

# Availability and fractionation of Cu, Pb and Zn in an acid soil from Galicia (NW Spain) amended with municipal solid waste compost

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*Fraccionamiento y disponibilidad de Cu, Zn y Pb en un suelo ácido de Galicia (NW España) enmendado con compost de residuos sólidos urbanos*  
*Disponibilidade e fracionamento do Cu, Pb e Zn num solo ácido da Galiza (NW Espanha) corrigido com um composto de resíduos sólidos urbanos*

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## ABSTRACT

The potential availability and chemical fractionation of trace elements in soil after compost addition was studied in a laboratory incubation. An acid agricultural soil, developed on biotitic schists, was amended with two rates of a municipal solid waste compost (3% and 6% weight) or lime (2.5 g CaCO<sub>3</sub> kg<sup>-1</sup>, calculated to raise soil pH to values close to those of the compost-amended soil), and incubated in the laboratory for five months. Compost addition increased the availability of Cu, Pb and Zn (measured in DTPA extract) with respect to the control, whereas lime reduced it. Compost amendment increased soil total Cu, Pb and Zn concentrations, and their chemical distribution, assessed by means of the BCR (Bureau Communautaire de Référence) fractionation scheme, was different for each element. Compost addition increased Pb in the reducible (iron oxide-bound) fraction, Cu in the organic and iron oxide fractions, and Zn in the soluble and reducible fractions. Lime did not change the fractionation or total concentrations of the metals analysed. The most problematic element in the compost-amended soil from an environmental point of view is Zn, because it was found in highly mobile forms, while for Cu and Pb this risk is low.

## RESUMEN

Se estudió la disponibilidad potencial y el fraccionamiento químico de metales traza en el suelo tras la adición de compost en un experimento de laboratorio. Un suelo agrícola ácido, desarrollado sobre esquistos biotíticos, fue enmendado con dos dosis de un compost de residuos sólidos urbanos (3% y 6% en peso) e incubado en laboratorio durante cinco meses. Con fines comparativos, también se estudió el suelo enmendado con una dosis de cal (2,5 g CaCO<sub>3</sub> kg<sup>-1</sup>) calculada para llevar el pH a valores próximos a los del suelo enmendado con compost. La enmienda con compost aumentó la disponibilidad de Cu, Pb y Zn con respecto al suelo sin enmendar, mientras que la cal tuvo el efecto contrario. El compost también incrementó las concentraciones totales de Cu, Pb y Zn, modificando su fraccionamiento químico, mientras que la cal no tuvo efecto en este sentido. La adición de compost aumentó la concentración de Pb en la fracción reducible (unido a óxidos), la de Cu en las fracciones oxidable (unido a materia orgánica) y reducible, y la de Zn en las fracciones soluble y reducible. El elemento más problemático en el suelo enmendado con compost es el Zn, al estar presente en formas de alta movilidad, en contraste con lo que sucede en el caso de Cu y Pb.

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## RESUMO

*Estudámos num ensaio laboratorial a disponibilidade potencial e o fracionamento químico de elementos metálicos vestigiais no solo após a adição de um material compostado. Usou-se um solo agrícola ácido, desenvolvido em xistos de biotite, ao qual se adicionaram duas doses de um composto de resíduos sólidos urbanos (3% e 6% em peso) e que foi incubado no laboratório durante um período de cinco meses. Para comparação, o solo foi igualmente tratado com uma dose de calcário (2,5 g CaCO<sub>3</sub> kg<sup>-1</sup>). A adição de composto aumentou a disponibilidade de Cu, Pb e Zn do solo testemunha, enquanto que o calcário teve o efeito oposto. O composto aumentou também as concentrações totais de Cu, Pb e Zn, modificando o seu fracionamento químico, enquanto que o calcário não produziu qualquer efeito. O composto aumentou a concentração de Pb na fração redutível do solo, o Cu nas frações oxidáveis e redutíveis, e o Zn nas frações solúveis e redutíveis. O elemento mais problemático no solo tratado com o composto foi o Zn, por estar presente sob formas de alta mobilidade, em contraste com o que aconteceu no caso de Cu e Pb.*

