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Exploratory analysis of trace elements in soils and plants affected by a gossan in the Semiárido¹

Análise exploratória de elementos-traço em solos e plantas afetados por um gossan no Semiárido

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HIGHLIGHTS:

Concentration of metals extracted by DTPA method was higher than Mehlich-1, with exception of Zn.

Soils under the presence of the gossan had high Pb and Zn but no sign of toxicity in plants.

Plants showed low bioconcentration of potential toxic elements due to its low availability in the soil.

ABSTRACT: Trace elements in high concentrations are a huge problem worldwide. Monitoring of natural areas with a high concentration of these elements, such as soils under the influence of gossans, is important since there is little information available. This study aimed to evaluate the pseudo total and available concentrations of Fe, Mn, Pb, and Zn in the soil and its accumulation in native and cultivated plants in the area under the influence of gossan in the Semiárido region in Brazil. Soil samples (0-20 cm) were collected in the North, South, and center areas of the gossan and five transections separated by 1000 m. The pseudo total (EPA3050A) and available concentration (DTPA, Mehlich) of Fe, Mn, Pb, and Zn were determined. In the same points, plant samples were collected and processed to determine the concentration and bioconcentration factor of Fe, Mn, Pb, and Zn. Soils under the influence of the gossan had high Pb and Zn concentration (exceeding soil reference values), indicating that care should be taken in these areas to prevent any risk for the local community. Plants showed different accumulation patterns, with a higher accumulation of trace elements in the shoot (Fe, Mn, Zn), while Pb was primarily accumulated in the roots. Even though soil concentrations were high, plants showed in general, low bioconcentration factor of the potentially toxic elements, except for Zn, indicating that soil conditions limit their availability.

Key words: Irecê Plateau, heavy metals, bioconcentration factor, lead, zinc

RESUMO: Elementos-traços em altas concentrações são um grande problema a nível mundial. O monitoramento de áreas naturais com alta concentração destes elementos, como solos sob influência de gossans, é importante, por existir pouca informação disponível. Objetivou-se neste trabalho avaliar as concentrações pseudototais e disponíveis de Fe, Mn, Pb e Zn no solo e seu acúmulo em plantas nativas e cultivadas na área sob influência de gossan no semiárido no Brasil. Amostras de solo (0-20 cm) foram coletadas no norte, sul e área central do gossan e em cinco transecções separadas em 1000 m. Determinou-se a concentração pseudototal (EPA3050A) e disponível (DTPA, Mehlich) de Fe, Mn, Pb e Zn. Nos mesmos pontos, amostras de plantas foram coletadas e processadas para determinação da concentração e fator de bioconcentração de Fe, Mn, Pb e Zn. Os solos sob influência do gossan apresentaram alta concentração de Pb e Zn (excedendo os valores de referência de qualidade), indicando que cuidados devem ser tomados nessas áreas para evitar qualquer risco para a comunidade local. As plantas apresentaram diferentes padrões de acumulação, com maior acúmulo de elementos-traço na parte aérea (Fe, Mn, Zn), enquanto o Pb foi acumulado principalmente nas raízes. Embora as concentrações no solo fossem altas, as plantas apresentaram, em geral, baixo fator de bioconcentração dos potenciais elementos tóxicos, com exceção do Zn, indicando que as condições do solo limitam sua disponibilidade.

Palavras-chave: Platô de Irecê, metais pesados, fator de bioconcentração, chumbo, zinco

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INTRODUCTION

Trace elements are known by their toxic potential and can be classified by their function in plants; as essentials (Cu, Fe, Mn, Zn) and not essentials (As, Cd, Pb, Cr) (Ali et al., 2013). Naturally, contaminated soils are formed from parent material with a high concentration of trace elements. Gossans, defined as great masses of residual material of iron oxyhydroxide, originated from sulfide mineralization of pyrite and, deposited on the surface, with high concentrations of trace elements due to the processes during its formation, where trace elements are strongly adsorbed into the fine precipitates (Atapour & Aftabi, 2007; Faure, 1991). However, some of these soils have high natural fertility, and they are used for agriculture. High trace elements concentration in soils predispose a high absorption of these elements by plants, which are the main source of human exposure for trace elements, contributing to nearly 90% of trace elements consumption in humans (Khan et al., 2015). Generally, the procedure used to explore the possible effects of trace elements toxicity in plants or their impact on human health is the determination of pseudo total and available concentrations in the soil (Alloway, 2013). Accumulation of these elements can also range according to the plant species, organ, genotype, and studied ion (Li et al., 2007; Kabata-Pendias, 2010).

