Food Chemistry 115 (2009) 1405–1410

Contents lists available at [ScienceDirect](http://www.sciencedirect.com/science/journal/03088146)

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Evaluation of some metals in Brazilian coffees cultivated during the process of conversion from conventional to organic agriculture

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article info

Article history: Received 29 July 2008 Received in revised form 10 December 2008 Accepted 26 January 2009

Keywords: Principal component analysis Organic coffee Cluster analysis Available metals in soil Nutrients and toxic elements

ABSTRACT

The aim of this study was to evaluate the presence of nutrients and toxic elements in coffees cultivated during the process of conversion, on organic agriculture, in southwest Bahia, Brazil. Levels of the nutrients and toxic elements were determined in samples of soils and coffee tissues from two transitional organic farms by atomic absorption spectrometry (FAAS). The metals in soil samples were extracted by Mehlich1 and USEPA-3050 procedures. Coffee samples from both farms presented relatively high levels of Cd, Zn and Cu (0.75, 45.4 and 14.9 μ g g⁻¹, respectively), but were still below the limits specified by the Brazilian Food Legislation. The application of statistical methods showed that this finding can be attributed to the addition of high amounts of organic matter during the flowering tree period which can act on the bioavailability of metal ions in soils.

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1. Introduction

Organic farming holds an increasingly important position in current agriculture. Hardly any discussion about the future of modern agriculture is undertaken without considering organic farming. The International Federation of Organic Agriculture Movements (IFOAM) has estimated a global production growth rate of between 20 and 30 percent, annually. This movement, which was founded in 1972, has 770 member organizations in 107 countries. Europe and the USA are leading the way in this development, but in developing countries organic farming is also seen as a rewarding alternative to the current problematic agricultural situation in agriculture ([Bibin, Siba, Raj, & Pramod, 2007; Figueiredo, Assis, &](#page-4-0) [Reydon, 2003; Siu, Mejia, & Saavedra, 2007](#page-4-0)).

Long-term agricultural use of inorganic fertilizers increases the total content of heavy metals in cultivated soils. Metal accumulation in soil is likely to gradually generate health and environmental risks. In order to assure the safety of agricultural products and to protect arable land, the heavy metal concentrations in composts, particularly the heavy metal loading rates (into the soil) have to be reviewed in detail in both conventional and organic farming ([Dungan & Dees, 2007; Evangelou, Klöss, Ebel, & Schaeffer, 2007;](#page-4-0)

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[Muthanna, Viklander, Gjesdahl, & Thorolfsson, 2007; Overesch,](#page-4-0) [Rinklebe, Broll, & Neue, 2007; Tsadilas, Karaivazoglou, Tsotsolis,](#page-4-0) [Stamatiadis, & Samaras, 2005\)](#page-4-0).

The demand for organic products has created new export opportunities for the developing world. Typically, organic exports are sold at impressive premiums, often at prices 20 percent higher than those of identical items produced on non-organic farms [\(Cua](#page-4-0)[dra & Rydberg, 2006; Lorena et al., 2007](#page-4-0)).

The time between the start of organic management and cultivation of crops is known as the conversion period. In Brazil, the minimum conversion period for plant products, produced annually, is 12 months prior to the start of the production cycle. For perennial plants (including coffee cultivation) the conversion period is 24 months from the date of starting organic management. Depending upon the past use of the land and ecological situations, the certification agency can extend or reduce the minimum conversion period ([Figueiredo et al., 2003\)](#page-4-0).

During the transition from conventional to organic farming practices, nutrients from organic fertilizers are often mineralized before or after the time when plants can best use them for their growth. When this happens, farmers obtain poor yields from their crops while nutrients intended for plant growth may be leached into the groundwater or carried into rivers by runoff. This lack of synchrony between nutrient release and nutrient uptake occurs primarily because the soil undergoes chemical and biological changes during the transition from synthetic to organic inputs.