## 1. Introduction

The use of organic amendments in agriculture allows the restoration of soil fertility, thus counteracting the organic matter loss that has been recognized as a major process of soil degradation (C.E.C. 2002; Jones et al. 2004; Diacono and Montemurro 2010). As urban waste increases with the rise of world and urban population, composting is a valuable alternative for the conversion of wastes into safe soil amendments (Hargreaves et al. 2008; Barral et al. 2009; Farrell and Jones 2009). Compost amendments have been reported to ameliorate soil physical condition (Deeb et al. 2016; Xin et al. 2016), raise the pH of acid soils (García-Gil et al. 2004) and improve nutrient cycling (He et al. 2001; Barral et al. 2011). However, potential negative effects of some kinds of composts can also exist. This is the case of the high concentrations of trace elements in some composts derived from urban wastes, such as municipal solid waste (MSW) or sewage sludge (Smith 2009; Lopes et al. 2011; Paradelo et al. 2011; Cambier et al. 2014), where Cu, Pb and Zn are usually the most problematic due to their presence in vegetables and other human food materials (Chaney et al. 2001).

The environmental risk derived from the agricultural use of compost relies to a great extent on the amount of trace elements in the compost, as well as on their dynamics once they reach the soil. The behaviour of metals in soil is a function of their concentrations and chemical forms in compost, but also of their interactions with soil components (Smith 2009). In addition, compost modifies both soil organic matter contents and pH, two properties that control the fate of trace elements in soil by affecting their solubility. It is widely recognised that pH is one of the most important soil properties that determines the solubility of metals in soils (Bohn et al. 2001), whereas organic matter has a high reactivity to complex metallic elements by functional groups such as hydroxyl and carboxylic groups (Stevenson 1994). Therefore, metal forms that were previously present in the soil can be modified by the changes of soil physicochemical properties induced by compost addition, and this can affect their mobility and bioavailability. Under these circumstances, the behaviour of trace elements cannot be accurately estimated just from compost analyses, so studies in amended soils become an essential tool with which to understand the potential risks related to the accumulation of trace elements in soils due to this agricultural practice.

### KEY WORDS

Compost, soil pollution, urban wastes, copper, zinc, lead.

### PALABRAS

#### CLAVE

Compost, contaminación de suelos, residuos urbanos, cobre, zinc, plomo.

### PALAVRAS-

#### CHAVE

Composto, contaminação de solos, resíduos sólidos urbanos, cobre, zinco, chumbo.

In this context, the objective of our work was to study the potential risk related to trace elements entering the soil due to compost amendment. For this, a laboratory incubation was performed using a representative agricultural acid soil from the Atlantic region of Spain, which was amended with two doses of a MSW compost. Fractionation and availability of Cu, Pb, and Zn (the most commonly problematic metals in compost) were studied throughout the incubation by means of chemical analyses.

## 2. Materials and methods

### 2.1. Soil and compost

The soil used in the experiment was an agricultural soil developed over biotite-rich schists, located in the town of Ordes (near Santiago de Compostela, Spain, 43°N 8°W), 180 m above mean sea level, in an oceanic climate (mean annual temperature 12.3 °C; mean annual rainfall 1900 mm). Currently, arable soils in the region alternate between

forage (ryegrass and white clover) and agricultural (maize and wheat) crops, the most common rotation being a forage crop in winter and maize in summer (Verde et al. 2005). As is usual for soils of the region, it has an acid pH and high organic carbon concentration (Table 1). Similar soils in the area are classified as Haplic Umbrisols (IUSS Working Group WRB 2014) or Humic Dystrudepts (Soil Survey Staff 2006).

