4
Zn	mg kg^{-1}	391 ± 2	446 ± 8	537 ± 2	24 ± 1
Available Heavy Metal Content					
Cd	mg kg^{-1}	4.1 ± 0.1	0.9 ± 0.2	1.6 ± 0.2	0.02 ± 0.03
Co	mg kg^{-1}	0.7 ± 0.1	0.2 ± 0.1	0.4 ± 0.1	0.01 ± 0.01
Cr	mg kg^{-1}	0.4 ± 0.1	0.1 ± 0.1	0.2 ± 0.1	0.09 ± 0.04
Cu	mg kg^{-1}	9.1 ± 0.2	3.9 ± 0.2	0.6 ± 0.2	0.39 ± 0.01
Mn	mg kg^{-1}	17.9 ± 1.3	3.6 ± 0.7	3.8 ± 0.6	1.72 ± 0.09
Ni	mg kg^{-1}	2.0 ± 0.1	0.6 ± 0.1	0.6 ± 0.1	0.16 ± 0.01
Pb	mg kg^{-1}	3.0 ± 0.1	1.3 ± 0.4	1.9 ± 0.8	0.53 ± 0.04
Zn	mg kg^{-1}	61.1 ± 1.5	15.1 ± 0.3	16.1 ± 2.3	0.34 ± 0.17

^a Average values \pm standard deviation ($n = 3$); PV: pore volume; SSA: specific surface area; ^b: organic carbon (Walkley–Black); ^c: available P (Mehlich-1); nd: not determined; db: on dry-basis. Modified from Chagas et al. [29].

2.2. Field Trial Area and Experimental Design

The study was conducted at the experimental farm at the University of Brasilia, Brazil (15°56'45" S, 47°55'43" W; 1095 m). The region's climate is classified as type Aw (tropical seasonal savanna), with a rainy season between October and March and a dry season defined from April to September. The average annual rainfall between 2001 and 2017 is 1500 mm, and air temperature ranges between 17 and 22 °C. The climate data were obtained according to the Climatological Standard Normals [30]. The soil in the area is classified as a clayey Oxisol (Typic Haplustox) [31], Gibbsic Ferralsol [32], or Latossolo Vermelho according to the Brazilian Soil Classification [33]. The soil chemical and physical properties (0–0.20 m) before the establishment of the experiment are shown in Table 1. The experiment was set up in an area used for more than 20 years with a low-productivity pasture. Before starting the experiment, the soil was not used for annual crop production. Therefore, as a typical Oxisol of the Central region of Brazil, the soil presented low levels of available nutrients (Table 1). The direct effect of biochar over successive corn cropping (two seasons; 2014/2015 and 2015/2016) was conducted in this experiment beginning in November 2014 as described in our previous study [11]. To study the one-year residual effect, the application of SSB was stopped in the 2016/2017 growing season. Therefore, the present study shows results from the residual growing season (2016/2017).

Sewage sludge biochar is a multielement material. However, it is unbalanced in nutrients for plants. According to Faria et al. [11], potassium is a missing component in this type of biochar. Thus, combining biochar with mineral sources is an essential strategy to fully provide different macro- and micronutrients. In general, the co-application between fertilizer and biochar has previously been recommended by other works [11,34,35]. Thus, in the present study, six treatments were set up in a randomized block design with four repetitions. The following treatments were assessed: (1) Control: without mineral fertilizer and biochar; (2) NPK: with mineral fertilizer; (3) SSB300: with biochar produced at 300 °C; (4) SSB300+NPK; (5) SSB500: with biochar produced at 500 °C; and (6) SSB500+NPK. Each plot measured 20 m² (5 × 4 m).

2.3. Mineral Fertilizer Application

Before starting the study, the experimental area received the “corrective fertilization” as is commonly suggested for the Cerrado region [11]. Details on corrective fertilization and lime application are shown in Supplementary Materials Table S1. As a maintenance fertilizer application, the mineral fertilizer was applied in all years, including the 2016/2017 cropping season. At planting, 714 kg ha⁻¹ of mineral fertilizer (formula 4–14–8, corresponding to the percentage of N, P₂O₅, and K₂O, respectively) were applied, which corresponded to the application of 45 kg ha⁻¹ of P, 48 kg ha⁻¹ of K and 30 kg ha⁻¹ of N. A side-dressing fertilizer application of 150 kg N ha⁻¹ was split into two applications of 75 kg N ha⁻¹. The first application occurred when the corn had 4 to 6 leaves (V4 stage) and the second application of 75 kg N ha⁻¹ when the corn had 8 to 10 leaves (V6 stage).

