

# Mobility of Cu and Zn in Soil Amended with Composts at Different Degrees of Maturity

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Received: 7 December 2015 / Accepted: 25 July 2016 / Published online: 12 August 2016  
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## Abstract

**Purpose** This work tackles the mobility of heavy metals in soil after compost addition, with particular attention to the effects of compost feedstock and compost maturity.

**Methods** Three types of organic wastes: MSW (municipal solid waste), MSW + GW (municipal solid waste + green waste) and SS + GW (sewage sludge + green waste), were composted and sampled at three different composting times (0, 6 and 12 weeks). These samples were mixed with a cultivated horizon from a silt loam soil in order to pursue column leaching experiments. Columns were built up in three replicates and submitted to leaching with dilute CaCl<sub>2</sub> solution, during 1 h, nearly each week during 2 months. Leachates were analysed for organic and inorganic C, pH, Zn and Cu.

**Results** The leaching of Cu and dissolved organic C were closely related, while Zn showed a different behaviour. Compost feedstock was the factor with the highest influence in element leaching, which decreased in the sequence

MSW > MSW + GW > SS + GW. Within composts obtained from the same feedstock, maturity also had an influence on element leaching, which was expressed at two levels: (1) during the first days after compost addition to soil, leaching decreases at increasing degrees of compost maturity; (2) cumulated amounts of Cu and organic C leached during the 2-month experiments increased with compost maturity. The limited variations of pH during leaching experiments showed no clear relationship neither with the maturation of compost, nor with the mobility of the studied heavy metals.

**Keywords** Trace element · Zinc · Copper · Mobility · Organic waste · Organic amendment

## Introduction

Amendment of agricultural soils with organic waste composts increases their organic matter content, provides short and medium term essential nutrients to plants, and improves their physicochemical and biochemical properties [1–5]. However, composts contain a variety of contaminants, in particular heavy metals, which are not biodegradable, so much that their contents in amended soil horizons may increase after several spreadings. According to various studies, this impact occurs most often with the elements Cu, Pb and Zn [6–9].

The load of trace metals present in compost is a function of several factors. Despite the apparently random contribution of sporadic metallic impurities [10], the source of organic waste used for compost production is often the most influential factor. In particular, municipal solid waste (MSW) and sewage sludge are commonly cited as problematic feedstocks [8, 11–13]. Processing may also have an

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impact on trace metal contents, typically the source-segregation step before the composting of urban wastes [14], as well as the length of treatment and other factors of the process (irrigation, finishing, composition of the equipment...). These factors influence not only the contents of contaminants, but also their physicochemical properties. In particular, the humification degree of organic matter influences the speciation, solubility, mobility and bioavailability of trace metals, which can be strongly fixed to insoluble humic substances or under the form of mobile complexes with dissolved organic matter [15, 16]. This will play a decisive role on environmental risk after compost addition to soil. For example, free ions can be directly assimilated by plants and affect soil organisms, whereas all soluble species, including DOM complexed metals, may migrate to ground and surface waters. These processes are controlled by edaphic factors such as carbonates, organic matter, pH and redox potential, by the chemical and physical forms of the metals in compost [9], and by eventual changes in compost composition and soil conditions over time [17, 18].

Among these factors, we focus here on the feedstock and the length of the composting process. It has been commonly observed that the most mobile forms of trace metals decrease with composting time, due to the formation of stable complexes with humified organic matter [19–23]. On the contrary, soluble organic compounds, issued from unstabilised composts and/or from early degradation of organic amendments by soil microorganisms can form soluble complexes with metals and favour their leaching through the soil layers [24–26]. Several works cited above also underlined differences between Cu and Zn among studied trace metals.

The objective of our study was to unravel the influence of compost maturity upon the mobility of copper and zinc in the short term after addition to soil. For this, we amended an agricultural soil with three different types of urban waste composts, sampled at different moments of composting in order to obtain amendments at different stages of maturity. Beyond chemical extractions applied on compost samples, the mobility of Cu and Zn was assessed through a leaching experiment with soil columns. These metals were chosen since they are among the most important contaminants of recycled organic wastes in terms of inputs to agricultural soils and, unlike Pb, they are commonly quantifiable in leachates of arable amended soils.

## Materials and Methods

### Soil and Composts

The soil was sampled in the ploughed layer of a control plot, i.e., an unamended plot receiving only mineral N

fertiliser since 1998, of the QualiAgro experiment ([www7.inra.fr/qualiagro\\_eng/](http://www7.inra.fr/qualiagro_eng/)). The QualiAgro long term field experiment has been initiated in 1998 by INRA and Veolia R&I. The field is located at Feucherolles, 35 km west of Paris [27]. Mean annual temperature is 11 °C and mean annual rainfall is 582 mm year<sup>-1</sup> (average data between 1989 and 2009 from the nearby meteorological station). The soil is a silt loam Luvisol, according to IUSS-FAO classification and the USDA texture triangle [28]. A sample representative of the study plough layer has been obtained by mixing ten 0–28 cm cores, prepared according to the norm ISO 11464 [29] and submitted to chemical analyses (“Compost and Soil Analyses” section). A similar sample of the same horizon was sieved to 4 mm and kept moist at 4 °C until use for pot experiments (“Leaching Experiment” section).

Three types of organic wastes were composted in pilot-scale reactors:

- a fraction of municipal solid waste collected separately from packaging, further sorted on-site (named “residual municipal solid waste”, abbreviated MSW),
- a mixture of this fraction with green waste (MSW + GW),
- a mixture of sewage sludge with green waste (SS + GW).

