Co-application of wood biochar and paper mill biosolids affects yield and short-term nitrogen and phosphorus availability in temperate loamy soils1

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ARTICLE

Co-application of wood biochar and paper mill biosolids affects yield and short-term nitrogen and phosphorus availability in temperate loamy soils[1]

Bernard Gagnon, Noura Ziadi, and Eric Manirakiza

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Abstract: Amending croplands with forest residues may help in restoring soil properties in fields subject to intensive land management. Despite their known benefits when applied separately, co-application of wood biochar with paper mill biosolids (PB) has seen little investigation under field conditions. A study was initiated in Québec, QC, Canada, to determine the effect of a single application of wood biochar with and without PB on the nitrogen (N) and phosphorus (P) availability of two pH-neutral to alkaline loamy soils. Biochar at 0, 10, and 20 Mg dry weight·ha^{-1} and PB at 30 Mg wet weight·ha^{-1} were applied before planting of corn (*Zea mays* L.) and soybean [*Glycine max* (L.) Merr.] in 2018. Residual effect of this co-application was determined under soybean and corn in the subsequent year. In both years, corn received supplemental N and P from mineral fertilizers according to local agronomic recommendations. Co-applying biochar and PB reduced soil NO_3-N availability in the year of application and decreased corn yield by 1.0 Mg·ha^{-1} compared with biochar or PB applied alone, but these amendments did not affect soybean yields. In the following year, the previous biochar addition increased soybean yield by 0.6 Mg·ha^{-1} but had little effect on corn. For both years, biochar addition induced a large increase in soil Mehlich-3 P. This study revealed that wood biochar positively impacted P status of these soils but was not a source of N to crops even when co-applied with PB.

Key words: grain corn, N availability, paper mill biosolids, P availability, soybean, wood biochar.

Résumé : Amender les terres agricoles avec des résidus forestiers pourrait contribuer à restaurer certaines propriétés des sols exploités de façon intensive. Malgré les avantages qu'on leur connaît quand ils sont appliqués séparément, on s'est relativement peu intéressé aux effets du biocharbon épandu au champ avec des biosolides papetiers (BP). Les auteurs ont entrepris une étude à Québec (Québec, Canada) en vue de préciser les effets d'une application unique de biocharbon de bois avec ou sans BP sur la disponibilité de l'azote (N) et du phosphore (P) dans deux sols loameux dont le pH variait de neutre à alcalin. Dans cette optique, en 2018, ils ont épandu 0, 10 ou 20 Mg (poids sec) de biocharbon par hectare et 30 Mg (poids humide) de BP par hectare au sol avant de semer du maïs (*Zea mays* L.) et du soja [*Glycine max* (L.) Merr.], puis ont déterminé les effets résiduels sur chaque culture, l'année subséquente. Pour les deux années, pour le maïs, les auteurs ont appliqué une quantité supplémentaire de N et de P sous forme d'engrais minéral, conformément aux recommandations locales. L'application de biocharbon et de BP a réduit la disponibilité du N-NO_3 dans le sol l'année même d'application et a diminué le rendement du maïs de 1,0 Mg par hectare, comparativement à l'application de l'un ou l'autre des amendements seul. Cependant, aucun effet n'a été observé sur le rendement du soja. L'année suivante, l'addition de biocharbon un an plus tôt a accru le rendement du soja de 0,6 Mg par hectare, mais a eu peu d'effet sur le maïs. Pour les deux années, l'addition de biocharbon a entraîné une hausse importante de la concentration en P Mehlich-3 dans le sol. Les résultats indiquent que le biocharbon de bois a une

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incidence positive sur le bilan du P dans les sols examinés, mais ne procure pas de N aux cultures, même lorsqu'il est appliqué avec des BP. [Traduit par la Rédaction]

Mots-clés : maïs-grain, N disponible, biosolides papetiers, P disponible, soja, biocharbon de bois.

Introduction

A large amount of residues such as treated wastewater biosolids from paper and pulp mills (1.5×10^6 dry Mg) are produced annually by the Canadian forest industry (Pervaiz and Sain 2015). More recently, a biochar production sector has been gradually emerging, utilizing feedstock of little economic value such as wood chips, forest clear cutting, and insect-infested trees (Matovic 2011). Agricultural use of forest residues would benefit soil properties, particularly when the rotation consists of solely of corn and soybean crops, which are conducive to less healthy soil (Karlen et al. 2006; Wade et al. 2020). In addition, this offers an opportunity to recycle and manage soil nutrients in a circular economy efficiently (Camberato et al. 2006; Gao and DeLuca 2020).

Biochar, a carbon (C)-rich and recalcitrant solid material produced through the thermochemical conversion of biomass (Lehmann and Joseph 2015), is viewed as a way to mitigate greenhouse gas emissions and sequester C in the soil (Lehmann et al. 2006; Galinato et al. 2011). It also has the capacity to enhance soil fertility and increase crop production (Jeffery et al. 2011; Liu et al. 2013). However, results are inconsistent under temperate conditions, with negative (Gaskin et al. 2010; Nelissen et al. 2015; Haider et al. 2017), neutral (Jones et al. 2012; Borchard et al. 2014; Tammeorg et al. 2014; Soinne et al. 2020), or positive (Hammond et al. 2013; Backer et al. 2016; Laird et al. 2017) effects of wood biochar on crop yields, with response depending on crop, soil, and biochar type (Rajkovich et al. 2012). Globally, modest yield increases (<3%) were reported in temperate fields due to inherently good productivity (Jeffery et al. 2017), with rates of 20 $Mg \cdot ha^{-1}$ or less usually bringing the most benefits (Rajkovich et al. 2012; Hammond et al. 2013). Use of wood biochar in temperate climates is unfortunately minimal at present time due to variable crop results, high market price, and weak incentive policy, which do not motivate farmers to invest in biochars beyond their role in climate change mitigation (Galinato et al. 2011; Soinne et al. 2020).

Much work is being done these days to combine the use of biochar with organic fertilizers to improve crop response. This is because of the direct supply of nutrients and their retention, stabilization of soil organic matter, and increase in water-holding capacity (Liu et al. 2012; Agegnehu et al. 2017). Moreover, co-amendment is particularly recommended with wood biochar because this material applied alone tends to reduce microbial abundance and enzyme activities in coarse-textured soils (Gul et al. 2015). Considering their attributes, combined paper mill biosolids (PB) — a mixture of treated wastewater primary and secondary sludge — could be valuable material because they are widely available and are a good source of organic N and P as well as organic matter (Camberato et al. 2006).

