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Natural concentrations and reference values of heavy metals in sedimentary soils in the Brazilian Amazon

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Abstract The soils of the Brazilian Amazon exhibit large geochemical diversity reflecting the different soil formation processes in an area covering 49% of the Brazilian territory. Soil contamination by heavy metals is one of the threats to the sustainability of this Biome but establishing quality reference values (QRVs) for the region is a challenging owing to the immense territorial area of the Amazon. This study aimed to determine the natural background of heavy metals in soils from the southwestern Brazilian Amazon in order to propose QRVs for Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sb, and Zn for alluvial sedimentary soils. One hundred and twenty-eight soil samples were collected at a depth of 0.0–0.2 m in sites with minimal anthropogenic interference. Soil sample digestion was based on the EPA 3051A method and metal concentrations were determined by ICP-OES. QRVs calculated for the southwestern Brazilian Amazon are among the lowest recorded in Brazil (mg kg^{-1}): Ba (16.5), Cd (0.1), Cr (6.9), Cu (2.8), Fe (15.4), Mn (13.4), Ni (1.7), Pb (4.4), Sb (0.9), and Zn (5.7). The low metal concentration is likely a result of the sedimentary origin of the soils. The results of this study can serve as a basis for defining public policies to investigate the environmental impacts resulting from changes in land use in areas of the Brazilian Amazon.

Keywords Amazon soils · Soil contamination · Trace elements · Soil quality

Introduction

The Brazilian Amazon encompasses more than 5 million km^2 and covers the northern and some of the northeastern and western parts of the country (IBGE 2017). Considered to be the most biodiverse biome, it has great socioenvironmental and economic importance. The Brazilian Amazon also hosts industrial, extractive, agricultural, and tourism activities (Hall and Harris 2013; Hoefle 2016). However, in recent decades, the intensification of anthropic activities, such as deforestation, artificial fires, agricultural activities, mining, and highway and hydroelectric dam construction have led to the

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deterioration of the Brazilian Amazon's natural resources (Brown et al. 2016; Faria and Almeida 2016; Mendes et al. 2017; Moura et al. 2013; Solar et al. 2016).

These anthropogenic activities favor the enrichment of heavy metals in soils. This potential contamination can adversely affect the health of fauna and flora, damaging the balance of the ecosystem (Pierzynski et al. 2015). Metal exposure can have deleterious effects on humans, such as cardiovascular problems (Myong et al. 2014), central nervous system disorders (Mason et al. 2014), and cancer (Zhang et al. 2014). Monitoring of contaminated or remediated areas requires knowledge of soil quality reference values (QRVs), which should reflect the natural levels of metals in soils (i.e., indicative of their inherent quality) (Preston et al. 2014). Knowing QRVs makes it possible to evaluate the level of contamination or its absence, which can stave off socioeconomic and environmental damage (Biondi et al. 2011).

In Brazil, it is difficult to establish QRVs due to territorial extension and geological and pedological diversity. Differences in the physicochemical attributes of soils and their geological contexts modify the dynamics of metals in the environment. The Amazon has several sedimentary deposits that are constantly receiving large influxes of fluvial materials (Silva et al. 2009; Villar et al. 2018). Alluvial sedimentary soils are formed by the deposition of sediments transported by rivers. The composition of these soils is influenced mainly by the origin of the transported material (Grygar and Popelka 2016; Ogg et al. 2017). These sediments can form soils with low ion retention capacities, favoring the leaching of contaminants into the water table (Gloaguen and Passe 2017).

Although several studies of heavy metals in soils have been carried out around the world, such studies are incipient in the Amazon region. The presence of these elements in soils with a low capacity to retain them increases the need to understand and monitor their concentrations. Only a set of studies, conducted in different pedological conditions of the immense Amazon region, can provide a suitable geochemical background for the establishment of QRVs. In this context, the objective of this work was to determine the natural contents of Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sb, and Zn to propose their QRVs in alluvial sedimentary soils of southwestern Brazilian Amazon.

