Science of the Total Environment 739 (2020) 140013

Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Direct and residual effect of biochar derived from biosolids on soil phosphorus pools: A four-year field assessment

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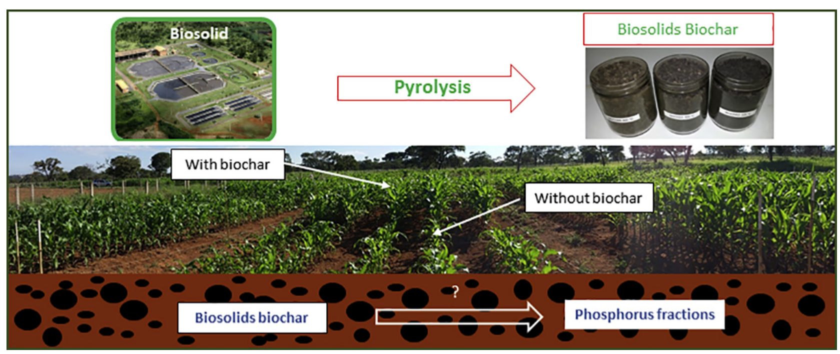
HIGHLIGHTS

- P fractions in soil amended with biosolids biochar (BBC) were investigated.
- BBC, regardless of pyrolysis temperature, increased soil total P and all P fractions.
- In a BBC-amended soil, available P was the only fraction capable of predicting corn yield.
- BBC produced at 300 °C was able to replace mineral fertilization.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 19 February 2020
Received in revised form 29 April 2020
Accepted 4 June 2020
Available online 6 June 2020

Editor: Charlotte Poschenrieder

Keywords:
Pyrolysis
Sewage sludge
Soil phosphorus fraction

ABSTRACT

Measures to improve the use of phosphorus (P), either by improved efficiency or reuse, are needed worldwide in order to preserve a finite resource and ensure that farmers have access to it. Currently, the rapidly growing global population has generated an increased demand for this mineral. Sustainably disposing for the massive amount of globally produced biosolids and alternative sources of P for agriculture are two major challenges to address. In this scenario, biosolids-derived biochar (BBC) has been presented as a win-win opportunity. However, the BBC-P dynamics in soil over consecutive cropping seasons remain unclear. Direct (first and second cropping season) and residual (third and fourth cropping season) effects of BBC on soil P fractions, P uptake and corn grain yield were assessed. Additionally, the relationships between soil P pools and grain yield were investigated by multivariate and multiple linear regression analysis. In a field experiment, BBC produced at 300 °C (BC 300) and 500 °C (BC 500) were applied to an Oxisol at a rate of 15 t ha^{-1}. Soil total P and its fractions (organic P, inorganic P, and available P) were determined. Phosphorus uptake and corn grain yield were also evaluated. BBC, regardless of pyrolysis temperature, increased soil total P and all P fractions. Moreover, BBC maintained high soil P contents for at least two years after stopping its application. These results suggest that BBC may act as a slow-release P fertilizer. Surprisingly, soil P fractions were unaffected by different pyrolysis temperatures, but BC 300 promoted higher grain yield than BC 500 in the third and fourth cropping seasons. Overall, the results confirmed that under direct application both biochars can replace mineral fertilization for corn production; and when considering the residual effect, BC 300 showed a higher potential to be utilized as a soil amendment for P supply.

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https://doi.org/10.1016/j.scitotenv.2020.140013
0048-9697/

1. Introduction

Sewage sludge production has accompanied the rapid population growth in recent decades around the world (Collivignarelli et al., 2019). Globally, an estimated 9.5 million m^3 of human sewage and 900 million m^3 of municipal wastewater are produced every day (Mateo-Sagasta et al., 2015). After further treatment the sewage sludge is referred to as biosolids, and this product has beneficial properties for various applications (e.g., soil amendment, construction sector, alternative fuels) (Collivignarelli et al., 2019). The application of biosolids to agricultural land is regulated by different legislation. For example, in the European Union by Directive 2008/98/EC (EP/CEU, 2008), in the US by 40 CFR Part 503 (US EPA, 2015), and in Brazil by Resolution 375 (Brazil, Ministry of the Environment, 2006). In Australia, each state has a specific regulation (EPA Victoria, 2004). All these regulations limit the use of biosolids on agricultural land, mainly as a precautionary measure due to the presence of organic and inorganic pollutants, as well as pathogens (Paz-Ferreiro et al., 2018).

Thermal treatment via pyrolysis is an effective technological alternative to transform biosolids into an agricultural input (Paz-Ferreiro et al., 2018). The solid product of biosolids pyrolysis, rich in carbon and plant nutrients, is called biosolids biochar (BBC). Concerns regarding potential soil contamination with heavy metal due to BBC amendment exist (Shepherd et al., 2017; Yang et al., 2018). The availability of heavy metals in soils amended with SBB is affected by several factors, including the type of sewage, pyrolysis temperature and soil characteristics (Yang et al., 2018; Figueiredo et al., 2019a). In general, non-industrial sewage, subjected to tertiary treatment, results in biosolids with a lower concentration of heavy metals and a higher concentration of nutrients such as P and N. Recent studies have shown that despite increasing the concentration of total heavy metals in relation to the raw material, this thermal process reduces the bioavailability of metals such as cadmium, arsenic, mercury and lead (Paz-Ferreiro et al., 2018; Figueiredo et al., 2019a).

Nutrient content in biosolids depends on the untreated water source, chemicals used for purification and unit operations used (Torri et al., 2017). In most treatment systems P is removed from wastewater by biological treatment or physiochemical precipitation (De-Bashan and Bashan, 2004). As a result, at the end of treatment almost all of the sewage P remains in the biosolids. Among multiple feedstocks (e.g. manure, woody and herbaceous) used for producing biochar, biosolids are considered P-rich feedstocks, with higher water-soluble P and total P content (Li et al., 2019). Application of BBC to soil can increase P availability by up to 38 times (Yue et al., 2017) thus providing higher crop yields (Khan et al., 2013; Faria et al., 2018). Therefore, the combination of massive amounts of P-rich biosolids with the imminent scarcity of mineral P reserves makes BBC production a win-win opportunity.

In general, biochar can act either as a short-, mid-, or long-term P fertilizer which will depend on the feedstock and pyrolysis temperature used during production and the quantity applied to the soil (Glaser and Lehr, 2019). The concentration and chemical forms of P in BBC are also influenced by pyrolysis temperature, heating rate and residence time (Adhikari et al., 2019). In general, higher pyrolysis temperatures result in biochars rich in more stable inorganic P forms (Li et al., 2019).

The P contained in biochars, when applied to the soil, can be released and used by plants. However, release of this P is part of the biochar aging process (Li et al., 2019) and may be difficult to predict over time. This may hinder farmer's adoption of biochar as a soil amendment. In this sense, determining the organic and inorganic pools of P in soil may be a simple strategy to understand the function of biochar as a P source for plants.

