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Assessing arsenic, cadmium, and lead contents in major crops in Brazil for food safety purposes



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ABSTRACT

The food chain is one of the major sources of human exposure to non-essential trace elements (TEs) present in soils. Human exposure to contaminated food is a worldwide health concern and a food safety issue that threatens agricultural trade. To assess the quality of Brazilian food products with respect to non-essential TEs, we evaluated arsenic (As), cadmium (Cd), and lead (Pb) contents in five major crops grown in Brazil: rice, wheat, corn, soybeans, and potatoes. The samples were collected from field trials with a record of long-term use of phosphate fertilizers in the states of Mato Grosso and Minas Gerais, Brazil. The TE concentrations in soils were all below the maximum allowable concentrations for agricultural soils. The mean concentrations of As, Cd, and Pb ($\mu\text{g kg}^{-1}$ dry weight) were as follows: below the detection limit <15, 29, and <40 for rice; 19, 23, and 64 for wheat; 47, 40, and 95 for corn; 65, 23, and 106 for soybeans; and 59, 22, and <40 for potatoes, respectively. Significant differences were found in the As and Cd contents of the different wheat cultivars. The levels of As, Cd, and Pb found in the studied crops are well below the values reported in the literature and are in accordance with the *Codex Alimentarius* and the European Union and Brazilian guidelines, indicating that the concentrations of these elements in the crops do not pose a risk to human health.

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1. Introduction

Food security is achieved at the individual, household, national, regional, and global levels when all people at all times have physical and economic access to sufficient, safe, and nutritious food to meet their dietary needs and preferences for an active and healthy life (Pinstrup-Andersen, 2009). In spite of the existence of disagreements regarding the concept of food

security (Coleman-Jensen, 2010; Renzaho and Mellor, 2010), the qualitative aspect of the abovementioned statement requires that any non-essential elements in food must be present at concentrations that do not pose a risk to human health. In fact, food safety is one of the five dimensions of food security that still lacks an appropriate suite of indicators to be assessed (Coates, 2013).

Some trace elements (TEs) are essential to the vital functions of humans, while others have no known beneficial biological function and may be potentially harmful to human health when present in high concentrations in food and in the environment (Barker and Pilbeam, 2007). Food ingestion is a major source of human exposure to non-essential TEs such as arsenic (As), lead (Pb), and cadmium (Cd) (Kabata-Pendias and Mukherjee, 2007).

Arsenic, Pb, and Cd are known to have many toxic effects in humans and are ranked first, second, and seventh, respectively,

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in the priority list of hazardous substances compiled by the US Environmental Protection Agency (USEPA) (ATSDR, 2011). Arsenic-exposed humans may develop skin lesions, neuropathy, gastrointestinal diseases, cardiovascular diseases, cancer, and other ailments (ATSDR, 2007a). Lead exposure may cause changes in the neurologic system, leading to loss of neurological function (ATSDR, 2007b). High Cd intake through Cd-contaminated food or water can cause stomach irritation, resulting in vomiting and diarrhea, while the long-term intake of low levels of Cd can cause kidney disease and bone fragility (ATSDR, 2008).

Environmental pollution (in air, water, and soil) may be one of the sources of toxic TEs in agricultural areas. Likewise, agricultural inputs such as phosphate (P) fertilizers might represent a diffuse source of TEs in soils, especially those requiring high inputs of P fertilizers for adequate yields (Corguinha et al., 2012). While non-essential TEs are present in many agricultural soils, their accumulation by plants and their translocation to edible and harvested parts depend on many factors, including climatic factors, soil attributes, input rates, and plant species and genotypes (McLaughlin et al., 1999).

Most of the daily human intake of non-essential TEs is due to the consumption of vegetables and cereals (Kabata-Pendias and Mukherjee, 2007). Crops such as rice, corn, wheat, potatoes, and soybeans are widely used in food and feed. These crops are the basis of the human diet in many countries and can be an important source of contamination of non-essential TEs in human and animal diets. This is especially true for rice, a crop that has a high capacity for As and Cd uptake (Peralta-Videa et al., 2009). Soybeans, corn, and rice are among the most important crops in terms of cultivated area in Brazil, whereas wheat and potatoes are crops that demand high rates of agricultural inputs that might represent sources of non-essential TEs to soils.

Human exposure to contaminated food is a general health concern in Brazil, as it is worldwide, which speaks to the necessity of evaluating food safety with respect to the presence of non-essential trace elements in the edible parts of major food crops. Such evaluation is especially needed for crops grown under field conditions, as studies conducted under such circumstances are quite rare in Brazil.

In view of the concern for food safety, this study assessed the contents of As, Cd, and Pb in the edible parts of five major crops (rice, potatoes, wheat, corn, and soybeans) cultivated in areas that use high rates of P fertilizers in Brazil. To the best of our knowledge, this is the first paper dealing with assessment of such trace-elements in field areas, which is relevant not only for food security purposes, but also for commercial and economics aspects.

2. Materials and methods

Soil and crop samples were obtained from field experiments conducted in Mato Grosso and Minas Gerais States, Brazil, during the 2010/11 growing season. All areas in which the field experiments are located have been using high application rates of phosphate fertilizers for several years (at least 8 years). Composite soil samples (500 g) were made up of 10 separate subsamples, which were collected from the 0 to 20 cm soil layer and then stored in plastic bags until analysis. A composite soil sample from uncultivated soils (reference soil) was also collected near each experimental field to investigate changes in soil attributes (mainly As, Cd, and Pb contents) due to cultivation. Additional details of these experiments are presented below.