Materials and methods

Study area and soil sampling

The study area (Fig. 1) is located in the southwestern region of the Amazon rainforest (7 ° 30' 24" S and 63° 04' 56" W). The climate, according to classification of Köppen, is type Am (tropical rainy). The average annual rainfall and temperature are 2500 mm and 26 °C, respectively, and the relative humidity is high and varies from 85 to 90% (Campos et al. 2014). The vegetation is mostly fields and forests (Campos et al. 2010). The predominant relief is of the "tray" type, with low gradients and slightly undulating edges. The geology is formed of ancient alluvium, originating from the Holocene, with pluvial-fluvial and lacustrine sediments (Braun and Ramos 1959; Campos et al. 2012).

Samples were collected at depths of 0–20 cm in areas with natural conditions or minimal anthropogenic interference. At each point, three simple samples were collected with the aid of a stainless-steel auger to form a composite. We collected, in all, 128 composite samples. Next, the samples were air-dried and passed through a stainless-steel sieve with a 2-mm aperture for chemical and physical characterization.

Chemical and physical characterization of soils

The granulometric fraction was analyzed using the Bouyoucos densimeter method. We also measured pH in water (1:2.5 *v*/*v*) and extracted P, Na^+, and K^+ with Mehlich-1 and determined their contents using photocolorimetry and flame photometry, respectively. Ca^{2+}, Mg^{2+}, and Al^{3+}were extracted by KCl 1 mol L^{-1} and determined by complexometry (Donagema et al. 2011). Soil organic matter (SOM) was estimated from the determination of the organic carbon content using the modified Walkley-Black method (Silva et al. 1999).

Extraction of the metals Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sb, and Zn was performed using the EPA method 3051A (USEPA, 1998). Substrates of the soils were sprayed in agate mortar and subsequently sieved through a 0.15-mm mesh stainless-steel screen. Next, 0.500 g of the samples was transferred to Teflon tubes, and 9 mL of HNO3 + 3 mL of HCl was added. The product was then digested in a microwave oven (Mars Xpress,) at 175 °C for 4 min and 30 s. The extracts were transferred to 25-mL certified flasks and filtered with a blue-band filter paper. Their

