

# Plant Macro- and Micronutrient Dynamics in a Biochar-Amended Wetland Muck

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**Abstract** Biochar is an organic carbon (OC) and plant nutrient-rich substance that may be an ideal amendment for bolstering soil organic matter and nutrient contents. Two biochars were produced by pyrolysis at 350 °C from pine chips (*Pinus taeda*) and swine manure solids (*Sus scrofa domestica*). The biochar total elemental composition was quantified using inductively coupled plasma spectrometer and their surface chemical composition examined using a combination of scanning electron microscopy (SEM) and electron dispersive spectroscopy (EDS). The biochars were mixed into triplicate pots containing Lauderhill muck (Euic, hyperthermic Lithic Haplosaprists) at 0, 2.5, 5, and 10 % (dry mass). Four simulated water infiltration events were conducted during the 124-day incubation to assess the potential alteration in the leaching potential of soluble soil nutrients. At termination, the muck's fertility characteristics

were assessed, and dissolved cations were measured in water leachates. Neither biochars significantly increased the muck's OC contents. Swine manure biochar contained higher K, Mg, Na, and P concentrations, and these differences were observable in SEM and EDS as differing amounts of surface-precipitated Mg and K salts. Correspondingly, swine manure biochar at all three applications rates significantly increased Mehlich 1-s K, P, Mg, and Na concentrations. Pine chip biochar only improved the Mehlich 1-extractable K concentration but did reduce soluble P concentrations. Water leachates from swine manure biochar treated wetland soil contained significantly higher soluble P concentrations that could create water quality issue in downstream ecosystems.

**Keywords** Biochar · Wetlands · Soluble nutrients · Leaching

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## 1 Introduction

Histosols in the Florida Everglades Agricultural Area (EAA) are organic carbon (OC)-rich muck soils that formed in a limestone-basin from the accumulation of plant debris (Stephens 1969). These Histosols are inherently fertile and have been drained for agricultural production for over 150 years (Stephens 1956). Their drainage has resulted in enhanced microbial OC decomposition (Tate 1979) causing subsidence rates of up to 4 cm/year (Stephens et al. 1984). The Histosols were initially several meters thick in areas, but high subsidence rates

has caused some Histosols to be only 1–2 m thick before contact with the limestone bedrock (Ingerbritsen et al. 2013).

The long-term (next 50 years) sustainability of agriculture in the EAA will be a challenge, with nearly one-half of the south Florida Everglades projected to have Histosol thickness reduced to <20 cm (Snyder et al. 1978). A potential strategy to restore wetlands is the use of biochar (Ballantine et al. 2012). In a recent study, biochar produced from bone feedstock was applied to wetland soils in Jordan, resulting in higher extractable P concentration (Barnea et al. 2012). With respect to subsidizing Histosols, however, utilization of biochar for adding organic compounds back into the OC pool and for improving inorganic fertility has not been well investigated.

With biochar utilization in Histosols, careful selection of feedstock type is needed. Biochars produced from animal manures are P-enriched while biochars produced from lignocellulosic feedstocks are nutrient-poor (Novak et al. 2013). If a manure-based biochar were applied to a Histosol, any soluble P released and transported via drainage canals could result in water quality impairment of connected surface water systems (Litaor et al. 2008).

Biochars have also been reported to bind with soluble plant nutrients and reduce their downward movement. Biochars produced from Brazilian pepperwood (*Schinus terebinthifolius*) after addition to a sandy soil reduced nitrate, ammonium, and phosphorus leaching, presumably by binding onto soil cation exchange sites (Yao et al. 2012). In the same study, however, biochar made from poultry litter had higher P concentrations in water leachates. Laird et al. (2010) reported reduced N and P leaching from a mid-west Mollisol treated with a hardwood biochar, whereas Singh et al. (2010) reported mixed results of ammonium movement through an Alfisol and Vertisol treated with biochars produced from poultry manure and *Eucalyptus* chips (*Eucalyptus* sp.).

