 Original Article

Analysis and assessment of heavy metals in urban surface soils of Teresina, Piauí State, Brazil: a study based on multivariate analysis

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Abstract
The concentration of Cu, Pb, Zn, Cr, Ni and Mn in the surface soil samples from Teresina city were measured by flame atomic absorption spectrometry (FAAS). A total of 28 composite soil samples were collected at depths 0-10 cm, including residential districts, urban parks, green areas, vegetable-garden, heavy- and low-traffic density areas. Principal component analysis (PCA) and hierarchical cluster analysis (HCA) were used to analyze the data and identify possible sources of these heavy metals. The results of the multivariate analysis showed that Cu, Pb, Zn and Cr were associated mainly to anthropogenic activities, such as, fertilizer and vehicle emission. While Ni and Mn were controlled by original materials and therefore interpreted as natural sources.

Keywords: urban soil; metal; multivariate analysis

Análise e avaliação dos metais pesados em solos superficiais urbanos de Teresina, Estado do Piauí, Brasil: um estudo baseado em análise multivariada

Resumo
A concentração de Cu, Pb, Zn, Cr, Ni and Mn nas amostras de solo superficial da cidade de Teresina foram medidas por espectroscopia de absorção atômica em chama (FAAS). Um total de 28 amostras compostas de solo foram coletadas numa profundidade de 0–10 cm, incluindo distritos residenciais, parques urbanos, áreas verdes, hortas, áreas de densidade de tráfico baixo e alto. Análise por componentes principais (PCA) e análise por agrupamento hierárquico (HCA) foram usadas para analisar os dados e identificar as possíveis fontes desses metais pesados. Os resultados da análise multivariada mostraram que Cu, Pb, Zn e Cr foram associados, principalmente, às atividades antropogênicas, tais como, fertilizantes e emissões veiculares. Enquanto Ni e Mn foram controlados por material parente, portanto interpretado como fonte natural.

Palavras chave: solo urbano, metal, análise multivariada

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Introduction

Urban soils are known to have peculiar characteristics such as unpredictable layering, poor structure, and high concentrations of trace elements (Kabata-Pendias & Pendias, 1984). They are the ‘recipients’ for large amounts of heavy metals from a variety of sources including industrial wastes, vehicle emissions, coal burning waste, and many others. In areas where public gardens and parks are exposed to significant pollution levels, dust from the ground may have toxic effects when an inhalation or an ingestion occurs among humans, particularly if they are children, who pose major health hazards (Manta et al., 2002; Sanchez-Camazano et al., 1994; Tüzen, 2003).

Several researchs have indicated the need of a better understanding of urban soils pollution (Li et al., 2001; Lu et al., 2003; Madrid et al., 2002; Manta et al., 2002; Wilcke et al., 1998), and, most of this research has been concentrated on the distribution of heavy metal in urban soils (Chen et al., 1997) and on their chemical forms (Ge et al., 2000; Li et al., 2001), distribution and movement in the soil profile (Yang et al.,2006).

There are two main sources of heavy metals in the soil: (i) natural background, which represents that the heavy metal concentration is derived from parent rocks; (ii) anthropogenic contamination, including application of agrochemicals, addition of organic amendments, animal manure, mineral fertilizer and sewage sludge. Generally, there are more heavier metals in soils which were originated from anthropogenic sources than the ones derived from natural sources (Nriagu & Pacyna, 1988).

Heavy metals are considered to be one of the main sources of the environmental pollution, since they have a significant effect on soil ecological quality. Some of them are toxic even if their concentration is very low and their toxicity increases with the accumulation of water in the soils. (Bradl, 2004; Li et al., 2001; Sastre et al., 2002). Cu, Pb, and Zn are common anthropogenic elements in urban environment (Alexandrovskaya & Alexandrovskiy, 2000; Yang et al., 2006).

Anthropogenic source not only leads to increasing levels of heavy metals owing to mining, industrial and energy production, agricultural, construction, vehicle exhaust, waste disposal, as well as coal fossil fuel combustion (Li et al., 2001; Wong & Mak, 1997), but also, as time goes by, it can lead to anomalous enrichment, causing metal contamination of the surface soils. The prolonged presence of the contaminants in the urban environment, particularly in soils, and their close proximity to the human population can significantly amplify the exposure of the urban population to metals via inhalation, ingestion and dermal contact (Mielke & Reagan, 1998; Mielke et al., 1999).