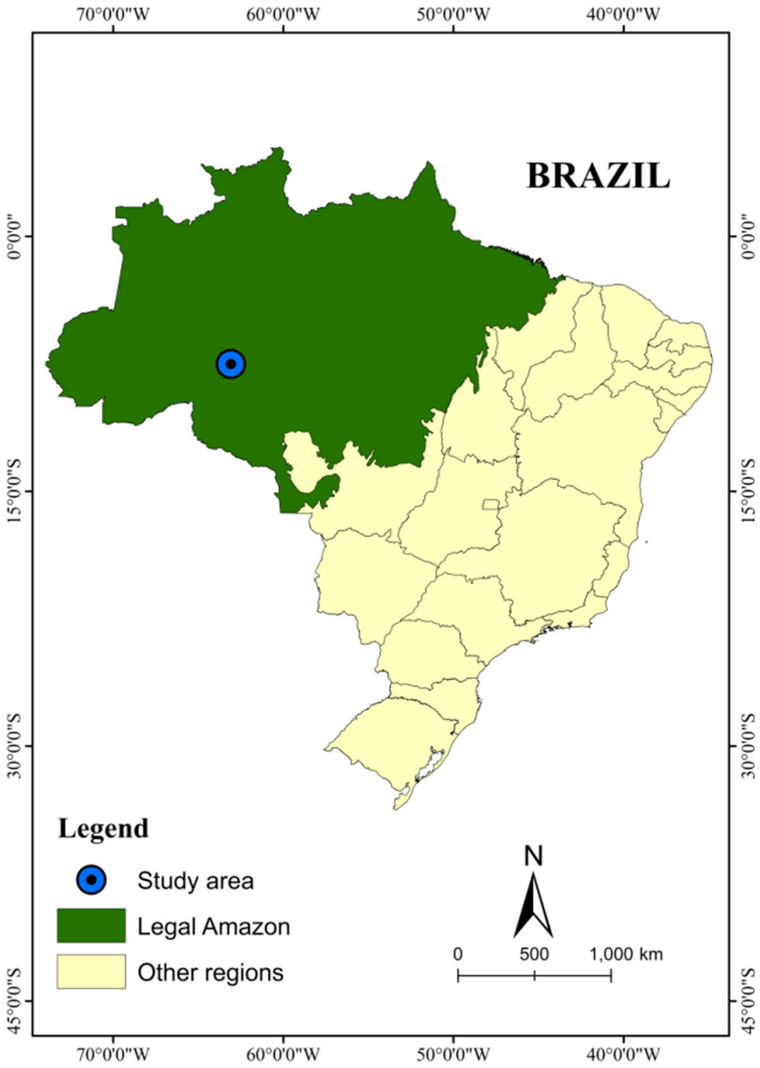

Fig. 1 Location of the study area in southwestern Brazilian Amazon

volume was completed using ultrapure water. The analyses were performed in duplicate. The quality standard of the samples was checked using blank and certified soil samples (SRM 2709 San Joaquin Soil). The concentrations of the elements were determined by inductively coupled plasma optical emission spectrometry (ICP-OES Perkin Elmer 7000 DV).

Statistical analyses

The data were analyzed using univariate statistical methods (mean, median, minimum, maximum, standard deviation, and box plots). The metal QRVs were determined based on the 75th and 90th percentile of the sample set, and anomalous values were eliminated based on the recommendation of Conama (2009). The statistical procedures were performed using STATISTICA v.10 software.

Results and discussion

Physical and chemical characterization of soil

The analyzed soils are acidic ($\mathrm{pH} \leq 5.5$) and present low natural fertility (Ca < 2.5 cmolc dm^{-3}, Mg < 4.5 cmolc dm^{-3}, $P < 12.5$ mg dm^{-3}; < 0.2 cmolc dm^{-3}; cation exchange capacity (CEC) below 6.5 cmolc dm^{-3}) (Table 1), corroborating the results by Delarmelinda et al. (2017). These authors attributed the low levels of basic cations to the poverty of the soil-forming sediments of the region and the high rainfall indices

Table 1 Descriptive statistics of chemical and physical attributes of soils samples

	pH	P mg dm^{-3}	Na cmol$_c$ dm^{-3}	K	Ca	Mg	Al	CEC$_{(t)}$[b]	SOC[c] g kg^{-1}	Clay	Silt	Sand
Mean	4.7	3.3	0.2	0.08	1.1	0.5	0.2	3.3	27.4	242.7	558.6	198.5
Median	4.7	3	0.2	0.1	1.1	0.3	0.2	2	27.3	239.6	558.2	199.5
Minimum	4.1	0.2	0.03	0.05	0.9	0.2	0	1.7	23.4	156.8	451.1	152.3
Maximum	5.5	12.5	0.3	0.1	2.3	4.3	0.6	6.2	32.3	354	667.6	236.6
SD[a]	0.2	1.3	0.04	0.02	0.2	0.7	0.08	0.7	1.9	30.8	33.1	20

[a] Standard deviation

[b] Cation exchange capacity

[c] Soil organic carbon

that contributed to the low CTC values, and favored leaching of bases.

It was verified that the average sand, silt, and clay contents were 24%, 56%, and 20%, respectively. The texture of the soil was classified according to the criteria of the FAO (2006) as silt loam, which is justified by the alluvial nature of the constituent sediments of the source material (Campos et al. 2010). Along with the low pH, the sandy nature of the studied soils may enhance the mobility and losses of heavy metals in the soil by leaching and/or surface runoff in an event of soil contamination. Birani et al. (2015) found highly significant correlations between PTE contents and clay contents in Oxisols of Amazon region, indicating an association of most metals with more clayey soils. It is likely that metals are bound mostly by covalence on kaolinite and iron and aluminum oxides (Araújo et al. 2002). The adsorption mechanisms in these clay minerals take place by high energy covalent binding (specific adsorption), a common reaction between soil oxides and metallic ions (Naidu et al. 1998).