We tested the hypothesis that two different biochar-types applied to a Histosol in the EAA could re-build the OC pool and supply the wetland soil with extractable plant macro- and micro-nutrients. Attention was also focused on the impact of both biochars on nutrient retention and water-soluble P release. In the EAA, reducing soluble P movement from agricultural activity into the Everglades is a major water quality goal (Daroub et al. 2002; Rice et al. 2002).

Our objectives were: (1) to characterize the total elemental composition and identify salts on biochar surfaces after pyrolysis of pine chip and swine solid feedstocks, (2) to determine if these biochars increase the OC pool and plant available nutrient concentrations of an wetland soil, and (3) to assess if these two biochars added to the muck soil results in significant releases of soluble cations after successive water infiltration events.

## 2 Material and Methods

### 2.1 Site Description, Soil Sampling, and Characterization Procedures

In this study, samples from a Lauderhill muck soil were collected from a nearly level (0–1 % slope) field located in Palm Beach County, approximately 20 km east of Belle Glade, Florida, USA (26.72305°N, 80.45794°W). Previously, the field has been in crop production (sugarcane) for several decades. The muck soil has a profile composed of black and brown-colored sapric (well-decomposed) residues from plant leaves and roots. Samples were collected using a shovel from the top part of the profile (0 to 30 cm thick) and were placed in sealed plastic bags.

The muck samples were initially air-dried, and then later, subsamples were placed in an oven for further drying at 105 °C to determine moisture content of the air-dried muck soil. The gravimetric moisture content was determined to be 60.8 % (w/w). The muck samples were then ground through an 8-mm sieve, placed into sealed plastic bags, and then refrigerated at 5 °C.

No particle size analysis of the Lauderhill muck was conducted due to the predominance of sapric material. Total C and N were determined by dry combustion (LECO TruSpec CN analyzer, LECO Corp., St. Joseph, MI). The pH of the muck soil in deionized water was 6.5 (1:2w/v). Total elemental composition of the muck soil was determined using EPA method 3051A acid digestion method (USEPA 1996). Elements in the digestion solutions were quantified using an inductively coupled plasma (ICP) spectrometer as outlined previously (Novak et al. 2009). Muck samples were also extracted for fertility characteristics using Mehlich 1 extracting solution by the Clemson University Soil Testing Laboratory (<http://www.clemson.edu/agsvin/interest.htm>). Cations in the extraction solutions were measured

using ICP and were summed for cation exchange capacity (CEC) estimations.

## 2.2 Biochar Production and Chemical Characterization

Fresh pine chips were collected from a United States Forest Service tree harvesting operation near Cordesville, SC, and the swine manure solids were obtained from a liquid–solid separation facility in Sampson Co., NC. The pine chips and swine solids were initially air-dried and ground into 6-mm flakes. The flakes were pyrolyzed at 350 °C using a Lindberg electric box oven as described by Novak et al. (2013). After pyrolysis, the biochars were further ground to pass through a 0.25-mm sieve and were stored in a dessicator.

The pine chip and swine solid biochars were characterized for their elemental concentrations of C, H, N, S, O (by difference), and ash contents by Hazen Research Inc. (Golden, CO) using ASTM D 3172 (ASTM 2006). Additional elements in the biochars were measured using acid-digestion procedures and ICP quantification methods as described above (Table 2). The volatile matter and ash contents of the biochars produced from pine chip and swine solids at 350 °C were 450 and 460 g/kg, and 15 and 320 g/kg, respectively. The pine chip and swine solid biochars also had pH values in deionized water of 4.4 and 9.1, respectively (1 % w/w shaken for 24 h at 200 rpm).

## 2.3 Scanning Electron Microscopic and Energy Dispersive Spectroscopy of Biochar

The SEM images were collected using a JEOL 6500 SEM (Joel, Inc. Tokyo, Japan) at the University of Minnesota–Surface Characterization Laboratory. The biochars were attached to the SEM mount by an adhesive pad (PELCO Tabs™, Ted Pella, Inc.; Redding, CA) and then dried at 105 °C for 24 h to remove trapped water vapor to reduce vacuum equilibration times (or sample degassing times). The EDS data were conducted of the observed surface-precipitated salts to assess qualitative chemical composition (Thermo-Noran Vantage system). There was no surface coating used for this imaging. Surface porosity and salt precipitate coverage percentages were calculated using software NIH ImageJ (with JPor; Grove and Jerram 2011). Briefly, the SEM images were converted to bitmap code, and the threshold was set to identify the pores or salts (threshold settings). This numerical estimation has provided

accurate porosity estimations for digital images compared with more traditional assessment methods across a variety of materials (Janseen et al. 2011).