The compost employed was an aerobic compost obtained from the source-separated organic fraction of MSW, provided by an industrial composting facility in Galicia (Spain). It has an alkaline pH and Cu, Pb and Zn concentrations (Table 1) that are higher than the threshold levels allowed in the Spanish regulation for organic amendments (Spanish Ministry of Agriculture 2005).

### 2.2. Incubation with compost

For the experiment, a 30-kg composite sample from the top 10 cm of the Ap horizon of the soil was used. The sieved soil (< 4 mm) was divided in four equal parts (~8 kg each) and mixed by hand with the compost at two rates: 3% and 6% (dry weight), roughly equivalent to 60 and

**Table 1.** Properties of soil and compost. TOC: total organic carbon; EC: electrical conductivity

	pH <sub>w</sub>	EC dS m <sup>-1</sup>	Texture	TOC g kg <sup>-1</sup>	Total N g kg <sup>-1</sup>	C/N	Cu mg kg <sup>-1</sup>	Zn mg kg <sup>-1</sup>	Pb mg kg <sup>-1</sup>
<b>Soil</b>	5.7	-	Sandy-clay loam	18.6	2.0	9	22	66	14
<b>Compost</b>	8.7	5.1	-	220	18	12	356	646	200

120 Mg ha<sup>-1</sup>, (treatments S+3 and S+6, respectively), or with 2.5 g kg<sup>-1</sup> of CaCO<sub>3</sub>. This dose of carbonate, needed to raise soil pH to 6.5, was calculated using the incubation method as described by Guitián and Carballas (1976). The fourth part of soil was not amended and used as a control.

For the incubation experiment, 2.5 kg of each mixture and the control soil were placed in PVC pots (three replicates per treatment) and incubated in the laboratory in the dark at 25 °C for 150 days, a longer time than is usual

in incubation studies, intended to simulate a whole agricultural season. Soil moisture was maintained throughout the experiment at around 80% of their field capacity (0.2 g g<sup>-1</sup>), replacing weight losses with distilled water. Every time that the moisture was adjusted, the soil was homogenised and aerated.

Composite samples were taken monthly at random from each pot, air-dried and sieved (< 2 mm) for analysis. Soil pH was determined in a water suspension and in 0.1 N KCl

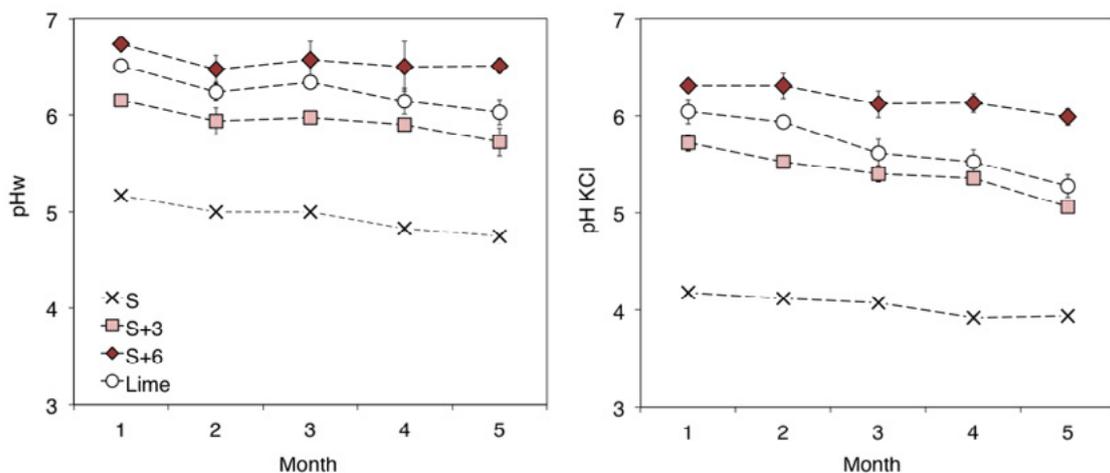
(1:2.5 sample:solution ratio). For the determination of available Cu, Zn and Pb, 20 g of soil were extracted with 40 mL of a 0.05 M diethylene triamine pentaacetic acid (DTPA) + 0.01 M  $\text{CaCl}_2$  + 0.1 M triethanolamine solution adjusted to pH 7.3, following the method by Lindsay and Norvell (1978). The extracts were analyzed for Cu, Zn, and Pb using flame atomic absorption spectrometry (Varian SpectraAA 220FS). Metal distribution in chemical fractions was studied at the end of the incubation using the BCR (Bureau Communautaire de Reference) sequential extraction procedure as described by Rauret et al. (2000) and Pueyo et al. (2003). This method consists of three steps and a residual extraction, and defines four operational fractions: an acid-soluble fraction, a reducible fraction that is mainly associated with iron and manganese oxides, an oxidizable fraction associated with organic matter, and a residual fraction which is associated with silicates and other mineral material. The Cu, Pb, and Zn concentrations in all the BCR extracts were determined by inductively coupled plasma mass spectrometry (VARIAN 820-MS). Blanks were run in parallel to all the determinations. To confirm the accuracy of the extraction procedure and analysis, the certified reference material CRM701 was also analysed in parallel to the soil samples. The recovery of the elements in the different fractions was between 81-109% of the certified values.