Heavy metal recovery in the certified sample

Digestion by the 3051A method yields the environmentally available levels of heavy metals because this method does not use substances capable of destroying soil silicates. In this sense, the National

Table 2 Recovery of heavy metals in the reference soils (SRM 2709—San Joaquin) based on the USEPA method 3051A

Metal	Determined value mg kg^{-1}	Certified value (NIST)[a]	Determined recovery[b] %	Leaching recovery[c]	Leaching-based recovery[d]
Ba	558.53 ± 181.5	968 ± 40	58	51	114
Cd	0.52 ± 0.12	0.38 ± 0.01	137	ND	ND
Cr	69.33 ± 17.45	130 ± 4	53	54	99
Cu	26.12 ± 5.80	34 ± 0.7	77	82	94
Fe	19,271 ± 4449.5	35,000 ± 1100	55	60	91
Mn	392.11 ± 79.6	538 ± 17	73	74	98
Ni	57.15 ± 12.6	88 ± 5	65	67	97
Pb	9.68 ± 1.91	18.9 ± 0.5	51	54	94
Sb	2 ± 0.3	7.9 ± 0.6	25	ND	ND
Zn	72.02 ± 19.04	106 ± 3	68	75	90

[a] NIST: National Institute of Standards and Technology

[b] % Determined recovery = (determined value/certified value) × 100

[c] % Leaching recovery = (leaching median (NIST) / certified value) × 100

[d] % Leaching based recovery = (determined recovery /leaching recovery) × 100

ND not determined. Source: Preston et al. (2014)

Table 3 Descriptive statistics of heavy metal contents

Metals	Mean mg kg^{-1}	Median	Max	Min	das	CV^{b} %
Ba	19.3	16.6	293.2	1.4	26.1	135
Cd	0.1	0.1	0.2	0.1	0.1	31
Cr	8.7	7.4	32.5	4.4	6.4	73
Cu	3.3	3.0	8.2	1.3	1.3	40
Fe	15,462	15,392	23,660	8660	3839	25
Mn	15.1	13.7	79.1	8.7	7.4	49
Ni	2.5	1.7	13.7	0.8	2.9	116
Pb	4.2	4.5	9.1	2.1	1.2	28
Sb	1.0	0.8	2.4	0.1	0.6	64
Zn	6.3	5.8	18.5	0.8	2.2	35

[a] Standard deviation

[b] Coefficient of variation

Institute of Standards and Technology (NIST) recommends that this method be compared with recoveries based on leached values (Biondi et al. 2011). Recovery rates were considered to be satisfactory for all metals and ranged from 90 to 114% (Table 2). These results indicate the quality and reliability of the analyses.

Natural contents of heavy metals and QRVs

The mean natural contents of heavy metals were (mg kg^{-1}): Fe (15,462.0), Ba (19.3), Mn (15.1), Cr (8.7), Zn (6.3), Pb (4.5), Cu (3.3), Ni (2.5), Sb (1.0), and Cd (0.1) (Table 3). Natural contents of these metals in the Amazon sedimentary soils were lower than other Brazilian regions (Paye et al. 2010; Biondi et al. 2011; Preston et al. 2014; Almeida et al. 2016). The sediments carried by the Madeira River and deposited during its course in sedimentary basins are primarily responsible for the low natural contents found because the source of the Madeira River is in the Andes region; it carries materials low in metals that are mainly composed of kaolinite clay, chlorite, illite, and smectite minerals (Guyot et al. 2007; Martinelli et al. 1993). Queiroz et al. (2011) and Horbe et al. (2013) verified that the sediments of the Madeira River are more homogeneous and impoverished than those of other rivers in the Amazon basin, which indicates that sediments with different compositions can form soils with different concentrations of heavy metals, thereby confirming the determinant role of the parent material in the distribution of metals in soils.

