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Metal Analysis of Leachate from the Organic Fraction of Urban Solid Waste (MSW) from the Municipality of Belém/PA

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Abstract: In this study, the analysis of metals in the solubilized extract of the organic fraction of Urban Solid Waste (MSW) from the municipality of Belém do Pará was carried out. The waste used in this research was collected in residential areas, through door-to-door collection, with the points and neighborhoods served in the municipality of Belém determined by the sectorization of these locations, with family income as the main parameter. The MSW was collected and transported to the segregation area. Gravimetric analysis of MSW was carried out and the selected organic and paper fractions were subjected to drying, crushing and sieving pre-treatment. Next, the solubilized extract of the organic fraction of MSW was obtained, following the method set out in NBR nº 10.006/2004 of the Brazilian Association of Technical Standards. The values obtained were compared with CONAMA Resolutions nº 357/2005, 396/2008 and 430/2011, in addition to being compared with results of bibliographical research. The results indicated that these wastes do not comply with environmental and health regulations. Although a highly significant association was found between chromium and boron through Pearson's correlation, the remaining strong correlations between other elements did not reach statistical significance. Furthermore, a similarity was observed in the solubilization conditions of these wastes with those found in landfill leachate.

Keywords: municipal solid waste; leachate; heavy metals; Pearson correlation

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1. Introduction

The generation of solid waste is one of the biggest environmental problems in the world today. In developing countries, improper disposal of municipal solid waste (MSW) is a major environmental and health concern. In addition to the lack of financial resources, there is a lack of administrative will, financial capacity and poor management of the complex multidimensional solid waste management system in developing countries TSAI et al., [1].

Because of urbanization and global population expansion, 2.01 billion metric tons of MSW were generated globally in 2016, and the amount is expected to reach 2.59 billion metric tons in 2030 [2,3].

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Around 37% of the total waste generated is disposed of in landfills, 33% is dumped in the open, 19% is recycled and 11% is incinerated KAZA et al., [3].

In 2021, the Northern Region of Brazil generated approximately 36% of Urban Solid Waste (MSW) disposed of appropriately, with 64% incorrectly disposed of [4]. A gradual increase in the amount of solid waste leads to several problems in the transport, storage and disposal of this waste and makes the efficient management of solid waste complicated. SILVA et al., [5]. MSW is considered heterogeneous due to the presence of hazardous and non-hazardous materials, but it is important to highlight that when non-hazardous pollutants continue to accumulate in the environment, after a certain level, it also becomes dangerous for living beings KAZA et al., [3].

Consequently, MSW needs proper disposal and management; Otherwise, they may represent a direct threat to the environment and people's health due to inadequate management RENOU et al., [6]. The most used methods of MSW management in developing countries are landfills and open dumping, which are far below acceptable or recommended standards due to the lack of leachate collection and treatment systems [7–9]. Como resultado, esses lixiviados migram vertical e lateralmente para o meio ambiente [10–12]. These migrated leachates are the main sources of pollutants in soil, water, and the environment [12]

The bioavailability of toxic elements (heavy metals) in the soil depends mainly on soil physicochemical processes such as sorption, complexation, and redox processes Rodrigues et al., [13]. These processes depend on soil characteristics, such as soil pH, soil organic matter content, percentage of clay and non-crystalline metal oxides Rodrigues et al., [13]. Metallic pollutants are not biodegradable and have a high chance of accumulating in biological systems over time [14,15].

MSW generally comprises toxic and carcinogenic heavy metals in different forms that can be leached into soil and water bodies. Arsenic (As), cadmium (Cd), chromium (Cr) and nickel (Ni) are classified as group 1 carcinogens by the International Agency for Research on Cancer, which are also toxic in nature [16].

Furthermore, the enrichment of heavy metals can cause serious ecological risks when absorbed by different aquatic organisms, thus entering the complex food chain DASH et al.,[17]. It also has direct impacts on the functioning of soil enzymes Singh et al., [18] and microbial biomass [19,20]. Heavy metals not only degrade soil quality, but also intrude and impede the growth of the microbial community Stamps et al., [21]. For example, lead (Pb) and Zn have detrimental effects on bacteria, Cd impacts on fungal population, and Cr affects soil bacterial diversity [22–24].

Due to its characteristics, leachate requires adequate treatment so that the values of its physical, chemical, and biological parameters meet the limits established by current legislation and do not cause impacts on the environment [25]. Biological treatment processes are the most recommended for leachate, given their ability to remove organic matter, however, this is something that must be evaluated, as these compounds have variable flow rates and organic load, which can harm treatment units LANGE et al ., [26]. Given the above, this work proposes to systematically investigate in the short term the distribution of heavy metals in the form of oxides and salts in the leachate extract obtained via chemical and microbiological transformation of the organic fraction of municipal solid waste (MSW).