2.1. Rice experiments

Soil and rice (*Oryza sativa* L.) samples were collected from an experimental field at the Empresa de Pesquisa Agropecuária de

Minas Gerais (Epamig) experimental station, located in Patos de Minas (MG), Brazil (18°31'6.63"S, 46°26'25.13"W). The experiment was conducted in field plots of 5 × 1.5 m arranged in a randomized block design with three replicates ($n = 60$). The plots were previously fertilized with a single application of 400 kg ha⁻¹ of commercial fertilizer formula NPK 08-28-16 + micronutrients applied at the beginning of the growing season and then cultivated with the following upland rice cultivars and accessions: (1) BRSMG Curinga; (2) MG1097-2; (3) BRA042048; (4) BRSGO Serra Dourada; (5) Canastra; (6) BRSMG Conai; (7) MG1097-7; (8) MG1097-4; (9) MG1097-9; (10) MG1097-16; (11) BRSMG Caravera; (12) BRSMG Relâmpago; (13) CG3-118-6; (14) CMG1511; (15) CMG1152; (16) CMG1164; (17) CMG1128; (18) CMG1271; (19) CMG1509; and (20) CMG1284. Additional nitrogen was top-dressed at a rate of 500 kg ha⁻¹ of ammonium sulfate, 25 and 45 days after germination. One composite sample of rice grains (200–300 mg) was harvest at the mature stage by hand from each experimental field plot and stored in a cold chamber (4 °C).

2.2. Wheat experiments

Soil and wheat (*Triticum aestivum* L.) samples were collected from an experimental field at Cooperativa Agropecuária do Alto Paranaíba (Coopadap), located in São Gotardo (MG), Brazil, (19°12'24.48"S, 46°10'9.27"W). The experiment was conducted in field plots of 2 × 5 m arranged in a randomized block design with three replicates ($n = 30$), cultivated without irrigation and with the following wheat cultivars and accessions: (1) CD108; (2) Brilhante; (3) PF020122; (4) PF020037; (5) PF04006; (6) BR18; (7) BR220; (8) BR229; (9) IPF79812; and (10) IPF79813. The area was previously fertilized with 300 kg ha⁻¹ of the commercial fertilizer formula NPK 08-28-16. Additional nitrogen and potassium was top-dressed at a rate of 50 kg ha⁻¹ of the commercial fertilizer formula NPK 20-00-20, 15 days after germination. One composite sample of wheat grains (200–300 mg) was harvest at the mature stage by hand from each experimental field plot and stored in a cold chamber (4 °C).

2.3. Soybean and corn experiments

Soil, soybean (*Glycine max* L.), and corn (*Zea mays* L.) samples were collected from a field experiment at the Fundação de Apoio à Pesquisa Agropecuária de Mato Grosso (Fundação MT) experimental station, located in Itiquira (MT), Brazil, (17°09'20"S, 54°45'11"W). These areas have a well-known record of long-term application of phosphate fertilizers (up to 2700 kg of P₂O₅ per hectare) due to a history of 30 years of grain production. The experiments involved cultivation under crop rotation for 3 years and were established in field plots of 20 m × 30 m arranged in a randomized block design with four replicates (soybean: $n = 28$; corn: $n = 16$). The soybean treatments consisted of seven crop rotations: (1) soybean monoculture in a conventional tillage system (S-CT); (2) soybean monoculture in a non-tillage system (S-NT); (3) soybean/millet (S/M); (4) soybean/*Brachiaria* (S/B); (5) soybean/*Sunnhemp* (S/S); (6) soybean/corn + *Brachiaria* (S/C + B); and (7) soybean/corn (S/C). The corn treatments consisted of three crop rotations: (1) corn + *Brachiaria* (C + B); (2) soybean/corn + *Brachiaria* (S/C + B); and (3) soybean/corn (S/C). The plots received single superphosphate at an annual rate of 225 kg ha⁻¹ and KCl at an annual rate of 150 kg ha⁻¹. Glyphosate was applied at a rate of 2.5 L ha⁻¹. The area was cultivated without irrigation. Composite samples of soybean and corn grains (200–300 mg) were harvest at the mature stage by hand from each experimental field plot and stored in a cold chamber (4 °C).

2.4. Potato experiments

Soil and potato samples (*Solanum tuberosum* L.) were collected from five experimental areas: Lavras (area I – 21°14'12"S, 45°59'35"W), Ipuíuna (areas II – 22°05'00"S, 46°11'04"W – and III – 22°05'12"S, 46°11'16"W), Gonçalves (area IV – 22°41'31"S, 45°57'30"W), and Senador Amaral (area V – 22°35'18"S, 46°10'59"W) in Minas Gerais, Brazil. Each area was previously fertilized with 4000 kg ha⁻¹ of the commercial fertilizer formula NPK 04-14-08. Additional nitrogen was top-dressed at a rate of 350 kg ha⁻¹ of ammonium sulfate, 30–40 days after planting. All areas were cultivated under conventional tillage and without irrigation. Other cultural practices, including pesticide applications, were performed according to the procedures commonly employed for potato cultivation in the highlands of Minas Gerais State (Rodrigues et al., 2009). The experiments were conducted in field plots of 0.30 × 0.80 m arranged in a randomized block design with 10 plants in each line and three replicates (n = 55). Potato tuber samples (900–1000 g) were harvested at the mature stage by hand from each studied area, washed with distilled water, peeled and storage in a cold chamber (4 °C).

2.5. Sample preparation and chemical analysis

The samples were prepared and analyzed following a rigid quality assurance/quality control (QA/QC) program to ensure accurate and reliable analytical data. The plant samples (harvested products) were rinsed with distilled water, oven dried at 65 °C, and ground to <0.38 mm using a stainless steel mill before analysis, while the soil samples were air-dried and ground and sieved to <2 mm.