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To mineralize sufficient nutrients for plant growth, the relatively small populations of soil organisms in transitional farms need to have access to high amounts of organic matter. However, these soil organisms will continue to decompose the added organic matter, even after plant growth. Therefore, the mineralized excess of nutrients may contaminate groundwater or streams [\(Figueiredo et al.,](#page-4-0) [2003; Tsadilas et al., 2005](#page-4-0)).

Organic matter can act either to mobilize or immobilize metals in soil. The solubility of metals is determined by the solubility of the associated organic matter. Often, decomposition-driven formation of products that are smaller and more soluble is an important factor in increasing the solubility of such metals. Organic acids act as ligands for many metals, enhancing movement in percolating water, down through the soil profile ([Ana, Stervin, & Jesús, 2007;](#page-4-0) [Bibin et al., 2007; Rieuwerts, Ashmore, Farago, & Thornton, 2006;](#page-4-0) [Sinha et al., 2006](#page-4-0)).

The aims of this work are to: (I) evaluate the availability of nutrients and heavy metals in coffees cultivated during the process of conversion to organic agriculture in southwest Bahia, Brazil and (II) evaluate the environmental impact of the conversion period.

2. Material and methods

2.1. Site description

The area studied is located in the southwest of the state of Bahia, Brazil, close to Vitória da Conquista City. The area has a typical transitional forest climate, with an annual average temperature of 23 \degree C and rainfall of 900 mm, which is very suitable for the growth of coffee. Two transitional organic farms (organic and organic shaded methods) were included in this study. The organic coffee farm (latitude 14° 49′ 3.8″ S, longitude 40° 31′ 33.7″ W and altitude of 901 m) and the organic shaded coffee farm (latitude 14 \degree 48' 53.2" S, longitude 40 \degree 31' 21.8" W and altitude of 891 m) are located in the same municipal district (Barra do Choça City). As reference, soils from the forest reservation sites, located around the two organic farms in study, were also included.

2.2. Sampling

Sampling was carried out in April (flowering period) and August (harvesting period). Ten samples of soils and coffee plant tissues (leaf and fruit) were collected from each of the two coffee cultivation operations. Each soil sample (about 1 kg) was a composite of five sub-samples taken from depths of 0 to 20 cm. The coffee samples were collected from the associated soil-sampling sites for comparison purposes. All samples were stored in polyethylene bags in the field and transferred to the laboratory within 3 h for preparation.

2.3. Reagents

All solutions were prepared using high purity deionized water obtained from a Millipore water purification system (Millipore, São Paulo, SP, Brazil).

For extraction procedures, the following were used: nitric acid (HNO3, 65%, p.p., Merck, Rio de Janeiro, RJ, Brazil); hydrochloric acid (HCl 37%, p.a., Merck, Rio de Janeiro, RJ, Brazil) and perchloric acid (HClO₄ 70%, p.a., Merck, Rio de Janeiro, RJ, Brazil).

For calibration of AAS analytical reference, solutions in 1.0% (v/ v) HNO $_3$ prepared by successive dilution of 1000 mg l $^{\rm -1}$ of Ca, Mg, Fe, Mn, Zn, Cu, Cr, Cd, Pb and Ni Titrisol standard solutions (Merck, Darmstadt, Germany), were used. All solutions were stored in decontaminated polypropylene bottles (Nalge Company, Rochester, NY, USA).

2.4. Apparatus

For measurements, an air–acetylene flame atomic absorption spectrometer (Perkin Elmer AAnalyst 200, USA) equipped with a deuterium background corrector was used.

2.5. Preparation of plant tissue samples

All glassware and polyethylene flasks used were cleaned with detergent solutions, soaked in 10% (v/v) nitric acid for 24 h, rinsed with Millipure water, and dried in polypropylene containers. Extractions were replicated three times for each sample. The procedures are given below.