They were composted indoor, in insulated 170 L-reactors, with forced aeration through perforated bottoms up to top events. The aeration was driven by the continuous controls of temperature and O<sub>2</sub> concentration of exhaust air, in order to maintain a high temperature stage during weeks and O<sub>2</sub> concentration around 10 %. Composting was performed over a 12-week period with turning after 2 and 6 weeks. The mass loss during composting was measured by weighting the reactors before each turning and sampling. In addition to initial mixtures (0 week), the composts were sampled after 6 and 12 weeks of composting. All these organic samples were dried at 40 °C during at least 10 days, and ground below 2.5 mm. Non-composted and composted organic wastes are generally called hereinafter “composts”.

### Compost and Soil Analyses

To calculate the rates of compost addition to soil in pot experiments, estimations of OM contents were based on weight losses at 480 °C (results not presented). Apart from these first determinations, all physicochemical analyses of soil and dried compost samples were performed at the Laboratoire d’Analyses des Sols (LAS, INRA, Arras, France), according to standard methods resumed below. The soil sample (“Soil and Composts” section) was analysed once, but every dried ground compost stock was sampled three times in our lab and the triplicates were sent

separately to the LAS. This laboratory fulfils the requirements of the standard NF EN ISO/CEI 17025 for the analyses of food products and agronomic quality of soils (<http://www6.npc.inra.fr/las/Assurance-qualite/Accreditation>).

Total carbon and nitrogen were measured from catalytic combustion of representative finely ground samples (ISO 10694 and 13878 [29]), and organic carbon (OC) was deduced from total C by subtracting C corresponding to the total carbonate content (ISO 10693 [29]). Standard pH measurements in water were done on soil (ISO 10390 [29]) and compost samples (NF EN 12176 [30]). Electrical conductivity was measured at 25 °C on 1:25 water extracts of composts. Particle size analysis of soil was performed after organic matter destruction with H<sub>2</sub>O<sub>2</sub> and dispersion with Na-hexametaphosphate (NF X 31-107 [29]). Total trace element contents were measured on soil after HF-HClO<sub>4</sub> digestion (NF X 31-147 [29]), and after *aqua regia* digestion for composts (NF EN 13346 [29]). EDTA-extractable elements were determined on soil and composts according to the “BCR” method [31]. Exchangeable elements were determined from extraction in 0.01 M CaCl<sub>2</sub> solution (ratio 1:10, shaking 2 h, centrifugation and filtration [32]). Besides trace metals, pH and DOC (dissolved organic C) have been measured on the CaCl<sub>2</sub> extracts of compost samples.

### Biochemical Fractionation of Compost Organic Matter

Compost samples, dried and ground to 2.5 mm, were fractionated using a modified Van Soest procedure [33, 34] as described in the Standard XPU 44 162. Four organic fractions, i.e., soluble in water with neutral detergent (SOL), hemicellulose-like (HEM), cellulose-like (CEL) and lignin-like (LIG) were measured, once for each sample.

### Potential Carbon Mineralisation

To evaluate the potential mineralization of organic C in the initial feedstocks and its evolution during composting, 28-day incubations were performed at 28 ± 1 °C in the dark and under aerobic conditions with the equivalent of 25 g of dry soil. All ground organic samples, dried at 40 °C, were mixed with the fresh soil, with a ratio based on organic C (2 g OC per kg of dry soil). The water content of the mixtures was adjusted to 80 % of field capacity (pF 2.8) with Milli-Q water and an adjusted volume of KNO<sub>3</sub> solution carrying 25 mg N-NO<sub>3</sub> per kg dry soil. Moisture was maintained during the incubation period by weighing and adding de-ionized water, if necessary. C mineralisation was also measured in soil alone as control. All incubations

were done in four replicates in hermetically closed 1-L glass jars. In each glass jar, CO<sub>2</sub> was trapped in 20 mL of 1 M NaOH replaced after 1, 3, 7, 14, 21 and 28 days of incubation. The jars were opened when replacing the NaOH traps to enable the atmosphere to be renewed and maintain aerobic conditions. The CO<sub>2</sub> trapped in NaOH during incubation experiments was analysed by colorimetry after titration with 0.25 M sulphuric acid on a continuous flow analyser (Skalar, Breda, NL). Results of mineralized C, after subtracting the average obtained for controls, were expressed as percentage of the OC content of the amendment.

### Leaching Experiment

Mixtures of composts with the control soil were prepared in proportion corresponding to the seven applications on the experimental field during 11 years, i.e., about 28 t OC/ha. In the lab, 13 g of dry material were intimately mixed in two steps into 1 kg of fresh soil for each pot, being about 12 cm in diameter at half-height. At the bottom of the pot, 2 Nylon canvas (34 µm mesh) were spread under 50 g of Fontainebleau sand previously washed with dilute nitric acid, carefully rinsed, then heated at 500 °C and rehydrated with pure water. All pots were prepared in triplicates, leading to 3 solutions per treatment and per chosen leaching date. The first experiment included mixtures of soil with the 3 types of non-composted feedstock and the control soil material (without amendment). A second and a third series used the 6 and the 12-week composts.

A solution that simulates rain water (0.02 g CaCl<sub>2</sub>/L, saturated with air) was used for progressively adjusting the water content up to the retention capacity, during about 6 h until first drops. Then the same solution was added to force periodic leaching, every week during about 2 months, using a small rainfall simulator above the sample surface, regulated to deliver 0.1 L during 1 h (equivalent to 7 mm). The leachates were collected in a polypropylene vial, weighted, and 2 aliquots were kept, one for metal analyses after acidification with ultrapure HNO<sub>3</sub>, the second one for measuring pH, organic and inorganic carbon (DOC and IC).