Co-application of biochar and biosolids has been the subject of few studies regarding soil N and P availability under temperate climates. Knowles et al. (2011) found that the co-application of sewage biosolids and pine (*Pinus* spp.) biochar increased the retention of NO_3-N, which can benefit the agroecosystem by reducing the NO_3-N leaching, but they decreased pasture growth. Manirakiza et al. (2019) also reported a reduction in soil N availability in an incubation study using pine biochar co-applied with PB. This decrease could be attributed to inorganic N adsorption on biochar (Shaaban et al. 2018; Manirakiza et al. 2019) and also to volatile matter content and the C/N ratio of material (Deenik et al. 2010; Gao and DeLuca 2016; Nguyen et al. 2017). Nonetheless, Lu et al. (2020) observed an increase in total annual dry matter (DM) grass accumulation when pinewood biochar was added to a municipal biosolid rich in inorganic N. Lentz et al. (2014) and Ippolito et al. (2016) concluded that combining hardwood biochar with dairy manure utilized N more effectively, as it eliminated potential yield reduction caused by biochar and maximized manure net N mineralization potential.

Plant N and P nutrition following biochar and organic amendments can be assessed using in situ crop diagnosis in complement to soil analysis. The concept behind this is that plant nutrient availability is not simply determined by soil parameters, but it is also highly dependent upon many other local environmental variables that determine the plant growth rate and the root adsorption capacity (Lemaire et al. 2021). Crop nutrition diagnosis is performed on whole plants during the vegetative phase until flowering. It has proven its effectiveness for both plant N and P status in the temperate conditions of eastern Canada for various crops, including corn (Ziadi et al. 2008; Gagnon et al. 2020), and it could address variations in yield induced by differences in crop N and P availability (Lemaire and Meynard 1997). An N nutrition index (NNI) or P nutrition index (PNI) of approximately 1.0 indicates well-balanced plant nutrition, whereas lower values indicate an N or P deficiency (Gastal et al. 2015).

The objective of this study was, therefore, to determine and monitor, during two growing seasons, the effect of a single application of wood biochar with and without PB on soil N and P availability for corn and soybean grown in two pH-neutral to alkaline loamy soils under temperate climatic conditions. The hypothesis was that co-application of biochar and PB could enhance crop yield by retaining inorganic N and P once available and improving their supply to plants.

Materials and Methods

Material production

The biochar consisted of forest biomass (bark and wood) collected from various sources (harvest clear cutting, timber mill, and reject wood processing) that was subjected to carbonization at high temperature (900–950 °C) in a steam-powered wood boiler (Phénix, Boralex Énergie S.E.C., Senneterre, QC, Canada). Along with fast and slow pyrolysis, this process can be used to produce biochars (Spokas et al. 2011). Briefly, the combustion process is continuous, with feedstock entering on a mobile grate at controlled speed (6–7 $m \cdot h^{-1}$) and staying there for approximately 60 min. The oxygen required for the combustion is injected under the grate and passes through it to supply the combustion chamber. Fly ash as it is produced is carried along by this upward air movement and captured using a multicyclone and an electrostatic precipitator to form the biochar. The remaining material on the grate after combustion is evacuated by a conveyor.

The other material used (PB) consisted of combined primary and secondary de-inking sludge from treated paper-recycling wastewater (Les Entreprises Rolland Inc., Lévis, QC, Canada).

Material analysis

Composite samples of each material were analyzed for their properties. To determine the pH of the PB, 5.0 g of fresh material was placed in 20 mL of distilled water, which was agitated for 30 min, left to stand for 30 min, and then measured using a glass electrode. For biochar, 1.0 g of fresh material was placed in 20 mL of distilled water, which was shaken for 1.5 h, then centrifuged for 15 min at 15 000 $r \cdot min^{-1}$, and filtered through a grade 410 filter paper (Rajkovich et al. 2012). Moisture content was determined after drying the materials at 55 °C to constant weight. Potential cation-exchange capacity (CEC) was determined by saturating 1.0 g dry material with 1 $mol \cdot L^{-1}$ ammonium acetate pH 7.0 and then replacing by the addition of 2 $mol \cdot L^{-1}$ KCl, as described by Rajkovich et al. (2012).

Major total nutrients (N, P, and K) were determined on 0.25–0.30 g fresh weight for PB and 0.16 g fresh weight for biochar by wet acid digestion in presence of H_2SO_4–H_2SeO_3 (Isaac and Johnson 1976). Concentrations of N and P in acid extracts were measured by colorimetry using a continuous-flow injection auto-analyzer (QuickChem 8000 FIA+ analyzer, Lachat Instruments, Loveland, CO, USA) with the salicylate–nitroprusside procedure for total N and the vanadomolybdate reaction for total P. The K concentrations were determined using an inductively coupled plasma optical emission spectrometer (ICP-OES, Perkin Elmer Optima 4300DV, Shelton, CT, USA).

The 2 $mol \cdot L^{-1}$ KCl-extractable NO_3-N and NH_4-N contents were obtained by shaking a solution of 1/4 PB or 1/20 biochar (*w*/*w*, fresh weight) for 1.5 h, followed by centrifugation (15 min, 15 000 $r \cdot min^{-1}$) and filtration. The concentrations of NO_3-N and NH_4-N in the extracts were measured using the auto-analyzer with the Cd–Cu reduction procedure for NO_3-N and the salicylate–nitroprusside procedure for NH_4-N.

Total C was determined on 0.20 mm finely ground samples by dry combustion on a Vario Macro CN (Elementar, Hanau, Germany) for PB and a LECO TruSpec Micro (LECO Corp., St. Joseph, MI, USA) for biochar. Material ground samples were also treated with HCl to eliminate carbonates and determine organic C. The proximate analysis was performed on dry samples (<1 mm) of biochar using a TGA701 (LECO Corp.) to assess the contents in volatile matter, fixed C, and ash (ASTM 2015). Biochar samples were also analyzed for their specific surface area using Brunauer–Emmett–Teller (BET)-N_2 multilayer adsorption isotherms at 77K (−196 °C) collected on a Micromeritics ASAP 2020.

Site description

A rain-fed field trial was conducted during two growing seasons (2018 and 2019) at the St-Augustin-de-Desmaures Research Farm of Agriculture and Agri-Food Canada near Québec, QC, Canada (46°44′N, 71°31′W) on two adjacent sites located 10 m apart. The field was under conventional tillage with mouldboard ploughing in fall, and the preceding crop was oat (*Avena sativa* L.). The soil, classified as Orthic Humic Gleysol, was a Chaloupe in association with Champlain series developed on a surrounding limestone bedrock, which is prone to compaction when managed moist (Raymond et al. 1976).

The soil (0–15 cm) of each site was sampled ($n = 4$) before applying materials and analyzed for pH (H_2O), total C, NH_4-N + NO_3-N, Mehlich-3-extractable P, K, and Al, and particle size (Table 1). Both sites were of loam texture, imperfectly flat drained, with a pH ranging from neutral to slightly alkaline. They were classified as poor in P and medium in K, according to local soil test guidelines (CRAAQ 2010). Considering the site properties, soil pH would be a less dominant factor here in explaining response to biochar addition (Gao and DeLuca 2020).

