

Determination of Metals in a Soil Occupied by Necropolis in Southern Brazil: The Use of X - Ray Fluorescence Technique (XRF)

Determinação de Metais no Solo Ocupado por Necrópole no Sul do Brasil: Uso da Técnica de Fluorescência de Raios-X (XRF)

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Abstract

Necropolis are areas prone to severe environmental problems due to contamination of both soil and water resources. The study aimed to analyze the concentration of three heavy metals (Copper, Vanadium, and Zinc) in the soil (Oxisol) of a cemetery located in Frederico Westphalen municipality, Rio Grande do Sul State (RS), Brazil. An auger drill was used to collect soil samples in 10 different points and 7 distinct depths with 0 to 300 cm variation. The Energy Dispersive X-Ray Fluorescence technique was applied to estimate the metals concentration in the soil. To expand the spatial attention of the chemical elements, we used Software Surfer 8. That process generated maps through a method of interpolation, Kriging. Some features showed high concentrations between 0 to 300 cm depths, which can be correlated to the activities in question. The heavy metals Vanadium and Zinc showed a weak correlation with the deep, which indicated that those concentrations increase following the depth. However, within this context, none of the elements showed value superior to the quality imposed by the current legislation in the Rio Grande do Sul state.

Keywords: Groundwater pollution; Contamination; Necrochorume.

Resumo

As necrópoles são áreas propensas a graves problemas ambientais, que resultam na contaminação de solos e dos recursos hídricos. Aliado a isso, buscou-se analisar as concentrações de três metais pesados (Cobre, Vanádio e Zinco) em um solo ocupado por cemitério no município de Frederico Westphalen - RS. Com o auxílio de trado holandês, foram realizadas coletas em 10 diferentes pontos e 7 profundidades distintas, com variação de 0 a 300 cm. A estimativa da concentração dos metais, se deu a partir do uso da técnica de Fluorescência de Raios - X por Dispersão em Energia. As informações foram interpoladas por meio do software ArcGIS, versão 10.2. Todos os elementos apresentaram alterações nas concentrações nas profundidades de 0 a 300 cm, que pode ter correlação com a atividade em questão. Foi identificada correlação entre a profundidade e as concentrações de zinco e vanádio, além de uma correlação entre as concentrações dos elementos analisados. Apenas algumas profundidades estiveram dentro do valor máximo permitido pela legislação vigente para o Estado do Rio Grande do Sul.

Palavras-chave: Águas subterrâneas; Cemitério; Necrochorume

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1 Introduction

Cemetery is a word originally from Greek *koimētērion* and of latin *coemeterium*, it means dormitory, enclosure where the dead are kept, place to sleep. Synonym words for cemetery are necropolis, sepulchre, sheep, sacred ground, feet together city and last dwelling place.¹ (FUNASA 2007). According to Fiedler et al. (2012) cemeteries can be seen as a kind of private landfill sites and since the 1950s they have been object of studies due to the risks they can generate to the environment. Cemeteries pose a risk of contamination to underground water and water bodies; through the decomposition process of corpses bacteria and viruses proliferate and release toxic substances that are a menace to health.

From the public health point of view the main risks that can definitely be associated with activities within cemeteries are: possibilities of occurrence of dissemination of diseases through direct contact with microorganisms and contamination of both sources of water useful to human consumption and water bodies (Silva & Malagutti Filho 2008).

According to Sória and Ramires (2004), initial environmental reactions are noted strongly by the stench in the area due to emanation of funeral gases, inadequate grave work for burials, insufficient depth for interment, improper top cover to fill in the grave site as well as the type of soil. Studies in Iowa and New York states in the nineteenth century found high levels of Arsenic (As) in the groundwater downstream of the backyard of the cemeteries, as well as highest levels of Copper (Cu), Zinc (Zn) and Lead (Pb), which are associated with coffins as stated by Knoefes and McGee (2002) and Fiedler et al. (2012).

From 2003, the National Environment Council of Brazil - CONAMA passed the resolution n° 335/2003 - (CONAMA 2003) and demanded adjustments to be made in cemeteries to minimize their impact in nature and on the population. Since then, all areas occupied by cemeteries must have an environmental licence. Thus, we sought to determine a potential chemical pollution of the soil in a local cemetery in Frederico Westphalen municipality. The analytical technique applied was the *Energy Dispersive X-ray Fluorescence* (EDXRF) analysis. Inside of the area of study was especially analysed the concentration of the heavy metals: Copper, Vanadium and Zinc.

¹ sheep, sacred ground, and last dwelling place are Brazilian Portuguese regional meanings for the word cemetery.

2 Methodology

2.1 Description of The Study Area

Frederico Westphalen is located in the northwest state of Rio Grande do Sul, Brazil, in the latitude 27°21'33"S and longitude 53° 23'40" W, and covers an area of 264.843 km² with a population of 28.843 inhabitants (IBGE 2010). According to Rio Grande do Sul State Environment and Infrastructure Department - SEMA (2004) the city belongs to the hydrographical basin of Uruguay River (U) and sub-basin of Varzea River (U - 100). The climate in the region is of a humid subtropical type (Moreno 1961), with precipitations well distributed throughout the year.

Jardim da Saudade cemetery is situated at a 150 km distance from the riverbank Uruguai in the northward direction, with an average altitude of 555 m. The cemetery was opened in 1930 and currently has approximately three thousand graves and receives seven corpses a month on average. The area occupied by the necropolis is of 1.2 hectares.

At the site there are two main different types of graves: one where cadavers are buried in direct contact with the soil and another where they are buried on existent constructions made from prefabricated panels. According to Kemerich et al. (2012b) those graves are normally "drawers" where corpses are placed an average of one meter above the ground.

With reference to the local geology, the city is inside the basalt lava flows of Serra Geral Formation, Paranapanema facies, and that formation has undergone physical and chemical weathering processes (Geological Survey of Brazil/CPRM 2006). In relation to the hydrogeology, as reported by Machado and Freitas (2005), that location is where the Serra Geral Aquifer Systems (SASG)² I and II are found. The Serra Geral Aquifer System is formed by basaltic lava flows, amygdaloides and cracks capped with thick reddish soil.