### Leachate Analysis

The pH was measured using a combined LL-glass electrode (Metrohm, Herisau, Switzerland). DOC was determined as the difference between total carbon and inorganic carbon, measured by using an analyser TOC5050A (Shimadzu, Kyoto, Japan). Zn and Cu were analysed in the acidified leachates by atomic absorption spectrometry (Varian SpectraAA 220), by flame for Zn, using a quartz concentrating tube, and by graphite furnace for Cu

(GTA110, Varian, Victoria, Australia). Quality control was conducted by blank analyses and, for Cu by GTA through triplicate analyses of each leachate sample, leading to variation coefficients of 11 % in average, and by periodic addition of internal standard, showing recovery rates between 84 and 108 %.

## Statistics

Statistical analyses including one-way ANOVA were performed to assess the effect of compost type and composting time on element leaching, using the R statistical package for MacOSX.

## Results

### Soil Properties

Table 1 presents relevant properties of the horizon sampled for the present study. Concerning total trace metal contents, references to common similar cultivated horizons are given. They confirm that this sample is representative of arable horizons of the region, without remarkable metal contamination.

### Compost Properties

Records of temperature and O<sub>2</sub> concentration during composting have been suitable, with temperature reaching 77, 80 and 72 °C, for MSW, MSW + GW and SGW + GW respectively. Table 2 shows the main properties of initial waste mixtures and composts after increasing composting periods. A pH increase was observed in all cases between 0 and 6 weeks, with final

values around 8 for all the composts. Electrical conductivity increased during the composting of MSW, whose final values were the highest among the three composts studied. The OC contents decreased progressively, due to organic matter mineralization, inducing a relative increase of other component concentrations, such as carbonates and N. However, total N content decreased during the composting of SS + GW, indicating a significant loss of nitrogen during the first weeks of composting for that mixture. As a result, C/N remained fairly constant for SS + GW, while it decreased regularly for the other composts. DOC measured in CaCl<sub>2</sub> extracts decreased versus composting time in all cases, the highest values being recorded for the MSW compost, as for EC.

The amount of OC mineralized during standard soil-compost incubations decreased with increasing duration of composting (Table 2), which reflects the progressive stabilization of organic matter during composting. Overall, OC mineralization of the different feedstocks decreased in the sequence MSW > MSW + GW > SS + GW. It is likely that MSW needs more time to stabilize as compared to other feedstock due to the presence of highly degradable waste such as food waste. In turn, the presence of green waste in the mixture reduces the capacity of mineralization, due to the input of scarcely mineralizable components such as lignin. The increase of index of residual carbon I<sub>ROC</sub>, integrating parameters from C mineralization kinetic and biochemical fractions, also indicated the progressive stabilization of OM during composting, as well as the initial differences of OM stability in the different feedstocks, and certain convergence between them after 12 weeks of composting (Table 2).

Figure 1, presenting the evolution of OM biochemical fractions in percentages of initial dry mass, shows that nearly all loss of dry matter occurring during composting

**Table 1** Characteristics of the study horizon (all contents expressed per kg of oven-dried soil)

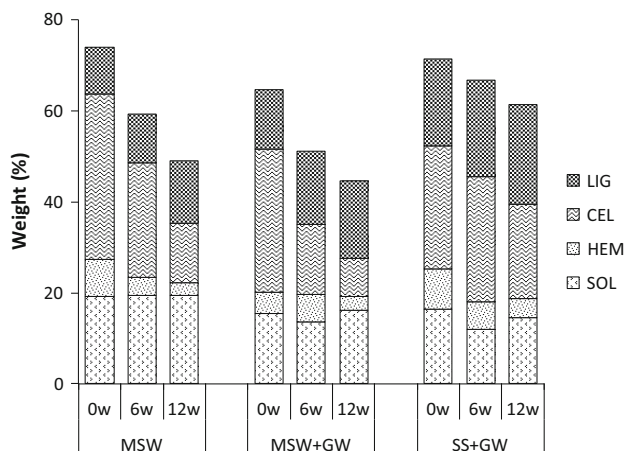
| Major components  | Organic C<br>(g kg <sup>-1</sup> ) | N total<br>(g kg <sup>-1</sup> ) | pH<br>H <sub>2</sub> O | pH<br>CaCl <sub>2</sub> | Clay<br>(<2 μm)<br>(g kg <sup>-1</sup> ) | Silt<br>(2–50 μm)<br>(g kg <sup>-1</sup> ) | Sand<br>(50–2000 μm)<br>(g kg <sup>-1</sup> ) | CEC<br>(cmol <sup>+</sup> kg <sup>-1</sup> ) |
|---|------------------------------------|----------------------------------|------------------------|-------------------------|--|--|---|--|
|   | 9.4                                | 0.98                             | 6.7                    | 5.9                     | 170                                      | 768  | 63  | 8.8  |
| Total contents of trace<br>metals (TM)<br>(mg kg <sup>-1</sup> )      |                                    |                                  | Cd<br>0.20             | Cr<br>45.5              | Cu<br>12.2                               | Ni<br>16.2                                 | Pb<br>21.7                                    | Zn<br>47.0                                   |
| Regional medians <sup>a</sup>   |                                    |                                  | 0.30                   | 44                      | 12                                       | 19   | 26  | 55   |
| EDTA extracted trace<br>metals<br>(mg kg <sup>-1</sup> )              |                                    |                                  | Cd<br>0.14             | Cr<br>0.07              | Cu<br>4.17                               | Ni<br>1.54                                 | Pb<br>5.96                                    | Zn<br>2.78                                   |
| CaCl <sub>2</sub> extracted trace<br>metals<br>(μg kg <sup>-1</sup> ) |                                    |                                  | Cd<br>9.7              | Cr<br><10               | Cu<br>20                                 | Ni<br>38                                   | Pb<br><3                                      | Zn<br>93                                     |

<sup>a</sup> Median TM contents of cultivated horizons of soils developed on decarbonated loess deposits of the Paris Basin, with clay content in the range 10–21 % [44]