The chemical properties of the soil samples were determined following the methodology proposed by Silva (2009). Briefly, soil pH was determined in a 1:2.5 soil:water suspension. Soil organic matter content was determined using the potassium dichromate (K₂Cr₂O₇) method, which consists in oxidizing an aliquot of soil (0.5 g) with a solution of K₂Cr₂O₇ + H₂SO₄ at 160 °C. The excess of dichromate is titrated with 0.25 mol L⁻¹ FeSO₄. Calcium and Mg were extracted mixing a volumetric soil aliquot (10 cm³) with 100 mL of KCl (1 mol L⁻¹) at room temperature overnight. Available soil P, K, Fe, Zn, Mn, and Cu were extracted using 100 mL of the Mehlich-1 solution (0.05 mol L⁻¹ HCl + 0.0125 mol L⁻¹ H₂SO₄) reacted with 10 cm³ of soil sample. The CEC was calculated as the sum of Ca, Mg, K, and Al (extracted by 1 mol L⁻¹ KCl) (Silva, 2009). Calcium, Mg, Fe, Mn, Zn, and Cu concentrations in the extracted solutions were determined by flame atomic absorption spectrometer (F-AAS, Perkin-Elmer® AAnalyst™800 – Waltham, MA, USA), using either certified or Sigma–Aldrich® single elements

AAS standards for QA/QC. Available K was measured by flame photometry and available P was determined by colorimetry.

The soil and harvested product samples were microwave-digested according to USEPA Method 3051A (USEPA, 1998) using a CEM® Mars-5 microwave system (Matthews, NC, USA), to determine the concentrations of As, Cd, and Pb. An aliquot of a 0.5-g sample was combined with 10 mL of HNO₃ in Teflon® PTFE vessels and digested for 10 min in the microwave. All the reagents used (Sigma–Aldrich® – St Louis, MO, USA or Merck® – Darmstadt, Germany) were of high purity, and the HNO₃ was distilled prior to use in the digestions.

The As, Cd and Pb concentrations in the digested solutions were determined by graphite-furnace atomic absorption spectrometer (GF-AAS, Perkin-Elmer® AAnalyst™800 – Waltham, MA, USA). The analytical detection limits (DLs) for As, Cd, and Pb in the plant extracts were calculated as $DL = 3 \cdot S + \bar{X}$, where S is the standard deviation of the blank digestion and \bar{X} is the mean TE content in the blank samples (n = 7). The calculated detection limits for plants were approximately 7 µg Cd kg⁻¹, 15 µg As kg⁻¹ and 40 µg Pb kg⁻¹.

Standard reference materials from the Institute for Reference Materials and Measurements (BCR® 482 Lichen and BCR® 142R Sandy Soil – Geel, Belgium) and from the National Institute of Standards and Technology (SRM 2710 Montana Soil – Gaithersburg, MD, USA) were used to substantiate the accuracy of the analytical results obtained. Blank and certified reference samples were analyzed along with every batch of digestion.

The R software (R-2.13.1 version) was used for the statistical analyses. The differences were determined by an ANOVA and Tukey's test to investigate statistically significant differences at p < 0.05.

3. Results and discussion

The mean As, Cd, and Pb concentrations obtained from the repeated analysis (n = 12) of the standard reference materials are presented in Table 1. The TE content recoveries in the certified samples exhibited reliable analytical data accuracy for As, Cd, and Pb analysis.

3.1. As, Cd and Pb contents in rice

Table 2 presents the soil chemical properties, and Table 3 displays the As, Cd, and Pb contents in the uncultivated soil (reference) and in the area cultivated with rice. The results show a high natural P content in these soils, which might be related to the P-rich parent material (tuffite). Motta et al. (2002) obtained similar results studying soils from the same region (Patos de Minas, Minas

Table 1
Recovery of As, Cd and Pb from reference standards.

		Reference material	Element [†]	Mean recovery ^{**}
BCR® – 482 Lichen	As	µg kg ⁻¹ 850 ± 70	µg kg ⁻¹ 803 ± 90	% 94
	Cd	560 ± 20	474 ± 30	85
	Pb	40,900 ± 1400	41,326 ± 6004	101
BCR® – 142R Light Sandy Soil	As	µg kg ⁻¹ –	µg kg ⁻¹ –	% –
	Cd	249 ± 10	230 ± 46	92
	Pb	25,700 ± 1600	21,178 ± 3300	82
NIST 2710 Montana Soil I	As	mg kg ⁻¹ 1540 ± 10	mg kg ⁻¹ 1709 ± 73	% 111
	Cd	12.3 ± 0.3	14 ± 1.8	114
	Pb	5520 ± 30	5766 ± 195	105

–: value not certified.

[†] Mean of 12 measurements of standard reference material samples.

^{**} Mean recovery (%) = (Mean_{determined concentration}/Mean_{certified value}) × 100%.

Table 2
Soil chemical characteristics of rice areas.

Soil – rice	pH	P (mg dm ⁻³)	K (mg dm ⁻³)	Ca (mg dm ⁻³)	Mg (cmol _c dm ⁻³)	CEC (cmol _c dm ⁻³)	OM (g kg ⁻¹)	Fe (mg dm ⁻³)	Zn (mg dm ⁻³)	Cu (mg dm ⁻³)	Mn (mg dm ⁻³)
Reference	5.8 ± 0.4	34 ± 3	36 ± 4	0.7 ± 0.2	0.5 ± 0.01	1.4 ± 0.1	31 ± 1	43 ± 2	1.1 ± 0.1	10 ± 0.3	43 ± 7
Cultivated	4.8 ± 0.3	55 ± 8	28 ± 5	0.8 ± 0.3	0.4 ± 0.01	1.8 ± 0.3	36 ± 2	45 ± 2	2.6 ± 0.7	11 ± 0.6	77 ± 13

Values are the means ± standard deviation (SD) (n = 3).