2.4. Biochar Application

Biochars were applied in the first two growing seasons at a dose of 15 t ha⁻¹ (dry weight) per season and were incorporated into the 0–0.20 m layer of the entire soil area before sowing. The SSB dose was based on the previous study, which indicated that higher yields were obtained with the application of 10–20 t ha⁻¹ of SSB [12].

2.5. Planting and Maize Harvest

As in the previous growing seasons, in December 2016, the maize hybrid cultivar LG 6030 was planted with rows spaced 0.90 m with a plant density of 60,000 plants ha⁻¹ and harvested in May 2017. Supplementary Table S1 shows the main agricultural practices conducted in the experiment from November 2014 to May 2017.

2.6. Plant Collection and Analysis

In March 2017, at maize R2 stage (at full bloom), eight leaves (first leaf bellow and opposite to the corn ear) from eight plants per plot were randomly selected and placed in paper bags for nutrient analysis as described by Malavolta et al. [36]. According to Coelho et al. [37], a leaf is the most frequently chosen control organ to represent the whole plant mainly because of its sensitivity to variation of nutrient supply in the soil. P was analyzed by the metavanadate colorimetric method, total nitrogen by the semi-micro Kjeldahl method, K with atomic absorption spectrometry, whereas, Ca, Mg Cu, and Zn was determined by atomic absorption spectrophotometry. Corn nutrient uptake was obtained based on the dry matter yield (DMY, kg ha^{-1}) and the leaf nutrient content (in kg ha^{-1}). Corn grain yield was determined at harvesting in May of 2017, considering 15 plants plot^{-1} .

2.7. Soil Sampling and Analysis

In May 2017, after the corn harvesting, soil samples were collected randomly in the plots, at the 0–0.20 m soil layer, using a Dutch auger. The collected samples were air-dried and passed through a 2 mm sieve to determine P, K, Ca, Mg, pH, CEC, and the sum of bases (SB) according to procedures by Teixeira et al. [38]. Soil pH was analyzed in CaCl_2 using a 1:2.5 (*v:v*) soil:solution ratio suspension. Soil P and K contents were determined with the Mehlich-1 extractor, whereas, Ca and Mg were extracted with a 1 mol L^{-1} KCl solution.

2.8. Statistical Analyses

Data followed a normal distribution according to the Lilliefors test and had homogeneity of variance by Bartlett's test. In addition, the residues showed homoscedasticity by the Cochran test. Data were analyzed using a one-way ANOVA, followed by Tukey's HSD test ($p < 0.05$) as a post hoc to detect statistically significant differences among all treatments. All statistical analyses were performed using the XLSTAT 2013 software [39].

3. Results and Discussion

3.1. Physicochemical Properties of Feedstock, Biochar, and Soil

The physicochemical properties of the products after the pyrolysis are shown in Table 1. The proportions of the fixed carbon, ash and volatile materials, yield of the SSB300 and SSB500 are aligned with other works [17,40]. Overall, fixed carbon, ash, pore-volume, specific surface area and yield increased with pyrolysis temperature. On the other hand, volatile materials decreased with increasing biochar temperature. This may reduce the soluble nutrient fractions, especially P, due to increasing the pyrolysis temperature [19].