The leaf and fruit (green coffee) samples were washed with tap water, followed by neutral detergent, 3% (v/v) hydrochloric acid, and deionized water, to remove soil particles or dust adhering to the plant surface, and then dried with tissue paper. The leaf samples were then oven-dried at 60 °C for 48 h to a constant weight. Prior to chemical analysis, the dry leaf samples were ground into fine powder in a stainless steel mill and the green coffee samples were roasted in a laboratory roaster in 150 g batches at 300 \degree C. The extractions were performed using 1 g portions of powdered dry leaf dissolved in 10 ml of 65% nitric acid (puriss, Merck) heated under reflux. After dissolution, 2 ml of concentrated perchloric acid were added and the whole heated until formation of nitrous fumes stopped. The solution was placed in a 50 ml volumetric flask and made up to volume with Millipure water. The same procedures were used for the preparation of coffee using 0.500 g of powdered dry grain [\(Aucejo, Chover, Sáez, & Estellés, 2007; Maenpaa, Kukko](#page-4-0)[nen, & Lydy, 2002; Neunhäuserer, Berreck, & Insam, 2001; Rieuw](#page-4-0)[erts et al., 2006; Santos, Lauria, & Porto, 2004; Siripatrawan &](#page-4-0) [Harte, 2007; Yu et al., 2004](#page-4-0)).

2.6. Preparation of soil samples

2.6.1. General

After removal of visible pieces of plant residue, the soils were dried at 60° C, homogenized, gently crushed and sieved to <2 mm. A portion of each sample was ground to particle sizes of less than $63 \mu m$ for metal extraction.

2.6.2. Available metals in soil

The available element is the portion of the element that can take part in a range of chemical and biological reactions. To some extent, the terms "available element" and "extractable element" are synonymous. Since the bioavailability of metal ions in soils is largely governed by the chemical equilibrium of metal ions in solid and solution phases, adsorption reactions are important for determining the availability of metals to plants and their mobility throughout the soil. To measure extractable elements, soil samples were shaken in an aqueous solution containing a chemical chosen to displace that portion of the element that is supposed to be readily available for uptake by growing plants. The use of chemical extractants such as strong chelating agents (DTPA method), mild neutral salts (ammonium acetate method) and dilute acids (Mehlich methods), can provide assessment of potential metal availability to plants. Such tests, however, tend to be metal-, soil- or cropspecies specific.

For available metals, soils were extracted by two procedures: Mehlich1 (5 g samples of soil were digested using 0.05 M HCl and $1 M H₂SO₄$) and DTPA (8 g of soil were digested using 0.005 M diethylenetriamine pentaacetic acid) [\(Houba, Lexmond,](#page-4-0) [Novozamsky, & Van der Lee, 1996; Huang, Kuo, & Bembenek,](#page-4-0) [2003; Qing-wei, Hang, & Fang-yan, 2007](#page-4-0)).

2.6.3. Total metals in soil

General acid digestion procedures are available for total elemental analysis in soils. Various digestion reagents are used, including concentrated HNO₃, aqua regia (HNO₃–HCl 1:3 v/v), USEPA-3050 ($HNO_3-H_2O_2$), USEPA-3052 (HCl–HF) and HNO_3- HClO4. The use of the USEPA-3050 method can measure the metals adsorbed by soil constituents, such as carbonates, phosphates and other components that could become environmentally available. By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

For total metals, soil was digested by the USEPA SW 846-3050 procedure (0.5 g samples of soil were digested with concentrated nitric acid, concentrated hydrochloric acid and hydrogen peroxide, using open vessel digestion). After digestion, the samples were filtered and transferred into 50 ml volumetric flasks, brought to volume, and transferred into polyethylene flasks for analysis. The values were compared with their accumulation in the green coffee samples collected.