Despite recent advances in research with the application of P-rich biochar to soil, approximately 85% of experiments have been conducted in pots (Li et al., 2019). For this reason there is still little known regarding the dynamics of P-rich biochars under natural soil conditions and interactions with soil microorganisms. In addition, knowing the chemical forms of P in soil after BBC application is important to understand how the biochar acts as a source of P for plants. For example, Glaser and Lehr (2019) concluded that application of biochar to acidic soil significantly increased plant-P availability by a factor of 5.1, while there was no significant effect in alkaline soils. Forms of P in BBC (Schneider and Haderlein, 2016) and in biochars from several feedstocks, including wood and crop residues (Melia et al., 2019) are associated with Ca and may present alkaline reactions in acidic soils, promoting greater P availability. In fact, biochar total Ca concentration is closely related to P availability (Buss et al., 2018).

Some studies have indicated that biochar releases P more slowly than raw material from which it is derived (Liang et al., 2014; Jin et al., 2016). Thus, biochar has potential P fertilizer value and may act as a slow-release fertilizer in soils (Schneider and Haderlein, 2016). Previous studies have shown occasional effects of 15 t ha^{-1} of BBC on the soil available P with increases that reach up to 1150% in relation to the control (Faria et al., 2018; Figueiredo et al., 2019b). However, for BBC to be used as a source of P for plants much remains to be understood, mainly with regards to the forms and concentration of P in BBC, as well as its dynamics in soils. The characteristics of P release from biochar and its long-term sustained-release mechanism in the soil are not yet fully understood (Li et al., 2019) as few studies assessed BBC in field conditions, with most presenting results over a maximum of two years (Farrell et al., 2014; Figueiredo et al., 2019b). Our previous studies demonstrated the short-term benefits of biosolid biochar application on various aspects of soil chemistry (Faria et al., 2018; Figueiredo et al., 2019a; Figueiredo et al., 2019b). The present study elucidates the effect of biochar on soil phosphorus fractions under a long-term cropping seasons sequence.

The following hypotheses were proposed for biosolids obtained from tertiary treatment: (*i*) BBCs produced at different pyrolysis temperatures promote different concentrations of soil P fractions and different corn yield; (*ii*) BBC has a residual effect on corn yield and soil P fractions.

2. Material and methods

2.1. Acquisition and characterization of biosolid biochar

Biochars were produced from biosolid samples collected at the wastewater treatment plant (WWTP) belonging to the Environmental Sanitation Company of the Federal District, Brasília, DF, Brazil. The origin of the sewage is mostly domestic and the WWTP utilizes a tertiary treatment system including the addition of aluminum sulfate. In this system not only does anaerobic sludge decomposition occur, but specific nutrients such as P and N are removed from the liquid effluent, which remain in the final biosolids biomass.

The biosolids were air-dried (approximately to 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 (Linn Elektro Therm, Eschenfelden, Germany) with 610 × 610 × 590 mm (w × d × h) at a mean temperature increase rate of 2.5 °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 with volume of 30 L 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. In total, 16 thermal treatment batches were carried out to produce 480 kg of biochar. From each batch produced, aliquots of approximately 200 g were taken to perform the physical-chemical analyses. Table 1 presents the general characteristics of the biosolids and biochars used in this study. Background information on biosolid and biochar physicochemical characterization is available in our previous works (Figueiredo et al., 2018; Figueiredo et al., 2019a).

Table 1
Characteristics of the biosolids and the biochars.
Modified from Figueiredo et al. (2018) and Figueiredo et al. (2019a).

Property	Biosolids	BC 300	BC 500
Carbon (C) (%)	21.0 ± 0.4	23.4 ± 0.4	19.0 ± 0.2
Hydrogen (H) (%)	4.2 ± 0.1	3.6 ± 0.1	1.7 ± 0.1
H/C	2.4 ± 0.1	1.8 ± 0.1	1.1 ± 0.1
Nitrogen (N) (%)	3.0 ± 0.1	3.3 ± 0.1	2.3 ± 0.1
C/N	7.0 ± 0.1	7.0 ± 0.1	8.3 ± 0.1
NO_3^- (mg kg^{-1})	23.3 ± 3.4	17.5 ± 2.8	5.8 ± 0.9
NH_4^+ (mg kg^{-1})	461.2 ± 36.0	431.9 ± 31.0	169.3 ± 19.8
pH ($CaCl_2$)	4.8 ± 0.4	5.8 ± 0.2	6.5 ± 0.3
P (g kg^{-1})	35.7 ± 2.8	41.1 ± 3.2	61.3 ± 5.6
K (g kg^{-1})	0.8 ± 0.1	1.06 ± 0.1	1.25 ± 0.1
Ca (g kg^{-1})	6.6 ± 0.1	6.7 ± 0.2	8.2 ± 0.3
Mg (g kg^{-1})	0.8 ± 0.1	1.8 ± 0.1	1.7 ± 0.1
S (g kg^{-1})	6.7 ± 0.2	15.1 ± 1.0	7.4 ± 0.4
Total Cu (mg kg^{-1})	115 ± 1	148 ± 1	145 ± 1
Total Pb (mg kg^{-1})	207 ± 1	256 ± 3	265 ± 1
Total Zn (mg kg^{-1})	306 ± 1	321 ± 1	411 ± 5
Total Cr (mg kg^{-1})	100 ± 1	106 ± 2	136 ± 1
Total Co (mg kg^{-1})	20 ± 1	22 ± 1	25 ± 1
Total Mn (mg kg^{-1})	56 ± 1	58 ± 1	80 ± 2
FA (g kg^{-1})	nd	24.3 ± 1.8	4.3 ± 0.8
HA (g kg^{-1})	nd	19.3 ± 1.3	1.1 ± 0.1
Humin (g kg^{-1})	nd	74.8 ± 4.8	95.6 ± 6.8
Helminths (a)	nd	0	0
Thermotolerant coliforms (b)	nd	<1	<1
PV (mL g^{-1})	0.022 ± 0.001	0.027 ± 0.001	0.053 ± 0.002
SSA (m^2 g^{-1})	18.2 ± 1.2	20.2 ± 1.8	52.5 ± 4.3
Moisture content (65 °C) (g g^{-1})	0.17 ± 0.01	0.004 ± 0.001	0.003 ± 0.001
Ash (g g^{-1})	0.54 ± 0.03	0.59 ± 0.02	0.79 ± 0.04
Yield (%)	–	86 ± 8	65 ± 4

Average values ± standard deviation ($n = 3$); FA - fulvic acid; HA - humic acid; PV - pore volume; SSA - specific surface area; [a](MPN/g dry matter); [b](viable eggs/g dry matter); nd – not determined.

2.2. Field trial area

The field trial was conducted at an experimental farm located at latitude 15°56′ 45″ S, longitude 47°55′43″ W and elevation of 1095 m. The climate of the region is type Aw (tropical seasonal savanna, Köppen). The regional climate is classified as tropical savanna-Aw (Köppen classification), with a rainy season from October to March and a dry season from April to September. Fig. S1 shows the precipitation in the experimental area over four cropping seasons highlighting January when 82% of dry spell occur in the region (Silva et al., 2017).