Field experiment

The experimental layout was a randomized complete block design with four replicates, and plot size was 3 m × 5 m. Field crops on the two experimental sites consisted of a rotation of grain corn–soybean and soybean–grain corn, respectively.

On 22 May 2018, wood biochar at 0, 10, and 20 Mg dry $weight \cdot ha^{-1}$ was applied to the bare soil surface with and without 30 Mg wet weight $PB \cdot ha^{-1}$. One additional

Table 1. Main properties of studied sites.

Parameter	pH (H_2O)	Total C ($g \cdot kg^{-1}$)	Sand ($g \cdot kg^{-1}$)	Clay ($g \cdot kg^{-1}$)	$NO_3 + NH_4$-N ($mg \cdot kg^{-1}$)	Mehlich-3 P ($mg \cdot kg^{-1}$)	Mehlich-3 K ($mg \cdot kg^{-1}$)	Mehlich-3 Al ($mg \cdot kg^{-1}$)
Site 1	6.9 ± 0.2	10.6 ± 0.9	353 ± 9	162 ± 3	7 ± 0	24 ± 3	85 ± 13	990 ± 26
Site 2	7.7 ± 0.2	12.7 ± 0.4	485 ± 18	148 ± 3	8 ± 0	20 ± 3	67 ± 2	711 ± 168

Note: Mean of four subsamples ± standard deviation.

Table 2. Amount of fertilizer mineral nitrogen (N) and phosphorus (P) applied to corn in each treatment in the 2 yr of field experiment.

	Year of application (2018)		Residual year (2019)	
	$kg\ N \cdot ha^{-1}$	$kg\ P \cdot ha^{-1}$	$kg\ N \cdot ha^{-1}$	$kg\ P \cdot ha^{-1}$
Untreated	0	0.0	0	0.0
NP	150	30.5	150	30.5
Biochar 10 Mg	150	8.7	150	8.7
Biochar 20 Mg	150	0.0	150	0.0
PB	120	19.6	140	19.6
PB + biochar 10 Mg	120	0.0	140	0.0
PB + biochar 20 Mg	120	0.0	140	0.0

Note: NP, mineral NP fertilizer; PB, paper mill biosolids. Values for biochar and PB nutrient contribution were estimated from the company records. According to provincial recommendations (CRAAQ 2010), 150 $kg\ N \cdot ha^{-1}$ and 30.5 $kg\ P \cdot ha^{-1}$ were needed in these sites for grain corn.

treatment receiving no material was designed as mineral NP fertilizer (NP), for a total of seven treatments. Biochar and PB were weighed in small bins, according to the prescribed rate, and manually applied using a rake to an area of 3 m^2 at a time. They were incorporated in the same day at 10 cm depth using a rotary tiller. No material was applied in 2019. The sites were roto-tilled the next spring to prepare the seedbed for the following crops.

Mineral fertilizer N and P were added to corn, except in the unamended control, as Ca-NO_3NH_4 and triple superphosphate, respectively, to ensure adequate nutrient supply (Table 2; CRAAQ 2010). Rates were determined assuming a contribution for PB of 30% of organic N and no N for the biochar (OMAFRA 2012; CRAAQ 2013) and 80% of total P for PB and biochar (CRAAQ 2013), based on company records before application (J. Bégin, personal communication, 2018). The fertilizer N was split, with 50 $kg\ N \cdot ha^{-1}$ being surface broadcast by hand before planting and the rest being band applied at 5 cm depth, 10–15 cm from the plants, at the V7 corn stage. The fertilizer P was all surface broadcast before planting. No fertilizer K was applied. The soybean crop did not receive any fertilization, except in the NP treatment, where 20 $kg\ N \cdot ha^{-1}$ as Ca-NO_3NH_4 was broadcast applied at sowing. The same amounts and procedures were applied for fertilization in the residual year, except fertilizer N was increased to 140 $kg\ N \cdot ha^{-1}$ in the PB-amended plots to take into account material decomposition.

Grain corn ['Elite E49A12R' (2325 corn heat units)] was planted with a 0.76 m inter-row spacing at 88 300 $plants \cdot ha^{-1}$ using a modified mechanical two-row corn planter (Nodet-Gougis) on 24 May 2018 and 23 May 2019. Soybean ['Elite Podaga' (2400 corn heat units)] was sown on the same days with the same planter at a 0.38 m inter-row spacing and 381 000 plants ha^{-1}. To control weeds, glyphosate at 1.67 $L \cdot ha^{-1}$ was applied each year to each crop at the end of June.

Grain yield was determined at maturity by manually harvesting one 4 m long inner row for corn and two 1 m long inner rows for soybean in the middle of each plot. Harvest took place on 15 Oct. 2018 and 4 Nov. 2019 for corn and 1 Oct. 2018 and 3 Oct. 2019 for soybean. Soybean was dried at 55 °C in a forced-draft oven until a constant weight was reached, and then grain and straw were mechanically separated, cleaned, and weighed. Corn ears were dried at 55 °C and mechanically shelled afterwards. Corn stalks were weighed in the field and mechanically chopped, and a subsample was kept for DM determination. Grain yield was adjusted to a moisture content of 155 $g \cdot kg^{-1}$ for corn and 130 $g \cdot kg^{-1}$ for soybean. Specific grain weight ($kg \cdot hL^{-1}$) was determined for corn, and number of grains per kilogram was determined for soybean.

Plant sampling and analysis

Evaluation of in-season plant N and P nutrition status was conducted at corn tasseling (VT stage; Ritchie et al. 1993) and soybean beginning bloom

Table 3. Monthly temperatures and rainfall during the growing seasons of study and the 30 yr average (1981–2010).

Parameters	May	June	July	August	September	October	Mean
Air temperature (°C)							
2018	11.4	15.1	20.8	20.1	14.1	4.1	14.3
2019	9.2	15.1	20.3	17.8	12.7	7.0	13.7
30 yr average	11.2	16.4	19.3	18.1	12.7	6.6	14.1
Rainfall (mm)							Total
2018	56	135	101	101	109	113	614
2019	104	169	47	102	129	132	682
30 yr average	116	111	121	104	116	98	667

Note: Reported data were retrieved from a weather station located <10 km from the experimental site (Environment Canada 2020).

(R1 stage; Fehr and Caviness 1977) each year. To this end, whole plants were cut at ground level using pruning scissors from a 1 m section of a row within each plot and dried at 55 °C in a forced-draft oven until reaching constant weight for DM determination and laboratory analyses.

Samples of plant tissue and grain for corn and soybean were ground to 1 and 0.25 mm, respectively. Subsamples of 0.1 g were wet-acid digested as with biochar and PB (Isaac and Johnson 1976). Concentrations of N and P were measured by colorimetry on the auto-analyzer. Total plant N accumulation for harvest was obtained by adding together the N accumulation of grain and straw calculated by multiplying the DM yield by their respective tissue N concentrations. The same calculation was performed for P.

