Biology and Fertility of Soils
February 2013, Volume 49, Issue 2, pp 245-250
DOI: 10.1007/s00374-012-0746-6; Print ISSN: 0178-2762; Online ISSN: 1432-0789

# Phosphorus Availability in Soils Amended with Wheat Residue Char

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## Abstract

Plant availability and risk for leaching and/or runoff losses of phosphorus (P) from soils depends among others on P concentration in the soil solution. Water soluble P in soil measures soil solution P concentration. The aim of this study was to understand the effect of wheat residue char (biochar) addition on water soluble P concentration in a wide range of biochar amended soils. Eleven agricultural fields representing dominant soil texture classes of Swedish agricultural lands were chosen. Concentrations of water soluble P in the soils and in biochar were measured prior to biochar incorporation to soils in the laboratory. Experiments with three dominant soil textures- silt loam, clay loam and an intermediate loam soil with different rates of biochar addition (i.e., 0.5, 1, 2 and 4%; w/w) showed that the highest concentration of water soluble P was achieved at an application rate of 1%. At higher application rates, P concentrations decreased which coincided with a pH increase of 0.3 - 0.7 units. When the eleven soils were amended with 1% (w/w) biochar, water soluble P concentrations increased in most of the soils ranging from 11 to 253%. However, much of the water soluble P added through the biochar was retained (33 - 100%). We concluded that - wheat residue char can act as a source of soluble P; and low and high additions of biochar can have different effects on soil solution P concentration due to possible reactions with Ca and Mg added with biochar.

*Key words:* biochar, water soluble phosphorus, phosphorus saturation, phosphorus retention, clay soils, sandy soils.

## Introduction

Phosphorus (P) is an essential macronutrient for plants and is added to soil in significant amounts when biochar is used as soil amendment. Concentrations of P (and other nutrients) in biochars are several times higher than in other organic materials because the charring process reduces the biomass volume by 60% to 90% (Brown 2009).

Field applications of biochar have been shown to increase soil fertility and productivity by improving soil physical (e.g. Liang et al. 2006; Downie et al. 2009), chemical (e.g. Glaser et al. 2002; Major et al. 2009), and biological properties (e.g. Pietikainen et al. 2000; Rondon et al. 2007). Biochar increases water and nutrient retention capacity in soil (Verheijen et al. 2010) and improves nutrient use efficiency of growing plants (Chan et al. 2007). Thereby, increased crop yields were recorded in fields amended with biochar (Iswaran et al. 1980; Lehman et al. 2003; Yamoto et al. 2006; Chan et al. 2007; Rondon et al. 2007). However, crop yield is often influenced by the availability of P and the effect of biochar application on P availability in soil is not well understood.

Instant availability of P for plant uptake (Van Der Paauw 1971; Beegle 2005) and for leaching and/or runoff losses (Maguire et al. 2005; Ulén et al. 2010; Parvage et al. 2011) depends on the concentration of P in soil solution. An estimation of water soluble P (WSP) in soil is a direct and simple measure of P concentration in soil solution (Van Der Paauw 1971; Self-Davis et al. 2000; Beegle 2005; Ulén et al. 2010).

The aim of this study was to investigate changes in WSP concentrations after addition of wheat residue char (biochar) in a range of soils with different textural classes. The hypotheses tested were: (i) biochar increases WSP in soil after application due to high concentrations of WSP in the ash fraction of biochar; (ii) WSP added with biochar is partly adsorbed on soil particles in accordance with the number of soil P binding sites, i.e., clay content, available Al, Fe and, Ca, and organic C; and (iii) calcium present in biochar increases soil pH and may precipitate WSP in soil.