## 2.4 Biochar Incubation and Water Leaching of the Lauderhill Muck

The Lauderhill muck soil and biochar incubation experiment consisted of three replications of two biochar treatments at four rates, a control (no biochar) and biochars added at 2.5, 5, and 10 % on a dry weight basis. With a bulk density of 0.2 g/cm<sup>3</sup> and to a depth of 15 cm in the muck soil, these w/w rates equate to biochar field application rates of 10, 20, and 40 t/ha. The treatments were placed into pots similar to those described by Novak et al. (2009). Afterwards, sufficient deionized water was added to each pot to bring it up to 125 % of field capacity, which is typical for this muck soil (Alan Wright, University of Florida, Belle Glade, FL, personal communication, 2010). The pots were placed in a randomized order on a bench surface and incubated for 124 days under laboratory conditions that ranged from 18 to 29 °C and 22 to 85 % relative humidity. Throughout the incubation time course, the moisture contents of the pots were adjusted twice per week with sufficient deionized water to maintain them at 125 % field capacity. On days 34, 62, 90, and 124 of incubation, 250 mL of deionized water was allowed to infiltrate through each pot. This water volume was determined in pretrial tests to provide sufficient leachate volume (≈190 mL) necessary for subsequent elemental characterization. The leachates were then frozen at 0 °C until analyzed for K, Na, Mg, P, Mn, Cu, and Zn concentrations using ICP.

At termination, each pot was allowed to air-dry for several days and the contents removed for final drying. Portion of each muck sample were then analyzed by the Clemson Soil Testing Laboratory for pH and soil fertility levels using Mehlich 1 extraction solution.

## 2.5 Statistical Analyses

A Duncan's multiple-range test via a SAS PROC ANOVA (v10. SAS Institute, Cary, NC) was used to test the Mehlich 1-extractable K, P, Mg, Na, Mn, Cu, and Zn concentrations from the muck as influenced by the sources of biochar feedstock, biochar application rate, number of leaching events, and interaction among these treatment variables. A similar procedure was used

to determine the impact of these variables on comparable elements in the water leachates.

### 3 Results and Discussion

#### 3.1 Total and Extractable Elements in the Lauderhill Muck and Biochars

The Lauderhill muck had a TOC content of 46.93 and a total N (TN) content of 2.65 g/kg (*w/w*). The muck also contained plant extractable macro-nutrients (Ca, K, Mg, P) and micro-nutrients (Zn, Table 1). The relatively high concentrations of Ca, Mg, and Na in the surface layers of the muck profile were expected considering the close contact (<1 m) to the calcareous bedrock and the proximity of the site to coastal salt water ( $\approx 65$  km). The choice of feedstock and the low pyrolysis temperature had an influence on the total chemical characteristics of the two biochars (Table 2). Pine chips as a feedstock for biochar production at the mild pyrolysis temperature  $\approx 350$  °C are known to be C- and O-enriched but N-poor due to residual lignin and cellulose-type structures composing most of the biochar organic composition (Novak et al. 2013). Biochar produced from swine manure solids, on the other hand, is higher in ash content (320 vs. 15.3 g/kg) because of the abundance of macro-(N, Ca, K, Mg, and P) and micro-nutrients (B, Cu, Mn, and Zn) compared with pine chip biochar. Of particular importance for the nutrient values in swine solid biochar are the higher concentrations of P and K compared with pine chip biochar.