2.2 Sampling Points for Soil Analysis

In accordance with the methodology recommended by Kemerich et al. (2012a) and taking into consideration the local topography and natural flow of surface water, ten samples were subject to analysis. Using Software ArcGIS, the collected points were mapped from P1 to P10 (Figure 1). P1 had the highest sampling altitude, with 564 m, and represented the control point, whereas P10 showed the lowest sampling altitude (545 m). Figure 1 represents the total area

² SASG is the Serra Geral Aquifer System.

of necropolis, the surface water contour lines and sampling points. These points were obtained using a handheld GPS (Global Position System), Garmin Extrex model, using the Universal Transverse Mercator (UTM) coordinate system, 22 S spindle, with the SIRGAS 2000 datum.

An auger was used to collect soil samples and they were stored in plastic bags. The identification of location and depth was performed subsequently. In the ten sampling points soil samples were collected in seven levels in the soil profile: one surface sample, and subsequently samples at the depths of 50 cm, 100, 150, 200, 250 cm, and a last sample at a depth of 300 cm.

2.3 Technique Used in The Analysis

The X-Ray Fluorescence technique (XRF) allows to analyse and monitor different kinds of samples; for example, for environmental samples it is a tried and proved analytical method. For the last twenty years, XRF has evolved considerably, especially with reference to environmental samples (Melquiades & Appoloni 2004; Vanhoof, Corthouts & Tirez 2004).

The Energy Dispersive X-Ray Fluorescence (EDXRF) facilitates the application of the non-destructive, methodological quantitative and qualitative approaches. EDXRF requires little sample preparation and fits into

the green chemistry, when compared to the Inductively Coupled Plasma Mass (ICP-MS) and the Atomic Absorption Spectrometry (AAS) techniques; thus, EDXRF is seen as a kind of low-cost analysis. The main benefit of this method is its non-destructive feature; this advantage allows it to be used with other techniques as well.

According to the International Union of Pure and Applied Chemistry (IUPAC 1997), the characteristic X-ray radiation can be defined as resultant of the interaction between particles of high-energy or photon with matter. Since each element releases certain energies during the process, it becomes viable to access the qualitative information of the sample. Similarly, the intensity of energy emitted is in indirect proportion to the quantity of the element in the sample (Jenkins 1999).

2.4 Sample preparation, estimation of metals and statistical analysis

In the Laboratory of Environmental Monitoring and Planning (LMPA) of the Federal University of Santa Maria (UFSM), campus Frederico Westphalen, 20 g of soil (Oxisol) were dried in an oven at a temperature of 105 °C (centigrade) for two hours. To conduct the analysis, a specific glass laboratory equipment (Becker 50 mL) was used for the deposition process.

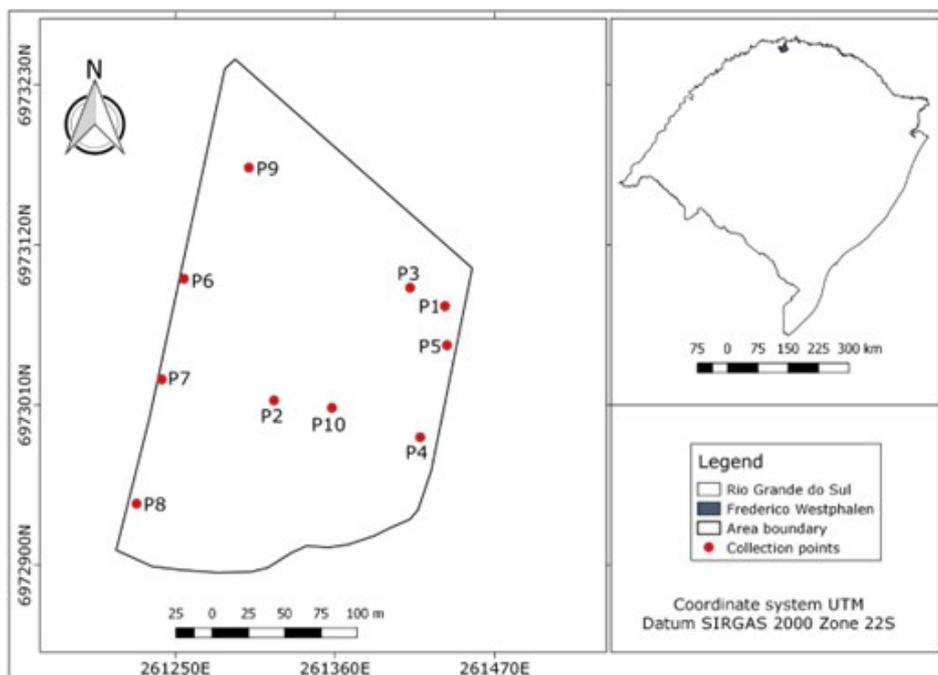


Figure 1 Location of the study area and a cartogram of the cemetery site with and sample points.

After applying the coning and quartering technique, the oven drying method and grinding the samples using a pestle and mortar, they were passed through a sieve to obtain a granulometry inferior to 125 μm . To obtain the measurements through the X-Ray Fluorescence technique first, 3 g of sample were laid in cells covered with 2,5 μm Mylar[®] film. The soil samples were analysed in the Applied Nuclear Physics Laboratory of the State University of Londrina (UEL).

The analysis was performed using the Shimadzu EDX 720 Energy Dispersive X-Ray Fluorescence Spectrometer that has a rhodium X-Ray tube, Si (Li) detector cooled with liquid nitrogen. To perform the analysis two routine

measurements were used: Na-Sc 15 kV, 980 μA , 200s; Ti-U 50kV, 500 μA , 100 s. The quantification of the samples was executed by using the fundamental parameters method.

The reference quality values advised by the approved resolution of Foundation for Environmental Protection Henrique Luis Roessler/FEPAM 085/2014 (FEPAM 2014) about evaluation of metals are represented on Table 1.

Action Software (ESTATCAMP 2014) was used for statistical analysis through correlation coefficient. Thusly, it was possible to determine the *Pearson* correlation between the variables analysed. The Inverse Distance Weighted (IDW) interpolation method was used to distinguish the results.

Table 1 Quality Reference Values (QRV) by Resolution 085/2014 (FEPAM 2014) for Plateau Volcanic Rocks Region.