Lack of knowledge in naturally contaminated soils, especially under the influence of gossans, highlights the necessity to study areas with high trace elements concentration with the finality of monitoring the possible health risks to the local community and implications in plant growth and development. Therefore, the objective of this study was to evaluate the pseudo total and available concentrations of Fe, Mn, Pb, and Zn in the soil and its accumulation in native and cultivated plants in the area under the influence of gossan in Lapão-Irecê, Bahia, Brazil.

MATERIAL AND METHODS

The gossan is in Lapão-Irecê, Bahia, Brazil, and it has climate type BSh, according to Köppen & Geiger (1939).

The identification of the gossan was based on a geochemical map for the prospection of metals and phosphates (Bahia, 1997). The types of soils present in the area are Oxisols and Inceptisols (Paiva, 2010). In general, the soils of the area tend to alkaline and with high natural fertility (Silva et al., 1993). The area is covered by typical 'Caatinga' vegetation (*Cereus* sp., *Anadenanthera* sp., etc.); also, maize, bean, and castor cultivation were observed.

Soil samples were collected perpendicularly to the gossan in five transections of 800 m long and with 1000 m between them, totalizing 4000 m in the direction from West to East of the gossan. A central point in each transection was sampled in the gossan; from this point, five samples were taken in direction from North to South, with 25, 50, 100, 200, and 400 m from the central point. These points were organized in three main zones: North, Center, and South, to have an overview of the general distribution of trace elements in the area (Table 1).

The Center represents the area directly affected by the gossan, and North and South the distribution in both directions. Each point was sampled at 0-0.20 m soil layer. Samples were sieved in a 2 mm nylon sieve and stored for analysis. Native and cultivated plants were randomly collected at the same point where the soil was sampled, totalizing 102 samples. Shoot and roots of each plant were collected. The list of plants sampled is shown in Table 2. Plant samples were washed with 0.5 M HCl and dried at 60 °C, mixed, milled, and stored in plastic bags before the analysis.

Pseudo total soil concentrations of Fe, Pb, Mn, and Zn were analyzed according to the procedures of EPA 3050A (USEPA, 2007). For available Fe, Pb, Mn, and Zn concentrations, two extraction methods were used: Mehlich-1 and DTPA (EMBRAPA, 2009). CONAMA N°420 (2009) was used to compare the results with reference values, where three reference values are defined as follows: quality reference value (concentration of the element in soils in natural conditions or non-contaminated), prevention value (limit value of the element in soils that can sustain its primary functions) and investigation value (limit value of an element in the soil that may cause potential risks to human health).

Table 1. Geographic coordinates (UTM) of soil and plant sampling points in the area influenced by gossan in Lapão, Irecê Plateau, BA, Brazil

Ubication	East	South	Ubication	East	South	Ubication	East	South
	185904	8736710		185904	8736335		185904	8736110
	185904	8736510		185904	8736310		185904	8736210
	185904	8736410		185904	8735910		185904	8736260
	185904	8736360		186904	8736335		185904	8736285
	186904	8736710		186904	8736310		186904	8736110
	186904	8736510		186904	8735910		186904	8736210
	186904	8736410		187904	8736335		186904	8736260
	186904	8736360		187904	8736310		186904	8736285
	187904	8736710		187904	8735910		187904	8736110
North (n = 20)	187904	8736510	Center (n = 15)	188904	8736335	South (n = 20)	187904	8736210
	187904	8736410		188904	8736310		187904	8736260
	187904	8736360		188904	8735910		187904	8736285
	188904	8736710		189904	8736335		188904	8736110
	188904	8736510		189904	8736310		188904	8736210
	188904	8736410		189904	8735910		188904	8736260
	188904	8736360					188904	8736285
	189904	8736710					189904	8736110
	189904	8736510					189904	8736210
	189904	8736410					189904	8736260
	189904	8736360					189904	8736285

