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Leaching and fractionation of heavy metals in mining soils amended with biochar

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ABSTRACT

Contaminated mining soils might pose risk to the environment due to leaching of heavy metals into ground water, especially under acid conditions. Biochar might be an option for remediation of contaminated mining soils. The aim of this study was to evaluate the effect of soil acidification and biochar (BC) application on Cd, Pb and Zn mobility and chemical fractionation in two mining soils (A and B). Fifteen leaching columns per soil were packed, applying the following treatments: soil + 3% BC; acidified soil; acidified soil + 3% BC and control soil + $CaCO_3$. The control treatment was constituted by the original polluted soil (soil A or soil B). BC was produced by slow pyrolysis at 700 °C from sugarcane straw. Ten leaching events were performed and Cd, Pb, Zn and pH were determined for each leaching. A sequential extraction procedure was performed after leaching was finished. The following fractions were evaluated: mobile, exchangeable, bound to Mn oxide, bound to OM, bound to Fe oxide and residual. In the polluted control soil metal mobility was very low. In acidified soils metal mobility increased drastically and BC application reduced the Cd (57–73%), Pb (45–55%) and Zn (46%) concentrations in the leachate. With the sequential extraction it was observed that Cd was mostly bound to the mobile fraction (15–35%) and exchangeable fraction (7–38%), Pb was mainly bound to the Mn oxide fraction (32–70%) and OM (31–43%) and Zn was principally bound to the residual fraction (31–68%). Cadmium presented the highest mobility index (33–53%) and Pb the lowest (1–3%), which is related to the fractions they are bound in the soil. Field studies would be necessary to confirm the effectiveness of BC application on heavy metals' retention, prior to large scale application.

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1. Introduction

Former mining areas are an environmental concern due to the high levels of potentially toxic elements, such as cadmium, lead, zinc, etc. These inorganic contaminants might spread to surrounding environments through wind erosion (Quinton and Catt, 2007), leaching to groundwater or even to surface water. An environmentally friendly alternative for reclaiming such areas is phytostabilization, which is usually complicated in such environments due to several factors. Besides phytotoxicity by heavy metals, other factors such as extreme pH values, low fertility, low water-holding capacity and poor soil structure prevent plant establishment (Fellet et al., 2011; Mendez and Maier, 2008).

Several techniques have been used for land reclamation, such as liming (Tlustoš et al., 2006), incorporation of organic amendments (Clemente and Bernal, 2006) and most recently the use of biochar has been suggested as an amendment for *in situ* stabilization of heavy metals in contaminated sites (Beesley et al., 2011; Lu et al., 2014; Paz-Ferreiro et al., 2014; Uchimiya et al., 2010). Biochar is the solid material produced by pyrolysis of carbon rich residues. The use of biochar for in situ heavy metal remediation is very attractive due to its low cost, particularly if organic biomass that would be disposed is used for its production, and due to its relative stability in the environment, which could render metal immobilization for longer periods as compared to other organic materials (Beesley et al., 2011). The primary mechanisms of metal immobilization by biochar in soils include an increase in soil pH, ion exchange, physical sorption and precipitation as oxi-hydroxides, with

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carbonate or phosphate (Cao et al., 2009; Park et al., 2011; Uchimiya et al., 2010).

Soil pH is one of the main soil attributes controlling metal mobility in contaminated sites (e.g. abandoned mine areas) either by direct proton (H$^+$) competition on colloids surface or by influencing other soil properties [e.g. CEC (cation exchange capacity), PZC (point of zero charge, etc.]. Some mine areas have naturally near neutral pH (Fellet et al., 2011), which is expected to result on low metal mobility. However, soil pH is subjected to temporal variations. As an example, the use of ammonium fertilizers, the introduction of nitrogen fixing species for revegetation purposes and acid rain could all lower soil pH values, resulting in increased mobility of metals and associated environmental risks.

The mobility of heavy metals in biochar amended soils has been a recent subject of research, both in the field and under laboratory conditions (Paz-Ferreiro et al., 2014). Column leaching studies with biochar addition to heavy metal contaminated substrates have been used in the attempt to predict its effect on metal mobility in the field. For instance, Beesley and Marmiroli (2011) observed sorption of Cd and Zn to biochar's surfaces assisted a 300 and 45-fold reduction in their leachate concentrations, respectively, in a contaminated soil with pH 6.2. However, in some cases the opposite behavior has been also reported. Kelly et al. (2014) found that biochar incorporation into acidic mine materials ($pH \approx 3.0$) caused an increase of Cd and Zn concentrations in the leachate. Thus, these authors recommend that biochar should be carefully evaluated for each specific site prior to large-scale application in order to prevent possible negative impacts on water quality.

In Brazil, an important zinc mining area is located in Vazante, Minas Gerais state. This area is a non-sulfide zinc deposit (Hitzman et al., 2003) where much higher than background concentrations of Zn, Pb and Cd have been recently reported in soils from the area (Lopes et al., 2015). Column leaching studies are an important tool to evaluate metal mobility in soils, but it is a limited technique to infer the metal distribution among soil fractions. Therefore, sequential extraction methods, based on the general principle of extracting a soil sample with chemical solutions of increasing extraction strength, may provide useful information on metal distribution in different soil phases (Sparks, 2003). The aims of this study were: (i) to evaluate the effect of biochar incorporation into a contaminated mine soil, under natural and intentionally acidified conditions, on Zn, Pb and Cd leaching in columns; (ii) to evaluate the effect of the treatments on the distribution of these metals among soil phases, after the last leaching event.