Chemical Element	Percentiles (Concentration of mg.kg^{-1})*	
	75	90
Copper (Cu)	165	203
Vanadium (V)	473	567
Zinc (Zn)	102	120

Source: *Text adapted from Resolution FEPAM 085/2014 (FEPAM 2014).

3 Results and Discussion

Table 2 shows the Quality Reference Value (QRV) of the analysed elements, for the Geological/Geomorphological Region of the Plateau Volcanic Rocks, according to FEPAM (2014).

3.1 Copper (Cu)

The different concentrations of Cu in the area occupied by the cemetery from points P1 to P10 within the depths of 0, 50, 100, and 150 cm are represented in Figure 2. For this chemical element, at depths from 0 (surface) to 150 cm, the concentrations were above the QRV established by FEPAM (2014) for the 75th (165 mg.kg^{-1}) and 90 (203 mg.kg^{-1}) percentiles.

Copper's main anthropogenic sources are: mining, casting, burning coal and those are originators of Cu in both energy supply and incineration of waste from municipalities (CETESB 2012). Copper, like other heavy metals, is not biodegradable and its complexation in the soil is very dynamic and is affected directly by its surroundings which changes mainly by the amount of organic matter, pH and the Cation Exchange Capacity (Sodr e & Lenzi 2001). Wang, Liu, and Qin (2019) describe that Cu is one of the heavy metals in which exceeds the contaminant standard

limits defined by the National Soil Pollution Investigation Bulletin.

The total contents of Cu in the study area are inside of the Brazilian soil ranges with a concentration of 1 to 126 mg.kg^{-1} (Malavolta 1980). According to Chrostowska and Skalmowski (1997) the accepted proportion of Cu in solid waste compost varies between 300 to 800 mg.kg^{-1} (Poland), whereas in countries such as Austria is 400 mg.kg^{-1} and in Spain is 1,750 mg.kg^{-1} . In studies conducted in cemetery areas, the authors describe Cu procedures between undetected and 245.40 mg.kg^{-1} (Jonker & Olivier 2012; Silva et al. 2019; Spongberg & Becks 1999; Żychowski 2021).

As reported by Costa (2005) the behaviour of heavy metals will depend on the type of soil, in other words, the type of organic matter within their contents, the iron oxide, the types of aluminium, the concentration of minerals in clay, the CEC, the aggregation, the humidity, and other factors.

The highest Cu concentration (519 mg.kg^{-1}) in P7 and P1 were found at a depth of 0 cm, while the lowest concentration was found in P3 (339 mg.kg^{-1}). Both values are above the range recommended by Resolution 085/2014 (FEPAM 2014). At a depth of 50 cm, the highest concentration was observed at P7 (599 mg.kg^{-1}) and the lowest concentration at P9 (407 mg.kg^{-1}).

Table 2 The concentration of chemical elements in the analysed depths in the 75 and 90 percentiles.

Depths (cm)	Elements (Concentration in mg.kg ⁻¹) *					
	Copper		Vanadium		Zinc	
	Percentiles		Percentiles		Percentiles	
	75	90	75	90	75	90
0	494	519	2,062	2,157	236	265
50	528	558	2,158	2,193	209	225
100	508	538	2,075	2,106	214	216
150	499	718	1,976	2,022	211	218
200	560	890	2,297	2,420	233	242
250	538	602	2,088	2,241	204	219
300	458	562	2,073	2,271	204	214

Source: Text adapted from FEPAM (2014).

