



Increased zinc and copper availability in organic waste amended soil potentially involving distinct release mechanisms[☆]



Marie Tella ^{a, b}, Matthieu N. Bravin ^c, Laurent Thuriès ^c, Patrick Cazevieille ^a,
 Claire Chevassus-Rosset ^a, Blanche Collin ^{b, d}, Perrine Chaurand ^b, Samuel Legros ^a,
 Emmanuel Doelsch ^{a, *}

^a CIRAD, UPR Recyclage et risque, F-34398, Montpellier, France

^b Aix-Marseille Univ., CNRS, IRD, CEREGE UMR 7330, F-13545, Aix en Provence cedex 4, France

^c CIRAD, UPR Recyclage et risque, F-97408, Saint-Denis, Réunion, France

^d CEA, IBEB, Laboratory of Microbial Ecology of the Rhizosphere and Extreme Environments, Saint-Paul-lez-Durance, F-13108, France

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ABSTRACT

This study aimed at determining the fate of trace elements (TE) following soil organic waste (OW) application. We used a unique combination of X-ray absorption spectroscopy analyses, to determine TE speciation, with incubation experiments for *in situ* monitoring of TE availability patterns over a time course with the technique of the diffusive gradients in thin films (DGT). We showed that copper (Cu) and zinc (Zn) availability were both increased in OW-amended soil, but their release was controlled by distinct mechanisms. Zn speciation in OW was found to be dominated by an inorganic species, i.e. Zn sorbed on Fe oxides. Zn desorption from Fe oxides could explain the increase in Zn availability in OW-amended soil. Cu speciation in OW was dominated by organic species. Cu release through the mineralization of organic carbon from OW was responsible for the increase in Cu availability.

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1. Introduction

The world population has grown substantially over the past century, while becoming more urban. Waste production has in turn risen tenfold and is expected to double again by 2025 (Hoorweg et al., 2013). There is now a global consensus on the necessity to recycle resources to ensure sustainable development (Lehmann, 2013; Schiermeier, 2013). Organic wastes (OW) can be effectively recycled by spreading them on cultivated soils, with many beneficial effects, such as: (i) improving the soil physical properties; (ii) providing major nutrients, thereby reducing the use of inorganic fertilizers, and (iii) improving soilborne microflora and fauna activity by providing organic carbon. These advantages should be assessed together with the potential (eco)toxicological impacts due to the presence of trace elements (TEs) and other contaminants in OW. Many studies have highlighted that OW application in crop

fields can drastically boost soil TE levels (Belon et al., 2012; Hargreaves et al., 2008; McBride, 2003; Nicholson et al., 2003). Zinc (Zn) and copper (Cu) account for the highest TE inputs in agricultural soils in many countries. Livestock manure is always identified as the major source of these TEs (Belon et al., 2012). Although the presence of TEs in OW has been demonstrated in many studies, major critical knowledge gaps must be overcome to be able to predict the fate of these potentially toxic compounds following OW application. The first major limitation is the lack of speciation data on TEs in OW. To date, most results concerning TE speciation in OW are inconsistent due to several factors. First, OW are complex matrices composed of different organic and/or inorganic phases (solid and liquid for manure) (Tella et al., 2013). Second, there is still no fully reliable benchmark method available to study TE speciation in such complex materials. Chemical extraction methods are often used but are criticized due their lack of clear chemical selectivity and the variability in the results obtained according to the protocols used (Doelsch et al., 2006b, 2008; Romeo et al., 2014). In order to overcome this problem, X-ray absorption spectroscopy (XAS) has been used to describe the speciation of target elements. XAS is sensitive to the oxidation state as well as the

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* Corresponding author.

E-mail address: doelsch@cirad.fr (E. Doelsch).

Table 1
Organic waste and soil characteristics.

Sample ID	Type of organic wastes and geographical origin	Origin		Treatment	Zn mg kg ⁻¹	Cu mg kg ⁻¹	C Organic % Mass	pH 1:2 soil:Water ratio
		Geographical	Production and type of soil					
OW1	Fine fraction from municipal solid waste compost _ Madagascar (sample ID in Tella et al. (2013): FF-MSWC_M)	Madagascar	Urban	Stockpiled (>10 years)	1900	280	8	7.8
OW2	Fine fraction from municipal solid waste compost _ Senegal (sample ID in Tella et al. (2013): FF-MSWC_S)	Senegal	Urban	Stockpiled (>10 years)	2190	330	9	7.6
OW3	Municipal solid waste compost _ Madagascar (sample ID in Tella et al. (2013): MSWC_M)	Madagascar	Urban	Composted	240	48	16	9.0
OW4	Municipal solid waste compost _ Senegal (sample ID in Tella et al. (2013): MSWC_S)	Senegal	Urban	Composted	100	24	24	8.6
OW5	Sewage sludge _ Senegal	Senegal	Urban	Dried	1000	340	25	6.8
OW6	Manure compost from pig slurry _ France (sample ID in Tella et al. (2013): MC_Pi_F)	France, Réunion	Agricultural	Stockpiled (some months)	700	180	30	8.4
Soil		France, Réunion	Nitisol		170	34	2	5.8