2. Materials and Methods

The process flowchart illustrated in Figure 1 generally summarizes the strategy applied, as well as the methodology used in the process, described systematically in a logical sequence of ideas, methods and procedures involving all stages of the process, for sustainable disposal and characterization of Urban Solid Waste (MSW), which was carried out through the analysis of metals in the leachate.

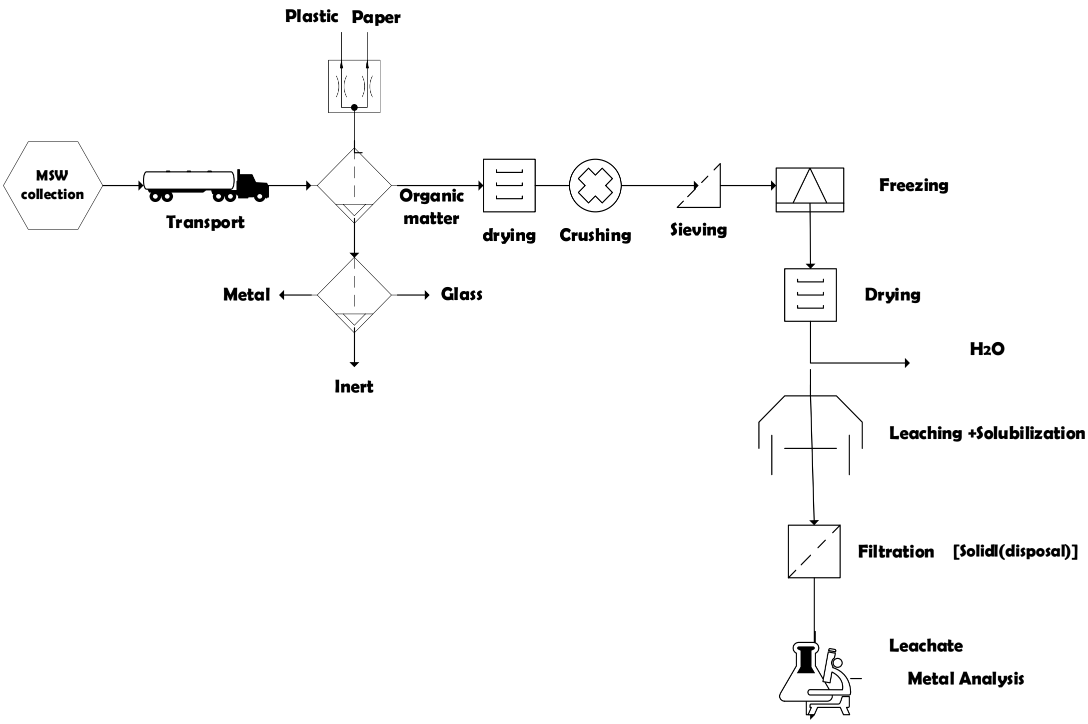

Figure 1. Flowchart of strategy adopted in the research.

2.1. Characterization of the Study Area

This study followed a methodology similar to that of Assunção et al. [27] and Pereira et al. [28], delimiting the research area in the municipality of Belém, in the state of Pará, where the company TERRAPLENA LTDA is responsible for collecting solid waste in Lot 1 (Figure 2), according to the Municipal Basic Sanitation Plan [29]. Understanding the specific context of basic sanitation in this region is extremely important. The studied area encompasses 21 neighborhoods that represent a significant part of the city, and includes 37 routes, highlighting the extent of coverage of services related to basic sanitation provided by TERRAPLENA LTDA, such as solid waste collection, water and sewage treatment, among others. This information is crucial for assessing the effectiveness and coverage of sanitation services in the region, as well as for identifying challenges or areas in need of improvement. The neighborhoods served by the company include: Cremação, Nazaré, Terra Firme, Batista Campos, Cidade Velha, Jurunas, Condor, Guamá, Universitário, Canudos, São Braz, Fátima, Reduto, Umarizal, Marco, Curió-Utinga, Aurá, Águas Lindas, Guanabara, Campina, Castanheira, in addition to the district of Mosqueiro [150]. It is essential to consider the socioeconomic diversity of neighborhoods, as this directly influences the demands and needs of the population in relation to basic sanitation services. By analyzing this distribution, it is possible to direct public policies and investments more effectively, seeking to improve the living and health conditions of local communities. This strategic approach allows for a more precise allocation of resources, meeting the specific needs of each demographic group and promoting an equitable distribution of the benefits of basic sanitation, according to de Assunção et al. [27] and Pereira et al. [28].