The in-season NNI was calculated using the equations of critical N of corn validated in eastern Canada (Ziadi et al. 2008) and critical N of soybean (Divito et al. 2016):

$$\mathrm{NNI} = N/(34.0 \times W^{-0.37}) \text{ for corn,}$$

$$\mathrm{NNI} = N/(37.0 \times W^{-0.08}) \text{ for soybean,}$$

where N is the whole plant N concentration in g·kg^{-1} DM and W is the shoot biomass in Mg DM·ha^{-1}.

The in-season PNI was only calculated for corn, using the equation developed in eastern Canada by Gagnon et al. (2020) under nonlimiting N conditions:

$$\mathrm{PNI} = P/(0.82 + 0.097 \times N) \text{ for corn}$$

with P and N as whole plant P and N concentration expressed in g·kg^{-1} DM.

Soil sampling and analysis

Soils were sampled 1 mo after planting (corn only) — i.e., before sidedress N application — at time of NNI measurement and at crop harvest in both years. Soils were also sampled in early May the year after material application. Samples consisted of fives cores (0–15 cm layer) taken at random from each plot with a 2.5 cm diameter hand-held soil probe (JMC Backsaver N-2, Clements Associates Inc., Newton, IA, USA).

A subsample of 2.5 g of field-moist soil was extracted with 20 mL 2 mol·L^{-1} KCl for 30 min on a reciprocal shaker before filtering (Maynard et al. 2008). Both the NO_3-N and NH_4-N concentrations in the soil extracts were quantified with the auto-analyzer using the same procedures as for biochar and PB characterization. Data were reported on a dry-weight basis, taking into account the soil moisture content determined by oven-drying a 20 g subsample at 105 °C for 24 h. An air-dried subsample sieved to <2 mm was extracted by the Mehlich-3 solution (Mehlich 1984), and concentrations in soil-available P were determined by colorimetry (Beckman Coulter DU720, Mississauga, ON, Canada) using the ascorbic acid – molybdate reaction (Murphy and Riley 1962).

Statistical analysis

All data were subjected to a Bartlett's test to check for homogeneity of variances and no transformation was needed. Data analysis was performed using the MIXED procedure of SAS version 12.1 (SAS Institute 2010). Analysis was done by separate site, due to a different rotation sequence, and by year to differentiate year of material application from residual year. Main treatment effects were compared using orthogonal polynomial contrasts for biochar rate and biochar rate × PB and single degree-of-freedom contrasts otherwise (NP vs. untreated, NP vs. PB, NP vs. biochar + PB, and biochar vs. biochar + PB). The contrast for biochar rate used NP as 0 Mg·ha^{-1} in corn due to N addition and untreated control in soybean. Statistical significance was defined as $p \leq 0.05$.

Results and Discussion

Climatic conditions

The summer growing conditions (July–September) in 2018 were warmer than the 1981–2010 average (Table 3). By contrast, mean temperatures in May and June 2019 were cooler (–1.7 °C), which delayed early crop growth. Total rainfall in both years was close to the 1981–2010

Table 4. Main characteristics of the biochar and paper mill biosolids (PB) used in the study (dry matter basis except moisture).

Parameter	Wood biochar	PB
pH (H_2O)	11.7 ± 0.0	7.3 ± 0.2
Moisture ($g{\cdot}kg^{-1}$)	355 ± 3	606 ± 7
CEC ($cmol{\cdot}kg^{-1}$)	58.9 ± 1.9	68.8 ± 3.5
Ash ($g{\cdot}kg^{-1}$)	685 ± 6	—
Volatile matter ($g{\cdot}kg^{-1}$)	205 ± 9	—
Fixed carbon ($g{\cdot}kg^{-1}$)	108 ± 15	—
BET surface area ($m^2{\cdot}g^{-1}$)	87 ± 2	—
Total carbon ($g{\cdot}kg^{-1}$)	198 ± 25	292 ± 76
Organic carbon ($g{\cdot}kg^{-1}$)	152 ± 19	154 ± 3
Total N ($g{\cdot}kg^{-1}$)	0.9 ± 0.1	11.5 ± 0.3
NO_3-N + NH_4-N ($g{\cdot}kg^{-1}$)	0.05 ± 0.00	2.3 ± 0.1
C/N ratio	212 ± 16	28 ± 2
H/C molar ratio	0.35	—
O/C molar ratio	0.41	—
Total P ($g{\cdot}kg^{-1}$)	4.3 ± 0.2	2.3 ± 0.2
Total K ($g{\cdot}kg^{-1}$)	19.3 ± 1.3	0.7 ± 0.0

Note: CEC, cation-exchange capacity; BET, Brunauer–Emmett–Teller. Mean of six subsamples except pH (4) ± standard deviation.

average. However, the rain received in 2019 was more variable across the season, and only 40% of the regional average was received in July.

Biochar properties

The assessed wood biochar was alkaline with a high volatile matter content (>20%) and a low fixed C (Table 4), meaning that it was more easily degraded in soil once land applied (Zimmerman 2010). This material also possessed a moderate C stability, owning to its O/C ratio ≤0.4 and volatile matter/fixed C <3.0 (Spokas 2010; Klasson 2017). Its BET surface area, close to 100 $m^2{\cdot}g^{-1}$, should positively influence soil biota and promote nutrient retention (Atkinson et al. 2010; Schimmelpfennig and Glaser 2012). Compared with other wood-based biochars (Ippolito et al. 2020), this material was denser (450 $kg{\cdot}m^{-3}$) and richer in ash. Nevertheless, its H/C molar ratio <0.7 was indicative of thermochemical conversion producing fused aromatic ring structures (Klasson 2017). The biochar had a fine particle size distribution with 60% <100 mesh (0.150 mm).

In-season crop N and P nutritional status

The NNI and PNI measured at corn tasseling or soybean beginning bloom give a direct indication of the plant nutrition status at this period of the season as related to amendment addition. In this study, this approach was used in complement to soil analysis for evaluating the performance of each cropping system influenced by the local environment-management conditions and to relate the indices to the yield and quality of crop (Lemaire et al. 2021).

The NNI of corn plants was steadily increased by all fertilized treatments, compared with the untreated control in both years ($p < 0.001$; Tables 5 and 6). Except for lower values in the residual year, likely attributable to poorer early-season growth conditions (Table 3), the NNI was close to 1.0, meaning balanced N nutrition (Gastal et al. 2015). Overall, NNI was unaffected by material application, but a trend ($p = 0.09$) was observed in the year of application for lower values in biochar with PB co-applied compared with NP (0.87 vs. 0.96; Table 5). For both years, the NNI of corn was closely related to grain yield ($r^2 > 0.96$). This means that biochar with N supplementation adequately met the corn N requirements in this soil.