2.7. Analytical methods

K, Na, Ca, Mg, Cu, Fe, Mn, Cd, Zn and Pb levels were determined directly in the extract solution by atomic absorption spectrometry (AAS). The instrument response was periodically checked with known standards. An air acetylene flame and a hollow cathode lamp were used for all samples. Calibration curves were prepared using dilutions of stock solutions. All the analyses were done in

Table 1 Mean total metal concentrations (mg kg^{-1}), pH and OM (%) of the crop and natural soils.

	Organic farm	Organic shaded farm	Natural soils
pH	4.14 ± 0.10 a	4.98 ± 0.20 b	4.65 ± 0.10 c
OM	6.23 ± 0.30 a	5.42 ± 0.50 b	3.14 ± 0.10 c
Cr	$70.0 \pm 4 a$	$81.1 \pm 9a$	51.3 ± 1 b
Ni	1.09 ± 0.60 a	1.92 ± 1 a	0.54 ± 0.10 b
Cu	10.9 ± 0.50 a	10.8 ± 1 a	7.10 ± 0.30 b
Cd	3.62 ± 0.30 a	$3.98 \pm 1 a$	0.27 ± 0.10 b
Pb	12.6 ± 0.4 a	13.5 ± 1 a	33.0 ± 1 b
Mn	$68.9 \pm 13 a$	$66.0 \pm 19 a$	$221 \pm 12 b$
Zn.	40.4 ± 7 a	$45.7 \pm 5 h$	$52.5 \pm 6c$

Means followed by different letters for the same variable are significantly different at confidence interval 95%, using a paired t-test analysis.

triplicate and the mean values were used for statistical data evaluation.

2.8. Statistical data evaluation

In order to test whether there was a significant difference in physicochemical parameters and metal contents between results obtained in crop soils (organic and organic shaded managements) and natural soils (forest reservation), paired two-tailed t-tests were performed. Correlation analysis (Pearson r) was performed between the contents of Ca, Mg, Fe, Mn, Zn, Cu, Cr, Cd, Pb and Ni in coffee tissues (leaf and fruit) and soil samples extracted by the two procedures (Mehlich1 and EPA3050). All statistical analyses were performed using the statistical package SPSS 12.0 for Windows (SPSS, USA, 2001).

3. Results and discussion

3.1. Mineral nutrients and toxic elements in soils

The physicochemical parameters and metal contents in crop soils (organic and organic shaded managements) and natural soils (forest reservation) are shown in Table 1. The results show that concentrations of all metals except Zn did not present any significant difference between the soils samples collected in organic farm and organic shaded farms. There were increases in the concentrations of Ni, Cr and Cu in the two crop soils (organic and organic shaded managements) compared to the natural soils. The reverse occurred for Zn and Pb contents, which were higher in natural soils than in the crop soils. The differences in mean metal contents between natural soils and soils of organic farms, indicate the export of Zn and Pb by the crop plants in the organic cultivations. For the two sites, there was a significant difference between the total Cd contents of crop and natural soils. The total Cd contents in the two crop soils (organic and organic shaded managements) are 3.98 and 3.62 mg kg^{-1} , respectively, whereas the mean concentration of Cd in the natural soils was 0.27 mg kg^{-1} .

3.2. Available elements in soils

Table 2 shows the concentrations and standard deviations for the heavy metals extracted by the Mehlich1 and USEPA-3050 methods and the percentage of metal available. The results from two extractions showed almost no variation in Cd, Pb, Cr, Zn and

Table 2

Amounts of total sorbed (USEPA-3050), extractable metal (Mehlich1) and available fraction (as a percentage of total metal concentrations).

^a <Detection limit

b Not available.

Table 3

Metal concentration (µg $\rm g^{-1}$) in five organic residues used as fertilizer.