Multivariate statistical techniques, such as principal component analysis (PCA) and hierarchical cluster analysis (HCA), are powerful tools to segregate sources contributing to observed pollution. These techniques have been used both to differentiate among various natural sources that cause modifications in soil composition (Atgın et al., 2000; Carrasco et al., 1991; Chen et al., 1991; Güvenç et al., 2003) and to identify pollution sources affecting the heavy metal content in the soil (Davies, 1997; Dudka, 1992; Sweet et al., 1993; Xhoffer et al., 1991).

The aims of the current study were: (1) to determine the concentration of heavy metals in urban soil from vegetable-garden, green areas, urban parks, heavy- and low-traffic density areas in urban soils; (2) to assess possible sources of heavy metal contamination by multivariate statistical.

Material and Methods

The study area

Teresina is located in the centre-north of Piauí state, Brazil (05° 05´ S, 42° 48´W). It occupies a surface of 1679.8 km^2 and has approximately 0.8 million inhabitants. The average annual temperature is 26,3 °C and an average annual rainfall is 1478 mm. The Figure 1 shows the sampling locations in Teresina.

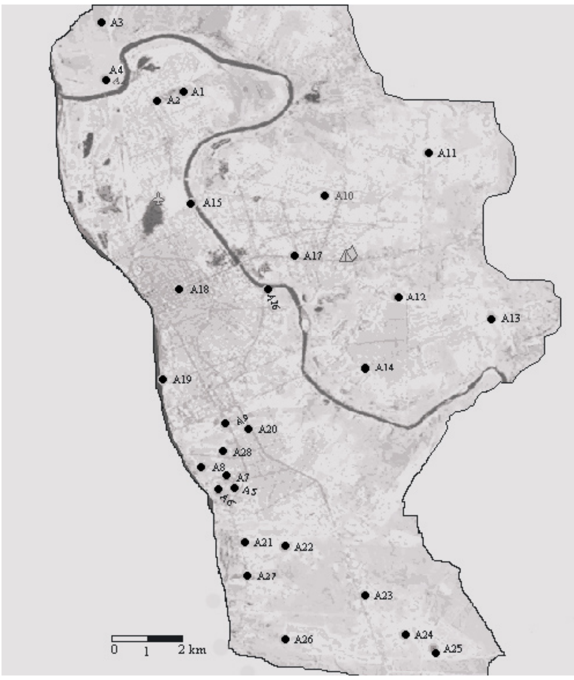

Figure 1. Location of the study area in Teresina County, Piaui State, Brazil.

Sampling

A total of 28 urban soils samples (0–10 cm depth) were collected in different areas including urban parks, residential districts, vegetable-gardens, heavy- and low-traffic density areas in Teresina (Figure 1).

At every sampling site, five sub-samples were collected (10 x 10 cm surface), which were mixed and homogenized to obtain a composite sample. The collected soil samples were stored in polyethylene bags, in room temperature.

Reagents and analytical

All acids (HNO_3 and HCl) used in this work were purchased from Merck and suprapur quality. Only deionized water (Milli-Q Millipore 18.2 MΩ.cm resistivity) was used to prepare all solutions. All the polyethylene bottles and glassware were cleaned by soaking in dilute HNO_3 10% to remove any adhered impurities and rinsed thoroughly with deionized water before use.

As for the elements (Cu, Pb, Zn, Cr and Ni), five standard solutions of different concentrations were prepared by dilution in 1 M HNO_3 of single-element standard solution of 1000 mg.L^{-1} (Cu, Pb, Zn, Cr and Ni), supplied by Fluka and ampoule of 1000 mg (Mn) supplied by Merck. Within the linear concentration range for measured. The calibration curve was prepared for each of the metals investigated by least square fitting.

Chemical analysis

Soil samples were air dried, sieved through a 2-mm plastic sieve and stored in polypropylene bags. Three replicate of each soil sample were analyzed to soil pH, CEC and TOM, according with Raij et al., (2001).