Conversely, the NNI of soybean plants was not affected by any treatments, including the control, in both years, and all values were around 1.0 (Tables 7 and 8). This is expected, since soybean derives between 50% and 60% of its total N from biological N_2 fixation and is more dependent on soil conditions (pH, moisture) than N fertilization (Salvagiotti et al. 2008).

The PNI of corn plants was not affected by treatments in any of the years except for the untreated control, where the plants accumulated P in their tissues in absence of N supply (Lemaire et al. 2019; Tables 5 and 6). Values of corn PNI ranged between 0.92 and 1.08, which indicated a good P nutrition. For soybean, no research has yet been done to develop a PNI, so tissue P at time of NNI sampling was used as indicator of P nutritional status. The soybean plant P was unaffected by treatments in the year of material application but was increased by PB in residual year (Tables 7 and 8). Concentrations of soybean tissue P were 3.0–3.4 $g{\cdot}kg^{-1}$ in both years, which were close to the critical ranges for sufficient concentration found by Stammer and Mallarino (2018) but with younger developed plants (3.3–4.1 $g{\cdot}kg^{-1}$; V5–V6 stage). This also can mean a good P nutrition for this crop.

Soil N and P availability

The availability of soil NO_3-N varied with crop rotation. In the corn-soybean rotation, addition of mineral fertilizer N in the year of material application increased the soil NO_3-N content 1 mo after corn planting (V7 stage) and at VT stage (Fig. 1). However, at harvest, only PB addition induced a soil NO_3-N increase relative to the untreated control. For all sampling dates, biochar alone (VT stage) or with PB (V7 and R6 stages), irrespective of rate, reduced the soil NO_3-N availability even if mineral N supplementation was provided. The soil NH_4-N was low (0–3 $mg{\cdot}kg^{-1}$) and not significantly affected by biochar addition (data not shown). This reduction in soil NO_3-N availability, which was widely reported with biochars (Nguyen et al. 2017; Gao et al. 2019), benefits NO_3-N retention, preventing leaching losses, but may lead to insufficient N supply to crops. It was reported that biochars produced from wood

Table 5. Effect of biochar, paper mill biosolids (PB), and mineral fertilizer (NP) on corn growth in the year of application (2018).

Treatment	N nutrition index (no unit)	P nutrition index (no unit)	Grain yield ($Mg \cdot ha^{-1}$)	Specific grain weight ($kg \cdot hL^{-1}$)	Grain N ($g \cdot kg^{-1}$)	Grain P ($g \cdot kg^{-1}$)	Plant N uptake ($kg \cdot ha^{-1}$)	Plant P uptake ($kg \cdot ha^{-1}$)
Untreated	0.50	1.34	3.5	72	10.8	3.2	58	19
NP	0.96	1.05	11.2	75	11.7	2.4	176	29
Biochar 10 Mg	0.93	1.01	11.4	75	11.0	2.5	164	29
Biochar 20 Mg	0.93	1.07	11.4	74	11.1	2.6	160	30
PB	0.95	1.05	11.5	75	10.5	2.6	157	31
PB + biochar 10 Mg	0.86	1.05	10.5	74	10.3	2.7	148	30
PB + biochar 20 Mg	0.88	1.08	10.5	73	10.3	2.7	147	30
LSD (5%)	0.12	0.09	0.7	1	1.1	0.2	18	3
				Statistical analysis (*F* value)				
Treatment	14.6***	12.0***	99.8***	5.6**	1.7	13.0***	28.5***	11.0***
Contrasts								
NP vs. untreated	<0.001	<0.001	<0.001	<0.001	0.09	<0.001	<0.001	<0.001
NP vs. PB	0.84	0.87	0.32	0.92	0.034	0.035	0.040	0.14
Biochar, linear	0.61	0.68	0.54	0.30	0.23	0.13	0.08	0.54
Biochar × PB, linear	0.28	0.42	0.009	0.024	0.69	0.38	0.25	0.62
Biochar vs. biochar + PB	0.17	0.41	0.001	0.049	0.06	0.010	0.029	0.47
NP vs. biochar + PB	0.09	0.69	0.033	0.038	0.005	0.001	0.001	0.23

Note: Statistical significance at 1% and 0.1% denoted by ** and ***, respectively. LSD, least significant difference.

Table 6. Effect of biochar, paper mill biosolids (PB), and mineral fertilizer (NP) on corn growth in the residual year (2019).

Treatment	N nutrition index (no unit)	P nutrition index (no unit)	Grain yield (Mg·ha^{-1})	Specific grain weight (kg·hL^{-1})	Grain N (g·kg^{-1})	Grain P (g·kg^{-1})	Plant N uptake (kg·ha^{-1})	Plant P uptake (kg·ha^{-1})
Untreated	0.41	1.19	2.3	68	9.6	3.5	33	12
NP	0.81	0.92	9.1	69	11.3	2.6	127	25
Biochar 10 Mg	0.87	0.92	9.2	69	11.0	2.5	126	25
Biochar 20 Mg	0.80	0.95	9.0	69	11.6	2.7	126	25
PB	0.81	0.99	8.8	69	11.6	2.7	127	25
PB + biochar 10 Mg	0.86	0.97	9.3	69	11.3	2.6	123	25
PB + biochar 20 Mg	0.93	1.01	9.6	70	11.2	2.8	128	27
LSD (5%)	0.14	0.11	1.1	1	1.2	0.2	15	3
				Statistical analysis (*F* value)				
Treatment	11.2***	4.3**	43.3***	3.1*	2.2	15.3***	43.5***	20.4***
Contrasts								
NP vs. untreated	<0.001	<0.001	<0.001	0.25	0.012	<0.001	<0.001	<0.001
NP vs. PB	0.91	0.19	0.49	0.69	0.60	0.42	0.91	0.88
Biochar, linear	0.91	0.53	0.87	0.41	0.58	0.61	0.99	0.98
Biochar × PB, linear	0.10	0.76	0.14	0.18	0.49	0.40	0.88	0.09
Biochar vs. biochar + PB	0.21	0.23	0.47	0.14	0.84	0.17	0.96	0.28
NP vs. biochar + PB	0.13	0.15	0.53	0.09	0.92	0.28	0.90	0.40

Note: Statistical significance at 5%, 1%, and 0.1% denoted by *, **, and ***, respectively. LSD, least significant difference.

Table 7. Effect of biochar, paper mill biosolids (PB), and mineral fertilizer (NP) on soybean growth in the year of application (2018).