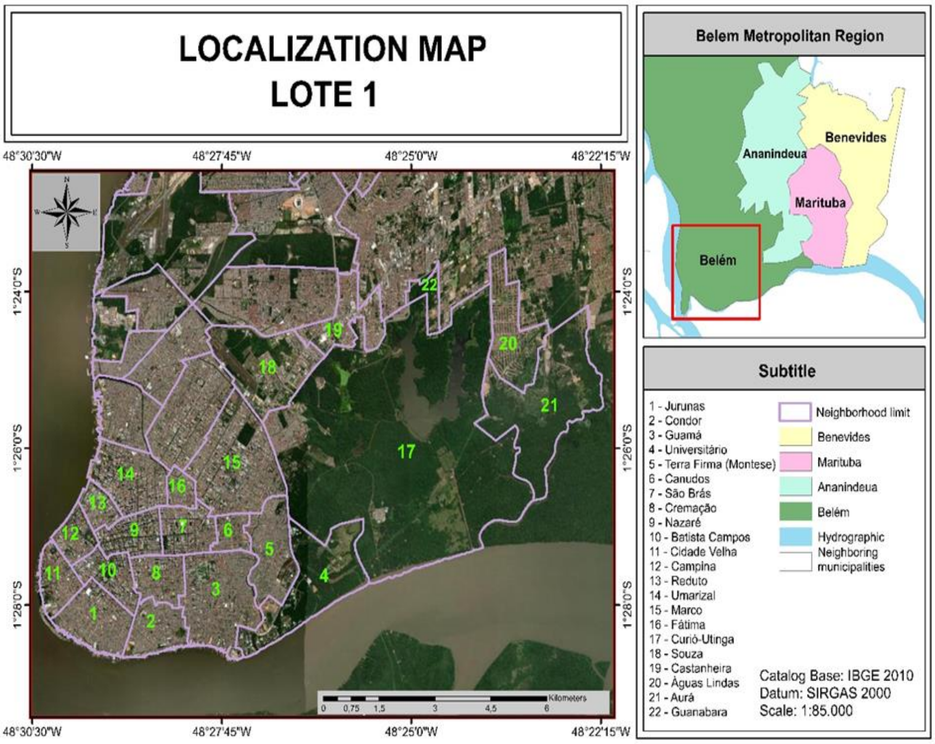

Figure 2. Location of the waste collection and transportation area belonging to Lot 1, in the munipality of Belém/PA.

To carry out a complete characterization of urban solid waste (MSW) in the 21 neighborhoods served by the Company TERRAPLENA LTDA, a comprehensive and viable work plan was drawn up. This plan involved several steps, including quantitative and qualitative analyzes of waste. Initially, neighborhood visits were carried out to understand the waste flow and determine the volume to be collected. Then, samples were taken of the newly deposited waste, followed by transportation of these samples to specific locations where the gravimetric composition was determined. Finally, the samples were prepared for detailed laboratory analysis. This comprehensive plan allowed for an in-depth understanding of the nature and quantity of waste in each neighborhood, providing a solid foundation for future waste management strategies. During the visits carried out on site, considering both the number of itineraries (37 in total) and their geographical extension, together with the economic similarity of some neighborhoods, it was suggested to group the itineraries into nine distinct sectors (as illustrated in Figure 3). This categorization was carried out based on socioeconomic characteristics and geographic proximity to the neighborhoods, aiming to facilitate the carrying out of targeted campaigns. The main objective of these campaigns was to carry out a comprehensive gravimetric analysis of the entire collection and transport area managed by the aforementioned company in the municipality of Belém.