In general, the pyrolysis of the SS enriched the macronutrients in the biochar. SSB300 showed a higher yield than SSB500, corroborating a previous study [41]. After the biochar amendment, phosphorus was the nutrient most affected. After two years of biochar application, the increase in total P was around four times greater than the total P accumulation provided by a no-till system conducted in the same soil type for 17 years [42]. The application of SSB either exclusive (SSB300 and SSB500) or combined with NPK (SSB300+NPK and SSB500+NPK) to the soil increased P contents considerably compared to NPK and the control (Figure 1; $p < 0.05$). The initial SS was rich in P [43] and, during pyrolysis, there are losses of some gaseous elements, containing C, H, O and N atoms [44,45], increasing, even more, the P concentration in the final SSB. In general, P in SS is mainly found as aluminum phosphate [46], a form that is resistant to volatilization below 700 °C [47]. For those reasons, treating SS by thermochemical conversion has great potential to produce a rich source of P for agriculture, mainly to be applied in highly weathered soils that are naturally deficient in P and need large inputs of P fertilizers [48]. Furthermore, the application of SSB to the soil may improve the quantity and availability of P for plants by reducing the mechanisms of specific desorption with Al and Fe oxides [49]. These mechanisms are common in Brazilian Oxisols and represent the main process responsible for reducing phosphate fertilization efficiency in the Central region of Brazil [48]. Furthermore, highly soluble sources are able to rapidly release P to plants after dissolution in soil solution but may have the adverse

effect of prompting exposure of P to adsorption sites [42]. Therefore, our results highlight the importance of using biochar in tropical soils, as reported by Jeffrey et al. [15].

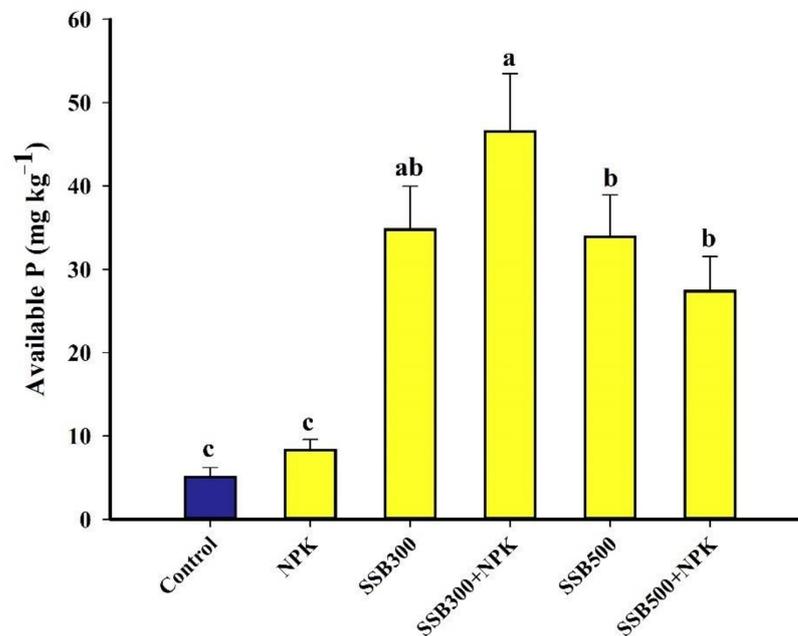


Figure 1. Soil available P under the one-year residual effect of biochars and mineral fertilizer, where: Control—without mineral fertilizer and biochar; NPK—with mineral fertilizer; SSB300—with biochar produced at 300 °C; SSB300+NPK—with biochar produced at 300 °C and mineral fertilizer; SSB500—with biochar produced at 500 °C; and SSB500+NPK—with biochar produced at 500 °C and mineral fertilizer. Different letters indicate that the fertilizations differed significantly by Tukey’s test ($p < 0.05$). Error bars represent the standard deviation ($n = 4$).

Despite the higher P content in the SSB produced at 500 °C, there was no significant difference between SSB300 and SSB500 in soil P residual contents ($p > 0.05$). In general, lower temperatures are recommended to obtain richer biochars in readily available forms of nutrients, with a better prospect to be used as fertilizers [50]. When 20 t ha⁻¹ of wood-derived biochar, produced at 300 °C and 350 °C, was applied to the soil, available P increased, especially when added in combination with NPK fertilizers [51]. In the present study, the concentration of soil available P after suspending the SSB application was higher than in the NPK treatment, where P is annually applied. Available soil P contents in plots that received SSB were around five times higher than the control and the NPK treatments, respectively. These results reinforce conclusions by Yuan et al. [13], who indicated that SSB might be used as a slow-release phosphate fertilizer that would increase soil fertility over the years. In the present study, the soil available P contents in both biochar treatments under residual effect were similar to those found in the first two years of the same experiment when SSB has been applied annually [11].

