



Chemical characterization and evaluation of composts as organic amendments for immobilizing cadmium

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ABSTRACT

The ability of three composted materials to immobilize cadmium (Cd) was examined in order to assess their potential for recovering soils contaminated with this metal. Composted pine bark (PB) pH 5.6, spent mushroom compost (SM) pH 8.0, and composted pruning waste + biosolids (BS) pH 6.9 (containing 81%, 75% and 47% total organic matter, respectively) were characterized. FT-IR and CP-MAS ¹³C NMR spectroscopy indicated the BS and SM to have a higher percentage of aliphatic and carboxyl groups than PB. The composts were artificially contaminated with Cd (80 and 200 mg kg⁻¹) and, after 4 weeks incubation, subjected to sequential extraction. In column leaching experiments, the total Cd leached from the composts exposed to both Cd treatments was similar, but much less leached from the BS (0.2%) than the PB (4.0%) or SM (0.7%). The greater capacity of BS to immobilize Cd was attributed to the greater humification of its organic matter and higher content of inorganic components, particularly Fe.

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1. Introduction

The pollution of soils by heavy metals is a serious environmental problem affecting many territories around the world. In Spain, the soil of some 4532 ha may be contaminated by such metals, excluding those also contaminated with radioactive waste and mine tailings (Bernal et al., 2007). Among the heavy metals, cadmium (Cd) is one of the most toxic to enter the food chain. It is more mobile and more soluble than other metals, and therefore more likely to leach into the deeper layers of the soil, contaminating the groundwater. In situ addition of organic amendments to contaminated soil aims to reduce the availability of heavy metals (Chen et al., 2000; Basta et al., 2001; Li et al., 2001; Bolan et al., 2003; Walker et al., 2003; Lee et al., 2004). Organic amendments provide sites for their adsorption, favoring their immobilization (Shuman et al., 2002).

Composted materials provide a suitable organic amendment for the remediation of soils contaminated with heavy metals (Kiikkila et al., 2001; Shuman et al., 2002; van Herwijnen et al., 2007). Bio-solid composts, manure, peat, paper mill waste have all been used to immobilize Pb, Cd and Zn (Basta et al., 2001; Bolan et al., 2003; Adriano et al., 2004). Although composts – especially biosolids – may contain heavy metals themselves, they can actually reduce the bioavailability of these metals in the soil (Merrington and

Smernik, 2004). Currently there is intense debate surrounding which fraction – organic or inorganic – is responsible for their immobilization (Li et al., 2001; Stacey et al., 2001; Bolan et al., 2003; Hettiarachchi et al., 2003; Merrington et al., 2003; Antoniadis et al., 2007; Kumpiene et al., 2008). However, different materials and technologies used in composting mean that compost variables such as the organic matter, nitrogen, dissolved organic carbon (DOC), nitrate and soluble sugar concentrations, the pH and the electrical conductivity (EC) can vary significantly (Zmora-Nahum et al., 2007). It is therefore difficult to predict compost behavior with respect to the adsorption/mobility of metals.

For an amendment to be effective it must be able to make heavy metals less bioavailable. However, not all organic amendments achieve this (van Herwijnen et al., 2007). The addition of organic matter to soil may increase the DOC, encouraging the mobility of heavy metals (Antoniadis and Alloway, 2002). Some functional groups such as hydroxyl, phenolic, carboxyl and sulphhydryl groups control the adsorption, formation and activity of soil–metal complexes (Lee et al., 2004; Karlsson et al., 2007), with carboxyl groups being the most commonly involved (Alloway, 1995).

The solubility of metal contaminants dictates – at least to some extent – their mobility and bioavailability (Basta et al., 2001). For the study of such mobility, leaching column experiments have proven useful (Eymar et al., 2002). However, few studies have been performed to determine how metals leach from organic amendments (i.e., to assess their ability to immobilize metals) before being applied to soils. The aim of the present study was to

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determine the capacity of composted materials commonly used as organic amendments or growth media (pine bark, spent mushroom and pruning waste + biosolids composts) to immobilize Cd, and to identify the chemical characteristics of these materials influencing metal immobilization.

2. Methods

2.1. Composted materials

Composts produced from various materials were obtained from different composting plants in Madrid, Cuenca and Avila (Spain), respectively:

- Pruning waste + biosolids (BS) (1:3 w/w). This mix is composted in tunnels with forced air for 14 days and matured in the open air. Composting is completed in about 5–6 months.
- Spent mushroom compost (SM). An initial compost is produced from wheat straw (55%), grape marc (15%) and poultry litter (30%) (w/w); this is commonly used as a mushroom (*Agaricus bisporus*) cultivation medium. When such cultivation is finished further straw used in the cultivation of another mushroom (*Pleurotus ostreatus*) is added (30% v/v), and this mixture composted in the open air. Composting is completed in about 6 months.
- Pine bark compost (PB). Pine bark (mix different species of pines: *Pinus sylvestris*, *Pinus pinaster* and *Pinus pinea*) is composted for 4 months in the open air, adding urea and water. The resultant mixture is then milled and sieved and allowed to mature for a further 4 months. Peat and wood fiber (20%) are then added.