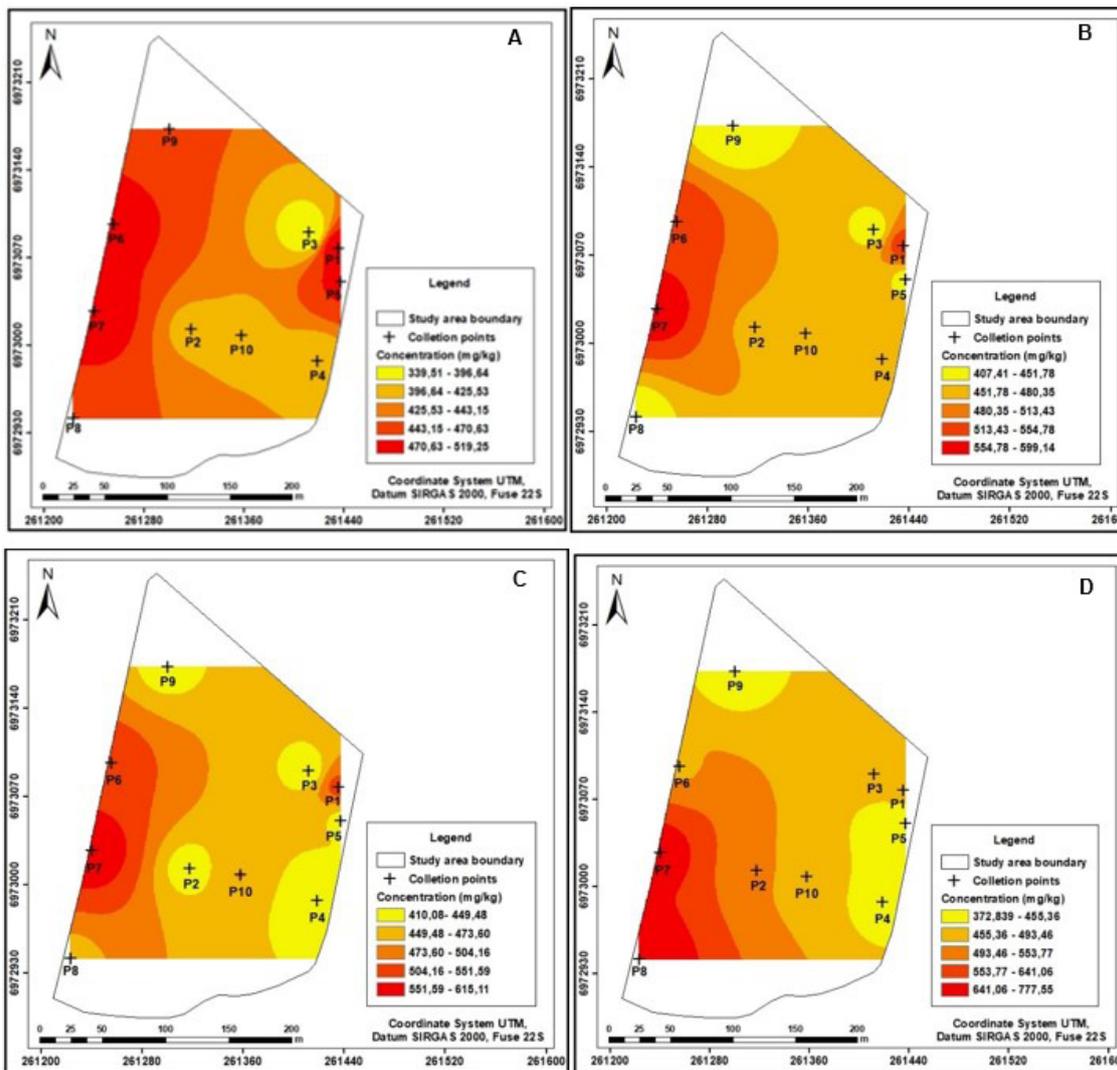


Figure 2 Concentration of Copper inside of Jardim da Saudade Cemetery in the depths of A. 0; B. 50; C. 100; D. 150 cm.

The highest concentration obtained at a depth of 100 cm was located in P7 (615 mg.kg⁻¹), and the lowest in P4 (410 mg.kg⁻¹). At a depth of 150 cm, the highest concentration was found in P8 (776 mg.kg⁻¹) and the lowest in P5 (373 mg.kg⁻¹). Thus, at these depths, all concentrations obtained exceeded the QRV in both percentiles (75 and 90). The Copper variation within the study area in the 200, 250, and 300 cm depths are represented in Figure 3. At a depth of 200 cm, the highest concentration was found in P8 (964 mg.kg⁻¹) and the lowest in P5 (336 mg.kg⁻¹). Both values are above the range recommended by FEPAM (2014) for the 75 and 90 percentiles.

Cu at a depth of 250 cm presented values below the Detection Limit (< DL) of the EDXRF technique at points P5, P7, and P10, and below that established by legislation at P9 (71 mg.kg⁻¹). At a depth of 250 cm, the values ranged

from < DL (P5, P7, and P10) to 1,010 mg.kg⁻¹ (P8), where, except for concentrations < DL, the others were above that established by FEPAM (2014) for both percentiles. Cu at a depth of 300 cm presented values within the range recommended by FEPAM (2014) at points P1, P5, P7, and P8 (< DL). The other sampling points presented concentrations above the maximum allowed concentration, up to a maximum of 591 mg.kg⁻¹ (P6). Kemerich et al. (2012a), in a study carried out on cemetery soil in the location of Seberi, about 15 km away from the study area, found a maximum Cu concentration close to 600 mg.kg⁻¹, and the lowest concentration was 330 mg.kg⁻¹, which are different from the ones found at the present research. CPRM (2006) indicates that this region shows the presence of native Copper, thus this element can be of natural origin and not necessarily due to the cemetery activity.

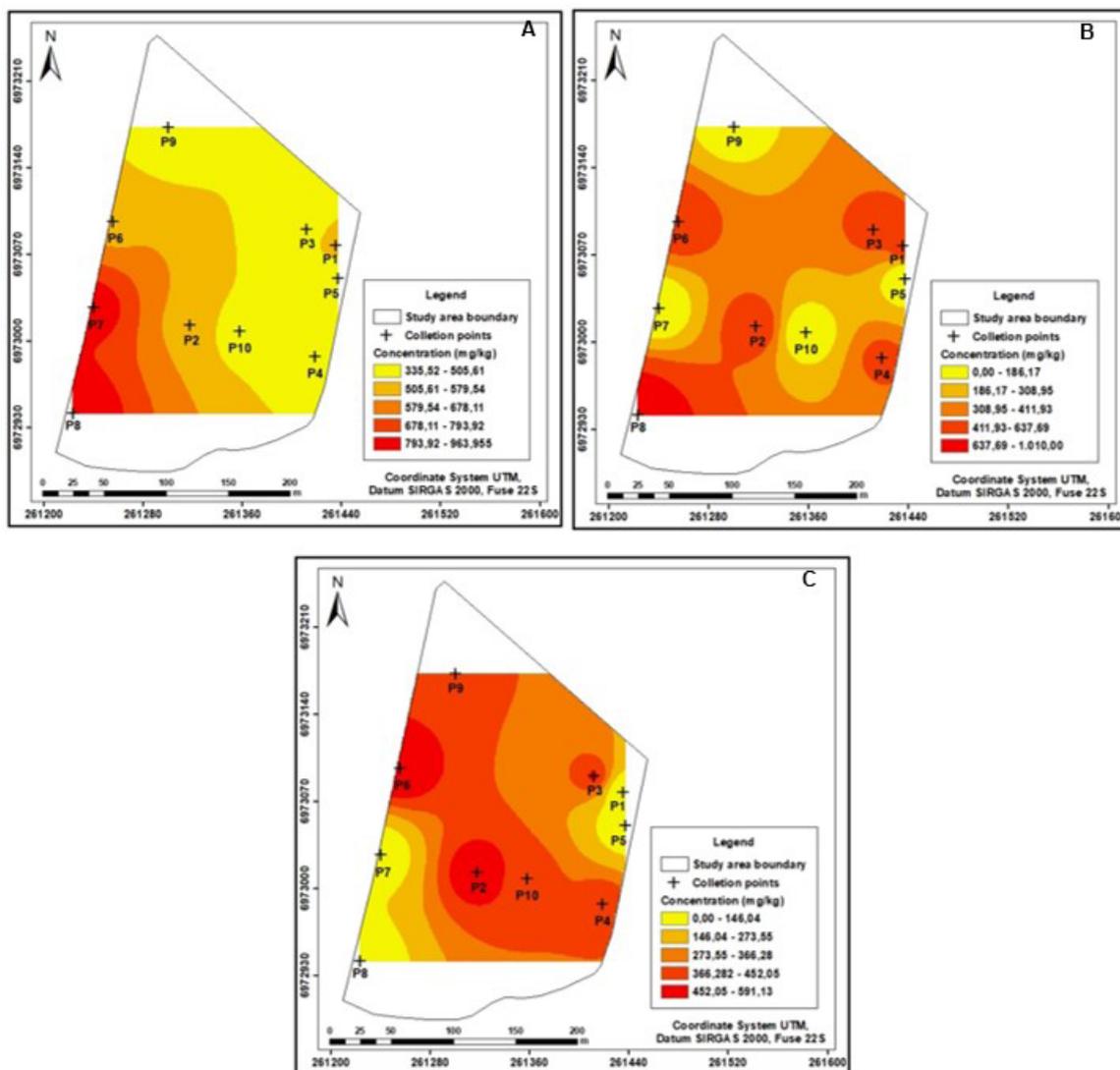


Figure 3 Concentration of Copper in the: A. 200; B. 250; C. 300 cm depths inside of Jardim da Saudade Cemetery.