The study was conducted for four years (2014/2015, 2015/2016, 2016/2017, and 2017/2018 - 1st, 2nd, 3rd, and 4th season cropping, respectively) in a soil classified as Red-Yellow Latosol (Typic Haplustox), with clayey texture (Soil Survey Staff, 2014). Before initiating the experiment, soil samples were collected for physical and chemical characterization. The main characteristics of the soil are shown in Table 2.

2.3. Soil preparation, crop management, and amendments application over the four cropping seasons

2.3.1. Soil preparation, liming and corrective fertilization

Fig. S2 and Table S1 show the sequence of operations performed in the experimental area over the four cropping seasons. Prior to the start of the experiment, the area was under low-productivity pasture and soil presented low nutrient contents. Since the soil had "low" P (2.3 mg kg^{-1}) and K (61.0 mg kg^{-1}) contents, "corrective fertilization" practices were needed as is commonly suggested for the Central region of Brazil (Zinn and Lal, 2013). Therefore, before establishing the experiment, in November 2014, the total area was ploughed, harrowed and fertilized with 200 kg ha^{-1} of P_2O_5 (single superphosphate – 1110 kg ha^{-1}) and 51 kg ha^{-1} of K_2O (potassium chloride – 85 kg ha^{-1}). Before the 2nd cropping season (in November 2015), the soil was analyzed and again indicated the need for "corrective fertilization". Therefore, before corn planting, all the experimental plots received 100 kg ha^{-1} of P_2O_5 (single superphosphate – 555 kg ha^{-1}) and 60 kg ha^{-1} of K_2O (potassium chloride – 100 kg ha^{-1}).

2.3.2. Establishment of the experiment

After soil preparation, liming and corrective fertilization, the experiment was established. Each experimental plot measured 20 m^2 (5 m × 4 m) and we studied the following treatments: 1) Control: without biochar and without fertilizer NPK; 2) NPK: with fertilizer NPK; 3) BC300: with biochar produced at the pyrolysis temperature of 300 °C; 4) BC500: with biochar produced at the pyrolysis temperature of 500 °C.

2.3.3. Biochar application

In the two first cropping seasons (2014/2015 and 2015/2016) both biochars were applied at 15 t ha^{-1} (dry weight), corresponding to 0.7% of biochar per soil weight (w/w). Biochars were spread manually and incorporated into the soil in the 0–20 cm layer using a rotary hoe before corn planting in both cropping seasons. The amount of biochar applied was based on the review study of Jeffery et al. (2011), where it is indicated that only doses higher than 10 t ha^{-1} could increase crop productivity. Also, preliminary results from Sousa and Figueiredo (2016) demonstrated that higher productivities were obtained with the application of 10 to 20 t ha^{-1} of BBC. No biochar was applied in the two last cropping seasons (2016/2017 and 2017/2018), remaining only its residual effect.

2.3.4. Mineral fertilizer application

Mineral fertilization (NPK) was performed in the four cropping seasons. In each cropping season, mineral fertilization was performed at the planting (in December) and as side-dressing fertilization (in December and January). Commercial NPK fertilizer (adubos araguaia®) was used in granular form (3–4 mm). The NPK formula was comprised by urea, single and triple superphosphate, and potassium chloride as source of nitrogen, phosphorus and potassium, respectively. At the planting the mineral fertilization consisted of applying 714 kg ha^{-1} of NPK, corresponding, therefore, to 30 kg ha^{-1} of N (as urea), 45 kg ha^{-1} of P_2O_5 (as single and triple superphosphates) and 48 kg ha^{-1} of K_2O (as potassium chloride). As side-dressing fertilization 75 kg N ha^{-1} + 48 kg K ha^{-1} at V4 stage and 75 kg N ha^{-1} at V6 stage were applied according to crop-specific requirements in the Cerrado region and based on soil chemical analysis to obtain a grain yield of approximately 10 t ha^{-1} (Sousa and Lobato, 2004).

2.3.5. Planting, management and corn harvest

In all cropping seasons the corn hybrid LG 6030 was cultivated in each plot, composed of 5 rows spaced at 0.9 m and with 6 plants m^{-1}, resulting in 66,666 plants ha^{-1}. Only mechanical treatments were performed periodically for the control of weeds, pests and diseases. Every year corn harvest took place in May.

Table 2
Soil chemical and physical properties prior to the establishment of the experiment[a].
Modified from Faria et al. (2018).

P	pH	K^+	Ca^{2+}	Mg^{2+}	H + Al	SB	Al^{3+}	V	Sand	Silt	Clay
mg kg^{-1}	(H_2O)	cmol$_c$ kg^{-1}						%	g kg^{-1}		
2.3	4.9	0.1	2.4	0.9	5.9	3.4	0.0	36.6	45	360	595

[a] All values were obtained from only one soil sample comprised of 10 subsamples collected in the experimental area ($n = 1$). P and K: extracted with Mehlich-1 (HCl 0.05 M+ H_2SO_4 0.0125 M); Ca and Mg: extracted with 1 M KCl; H + Al: extracted with calcium acetate buffer solution at pH 7.0; SB = sum of bases; V: base saturation.

2.4. Soil and plant analysis

One week after the corn harvest soil samples were collected in the 0–20 cm layer to determine the total, organic, inorganic and available P contents. Collection was performed using a Dutch auger, taking 5 subsamples in each plot. A circle with a radius of 1 m was drawn from the central point of the plot. The soil subsamples were collected randomly in points in the circle line around the central point of the plot. The 5 subsamples weighed around 1 kg and were put into a rigid plastic pot and mixed to form one composite sample. An aliquot of approximately 200 g of soil sample was used for performing all analyses. For each treatment, four field replicates were analyzed.

Prior to the analyses, the samples were air-dried and passed through a 2 mm mesh. P fractionation was performed according to Hedley's simplified method (Hedley et al., 1982). The chemicals used for the P analysis were H_2SO_4 (95–97%), H_2O_2 (30%), HCl (37%) and $MgCl_2$ (ACS reagent grade; heavy metal <0.0005%). All chemicals were of analytical grade and supplied from Merck. In addition, a certified P standard solution (Sigma Aldrich; 100 mg/L) was used for the analysis of P.

2.4.1. Soil total P

Total P was determined by acid digestion with H_2SO_4 and H_2O_2 in the presence of a $MgCl_2$ saturated solution (Brookes and Powlson, 1981; Hedley et al., 1982). In a digestion tube, 7.5 mL of H_2SO_4 18 M and 1 mL of a $MgCl_2$ saturated solution were added to a 0.15 g sieved (<2 mm) soil sample and heated in a digestion block for 2 h at 200 °C. Temperature was then reduced to 100 °C for 2 more hours, with two additions of H_2O_2 (2 mL each) in 1-hour intervals. The block was then turned off after 2.5 more hours at 180 °C, and P content in the extracts was determined colorimetrically (Murphy and Riley, 1962) after volume completion to 50 mL with distilled water.