## Materials and Methods

### *Experimental design*

The experiment was divided into two steps. In an initial screening experiment, three soils representing dominant soil textures - Skottorp (silt loam), Flinkesta (clay loam) and Flinkesta (an intermediate loam soil) were amended with biochar at rates of 0.5%, 1%, 2% and 4% (w/w) to find out the suitable amounts of biochar addition that would yield maximum WSP concentration in biochar amended soil. Mixtures of 6 g of dry soil and corresponding amounts of biochar (e.g. 0.03 g, 0.06 g, and so on) were transferred into 50 ml plastic centrifuge tubes and 18 ml of distilled water was added. The suspensions were then incubated for 16 hours to facilitate equilibrium concentrations between the solid and liquid phase and, were gently shaken (end over end) during the entire incubation period. Thereafter, the suspensions were centrifuged for 20 minutes at 2817g. The supernatants were then passed through 0.45 µm membrane filters (Schleicher and Schüll GmbH, Dassel, Germany) and analysed for WSP colorimetrically (Murphy and Riley 1962). The maximum WSP concentrations were measured at an addition of 1 % biochar.

In the main experiment all soils were experiment, all soils were therefore amended with 1% biochar and the same incubation time and analytical procedures were applied as described above. All experiments were replicated three times.

*Biochar analysis*

The biochar used was from a Swedish commercial producer named EcoEra AB (Östra Tommarp, Sweden) and originates from a mixture of seed coat, chaff and residues from winter wheat. The chemical composition of the biochar is given in Table 1.

Water soluble P, calcium (Ca), potassium (K), and magnesium (Mg) in the biochar were measured by inductively coupled plasma - atomic emission spectrophotometer (ICP-AES Optima 5300) after shaking biochar with water for 1 hour as proposed by Self-Davis et al. (2000) using a ratio of biochar to water of 5:1. In addition, water soluble chlorine (Cl) and sulfur (S) in the biochar were determined on an ion chromatography system (ICS-90) from the same extract. Total contents of P, K, Ca, Mg and Fe (iron) in the biochar were measured with ICP-AES Optima 5300 and S and Cl in ICS-90 after digestion with 7 M $HNO_3$ (SIS 1997). Concentrations of total C and total N in the biochar were determined using dry combustion of biochar at $1050^0$ C for 5 min, whereby the concentrations of C and N in the vapor were measured with a LECO CN2000 analyzer (LECO Cooperation 2003). Heavy metal contents (i.e., lead, Pb; cadmium, Cd; and mercury, Hg) in the biochar were provided by the producer.

*Soil analyses*

Eleven agricultural fields located in eight counties (Table 2) were chosen, representing the dominant soil texture classes of Swedish agricultural lands. Eight subsamples, taken from 0-10 cm depth after autumn harvest from each field, were pooled and thoroughly mixed. Samples were dried at $40^0$ C, grounded and passed through a 2 mm sieve. Dried samples of < 2 mm diameter were used for further experimentations.

Water soluble P in soils was determined colorimetrically (Murphy and Riley 1962) by using a Shimadzu UV-1201 spectrophotometer after shaking soil with water for 1 hour applying a ratio of soil to water of 3:1. Plant available P, Al, Fe and Ca in soils and biochar were extracted with ammonium acetate lactate (AL extractable) at pH 3.75 (Egnér et al. 1960) and analysed by ICP-AES Optima 5300. The degree of P saturation (DPS) in soils was calculated as the ratio of the elements on a molar basis in the AL extract using the equation:

DPS = [AL extractable P ÷ (AL extractable Fe + AL extractable Al)] x 100------------- (i)

Concentrations of organic C were determined after combustion of soils at 1250°C for 5 minutes with LECO CN2000 analyser (LECO Corporation 2003). The pH was determined in distilled water using a glass electrode; the ratio of soil (and/or biochar) to water was 5:1. Strongly bound P (2M HCl – P) was determined after digestion with 2 M HCl for 2 hours. Digested suspensions were filtered and the concentrations of P were measured by ICP-AES (KLS 1965). Soil texture was analysed using the pipette method and classified according to the Food and Agricultural Organization of the United Nations (FAO-ISRIC 1990).