3.2 Vanadium (V)

The variation of Vanadium concentration within the study area from points P1 to P10 in the depths of 0, 50, 100, and 150 cm is represented in Figure 4. In general, most concentrations obtained at these depths were above the QRV established by FEPAM (2014) for the 75th and 90th percentiles, except for P10 at depth of 100 cm. In superficial samples, concentrations ranged from 1,533 mg.kg⁻¹ (P4) to 2,291 mg.kg⁻¹ (P10). At 50 cm, the values ranged from 1,624 mg.kg⁻¹ (P10) to 2,218 mg.kg⁻¹ (P1). At a depth of 100 cm, P10 had the lowest concentration, 25.20 mg.kg⁻¹ (This value is below the QRV for both percentiles), and the highest concentration was present at P7 (2,184.62 mg.kg⁻¹). For a depth of 150 cm, the highest concentrations were found

in P4 (2,045 mg.kg⁻¹), and the lowest concentration was found in P6 (1,260 mg.kg⁻¹).

The variation of Vanadium concentration in the depths of 200, 250, and 300 cm within the study area is represented in Figure 5. For a depth of 200 cm, the highest concentration was present at P3 (2,442 mg.kg⁻¹) and the lowest at P5 (1,395 mg.kg⁻¹). Aimed at a depth of 250 cm, concentrations ranged from < DL at sampling points P5, P7, and P10 to 2,246 mg.kg⁻¹ (P2). P9 had a concentration lowest than the VMP established by FEPAM (2014) with 21.84 mg.kg⁻¹. At a depth of 300 cm, the highest concentration of Vanadium was presented at P6 (2.293 mg.kg⁻¹ and the lowest concentrations were observed at points P1, P5, P7, and P8 (< DL), which were also below the QRV.

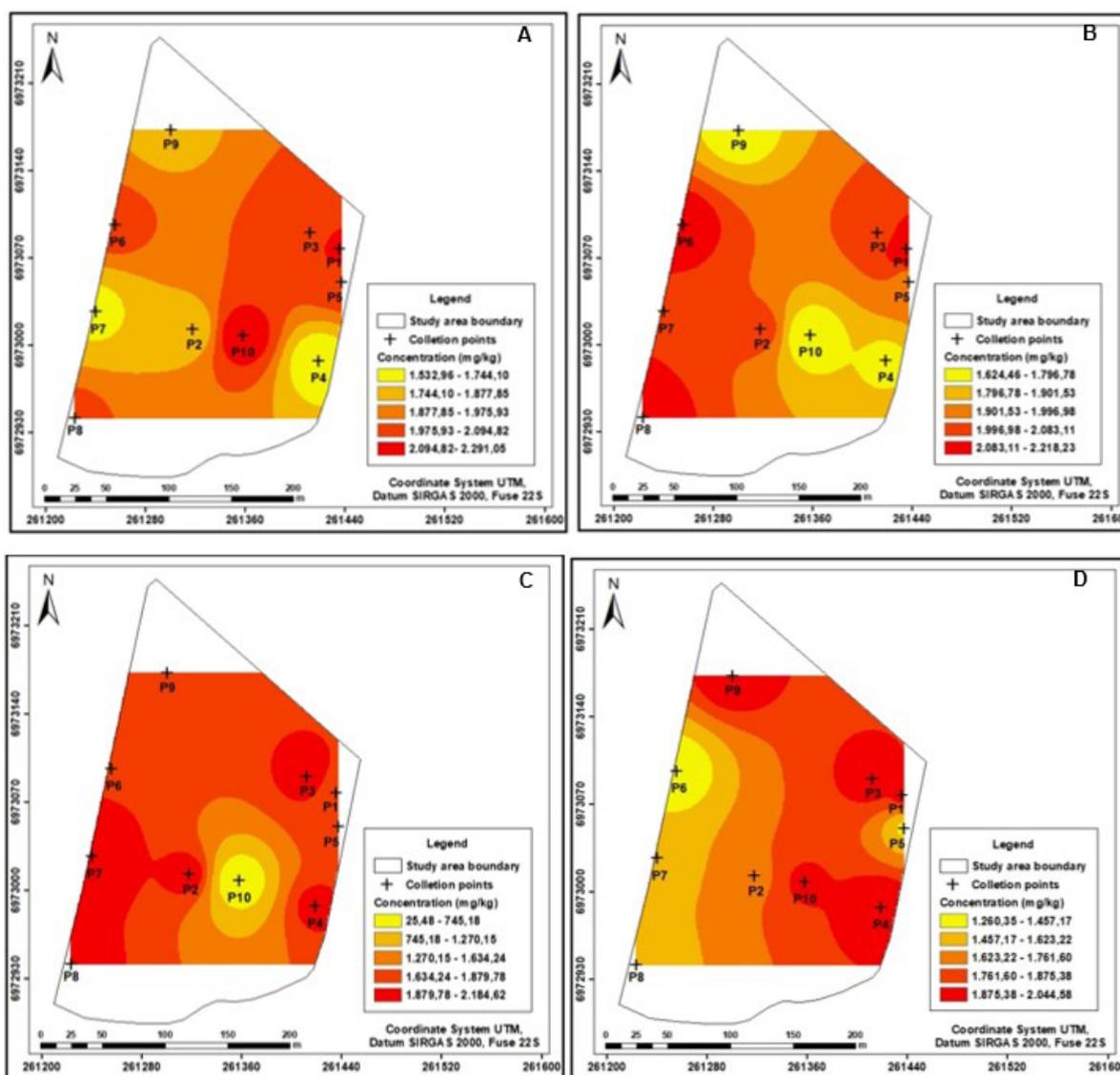


Figure 4 Vanadium Concentration in the depths of: A. 0; B. 50; C. 100; D. 150 cm inside of the Jardim da Saudade Cemetery.