2.4.2. Soil organic and inorganic P

Organic P was determined after extraction for 16 h with H_2SO_4 2.0 mol L^{-1}, in the 1:8 soil:solution ratio, of two soil sub-samples with 2 g each: one previously submitted to ignition at 550 °C for 1.5 h and the other not. For each treatment, the four field replicates were analyzed. The organic P was then obtained by the difference in P content of the two acid extracts (Hance and Anderson, 1962; Olsen and Sommers, 1982), analyzed by spectrophotometry at 820 nm (Murphy and Riley, 1962). Thus, the total inorganic soil P was obtained by the total P and organic P difference.

2.4.3. Soil available P

The available P content was determined according to the Mehlich-1 (0.05 M HCl + 0.025 M H_2SO_4) method. Phosphorus concentration in the extracts was measured by the colorimetric method of Murphy and Riley (1962), using spectrophotometer (Tecnal, UV-5100) at an 882 nm wavelength.

2.5. Experimental design and statistical analysis

The experiment consisted of four treatments in a complete randomized block design with four field replicates, totaling 16 plots. To compare the treatment and cropping season effects along the years the data were analyzed considering an incomplete randomized design with repeated measures in time using a linear mixed model. Treatment (T), cropping season (CS) and their interaction ($T \times CS$) were included as fixed effects. Model parameters were estimated by the restricted maximum likelihood method (REML). The comparison of means was conducted with Tukey's test ($P < 0.05$). Normality and homoscedasticity of the residues were evaluated by the Lilliefors and Cochran's C tests, respectively. Scatter plots also were employed to reveal the relationships between variables (soil P fractions, uptake P and grain yield), and Pearson's correlation coefficients were applied to test the significance of such relationships. Additionally, data were also submitted to a multiple linear regression analysis. The regression was performed according to the stepwise procedure in order to select among soil P variables the best predictor to grain yield. All predictors were checked for collinearity based on the variance inflation factor (VIF). The analyses were carried out using the software XLSTAT 2011 (Addinsoft, 2013).

3. Results and discussion

3.1. Soil total P

In each cropping seasons, biochar treatments (BC 300 and BC 500) increased soil total P compared to the other treatments (Fig. 1a). Overall, when considering the average of the four cropping seasons, treatments with biochars had twice the total P content than the other treatments (Fig. 1b). In the two applications (first and second cropping seasons), the biochars, on average, added 446.60 mg kg^{-1} or 892.0 kg ha^{-1} of total P to soil (Fig. 1a), representing an important soil P reserve for various consecutive crops. 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 (Zancanaro de Oliveira et al., 2020). These results corroborate previous studies which showed that BBC is a P-rich material (Li et al., 2019). The accumulation of P in biosolids is dependent on the type of sludge and the treatment system used in WWTP including chemical precipitation, enhanced biological P removal (EBPR), struvite and Ca-P crystallisation, and thermochemical treatment

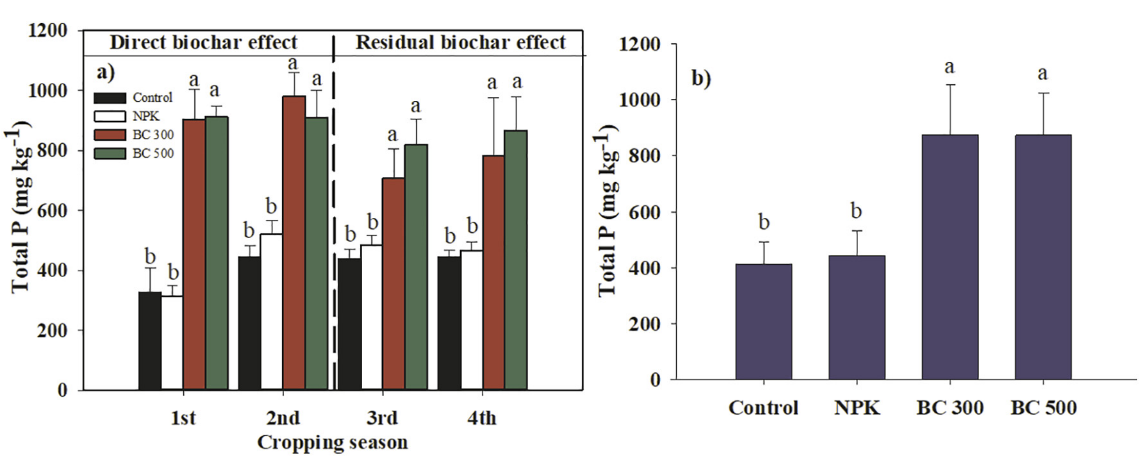

Fig. 1. Total P in soil at the end of each cropping season (a) and average value of total P from four cropping seasons (b). Treatments: control; NPK; BC 300 and BC 500. BC: biochar; 300 and 500 indicate pyrolysis temperature (in °C). Same letters within treatments indicate no difference. Error bars represent the standard deviation ($n = 4$).

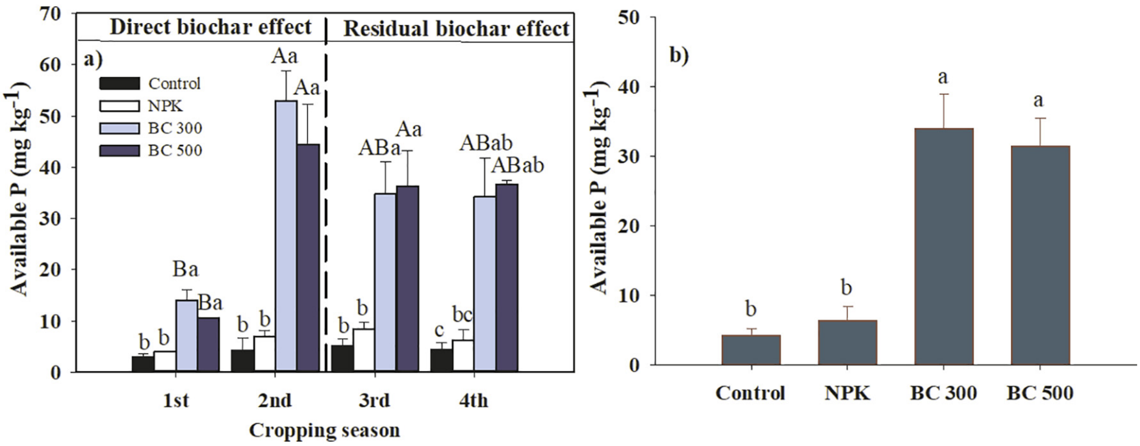

Fig. 2. Available P in soil at the end of each cropping season (a) and average value of available P from four cropping seasons (b). Treatments: control; NPK; BC 300 and BC 500. BC: biochar; 300 and 500 indicate pyrolysis temperature (in °C). Same lowercase letters within treatments and uppercase letters within cropping seasons indicate no difference. Error bars represent the standard deviation ($n = 4$).

(Melia et al., 2017). In the present study, the accumulation of P in biosolids was probably achieved through the addition of salts of Al for P precipitation during sewage treatment. Surprisingly, despite its higher P content, BC 500 did not increase the soil total P content compared to BC 300 ($P > 0.05$; Fig. 1a, b). It is possible that a small portion of BC 500 may have moved to depths below the sampled soil layer. The high levels of total P found in BBC treatments are a result of the high doses applied and the high levels of total P present in BBC, which are further concentrated by pyrolysis due to the loss of other more unstable compounds during the process (Hossain et al., 2011; Kleemann et al., 2017; Figueiredo et al., 2018).