Gerais State, Brazil). Although the P content is naturally high, the studied area has a record of long-term application of P fertilizers (e.g., 20 years), resulting in a greater soil P content in the cultivated area. Nonetheless, the As, Cd, and Pb contents of the cultivated and non-cultivated soils were not significantly different ($p < 0.05$). Furthermore, there were no significant correlations between the P content and the contents of the studied TEs in the soil or in the rice grains (data not shown, since all p values were greater than 0.20). The contents of As, Cd, and Pb in the studied soils were all lower than the range of maximum allowable concentrations of TEs in agricultural soils (As: 15–20; Cd: 1–5; Pb: 20–300 mg kg⁻¹) (Kabata-Pendias and Mukherjee, 2007).

Although rice is a crop with a high potential to accumulate contaminants such as As (Peralta-Videa et al., 2009), the low contents of As and Pb in the soil (Table 3) resulted in As and Pb levels below the detection limit (DL) for the rice samples (DL As: 15 µg kg⁻¹ and Pb: 40 µg kg⁻¹), indicating no grain contamination by these TEs. Zhang et al. (2011) evaluated the As, Cd, and Pb contents in rice grains grown in China and detected As concentrations below 600 µg kg⁻¹ and Pb contents below 5000 µg kg⁻¹. The median concentration of inorganic As in white rice samples collected in the United States was 112 µg kg⁻¹ (Lamont, 2003).

The average Cd concentration in rice grains was 29 µg kg⁻¹ dry weight (DW) (Fig. 1). The threshold level proposed by the Codex Alimentarius Commission is 400 µg kg⁻¹ fresh weight (FW) (Joint FAO/WHO Food Standards Programme, 2001). Assuming that a rice grain contains approximately 23% water, the upper limit for Cd on a dry basis would be 520 µg kg⁻¹. The threshold level proposed by the European Commission is 200 µg kg⁻¹ FW, which is 260 µg kg⁻¹ DW (European Commission, 2006). In our study, none of the evaluated cultivars or accessions presented Cd concentrations in rice grains in excess of the abovementioned maximum allowable concentrations. Zhang et al. (2011) reported Cd concentrations up to 3000 µg kg⁻¹ in rice grains cultivated in China, yet most of the studied samples had values below 400 µg kg⁻¹, which are in accordance with the Codex Alimentarius guidelines.

In our study, the Cd concentrations in the rice grains were not significantly different among the cultivars and accessions (Fig. 1). Earlier investigations have found significant genotype variation in Cd concentration in rice grains cultivated in China (Cheng et al., 2006) and Japan (Arao and Ae, 2003). Such differences may be related to physiological and morphological characteristics (Yan et al., 2010), which seem not to affect the Cd content in the cultivars considered in the present study.

Table 3
Arsenic, cadmium and lead contents (mg kg⁻¹) in soils from rice areas.

Soil – rice	As	Cd	Pb
Reference	12 ± 2.0	0.05 ± 0.01	18 ± 2
Cultivated	11 ± 0.5	0.07 ± 0.03	16 ± 2

Values are the means ± standard deviation (SD) (n = 3).

3.2. As, Cd and Pb concentrations in wheat

Except for iron (Fe) and copper (Cu), the values for most soil attributes in the area cultivated with wheat are greater than those in the reference area (Table 4). For As, Cd, and Pb, the greatest difference between the cultivated and reference areas was in the level of Cd (Table 5). No significant correlations between P and the TE contents in the soil and wheat grains were detected (data not shown, since all p values were greater than 0.20).

The average As concentration in the wheat grains (19 µg kg⁻¹) (Fig. 2) is lower than that proposed by Brazilian legislation for cereals and their products (limit: 1000 µg kg⁻¹) (ANVISA, 1998). There is no As threshold level proposed by the Codex Alimentarius Commission or the European Union for cereal or related products (Joint FAO/WHO Food Standards Programme, 2001; European Commission, 2006). Škrbic and Onjia (2007) found an average of 83 µg kg⁻¹ DW in wheat grains grown in Serbia. Huang et al. (2008) evaluated different trace element levels in wheat grains cultivated in different areas of Kunshan, China, and found a range from 29 to 86 µg kg⁻¹ DW for As concentrations, while Liu et al. (2009) detected As concentrations ranging from 110 to 160 µg kg⁻¹ DW in Zhengzhou, China. In wheat grains grown in Italy, the highest As concentration (60 µg kg⁻¹ DW) (Cubadda et al., 2010) was 2-fold higher than the highest the values observed in our study (27 µg kg⁻¹ DW).

A significant difference among the cultivars was observed only for the cultivar CD108, which showed the lowest As concentration (Fig. 2). Zhang et al. (2009) found differences in As concentrations in grains among wheat cultivars grown in China. However, wheat cultivars grown in Italy exhibited no differences in As concentration (Cubadda et al., 2010).