distance, number and nature of atoms immediately surrounding the selected element. This spectroscopy method was used for pig slurry, revealing that Cu is mainly (96%) in sulfide form (Cu₂S) with a Cu(I) oxidation state (Legros et al., 2010a) and that 14% of Zn is present as ZnS (Legros et al., 2010b). The detected presence of sulfide was unexpected and reported for the first time in animal manure. Similar Cu and Zn speciation was recently reported in sewage sludge (Donner et al., 2011). These results are crucial for understanding the fate of Cu and Zn from these OW when applied to agricultural soils (Legros et al., 2013).

The second major limitation is the lack of knowledge on TE dynamics following soil OW application. While some authors have suggested that organic matter is the major sorbent of TEs in OW (McBride, 2003), others have indicated that inorganic constituents (Fe and Mn oxides) play a dominant role (Hettiarachchi et al., 2006) and debate regarding the nature and stability of key sorbent phases is ongoing (Mamindy-Pajany et al., 2014). If the prevailing sorbent is organic matter, then TEs may be progressively released over the time course due to organic compound mineralization by soil micro-organisms. Alternatively, if contaminants are sorbed by inorganic waste constituents, their behavior may change as a result of changing physico-chemical conditions (pH or dissolved organic carbon (DOC) concentration) in OW-amended soils (Doelsch et al., 2009, 2010). Whatever the processes involved, there is a lack of experimental evidence to be able to determine the main processes that control the TE availability in OW-amended soils.

This study aimed at combining TE speciation characterization in OW with TE availability when OWs are applied on soils. XAS analyses were first used to decipher the TE-bearing components within OW. Then an incubation experiment was carried out using one soil and selected OW applied at doses consistent with current agricultural conditions. Mineralization of organic carbon derived from OW, variations in the physicochemical properties of the soil solution (pH, DOC concentration) and TE availability in OW-amended soils were monitored over a time course during an incubation experiment. This combined approach should enable us to identify parameters controlling the fate of trace elements following OW soil application and to contribute to the debate regarding the nature and stability of TE-bearing components during agricultural OW recycling.

2. Experimental

2.1. Organic wastes and soil characteristics

This study was focused on six OW selected among OW

previously studied (Tella et al., 2013). The waste types were: fine fractions from municipal solid waste compost (OW1 and OW2), municipal solid waste compost (OW3 and OW4), sewage sludge (OW5) and pig manure compost (OW6). The characteristics of the six OW are displayed in Table 1. The selected soil was a Nitisol that was sampled in the 0–25 cm horizon at La Mare (20°53'24"S, 55°32'13"E) on the island of Réunion. This soil was selected for its intermediate physicochemical properties (pH = 6, 2.3 g C_{org} kg⁻¹) and its naturally low Zn and Cu concentrations (170 and 34 mg kg⁻¹, respectively). A more complete description of the soil and sampling procedure can be found elsewhere (Doelsch et al., 2010).

2.2. X-ray absorption spectroscopy

Zinc K-edge (9.66 keV) and Cu K-edge (8.99 keV) X-ray absorption spectra for the six OW were recorded on the FAME beamline at ESRF (Grenoble, France) and SAMBA beamline at SOLEIL (Gif-sur-Yvette, France). Unfortunately, Zn and Cu concentrations in OW4 were too low for EXAFS acquisition. Spectra were thus recorded for the fine size-fraction of this waste (0.2–20 µm, see Tella et al. (2013) for details) in which 27 and 36% of total Cu and Zn were recovered, respectively. Spectra acquisition was performed on freeze-dried samples pressed into thin pellets at liquid helium temperature to prevent X-ray beam sample damage. Measurements were carried out in fluorescence mode with a 30-elements solid-state Ge detector. To obtain representative spectra, each scan was focused on a different specimen position. The energy was calibrated using Zn or Cu foils (threshold energy taken at the zero-crossing point of the second derivative spectrum). Each spectrum was the sum of at least three scans. Normalization and data reduction was performed according to standard methods (Doelsch et al., 2006a) using Athena software (Ravel and Newville, 2005).

The Zn and Cu-bearing components in the samples were speciated as follows: (i) principal component analysis (PCA) of EXAFS spectra database was performed to determine the minimum number of components; (ii) using a library of Zn and Cu model compounds, target transformation (SPOIL function) was used to identify the probable components, and (iii) those components were quantified from the experimental EXAFS spectra with least-square linear combination fitting (LCF) (Beauchemin et al., 2002; Manceau et al., 2002). Details on the database of model compounds, PCA, SPOIL function and LCF results are given in Supplementary material (SI-1 and 2). The Zn and Cu K-edge EXAFS spectra, corresponding Fourier transforms and LCF fits are reported in Figs S1, S2 and SI-3.

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