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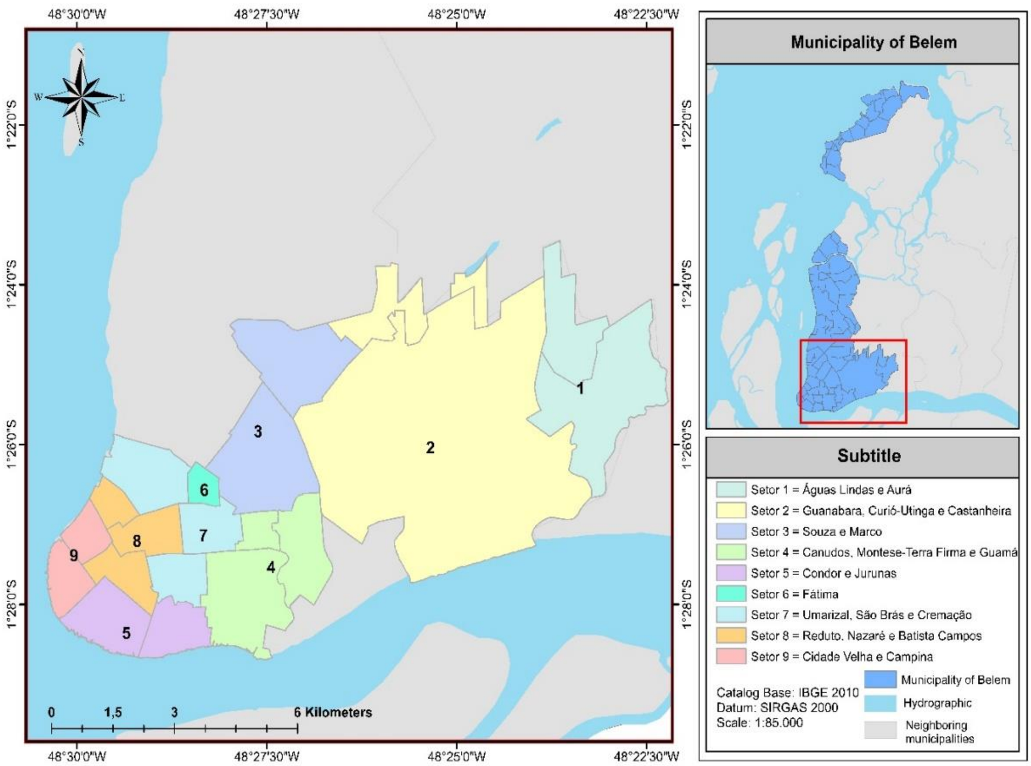

Figure 3. Map of sectorization of the neighborhoods of Belém/PA belonging to Lot 1.

The neighborhoods were categorized according to family income, following a methodology adapted from the IBGE guidelines [30]. This methodology classifies the Brazilian population into five classes (A, B, C, D and E), using the minimum wage as a reference point. It is important to highlight that, in the municipality of Belém, there are no neighborhoods classified in classes A and B. Most neighborhoods, around 61%, are classified in class E, Assuncao et al., [27]. The neighborhoods were categorized according to family income, following a methodology adapted from the IBGE guidelines [30]. This methodology classifies the Brazilian population into five classes (A, B, C, D and E), using the minimum wage as a reference point. It is important to highlight that, in the municipality of Belém, there are no neighborhoods classified in classes A and B. The vast majority of neighborhoods, around 61%, are classified in class E, Assuncao et al., [27].

Table 1. Determination of the grouping of sectors into regions.

Region	Class	Sectors	Neighborhoods
1	E	1, 2 e 3	Aurá, Águas Lindas, Curió-Utinga, Guanabara, Castanheira, Souza e Marco.
2	D	4, 5 e 6	Canudos, Terra Firme, Guamá, Condor, Jurunas e Fátima.
3	C	7, 8 e 9	Umarizal, São Brás, Cremação, Batista Campos, Nazaré, Reduto, Campina e Cidade Velha.

2.3. Calculation of the Mass Required for the Gravimetric Composition

To calculate the volume of samples necessary to determine the gravimetric composition of the total mass of waste collected, we used STATIDISK 13.0 software, a tool widely used in statistical analyses, including sample size calculations in experiments. We consider the capacity of the collection truck as essential parameters, fundamental for understanding the variability and characteristics of the population. We adopted a volume of 15 m^3 as the population size for each route, with a significance level of 5% in statistical tests and a confidence level of 95%, indicating the desired reliability in the results. Additionally, we set a margin of error of 10%. Based on these parameters, the simulation resulted in an approximate sample mass of 100 kg [31]. This calculated sample mass

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is the representative quantity required to perform an accurate and reliable analysis of the gravimetric composition of the waste. The choice of significance level, confidence level and margin of error reflects the concern with the statistical validity of the results, as highlighted by Assunção et al. [27] and Pereira et al. [28].