SSB500 alone or combined with NPK (SSB500+NPK) promoted similar content of soil available P content, despite the higher amount of P, applied via the combination of SSB500 with NPK. The higher corn productivity justifies the lack of differences within the treatment receiving the combination (SSB500+NPK) compared to SSB500 (Figure 3), reflecting the higher P uptake by the plant and consequently decreasing the residual P content available in the soil. Likewise, the absence of difference in available P content between the control and NPK treatments was also justified by the higher productivity and higher P uptake by corn in the NPK treatment, as shown later (Figure 3).

The present study demonstrated that SSB has great potential in providing available P to the soil even one year after suspending its application. However, biochar’s residual

effect on soil P availability was dependent on the pyrolysis temperature and NPK use. Soil amended with SSB300+NPK had 11.79 mg P kg⁻¹ and 20 mg P kg⁻¹, significantly higher than with SSB300 and SSB500+NPK, respectively. Despite its higher total P content, SSB500+NPK presents lower soil P than SSB300+NPK. A small portion of SSB500 may have moved to depths below the sampled soil layer. Furthermore, the predominance of recalcitrant forms of P in biochars made at higher temperatures [52] may explain the lower content of the soil available P in the SSB500+NPK. Unlike our results, when other feedstock-derived biochars (e.g., peanut hull and pine chip) were used, no residual effects of soil P were noticed [53]. Biochars from plant residues did not maintain the increase in P availability over the years due to the low content of this nutrient in the feedstocks [54,55].

Under the one-year residual effect, biochars did not affect soil pH (Table 2). Similarly, in our previous study [11], under the direct influence, SSB did not alter soil pH. Nevertheless, it is well-known the liming effect of biochar in acidic soils [56,57], mostly when the biochar is obtained under higher pyrolysis temperature [5,43,57]. This liming effect is commonly associated with increases in alkaline elements—Ca and Mg [43] due to gaseous losses of other elements such as C, H, O and N during the pyrolysis process. However, in the present study, this was not observed due to the previous application of lime in the whole experimental area in November 2014 (Supplementary Table S1).

Table 2. Soil chemical properties (average \pm standard deviation; $n = 4$) at the 0.00–0.20 m layer under the one-year residual effects of biochar and mineral fertilizer.

Treatment	pH		K		Ca		Mg	
	CaCl ₂		mg kg ⁻¹		cmol _c kg ⁻¹			
Control	5.01	$\pm 0.34a$	48.90	$\pm 3.90b$	2.36	$\pm 0.53a$	0.57	$\pm 0.18a$
NPK	5.21	$\pm 0.32a$	77.40	$\pm 16.05a$	2.88	$\pm 0.59a$	0.65	$\pm 0.24a$
SSB300	4.76	$\pm 0.54a$	48.83	$\pm 7.62b$	2.32	$\pm 1.28a$	0.53	$\pm 0.36a$
SSB300+NPK	4.87	$\pm 0.21a$	77.06	$\pm 26.35a$	2.68	$\pm 0.59a$	0.46	$\pm 0.10a$
SSB500	5.18	$\pm 0.23a$	49.37	$\pm 7.97b$	2.87	$\pm 1.04a$	0.68	$\pm 0.31a$
SSB500+NPK	4.77	$\pm 0.15a$	66.11	$\pm 14.29ab$	2.23	$\pm 0.60a$	0.46	$\pm 0.17a$

Different letters indicate that the fertilizations differed significantly by Tukey's test ($p < 0.05$). Soil K content was determined with the Mehlich-1 extractor, Ca and Mg with the KCl (1 mol L⁻¹) extractor solution.