**Table 1** Total and Mehlich 1-extractable elements in a Lauderhill muck soil prior to adding biochar

Elements	Total <sup>a</sup>	Mehlich 1
	mg/kg	
B	13	1.0
Ca	86,085	5,625
Cu	85	0.1
K	1,050	123
Mg	2,616	577
Mn	231	2.3
Na	171	48
P	657	12.4
Zn	69	1.9

<sup>a</sup>EPA method 3051A

**Table 2** Total elemental composition of biochars produced from pine chips and swine manure solids feedstocks

Element	Elemental composition	
	Pine chip <sup>a</sup>	Swine solids
	g/kg	
C	747	508
H	50	50
N	4.5	37
O	184	66
S	0.1	8
	mg/kg	
B <sup>b</sup>	6.0	52
Ca	3,317	47,347
Cu	6.0	1,653
K	1,926	19,274
Mg	866	24,606
Mn	83	1,716
Na	27	6,189
P	206	40,250
Zn	32	3,778

<sup>a</sup> Portions of results were published in Novak et al. (2013)

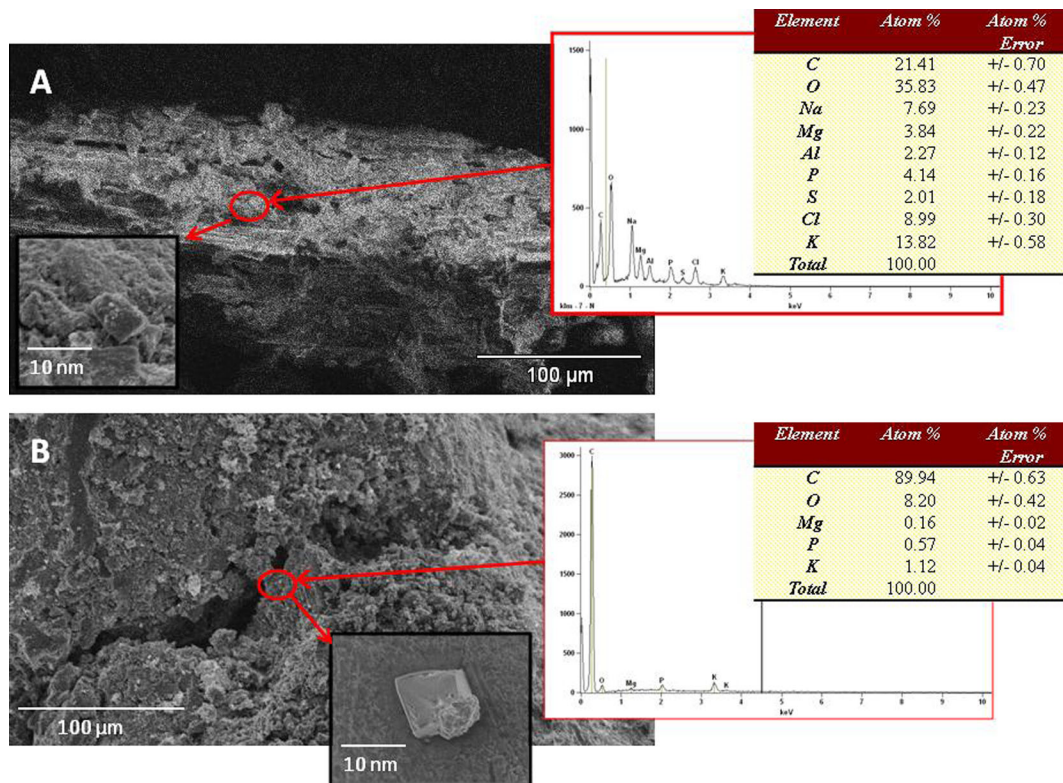
<sup>b</sup> EPA method 3051A

#### 3.2 Biochar SEM and EDS Images

Spectroscopic images of the two fresh (not incubated in muck soil) biochars used in this study exemplify dramatic differences in their surface pore structure and chemical composition (Fig. 1a and b). The overall surface porosity for the swine manure biochar was 31.5 % and 50.7 % for the pine chip. A large crevice appears in the 100- $\mu$ m scan of the pine chip biochar (Fig. 1b).