### 2.3. Statistics

Linear regression model analysis and one-way ANOVA were performed to assess the effect of the treatments on trace element concentrations; significance of the differences between the treatments and the control soil was assessed by orthogonal contrasts, using the R statistical package for MacOSX (version R 3.1.3; R Core Team 2015).

## 3. Results and discussion

In all treatments, pH decreased progressively throughout the incubation (**Figure 1**), likely an effect of the acidification produced by  $\text{CO}_2$  release during mineralization of labile organic C, as commonly observed in laboratory studies with compost-amended soils (Paradelo et al. 2009). Compost addition increased the soil acidic pH ( $P < 0.001$ ) up to values close to neutrality, with a more marked effect at the highest dose. The values of pH in KCl were in all cases lower than the pH in water, but in the compost-amended soils the differences were lower than in the control soil, an indicative that the amendment reduced the amount of exchangeable



**Figure 1.** Soil pH evolution during the incubation. Error bars represent twice the standard deviation of the mean ( $n = 3$ ). S: control soil; S+3: soil amended with 3% compost; S+6: soil amended with 6% compost. Lime: soil amended with 2.5 g  $\text{CaCO}_3$   $\text{kg}^{-1}$ .

aluminium. The increase of the pH of acid soils after amendment with MSW compost is due to the buffering capacity of organic matter, as well as to the presence of significant amounts of carbonates in MSW (Vassilev et al. 1999; Paradelo et al. 2011). Since soil acidity is one of the main limitations for crop production in extensive areas of the world, the liming capacity of compost should be considered as a major advantage of compost use in acid soils.

Both doses of compost increased the availability of Cu, Pb and Zn with respect to the control soil, as shown by their DTPA-extractable concentrations, whereas lime produced the opposite effect (Figure 2, Table 2). The decrease of metal availability observed in the limed soil is in agreement with results from other researchers (Khan and Jones 2009), and it is likely to be a consequence of the reduction of metal solubility at increasing pH. This decrease was not observed in the compost-amended soils, despite the increment of pH, due to the metal input of the compost. Extractable Cu and Pb concentrations were lower than the levels usually regarded as potentially toxic (20 mg kg<sup>-1</sup> for both elements), but extractable Zn was close to the concentration of 10 mg kg<sup>-1</sup> that could be toxic for certain crops (Wallace and Wallace 1994; O'Sullivan et al. 1997). During the incubation, available Zn and Pb concentrations decreased steadily in the compost-amended soil, suggesting that the interaction of compost with soil components reduced the availability of those elements. Contrary to Pb and Zn, Cu availability increased slightly during the incubation. Increases of Cu mobility in compost-amended soils have been commonly observed (Hernando et al. 1989; Paradelo et al. 2017), which are likely to be a consequence of the formation of soluble complexes of Cu with organic carbon (Temminghoff et al. 1997).