Table 2. List of plant species assessed in the area under the influence of the gossan in Lapão-Irecê, Brazil

Plant order	Common name	Scientific name	Origin
1	Angico	<i>Anadenanthera colubrina</i>	Native
2	Banana	<i>Musa</i> spp.	Cultivated
3	Barbados nut	<i>Jatropha curcas</i>	Cultivated
4	Bean	<i>Phaseolus vulgaris</i>	Cultivated
5	Brazilian copal	<i>Hymenaea courberil</i>	Native
6	Buffelgrass	<i>Cenchrus ciliaris</i>	Cultivated
7	Cactus pear	<i>Opuntia ficus-indica</i>	Cultivated
8	Castor bean	<i>Ricinus communis</i>	Cultivated
9	Jocote	<i>Spondias purpurea</i>	Cultivated
10	Jurema	<i>Balizia pedicellaris</i>	Native
11	Maize	<i>Zea mays</i>	Cultivated
12	Mucambo	<i>Balfourodendron riedelianum</i>	Native
13	Peruvian pepper	<i>Schinus molle</i>	Cultivated
14	Saguaro	<i>Cereus giganteus</i>	Native
15	Sugar apple	<i>Annona squamosa</i>	Cultivated
16	Umbu caja	<i>Spondias</i> spp.	Cultivated
17	Umburaniha	<i>Bursera</i> sp.	Native

For plant analysis, shoot and roots dry samples were weighed (500 mg each), and digestion was carried out using 5 mL of HNO₃. The digested solution was filtered through a Whatman® Grade 42 filter paper and diluted before analysis. Concentrations of Fe, Mn, Pb, and Zn were determined in the filtrate using ICP-OES (EMBRAPA, 2009).

The bioconcentration factor is defined by Zhuang et al. (2007) as the relationship between the concentration of elements in plants and pseudo total concentration in soils in each point collected. From the results of soils and plants in each location sampled, the bioconcentration factor for Fe, Mn, Pb, and Zn, per plant was determined, and a mean value for each species was used for comparison.

For data analysis, the completely randomized design was considered, and the analysis of variance (ANOVA) was carried out ($p \leq 0.05$). When necessary, data transformations to meet assumptions of normality were performed using logarithm transformations. Finally, means were grouped using the Scott-Knott test ($p \leq 0.05$). Statistical analyses were carried out in statistical software R 3.2.

RESULTS AND DISCUSSION

Pseudo total and available concentrations (Mehlich 1 and DTPA) of Fe, Mn, Pb, and Zn, are presented in Table 3. Significant differences ($p \leq 0.05$) were observed between the sampling location for all the assessed elements. Pseudo total concentrations of Fe ranged from 8.0 ± 1.1 mg g⁻¹ in the center of gossan to 19.0 ± 4.0 mg g⁻¹ in the West; Mn concentrations ranged from 151 ± 71.5 µg g⁻¹ in the West to 736.4 ± 293.7 µg g⁻¹ in the center of gossan. Pb concentrations ranged from 9.3 ± 3.5 µg g⁻¹ in the West to 267.8 ± 287.3 µg g⁻¹ in the center of gossan; Zn concentrations ranged from 18.0 ± 11.9 to 435.6 ± 219.4 µg g⁻¹ in the center of gossan. In general, the elements tended to accumulate towards the East. This could be related to the wind direction because, in semi-arid areas, the wind is the main factor of erosion affecting the dispersion of these elements in the soil.

In Australia, Lottermoser et al. (2008) reported in soils near to the gossan, average pseudo total values of 79.9 mg g⁻¹ of Fe;

Table 3. Pseudo total and available concentration of Fe, Mn, Pb, and Zn in soils affected by the presence of gossan in Lapão-Irecê, Brazil