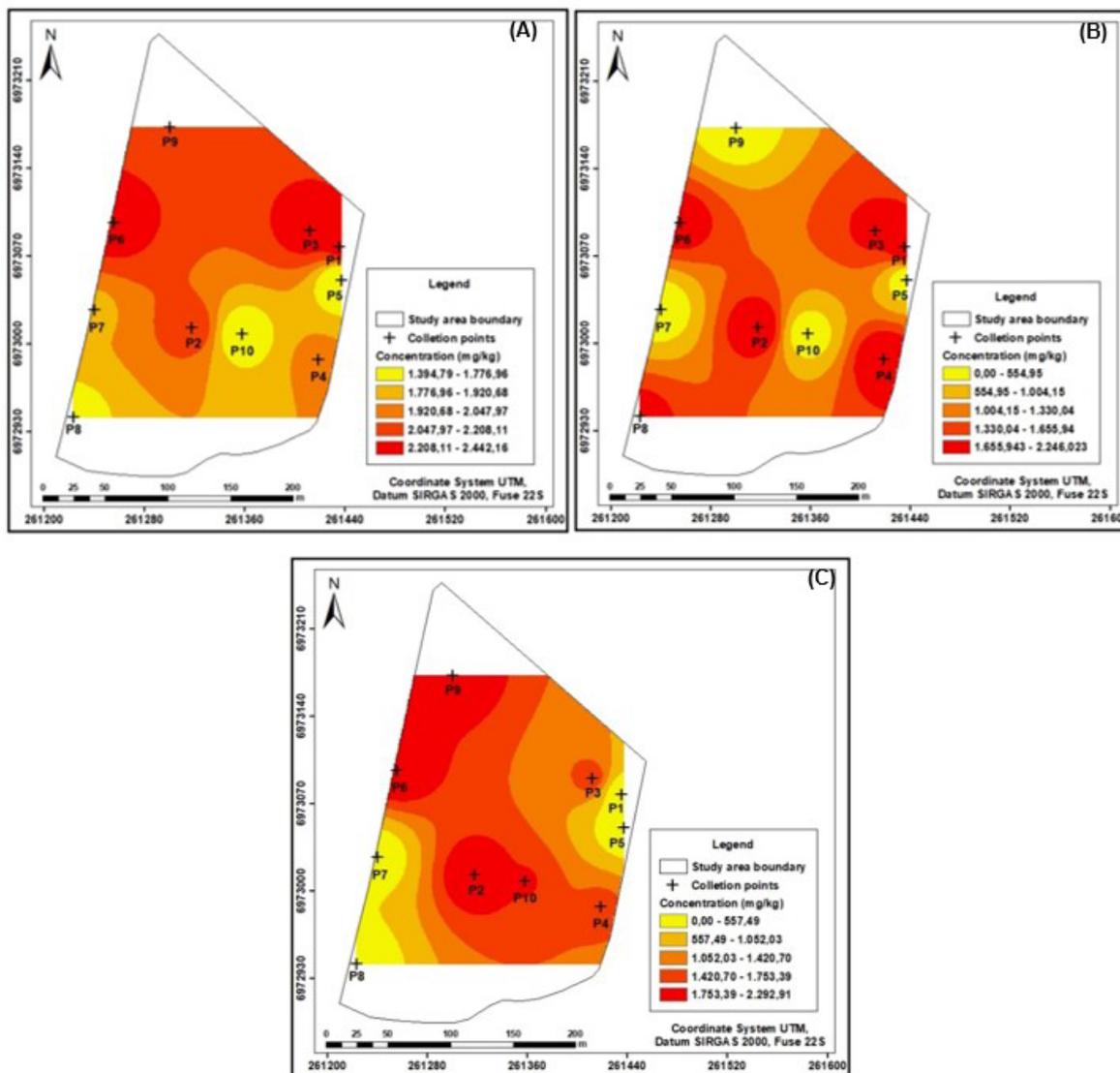


Figure 5 Concentration of Vanadium at: A. 200; B. 250; C. 300 cm depths inside of Jardim da Saudade Cemetery.

The weathering process in rocks and minerals during the formation of the soil can release vanadium in the form of the complexed anion, and it can stay in the soil or migrate to the hydrosphere (Shuquair 2002). According to the same author mentioned above, vanadium is one of the most common elements in the earth’s crust with trace amount of 100 - 150 mg.kg⁻¹.

3.3 Zinc (Zn)

The variation of Zn concentration inside of the study area from P1 to P10 in the depths of 0, 50, 100, and 150 cm are represented in Figure 6. In general, only at depths of 0 and 150 cm (P6), 50 cm (P8 and P9), and 100 cm (P10) the values were within the established by FEPAM (2014). The other depths and points were in disagreement with the

values proposed by the referred legislation. Wang, Liu and Qin (2019) describe that Zn is one of the heavy metals in which it exceeds the contaminant standard limits defined by the National Soil Pollution Investigation Bulletin, a government agency.

According to Medeiros (2012), Zn (from German word *zinke* - tooth) is a chemical element present in several natural environments (water and soil). Zinc presents itself as organic and inorganic and is available mainly as Zn or a complex with organic ligands (Santos 2005; Broadley 2007). In studies carried out in cemetery areas, the authors describe Zn concentrations between 2.64 and 112.40 mg.kg⁻¹ (Jonker & Olivier 2012; Silva et al. 2019; Spongberg & Becks 1999; Żychowski 2021). These values are well below those found in the present study.