On the other hand, for all cropping seasons evaluated, the application of mineral fertilizer (NPK) did not increase the soil total P content compared to the control (without biochar and without fertilizer NPK) ($P > 0.05$; Fig. 1a, b). This shows that the storage capacity of P in the soil after fertilization was low, even though the doses of mineral fertilizer were higher than the amount uptaken by the plants.

On average, in the two first years biochar application increased soil total P content by approximately 2.4 times in relation to the control (Fig. 1b). In the following two cropping seasons, without additional biochar amendment, treatments with biochars also presented higher total P, but with slightly smaller gain (1.9 times the control value). This result showed that the BBC amendment is able to maintain high soil total P levels for at least two years after concluding its application.

The current work clearly demonstrated the increase in the total P content promoted by the addition of biochar to the soil, using the control treatment as a baseline. However, in the present study it was not possible to differentiate accurately between the P released from biochar and P present in soil. Further studies, adopting specific analytical methodology, would be necessary to understand how phosphorus is released from biochar to soil.

3.2. Soil available P

The soil available P contents followed the same pattern as for total P. Again, both biochars, regardless of pyrolysis temperature, increased the levels of soil available P, with values higher than the control in all cropping seasons and higher than NPK in the three first years (Fig. 2a, b). In all cropping seasons both biochar treatments had similar available P contents in soil, showing that pyrolysis temperature had no effect on available P neither under direct application nor in the BBC residual phase.

Only the biochar treatments were influenced by cropping seasons ($P < 0.05$). In the BC 300 treatment the second season showed higher available P than the first year (Fig. 2a). No differences in available P were found between the other cropping seasons in BC 300 ($P > 0.05$). In the second and third cropping seasons BC 500 presented higher available P than in the first year. At the end of the fourth year BC 500 showed an available P content similar to the other treatments.

Even after P uptake by the corn harvest during the two first cropping seasons, BBC applications released around 89 kg ha^{-1} of available P more than the available P from the control. In the biochar treatments of the current study around 4% of total P was in forms available (extracted by Mehlich 1 solution) to plants. This proportion is considered average when compared to 0.04% obtained by Rehman et al. (2018) and 10% in the study of Bridle and Pritchard (2004). These results also demonstrate that there is great variability in soil P availability among several studies which evaluated BBC addition. This may be justified by differences in the origin of the biosolids (Torri et al., 2017). It may also be due to intrinsic factors of the pyrolysis process such as temperature, residence time and heating rate (Adhikari et al., 2019). Furthermore, differences between studies are mainly due to the P from BBC-mineral fraction interactions of different soil types (Wollmann et al., 2018).

Even after the stopping biochar application, the amount of available P from both BC 300 and BC 500 was sufficient to supply the standard nutritional requirements for corn (Sousa and Lobato, 2004; Faria et al., 2018) for at least four consecutive harvests, eliminating the need to apply phosphate fertilizers for maintenance.

Our results indicate that BBC acts as a slow-release fertilizer able to provide available P over time. This is a desirable feature for fertilizers that are used in soils with high P adsorption capacity. As mentioned earlier, contrary to our expectations, the available P level from BBC in soil along four years was unaffected by the different pyrolysis temperature. As described in previous studies, BBC produced at higher temperature has a significant loss of labile P fractions (Li et al., 2019), due to the formation of more stable and insoluble forms of P (Adhikari et al., 2019). This characteristic was also observed for other feedstocks. Xu et al. (2016) obtained higher labile and moderately labile P contents of plant residue biochars at temperatures between 300 °C and 500 °C, and lower contents at higher pyrolysis temperatures (>500 °C). In P fractionation studies, some authors have observed that most BBC-P is associated with Ca (Bridle and Pritchard, 2004; Schneider and Haderlein, 2016; Buss et al., 2018), which is more available compared to other types of phosphates such as those bound to Fe and Al (Cross and Schlesinger, 1995). This demonstrates that further studies should be conducted to better elucidate the relationship between P compounds in biochar and the soil available P content.

Biochar addition resulted in higher soil available P compared to the control soil in both phases (under direct and residual effects). On average, in the first cropping seasons, BBC application increased the

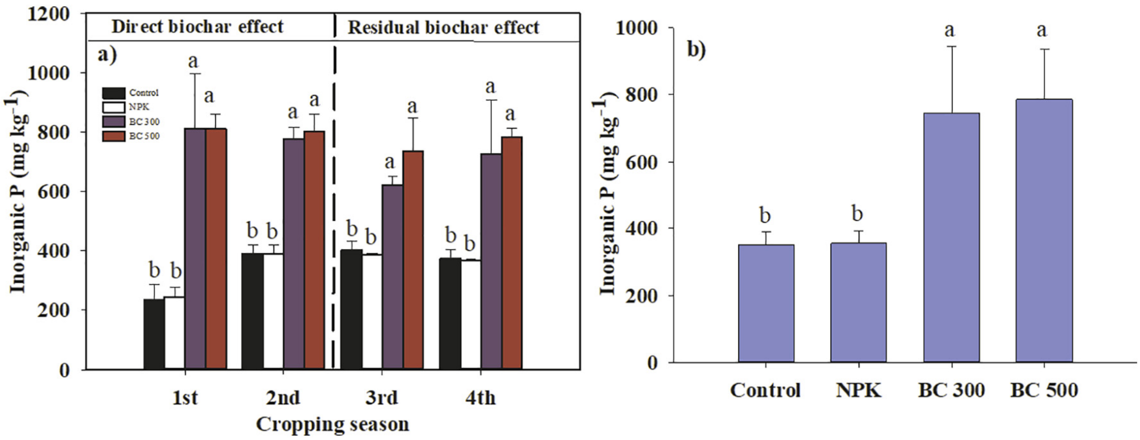

Fig. 3. Inorganic P in soil at the end of each cropping season (a) and average value of inorganic P from four cropping seasons (b). Treatments: control; NPK; BC 300 and BC 500. BC: biochar; 300 and 500 indicate pyrolysis temperature (in °C). Same letters within treatments indicate no difference. Error bars represent the standard deviation ($n = 4$).

available P content by around 8.5 times in relation to the control. Even in the two last cropping seasons BBC maintained available P in soil at around 7.4 times higher than the control treatment. Results of the current study reinforce that BBC is able to maintain available forms of P even under residual effects in a soil with high P-adsorption capacity (Zancanaro de Oliveira et al., 2020).

3.3. Soil inorganic P

Similar to total P and available P, BBC application, regardless of pyrolysis temperature, also increased the soil inorganic P content, compared to the control and NPK treatments in all cropping seasons (Fig. 3a). Inorganic P was unaffected by the cropping seasons ($P > 0.05$). Considering the four cropping seasons, inorganic P composed around 88% and 81% of total P on average in the treatments with and without biochar, respectively (Fig. 3b). This proportion reached 98% in the study by Rehman et al. (2018). This once again reinforces that there is high variability in inorganic P contents in biochars from different studies. The presence of these high inorganic P levels in the soil can be explained by the fact that most of the total biochar P is in the inorganic form (Hossain et al., 2011; Adhikari et al., 2019; Li et al., 2019).