2. Material and methods

2.1. Soil collection and characterization

Two soil samples, named Soil A and Soil B, were collected in a former zinc mining area in the city of Vazante, Minas Gerais state, Brazil (17°55′43″S; 46°49′15″W). The soil samples were selected based on differences in properties (i.e. soil color, texture and total concentration of heavy metals) and landscape position in order to represent the mining area. The area is an opencast mine, whose parent material is composed of phyllite with the presence of hematite, intercalations of shale and red phyllite (strong oxidation). The soils are classified as Technosol (IUSS/WRB, 2014). Soil samples were collected at 0–20 cm depth, air dried and passed through a 4-mm sieve. After homogenization subsamples were taken and passed through a 2-mm sieve for chemical and physical analysis.

The soil samples characteristics are shown in Table 1. Soil fertility testing was performed according to Raij et al. (2001), while pH was determined in a CaCl$_2$ solution (0.01 mol L^{-1}). Soil organic matter was determined colorimetrically after oxidation with sodium dichromate (Na$_2$Cr$_2$O$_7$.2H$_2$O). P, K, Ca and Mg were extracted by mixed cationic and anionic exchange resin (Raij et al., 1986); S was extracted by Ca$_3$O$_8$P$_2$. Al was extracted by KCl 1 mol L^{-1} and determined by titration with NaOH. Potential acidity (H + Al) was estimated by SMP (Shoemaker-MacLean-Pratt) buffer solution. Cation exchange capacity (CEC) was estimated by the sum of bases (Ca + Mg + K) and potential acidity; available micronutrients and heavy metals (Cd and Pb) were extracted by DTPA pH 7.3 (Lindsay and Norvell, 1978). The analysis of semi-total and total metal contents were carried out by using the EPA 3051A (USEPA, 2007) and EPA 3052 (USEPA, 1994) methods, respectively.

2.2. Biochar production and characterization

Sugarcane straw was used as feedstock for biochar (BC) production, as described in a previous study where BC prepared at temperatures of 400, 500, 600 and 700 °C were tested for retention capacity of Cd and Zn (Melo et al., 2013). In that study, the highest immobilization was observed in BC prepared at 700 °C and this BC was, therefore, used in the present experiment.

BC sample characterization is shown in Table 1. The sample pH and electrical conductivity (EC) were measured in a 1:10 (solid: solution ratio) extract after shaking with deionized water for 30 min and 24 h, respectively (Singh et al., 2010). The ash and

Table 1
Soil and biochar characteristics.

Soil attributes	QRV	Soil A	Soil B	Biochar attributes	
pH CaCl$_2$ 0.01M	–	5.8 ± 0.1	6.1 ± 0.1	Yield (%, w/w)	31 ± 1.0
SOM (g kg^{-1})	–	7.0 ± 0.5	7.0 ± 0.1	pH (1:10) in H$_2$O	10.2 ± 0.1
CEC (mmol$_c$ kg^{-1})	–	69 ± 13.5	97 ± 6.2	EC (dS m^{-1})	0.05 ± 0.0
Ca^{2+} (mmol$_c$ kg^{-1})	–	25 ± 4.6	38 ± 2.2	CEC (mmol$_c$ kg^{-1})	14 ± 2.0
Mg^{2+} (mmol$_c$ kg^{-1})	–	32 ± 8.7	50 ± 4.0	SA (m^2 g^{-1})	5.0 ± 0.6
Zn total[*] (mg kg^{-1})	60.0	2906 ± 15.8	2027 ± 14.1	C (%, w/w)[*]	69 ± 0.5
Zn semi-total[**] (mg kg^{-1})	–	698 ± 1.6	1065 ± 107	H (%, w/w)[*]	2.2 ± 0.1
Zn available[***] (mg kg^{-1})	–	56 ± 0.2	60 ± 1.3	N (%, w/w)[*]	0.9 ± 0.1
Pb total[*] (mg kg^{-1})	17.0	5700 ± 349	3688 ± 16.2	Ash (%, w/w)	13.4 ± 0.1
Pb semi-total[**] (mg kg^{-1})	–	2300 ± 251	2334 ± 165	Ca (g kg^{-1})	7.7 ± 0.3
Pb available[***] (mg kg^{-1})	–	136 ± 1.4	141 ± 4.0	Mg (g kg^{-1})	2.0 ± 0.2
Cd total[*] (mg kg^{-1})	0.5	11,3 ± 0.5	6.4 ± 0.2	K (g kg^{-1})	11.7 ± 0.4
Cd semi-total[**] (mg kg^{-1})	–	8.4 ± 0.3	4.5 ± 0.1	P (g kg^{-1})	0.9 ± 0.1
Cd available[***] (mg kg^{-1})	–	1.2 ± 0.2	0.9 ± 0.1	S (g kg^{-1})	1.9 ± 0.2

[**] EPA 3051a.

[***] DTPA pH 7.3. Values are mean (n = 3) ± standard deviation; EC = electrical conductivity; CEC = cation exchange capacity; SA = surface area (BET-N$_2$ method). QRV = quality reference values (CONAMA, 2009).

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