*Statistical analysis*

Treatment effects were statistically analysed by one-way ANOVA and Tukey adjusted multiple mean comparisons using the general linear model procedure of the statistical software MINITAB 16. Correlations between different P forms and their characteristic elements were determined by using the Pearson correlation method. Linear regression modeling was used to investigate relationships between variables. The confidence level used for the analysis was 95%.

## Results and discussion

*Addition of P with biochar*

The biochar material was rich in macronutrients consisting of 0.9% P, 2.9% N, 2.5% Ca, 1.8% K, 0.5% Mg, 0.2% S, and had a pH value of 8.9 (Table 1). In particular, concentrations of water soluble P, K, Cl and S were high (543, 8826, 1428 and 417 mg $kg^{-1}$ biochar, respectively) corresponding to 6% P, 49% K, 89% Cl, and 24% S of the total contents. In contrast, Ca and Mg in biochar had a low solubility in water amounting to only 0.1 and 1.5% of the total content, respectively. High solubility of macronutrients in water extract probably resulted from the ash in the charred material. Glaser et al. (2002) described ash as a source of readily available nutrients for plant uptake in soils amended with biochar. Christensen (1977) and Ohno and Erich (1990) observed significant increase of available plant nutrients in soils receiving ash after forest fire. Heavy metal contents in the biochar were low (Table 1), not exceeding the maximum applicable limits when applied to agricultural land within the EU (Pollak and Favoino 2004).

Table 1: Chemical compositions of the biochar used as soil amendment and the amount of different compounds added; values within parentheses express the percentage of the total content

| Characteristics | Concentrations | | Amount added with 1% (w/w) biochar* | |
|---|---|---|---|---|
| | Total | Water soluble | Total | Water soluble |
| | g $kg^{-1}$ | | kg $ha^{-1}$ | |
| Moisture | 140 | - | 1680 - 2100 | - |
| Ash | 205 | - | 2460 - 3075 | - |
| Carbon (C) | 637 | - | 7644 - 9555 | - |
| Nitrogen (N) | 29 | - | 348 - 435 | - |
| Sulfur (S) | 1.7 | 0.4 (25) | 20.4 - 25.5 | 4.8 - 6.0 |
| Chlorine (Cl) | 1.6 | 1.4 (89) | 19.2 - 24 | 16.8 - 21 |
| Calcium (Ca) | 24.9 | 0.03 (0.1) | 299 - 373 | 0.36 - 0.45 |
| Potassium (K) | 18.1 | 8.8 (49) | 217 - 271 | 106 - 132 |
| Phosphorus (P) | 9.1 | 0.5 (6.0) | 109 - 136 | 6.0 - 9.0 |
| Magnesium (Mg) | 5.1 | 0.08 (1.5) | 61.2 - 76.5 | 0.95 - 1.18 |
| Iron (Fe) | 1.9 | - | 22.8 - 28.5 | - |
| Lead (Pb) | <0.01 | - | <0.1 | - |
| Cadmium (Cd) | <0.001 | - | <0.01 | - |
| Mercury (Hg) | <0.0001 | - | <0.001 | - |

* Assuming the range of soil bulk density of 1.2 - 1.5 g $cm^{-3}$ for top 10cm soil

We have calculated that an application rate of 1% (w/w) biochar to the top 10 cm of soil on a hectare basis is equivalent to an amount of approximately 12 t in clay and 15 t in a sandy soil assuming a bulk density of 1.2 and 1.5 g $cm^{-3}$, respectively. Addition of 12 - 15 t of biochar in the soil means an input of 109 - 136 kg P and 348 - 435 kg N to soil (see Table 1), which is in agreement with other studies (Kishimoto and Sugiura 1985; Atkinson et al. 2010). The amount of WSP added with biochar was 6.0 - 9.0 kg $ha^{-1}$.