The NPK treatment presented similar P content to the control for all studied P fractions ($P > 0.05$). This was due to higher extraction of P by NPK fertilized plants, since the samples were collected after corn harvesting, which was confirmed by the higher grain yield obtained in the NPK application compared to the control, as will be discussed below.

In the two first cropping seasons biochars increased the inorganic content by a factor of 2.5 on average compared to the control (Fig. 3b). This factor was 1.9 in the biochar residual phase (3rd and 4th cropping seasons), representing the highest reduction among the P fractions. Inorganic P was probably the main source responsible to supply available P to soil during the cropping seasons.

3.4. Soil organic P

Organic P ranged from 96 mg kg^{-1} to 190 mg kg^{-1} among the biochar treatments and from 52 mg kg^{-1} to 82 mg kg^{-1} in those without biochar (Fig. 4a). Farrell et al. (2014) also noticed high variation in this fraction with values ranging from 113 to 438 mg kg^{-1} in an Australian soil amended with biochar produced from domestic green waste at 550 °C. This may have been the result of the aging process, which controls the biochar P changes in the soil (Li et al., 2019). There was no significant effect of the cropping season on organic P ($P > 0.05$). Only in the second cropping season, there was effect of the treatment on organic P. In this season, BC 300 increased organic P compared to the control and NPK ($P < 0.05$). There were no differences between the remaining treatments. Therefore, biochars produced at different temperature promoted similar organic P contents in all assessed cropping seasons.

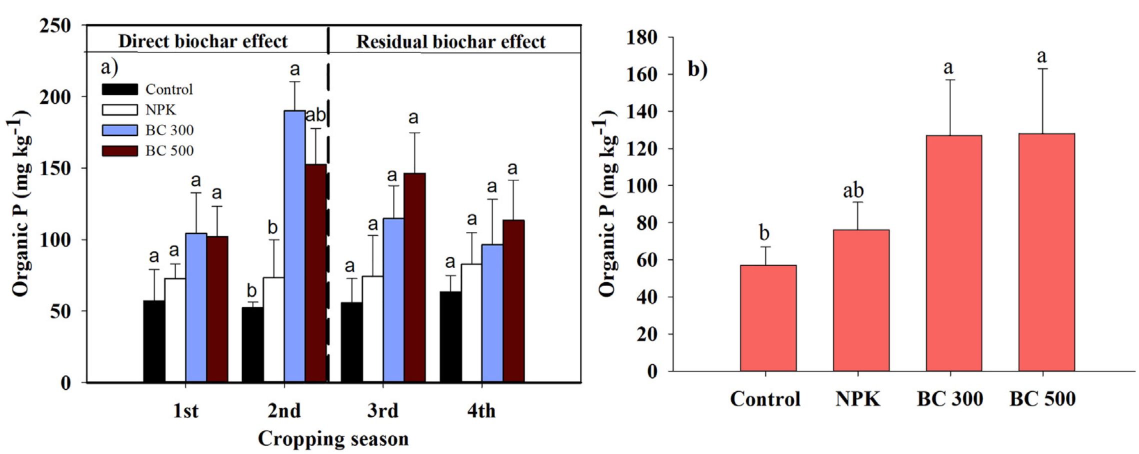

Fig. 4. Organic P in soil at the end of each cropping season (a) and average value of organic P from four cropping seasons (b). Treatments: control; NPK; BC 300 and BC 500. BC: biochar; 300 and 500 indicate pyrolysis temperature (in °C). Same letters within treatments indicate no difference. Error bars represent the standard deviation ($n = 4$).

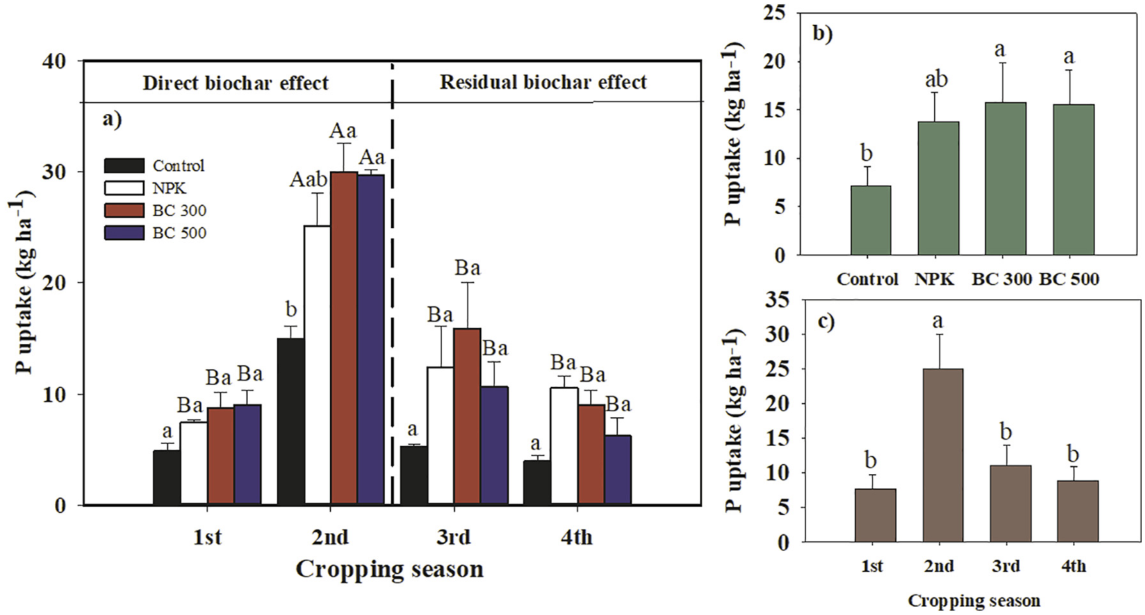

Fig. 5. P uptake by corn in each cropping season (a), average value of P uptake by treatment (b) and by cropping seasons (c). Treatments: control; NPK; BC 300 and BC 500. BC: biochar; 300 and 500 indicate pyrolysis temperature (in °C). Same lowercase letters within treatments (panels a and b) and within cropping seasons (panel c), and uppercase letters within cropping seasons (panel a) indicate no difference. Error bars represent the standard deviation ($n = 4$).

Considering the average content, both biochars promoted higher organic P than the control ($P < 0.05$). The NPK treatment was similar to both the control and biochars (Fig. 4b). Previous studies showed losses of organic P during pyrolysis or transformation of organic P into inorganic forms at temperatures equal or higher than 500 °C (Zhou et al., 2003). At lower pyrolysis temperatures the carboxyl and carbonyl groups would react with Ca and Mg, inhibiting a large-scale formation of insoluble P forms (Adhikari et al., 2019). Similarly, Xu et al. (2016) noticed transformation of organic P into inorganic or more stable organic fractions during the pyrolysis process of wheat straw, maize and peanut husk residues, revealing a decrease in this fraction when the temperature was increased. This transformation indicates that at higher pyrolysis temperatures more stable inorganic P compounds are formed (Li et al., 2019). Longer term studies are needed to assess the changes of biochar P in soil along successive cropping seasons.