Gloaguen and Passe (2017) assessed the heavy metal contents in the soil samples from the sedimentary basins of the Reconcavo Baiano and Tucano and presented contents of Pb (4.1 mg kg^{-1}) and Ni (2.6 mg kg^{-1}) similar to those in this study. Similar concentrations for Fe (15.1 g kg^{-1}) and concentrations five times lower for Mn (2.59 mg kg^{-1}) were also observed by Costa et al. (2017) in soils formed by alluvial deposits. In contrast, soils from Neogene sediments (Barreiras formation) yielded higher values for Cr, Zn, and Ni: 26.8 mg kg^{-1}, 7.7 mg kg^{-1}, and 6.6 mg kg^{-1}, respectively (Fadigas et al. 2010).

The levels of Cd in soils of the southwest of the Amazon varied between 0.1 and 0.2 mg kg^{-1}. They are considered to be low, which was expected due to the rare natural occurrence of Cd (0.01–2.00 mg kg^{-1}) (Alloway, 1990). Similar values were observed in soils formed from sedimentary deposits in the southern state of Amazonas (0.3 mg kg^{-1}) (Costa et al. 2017) and in the states of Pará and Amapá (0.3 mg kg^{-1}) (Fadigas et al. 2010). Cd is a well-studied metal due to its potential toxicological hazard (ATSDR 2017). It exhibits increased concentrations in soils generally associated with enrichment by anthropic sources, such as the use of phosphate fertilizers and industrial emissions. For instance, the Cd concentrations were clearly associated with anthropogenic sources as a consequence of P fertilization in sugarcane fields. All the soil samples analyzed presented Cd concentrations above the soil quality standard; 6 out of 60 sugarcane fields posing an unacceptable risk to human health since their Cd concentrations are above 3.0 mg kg^{-1} (Silva et al. 2016).