A more apparent difference was noted in the surface chemistry between the two biochars. This can be seen in EDS scan showing the elemental composition as well as the extreme 10 nm close up (Fig. 1a and b). The elemental composition of the swine manure biochar shown as an inlet reveals more diversity of ionic elements and a higher amount of surface precipitated salts. In fact, approximately,  $57.9 \pm 0.8$  % for the swine and  $17.8 \pm 0.45$  % for the pine chip biochar was covered with surface-precipitated salts. Additionally, there was a chemical compositional difference of the surface-precipitated salts, with the swine manure biochar having P salts comprised of more mono-, di-, and trivalent (i.e.,





**Fig. 1** Representative SEM images of fresh (not incubated in soil) biochars produced from **a** swine manure solids and **b** pine chip feedstocks. The *inset graph* illustrates the biochar surface at a higher magnification at the location of the energy dispersive

spectroscopy analysis, which is also shown as an insert graph. Note the drastic difference in the appearance (*white colors on swine solids*) of the biochar surface due to the precipitated salts

K, Na, Mg, Al) than the corresponding analysis of the pine chip surface salts (i.e., Mg, K, Fig. 1). This is important because cations with lower charge states are comparatively more soluble than polyvalent cations (Mengel and Kirkby 2001). This could be another reason for the different form of P salts in these biochars, with the manure biochar being composed of  $\text{AlPO}_4$  (a common form for commercial phosphate) and Mg and K phosphates on the pine chip biochar surface (Fig. 1). Whatever the reason for their presence, the salts in biochars that are composed of cations with higher valency will still possess different solubility properties, which would lead to differential nutrient leaching characteristics.

### 3.3 Biochar Incubation in the Lauderhill Muck

Adding biochar produced from swine solids significantly increased the extractable K, P, Mg, and Na concentrations relative to muck treated with pine chip biochars

with the noted concentration increases proportional to the amount of biochar applied (Table 3). This is consistent with the significant interaction determined between feedstock  $\times$  application rate (Table 1,  $P$  values  $<0.001$ ). Applying swine solid biochar at 2.5 to 10 % overloaded the Mehlich 1-extractable P and K levels in the muck, such that the magnitude of the increase was far in excess of that considered optimum for sugarcane production on muck soils in Florida (Mehlich 3-extractable; 37 kg P/ha and 233 kg K/ha, Rice et al. 2010). Mehlich 1-extractable Mg and Na concentrations after adding the swine solid biochar were also significantly increased relative to the control. With respect to biochar produced from pine chips, only Mehlich 1 K concentrations at the 10 % application rate were significantly improved relative to the muck control.

Addition of swine solid biochar also significantly improved some micronutrient contents of the muck. There were significant increases in Mehlich 1-extractable Mn (4.5 to 22.4 kg/ha) and Zn (3.7 to

**Table 3** Mean fertility characteristics in a Lauderhill muck after 124 days incubation with biochars produced from two feedstocks and applied at four rates ( $n=3$ )

Feedstock	Mehlich 1-extractable (kg/ha)			
	K	P	Mg	Na
Swine solids	1,276a	547a	2,195a	467a
Pine chip	471b	24b	1,573b	167b
Control	436b	21b	1,607b	138b
Application (g/100 g)				
10	1,165a	490a	2,067a	395a
5	796b	212b	1,845b	271b
2.5	659c	155c	1,740c	243b
0 (control)	436d	21d	1,607d	167c
Main treatment effect	<i>P</i> value			
Source of variation				
Feedstock	<0.001	<0.001	<0.001	<0.001
Application	<0.001	<0.001	<0.001	<0.001
Feedstock×application	<0.001	<0.001	<0.001	<0.001

Means within a column that are sorted by feedstock and application rate and followed by a different letter are significantly different using a Duncan's multiple-range test

8.4 kg/ha) after adding the swine solid biochar, particularly at the 10 % application rate. Both of these ranges are close to the Mehlich 3-extractable level of Mn (5.6 kg Mn/ha) and Cu (2.2 kg Cu/ha) recommended for sugarcane production in muck soils (Rice et al. 2010). In contrast, pine chip biochar did not increase concentrations of these two plant nutrients because its composition was nearly devoid of these two elements (<2.5 mg/kg, Table 1).