Applying SSB (either SSB300 or SSB500) alone did not affect the residual soil K contents compared to the control (Table 2). Even under direct effect, SSB alone was incapable of increasing the soil K content, confirming the low levels of this nutrient in SSB, as reported in our previous study [11]. This result shows the need to reapply potassium fertilizers with SSB in every cropping season. SS contains low levels of K because this element is water-soluble, and it is not maintained in the final dried-solid SS during the sewage treatment [58]. In a previous study that evaluated the use of SSB for growing radish, the amount of K was only satisfactory when higher doses ($>5\%$ w/w) of SSB were applied to soil [12]. The same was observed for sawdust-derived biochar when applied to soil—low soil K contents [51]. However, other feedstocks-derived biochars, such as those made from hardwood, peanut hull, and pine chips, increased soil K contents [50,52,58,59].

Soil Ca and Mg residual contents were not affected by the application of SSB or NPK (Table 2). Similar to the soil pH values, which were also not affected by SSB application, Ca and Mg contents may have been influenced by the previous application of lime (Ca and Mg-rich) in November 2014. Therefore, further studies should evaluate the residual alkaline effect of SSB in tropical soils, but without applying lime in long-term field trials.

SSB300 showed a one-year residual effect on the soil CEC, which was higher than the other treatments, except for SSB300+NPK (Figure 2). SSB500 either applied alone or in combination with NPK, showed similar CEC values compared to the control and NPK ($p < 0.05$). Biochars produced at lower pyrolysis temperatures are more negatively charged and easily oxidized, resulting in higher soil CEC [50]. Such differences between lower and higher pyrolysis biochars (e.g., 300 °C and 500 °C) may be explained by forming functional

groups on the surface of SSB300 during pyrolysis. Biochars produced at pyrolysis temperatures lower than 400 °C result in higher CEC due to carboxylic groups capable of adsorbing nutrients and improving soil fertility. In contrast, at higher pyrolysis temperatures, the CEC decreases [60]. Jien and Wang [61] verified increases in soil CEC when biochars made at lower temperatures were used. Therefore, this study confirms the residual effect of SSB300 to increase soil CEC at least one year after its application.

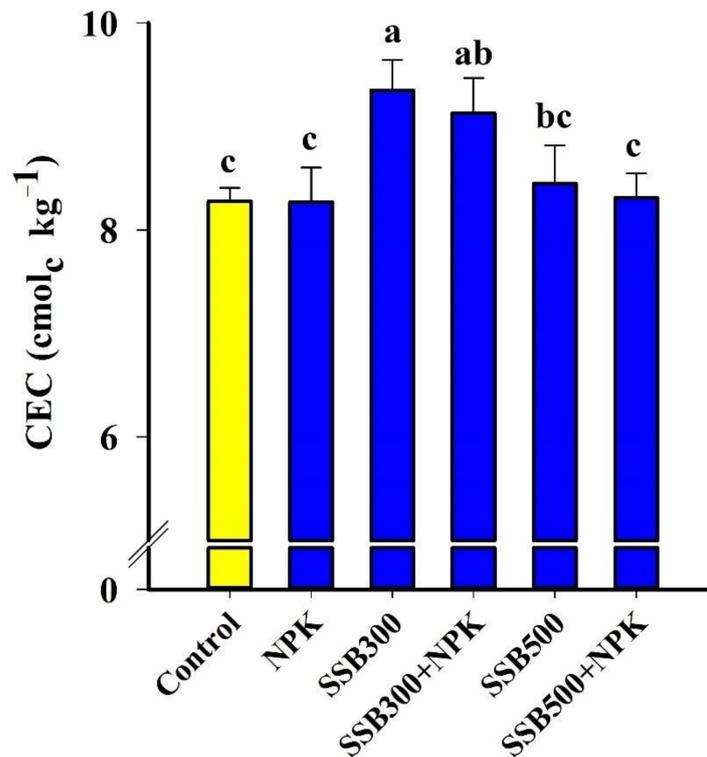


Figure 2. Cation exchange capacity (CEC) in soil amended with biochar and mineral fertilizer. Control—without mineral fertilizer and biochar; NPK—with mineral fertilizer; SSB300—with biochar produced at 300 °C; SSB300+NPK—with biochar produced at 300 °C and mineral fertilizer; SSB500—with biochar produced at 500 °C; and SSB500+NPK—with biochar produced at 500 °C and mineral fertilizer. Different letters indicate that the fertilizations differed significantly by Tukey’s test ($p < 0.05$). Error bars represent the standard deviation ($n = 4$).