	Residue 1	Residue 2	Residue 3	Residue 4	Residue 5
Cr	<0.08	< 0.08	< 0.08	< 0.08	<0.08
Ni	7.16 ± 0.70	5.41 ± 0.40	7.46 ± 0.80	6.63 ± 0.40	7.18 ± 0.50
Cu	75.4 ± 0.9	60.8 ± 1.7	71.5 ± 1.4	108 ± 0.7	102 ± 1.7
Cd	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Pb	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Mn	11.4 ± 0.3	23.9 ± 0.4	29.5 ± 0.2	36.5 ± 0.6	40.4 ± 0.4
Zn	253 ± 3.2	181 ± 1.7	121 ± 0.8	66.0 ± 0.4	448 ± 2.5

Cu concentrations between the replicates, as confirmed by low standard deviations. However there were very high variations, particularly for Pb and Cu, in the soil concentrations between the two collection periods. The Mehlich1 extraction showed partially significant decreases between the pre-flowering period and after-harvesting period, indicating that the reductions in potentially bioavailable Cd, Pb and Cr may be mainly attributed to the soil adsorption mechanisms, combined with removal of soluble metal for ground water or rivers by runoff, although the extraction process by plants, can also remove those elements of soils. However, the Cd, Pb and Cr concentrations in the plant tissues (April–August periods) presented in Table 4, do not corroborate the hypothesis of removal by plants. This happens, because, during the transition from conventional to organic farming practices, nutrients from organic fertilizers and their impurities (e.g. Cd, Pb and Cr) are often mineralized before or after the time when plants can best use them for their growth. In samples collected from farm 1 (organic management), the total Cd content of the soil, as determined by USEP-A-3050 extraction, showed no significant change between the time before the flowering tree period and the after harvesting period, indicating that the amount of Cd extracted by the plants was too small to be detected within the time frame of the two soil samplings.

The percentage increase in Cd and Pb concentrations in the available form in soil samples collected in April is likely caused by the addition of high amounts of organic matter during the flowering tree period compared to the soil collected in August (harvesting period). The added organic matter can act either to mobilize or to immobilize metals in soil. Because the metal cations that bond most strongly to organic soil matter tend to be the most rapidly adsorbed, the low copper concentration in soil samples collected in April can also be explained by addition of organic matter before the flowering tree period. When high levels of organic matter are added to soils, greater uptake of Cu^{2+} by plants occurs because the soluble organic matter acts as a ''cation carrier," facilitating metal diffusion from soil particles to roots. In contrast, the high levels of Cr in soil samples can be explained by the strongly hydrating cation $\mathsf{Cr}(\mathrm{H_2O})_{6}^{3+}$, having such an extremely slow water ex-

3.3. Influence of organic residue from the coffee processing used as fertilizer

Coffee processing generates residues in the form of pulp or husk. These represent approximately 78 per cent of the berry. This organic residue, the outer hull, is further reincorporated in the tillage soil as fertilizer since it is rich in mineral and organic compounds. In Brazil those residues are utilized as fertilizers in organic coffee farms.

Table 3 shows the concentrations and standard deviations for the heavy metals in organic residue samples collected in five coffee processing plants located around the two organic farms in study. All organic residue samples contained appreciable concentrations of Ni, Cu and Zn, that reflect an increase of those elements with the application of the organic residues from coffee processing as fertilizer in the crop soils. The differences in mean metal contents between natural soils and soils of organic farms [\(Table 1\)](#page-2-0) may be indicative of accumulation of Ni, Cu and Zn in crop soil provoked by use of organic residues from the coffee processing in the cultivations.

3.4. Toxic elements accumulation in coffee tissues

The Mehlich extractant has been widely used to assess the phytoavailability of many trace metals, and concentrations of Mehlichextractable trace metals have been reported to correlate well with plant uptake for Cd, Zn, Cu, Pb and Cr.