2.4. Gravimetric Collection and Composition

Solid waste collection was carried out from November 2021 to May 2022, the collection adopted was door-to-door, ensuring that the waste was collected in conditions similar to those in its generation, preserving its original composition, including the degree of humidity. After collection, the waste was sent to a space at the Federal University of Pará, where gravimetric characterization was carried out. As soon as the material arrived, the characterization process began immediately. For collections carried out at night, gravimetric characterization was conducted the following morning. The collections were carried out according to the schedule of TERRAPLENA's internal routes, taking place on Mondays, Wednesdays and Fridays, during the morning. The sampling process followed the guidelines established by the ABNT standard for Solid Waste Sampling [32].

2.5. Pre-Treatment of Samples (Drying, Crushing and Sieving Content)

The organic matter, composed of a mixture of carbohydrates, lipids, proteins and fibers, was selected based on the gravimetric composition of urban solid waste. With a moisture content of 60% due to inadequate disposal, which resulted in poorly packaged waste exposed to the environment, this organic matter was subjected to pre-treatment that included drying, crushing and sieving.

After pre-treatment, the material was placed in a cooling system (freezer) to avoid physical-chemical and microbiological degradation. The drying process was carried out in a thermal oven with air recirculation and analog temperature control, model De Leo Ltda, at 105ºC for a period of 24 hours. Of the 100 kg collected, 30 kg of wet organic matter were separated, which were subjected to the drying process, in which the moisture content was calculated. After the thermal drying process, 10 kg of dry organic matter was crushed using a TRAPP TRF 600 model knife mill, in the Materials Laboratory of the Faculty of Chemical Engineering USIMAT/UFPA. During this process, a 0.8 mm sieve was used for part of the material and a 5 mm sieve for the rest. After crushing, the material was weighed on a scale model WELMY CLASS 3 W200/S, with a maximum capacity of 200 kg and a minimum of 1 kg. The sieving of the crushed MSW was carried out using a PRODUTEST Telastem sieve system, with Mesh openings #14, #28, #35 and #48, in the Materials Laboratory of the Faculty of Chemical Engineering USIMAT/UFPA. Then, the pre-treated organic matter was placed in a freezer at 0°C, being used as raw material (Figure 4) for laboratory analysis.

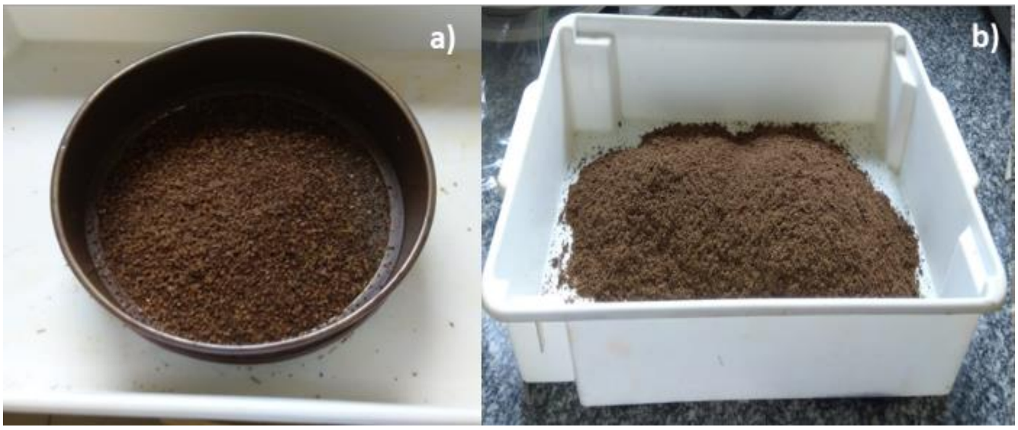

Figure 4. Pre-treated organic matter used as raw material for laboratory analyzes. **Legend*:** (a)Organic matter after crushing; (b) organic matter after sieving.

2.5.1. Laboratory Determinations

Leaching and Solubilization Tests

The leaching tests were carried out according to the methodology described by NBR 10.005 [33], following the procedures for waste with a solid content of 100%. For MSW solubilization tests, the methodology described by NBR 10.006 [34] was used, which establishes the requirements for obtaining the solubilized extract from solid waste, differentiating Class II A and II B waste according to NBR 10.004 [32]. Samples from the 9 sectors were regrouped for metal analysis purposes, totaling 3 samples. The samples from neighborhoods classified as E were called Region 1, those from class D neighborhoods were called Region 2, and those from class C neighborhoods were called Region 3.