**Table 2** Characteristics of organic wastes and composts

| Composting weeks | pH <sub>water</sub> | EC (dS m <sup>-1</sup> ) | OC (g kg <sup>-1</sup> ) | Total N (g kg <sup>-1</sup> ) | C/N         | CaCO <sub>3</sub> (g kg <sup>-1</sup> ) | DOC (g kg <sup>-1</sup> ) | %C <sub>min</sub> 3 day | %C <sub>min</sub> 7 day | %C <sub>min</sub> 28 day | I <sub>ROC</sub> (g kg <sup>-1</sup> ) |
|------------------|---------------------|--------------------------|--------------------------|-------------------------------|-------------|---|---------------------------|-------------------------|-------------------------|--------------------------|--|
| <b>MSW</b>       |                     |                          |                          |                               |             |   |                           |                         |                         |                          |  |
| 0                | 7.7<br>(0.0)        | 2.4<br>(0.0)             | 363<br>(4)               | 17<br>(0.2)                   | 22<br>(0.5) | 75<br>(1)                               | 31.4<br>(0.4)             | 15.9<br>(0.8)           | 40<br>(1.8)             | 52<br>(2.3)              | 244                                    |
| 6                | 8.3<br>(0.0)        | 2.5<br>(0.0)             | 300<br>(3)               | 19<br>(0.1)                   | 16<br>(0.2) | 131<br>(3)                              | 27.5<br>(0.4)             | 11.0<br>(0.6)           | 22<br>(1.7)             | 30<br>(2.2)              | 443                                    |
| 12               | 7.9<br>(0.0)        | 2.8<br>(0.0)             | 256<br>(3)               | 19<br>(0.1)                   | 14<br>(0.1) | 187<br>(2)                              | 16.8<br>(0.4)             | 4.6<br>(0.2)            | 6.6<br>(0.2)            | 12.2<br>(0.6)            | 710                                    |
| <b>MSW + GW</b>  |                     |                          |                          |                               |             |   |                           |                         |                         |                          |  |
| 0                | 7.7<br>(0.0)        | 1.6<br>(0.0)             | 338<br>(2)               | 13<br>(0.1)                   | 26<br>(0.2) | 46<br>(1)                               | 15.2<br>(0.9)             | 13.7<br>(0.7)           | 35<br>(2.3)             | 46<br>(2.7)              | 325                                    |
| 6                | 8.4<br>(0.0)        | 1.4<br>(0.0)             | 277<br>(4)               | 16<br>(0.3)                   | 18<br>(0.5) | 82<br>(7)                               | 9.7<br>(1.0)              | 4.3<br>(0.2)            | 6.9<br>(0.6)            | 13.3<br>(2.2)            | 649                                    |
| 12               | 8.2<br>(0.0)        | 1.6<br>(0.0)             | 241<br>(2)               | 17<br>(0.2)                   | 14<br>(0.0) | 95<br>(9)                               | 5.3<br>(0.1)              | 2.8<br>(0.1)            | 3.6<br>(0.2)            | 8.9<br>(1.2)             | 798                                    |
| <b>SS + GW</b>   |                     |                          |                          |                               |             |   |                           |                         |                         |                          |  |
| 0                | 7.2<br>(0.0)        | 1.2<br>(0.0)             | 392<br>(5)               | 22<br>(0.3)                   | 18<br>(0.5) | 34<br>(3)                               | 13.7<br>(0.2)             | 5.3<br>(0.4)            | 10.5<br>(0.6)           | 26<br>(2.9)              | 570                                    |
| 6                | 7.7<br>(0.0)        | 1.0<br>(0.0)             | 362<br>(9)               | 17<br>(1.1)                   | 21<br>(1.9) | 36<br>(4)                               | 8.6<br>(0.0)              | 1.9<br>(0.2)            | 3.3<br>(0.5)            | 8.8<br>(2.1)             | 642                                    |
| 12               | 7.7<br>(0.1)        | 1.0<br>(0.0)             | 337<br>(5)               | 19<br>(0.2)                   | 18<br>(0.4) | 47<br>(1)                               | 4.3<br>(0.1)              | 2.1<br>(0.1)            | 3.1<br>(0.3)            | 8.1<br>(1.1)             | 697                                    |

MSW municipal solid waste; GW green waste; SS sewage sludge. EC electric conductivity of 1:25 H<sub>2</sub>O extracts; OC Organic C; DOC soluble organic C in CaCl<sub>2</sub> extracts, in g per kg of dry matter at 105 °C; %C<sub>min</sub>: % of organic C mineralized in standard controlled conditions after 3, 7 and 28 days; I<sub>ROC</sub>: index of residual organic C in soils [45]. Values between brackets are standard deviations (SD) from replicate samples (n = 3, or n = 4 for C mineralized)



**Fig. 1** Biochemical fractionation of organic wastes at different times (0, 6 and 12 weeks), in percent of initial dry matter. MSW municipal solid waste; GW green waste; SS sewage sludge; 0w time zero of composting; 6w 6 weeks of composting; 12w 12 weeks of composting

can be attributed to cellulose and hemicellulose degradation, while the other organic fractions (lignin and SOL) remained fairly constant. The biochemical composition of the mixture SS + GW fluctuated less during composting as compared to MSW composts, and confirm the lower degree of evolution of this mixture, as already shown by other indicators.