Position	Fe (mg g ⁻¹)	Mn	Pb	Zn
		Pseudo total (µg g ⁻¹)		
EPA 3050 A				
East	15.7±6.9 a	442.6±291.2 b	31.2±17.5 b	181.2±103.1 b
Center	19.7±4.3 a	736.4±293.7 a	267.8±287.3 a	435.6±219.4 a
West	7.8±1.3 b	151.0±71.5 c	9.3±3.5 c	18.0±11.9 c
p	< 0.001	< 0.001	< 0.001	< 0.001
Available concentration (µg g ⁻¹)				
Mehlich-1 extraction				
East	< 0.01	20.6±15.1 b	< 0.01	12.2±9.5 b
Center	0.1±0.2	33.7±16.6 a	1.4±2.9	29.0±21.6 a
West	3.6±11.1	43.3±33.7 a	0.2±0.8	5.8±7.3 b
p	0.198	0.034	0.094	< 0.001
DTPA extraction				
East	10.1±5.6 a	51.3±28.7 a	8.8±4.9 b	15.2±6.7 a
Center	12.5±5.7 a	30.3±14.2 a	36.6±47.4 a	15.7±8.6 a
West	6.5±5.1 b	23.2±11.4 b	5.6±2.4 b	4.8±2.4 b
p	0.011	0.001	< 0.001	< 0.001

¹Means followed by the same letter belong to the same group by the Scott-Knott test ($p \leq 0.05$); p-value of F test

3800 µg g⁻¹ of Mn; 12000 µg g⁻¹ of Pb; and 1100 µg g⁻¹ of Zn; and at 500 m distance from the gossan, 15.3 mg g⁻¹ of Fe; 2000 µg g⁻¹ of Mn; 120 µg g⁻¹ of Pb, and 34 µg g⁻¹ of Zn. Atapour & Aftabi (2007), in Iran, reported ranges of 1.7-120 µg g⁻¹ of Fe; 8-2050 µg g⁻¹ of Mn; 7-204 µg g⁻¹ of Pb, and 9-2993 µg g⁻¹ of Mn. In general, the results observed in soils near the gossan in Bahia are below the values reported by Lottermoser et al. (2008) and within the range presented by Atapour & Aftabi (2007), except for Pb.

Mean pseudo total concentration of Fe and Mn in soils is within the range that is considered normal for these elements (Atapour & Aftabi, 2007). In the present study, Zn values were observed above 60 µg g⁻¹, considered the quality reference for this element in Brazil (CETESB, 2014). Although Zn values were high, Pb was the element that exceeded the value of prevention (72 µg g⁻¹), and in some cases the value of investigation (150 µg g⁻¹) defined by CETESB (2014), indicating that further research and monitoring is necessary to avoid possible risks to human health and ecosystems due to the high concentration of Pb in the soil (Guerra et al., 2012).

Available concentrations were in general, higher by DTPA than Mehlich-1 except for Zn, where Mehlich-1 extractor presented significantly higher values than DTPA ($p \leq 0.05$) at the center of the gossan. Fe values ranged from < 0.01 to 3.6 ± 11.1 µg g⁻¹ in Mehlich-1 extraction and from 6.5 ± 5.1 to 12.5 ± 5.7 µg g⁻¹ in DTPA extraction. About the Mn values, they ranged from 20.6 ± 15.1 to 43.3 ± 33.7 µg g⁻¹ in Mehlich-1 extraction and from 23.2 ± 11.4 to 51.3 ± 28.7 µg g⁻¹ for DTPA extraction. Pb values ranged from < 0.01 to 1.4 ± 2.9 µg g⁻¹ for Mehlich-1 extraction and from 5.6 ± 2.4 to 36.6 ± 47.4 µg g⁻¹ for DTPA extraction; and Zn values ranged from 5.8 ± 7.3 to 29 ± 21.6 µg g⁻¹ for Mehlich-1 extraction and from 4.8 ± 2.4 to 15.2 ± 6.7 µg g⁻¹.

Available concentrations in the East and the center were, in general, similar ($p > 0.05$), even though their pseudo total concentration were significantly different ($p \leq 0.05$), indicating that at the East, the soil presents conditions that favor its availability concerning the center. This result may be related

to differences in soil characteristics such as soil pH, organic matter, and clay content (Alloway, 2013). In general, available values in the gossan center, considering DTPA extraction, accounted for 0.07% for Fe, 4.11% for Mn, 13.71% for Pb, and 3.6% for Zn, of the total values.