The Cd grain concentrations varied from <15 to 83 µg kg⁻¹ DW (Fig. 2). The maximum permitted Cd level in a wheat grain proposed by the Codex Alimentarius Commission is 200 µg kg⁻¹

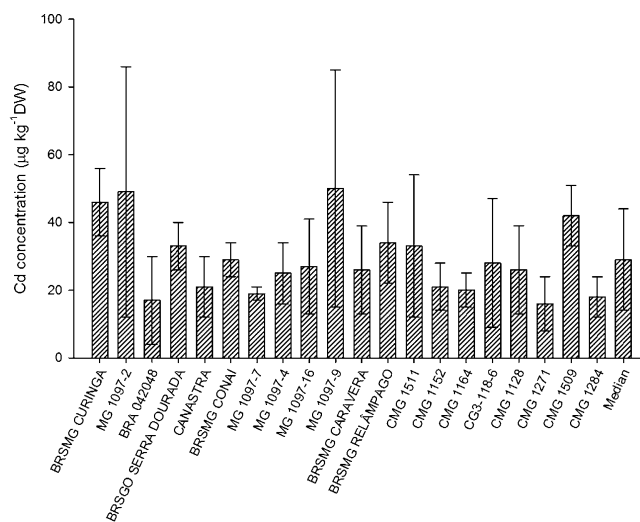


Fig. 1. Grain Cd concentration in rice cultivars and accessions. Values shown are mean ± SD (n = 3).

Table 4
Soil chemical characteristics of wheat areas.

Soil – wheat	pH	P (mg dm ⁻³)	K (mg dm ⁻³)	Ca (mg dm ⁻³)	Mg (cmol _c dm ⁻³)	CEC (cmol _c dm ⁻³)	OM (g kg ⁻¹)	Fe (mg dm ⁻³)	Zn (mg dm ⁻³)	Cu (mg dm ⁻³)	Mn (mg dm ⁻³)
Reference	5.2 ± 0.2	1.8 ± 0.3	25 ± 4	0.1 ± 0.02	0.1 ± 0.01	0.9 ± 0.2	31 ± 5	76 ± 7	0.4 ± 0.1	1.6 ± 0.07	2.7 ± 0.1
Cultivated	5.3 ± 0.4	27.3 ± 6.2	98 ± 12	2.3 ± 0.61	0.3 ± 0.04	3.1 ± 0.7	44 ± 2	39 ± 5	14.2 ± 1.1	1.4 ± 0.06	10.2 ± 2.1

Values are the means ± standard deviation (SD) (n = 3).

Table 5
Arsenic, cadmium and lead contents (mg kg⁻¹) in soils from wheat areas.

Soil – wheat	As	Cd	Pb
Reference	9 ± 3	0.010 ± 0.002	11 ± 2
Cultivated	11 ± 2	0.210 ± 0.020	14 ± 2

Values are the means ± standard deviation (SD) (n = 3).

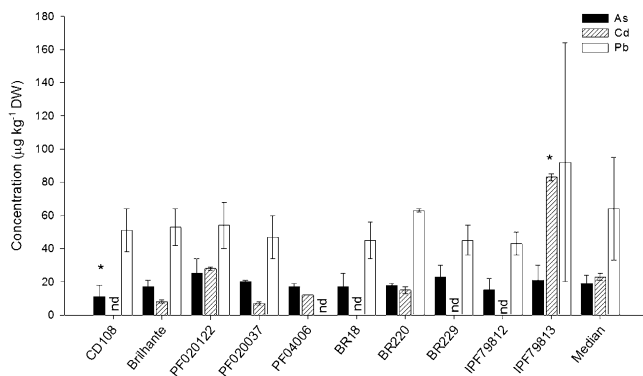


Fig. 2. Grain As, Cd and Pb concentrations in wheat cultivars and accessions. Values shown are mean ± SD (n = 3). nd = not detected. Detection limit: As = 15 µg kg⁻¹; Cd: 7 µg kg⁻¹; Pb: 40 µg kg⁻¹ (3051A). * Significantly different among other samples for the same trace-element.

FW (Joint FAO/WHO Food Standards Programme, 2001). Assuming that a wheat grain contains approximately 16% water, the upper limit for Cd on a dry basis would be 240 µg kg⁻¹ DW. In this study, the results for Cd in wheat grains did not exceed this maximum allowable concentration. In a study in China, the Cd concentrations of wheat grains ranged from 18 to 23 µg kg⁻¹ DW (Liu et al., 2009).

Differences between wheat cultivars with respect to Cd uptake, transport (Adams et al., 2004) and concentration (Wu et al., 2002; Jamali et al., 2009) have been reported in the literature. In our study, a significant difference was observed only for the accession IPF79813, which had the highest Cd concentration (Fig. 2).

The Pb concentration in wheat grains was, on average, 64 µg kg⁻¹ DW, and there were no differences among cultivars and accessions (Fig. 2). A limit of 200 µg kg⁻¹ FW (240 µg kg⁻¹ DW for wheat) has been proposed as the maximum Pb level for cereal grains (Joint FAO/WHO Food Standards Programme, 2001). The Pb concentration in wheat grains grown in Kunshan, China, ranged from 17 to 1158 µg kg⁻¹ DW (average concentration: 177 µg kg⁻¹ DW) (Huang et al., 2008), and those in Zhengzhou ranged from 99 to 1010 µg kg⁻¹ DW (Liu et al., 2009). For comparison, the average Pb concentrations reported in the literature for wheat

grains grown in Serbia was 366 µg kg⁻¹ DW (Škrbic and Onjia, 2007).

3.3. As, Cd and Pb concentrations in soybeans

The soybean experimental area has a record of long-term application of high rates of phosphate fertilizers (i.e., 30 years of grain production of soybean, maize, and sorghum) (Table 6), which could indicate possible soil contamination by fertilizer-derived TEs and their availability to plants (Molina et al., 2009). In the present study, there was a trend of increasing concentration of soil TEs (especially Cd) following cultivation (Table 7). However, we did not detect any correlations between P and the contents of As, Cd, and Pb in the soil or As and Pb in the soybean grains (data not shown, since all p values were greater than 0.20).