3.5. P uptake and corn grain yield

Fig. 5 shows the results of the interaction effects (treatment x cropping season) in each year evaluated (Fig. 5a), as well as the single effects of treatment (Fig. 5b) and cropping season (Fig. 5c) on P uptake. In the second cropping season, BBC and NPK treatments promoted similar P uptake by corn, an uptake which was significantly higher than the control (Fig. 5a). In the remaining cropping seasons, P uptake was not affected by treatments ($P > 0.05$).

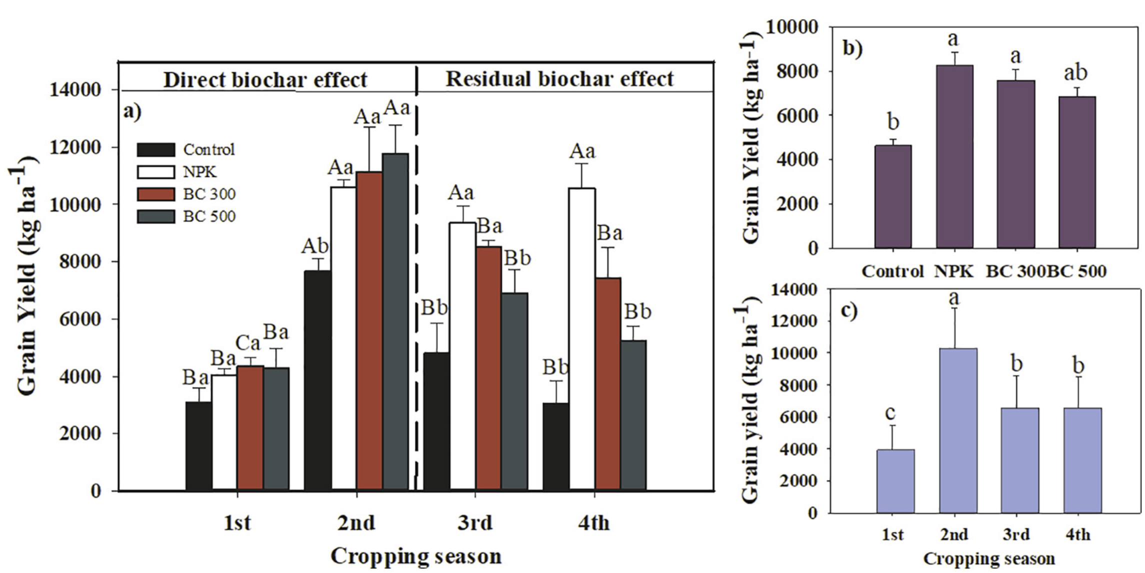

Fig. 6. Corn grain yield in each cropping season (a), average value of grain yield by treatment (b) and by cropping seasons (c). Treatments: control; NPK; BC 300 and BC 500. BC: biochar; 300 and 500 indicate pyrolysis temperature (in °C). Same lowercase letters within treatments (panel a and b) and within cropping seasons (panel c), and uppercase letters within cropping seasons (panel a) indicate no difference. Error bars represent the standard deviation ($n = 4$).

When taking into account the average P uptake from the four cropping seasons, biochar treatments presented higher values than the control (Fig. 5b), with both BC 300 and BC 500 exhibiting similar P uptake by corn. In relation to the control, biochars increased the P uptake by 190% and 223% in the phases of direct application and in the two following crops, respectively (Fig. 5a).

The P uptake for all cropping seasons with application of the biochars was found to be adequate for corn cultivation (Martinez et al., 1999). In general, the biochar treatments, regardless of the pyrolysis temperature, were able to provide sufficient soil P levels to maintain adequate P uptake by corn, even in the residual phase.

The highest P uptake was observed in the second year (Fig. 5c). This can be explained as a result of: i) higher available P content in soil, mainly as a consequence of a second amendment with biochar (Fig. 2a); ii) the second cropping season had the highest rainfall in January (Fig. S1). This period is considered crucial to explain corn productivity in the Cerrado region (Silva et al., 2014). During the second cropping season there were >250 mm in January of 2016, while for the remaining years this figure was below 170 mm. Less rainfall negatively impacted crop productivity, with a concomitant diminishing of plant P uptake.

The effects of treatment and cropping seasons on grain yield are shown in Fig. 6. Fig. 6a shows the results of treatment and cropping season. The effects of treatment (Fig. 6b) and of cropping season (Fig. 6c) on grain yield are also presented. In the first cropping season grain yield was unaffected by treatment ($P > 0.05$). In the second cropping season, BBC and NPK amendments increased grain yield compared to the control (Fig. 6a). These results confirm the possibility of replacing NPK fertilizers with BBC to increase the yields of several crops (Khan et al., 2013; Faria et al., 2018). These studies justified the increase in yields as a consequence of higher nutrient availability and highlighted P as the main factor.

During the residual phase (3rd and 4th cropping seasons) biochars showed a contrasting behavior. In this phase, BC 300 resulted in grain yields similar to NPK and higher than BC 500 (Fig. 6a). Crop yield for BC 500 did not differ from the control during the 3rd and 4th cropping season, demonstrating a slight exhaustion of its effect, probably due to the low availability of other nutrients such as N and K, since soil available P levels remained high in the both biochar treatments. Furthermore, BC 500 maintained P uptake levels similar to NPK in all cropping seasons (Fig. 6a). As previously mentioned, both biochars resulted in similar soil P contents (total and fractions) in the last two cropping seasons. Therefore, it can be concluded that other properties of BC 300, not related to P, were responsible for its better performance during these years. In fact, biosolids biochars produced at lower temperatures (around 300 °C) usually result in higher available N (Figueiredo et al., 2018; Glaser and Lehr, 2019).

Considering the average yields for the four cropping seasons (Fig. 6b), BC 300 promoted grain yields 64% 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 (Jeffery et al., 2017).

Table 4
Pearson correlation coefficients between soil P fractions, P uptake and corn grain yield under biochar treatments.

Variables	Total P	Organic P	Inorganic P	Available P	P uptake	Yield[a]
Total P		−0.03	0.94*	0.26	0.32	0.13
Organic P	−0.03		−0.16	−0.01	0.12	0.15
Inorganic P	0.94*	−0.16		0.25	0.33	0.11
Available P	0.26	−0.01	0.25		0.54*	0.62*
P uptake	0.32	0.12	0.33	0.54*		0.83*
Yield	0.13	0.15	0.11	0.62*	0.83*	

[a] Corn grain yield.
* Correlation is significant at 5% level.

Among the four cropping seasons (Fig. 6c), corn yield was the highest in the second year. As mentioned previously for P uptake, besides the reapplication of BBC in this year, the higher total rainfall obtained in January 2016 and the dry spell observed in January during the other years (Fig. S1) explain the difference in yields between the cropping seasons. In the Cerrado region, the dry spell on January is the main cause of decline in yield, as this is an important vegetative phase of the crop cycle (Silva et al., 2014).