Treatment	N nutrition index (no unit)	Plant P at beginning bloom ($g \cdot kg^{-1}$)	Grain yield ($Mg \cdot ha^{-1}$)	Grains (kg^{-1})	Grain N ($g \cdot kg^{-1}$)	Grain P ($g \cdot kg^{-1}$)	Plant N uptake ($kg \cdot ha^{-1}$)	Plant P uptake ($kg \cdot ha^{-1}$)
Untreated	1.04	3.1	4.3	6574	57	5.2	236	21
NP	0.99	3.0	4.3	6580	56	5.2	229	21
Biochar 10 Mg	1.03	3.0	4.3	6620	59	4.9	245	21
Biochar 20 Mg	1.06	3.2	4.5	6578	60	5.1	256	22
PB	1.00	3.1	4.7	6124	61	5.1	270	23
PB + biochar 10 Mg	1.08	3.1	4.8	6166	58	5.0	263	23
PB + biochar 20 Mg	1.00	3.4	4.7	6562	59	5.4	263	25
LSD (5%)	0.09	0.3	0.7	428	4	0.4	43	5
				Statistical analysis (*F* value)				
Treatment	1.5	1.6	1.5	3.3*	1.4	1.5	1.5	0.9
Contrasts								
NP vs. untreated	0.29	0.41	0.94	0.98	0.50	0.91	0.73	0.89
NP vs. PB	0.78	0.33	0.22	0.038	0.024	0.61	0.06	0.43
Biochar, linear	0.70	0.79	0.62	0.98	0.13	0.68	0.34	0.87
Biochar × PB, linear	0.94	0.09	0.92	0.046	0.42	0.09	0.75	0.34
Biochar vs. biochar + PB	0.95	0.13	0.21	0.12	0.41	0.13	0.43	0.13
NP vs. biochar + PB	0.21	0.05	0.16	0.24	0.14	0.75	0.07	0.14

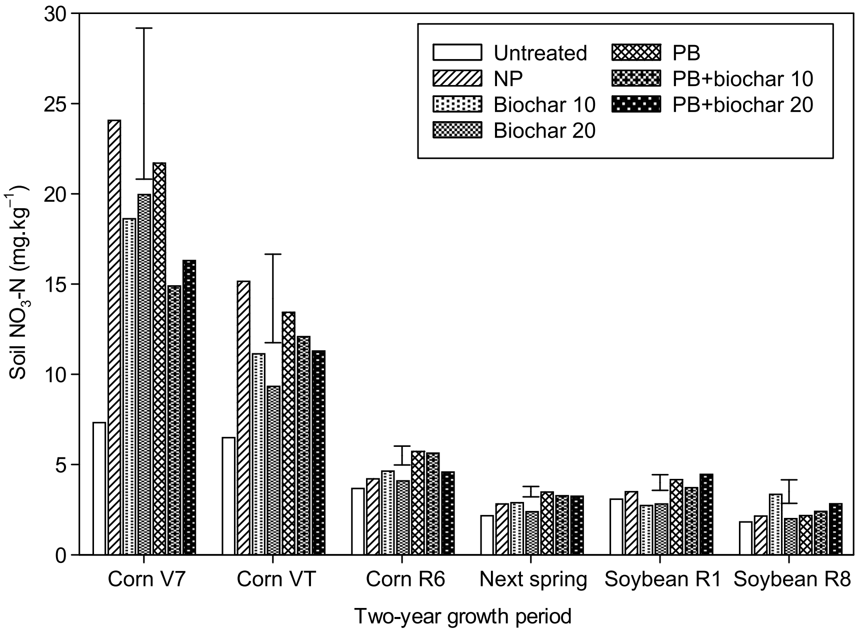
Note: Statistical significance at 5% denoted by *. LSD, least significant difference.

Table 8. Effect of biochar, paper mill biosolids (PB), and mineral fertilizer (NP) on soybean growth in the residual year (2019).

Treatment	N nutrition index (no unit)	Plant P at beginning bloom ($g \cdot kg^{-1}$)	Grain yield ($Mg \cdot ha^{-1}$)	Grains (kg^{-1})	Grain N ($g \cdot kg^{-1}$)	Grain P ($g \cdot kg^{-1}$)	Plant N uptake ($kg \cdot ha^{-1}$)	Plant P uptake ($kg \cdot ha^{-1}$)
Untreated	1.01	3.3	4.0	6955	61	5.7	227	21
NP	0.99	3.2	4.1	6664	61	5.6	234	21
Biochar 10 Mg	1.02	3.2	4.6	6730	60	5.4	260	23
Biochar 20 Mg	0.99	3.1	4.8	6904	60	5.4	268	24
PB	1.03	3.4	4.3	6683	63	5.7	256	23
PB + biochar 10 Mg	1.01	3.2	5.0	6605	61	5.5	287	25
PB + biochar 20 Mg	0.95	3.1	4.8	6914	61	5.5	269	25
LSD (5%)	0.09	0.3	0.7	307	2	0.3	40	3
				Statistical analysis (*F* value)				
Treatment	0.9	1.5	1.9	1.6	2.9*	1.2	2.0	2.1
Contrasts								
NP vs. untreated	0.70	0.46	0.78	0.06	0.32	0.43	0.71	0.83
NP vs. PB	0.38	0.039	0.48	0.90	0.06	0.47	0.26	0.26
Biochar, linear	0.75	0.15	0.037	0.73	0.41	0.10	0.043	0.042
Biochar × PB, linear	0.07	0.029	0.32	0.13	0.007	0.27	0.60	0.27
Biochar vs. biochar + PB	0.34	0.49	0.47	0.59	0.13	0.48	0.37	0.18
NP vs. biochar + PB	0.81	0.91	0.025	0.47	0.62	0.50	0.025	0.011

Note: Statistical significance at 5% denoted by *. LSD, least significant difference.

Fig. 1. Effect of biochar (10 and 20 $Mg{\cdot}ha^{-1}$), paper mill biosolids (PB), and mineral fertilizer (NP) on the soil NO_3-N content in the 0–15 cm layer under the corn–soybean rotation. No mineral fertilizer was applied in the soybean year. Vertical bars represent the least significant difference (5%) for mean separation at each sampling date.



biomass could cause soil N immobilization in the short term due to their low nutrient content, thus necessitating fertilizer application to avoid crop N deficiency (Gul and Whalen 2016). Nevertheless, Zheng et al. (2012) observed in their soil incubation a decrease in soil NO_3-N with mixed effect on NH_4-N after addition of an oak-derived biochar fertilized with NH_4NO_3 and attributed this to microbial immobilization rather than direct adsorption of inorganic N on biochar surface. Negative contribution to soil NO_3-N content was also reported in other studies when biochar was combined with NH_4NO_3 (Nelson et al. 2011; Nguyen et al. 2017).

In the soybean–corn rotation, the effect was related to the materials themselves, due to the absence of supplemental N fertilization. Contrarily to what was observed in corn, application of biochar did not affect the soil NO_3-N content in the year of application (Fig. 2). However, biochar co-applied with PB, particularly at the highest biochar rate, promoted soil NO_3-N increases at R1 and R8 stages of soybean to a level higher than biochar or PB applied alone. Hamer et al. (2004) observed that addition of glucose, which largely composes PB (McGovern et al. 1983), accelerated the wood biochar mineralization under controlled conditions due to enhanced growth in microbial biomass and decomposition of labile C compounds. This synergistic effect could also be explained indirectly by an increase in biological N_2 fixation induced by high soil K availability (135 mg Mehlich-3 $K{\cdot}kg^{-1}$ in biochar-amended plots compared with 63 $mg{\cdot}kg^{-1}$ in the untreated control; Mia et al. 2014). However, the soil NH_4-N was low (1–3 $mg{\cdot}kg^{-1}$) as for corn and unaffected by biochar addition (data not shown).