3.2. Nutrient Uptake by Maize

In general, the higher soil nutrient availability under the one-year residual effect of biochar reflected positively in nutrient uptake. Biochar also contributed to expanding the mineral fertilizer use efficiency since SSB with NPK increased the nutrient uptake by plants compared to the sole application of NPK (Table 3). Overall, in the present study, under the one-year residual effect, biochar promoted nutrient uptake values similar to the first two years of the experiment, under direct application of biochar [11]. The exception was the SSB500 alone that did not differ from the control in the present study under residual effect.

Table 3. Macronutrients uptake by corn (average \pm standard deviation; $n = 4$) after the first year under the one-year residual effects of sewage sludge biochar and mineral fertilizer.

Treatments	N		P		K	
	kg ha ⁻¹					
Control	52.44	$\pm 8.73c$	7.03	$\pm 0.23c$	62.11	$\pm 5.32c$
NPK	170.70	$\pm 27.69ab$	12.29	$\pm 3.74abc$	138.73	$\pm 18.80ab$
SSB300	141.09	$\pm 39.37ab$	17.19	$\pm 4.09ab$	119.27	$\pm 30.32bc$
SSB300+NPK	186.93	$\pm 50.78a$	21.87	$\pm 9.48a$	137.45	$\pm 52.96ab$
SSB500	110.92	$\pm 33.20c$	10.50	$\pm 0.87bc$	93.44	$\pm 7.042bc$
SSB500+NPK	194.14	$\pm 26.52a$	21.65	$\pm 5.05a$	157.04	$\pm 43.72a$

Treatments	Ca		Mg		S	
	kg ha ⁻¹					
Control	15.94	$\pm 3.23b$	12.95	$\pm 2.57b$	5.27	$\pm 0.79c$
NPK	29.54	$\pm 1.11ab$	18.22	$\pm 1.25ab$	12.20	$\pm 3.73ab$
SSB300	36.88	$\pm 6.39a$	26.95	$\pm 8.80a$	11.51	$\pm 2.58ab$
SSB300+NPK	33.11	$\pm 7.89a$	12.00	$\pm 6.45b$	13.42	$\pm 6.39ab$
SSB500	26.75	$\pm 3.37ab$	20.98	$\pm 3.81ab$	8.95	$\pm 0.55bc$
SSB500+NPK	35.08	$\pm 11.07a$	17.12	$\pm 3.53ab$	16.33	$\pm 3.15a$

Different letters indicate that the fertilizations differed significantly by Tukey's test ($p < 0.05$).

Application of sole SSB300 increased plant N uptake compared to the control. Moreover, it was found to be similar to the application of NPK. In general, SSB500 was less efficient than SSB300 to increase N assimilation by the plant. Higher NO_3^- and NH_4^+ contents in SSB300 (Table 1) reflected in increases in the N uptake by the plants in comparison to SSB500 ($p < 0.05$). Compared to SSB500, SSB300 has more N compounds resistant to volatilization in temperatures up to 300 °C [43]. Furthermore, N contents in biochars commonly decreased with higher pyrolysis temperatures [11,37,39]. In fact, SSBs produced at lower temperatures (around 300 °C) usually result in higher available N and, consequently, increase the N use efficiency by plants [10].

P uptake by maize was also higher under SSB300 application than the control ($p < 0.05$) and was similar to NPK. As a result, SSB300, even under one-year residual effect, was able to replace the soluble P fertilizers needed for corn production in the Cerrado soils. On the other hand, SSB500 showed no residual effect for P uptake, presenting the need to be supplemented with NPK. Although P contents in biochars increased with increasing temperatures (Table 1), no residual accumulation of soil P was observed. Probably, the presence of low solubility phosphate forms found in biochars produced at higher temperatures [62,63] explains the differences verified in the present study.