3.6. Relationships between soil P fractions, P uptake and corn grain yield

The correlation values between soil P fractions, P uptake and corn grain yield are shown considering all treatments (Table 3), only biochars (Table 4), and only non-biochar treatments (Table 5). When all treatments were assessed together a higher number of correlations were statistically significant ($P < 0.05$). On the other hand, the treatments including biochar had a lower number of significant correlations. The highest correlation values were obtained between total and inorganic P in the three groups assessed (Tables 3, 4 and 5). Organic P showed significant correlation with the other P fractions only when all treatments were compared. Furthermore, organic P had the lowest correlation with the other P fractions and was not correlated with either uptake P or yield in any of the evaluated groups. The high dispersion of organic P data (Fig. S3) and its microorganism-dependence to be mineralized in the soil (Li et al., 2019) may explain the lack of correlation between this fraction and the other variables.

When only the biochar treatments were evaluated (Table 4), among P fractions only the total P and inorganic P showed significant correlation ($P < 0.05$). As previously discussed, inorganic P made up over 80% of total P of biochar-amended soils ($P < 0.05$). This may explain the high correlation between the soil P pools. In addition, the low dispersion of data (Fig. S3) reinforces the strong relation between those two soil P fractions.

In all groups evaluated, available P showed a positive correlation ($P < 0.05$) with both P uptake and grain yield. When only biochars were considered, available P was the only P fraction that showed significant correlation with P uptake and grain yield. As discussed previously, the high availability of P in biochar-amended soil, in all cropping

Table 3
Pearson correlation coefficients between soil P fractions, P uptake and corn grain yield under all treatments.

Variables	Total P	Organic P	Inorganic P	Available P	P uptake	Yield[a]
Total P		0.31*	0.91*	0.68*	0.34*	0.20
Organic P	0.31*		0.34*	0.37*	0.20	0.18
Inorganic P	0.91*	0.34*		0.72*	0.35*	0.19
Available P	0.68*	0.37*	0.72*		0.49*	0.40*
P uptake	0.34*	0.20	0.35*	0.49*		0.69*
Yield	0.20	0.18	0.19	0.40*	0.69*	

[a] Corn grain yield.
* Correlation is significant at 5% level.

Table 5
Pearson correlation coefficients between soil P fractions, P uptake and corn grain yield under the control and NPK treatments.

Variables	Total P	Organic P	Inorganic P	Available P	P uptake	Yield[a]
Total P		0.06	0.80*	0.42*	0.05	0.40*
Organic P	0.06		−0.25	0.02	0.15	0.09
Inorganic P	0.80*	−0.25		0.52*	0.17	0.43*
Available P	0.42*	0.02	0.52*		0.39*	0.60*
P uptake	0.05	0.15	0.17	0.39*		0.54*
Yield	0.40*	0.09	0.43*	0.60*	0.54*	

[a] Corn grain yield.
* Correlation is significant at 5% level.

seasons, and its positive correlation with P uptake and grain yield showed that performance of biochar as a source of P depends on its ability to supply available P along the years. It should be highlighted that the increase in available P is not the only direct beneficial effect of biochar application when considering the dynamics of P in a soil-plant-biochar system. Indirect effects such as increased enzyme activity and rhizosphere low-molecular-weight organic acids concentration are also important variables (Bornø et al., 2018).

Table S2 shows the results of the multiple linear regression analysis performed according to stepwise criterium for P fractions. Stepwise regression analysis is a step by step approach where insignificant variables are removed from the regression analysis allowing only important variables to be present in the prediction models (Nazif et al., 2016). In the present study, stepwise procedure was applied to select one or a group of P fractions that could predict the grain yield. Two groups of samples were used: all treatments and only treatments with biochar addition. In both groups the only significant models ($P < 0.001$) included available P as a variable capable of predicting corn yield. Both total P and inorganic P showed variance inflation factor (VIF) above five indicating a high degree of collinearity between them and with other predictors (Fox et al., 2016). As discussed previously, in the current study, inorganic P made up >80% of soil total P. This may have been the cause of the high VIF values found for these fractions.

Even when biochars were assessed separately, available P was also considered the best predictor. In this case, the adjusted R^2 of the biochar model (0.4) was two times higher than that with all treatments together (0.2). This means that with biochar treatments the available P was able to explain 40% of yield variability. Therefore, available P can be used to predict corn grain yield, mainly in BBC-amended soils. The relationship between available P and grain yield is already well known in literature for many P sources applied to soils (Fixen and Grove, 1990). Nevertheless, in the current study, a preliminary model [Yield (kg ha^{-1}) $= 4009.7 + 104.2 *$ available P] is presented for the first time which shows the relationship between available P and corn grain yield using data from a field experiment with a sequence of four consecutive croppings in a BBC-amended soil. Further studies using external data are needed to validate these models.

4. Conclusions

The results of this study confirmed the hypothesis that BBC increases the total P and the organic, inorganic and available P fractions, maintaining these high values in the soil for at least two years after stopping its application. In the residual phase, the annual application of mineral fertilization (NPK) promoted corn yields similar to or higher than biochar application. However, compared to the control, mineral fertilization was not able to increase the soil P reserve in any of its fractions, demonstrating that reapplication of NPK is needed in each cropping season. Surprisingly, contrary to our second hypothesis, in the current study the pyrolysis temperature had no effect on soil P fractions throughout the four cropping seasons. Nevertheless, BC 300 stood out and promoted higher yield in the residual period (7396 kg ha^{-1} and 7416 kg ha^{-1} in the 3rd and 4th cropping season, respectively) than BC 500 (6126 kg ha^{-1} and 5236 kg ha^{-1} in the 3rd and 4th cropping season, respectively). BC 300 was also able to replace the mineral fertilization in all evaluated cropping seasons. BBC applied at the dose of 15 t ha^{-1} was able to maintain high content of all soil P fractions for at least two cropping seasons after stopping its application. In a BBC-amended soil, available P was the only fraction capable of predicting corn yield according to the following preliminary model [Yield (kg ha^{-1}) $=$ $4009.7 + 104.2 *$ available P]. Additional long-term studies should be focused on BBC as a slow-release phosphate fertilizer. Layers below 20 cm must also be considered for better understanding of the long-term P dynamics in soils.

CRediT authorship contribution statement

Cícero Célio de Figueiredo: Conceptualization, Writing - original draft, Formal analysis, Writing - review & editing. **Thamires Dutra Pinheiro:** Formal analysis, Methodology. **Luiz Eduardo Zacanaro de Oliveira:** Formal analysis, Methodology. **Alyson Silva de Araujo:** Formal analysis. **Thais Rodrigues Coser:** Methodology, Validation, Formal analysis. **Jorge Paz-Ferreiro:** Writing - review & editing, Visualization.

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.

Acknowledgements

This research was funded by Fundação de Apoio à Pesquisa do Distrito Federal, Brazil - grant number 00193.00002099/2018-65.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2020.140013.

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