In the following spring, plots receiving PB had higher soil NO_3-N content than the untreated control, whereas PB still increased soil NO_3-N at soybean beginning bloom and at corn R6 stage (Figs. 1 and 2). This indicated that PB released its organic N for more than one season. Previous biochar addition had little effect on soil NO_3-N availability in the residual year, and reduction observed early after material application was not noted. It was found that soil N immobilization following biochar was short-lived and largely attenuated beyond the first year (Deenik et al. 2011; Tammeorg et al. 2014). Thus, the biochar used, as for many other wood biochars (Nelissen et al. 2015; Ippolito et al. 2020; Romero et al. 2021), was not likely a source of N to crops, based on material characteristics such as total N and C/N ratio (Table 4) and provided no soil N contribution in the year of application or in the residual year.

Soil extractable Mehlich-3 P content was constantly and linearly increased by biochar, both in the year of application and in the following year (Figs. 3 and 4). Crop rotation affected soil Mehlich-3 P only through the mineral fertilizer P addition to corn receiving NP, biochar alone at 10 $Mg{\cdot}ha^{-1}$ and PB with no biochar (Table 2). The increase at corn VT stage in the residual year was substantial for NP and PB (Fig. 4) and probably attributable to the conditions being less favorable to early plant growth, combined with less rain at that time (Table 3), causing soil dryness and reducing cumulated plant P uptake. Contrarily to N, wood biochar was a good

Fig. 2. Effect of biochar (10 and 20 Mg·ha^{-1}), paper mill biosolids (PB), and mineral fertilizer (NP) on the soil NO_3-N content in the 0–15 cm layer under the soybean–corn rotation. No mineral fertilizer was applied in the soybean year. Vertical bars represent the least significant difference (5%) for mean separation at each sampling date.

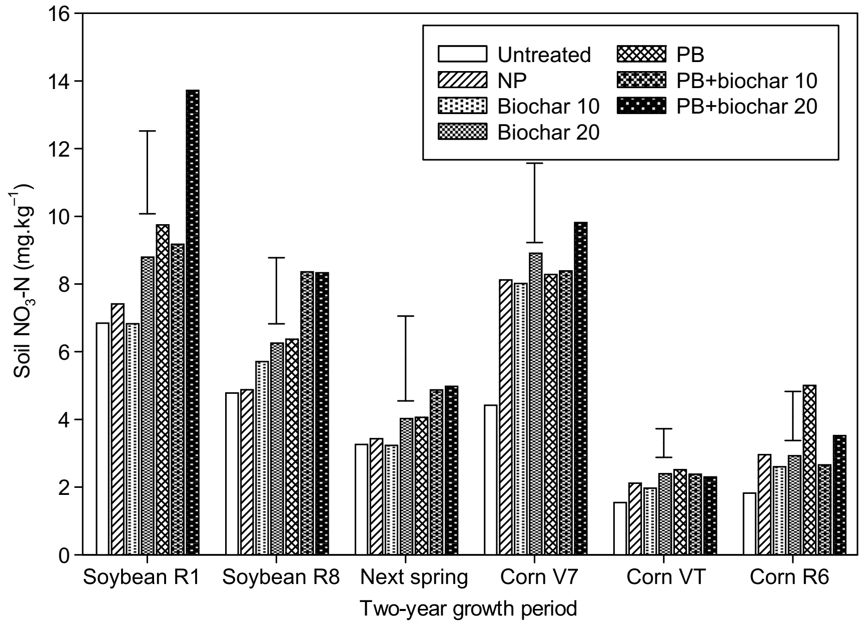
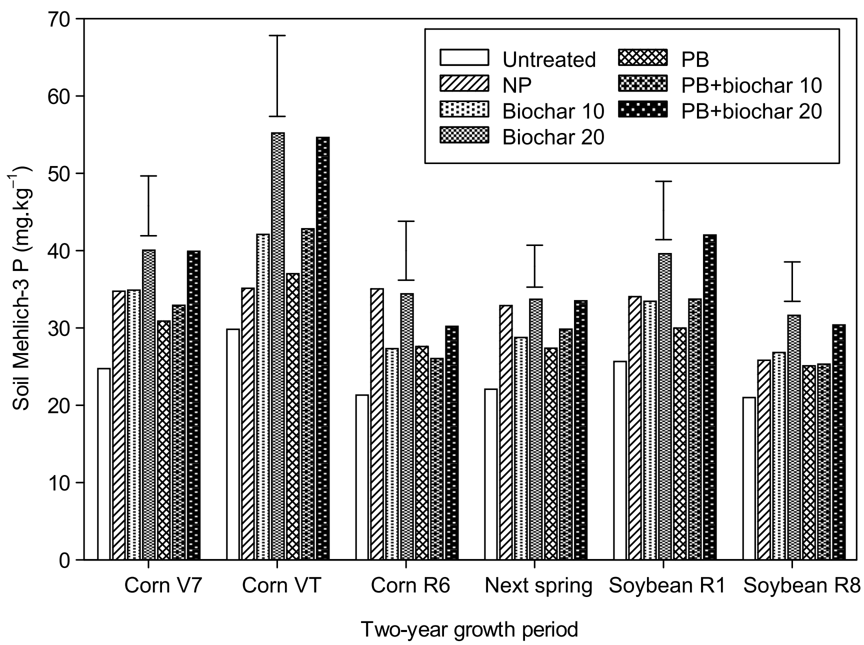
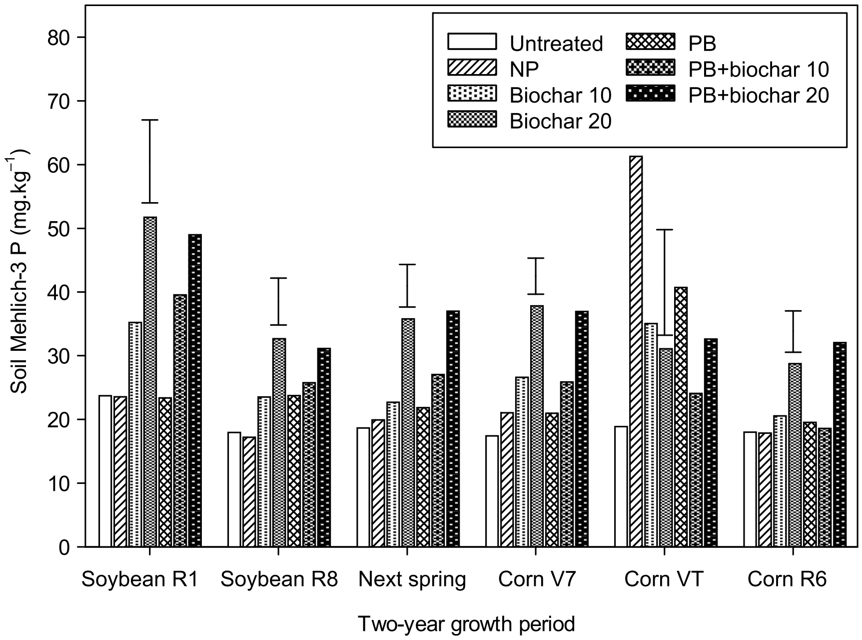