*Phosphorus in soils before and after biochar addition*

Background concentrations of WSP in soils ranged from 0.06 to 6.03 mg P $kg^{-1}$ soil, and AL extractable P concentrations ranged from 33 to 139 mg P $kg^{-1}$ soil (see Table 2). Soil organic C contents varied between 2.2% and 11.6%. Concentrations of strongly bound P (2 M HCl - P) ranged from 302 to 1017 mg P $kg^{-1}$ soil and were positively correlated with clay content ($R^2$ adjusted = 0.62, $p$ = 0.002; n=11). In the initial screening experiment, biochar addition increased WSP concentration in three soils due to direct addition of WSP from the biochar. However, addition of increasing amounts of biochar (i.e. control, 0.5, 1, 2 and 4%) did not result in a corresponding increase of WSP. The highest WSP concentrations were observed at a rate of 1 % biochar addition, thereafter, a significant ($p < 0.01$) decrease was observed when the rates of biochar application were increased from 1% to 4% in conjunction with a pH increase of 0.3 - 0.7 units.

Applying 1% biochar resulted only a slight shift in soil pH (0.1 - 0.2 units). The effect of different amount of biochar addition on soil WSP concentration and pH differed between soils, probably due to variations of the soil properties mentioned in Table 2. Similar to our findings, Wang et al. (2011) and Yao et al. (2011) also reported small increase of soil pH after biochar addition varying among soils.

Table 2: Properties of the soils: water soluble phosphorus (WSP); phosphorus, aluminium, iron and calcium in ammonium acetate lactate extract (extractable P, Al, Fe and Ca); degree of P saturation (DPS); strongly bound P (2M HCl - P); and organic carbon (C)

| Name of the soil and texture | Clay | WSP | Extractable P | 2M HCl - P | DPS | Extractable Al | Extractable Fe | Extractable Ca | pH | C |
|---|---|---|---|---|---|---|---|---|---|---|
| | % | | | mg $kg^{-1}$ | % | | mg $kg^{-1}$ | | | % |
| Skottorp *Silt loam* | 8 | 2.76 | 72 | 303 | 13 | 422 | 155 | 1590 | 6.4 | 3.1 |
| Hassla *Silt loam* | 8 | 1.11 | 88 | 332 | 12 | 564 | 209 | 1026 | 6 | 5.5 |
| Röbäcksdalen *Loam* | 10 | 0.66 | 114 | 490 | 12 | 440 | 740 | 1720 | 6.2 | 5.2 |
| Näsbygård *Loamy sand* | 11 | 4.74 | 83 | 302 | 26 | 178 | 200 | 1794 | 6.6 | 2.2 |
| Flinkesta *Loam* | 22 | 3.96 | 33 | 359 | 7 | 266 | 278 | 1094 | 5.4 | 3.3 |
| Östergötland *Clay loam* | 32 | 0.15 | 67 | 606 | 19 | 190 | 247 | 31596 | 7.7 | 2.9 |
| Östergötland *Silty clay loam* | 35 | 1.2 | 139 | 811 | 14 | 533 | 721 | 4604 | 6.2 | 8.0 |
| Flinkesta *Clay loam* | 38 | 6.03 | 34 | 531 | 7 | 220 | 364 | 1584 | 5.9 | 3.8 |
| Uppsala *Clay loam* | 39 | 0.06 | 51 | 694 | 4 | 665 | 986 | 5013 | 5.9 | 11.6 |
| Uppsala *Silty clay* | 55 | 0.63 | 81 | 1017 | 3 | 1366 | 1446 | 6809 | 5.3 | 3.4 |
| Södermanland *Clay* | 66 | 0.12 | 71 | 692 | 4 | 940 | 1233 | 1786 | 5.3 | 8.0 |

In the main experiment, applying 1% biochar to all soils, an increase of WSP ranging from 11% to 253% (Table 3) was found. However, in some soils, a relative decrease of 5% or no changes was also measured as compared to the untreated soils. In fact, when the expected increase of WSP in soils upon biochar addition was calculated, we found that a major portion of WSP (33 - 100%) of the biochar was retained (Table 3) upon mixing with soil.