There were no significant OC, CEC, or pH changes after adding these two biochars, even at the extremely high rates of 10 % (*w/w*). The OC contents of all biochar-treated muck soils were between 46.2 and 46.9 g/100 g which is not significantly different than the OC content of muck control 46.9 g/100 g ( $P=0.74$ , results not shown). The muck soils treated with both biochars maintained their pH values within 0.1 to 0.2 units of the control (6.4) while the control CEC (36.5 cmol/kg) was also similar to the biochar-treated mucks (35.9 to 36.9 cmol/kg). This muck soil is well-buffered due to the high OC content. Moreover, the

**Table 4** Mean dissolved K, P, Mg, and Na concentrations quantified by pooling amounts across four water leachates collected from Lauderhill muck after 124 days incubation with biochars produced from two feedstocks and applied at four rates

Feedstock	Leachate concentration (mg/L)			
	K	P	Mg	Na
Swine solids	39.1a	9.7a	9.1a	26.5a
Pine chip	18.5a	0.4b	6.6b	9.1b
Control	11.8c	0.2b	4.4c	6.5b
Application (g/100 g)				
10	38.6a	7.2a	9.6a	25.9a
5	28.4b	5.1b	7.9b	17.1b
2.5	19.3c	2.8c	6.3c	11.2c
0 (control)	11.8d	0.2d	4.4d	6.5d
Leaching no.				
1	44.6a	7.9a	12.6a	28.1a
2	20.7b	3.1b	6.3b	12.6b
3	20.2b	3.2b	5.8bc	12.8b
4	19.8b	3.1b	4.7c	12.4b
Main treatment effects				
Source of variation	<i>P</i> values			
Feedstock	<0.001	<0.001	<0.001	<0.001
Application	<0.001	<0.001	<0.001	<0.001
Feedstock×application	<0.001	<0.001	0.070	<0.001
Leaching no.	<0.001	<0.001	<0.001	<0.001
Feedstock×leaching no.	<0.001	<0.001	0.234	<0.001
Application×leaching no.	0.127	0.222	0.424	0.279
Feed×application×leaching no.	0.330	0.175	0.883	0.424

Means within a column that are sorted by feedstock application rate and leaching no. that are followed by a different letter are significantly different using a Duncan's multiple range test

sapric material in the muck contains functional groups capable of chelating or electrostatically binding cations, thus causing no significant impact on the CEC levels due to their resistance to extraction with Mehlich 1 reagent.

### 3.4 Dissolved Elements in Water Leachates

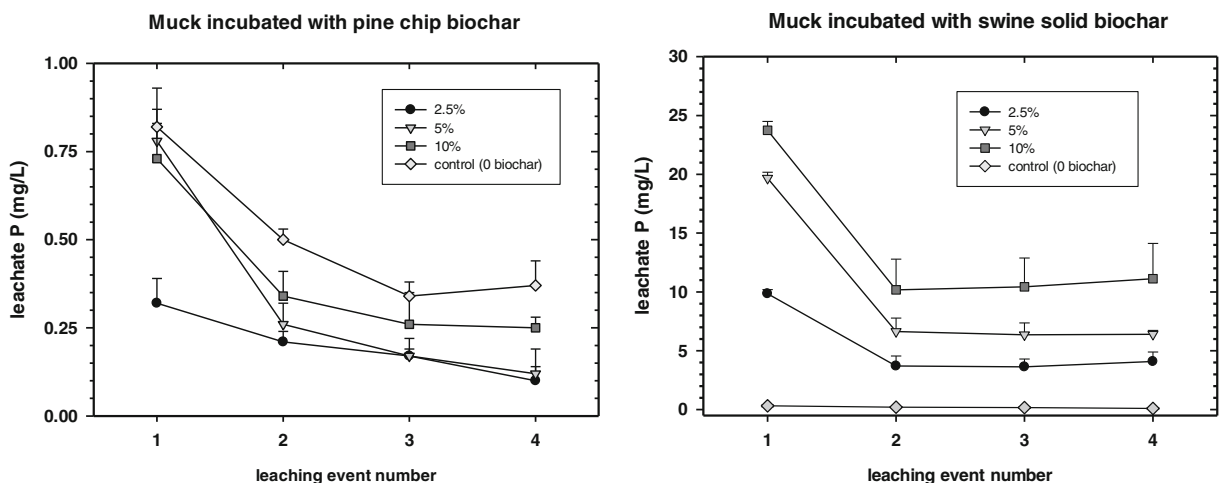
In all four leachates, there were non-measurable concentrations of water-soluble Mn, Cu, and Zn released from both biochar (data not included). This implies that these two biochars at this pyrolysis temperature were not a viable source of micro-nutrients for the Histosol.