For soil digestion, 1.5 grams of the sieved sample were accurately weighed and have been transferred to 50 mL volumetric flasks. Firstly, the pre-digestion step was done at room temperature for 16 h with 2 mL of HNO_3 and 6 mL of HCl. Then, the suspension was digested at approximately 100 °C for 4 h, in a reflux system. The solution was cooled, filtered and diluted to 25 mL with 1% nitric acid solution and stored in polyethylene bottles at 4 °C until analysis. Heavy metals contents in the final solutions were determined for Cu, Pb, Zn, Cr, Ni and Mn by flame (air-acetylene) atomic absorption spectrometry (Varian, model SpectrAA 220 FS).

For quality control, blank samples (Two blank for every 6 samples) and triplicated samples were routinely prepared and incorporated in the analysis to monitor the possibility of contamination during digestion and to provide quality control. The analytical precision, measured as relative standard deviation, was routinely 3-5%.

Operational parameters of FAAS are shown in Table 1.

Table 1. Operational parameters of FAAS (flame atomic absorption spectrometry).

Element	λ (nm)^a	F (nm)^b	C (mA)^c	LD^d (mg L^{-1})
Cu	324,7	0,5	4,0	0,014
Pb	217,0	1,0	5,0	0,027
Zn	213,9	1,0	5,0	0,001
Cr	357,9	0,2	7,0	0,019
Ni	232,0	0,2	4,0	0,016
Mn	403,1	0,2	5,0	0,057

a: wave length, b: crack opening, c: current of the lamp, d: detection limit

Statistical analysis

Descriptive statistics including mean, minimum, maximum and standard deviation were calculated for some soil properties and heavy metals contents in the analyzed samples.

To identify the relationship among heavy metal in urban soil and their possible sources, multivariate statistical analyses, such as principal component analysis and hierarchical cluster analysis, were performed using statistical software package SPSS®. In the PCA, varimax with Kaiser normalization was used as the rotation method in the analysis. Since the elemental concentrations varied greatly among the major and trace elements, the raw data were standardized before the execution of clustering in HCA. The data were standardized to the Z score (with a mean of 0 and a standard variation of 1). HCA was formulated according to the Ward-algorithmic method. Results were shown in a dendrogram where procedures in the hierarchical clustering solution and values of the distances between clusters (squared Euclidean distance) are represented (Lin et al., 2002).

Results and discussion

Descriptive statistics

Descriptive statistics for some physicochemical parameters and heavy metal contents of the analyzed soils are summarized in table 2. The application of the K-S test confirmed that most variables are normally distributed with the exception of Pb and Zn.

Table 2. Descriptive statistics of some properties and heavy metal contents in soil of Teresina County, Piaui State, Brazil.

Variable	symbol	Unit	Mean	SD^a	Min.	Max.	K-S test^b
pH in water	pH	--	6.4	1.0	4.4	8.9	0.99
Cation exchange capacity	CEC	mmol$_c$.dm^{-3}	71.0	19.6	33.0	109.4	0.80
Organic matter	TOM	g.dm^{-3}	18.8	12.2	4.1	55.8	0.22
Copper	Cu	mg kg^{-1}	5.4	3.8	1.3	17.1	0.13
Lead	Pb	mg kg^{-1}	7.7	7.9	1.5	36.4	0.03
Zinc	Zn	mg kg^{-1}	29.8	31.2	4.7	132.3	0.04
Chromium	Cr	mg kg^{-1}	7.8	3.2	3.0	18.3	0.89
Nickel	Ni	mg kg^{-1}	1.3	1.4	ND	6.1	0.39
Manganese	Mn	mg kg^{-1}	56.9	37.6	14.8	161.3	0.35

a: Standard deviation, b: Kolmogorov-Smirnov Test.

The pH values ranges in narrow intervals (4.4 to 8.9), what suggests acid to moderately alkaline conditions for all the soil samples. CEC showed a very broad interval of variation from 33.0 to 109.4 mmol$_c$.dm^{-3}, with mean value of 71.0 mmol$_c$.dm^{-3} organic matter contents of the analyzed soil ranged from 4.1 to 55.8 g.dm^{-3} with an average of 18.8 g.dm^{-3}. However, the levels of organic matter seem to suggest an important retention of heavy metal by this component. However, the distribution pattern of organic matter reflects the variable distribution of plant, grass and vegetation which cover the soil in the investigated area. According to these results, the bioavailability of trace elements would be expected to be higher as for the analyzed soils, although anthropogenic sources and some properties such as pH could facilitate the mobility of some trace elements.