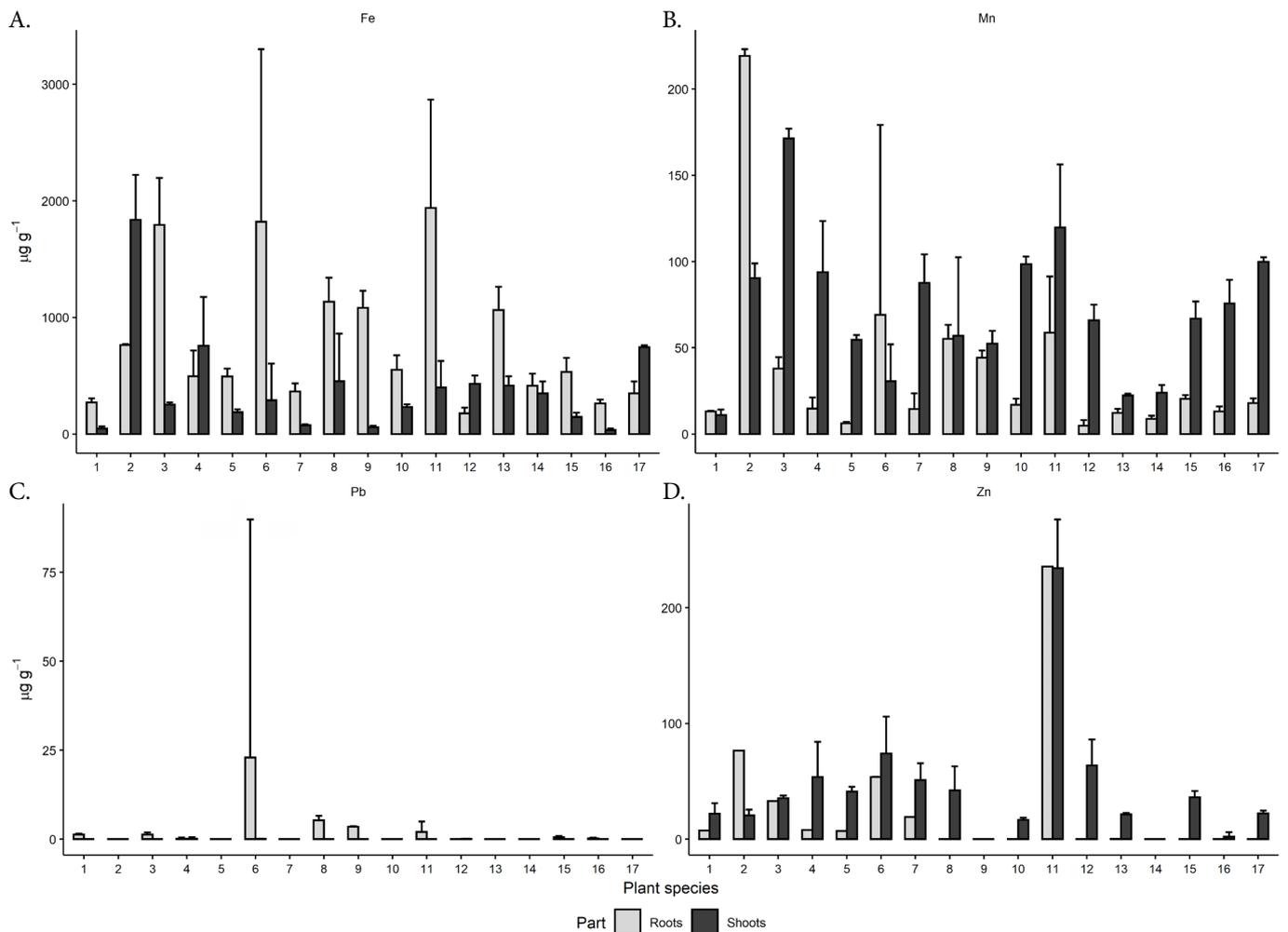
The results of the present study can be explained due to the nature of the studied soils that tend to alkalinity, soil for which DTPA was developed and affinity to bivalent cations, due to its high concentration of CaCl_2 in the solution (Hammer & Keller, 2002), especially Mn and Pb that are normally found in soils in the bivalent form. Zinc is highly adsorbed in the clay structures in comparison to the other elements studied (Alloway, 2013), which favors acid solutions such as Mehlich-1 in soils with a high content of clay, such as the ones studied in the present experiment, due to its higher capacity of dissolving structures that retain trace elements in soils (Abreu et al., 1995). Lottermoser et al. (2008) reported lower extraction concerning total values for all elements in comparison to the present experiment; and lower available values for Zn, similar values for Mn, and higher available values for Pb, in the three assessed areas even though the reported total values were higher.