Fig. 3. Effect of biochar (10 and 20 Mg·ha^{-1}), paper mill biosolids (PB), and mineral fertilizer (NP) on the soil Mehlich-3 P content in the 0–15 cm layer under the corn–soybean rotation. No mineral fertilizer was applied in the soybean year. Vertical bars represent the least significant difference (5%) for mean separation at each sampling date.



source of P and was found to be most efficient in promoting soil-available P in soil pH of 6–7.5 (Gao et al. 2019). The increase in soil Mehlich-3 P could be a direct contribution from biochar and (or) a direct attraction of cations (Al^{3+}, Fe^{3+}, and Ca^{2+}) on biochar surface (Xu et al. 2014) because of the alkaline nature of the soil (Table 1). Wood-derived biochars usually contain lower total P than other biochar types (Ippolito et al. 2020) and were reported to have no effect on soil P bioavailability (Glaser and Lehr 2019) unless they were co-applied with NP (Chathurika et al. 2016; Romero et al. 2021). In this study, wood biochar contained an appreciable amount

Fig. 4. Effect of biochar (10 and 20 Mg·ha^{-1}), paper mill biosolids (PB), and mineral fertilizer (NP) on the soil Mehlich-3 P content in the 0–15 cm layer under the soybean–corn rotation. No mineral fertilizer was applied in the soybean year. Vertical bars represent the least significant difference (5%) for mean separation at each sampling date.



of total P, more than other wood biochars evaluated in temperate conditions (4.3 vs. 0.2–1.8 g·kg^{-1}), and also higher ash content (Tammeorg et al. 2014; Laird et al. 2017; Soinne et al. 2020; Romero et al. 2021). Considering its attributes such as total P, ash content, and ratio of O/C and volatile matter/fixed C (Table 4), the present wood biochar shows a good compromise between P availability and C stability.

Crop yield

The soil of both sites was highly responsive to N addition under grain corn production, with an increase for the NP treatment of 7.7 Mg·ha^{-1} in 2018 and 6.8 Mg·ha^{-1} in 2019 compared with the untreated control (Tables 5 and 6). The corn yields were higher in 2018 than in 2019, likely due to more favorable growing conditions (Table 3), whereas the yields in soybean were similar between years (Tables 7 and 8). Both sites were in the low range for P fertility and P fixation capacities (Al <1100 mg·kg^{-1}; Pellerin et al. 2006), indicating that positive yield response to applied P could be expected.

In the year of application, all materials and NP treatments increased corn grain yield, specific grain weight, and plant N and P uptake (Table 5). As expected, biochar alone did not contribute to corn N nutrition and required fertilizer N supplementation to achieve high crop yields. This agrees with the study of Rajkovich et al. (2012), which reported little improvement in corn N uptake following addition of biochars of wood residues. For its part, the N supplementation with PB was insufficient to achieve comparable grain N and plant N uptake as with NP treatment, with PB showing lower than expected relative N effectiveness (5% vs. 9% in Joseph et al. 2017). In the residual year, treatments had no significant effect on corn, apart from that associated with the untreated control (Table 6).

Lack of yield enhancement with wood biochar under temperate or boreal conditions has been reported in many studies (Jones et al. 2012; Borchard et al. 2014; Tammeorg et al. 2014; Soinne et al. 2020). Crop yield response to biochar is complex and depends on numerous factors. In well-managed fertile temperate soils with sufficient nutrient supply, yield response to biochar was unlikely (Jay et al. 2015). Benefits are reported when soil conditions constrain productivity, such as low organic C, very high acidity, and limited water retention (Güereña et al. 2013; Laird et al. 2017). Backer et al. (2016) observed increases in corn yield on acidic loamy sand, but not on acidic sandy clay loam, when applying 20 Mg pine biochar·ha^{-1}. In a meta-analysis, Dai et al. (2020) reported that application of biochar with a high ash (>25%) or a low C (<50%) content, such as here (Table 4), is highly recommended for increasing plant productivity in sandy or acidic soils.

Conversely, soybean responded little to materials in the year of application, with only increases in grain size (lower grains per kilogram) and grain N concentration following PB addition (Table 7). This absence of response to biochar for soybean was also noted by Backer et al. (2016). In the residual year, however, previous biochar application alone or with PB increased grain yield by 0.6 Mg·ha^{-1} and plant N and P uptake (Table 8). This can be explained by the high supply of P, K, and other cations from this biochar, which were gradually released and benefited soybean growth (Rondon et al. 2007).

Co-application of PB and biochar, irrespective of biochar rate, decreased the grain yield by 1.0 $Mg \cdot ha^{-1}$ and reduced plant N uptake and grain quality as compared with biochar alone (Table 5). Several factors could contribute to this temporary reduction in soil N availability, such as biochar surface adsorption of NO_3-N and NH_4-N released from organic materials (Shaaban et al. 2018; Manirakiza et al. 2019), high volatile matter content in biochar (Deenik et al. 2010), and the C/N ratio of material (Gao and DeLuca 2016; Nguyen et al. 2017). Our results did not support these hypotheses because biochar with only N supplementation did not produce any negative effect and biochar with PB in absence of N fertilization promoted soil N mineralization in soybean. It may be reasonable to think that more than one factor may be involved, but the results of this study, particularly regarding soil properties, need further investigation.

Conclusion

Results indicated that the wood biochar was not a source of N to crops and needed fertilizer N supplementation to achieve high yield. In contrast, it was a good source of P, as it positively impacted the P availability status of these soils. Unfortunately, co-applying biochar and PB was detrimental to corn in the year of application, reducing soil NO_3-N availability and decreasing grain yield as compared with biochar or PB applied alone. Nonetheless, this negative effect lasted only 1 yr. By contrast, all amendments did not affect soybean in the year of application, but previous biochar addition increased grain yield in the residual year.

Future research will look at the long-term effect of such co-application due to the sorption capacity of biochar and gradual release of nutrients over time. This could also be evaluated on acidic soils because of the good liming value of this wood biochar. Different combinations of PB and biochar could also be assessed in the field.

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