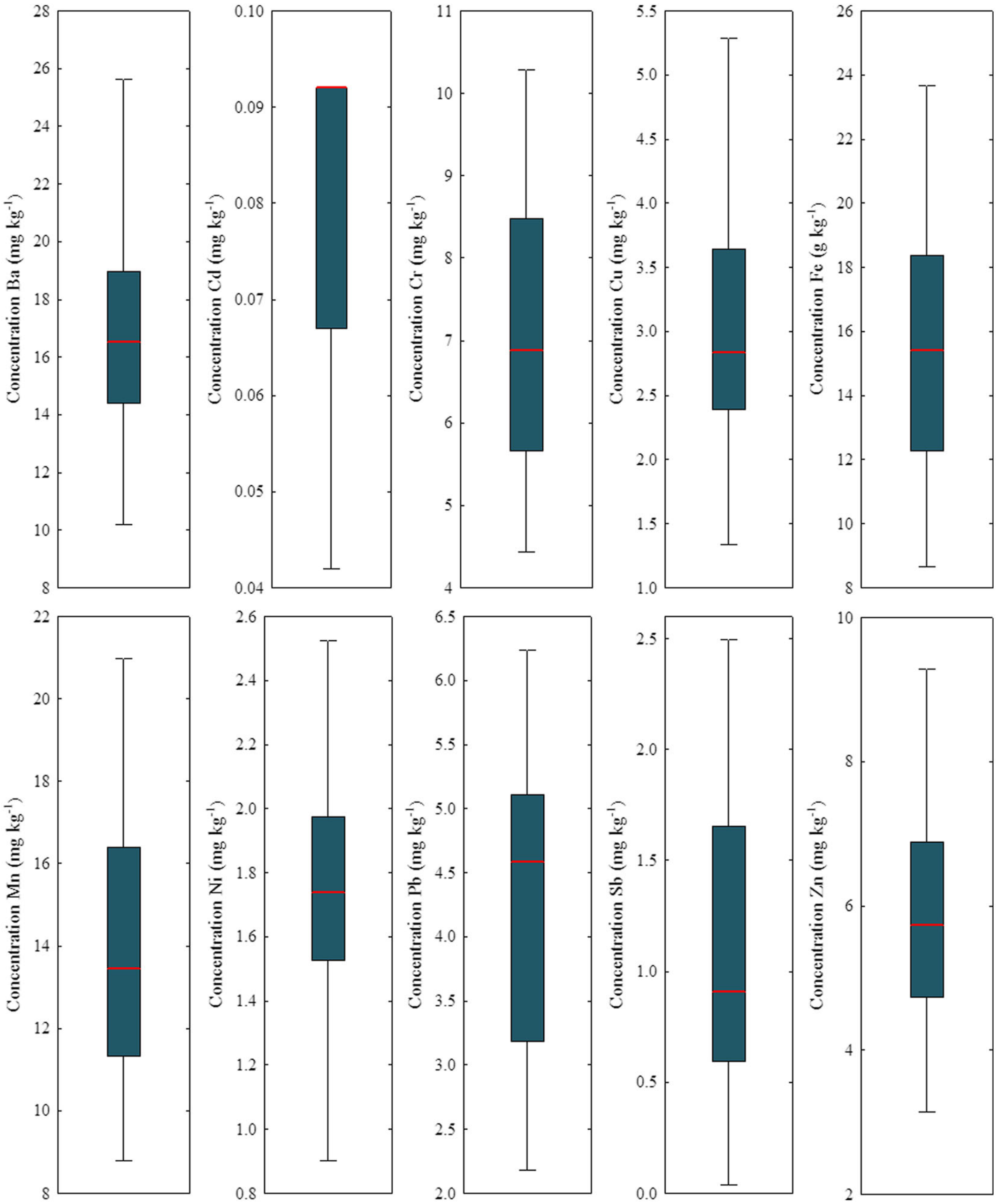

Fig. 2 Box plots for Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sb, and Zn after removal of the anomalous values. The horizontal lines on the inside of the boxes represent the QRVs of the corresponding metals

The Brazilian legislation, through Conama Resolution 420/2009, recommends that QRVs be established based on the 75th or 90th percentiles of the sample set after excluding anomalous values. Box plots were used to eliminate the anomalous values (Fig. 2). Cr, Cu, Ni, and Zn exhibited the greatest heterogeneity considering the number of outliers. The 75th and 90th percentiles presented small variations not exceeding 19% (Table 4); therefore, QRV for the soils of southwestern Amazon can be established using either 75th or 90th percentiles. The QRVs established for other states in Brazil were established using the 75th percentile, with the exception of Paraíba, which used the 90th percentile.

Table 4 QRVs for heavy metals in alluvial sedimentary soils from the southwestern Brazilian Amazon compared to values obtained by other Brazilian states

Metal	*n* (1)	*n* (2)	P75 (3)	P90 (4)	SP[a]	MG[b]	MT/RO[c]	RN[d]	PB[e]	PE[f]	ES[g]
Ba (mg kg^{-1})	127	1	16.5	16.9	75	171.4	–	58.9	87.9	84	–
Cd (mg kg^{-1})	126	2	0.1	0.1	< 0.5	1.0	< 0.3	0.1	0.06	0.5	0.13
Cr (mg kg^{-1})	120	8	6.9	7.1	40	86.6	44.8	30.9	11.2	35	54.1
Cu (mg kg^{-1})	123	5	2.8	3.4	35	12.2	20.6	13.7	28.8	5	5.9
Fe (g kg^{-1})	128	0	15.4	15.4	–	83.6	–	–	18.7	–	–
Mn (mg kg^{-1})	124	4	13.4	14.4	–	446.9	–	–	350.8	–	137.8
Ni (mg kg^{-1})	118	10	1.7	1.8	13	23	2.1	19.8	9.1	9	9.2
Pb (mg kg^{-1})	127	1	4.4	4.3	17	15.8	9.0	16.2	10	13	4.5
Sb (mg kg^{-1})	128	0	0.9	1.1	< 0.5	< 0.5	–	0.2	0.4	0.2	–
Zn (mg kg^{-1})	123	5	5.7	6.2	60	60	3.0	23.8	23.4	35	29.8

[a] State of São Paulo—Cetesb (2005)

[b] State of Minas Gerais—COPAM (2011)

[c] State of Mato Grosso e Rondônia—Santos and Alleoni (2013)

[d] State of Rio Grande do Norte—Preston et al. (2014)

[e] State of Paraíba—Almeida et al. (2016)

[f] State of Pernambuco—CPRH (2014)