Biochar produced from swine manure after incubation in the muck soil released significantly higher concentrations of soluble K and P relative to the muck treated with pine chip biochar and the control (Table 4). This is consistent with the elemental compositional analyses gathered using EDS (Fig. 1a). Soluble K and P measured in leachates from samples sorted by pooling both feedstocks and leaching events were proportional to the amount of biochar applied, which was manifested by a significant feedstock  $\times$  application rate interaction (Table 4,  $P < 0.001$ ). However, no significant differences in the mean K and P concentrations due to feedstock, application rate, or their interaction occurred after the first leaching event, which highlights the importance of conducting multiple leaching events. Overall, the release of 9.65 mg/L of

soluble P from this particular swine manure biochar application to these EAA mucks would create water quality impairment issues.

Significant amounts of Mg and Na were also released by the swine manure biochar compared to the pine chip biochar (Table 4). As more swine solid biochar was added to the muck soil, there was a significant increase in both of their concentrations. This condition was also confirmed by the highly significant feedstock  $\times$  application rate interaction (Table 4,  $P < 0.001$ ) and by the observations of these salts on the surface of the swine manure biochar (Fig. 1a).

Of particular concern was the continual release of soluble P from the swine manure biochar with successive water leaching events (Table 4, Fig. 2). More soluble P was released after the first leaching event at the highest swine manure biochar application amount (ca. 10 %, Table 4). This means that, if swine manure biochar were applied at 2.5 to 10 % (w/w), soluble P will be released into shallow ground water. In fact, even at the lowest application amount (ca. 2.5 %), the soluble P concentration was between 4 and 5 mg/L (Fig. 2). Soluble P concentrations released from pine chip biochar were one order of magnitude lower than the swine manure biochar. In fact, the soluble P concentrations from the muck treated with pine chip biochar continued to drop to  $< 0.5$  mg/L by the fourth leaching event (Fig. 2). This suggests that the pine chip biochar at 2.5 % reduced P leaching compared with the control and was able to control P movement.



**Fig. 2** Mean P concentrations measured in successive water leaching events during incubation of Lauderhill muck soil treated with different amounts of pine chip and swine solid biochar (error bars are 1 standard deviation)

## 4 Conclusions

Application of biochars produced from pine chip and swine manure solid feedstocks to a Lauderhill muck soil in the EAA of Florida revealed some significant differences between choice of feedstocks and application amount. Pine chip biochar had a significant effect on K but only the highest application rate. On the other hand, it was capable of reducing P leaching and may be considered a best management practice for reducing P movement from the EAA into adjacent waterways. Swine manure solid biochar added at all three applications rates (2.5, 5, and 10 %) to the muck soil, in contrast, significantly increased the Mehlich 1-extractable K, P, Mg, and Na. Water infiltration of the muck treated with swine manure solid biochar substantially increased mean soluble P concentrations in the collected leachates. The SEM scans of fresh swine solid biochar confirmed that P was bound in more water-soluble salts, which could explain the high concentrations observed in the water leachates. If applied to farm fields within the EAA using the application rates employed within this study, swine manure solid biochar may cause P-related water quality issues.

This research stresses the need to properly match anticipated soil benefits with proper biochar properties in order to avoid unintended *post facto* environmental and agronomic consequences. Research revealed in this study indicates that the soluble elements released from biochars are directly correlated to the chemistry of the surface-precipitated salts on the biochar. Swine solids biochar was not an appropriate amendment for this Histosol due to P release issues, but pine chip biochar is an environmentally safer amendment because it reduced P movement.

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