The addition of compost at the highest dose (6%) increased total Cu, Pb and Zn concentrations in the amended soil, modifying their chemical distribution in the BCR fractions compared to control soil, whereas lime and the low compost dose had no effect in any case (Figure 3, Table 2). In the non-amended soil, most Cu and Zn were

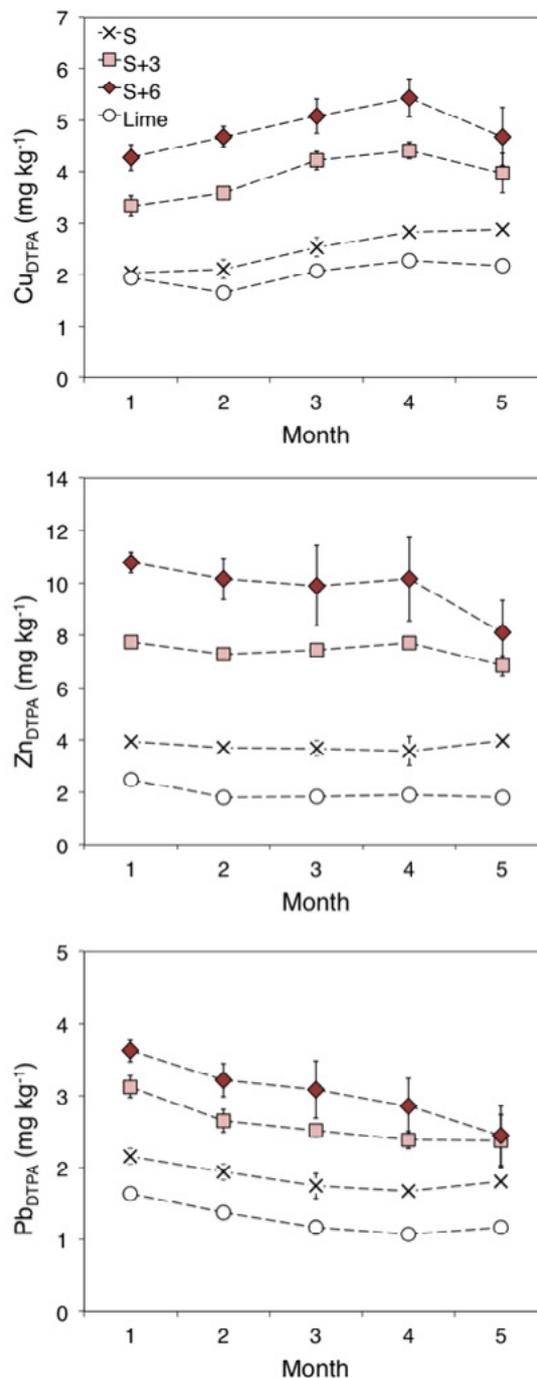


Figure 2. Evolution of DTPA-extractable elements with time. Error bars represent twice the standard deviation of the mean (n = 3). S: control soil; S+3: soil amended with 3% compost; S+6: soil amended with 6% compost. Lime: soil amended with 2.5 g CaCO<sub>3</sub> kg<sup>-1</sup>.

**Table 2.** Significance of differences in metal contents between amended soils and the control soil. S+3: soil amended with 3% compost; S+6: soil amended with 6% compost; Lime: soil amended with 2.5 g CaCO<sub>3</sub> kg<sup>-1</sup>. Significance is indicated as follows: ns: not significant; \* significant at a *P*-value of 0.05; \*\* significant at a *P*-value of 0.01; \*\*\* significant at a *P*-value of 0.001