### Trace Metals in Composts

Total contents of trace metals have been measured in all organic matrices and showed the expected relative concentration with composting time (Table 3). Overall, MSW composts contained more trace metals than SS + GW and MSW + GW, but in all cases their concentrations remained under the French regulatory limits for organic amendments. With respect to inputs of trace metals in soils which are also regulated, the inputs equivalent to our additions in pots, (assuming a mass of 3800 t/ha for the

**Table 3** Total trace metals in composts (after aqua-regia digestion, in mg kg<sup>-1</sup> DW)

| Weeks                  | Cd             | Cu             | Cr          | Ni          | Pb           | Zn           |
|------------------------|----------------|----------------|-------------|-------------|--------------|--------------|
| <b>MSW</b>             |                |                |             |             |              |              |
| 0                      | 0.61<br>(0.01) | 88<br>(2.5)    | 21<br>(0.6) | 20<br>(4.5) | 97<br>(1.4)  | 248<br>(2.6) |
| 6                      | 0.70<br>(0.04) | 126<br>(1.5)   | 28<br>(0.4) | 26<br>(3.2) | 110<br>(0.6) | 352<br>(3.8) |
| 12                     | 0.88<br>(0.04) | 161<br>(3.0)   | 43<br>(0.3) | 39<br>(4.4) | 138<br>(1.5) | 473<br>(6.2) |
| <b>MSW + GW</b>        |                |                |             |             |              |              |
| 0                      | <0.5           | 83<br>(5.3)    | 19<br>(2.1) | 17<br>(2.4) | 38<br>(1.0)  | 193<br>(2.6) |
| 6                      | 0.63<br>(0.03) | 98<br>(2.0)    | 23<br>(0.9) | 22<br>(0.3) | 52<br>(2.9)  | 254<br>(6.2) |
| 12                     | 0.80<br>(0.09) | 126<br>(7.8)   | 30<br>(0.9) | 29<br>(3.4) | 61<br>(3.0)  | 318<br>(4.0) |
| <b>SS + GW</b>         |                |                |             |             |              |              |
| 0                      | <0.5           | 75<br>(5.1)    | 32<br>(2.6) | 16<br>(4.2) | 38<br>(0.7)  | 223<br>(10)  |
| 6                      | 0.60<br>(0.04) | 93<br>(0.6)    | 34<br>(2.7) | 13<br>(2.1) | 44<br>(0.3)  | 273<br>(4.5) |
| 12                     | 0.76<br>(0.01) | 120*<br>(4.9)* | 65<br>(6.2) | 34<br>(10)  | 59<br>(1.4)  | 338<br>(5.0) |
| Regulatory limits [46] | 3              | 300            | 120         | 60          | 180          | 600          |

Values between brackets are SD from triplicate samples. Last line shows French regulatory limits for total contents of presented contaminants in similar organic amendments (mg kg<sup>-1</sup> DW)

MSW municipal solid waste; GW green waste; SS sewage sludge

\* For this analysis, one of the triplicates, about 3 times higher than the two others, was ignored

amended layer in the field), remained also under the regulatory limits for 10 years, in consistency with our aim of simulating 11 years of amendments (“Leaching Experiment” section).

Figure 2 shows the evolution of extractable trace elements during composting. In all cases, EDTA-extractable Zn steadily increased during composting. In percent of total Zn, variations were attenuated for the SS + GW compost, and reversed for MSW and MSW + GW. EDTA-extractable Cu showed a similar behaviour to that of Zn, but the concentrations and the variations were lower. In absolute values, it appeared roughly constant in MSW + GW composts.

CaCl<sub>2</sub>-extractable Zn varied during composting of MSW, with an apparent maximum after 6 weeks reaching 3.6 % of total Zn to decrease back to 1.6 % after 12 weeks. Similar trend was observed for CaCl<sub>2</sub>-extractable Cu during the composting of MSW although

the proportion of total Cu decreased during the entire composting process. Both CaCl<sub>2</sub>-extracted metals regularly decreased with composting time in SS + GW and MSW + GW mixtures down to about 1 mg/kg in absolute values, i.e., less than 1.1 % of total Cu, or less than 0.3 % of total Zn. The overall sequence of these readily mobile metal fractions in the composts was the following:

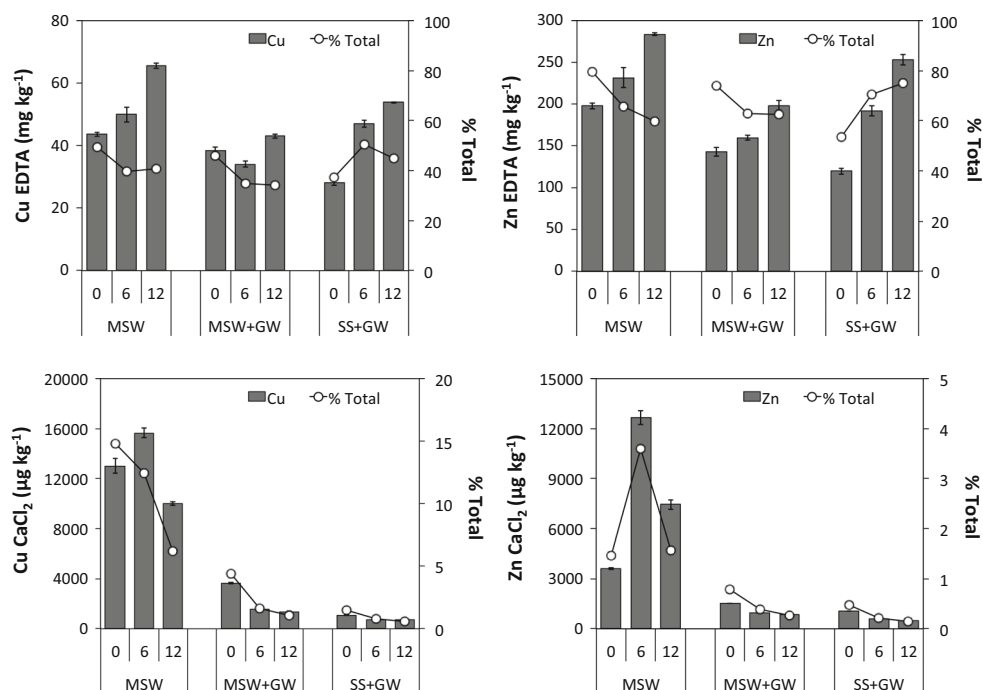
MSW > MSW + GW > SS + GW.

