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Research article

Heavy metals fractionation and desorption in pine bark amended mine soils

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ABSTRACT

The European Community Bureau of Reference method (BCR) was used for evaluating the effects of pine bark amendment (0, 24 and 48 g kg^{-1}) and ageing (1 and 30 days) on Cd, Cu, Ni, Pb and Zn fractionation, on samples from an acid mine soil. In addition, the stirred flow chamber technique was applied for analyzing heavy metals desorption from the unamended and pine bark amended mine soil. When the unamended soil were not subjected to ageing, the added heavy metals were mainly accumulated as soluble fraction (>90% for Cd, Ni and Zn; 71% for Cu; and 45% for Pb). Pine bark amendment and ageing had little effect on Cd, Ni and Zn fractionation, whereas important changes were detected for Cu and Pb in response to both pine bark amendment and ageing (decrease in the soluble fractions, and increase in less mobile fractions). Desorption experiments showed that both pine bark amendment and ageing decreased heavy metals release from the mine soil. The results of this study indicate that pine bark amendment could be used to increase heavy metals retention (especially in the case of Cu and Pb) in acid mine soils, thus reducing the risks of metal transfer to uncontaminated environmental zones.

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1. Introduction

Mining is one of the industrial activities with highest impact on the environment, resulting in pronounced land degradation (Masto et al., 2015). Metalliferous mining activities, especially opencast mining, generate huge amounts of spoil accumulation, often characterized by high heavy metal loads (Venkateswarlu et al., 2016). When mining ceases, some of those dumping sites suffer lack of maintenance, with poor management, input of external contaminants, sometimes through polluted flows (mainly runoff), thus increasing the risk of further contamination of water bodies. Since the metalliferous mining is widely extended along the word (Venkateswarlu et al., 2016), its impacts represents a global problem.

Specifically, mining areas devoted to metal extraction are usually characterized by high metal concentrations (Conesa et al., 2007) and strong acidity (Wong et al., 1998), thus facilitating the mobilization of most heavy metals. Moreover, the tailings are sometimes affected by polluted waters containing high concentrations of metals that cannot be effectively retained. Also, the presence of huge tailing ponds is very frequent in this type of mine exploitations. In areas with high precipitations, these ponds may become full, and waters with high concentrations of a mixture of different heavy metals and low pH values may run off trough the mine dumps and reach surrounding water bodies (Zhang et al., 2016). Therefore, restoration tasks are necessary to avoid heavy metal releases from mine dumps, and to increase the retention of heavy metals in the mine dumps after ponds overflows. The addition of certain materials to increase heavy metals retention within the soil, and/or to avoid their release, could be a good alternative in order to perform successful restoration tasks (Fellet et al., 2011; Abad-Valle et al., 2016; Manzano et al., 2016). Such kind of restoration practices could decrease the risks of environmental pollution derived from mine dumps (Mahar et al., 2015; Núñez-Delgado et al., 2015; Zornoza et al., 2016).

Technologies used to restore mine dumps are based on physical,

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chemical and biological procedures (Anawar, 2015). Physical stabilization methods usually implicate covering mine wastes with inert materials that prevent or hinder the spread of potential contaminants by wind, also reducing risks of water erosion. Chemical procedures essentially promote processes such as precipitation, oxidation-reduction, ion exchange and extraction. However, these methods have significant disadvantages, such as incomplete extraction of metals, special equipment requirements, and sometimes high and costly energy requirements (Anawar, 2015).

In recent years, the use of bio-sorbent materials has been promoted as an alternative to more conventional and costly methods. Low-cost bio-sorbents can effectively retain heavy metals (Akunwa et al., 2014), which is especially interesting in highly degraded areas such as mine tailings (Puga et al., 2016). Many bio-sorbents are rich in organic matter and nutrients, which can facilitate re-vegetation and promote medium-term global restoration of degraded environments. Previous studies have focused on algae (Kratochvil et al., 1998), sawdust (Acar and Malkoc, 2004), eucalyptus bark (Sarin and Pant, 2006), olive wastes (Malkoc et al., 2006; Pagnanelli et al., 2003), and residues from the manufacture of beer (Šillerová et al., 2013). We have previously studied materials such as forest residues (Seco-Reigosa et al., 2013a), mussel shell ash and other waste mixtures (Fernández-Pazos et al., 2013; Seco-Reigosa et al., 2013b; Fernández-Calviño et al., 2016).

Another by-product that can be an interesting bio-sorbent is pine bark. Previous studies showed high ability of pine bark to adsorb heavy metals (Al-Asheh et al., 2000; Ribé et al., 2009; Khokhotva, 2010; Mun et al., 2010; Ribé et al., 2012; Cutillas-Barreiro et al., 2014). However, the impact of pine bark amendments on heavy metals retention has not been tested in particularly vulnerable environments, such as mine soils. Moreover, the possible influence of pine bark on metals immobilization and release in mine soils might be due to its own adsorption capacity, or could be due to changes affecting the distribution of metals, and the latter can be investigated by means of fractionation methods supplemented by desorption studies.