[g] State of Espírito Santo—Paye et al. (2010)

n (1) total samples used to obtain the QRVs; *n* (2) number of anomalous values excluded through box plot analysis; P75 (3) 75th percentile; P90 (4) 90th percentile

The QRVs proposed for Ba, Cu, Cr, Fe, Ni, and Pb were lower than those established for other Brazilian states (Table 4). The value for Zn (6.2 mg kg^{-1}) was only larger than the Zn contents measured in the states of Mato Grosso and Rondonia (3.0 mg kg^{-1}). The QRV for Sb (1.1 mg kg^{-1}) was four times smaller than that determined for the Fernando de Noronha archipelago (4.6 mg kg^{-1}) and five times greater than the values determined for Pernambuco state and Rio Grande do Norte state (0.2 mg kg^{-1}). The value of Cd (0.1 mg kg^{-1}) was similar to that measured in Rio Grande do Norte (0.1 mg kg^{-1}) and ten times lower than measured in Minas Gerais (1.0 mg kg^{-1}). In the soils of southern Amazonia, Silva et al. (2017) proposed QRVs for Cr (8.9 mg kg^{-1}), Cu (3.9 mg kg^{-1}), and Ni (2.6 mg kg^{-1}) that were similar to those in this study: 7.1 mg kg^{-1}, 3.4 mg kg^{-1}, 1.8 mg kg^{-1}, respectively. On the other hand, the QRV for Pb in our soils (4.3 mg kg^{-1}) was only half of the amount determined by Silva et al. (2017), i.e., 7.0 mg kg^{-1}.

In the present study, it was possible to observe low levels of Fe (0.3 g kg^{-1}) and Mn (6.3 mg kg^{-1}), Ba (1.9 mg kg^{-1}), Zn (1.0 mg kg^{-1}), Cu (0.8 mg kg^{-1}), Ni (0.6 mg kg^{-1}), Cr (0.5 mg kg^{-1}), Pb (0.5 mg kg^{-1}), and Cd (0.04 mg kg^{-1}). These low levels also reflect the depleted composition of the sediments and the method of extraction used. On the other hand, soils from mafic and ultramafic rocks present QRVs ranging from 6 to 145 times higher than those of the present study (Alfaro et al. 2015). However, Fe and Mn are not among the metals that may present risks to the ecosystems in general because they are naturally abundant and their variation is mainly due to their oxireduction characteristics and origin material (Burt et al. 2003).

The heterogeneity of QRVs between Brazilian states and other places around the world is due to the geological diversity of Brazil and the occurrence of different pedogenetic processes in its soils. For alluvial sedimentary soils, the hydrography of the area is determined by the soil's metal content because sediment deposition is controlled by the velocity and flow of rivers and the composition of these materials influences the specific levels of heavy metals for each soil (Yao and Liu 2018). Therefore, the data presented here reinforce Conama's request for the establishment of QRVs in a stratified manner, which allows for a closer approximation of reality for each area. This importance is even clearer for areas of great territorial extension, as is the case for the Brazilian Amazon.

Conclusions

The soils of the Brazilian Amazon exhibit large geochemical diversity, reflecting the pronounced territorial area, and the different factors of soil formation and source materials. In this context, the present work seeks to contribute data of sedimentary soils of this region that can help guide environmental policies in the investigation of supposedly contaminated areas in this important region of Brazil. Our results reveal that the natural levels of heavy metals in sedimentary soils in Amazonia followed this order, with the most abundant metal listed first: Fe > Ba> Mn > Cr > Zn > Pb > Cu > Ni > Sb > Cd. The lower values recovered in this study are likely due to the significant weathering of the soils and the poverty of the materials of origin. As a consequence, the QRVs calculated for the southwestern Brazilian Amazon are among the lowest recorded in Brazil and presented the following values (mg kg^{-1}): Ba (16.5), Cd (0.1), Cr (6.9), Cu (2.8), Fe (15.4), Mn (13.4), Ni (1.7), Pb (4.4), Sb (0.9), and Zn (5.7). The results of this study can serve as a basis for defining public policies to investigate the environmental impacts resulting from changes in land use in areas of the Brazilian Amazon.

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