### Leaching Experiment

The range of variation of leachate pH throughout the pot experiments was small, as it is shown by comparing the pH values in the initial and final leachates (Table 4). The DOC concentration in the leachates was higher in the compost-amended soils than in the control soil, and decreased with time in all cases (Fig. 3, left). A significant effect of the type of compost ( $P < 0.001$ ) and composting time ( $P = 0.0013$ ) on DOC concentrations was found. Concerning the first factor, the following sequence was always observed for DOC leaching from amended soils: MSW > MSW + GW > SS + GW, which agrees with the sequence of decreasing OM stability and CaCl<sub>2</sub>-extracted DOC from the pure composts (Table 2). The effect of composting time was less clear. For the mixture SS + GW, a reduction of DOC with increasing composting time was clearly observed in the first leachates; then curves were practically similar. The MSW and MSW + GW composts showed a different complex behaviour. Soils amended with “6-week composts” showed the highest DOC concentrations in the first leachates, followed by the 0-week composts, with the lowest values for the soils amended with 12-week composts. Afterwards, DOC leaching decreased progressively in all amended soils, but the decrease was faster in the soils amended with 0- and 6-weeks composts than in the soils amended with the 12-weeks composts.

The leaching of Cu presented similarities with that of DOC: compost addition to the soil clearly increased leaching, which was maximal at the beginning of the experience. A significant effect of the type of compost ( $P < 0.001$ ) and composting time ( $P = 0.005$ ) on Cu concentrations was observed. Similarly to DOC, Cu concentration in the leachates followed the sequence MSW > MSW + GW > SS + GW. The patterns of leaching as a function of composting time for the soil amended with MSW and MSW + GW were also very similar to those of DOC leaching. However, for SS + GW and apart for the first leachates, Cu leaching rather increased with the length of composting.

**Fig. 2** EDTA-extractable and  $\text{CaCl}_2$ -extractable Cu and Zn. Columns present mass contents and *white symbols* the percentages with respect to total contents. Segments on columns represent standard deviations of the means ( $n = 3$ ). *MSW* municipal solid waste; *GW* green waste; *SS* sewage sludge



**Table 4** pH of the leachates (mean  $\pm$  standard deviation,  $n = 3$ )

|          | Weeks | Initial leachate pH | Final leachate pH |
|----------|-------|---------------------|-------------------|
| Control  | –     | 6.9 $\pm$ 0.2       | 7.7 $\pm$ 0.1     |
| MSW      | 0     | 7.6 $\pm$ 0.2       | 7.2 $\pm$ 0.03    |
| MSW      | 6     | 7.0 $\pm$ 0.04      | 7.6 $\pm$ 0.05    |
| MSW      | 12    | 7.2 $\pm$ 0.1       | 7.5 $\pm$ 0.04    |
| MSW + GW | 0     | 7.2 $\pm$ 0.3       | 7.3 $\pm$ 0.1     |
| MSW + GW | 6     | 7.3 $\pm$ 0.1       | 7.7 $\pm$ 0.1     |
| MSW + GW | 12    | 7.1 $\pm$ 0.05      | 7.5 $\pm$ 0.04    |
| SS + GW  | 0     | 7.0 $\pm$ 0.1       | 7.3 $\pm$ 0.03    |
| SS + GW  | 6     | 7.0 $\pm$ 0.1       | 7.5 $\pm$ 0.1     |
| SS + GW  | 12    | 7.0 $\pm$ 0.03      | 7.4 $\pm$ 0.1     |

*MSW* municipal solid waste; *GW* green waste; *SS* sewage sludge

Zinc leaching was also significantly affected by both the type of compost ( $P < 0.001$ ) and composting time ( $P < 0.001$ ). Unlike Cu and DOC, Zn concentrations in the first leachates decreased with compost amendment, except with the 6-week MSW compost which stood above the control (Fig. 3, right). This compost produced higher Zn concentrations in the leachates than the two other, but the differences were smaller than in the case of DOC and Cu. Concerning the effect of composting time on Zn leaching, a small regular decrease was observed only for the SS + GW compost. It has to be noticed that the peak of initial Zn leaching observed with the soil amended by the 6-week MSW compost was in agreement with the peak of  $\text{CaCl}_2$ -extractable Zn (Fig. 2).