In view of that all, in this work we studied the influence of pine bark amendments (rates of 0, 24 and 48 g kg^{-1}), and ageing (1 and 30 days), on the fractionation of five heavy metals (Cd, Cu, Ni, Pb and Zn) added simultaneously to a mine soil, as well as its influence on the release of these metals. Our working hypotheses is that the pine bark amendment can favor heavy metals retention by means of strong and irreversible bindings, facilitating that heavy metals are incorporated to more recalcitrant fractions, thus decreasing release. This kind of amendment could increase the purging capacity in mine soils, decreasing risks of surface and subsurface water pollution, as well as risks of transfer to plants and to the food chain. The main objectives of this work are to increase knowledge on appropriate recycling of pine bark, and to diminish the vulnerability of certain mine soils with limited capacity of removing heavy metals, using pine bark amendment to hamper the export of pollutants into water bodies or any other environmental compartment.

2. Material and methods

2.1. Soil and pine bark characteristics

The selected soil was sampled from a Cu mine dump area in Touro (Galicia, NW Spain), where intense mining was developed until the early 1980s (Arias et al., 1998). The soil was collected at a 20 cm depth, air-dried and sieved through a 2 mm mesh, and characterized by Fernández-Pazos et al. (2013). The pine bark was supplied by Geolia (Madrid, Spain), powdered (<2 mm), and characterized previously by Cutillas-Barreiro et al. (2014). The soil and pine bark characteristics are shown in Table S1 (Supplementary Material). The selected soil had sandy loam texture, and was classified as a Spolic Technosol (Dystric, Arenic) according to the World reference base for soil resources 2014 (Food and Agriculture Organization, 2015). This mine soil was ultra-acid (aqueous pH 3.0), with low organic carbon content (3 g kg^{-1}), high concentrations of Fe oxides (42 g kg^{-1} of amorphous Fe oxides), and lower concentrations of Mn and Al oxides (<1 g kg^{-1}), and presented a surface area of 27.8 m^2 g^{-1}. The total studied heavy metals concentrations were 773 mg kg^{-1} for Cu, 58 mg kg^{-1} for Zn, 5 mg kg^{-1} for Ni, 4 mg kg^{-1} for Pb, and 0.08 mg kg^{-1} for Cd. The mine soil presented high total concentrations of Fe (13.5%) and relatively low total Al concentrations (1.0%). The clay fraction of the mine soils in the sampling area is dominated by quartz, kaolinite, goethite, hydronium jarosite and metal oxides (Asensio and Covelo, 2015), whereas the concentrations of sulfates range from 1.5 to 5.0 g kg^{-1} (Cerqueira et al., 2012). The pine bark presented a strongly acid pH (4.5) and high organic carbon content (486 g kg^{-1}), with predominance of lignin (47.9%), followed by glucan (18.6%), and with surface area (0.36 m^2g^{-1}) lower than that of the mine soil. Its heavy metal content was quite low, with total concentrations being Cu < 0.1 mg kg^{-1}, Zn 7 mg kg^{-1}, Ni 2 mg kg^{-1}, Pb 0.2 mg kg^{-1}, and Cd 0.1 mg kg^{-1}.

2.2. Experimental design

The mine soil was amended with pine bark by adding 0, 24 and 48 g of pine bark per kg of soil. After that, a heavy metals mixture was added to the soil + pine bark mixtures. We used a heavy metals mixture, instead of individual heavy metal solutions, taking into account that toxic spills from metalliferous mines usually present high concentrations of various heavy metals. These experimental conditions allow the study of eventual differences in metal fractionation and release under competition with other metals. In brief, 2.7 mL of a heavy metal mixture (including Cd, Cu, Ni, Pb and Zn as nitrate salts) were added to 30 mg of each pine-bark/soil mixture (those added with doses of 0, 24, and 48 g kg^{-1}). In the heavy metal mixture, the concentration of each heavy metal was 15.7 mmol l^{-1}, resulting in an added concentration of 1.41 mmol kg^{-1} for each metal. Taking into account the previous contents of each metal in the untreated soils, the final concentrations of each metal in the soil after the addition of the heavy metals mixture were 13.57 mmol kg^{-1} for Cu, 2.30 mmol kg^{-1} for Zn, 1.50 mmol kg^{-1} for Ni, 1.43 mmol kg^{-1} for Pb, and 1.41 mmol kg^{-1} for Cd. All these samples were incubated for 1 and 30 days at soil field capacity (8.5% moisture, w/w), and heavy metal fractionation and desorption experiments were performed on aliquots taken from each sample after each of both ageing periods (incubation time 1 and 30 days). Heavy metals fractionation was also performed on untreated soil samples (without heavy metals addition and without pine bark amendment).

2.3. Heavy metals fractionation

Cd, Cu, Ni, Pb and Zn fractionation was carried out on untreated soils, on soils added with the heavy metals mixture but without pine bark, and on soils added with the heavy metals mixture and with two different doses of pine bark (24 and 48 g kg^{-1}). We used the European Community Bureau of Reference (BCR) method, with the modifications performed by Rauret et al. (1999), giving Soluble, Reducible, Oxidizable and Residual fractions (all of them measured, not estimated). To confirm the accuracy of the extraction procedure and analysis, the certified reference material CRM701 was also analyzed in parallel to the soil samples. The recovery of the elements in the different fractions was between 87 and 108% of the

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