Heavy metals contents

The mean value of heavy metals contents (Table 2) measured in the urban soils, in Teresina, followed a descending order: Mn>Zn>Cr>Pb>Cu>Ni. In a general form, the concentrations of heavy metals presented wide ranges, which are typical of urban soils (Li et al., 2001; Lu et al., 2003; Manta et al., 2002).

The metal concentrations in the studying area soils were, in general, low. These metals originated from anthropogenic activities are distributed in soil by the atmosphere within a distance that depends on the size of particles. The concentration of these metals in soil can vary considerably according to the strength and direction of the wind, soil type, composition and cation exchange capacity and pH. Usually pH influences the CEC of soil, which, consequently, affects the heavy metal mobility and distribution in soil.

The data from this study demonstrated low contents of Cu, Pb, Zn, Cr, Ni and Mn in urban soils of Teresina whether compared to other study (Imperato et al., 2003), specially held in urban soils. Considering the sampling locations, the highest levels of heavy metals were found in vegetable-gardens and elevated traffic density areas. Although, there are few companies in Teresina, the disturbance of soil chemistry could also be attributed to local anthropogenic activities.

The average concentrations of metals in Teresina are lower than the ones from other cities around the world, varying according to soil type and also to the degree of industrialization (Imperato et al., 2003, Li et al., 2001). Therefore, levels of heavy metal analyzed corresponded to background populations, whereas Cu, Pb and Zn presented moderately polluted samples. In vegetable-gardens soil the most important activity, which produces the highest concentrations of Cu in agricultural soils, is the use of commercial fertilizers and fungicidal sprays, which usually may contain a wide variety of heavy metals as impurities (Gimeno-García et al.,1996).

The highest copper concentration (17.1 mg kg^{-1}) were observed in vegetable-garden soil that received commercial fertilizers, pesticides and other scattered diffuse pollution sources such as traffic emissions and incineration. The results of Cu in Teresina soil samples did not reach dangerous levels of contamination, because the maximum amount was 17.13 mg kg^{-1} in a vegetable-garden, i.e., below the value reported by Kabata-Pendias & Pendias (1984).

Lead concentrations were in the range of (1.5–36.4 mg kg^{-1}) with an average of (7.7 mg kg^{-1}). The highest lead concentration was found in dense traffic areas, while the lowest (1.5 mg kg^{-1}) was observed in an urban park. This situation proves that anthropogenic activities are the main source of metals in soil. In the present study, the contribution of anthropogenic sources in the elevation of soil metal content is evident, because the highest concentrations were observed in neighbor dense traffic areas instead of urban parks.

The average zinc concentration was 29.8 mg kg^{-1} with range of 4.7-132.3 mg kg^{-1}. The high levels of zinc in the soil are associated mainly to the use of mineral and cattle slurry fertilizers and traffic emissions in dense traffic regions.

The soil chromium concentration varied from 3.0 to 18.3 mg kg^{-1} with a mean value of 7.8 mg kg^{-1}. Results presented by Lin et al. (2002) showed an average concentration of chromium of 4.9 mg kg^{-1}, while Loska et al. (2004) found 4 mg kg^{-1} in an uncontaminated soil from Poland. The soils from Vietnam presented higher levels of Cr with range of 80.8-116.7 mg kg^{-1} in non-urban soil and 23.2-174.5 mg kg^{-1} in urban soil (Thuy et al., 2000).

The average content of nickel was 1.3 mg kg^{-1}, with a maximum of 6.1 mg kg^{-1}, but in 25% of the samples it was not possible to determine its presence, because the concentration was below the detection limit of the technique used through this study. Similar results were observed for non-contaminated soils in Poland (Loska et al., 2004). In soils, Ni is observed during the organic phase, which increases their mobility and bioavailability under acidic conditions.

The manganese concentration ranged from 14.8 to 161.3 mg kg^{-1}, with an average of 56.9 mg kg^{-1}. The manganese is one of the most abundant trace elements in the lithosphere and is commonly found in rocks in a concentration varying from 350 to 2000 mg kg^{-1} depending on soil type. In a global scale, the Mn content is often distributed in approximately 200 to 800 mg kg^{-1} with an average of 545 mg kg^{-1} (Kabata-Pendias & Pendias, 1984).