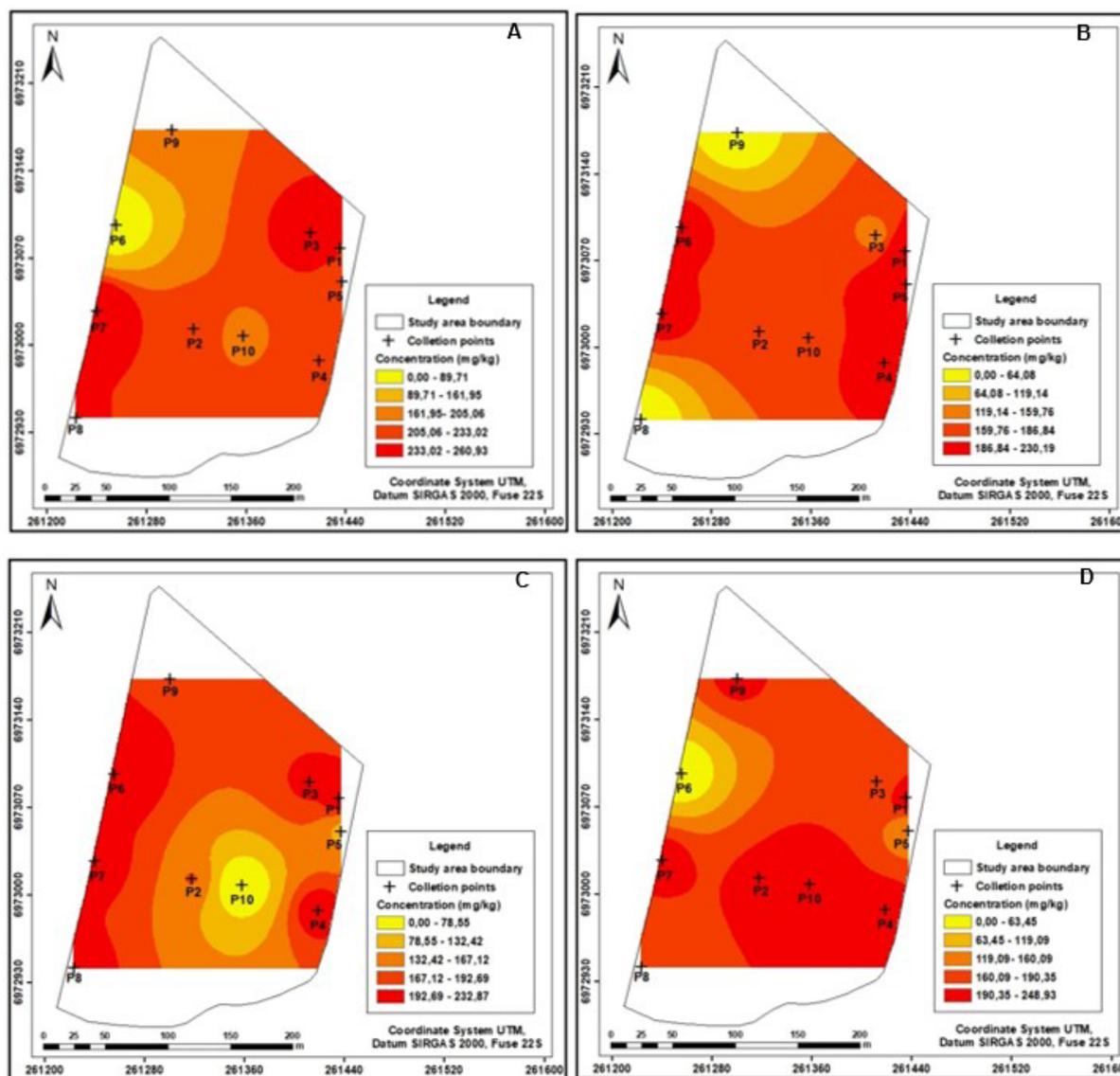


Figure 6 Zinc Concentration of A. 0; B. 50; C. 100; D. 150 cm depths inside of Jardim da Saudade Cemetery.

Zn is an important substance for various industrial sectors. It is used in electroplating, tinting, alloy and other utilities. In some localities near to Taiwan’s installation Zn is considered to be a serious contaminant due to the lack of environmental care (Chang et al. 2007).

In accordance with Iwegbue et al. (2007) the contents of Zn in Brazilian soils are $39 \pm 24 \text{ mg.kg}^{-1}$ on average. Zinc is vital for organisms because it is used as an essential cofactor in proteins. It is also indispensable for plant growth (Robson 1993).

At superficial depth, values ranged from <DL at P6 to 261 mg.kg^{-1} at P3. At 50 cm, the lowest concentrations were found at points P8 and P9 (<DL), which were within the established by legislation, and the highest at P5 (225

mg.kg^{-1}). By 100 cm, P10 had the lowest value (<DL), and the highest concentration was identified at P6 (233 mg.kg^{-1}). Finally, at 150 cm depth, the values ranged from <DL (P6) to 249 mg.kg^{-1} at P10.

The Kemerich et al. (2012a) investigation inside of a cemetery soil in Seberi municipality - RS showed highest concentration in the 0 cm depth (209 mg.kg^{-1}) and the lowest concentration presented a value below of the Limit of Detection; values similar to those found in this research.

The distribution of concentration of Zn in the depths of 200, 250, and 300 cm is represented in Figure 7. In general, only a depth of 250 cm (P5, P7, P9, and P10) and a depth of 300 cm (P1, P5, P7, and P8) concentration was within the limits established by law.