Generally, the differences between extractors observed in the present study were in concordance with Feng et al. (2005), in which DTPA was the extractor with higher extraction

capacity. However, Mantovani et al. (2004) observed that Mehlich-1 extracted higher values of Mn, Pb, and Zn in comparison to DTPA in different soil types.

Fe, Mn, Pb, and Zn concentrations in roots and shoots are shown in Figure 1. Significant differences ($p \leq 0.05$) were observed between the species assessed for all elements studied. In general, the concentrations in shoot and roots were significantly different ($p \leq 0.05$), evidencing that elements were mostly accumulated in roots. In the shoot, Fe (Figure 1A) had a concentration ranging from $34.27 \pm 13.35 \mu\text{g g}^{-1}$ (umbu-cajá) to $1836.97 \pm 386.81 \mu\text{g g}^{-1}$ (banana); Mn (Figure 1B), from $10.98 \pm 3.17 \mu\text{g g}^{-1}$ (angico) to $171.43 \pm 5.59 \mu\text{g g}^{-1}$ (barbados nut); and Pb (Figure 1C), from < 0.001 to $0.16 \pm 0.4 \mu\text{g g}^{-1}$ (bean). In roots, Fe concentration ranged from $177.22 \pm 48.22 \mu\text{g g}^{-1}$ (mucambo) to $1820.99 \pm 1481.84 \mu\text{g g}^{-1}$ (buffelgrass); Mn from $4.83 \pm 3.25 \mu\text{g g}^{-1}$ (mucambo) to $219.33 \pm 3.87 \mu\text{g g}^{-1}$ (banana); Pb from < 0.001 to $22.96 \pm 66.88 \mu\text{g g}^{-1}$ (buffelgrass); and Zn (Figure 1D) from < 0.001 to $235.64 \pm 217.83 \mu\text{g g}^{-1}$ (maize).

Lottermoser et al. (2008) found a similar range of Fe concentrations compared to the present study in soils affected by Pb-Zn gossans. Generally, values above $500 \mu\text{g g}^{-1}$ are considered toxic; however, it is important to consider the concentration of other elements (Broadley et al., 2012). It was observed that banana (*Musa spp.*), bean (*Phaseolus*



1 - Angico; 2 - Banana; 3 - Barbados nut; 4 - Bean; 5 - Brazilian copal; 6 - Buffel Grass; 7 - Cactus pear; 8 - Castor bean; 9 - Jocote; 10 - Jurema; 11 - Maize; 12 - Mucambo; 13 - Peruvian pepper; 14 - Saguaro; 15 - Sugar apple; 16 - Umbu caja; 17 - Umburaninha

Figure 1. Concentration of Fe (A), Mn (B), Pb (C), and Zn (D) in roots and shoot of different plant species assessed under the influence of a gossan in Lapão-Irecê, Brazil

vulgaris), and umburaninha (*Bursera* sp.) had concentrations, in general, above this limit, which indicates that could exist problems in the development of these species due to high Fe concentrations.

In the shoot, Nguyen et al. (2011), in Vietnam, in one of the largest mines of Pb and Zn of the country, they found higher concentrations of Mn in shoot and roots. However, in shoots, Lottermoser et al. (2008), in Australia, reported values that were, in general, higher than the ones found in the present study. However, most of the values from the shoot affected by the gossan were observed in a lower range than the value considered to be toxic for maize, which is the species with the lowest critical limit ($200 \mu\text{g g}^{-1}$) as described by Broadley et al. (2012). This indicates no limitation due to excessive concentration of Mn in the shoot, although roots present high concentration values.