Multivariate statistical analysis results

Principal component analysis

The results of PCA for heavy metal contents in soil of Teresina are shown in Table 3. The factor loadings or component loadings are the correlation coefficients between variables and factors. The squared factor loading is the percent of variance in that a variable can be explained by a factor. According to these results, the eigenvalues of the two extracted components are greater than the ones before and after the matrix rotation. As a consequence, heavy metals could be grouped into a two-component model that accounts for 70.7% of all the data variation. In the rotated component matrix, the first PC (PC1, variance of 45.8 %) included Cu, Pb, Zn and Cr, while the second PC (PC2, variance of 24.9%) was constituted by Ni and Mn. Spatial representation of the two rotated components is shown in Figure 2.

PC1, including Cu, Pb, Zn and Cr, can be defined as an anthropogenic component due to its high-variability observed in the present study. In the sampling sites, higher content of Cu might have come from the application of Cu-contained agrochemicals related to specific agronomic practices, whereas vehicle fume in dense traffic areas can also be the main source of Pb contents found in some soils. However, Zn and its compounds are also used in different manufactured goods (e.g., paints, cosmetics, automobile tires, batteries and electrical apparatus) and in mineral and

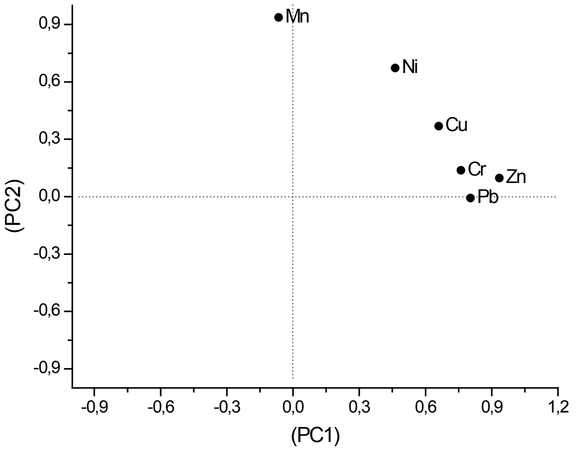

Figure 2. Principal component analysis loading plots for the rotated components

cattle slurry fertilizers, however higher content of Zn were found in dense traffic areas.

PC2 could be considered as a natural component, because the variability of the heavy metal seems to be controlled by parent rocks, moreover, Ni contents were lower than the other elements. This result suggested that the distribution of Mn and certain amounts of Ni had lithogenic control, and these two heavy metals were included in the second principal component.

Similarly, many authors have investigated the possible heavy metal sources in different countries. Micó et al. (2006) reported that Cu and Pb constituted an anthropogenic component, whereas the remaining elements (Mn and Ni) appeared to be associated with parent rock. Facchinelli et al. (2001) also drew conclusions about two groups of heavy metals where Cu and Zn were associated with specific agronomic practices, Pb was derived from car exhausts and all three metals were related to anthropogenic activities. On the other hand, Ni was controlled by parent rocks (Mn was not analyzed by these authors).

Considering this, it seems reasonable to conclude that Cu, Pb, Zn and Cr constitute an anthropogenic component, whereas the remaining elements (Mn and Ni) appear to be associated with parent rocks.

Table 3. Total variance explained and component matrixes for heavy metal contents (two-components extracted).

Component	Initial Eigenvalues			Extraction Sums of Squared Loadings			Rotation Sums of Squared Loadings		
	Total	% of Variance	Cumulative %	Total	% of Variance	Cumulative %	Total	% of Variance	Cumulative %
1	3.051	50.852	50.852	3.051	50.852	50.852	2.751	45.851	45.851
2	1.194	19.900	70.753	1.194	19.900	70.753	1.494	24.902	70.753
3	0.822	13.708	84.461						
4	0.447	7.449	91.910						
5	0.334	5.568	97.478						
6	0.151	2.522	100.000						

Elements	Component matrix		Rotated component matrix	
	PC1	PC2	PC1	PC2
Cu	0.753	0.073	0.660	0.369
Pb	0.733	-0.329	0.804	-0.007
Zn	0.894	-0.287	0.934	0.097
Cr	0.751	-0.179	0.760	0.138
Ni	0.694	0.430	0.463	0.673
Mn	0.317	0.884	-0.065	0.937