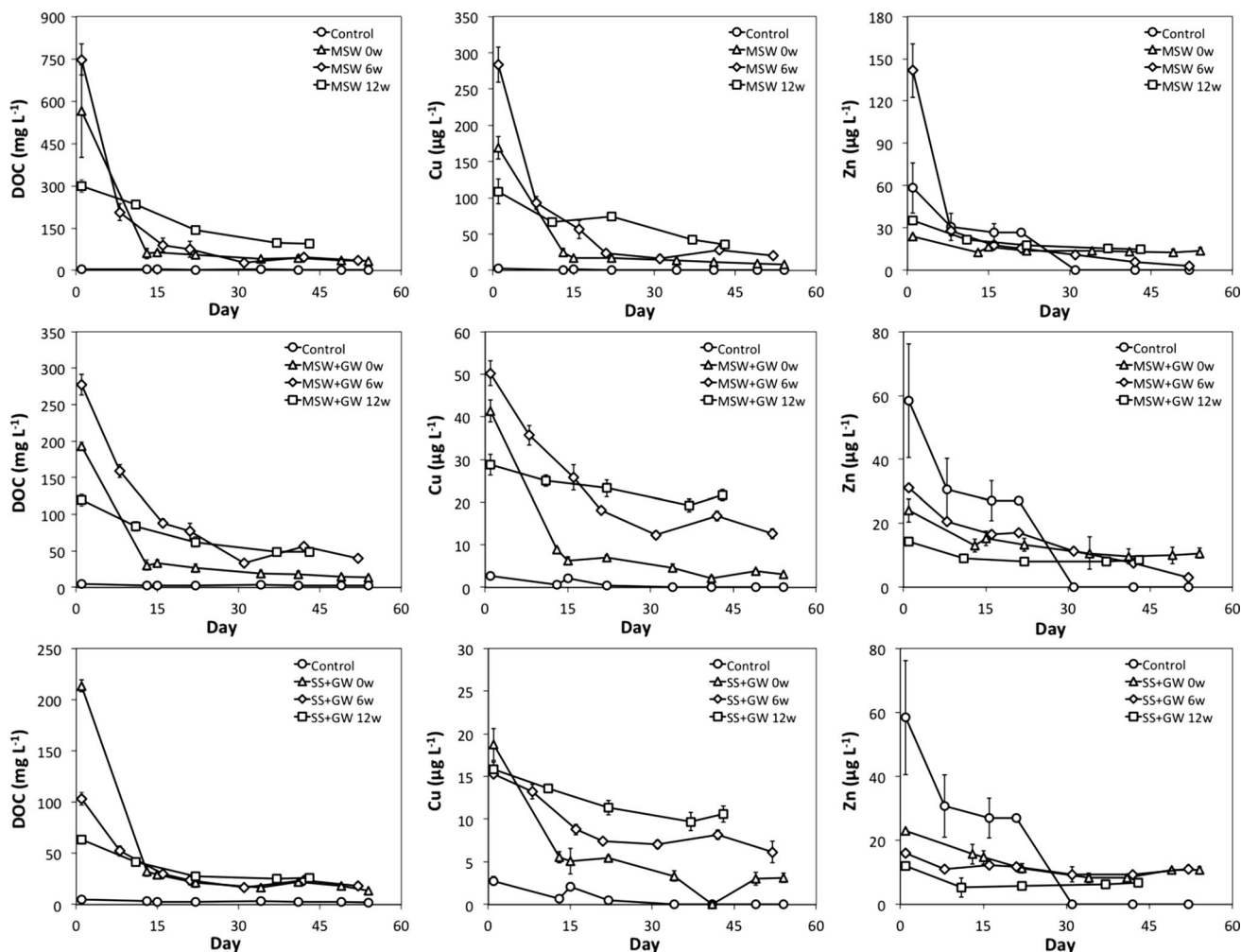
Finally, we calculated from concentrations and collected volumes the total amounts of DOC and trace elements

leached at the end of the experiment (Table 5). We observed higher amounts for the MSW compost with respect to the other two composts. Concerning the influence of composting length, a maximum of leached elements was generally observed for the 6-week composts, especially for Zn.

## Discussion

In order to study the factors influencing Cu and Zn mobility in soil shortly after amendment with urban waste products, we have worked here with three feedstocks at different levels of evolution (0, 6 and 12 weeks of composting). Differences in their degree of maturity were shown by common chemical indicators. In particular, the Van Soest fractionation scheme showed that cellulose and hemicellulose degradation were the main responsible for OM evolution during composting. This is in agreement with what has been commonly observed in the literature [34–36]. Besides, feedstock was an important factor determining the degree of compost maturity. In particular, MSW generated high amounts of soluble OM and leachable DOC, which remained relatively high throughout the entire composting process (Fig. 1; Table 2); inversely, the presence of GW favoured a faster stabilization and smaller amounts of readily soluble OM.

The relationship between trace element mobility and compost maturity was initially examined through chemical extractions on the composts (Fig. 2). A reduction of  $\text{CaCl}_2$ -extractable Cu and Zn with maturity was observed for the



**Fig. 3** From left to right: Water-soluble carbon (DOC), Cu and Zn leached from soil (Control) and soil amended with the composts at different stages of maturity. MSW municipal solid waste; GW green waste; SS sewage sludge; 0w time zero of composting; 6w 6 weeks of

composting; 12w 12 weeks of composting. The control curves are the same. Vertical segments represent standard deviations of the means ( $n = 3$ )

**Table 5** Total amounts of organic C and trace metals leached during the experiments and ratios metal/C

|          | Weeks | DOC (mg) | Zn ( $\mu\text{g}$ ) | Cu ( $\mu\text{g}$ ) | Zn/DOC ( $\text{mg g}^{-1}$ ) | Cu/DOC ( $\text{mg g}^{-1}$ ) |
|----------|-------|----------|----------------------|----------------------|-------------------------------|-------------------------------|
| Control  | –     | 1.3      | 11                   | 0.3                  | 8.2                           | 0.19                          |
| MSW      | 0     | 33       | 5.8                  | 9.8                  | 0.18                          | 0.30                          |
| MSW      | 6     | 88       | 15                   | 35                   | 0.17                          | 0.40                          |
| MSW      | 12    | 88       | 7.1                  | 35                   | 0.08                          | 0.40                          |
| MSW + GW | 0     | 15       | 5.7                  | 3.4                  | 0.38                          | 0.23                          |
| MSW + GW | 6     | 57       | 7.1                  | 12                   | 0.12                          | 0.21                          |
| MSW + GW | 12    | 38       | 3.5                  | 9.3                  | 0.09                          | 0.25                          |
| SS + GW  | 0     | 14       | 4.9                  | 1.9                  | 0.35                          | 0.14                          |
| SS + GW  | 6     | 20       | 5.6                  | 4.4                  | 0.27                          | 0.21                          |
| SS + GW  | 12    | 20       | 2.8                  | 4.9                  | 0.14                          | 0.25                          |

MSW municipal solid waste; GW green waste; SS sewage sludge; DOC dissolved organic carbon



co-composts containing GW, whereas MSW compost showed a more complex behaviour. Increasing complexation of metals by OM stabilised during composting can explain the reduction of their immediate mobility. Such mechanism of immobilization has been argued both for Cu and Zn [8]. On another side, EDTA-extractable Cu and Zn, assessing potential mobility, or the plant availability in a longer term, rather increased with maturity. EDTA is a strong extracting agent that targets several element pools, including soluble, exchangeable, organically-bound and inorganically-bound metals [31]. Given this low specificity, trends of this fraction during composting are more difficult to be interpreted and, like total metal contents, they must be also related to mass loss during mineralisation.