Values of Pb found in the present study, both in shoot and roots, were below the ones reported by Yang et al. (2014), in China, studying hyperaccumulator species. Similarly, Lottermoser et al. (2008) reported higher values than the present study. In general, Pb has low mobility in the soil and plant; therefore, it has low translocation to shoot (Li et al., 2007). Kabata-Pendias (2010) established ranges of trace elements tolerance for crops; the values observed are below the

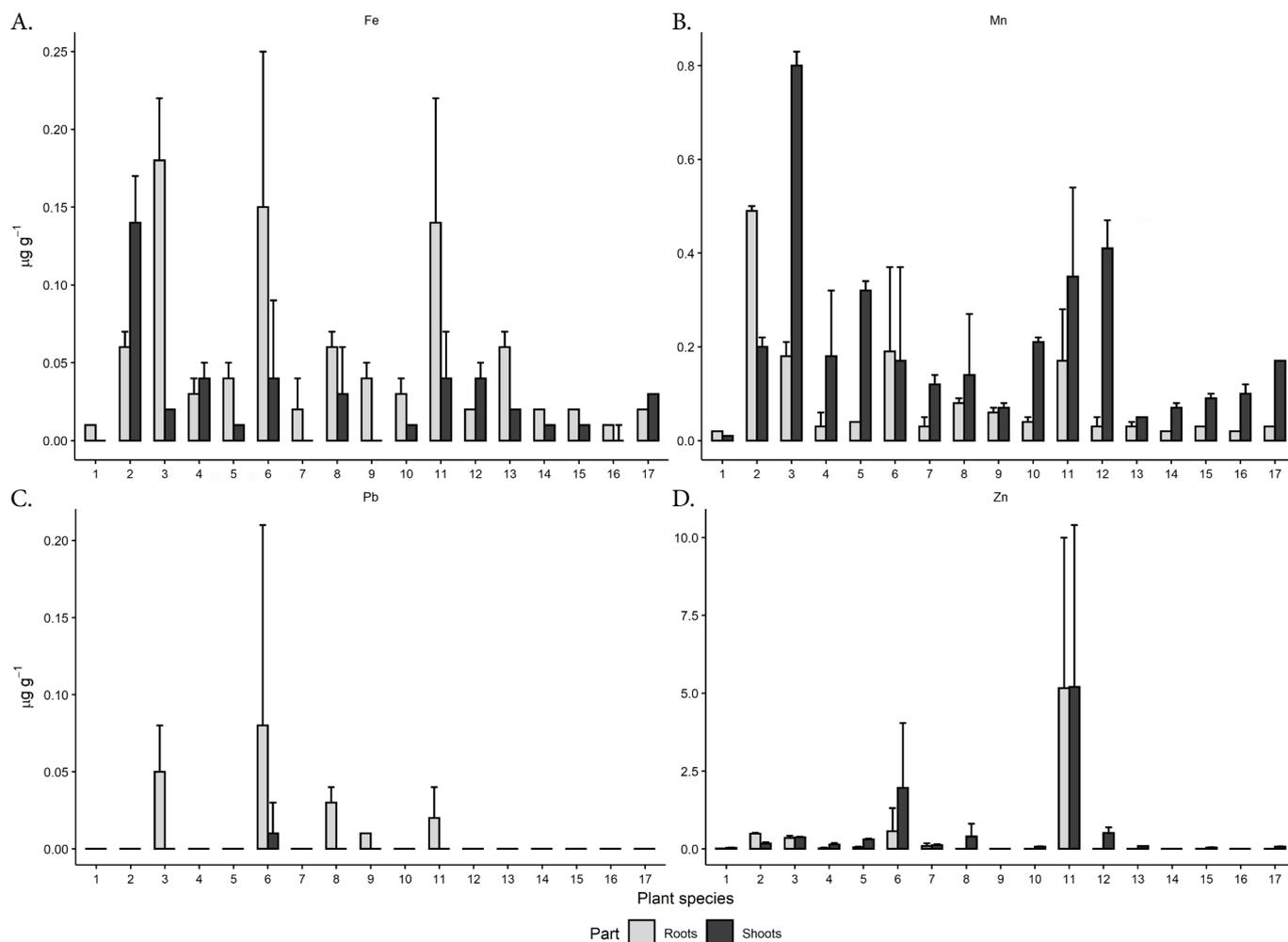
critical limit ($10 \mu\text{g g}^{-1}$), indicating no risk for Pb even though soil concentrations are high.