Extraction method: Principal component analysis; Rotation method: Varimax with Kaiser normalization

Hierarchical cluster analysis

Although performing HCA on variables rather than on cases is preferred in most research studies (Facchinelli et al., 2001; Yalcin et al., 2008), HCA was developed, in the present study, on soil samples, in order to identify similarities in metal contents between the analyzed soil samples. This approach was selected instead of trying to discriminate between the different sources of metals, which was already accounted by PCA. Thus, the aim in performing HCA was to identify the samples which represented different areas in where metal content followed a similar pattern (anthropogenic metal influence, background lithogenic metal levels, etc.). This different approach was preferred since, in that sense, the results provided by HCA and PCA, in this work, are complementary, although they are not quite different methods. PCA helped to group metals according to their different origin. Once this information is known, HCA allowed clustering the areas affected by the different metals, that is, affected or not affected by anthropogenic activities.

Four main clusters can be distinguished in the dendrogram shown in Figure 3, performed with the Ward method, which uses the squared Euclidean distance as a similarity measure. Cluster 1 includes two soil samples (cases 6 and 20) affected by higher Ni and Zn contents; whilesoil samples (cases 4 and 16) were included in cluster 2 due to its higher Mn contents. Soils associated in this two clusters had the lithogenic component was the predominant one. Soils samples (cases 9, 12, 13, 15, 17, 18 and 19) belonged to clusters 3. This cluster includes soils with heavy metal contents enriched of some elements (e.g., Cu, Pb and Zn). Soils (12, 15, 17, 18 and 19) are located close to local roads with dense tra□c, while, soils (9 and 13) were collected in vegetable-garden. For this group, it would be desirable to monitor these levels in order to avoid a continued increase of heavy metal contents as a consequence of human activities.

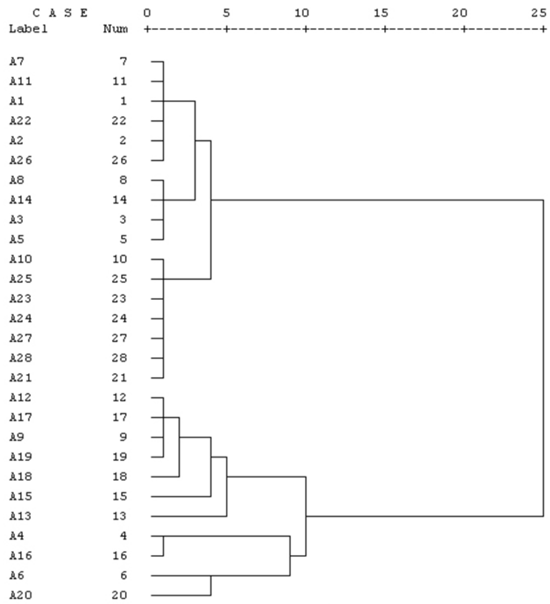

Figure 3. Dendrogram obtained by HCA (hierarchical cluster analysis) for heavy metal contents in soil samples (Ward method).

Finally, the last cluster (cluster 4) included 60% of the soils analyzed and presented low content for most of the elements. In these cases, normal levels of heavy metals were identified. Therefore, an acceptable quality of urban soils was detected in the studying area according to the heavy metal contents analyzed and to background values that may be fixed on the basis of levels recorded in soils included in cluster 4.

This group distribution corresponded to the locations of samples. Within this region, it seems clear that Ni and Mn have a lithogenic origin, whereas Cu and Pb have an anthropogenic origin in the urban soils. As for metal Zn, the origin varies among the different areas, depending on specific human activities that are locally relevant.

Conclusions

The results obtained in this study increase our knowledge of the heavy metal contents and its possible source as for the Teresina urban soils. This study also demonstrated that multivariate analysis methods helped to identify Cu, Pb, Zn and Cr with anthropogenic sources, while the remaining ones (Ni and Mn) were mainly associated with parent material of urban soils. The PCA performed identified the two principal components which control the variability of heavy metal in urban soil. Cu, Pb, Zn and Cr were associated to the same component (PC1) and wasdetermined, mainly, by anthropogenic component due to the large variability of contents in the same samples. Finally, the Ni and Mn contents (PC2) were controlled, principally, by soil parent rocks. The clusters distribution by HCA corresponded to the location of samples.

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