	Total Cu	Cu <sub>DTPA</sub>	Soluble Cu	Reducible Cu	Oxidable Cu	Residual Cu
<b>S+3</b>	ns	< 0.001***	ns	0.004**	< 0.001***	ns
<b>S+6</b>	0.02*	< 0.001***	0.017*	< 0.001***	< 0.001***	ns
<b>Lime</b>	ns	0.004**	< 0.001***	ns	0.03*	ns
	Total Zn	Zn <sub>DTPA</sub>	Soluble Zn	Reducible Zn	Oxidable Zn	Residual Zn
<b>S+3</b>	ns	< 0.001***	0.007**	0.03*	< 0.001***	ns
<b>S+6</b>	0.01*	< 0.001***	< 0.001***	< 0.001***	0.006**	ns
<b>Lime</b>	ns	< 0.001***	ns	ns	0.015*	ns
	Total Pb	Pb <sub>DTPA</sub>	Soluble Pb	Reducible Pb	Oxidable Pb	Residual Pb
<b>S+3</b>	ns	< 0.001***	0.026*	0.02*	0.02*	ns
<b>S+6</b>	0.006**	< 0.001***	0.048*	0.0006***	ns	ns
<b>Lime</b>	ns	< 0.001***	ns	ns	ns	ns

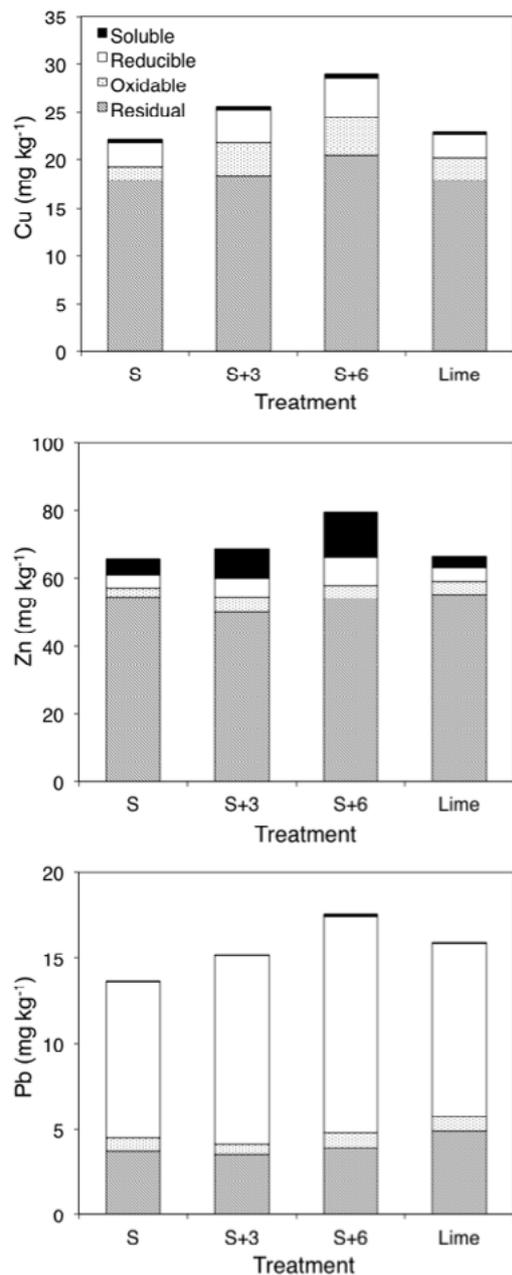
found in the residual fraction (~80% of the total), whereas Pb was mainly (65% of the total) in the reducible fraction. Compost addition modified these chemical distributions in some cases, and in a different way for each element studied. Both doses of compost increased Cu concentrations in the soluble, reducible and oxidizable fractions, and the highest dose also increased Cu in the residual fraction. The fraction that increased most for Cu was the organic-bound, as expected given the high affinity of this element for organic matter (Stevenson 1994). This preferential association of Cu to the organic-bound fraction has been observed in composts (Greenway and Song 2002; Paradelo et al. 2011) and compost-amended soils (Zheljazkov and Warman 2004).