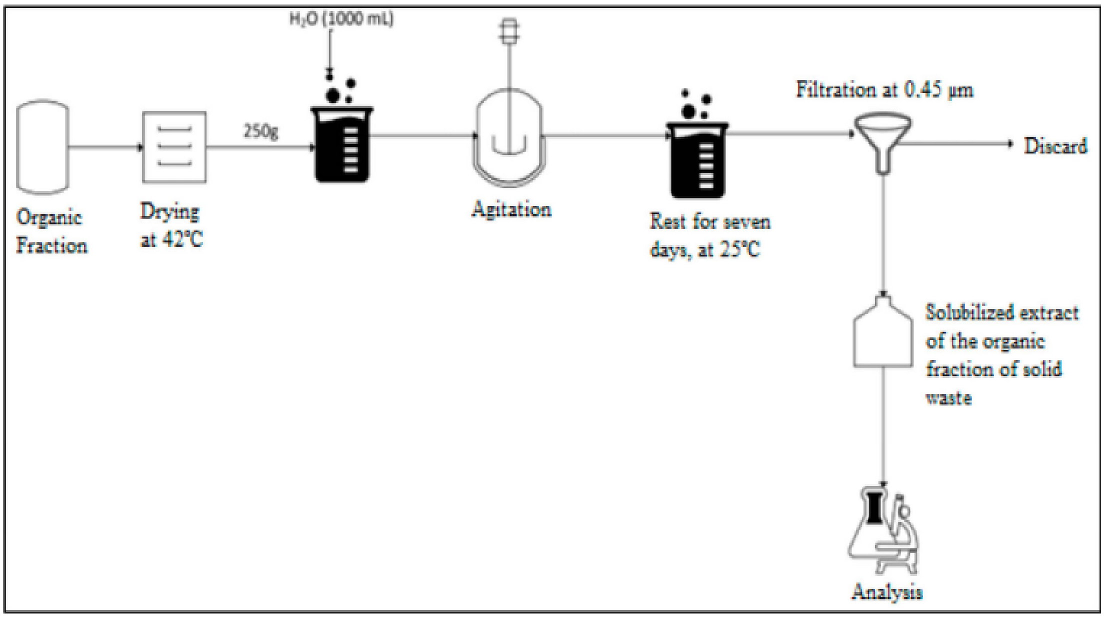

Figure 4. Tests carried out to obtain the solubilized extract.

Determination of Heavy Metals (Sample Preparation and Digestion)

The samples were collected in 100 ml polyethylene containers for the quantification of heavy metals and were preserved with 2 ml of high purity concentrated HNO3. The leachate was kept at 4 °C to minimize chemical and biological changes [35]. Subsequently, the samples were sent for metal analysis. The analyzes were carried out in the Laboratory of the Center for Agricultural and Environmental Technology accredited in the ISO 17025 System. All parameters were analyzed according to the procedure prescribed in Standard Methods, 23rd edition and the United States Environmental Protection Agency [35] . The attached Supplementary Table S2 presents a summary of the reference methods, units, measurement uncertainty and limit of quantification for the metals analyzed. Heavy metal concentrations in the leachate were determined by inductively coupled plasma mass spectrometry (ICP-OES, 5800 VDV, Agilent, USA) after digestion using a mixture of HCl and HNO3 in a microwave digester. The results of the analyzes presented were validated with the analysis of Certified Reference Standards (CRM) for homogeneous groups and blank analysis of methods, according to Supplementary Table S3 attached. For each Standard analyzed, recovery was calculated and compared with the criteria established by DOQ-CGCRE-008 (INMETRO, 2020). The analysis methods and method and operational conditions were the same as those used for the samples.

2.5.2. Method and Statistical Analysis

For data processing, metal results below the quantification limit of the proposed methods were excluded from the statistical analysis. The results of metals present in samples composed of leachate

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from regions R1, R2 and R3 were compared using bar graphs, aiming to visualize the concentrations of the main contaminants present in each sample. This approach facilitates the identification of patterns and discrepancies, in addition to allowing comparison with current standards for the levels of these metals in the environment. Organizing the results in Microsoft® Office Excel® 2010 spreadsheets, and subsequently analyzed in the statistical program R (version R_2.12), we calculated the Pearson correlation coefficient (r), also known as product correlation coefficient. moment, using the function = CORREL [1–8]. This coefficient measures the degree of linear correlation between two quantitative variables. A coefficient close to 1 indicates a strong direct correlation, while a coefficient close to -1 indicates a strong inverse correlation. Although the correlation coefficient is useful for examining the relationship between metals, its application is limited when dealing with multiple parameters simultaneously. This method was used to explore the relationships between different chemical elements, where strong correlations (positive or negative) can indicate elements with similar or opposite behaviors in different regions. To overcome this limitation, Principal Component Analysis (PCA) can be used to evaluate the relationship between several variables with minimal data loss. Additionally, a correlation matrix based on the Pearson method was used. PCA was applied to select the chemical elements (pollution indicators) that best characterize the properties of the leachate from the selected regions. This method allowed calculating the correlation between each of the metals, and graphs were generated to facilitate the visualization of the relationships between the variables [36–40].