In general, Zn concentrations were less than the values reported by Zhuang et al. (2007) and Nguyen et al. (2011). However, Lottermoser et al. (2008), in Australia, reported higher mean values of Zn for the assessed species. In general, Zn has very low availability in soils with pH near alkalinity (Alloway, 2013). However, a high concentration of this metal was observed in shoot and roots, and in some cases exceeding the critical limit ($< 100 \mu\text{g g}^{-1}$) established by Kabata-Pendias (2010).

Values of Fe, Mn, Pb, and Zn bioconcentration factors in roots and shoot are shown in Figure 2. In shoot, Fe bioconcentration (Figure 2A), was observed from < 0.001 to 0.14 ± 0.03 in banana; Mn (Figure 2B), from 0.01 in angico to 0.41 ± 0.06 in mucambo; Pb (Figure 2C), from < 0.001 to 0.01 ± 0.02 ; Zn (Figure 2D) from < 0.001 to 5.2 ± 5.2 in maize.

Bioconcentration factor of Fe was observed in the “medium” range, according to Kabata-Pendias (2010), and with values above the range of 10^{-3} - 10^{-2} , considered as normal for most plant species (Kabata-Pendias, 2010).

In the present study, bioconcentration was below the ones reported by Meera & Agumuthu (2012). Iron is one of the most abundant elements in the earth and especially in soils



1 - Angico; 2 - Banana; 3 - Barbados nut; 4 - Bean; 5 - Brazilian copal; 6 - Buffel Grass; 7 - Cactus pear; 8 - Castor bean; 9 - Jocote; 10 - Jurema; 11 - Maize; 12 - Mucambo; 13 - Peruvian pepper; 14 - Saguaro; 15 - Sugar apple; 16 - Umbu caja; 17 - Umburaninha

Figure 2. Bioconcentration factor of Fe (A), Mn (B), Pb (C), and Zn (D) of roots and shoot in the plant species assessed under the influence of a gossan in Lapão-Irecê, Brazil

derived from gossans since it is defined as a large mass of residual iron oxyhydroxide material (Atapour & Aftabi, 2007). Bioconcentration factors for Mn in all plants were below 1, indicating a low Mn concentration capacity in plant tissues from the soil's available concentration. However, these values are in the range considered normal for Mn for most plants (0.01-1.0) and still inside the range considered as "medium" (Kabata-Pendias, 2010). These results are like those reported by Ondo et al. (2013) that observed plants with bioconcentration factors below 1. Bioconcentrations factor for Pb were observed in the range "medium" (0.1-1.0), low (0.01-0.1) and according to Kabata-Pendias (2010), were inside the range (0.1-1.0) considered as a medium for most plants; also, these results are similar to the values reported by Yang et al. (2014). In general, the bioconcentration factor for Zn was among the highest, being registered in the range considered as "high" (> 1.0); these values are higher than the ones observed by Yang et al. (2014). The bioconcentration factor is important in determining which plant species is more efficient in extracting trace elements and evaluating the possible effects of this accumulation in the food chain. Zn was the most accumulated element compared to the other elements studied, even though the sampled soils are alkaline, which favors conditions to low availability of trace elements. In general, Zn can only cause human health problems due to acute intoxication and interference with the uptake of copper; hence, many of its effects are associated with copper deficiency (Plum et al., 2010). Nevertheless, higher absorption of Zn can interfere with the crop yield due to inhibition of root growth and flower production (Kabata-Pendias, 2010), even with the advanced homeostatic mechanisms in plants for zinc tolerance (Alloway, 2013).

CONCLUSIONS

1. Plants showed different accumulation patterns, with a higher accumulation of Fe, Mn, and Zn in the shoot, while Pb was mainly accumulated in the roots.

2. Even though the concentrations in soil were high, plants showed, in general, low bioconcentration factor of the potentially toxic elements, except for Zn with the maximum value observed in maize.

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