Table 3: Changes of water soluble P (WSP) in soils after addition of 1% biochar to each soil; values within parentheses express the percentage increased after biochar addition

| Name of the soil and texture | WSP, soil only | WSP measured, soil + 1 % biochar* | Portion of WSP from biochar retained in soil |
|---|---|---|---|
| | mg kg$^{-1}$ | | % |
| Skottorp *Silt loam* | 2.76 | 4.43 (61) | 69 |
| Hassla *Silt loam* | 1.11 | 1.55 (40) | 92 |
| Röbäcksdalen *Loam* | 0.66 | 0.95 (44) | 95 |
| Näsbygård *Loamy sand* | 4.74 | 5.25 (11) | 91 |
| Flinkesta *Loam* | 3.96 | 7.61 (92) | 33 |
| Östergötland *Clay loam* | 0.15 | 0.53 (253) | 93 |
| Östergötland *Silty clay loam* | 1.2 | 1.14 (-5) | 101 |
| Flinkesta *Clay loam* | 6.03 | 7.23 (20) | 78 |
| Uppsala *Clay loam* | 0.06 | 0.09 (50) | 100 |
| Uppsala *Silty clay* | 0.63 | 1.17 (86) | 90 |
| Södermanland *Clay* | 0.12 | 0.12 (0) | 100 |

*Water soluble P added through 1% biochar was 5.43 mg kg$^{-1}$

*Phosphorus retention in biochar-amended soils*

To find out whether there was any relationship between retention of WSP and soil parameters, AL extractable Al, Fe, and Ca, clay content, organic C, DPS and pH were correlated with retained portion of WSP. Pierzynski et al. (2005), Brady and Weil (2002), Hinsinger (2001) and others described retention of WSP in soils through adsorption to clay minerals, organic matter, and $Al^{3+}$, $Fe^{2+/3+}$ and $Ca^{2+}$ sites. Parvage et al. (2011) found a large potential for P retention in soils having low degrees of P saturation (DPS) after after adding soluble P to the soils. Nonetheless, no significant correlations were observed between the retained amount of WSP added through biochar and the soil properties tested. Only a weak correlation was found using all soil parameters as predictor variables against the WSP in a linear regression model ($R^2$ adjusted = 0.42, $p$ = 0.232; n=11) indicating that adsorption of P to soil binding sites might not be the main mechanism for WSP retention in soil amended with biochar.

Another possible retention mechanism for P in biochar amended soil is through binding to positively charged metal complexes formed on biochar surfaces (Beaton et al. 1959; DeLuca et al. 2009). A third possible mechanism could be precipitation of WSP with $Ca^{2+}$ (Hedley and McLaughlin 2005; Ulén and Snäll 2007) and, $Mg^{2+}$ (Sales et al. 1992; Rahaman et al. 2008) present in the solution coming from the dissolution of ash (Erich 1991; Etiégni and Campbell 1991) in the biochar. However, the actual concentrations of soluble Ca and Mg in the water extract of biochar (Table 1) were far below the precipitation limit for any kind of Ca-P (or Mg-P) compounds (Lindsay 1979).

A further reaction that could affect WSP concentration in biochar-amended soil is that Ca, Mg and K ions added with biochar may cause flocculation of colloidal soil P. As a consequence, larger soil colloids formed may not pass through the 0.45 µm filter whereby lower WSP concentrations are measured (Koopmans et al. 2005). The decrease in WSP in soil at increasing rates of biochar addition may also be explained by the flocculation mechanisms as the flocculation conditions for colloidal P were favored by adding more of those cations through more biochar, which also resulted in an increase of bulk soil pH by 0.3-0.7 units.

## Conclusions

This study showed that (i) wheat residue char can act as a source of soluble P although a major fraction of WSP in biochar was retained in soil; (ii) two mechanisms seem to be responsible for P retention - adsorption on soil P binding sites and flocculation of P through cations added with biochar and; (iii) biochar acted as a sink instead of a source of P when applied at high quantity (2 - 4% of soil weight for 0-10 cm).


## Acknowledgement

The project was partly financed by The Royal Swedish Academy of Agriculture and Forest research grant.


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