3. Results

3.1. Analysis of the Leachate Extract of the Organic Fraction

The physicochemical results obtained for the leached extract of the organic fraction for each region (R1, R2 and R3) are illustrated in Table 2, in which it was possible to verify Aluminum values between 5.198 and 1.775 mg/L, values for Barium 1, 33 and 0.44 mg/L, Boron in values between 1.16 and 1.30 mg/L, values for Lead between 0.03 and 0.05 mg/L, Copper between 0.22 and 0.35 mg/L L, Chromium in values between 0.055 and 0.075 mg/L, values for Iron between 28.5 and 160.4 mg/L, Manganese in values between 0.265 and 1.910 mg/L, Nickel between 0.06 and 0.15 mg/L L, Sodium values varied between 748 and 1047.8 mg/L, for Zinc the values were between 1.30 and 2.85 mg/L, for Calcium the value was 82.9 mg/L and finally the values found for Magnesium were between 39.83 and 61.92 mg/L. For the other elements that were not mentioned, the detected values were below the limit of quantification.

Table 2. Heavy metal concentrations in leachate extract samples in regions 1, 2 and 3.

Heavy metals (mg/L)	Regions		
	R1	R2	R3
Al	5,198	3,007	1,775
As	0,002	0,00875	<0,002*
Ba	1,33	0,44	0,95
B	1,19	1,30	1,16
Pb	0,05	0,03	<0,002*
Cu	0,35	0,22	0,23
Cr	0,060	0,075	0,055
Fe	32,2	160,4	28,5
Mn	0,265	1,910	1,205
Ni	0,06	0,14	0,15
Na	748	811,3	1047,8
Zn	2,85	1,30	1,55
Ca	82,9	<0,02*	<0,02*
Mg	39,83	56,82	61,92

Subtitle: *: Results below the method's quantification limit were not used in statistical analyses.

Figure 5 illustrates the results of the concentrations of some metals, such as boron (B), barium (Ba), calcium (Ca), magnesium (Mg) and sodium (Na), found in the three regions studied.

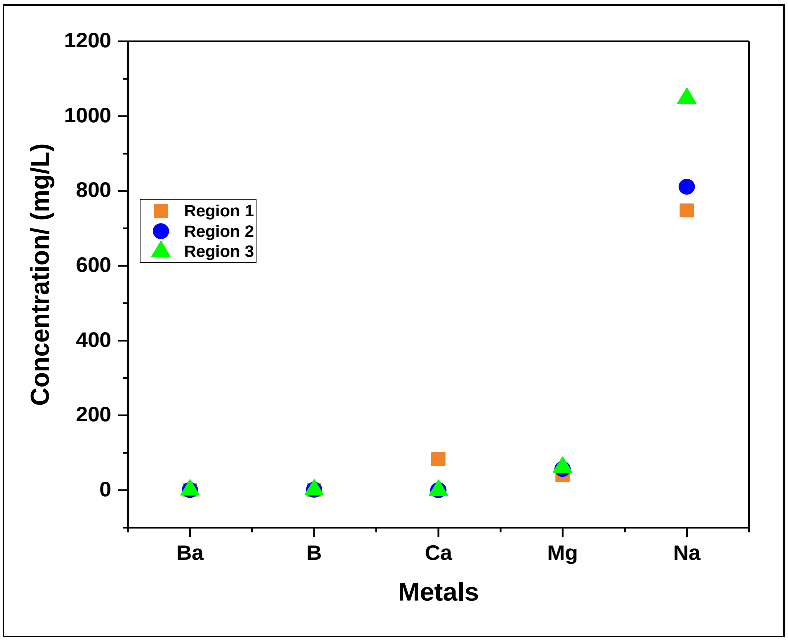

Figure 5. B, Ba, Ca, Mg and Na results for the regions.

The presence of boron and barium in solid waste leachate is mainly attributed to the use of consumer products that contain them in their formulations. For example, boron is found in products such as detergents, cleaning products, cosmetics and pesticides. When these products are discarded in landfills, the boron can dissolve in the water present in the waste and become part of the leachate. Likewise, barium is present in products such as alkaline batteries and certain types of paint. Disposal of these products in landfills can result in the release of barium into the leachate as they decompose [41,42].