Nevertheless, Xu et al. [64], studying the effect of wheat straw-derived biochar on P transformations in a Haplic Luvisol in China, observed that the incorporation of biochar produced at 300 °C increased soil CEC compared to the 500 °C. As previously highlighted in the present study, the soil CEC was also higher for SSB300 than SSB500. The higher CEC may have induced more negatively charged surfaces in soil [65], increasing anion repulsion and decreasing P adsorption by the soil [66]. Despite the growing number of research that aims to understand the SSB-derived P availability and uptake by plants, it is still necessary to comprehend how forms of this element are found and released over the years. According to DeLuca et al. [67], thermochemically treated feedstocks may contain various forms of P with different levels of availability. Generally, SSB is a more nutrient-rich material (especially for P) than biochars made from multiple other feedstocks [9].

As expected, SSB applied alone (SSB300 and SSB500) showed a little one-year residual effect on K uptake by the plants and was similar to the control treatment ($p < 0.05$). As mentioned above, SSB has a minimal amount of K (Table 1), which reinforces the need to blend or mix SSB with potassium fertilizers.

SSB300 applied one year earlier (SSB300 and SSB300+NPK) increased Ca uptake by the plants compared to the control ($p < 0.05$). Therefore, such results may be related to

the use of SSB300 that favored higher corn biomass yield and consequently increased Ca uptake. SSB500 only increased Ca uptake, compared to the control, when the NPK was applied together (SSB500+NPK). Regarding magnesium (Mg), the one-year residual SSB300 again increased its uptake by the plants compared to the control. Major et al. [68] reported Ca and Mg increases when wood-derived biochar was applied to soil four years earlier. These authors associated such increases with less Ca and Mg leaching, due to biochar's adsorption characteristics, after lime application.

All treatments, except SSB500, increased sulfur (S) uptake compared to the control ($p < 0.05$). As a result, the SSB500 did not show any residual effect on S uptake after one year of its suspension. SSB500 has approximately half the S content as the SSB300 (Table 1).

3.3. Maize Grain Yield

All the soil amendments (with SSB, NPK, and combinations) promoted higher corn grain yield than the control ($p < 0.05$; Figure 3). These increases are related to higher nutrient uptakes by using these inputs. In general, our results differ from those obtained in soils of temperate latitudes where biochar had no effect or potentially negative effects on yields [15].

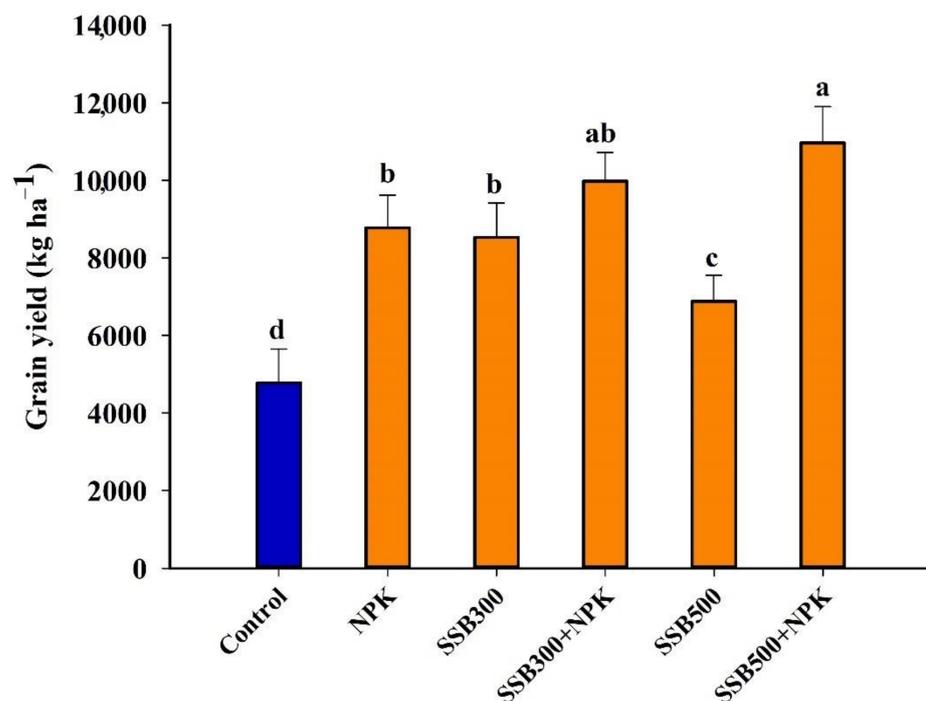


Figure 3. Maize grain yield response to the one-year residual effect of biochar and mineral fertilizer. Control—without mineral fertilizer and without biochar; NPK—with mineral fertilizer; SSB300—with biochar produced at 300 °C; SSB300+NPK—with biochar produced at 300 °C and mineral fertilizer; SSB500—with biochar produced at 500 °C; and SSB500+NPK—with biochar produced at 500 °C and mineral fertilizer. Different letters indicate that the fertilizations differed significantly by Tukey's test ($p < 0.05$). Error bars represent the standard deviation ($n = 4$).