Table 4 shows Cd, Pb, Cu, Zn and Cr concentrations in plant tissues (leaf and fruit) of the coffee collected in April (flowering tree period) and August (harvesting period) from each of the two coffee cultivation procedures. The leaf samples showed relatively high accumulations of zinc and cadmium in the two collection periods. Moreover, there were increases in Cd, Zn and Cu contents in leaves from the flowering tree period (April) to harvesting period (August). Such increase is likely caused by the addition of large amounts of organic matter in the flowering tree period. When high levels of organic matter are added to soils, greater uptake of Cd, Zn and Cu by plants occurs. Zinc and copper are essential micronutrients, and their accumulation is necessary for plant metabolism. The mechanism of Cd accumulation in plants has not yet been elucidated. It is possible that the uptake of this metal in roots is via a system involved in the transport of another essential divalent micronutrient, possibly Zn^{2+} . Cadmium is a chemical analogue of the latter, and plants may not be able to differentiate between the two ions. Different behavior was observed for Pb and Cr.

Concentration, μ g g $^{-1}$ (dry matter).

^a Not determined.

b <Detection limit.

Correlation matrix between the variables of coffee tissues (leaf and fruit) and soil samples extracted by two procedures (Mehlich1 and EPA3050).

Farm 1 (organic method).

Table 5

^b Farm 2 (organic shaded method).

Although these two elements presented high concentrations in soils [\(Table 1](#page-2-0)), they did not accumulate in plant tissues (leaf and fruit) ([Table 4](#page-3-0)).

In fruit (edible part), Cd, Zn and Cu were present at concentrations of: 0.70, 25.0 and 7.15 μ g g⁻¹, respectively, for samples collected on farm 1 and 0.75, 45.4 and 14.9 μ g g⁻¹, respectively, for samples collected on farm 2. Although the levels of these three elements were relatively high, they were below the maximum allowable limit specified by the Brazilian Food Legislation. The values defined by the Brazilian Food Legislation for Cd, Zn and Cu are 1.00, 50.0 and 30.0 μ g g⁻¹, respectively. However, the values for cadmium content, for the coffee samples from the two farms, exceeded the normal range for Brazilian coffees produced by traditional management (Małgorzata, Ewa, & Piotr, 2007; Éder & Elisabeth, 2001). For Zn and Cu, the concentrations found were in accordance with values reported in the literature (Martín, Pablos, & Gonzalez, 1999; Małgorzata et al., 2007; Éder & Elisabeth, 2001).

3.5. Correlations between metal concentration in soil and coffee tissues

Table 5 shows the correlation matrix between the contents of Ca, Mg, Fe, Mn, Zn, Cu, Cr, Cd, Pb and Ni in coffee tissues (leaf and fruit) and soil samples extracted by the two procedures (Mehlich1 and EPA3050). All variables show a significant correlation with at least one other variable. No strong correlations between the leaves and other variables are evident. High correlations are observed for the green coffee and soil extraction Mehlich1 pairs $(r = 0.912$ for farm 1) and $(r = 0.907$ for farm 2). This fact leads to the following preliminary considerations: the strong correlations between metal concentrations in the available fraction in soil and the amounts in coffee fruit reflect the importance of the Mehlich extractant for providing a suitable chemical evaluation of the amount of metals available for plant uptake. This could be caused by the addition of organic matter before the flowering tree period. When high levels of organic matter are added to soils, a greater uptake of metals and nutrients by plants occurs because the soluble organic matter acts as a ''cation carrier", facilitating metal diffusion from soil particles to roots (Houba et al., 1996; Huang et al., 2003; Micó, Recatala, Peris, & Sanchez, 2006; Qing-wei et al., 2007).

4. Conclusions

The results obtained from this study suggest that some heavy metals cannot be avoided or eliminated through the use of organic farm management techniques. Coffees cultivated during the process of conversion to organic agriculture in Southwest Bahia, Brazil, presented relatively high levels of Cd, Zn and Cu. The values for cadmium content for the coffee samples exceeded the normal range for Brazilian coffees produced by traditional management. However, the levels of these three elements were below the limit specified by the Brazilian Food Legislation. This characterization, with the aid of cluster analysis, is of great importance in establishing guidelines for the administration of organic farms and the improvement of coffee quality.

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