The average As concentration in soybean grains was 65 µg kg⁻¹ DW (Fig. 3). There is no threshold level proposed for As by the Codex Alimentarius Commission, the European Union, or Brazilian legislation for either beans or soybeans. However, there is a level proposed for vegetable oils, including soybean oil (maximum level = 100 µg kg⁻¹) (Joint FAO/WHO Food Standards Programme, 2001). Soybean grains cultivated near a lead/zinc mine area in Hunan, China, showed an average As concentration of 800 µg kg⁻¹ DW (Liu et al., 2005).

The results of Cd concentration in the soybean grains are detailed in Corguinha et al. (2012). The Pb concentration in soybean grains in our study ranged from 90 to 114 µg kg⁻¹ DW, with an average of 103 µg kg⁻¹ (Fig. 3). The threshold level proposed by the Codex Alimentarius Commission and the European Union is 200 µg kg⁻¹ FW (360 µg kg⁻¹ DW) for vegetables (Joint FAO/WHO Food Standards Programme, 2001; European Commission, 2006). Values in excess of the abovementioned threshold were reported by Cao et al. (2009), who found Pb concentrations ranging from 293 to 538 µg kg⁻¹ FW in soybean seeds cultivated in agricultural black soils in China. On the other hand, Liu et al. (2005) found an average Pb concentration of 200 µg kg⁻¹ DW in soybean grains cultivated near a lead/zinc mine area in Hunan, China, whereas Lavado et al. (2001) observed 800–850 µg Pb kg⁻¹ FW in grains collected from uncontaminated areas in Argentina.

Crop rotation is a management practice used to avoid soil exhaustion while improving the physical, chemical, and biological properties of the soil and helping control pests and diseases (EMBRAPA, 2004). This management practice might also help prevent the depletion or concentration of specific elements (e.g., TEs) caused by single crops. Although other studies have shown significant decreases in Cd and Pb concentrations in plants cultivated under a crop rotation system (Wu et al., 2011), in the

Table 6
Soil chemical characteristics of soybean and corn areas.

Soil – soybean	pH	P (mg dm ⁻³)	K (mg dm ⁻³)	Ca (mg dm ⁻³)	Mg (cmol _c dm ⁻³)	CEC (cmol _c dm ⁻³)	OM (g kg ⁻¹)	Fe (mg dm ⁻³)	Zn (mg dm ⁻³)	Cu (mg dm ⁻³)	Mn (mg dm ⁻³)
Reference	4.3 ± 0.3	1.2 ± 0.04	39 ± 8	0.1 ± 0.01	0.2 ± 0.01	1.8 ± 0.3	49 ± 5	106 ± 22	0.4 ± 0.06	0.8 ± 0.2	5.4 ± .22
Cultivated	5.3 ± 0.6	24.1 ± 11.0	100 ± 13	2.6 ± 1.00	0.8 ± 0.30	4.6 ± 1.2	37 ± 4	45 ± 14	7.6 ± 2.02	2.6 ± 0.6	20.1 ± 6.1

Values are the means ± standard deviation (SD) (n = 3).

Table 7Arsenic, cadmium and lead contents in (mg kg^{-1}) soils from soybean and corn areas.

Soil – soybean	As	Cd	Pb
Reference	13 ± 0.5	0.01 ± 0.002	10 ± 0.6
Cultivated	16 ± 2.0	0.11 ± 0.071	11 ± 1.0

Values are the means \pm standard deviation (SD) ($n=3$).

present study, no significant differences were detected in the As and Pb concentrations in the soybean grains grown under crop rotation (Fig. 3).

3.4. As, Cd and Pb concentrations in corn

Corn and soybeans were cultivated in the same experimental areas. Again, although the soil in the cultivated area had a higher P content than the reference soil (Table 6), suggesting a higher content of fertilizer-derived TEs (Table 7), no correlation was detected between the P and TE contents in the soil or corn grains (data not shown, since all p values were greater than 0.20).

The As concentration in the corn grains ranged from 42 to 51 $\mu\text{g kg}^{-1}$ DW (Fig. 4). There is no maximum permitted level of As in cereals and related products established by the *Codex Alimentarius* or the European Union (Joint FAO/WHO Food Standards Programme, 2001; European Commission, 2006), while the maximum level for cereals permitted by Brazilian legislation is 100 $\mu\text{g kg}^{-1}$ (ANVISA, 1998). Queirolo et al. (2000) evaluated the As concentration in corn grains cultivated in a region characterized by volcanic events in northern Chile and found that the As concentration (1850 $\mu\text{g kg}^{-1}$ FW) exceeded the Chilean National Standard for As (500 $\mu\text{g kg}^{-1}$ FW) by approximately 400%. The As concentration in corn grains (70 $\mu\text{g kg}^{-1}$ FW) grown in areas of long-term application of phosphate fertilizers in China was found to be significantly lower than the level found in the corn roots (1690 $\mu\text{g kg}^{-1}$ FW), which indicates that there is low transferability of As from roots to grains (Li et al., 2010).

The corn grains had an average Cd concentration of 40 $\mu\text{g kg}^{-1}$ DW (Fig. 4). The Cd values in all corn samples are lower than the maximum permitted for cereals by *Codex Alimentarius*, at 100 $\mu\text{g kg}^{-1}$ FW (Joint FAO/WHO Food Standards Programme, 2001). Assuming a corn grain contains approximately 12% water, the upper limit for Cd on a dry basis would be 110 mg kg^{-1} . Lavado et al. (2001) found a Cd concentration of 50 $\mu\text{g kg}^{-1}$ in corn grains, while the concentration found by Queirolo et al. (2000) was 4 $\mu\text{g kg}^{-1}$ FW.