According to our results, the increase in grain yields was due to the higher nutrient availability promoted by biochar application, mainly P. Even one year after applying SSB300 in the plots, the biochar supplied the necessary nutrients to reach the same maize yield as the one observed for NPK treatment. SSB300 promoted grain yields 44% greater than the control. These values were much higher than the 25% average increase in crop yields in biochar amended soils found for tropical regions [15]. In most long-term studies, biochars only expressed their potential for increasing productivity one year after appli-

cation [54,60,69–71]. As demonstrated in our previous work under the direct effect of biochar [11], using the SSB300 dispenses mineral fertilizer for at least one more cropping season after its application in Cerrado tropical soils. Further research will be needed to evaluate how long SSB300 will keep up with the same productivity of the NPK fertilizer. Nevertheless, 20 t ha⁻¹ of wood-derived biochar applied into tropical acidic soil was enough to maintain crop yields for at least four consecutive years after its application [60]. Similarly, for cultivating Cassava roots, the application of garden cuttings-derived biochar was able to increase productivity up to three successive years [69]. Corn yield response to SSB500 was only positive when it was blended with NPK, where SSB500+NPK was the best arrangement to increase corn grain yield ($p < 0.05$). Furthermore, biochars produced at higher pyrolysis temperatures (500 °C) when combined with mineral fertilizers can increase corn productivity by approximately 2 t ha⁻¹ compared to the application of NPK alone. This increase of 2 t ha⁻¹ in maize yield highlights the possibility of using SSB as a corrective fertilizer, followed by annual applications of soluble mineral fertilizers. In the Central region of Brazil, a common practice in agriculture is the use of “corrective fertilization practice” that includes the addition of fertilizers such as P and K before planting, depending on soil analysis. However, further studies need to assess the economic feasibility of applying SSB as a corrective fertilizer. The economic feasibility of using SSB should consider C price scenarios. The cost–benefit analysis showed that the optimal biochar application dose was 15 t ha⁻¹ for all C price scenarios, increasing gross margin by 21% and 53%, respectively, for 0 and US\$ 42 per ton CO₂ price scenarios [72]. Studies have also shown positive synergistic effects of biochars (corn straw, wood, and chicken litter-derived biochars) and NPK fertilizers in the soil for cotton, soybean, and sunflower productivities [14,51].

4. Conclusions

The results of the present study express the one-year residual effect of sewage sludge biochars on soil properties and maize yield. We concluded that there is a need to continue studying the residual effect of biochar for a longer period of time. It is possible to conclude that sewage sludge biochars showed one-year residual effects on soil properties, such as increasing soil P availability and cation exchange capacity. The one-year residual SSB contributed to increasing the macronutrient uptake by corn. Considering the exclusive application, SSB300 stood out and promoted higher grain yield in the one-year residual period (8524 kg ha⁻¹) than SSB500 (6886 kg ha⁻¹). Consequently, the biochar produced at 300 °C showed the potential to substitute mineral fertilizers over one year after its suspension. Both SSB made at 300 °C, and 500 °C combined with mineral fertilizer (NPK) are agronomically efficient to improve soil fertility, nutrient uptake, and increase maize yield. Additional studies should be focused on the long-term residual effects of SSB on the soil fertility status and grain yield. Furthermore, heavy metals dynamics in the soil–plant system need to be better understood in future research with SSB.

Supplementary Materials: The following are available online at <https://www.mdpi.com/2071-1050/13/4/2226/s1>, Table S1: Description of the seasonal agricultural practices and fertilizer inputs during the experimental period.

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