Compost increased the concentrations of Zn in the soluble, reducible and oxidizable fractions at both doses in comparison with the control, and the highest increase was clearly that of the soluble fraction (Figure 3, Table 2). This is in agreement with most studies in the literature, where Zn is commonly the element where the largest increase in labile forms is reported when compost is applied to soil, particularly under

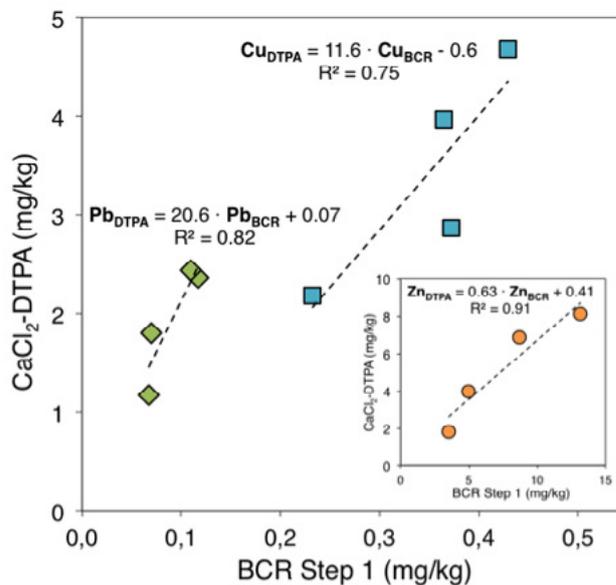
acidic soil conditions (Planquart et al. 1999). In the case of Pb, compost addition increased its concentration in the soluble and reducible fractions, especially in the latter, compared with the control soil. The fate of Pb derived from compost in soils is highly variable according to the literature, and while some authors have observed that Pb increases in the reducible fraction (Achiba et al. 2010), most studies point at a preference of Pb for organic-bound forms (Planquart et al. 1999; Businelli et al. 2009).

Finally, we have compared the amounts of elements extracted by DTPA and the first extraction of the BCR procedure (diluted acetic acid) in order to delve into the interpretation of the sequential extraction in terms of availability (Figure 4). Although both extractions are aimed at quantifying the elements that are more readily available, their results are similar only in the case of Zn. In turn, the amounts of Cu and Pb extracted by DTPA are 10-20 times higher than those extracted by the first step of the BCR procedure. Given that DTPA solubilises elements held as sorbed and organically bound phases, the differences between both extractions mean

that there is a significant fraction of Cu and Pb associated to organic matter that cannot be mobilised by the mild acid extraction of the BCR procedure.



**Figure 3.** Total concentrations and BCR fractionation of Cu, Pb and Zn in the soils after five months of incubation. S: control soil; S+3: soil amended with 3% compost; S+6: soil amended with 6% compost. Lime: soil amended with 2.5 g CaCO<sub>3</sub> kg<sup>-1</sup>.



**Figure 4.** Comparison of acetic acid-soluble elements (BCR Step 1) and Cl<sub>2</sub>Ca-DTPA-extractable elements at the end of the incubation.

## 4. Conclusions

Overall, this study showed that the MSW compost applied has a liming effect on acid soils, being able to raise soil pH in more than one unit at 3-6% doses. This effect must result in a reduction of Cu, Pb and Zn availability by decreasing their solubility as pH increases. However, compost addition promoted an increase of available Cu, Pb and Zn in the soil, even though pH values were comparable to the limed soil (for which metal availability was lower). This shows that the effect of higher pH was counteracted by the metal input coming from the MSW compost itself. The chemical fractionation of the metals in the soils showed that compost addition increased specially Pb in the reducible (iron oxide-bound) fraction, Cu in the oxidizable (organic matter-bound) and reducible fractions, and Zn in the soluble fraction, which is the most bioavailable. For this reason, Zn is the most problematic element regarding the potential environmental risk posed by MSW compost amendments in this study.

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