The average Pb concentration in corn grains was 95 $\mu\text{g kg}^{-1}$ DW (Fig. 4). The threshold level proposed by the *Codex Alimentarius* is 200 $\mu\text{g kg}^{-1}$ FW (230 $\mu\text{g kg}^{-1}$ DW) (Joint FAO/WHO Food Standards Programme, 2001). Lavado et al. (2001) found 800–

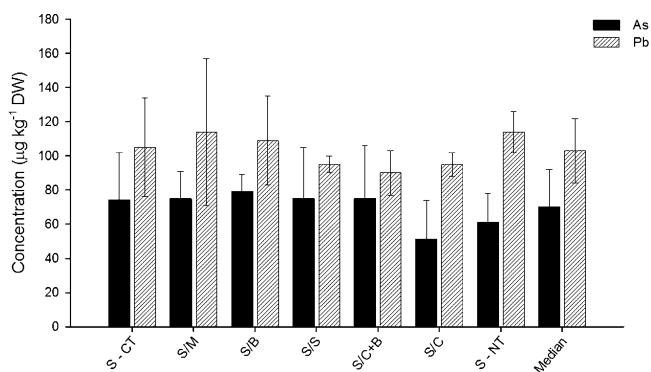


Fig. 3. Grain As and Pb concentrations in soybeans cultivated under crop rotation. Values shown are mean \pm SD ($n=3$).

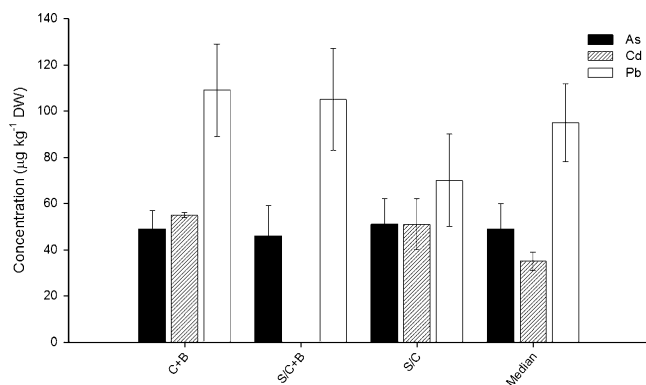


Fig. 4. Grain As, Cd and Pb concentrations in corn cultivated under crop rotation. Values shown are mean \pm SD ($n=3$).

980 $\mu\text{g Pb kg}^{-1}$ FW in corn grains. However, the average Pb concentration in maize grains cultivated in Socaire, Chile, was 24 $\mu\text{g kg}^{-1}$ FW (Queirolo et al., 2000).

In the present study, no differences were detected in the As, Cd, and Pb concentrations in corn grains under crop rotation (Fig. 4, $p < 0.05$). A short-rotation coppice of wood plants may provide efficient TE removal from soils, especially for Cd. However, there is no evidence that this practice interferes with the availability of As and Pb to plants (French et al., 2006). Aboveground corn biomass accumulates less Cd when grown under crop rotation than a monoculture system (Pavlíková et al., 2007).

3.5. As, Cd, and Pb concentrations in potatoes

Despite the previous use of P fertilizers for crop production, the potato experimental areas had a high single-dose application of P fertilizer that resulted in higher P levels in the cultivated soils than the reference soils (Table 8). The As and Pb contents in the soils of the five studied areas were not significantly different (Table 9), and there were no correlations between P and the TE contents in the soil and in the potato tubers (data not shown, since all p values were greater than 0.20).

The average As concentration in the potato tubers was 59 $\mu\text{g kg}^{-1}$ DW (Fig. 5). However, there is no maximum level of As permitted by Brazilian legislation (Anvisa), the *Codex Alimentarius*, or the European Union for tubers or roots. In potato tubers cultivated in Chile, Muñoz et al. (2002) detected an As concentration of 58 $\mu\text{g kg}^{-1}$ FW, and Schoof et al. (1999) found concentrations lower than the DL. High As levels were found in potatoes cultivated in northern Chile, in the villages of Socaire (860 $\mu\text{g kg}^{-1}$ FW) and Talabre (241 $\mu\text{g kg}^{-1}$ FW), representing a 3.5-fold difference in As content for tubers grown in different areas. These locations are volcano-influenced, most likely leading to the As contamination of the region's soils (Queirolo et al., 2000). In the present study, no differences were detected in the As levels in potato tubers grown in the different areas studied (Fig. 5), which were all uncontaminated areas.

Results concerning Cd concentrations in potato tubers are detailed in Corguinha et al. (2012). The presence of low Pb contents in the soil (Table 9) resulted in Pb levels below the detection limit (DL) for the potato tubers (DL: 40 $\mu\text{g kg}^{-1}$), indicating no product contamination by this TE. The maximum permitted level of Pb in peeled potatoes established by the *Codex Alimentarius* and the European Union is 100 $\mu\text{g kg}^{-1}$ FW. Llobet et al. (2003) found Pb concentrations in vegetables, including potatoes, from cities in Catalonia, Spain, that were less than 20 $\mu\text{g kg}^{-1}$ FW. De Pieri et al. (1997) reported values ranging from 30 to 60 $\mu\text{g kg}^{-1}$ FW for samples from three regions of the Lower Fraser Valley of British Columbia.

Table 8

Soil chemical characteristics of potato areas.