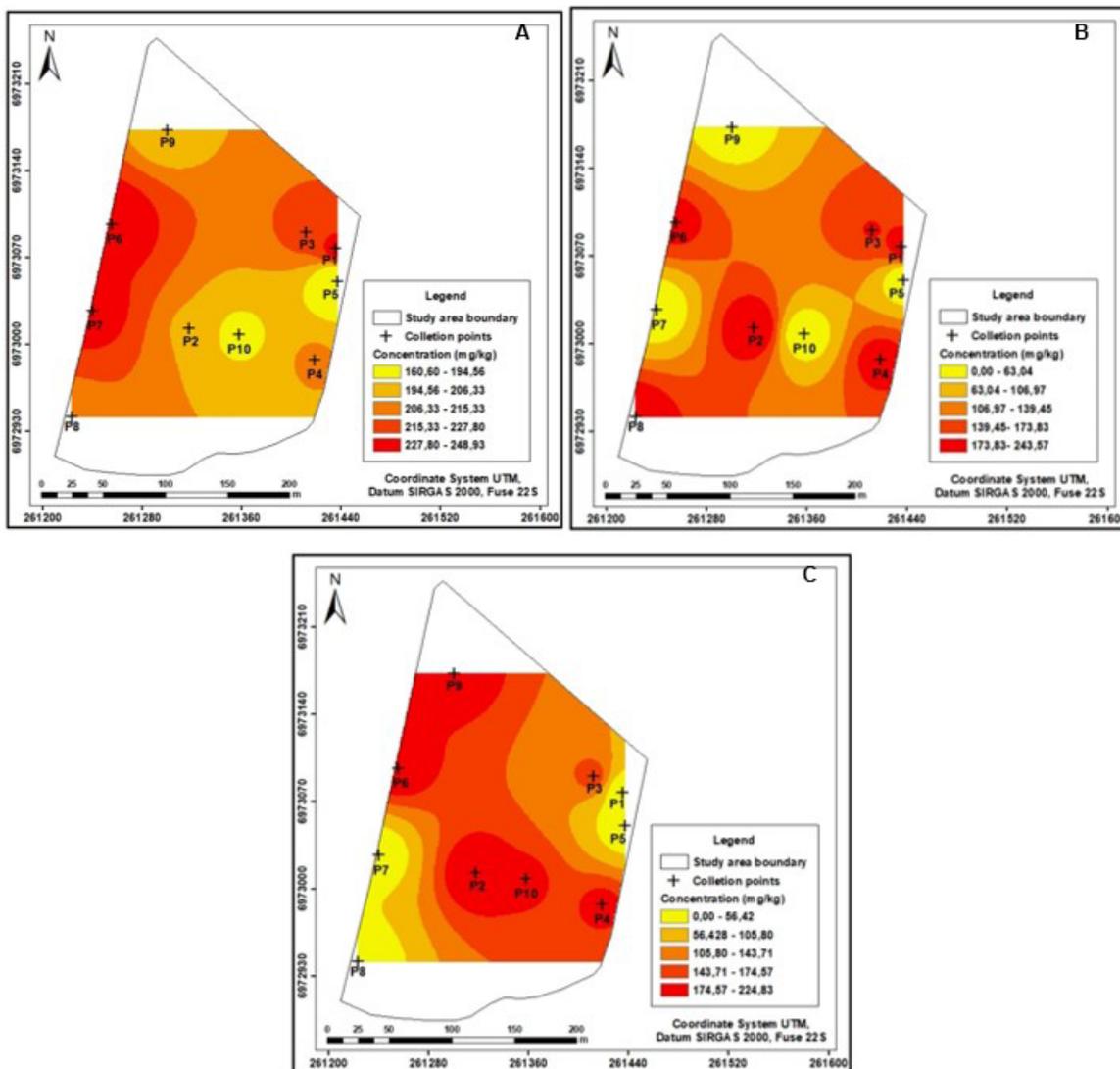


Figure 7 Zinc Concentration in the: A. 200; B. 250; C. 300 cm depths inside of Jardim da Saudade Cemetery.

At 200 cm, concentrations ranged from 161 mg.kg⁻¹ (P5) to 237 mg.kg⁻¹ (P1). At 250 cm, the lowest concentrations obtained were < DL (P5, P7, and P10), while the highest was present at P2 (244 mg.kg⁻¹). At this depth, in addition to the values < DL already described, P9 also presented a concentration (16.86 mg.kg⁻¹) within the established by FEPAM (2014) in both percentiles. At a depth of 300 cm, concentrations ranged from < DL at P1, P5, P7, and P8 to 225 mg.kg⁻¹ at P2.

Table 3 shows the statistical correlation for the level of 5% between the variable point, elevation, depths and the concentration of Copper, Vanadium and Zinc. The interpretation of the correlation data followed the values

proposed by Dancey and Reidy (2013), so the p values are classified as weak correlation (R-values between 0.10 to 0.30), moderate (R-values between 0.40 to 0.60), and strong (R-values between 0.70 to 1.00). The point variable showed a strong correlation with the elevation of the terrain (-0.97), a low correlation with the concentrations of Vanadium and Zinc (- 0.24). The depth showed a weak correlation with the concentrations of Vanadium (- 0.26) and Zinc (- 0.25). A moderate correlation was identified between copper concentrations and values of Vanadium (- 0.69) and Zinc (0.64). Finally, a strong correlation was present between the concentrations of Vanadium and Zinc (0.76).

Table 3 Pearson Correlation significance level of 5%.

Variants	Point	Elevation	Depth	Copper	Vanadium	Zinc
Point	1.00					
Elevation	-0.97*	1.00				
Depth	0.00	0.00	1.00			
Copper	-0.03	-0.01	-0.22	1.00		
Vanadium	-0.24*	0.23	-0.26*	0.69*	1.00	
Zinc	-0.24*	0.22	-0.25*	0.64*	0.76*	1.00

Source: Data generated via Action Software (ESTATCAMP 2014); *Significant correlations with $p < 0.05$; $N = 70$

4 Conclusion

The EDXRF technique was useful in analyzing the concentration of copper metals. Zinc and Vanadium in soil. Changes in concentrations were identified for all depths and elements analyzed by the technique used, mainly with the increase in depth, which may be related to the percolation of necroslurry by the soil profile.

Most concentrations were outside the limits established by State legislation, indicating a possible change in the natural quality of the soil. The correlation between copper and vanadium rates in depth was identified, in addition to the correlation between the three chemical elements. It is recommended to carry out additional studies with other analytical techniques and subsequent monitoring of water quality through wells.

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Pedro Daniel da Cunha Kemerich: conceptualization; methodology; writing – review and editing; supervision. **Fábio Luiz Melquiades:** Formal analysis, Methodology; Validation; Writing – original draft; Writing review and editing; Supervision. **Willian Fernando de Borba:** Conceptualization; Formal analysis, Methodology; Validation; Writing – original draft; Supervision; Visualization. **Gabriel D’Avilla Fernandes:** Conceptualization; Writing review and editing; Visualization. **Ademir Eloi Gerhardt:** Other contribution (Collection and preparation of soil samples). **Guilherme Barros:** Other contribution (Collection and preparation of soil samples). **Débora Bianchini:** Methodology; Writing review and editing. **Ericklis Edson Boito de Souza:** Methodology; Writing review and editing; Funding acquisition; Supervision. **Gabriela Souza Nascimento da Silva:** Writing review and editing; Other contribution (Translation).

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The authors declare no potential conflict of interest.

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