The results from compost analyses, especially from  $\text{CaCl}_2$  extractions, suggest that maturity would have the effect of decreasing metal leaching in compost-amended soils. In agreement with this, experiments with soil-compost mixtures showed that Cu and DOC leaching decreased with compost maturity during the very first days after compost addition to soil (Fig. 3). However, when considering the cumulated leached amounts of DOC and Cu through the whole experiment, and despite non-monotonous variations, they did increase with compost maturity, for each feedstock separately (Table 5), in relation with higher concentrations in the leachates between 15 and 60 days (Fig. 3). Similarly, Beesley and Dickinson [37] found higher concentrations of DOC and Cu in pore water of soils amended with composted green-waste compared to non-composted green wastes, but it was a matter of non-composted coarse woody material, inducing no initial DOC flush like the fresh organic wastes of our study.

In turn, the relatively low stability constants of organo-Zn complexes [15, 38] compared to Cu should limit the association of Zn with DOC and its retention by added organic solid substances. However, organic amendments appeared to reduce Zn solubility as compared to control soil. The variations of Zn mobility could be also influenced here by small pH variations. Finally with respect to composting length, despite a complex behaviour of 6-week composts, the overall trend was to reduce Zn mobility (Figs. 2, 3; Table 5). In summary, the influence of maturity on leaching can be important just after compost addition to soil, in the way that the most mature 12-week composts induce less leaching of DOC, Cu and Zn than fresh OM and 6-week composts. But shortly after, the impact of such unstable OM on leachates vanished, precisely because of its leaching and probably of its microbial degradation.

Many studies have focused on the influence of OM origins and transformations upon their metal complexing properties, involving aspects as their size, their hydrophobic, aromatic or proteic character, or the density and nature of their functional groups [39–41]. Data presented here on

Cu/DOC ratios in the leachates illustrate this multifactor influence (Table 5): on one side, Cu/DOC ratios increased with maturation of MSW and SS + GW products, tending towards strengthening Cu complexation; on another side, the less stable MSW composts show the highest Cu/DOC ratios, suggesting that soluble organic molecules issued from MSW present a particularly high affinity for copper. The progressive reduction of Zn/DOC ratio of the leachates with composting time (Table 5), opposed to Cu/DOC trends discussed above, appears in agreement with Gove et al. [42], who reported that Zn is more susceptible to complexation by fresh biosolid-derived organic matter than by composted materials.

Nevertheless, the influence of maturity is second only in importance for compost behaviour in soil; the most important factor remains compost feedstock, as it was extensively demonstrated in this work both by compost properties and element leaching in soil. From the present results, the incorporation of GW can be recommended to improve the stability of OM and decrease the mobility of the studied trace metals in MSW-based composts. However, due to problematic behaviour of 6-week MSW + GW composts, the length of composting should be at least 12 weeks. And although SS + GW compost properties appeared less sensitive to composting time, 12 weeks appeared also better than 6 weeks with respect to immediate mobility of organic C and Cu in soils amended by these products. On the longer term, i.e., about 2 months after incorporation in soil and according to results, mature composts will produce higher DOC and Cu leaching than immature composts. This is the drawback of lastingly increasing the soil OM content by using composts, which can induce a steady output of DOC and Cu [43]. On the contrary, Zn leaching here appeared lower in amended soils than in the control, unamended one. The environmental impact of Cu and Zn leaching may be limited, compared to other potential effects of amendments, but an environmental balance of urban compost amendments is out of scope of the present study.

## Conclusions

The present study enlightened the influences of original composition and maturity of composts on the availability of Cu and Zn within the products and on their mobility after addition to soil. As a consequence of relative concentration due to progressive OM mineralization, total and EDTA-extractable trace metals increased between 0 and 12 weeks of composting. In turn, exchangeable Cu and Zn decreased with increasing composting time. Concerning metal mobility in soil, compost feedstock has a strong influence in Cu and Zn leaching. MSW composts

induced relatively high levels of soluble OM and mobile Cu and Zn, and this disadvantage with respect to the other studied feedstock lasted over 12 weeks of composting and a few months after addition to soil in laboratory experiments. Addition of GW to MSW improved the stability of OM in composts and lowered the mobility of the two trace metals studied. Thus the association of GW to MSW and at least 3 months of maturation are recommended. Association of sewage sludge and green waste produced immediately more stable organic products, with levels of mobile metals lower or similar than those from mixtures with municipal solid waste. Within composts obtained from the same feedstocks, metal leaching is influenced by maturity: during the first days after compost addition to soil, higher maturity increases the stability of OM and decreases OC and Cu immediately leachable. However, after a few weeks of contact with soil, mature composts induced higher levels of soluble OC and Cu than less mature compost.

**Acknowledgments** The authors express their acknowledgement to Veolia Research & Innovation for its support, the Ecuadorian Government for the scholarship of Andrés Jara during his Master in France, and are grateful to Muriel Collin, Véronique Etievant, Christophe Labat, Jean-Noel Rampon and Amélie Trouvé for their help.

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