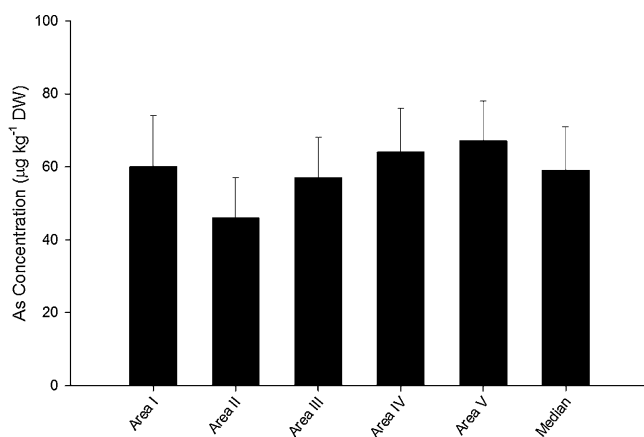
Area	Soil	pH	P (mg dm ⁻³)	K (mg dm ⁻³)	Ca (mg dm ⁻³)	Mg (cmol _c dm ⁻³)	CEC (cmol _c dm ⁻³)	OM (g kg ⁻¹)	Fe (mg dm ⁻³)	Zn (mg dm ⁻³)	Cu (mg dm ⁻³)	Mn (mg dm ⁻³)
Area I	Reference	6.0 ± 0.3	4.0 ± 0.1	30 ± 1	3.0 ± 0.3	0.6 ± 0.10	3.8 ± 0.2	19 ± 1	40 ± 6	4.2 ± 0.4	3.3 ± 0.4	45.7 ± 7.1
	Cultivated	5.5 ± 0.4	5.2 ± 0.2	53 ± 2	1.8 ± 0.2	0.6 ± 0.20	2.7 ± 0.3	22 ± 2	38 ± 2	5.3 ± 1.2	3.7 ± 0.3	21.9 ± 4.2
Area II	Reference	4.9 ± 0.4	4.0 ± 0.1	86 ± 4	0.3 ± 0.1	0.5 ± 0.01	14.7 ± 0.6	41 ± 3	106 ± 9	0.8 ± 0.2	0.6 ± 0.1	7.6 ± 2.0
	Cultivated	5.5 ± 0.6	5.2 ± 0.2	53 ± 5	1.8 ± 0.4	0.4 ± 0.02	2.7 ± 0.4	22 ± 2	64 ± 7	0.9 ± 0.1	0.5 ± 0.1	10.1 ± 3.1
Area III	Reference	4.9 ± 0.5	4.0 ± 0.1	86 ± 4	0.3 ± 0.1	0.5 ± 0.01	14.7 ± 0.6	41 ± 3	106 ± 9	0.8 ± 0.2	0.6 ± 0.1	7.6 ± 2.0
	Cultivated	5.5 ± 0.2	13.0 ± 0.4	55 ± 3	1.8 ± 0.4	0.4 ± 0.02	2.6 ± 0.2	27 ± 4	64 ± 6	1.0 ± 0.1	0.5 ± 0.1	9.6 ± 2.2
Area IV	Reference	5.0 ± 0.2	11.0 ± 0.6	101 ± 12	0.4 ± 0.1	0.3 ± 0.02	2.8 ± 0.4	134 ± 7	84 ± 10	2.2 ± 0.3	0.6 ± 0.2	37.2 ± 4.0
	Cultivated	5.0 ± 0.1	50.0 ± 0.8	123 ± 10	1.8 ± 0.3	0.5 ± 0.04	3.8 ± 0.5	46 ± 4	51 ± 7	3.4 ± 0.4	0.3 ± 0.1	36.8 ± 5.7
Area V	Reference	4.4 ± 0.4	8.2 ± 0.4	50 ± 3	0.3 ± 0.1	0.1 ± 0.01	2.8 ± 0.5	48 ± 4	98 ± 9	8.5 ± 0.2	1.7 ± 0.4	0.3 ± 0.1
	Cultivated	5.0 ± 0.3	51.0 ± 0.7	41 ± 2	1.1 ± 0.2	0.1 ± 0.02	1.7 ± 0.2	16 ± 2	119 ± 13	11.0 ± 0.5	1.7 ± 0.5	2.3 ± 0.2

Values are the means ± standard deviation (SD) (n = 3).

Table 9Arsenic and lead contents (mg kg⁻¹) in soils from potato areas.

Area	Soil	As	Cd	Pb
Area I	Reference	10 ± 3	0.01 ± 0.007	16 ± 1.0
	Cultivated	18 ± 6	0.04 ± 0.020	18 ± 1.4
Area II	Reference	12 ± 3	0.01 ± 0.004	10 ± 0.3
	Cultivated	17 ± 3	0.02 ± 0.001	10 ± 0.2
Area III	Reference	12 ± 3	0.01 ± 0.004	10 ± 0.3
	Cultivated	18 ± 2	0.02 ± 0.006	10 ± 0.7
Area IV	Reference	7 ± 2	0.07 ± 0.010	17 ± 0.7
	Cultivated	11 ± 3	0.10 ± 0.020	21 ± 0.5
Area V	Reference	7 ± 2	0.02 ± 0.002	23 ± 4.2
	Cultivated	11 ± 3	0.04 ± 0.006	17 ± 2.4

Values are the means ± standard deviation (SD) (n = 3).

**Fig. 5.** Tuber As concentration in potato cultivars from different agricultural areas. Values shown are mean ± SD (n = 3).

4. Conclusions

The long-term application of P fertilizers in the major crops evaluated in this study resulted in higher soil P contents in cultivated areas than in reference soils. However, the As, Cd, and Pb contents in the soils and in the studied crops did not follow the same trend. The As, Cd, and Pb (in µg kg⁻¹ dry weight) concentrations were <15, 29, and <40 for rice; 19, 23, and 64 for wheat; 47, 40, and 95 for soybeans; 65, 23, and 106 for corn; and 59, 22, and <40 for potatoes, respectively. The As, Cd, and Pb concentrations in the harvested products are in accordance with the *Codex Alimentarius*, European Union, and Brazilian guidelines.

Conflict of interest

The authors declare that there are no conflicts of interest.

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