2.2. Physico-chemical characterization

Electrical conductivity (EC) and pH were determined in water extracts (1:5 v/v) using a conductivitymeter Crison CM 2200 (Barcelona, Spain) and pH electrode Orion 720A (Beverly, MA, USA), respectively. The bulk density of the samples was determined in stainless steel cylinders by applying a suction pressure provided by a 10 cm column of water using a sandbox Eijkelkamp (Giesbeek, The Netherlands), and then drying the samples at 103 °C. The index particle size was obtained by determining the percentage (by weight) of particles with a diameter of >1 mm using a series of sieves (Filtro®). The concentrations of soluble Ca, Na, Cl⁻ and NO₃⁻ were determined in water extracts (1:5 v/v). Ca and Na were determined by atomic absorption spectroscopy (AAS) using AA Analyzer 800 Perkin Elmer (Waltham, MA, USA). Cl⁻ was measured using a selective electrode (Orion 720A). NO₃⁻ was determined colorimetrically as described by Cataldo et al. (1975) using a Shimadzu UV-160A spectrophotometer (Kyoto, Japan). Total N was determined by the Kjeldahl method in a Block Digest 12 apparatus Selecta (Barcelona, Spain). Total P was determined colorimetrically following the method of Murphy and Riley (1962) after digestion of the samples in *aqua regia* (HNO₃–HCl, 1:3). Total S was determined using a Leco CHNS-932 element analyzer (St Joseph, MI, USA). Total organic matter (TOM) was determined by combustion at 450 °C in a muffle furnace (Select-Horn, P Selecta); total organic carbon (TOC) was calculated as 58% of the TOM value (Walkley and Black, 1947). The effective cation exchange capacity (CEC_E) was determined at the natural pH of the composts using the method of Ansoarena (1994). Humic extract carbon (C_{EXT}) was determined following the method of Stevenson (1994). The C_{EXT}/TOC ratio was calculated to assess the degree of humification of the organic matter (Roletto et al., 1985; Clemente et al., 2006). The hydrosoluble organic carbon was measured spectrophotometrically using the

method of Hernández et al. (2007). The total concentrations of heavy metals (Cd, Cu, Fe, Mn, Pb and Zn) were determined by AAS after that the samples were dried (55 °C), sieved (<63 µm) and digested with *aqua regia* (HNO₃–HCl, 1:3) (McGrath and Cunliffe, 1985). All determinations were performed in quadruplicate.

2.3. Spectrometric characterization

All composts were subjected to Fourier-transformed infrared spectroscopy (FT-IR) using an IFS60v spectrometer Bruker (Billerica, MA, USA); readings were obtained in arbitrary units of diffuse reflectance. The composts were also subjected to cross-polarization and magic angle spinning nuclear magnetic resonance (¹³C-CP-MAS NMR) in a Bruker AV-400-WB apparatus (Billerica, MA, USA) (rotor spin rate 14 kHz; contact time 3 ms; acquisition time 2 s; 28,500 scans). Samples were packed in 4 mm zirconia rotors with Kel-F caps. The pulse sequence was applied with a ¹H ramp. Chemical-shift (δ) regions were divided into the main resonance regions according to Stevenson (1994). Relative areas were obtained from the integration of these regions.

2.4. Sample pretreatment

The pine bark (PB), spent mushroom (SM) and pruning waste + biosolids composts (BS) were artificially contaminated with Cd (CdSO₄·8/3H₂O) in the following manner: (1) 0 (control), (2) 80 mg Cd kg⁻¹ (80 Cd), or (3) 200 mg Cd kg⁻¹ (200 Cd) dry matter. Samples were incubated for 4 weeks under laboratory conditions (~18 °C and 38% relative humidity [RH]). All samples were shaken periodically. The moisture level was maintained at about 70% of the container capacity.

2.5. Cd extraction (DTPA-CaCl₂)

Extractable Cd was determined in 1:5 (v/v) DTPA-CaCl₂ (pH 2.6; DTPA concentration 0.002 M) extracts of fresh samples of compost BS, SM and PB (200 Cd treatment). All extracts (four replicates per sample) were shaken for 1 h and filtered through a 0.45 µm microfilter. Cd was determined by AAS.

2.6. Sequential extraction of Cd

Sequential Cd extraction was performed on the control and 200 Cd PB, SM and BS composts (four replicates per sample), following the method of Tessier et al. (1979). This provides the distribution of Cd in the FI fraction (the soluble and/or interchangeable fraction), the FII fraction (associated with carbonates and/or acid soluble), the FIII fraction (associated with Fe and Mn oxides), the FIV or oxidizable fraction (associated with organic matter), and the FV or residual fraction. The Cd content of every fraction was determined by AAS.

2.7. Column leaching

Seven hundred and fifty milliliters of the control, 80 Cd- and 200 Cd-treated PB, SM and BS composts were placed in methacrylate columns (50 cm high by 6 cm in diameter) attached to Büchner funnels, as described in Eymar et al. (2002) (four replicates per treatment). The columns were saturated with distilled water before irrigating; all were maintained under laboratory conditions (~18 °C, 38% RH) throughout the assay. Distilled water (100 mL, pH 6.0) was poured daily into each column for 30 days. Leachates were collected daily to determine their volume, pH, and Cd concentration by AAS.

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