These chemical elements, present in commonly used products, can represent an environmental challenge when improperly disposed of, contributing to the contamination of the leachate and, consequently, the environment. Therefore, it is important to consider waste management strategies that minimize the impact of these elements on the waste cycle [42].

Calcium may be present in solid waste leachate from several sources. For example, materials such as cardboard, paper and certain types of plastics may contain calcium as an additive. When these products are disposed of in landfills, calcium can be released into the leachate during the decomposition process. Additionally, calcium is present in food and organic waste, this essential mineral is found in many foods. When these wastes are landfilled, the calcium in them can be leached as they decompose [41]. The presence of this element requires adequate attention, as it can have environmental and health implications. Therefore, it is important to consider effective waste management strategies to deal with calcium and other contaminants present in leachate.

Food and other organic residues present in solid waste leachate naturally contain magnesium, and include a variety of materials of plant and animal origin. Magnesium is an essential mineral found in many foods and plants, and is present in different forms in organic waste. Here are some examples of organic waste that may contain magnesium: fresh fruits and vegetables are natural sources of magnesium; when the remains of these foods are discarded, they contribute to the presence of magnesium in organic waste; Fruit and vegetable peels, as well as food scraps, contain magnesium. These are often discarded as organic waste and can be composted to take advantage of their nutrients, including magnesium, leaves, twigs, grass clippings and other garden scraps contain magnesium, as plants absorb this mineral from the soil during their growth [42] .

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The maximum permitted values (VMP) for boron, barium and sodium are, respectively, 0.5 mg/L, 0.7 mg/L and 200 mg/L for human consumption, as established in Resolution 396/2008 [43]. Results for this research show that boron concentrations exceeded VMP in all three regions. Barium exceeded the limit in the leachate from regions R1 and R3, while sodium also exceeded the value allowed by the resolution in all regions. Although the predominant concentrations of heavy metals in this study are above the typical values established for groundwater in the Brazilian standard, they still do not violate release standards.

In the study conducted by Krugel [41], several metals were identified in the leachate from the waste treatment plant in Curitiba (PR), including barium, boron and manganese with concentrations of 10.87, 0.67 and 3.65 mg. /L, respectively. Only boron and manganese are above the maximum limit permitted according to CONAMA Resolution No. 430/2011[44]. According to FAO [45], barium is considered toxic to living beings due to the possibility of acute or chronic intoxication. As for boron, its classification is complex: it is considered possibly essential and its biological functions are still being studied by the scientific community, as mentioned by [45,46]

Compared to studies by Samadder et al. [47] who investigated the evaluation of physicochemical parameters and the detection of toxic metals in the soil and groundwater around solid waste disposal areas in a city in India. The results revealed considerable variations in calcium concentration. The maximum calcium concentration was 933 mg/kg, while the minimum was 143 mg/kg. As for magnesium, concentrations measured in the soil were remarkably high, with minimums and maximums of 292 mg/kg and 948 mg/kg, respectively. Around 60% of the soil samples showed magnesium concentrations above 500 mg/kg. In contrast, in this study, the values obtained for calcium (Ca) and magnesium (Mg) were lower.

Edokpayi et al. [48] conducted research on the levels of some main metals (Na, Mg and Ca) present in leachate from the Thohoyandou landfill, Limpopo province, South Africa. The results indicated that these levels did not exceed the limits considered safe for dispose of in water bodies. However, there is concern about the continued migration of salt-rich leachate to surface and underground water sources, which could increase salinity levels in river and underground aquifers.

Figure 6 presents the results of the concentrations of various metals, including aluminum (Al), arsenic (As) and lead (Pb), detected in the three regions analyzed. Discarded products such as aluminum cans, aluminum foil and food packaging contribute to the natural presence of aluminum in solid waste leachate.

Research indicates that the concentration of arsenic in leachate varies widely depending on the composition of the waste, landfill conditions and decomposition processes. Continuous monitoring and detailed studies are essential to better understand the dynamics of arsenic in leachate and develop effective mitigation strategies. This heavy metal can be found in leachate due to the decomposition of various types of waste that contain arsenic compounds, such as some chemicals such as pesticides and herbicides that contain arsenic compounds. Lead is one of the heavy metals frequently found in leachate from landfills and municipal solid waste. Its presence in leachate is a significant concern due to its environmental impacts and public health risks. This metal can be found in several forms, including lead-acid batteries, lead-based paint residues, especially in construction and demolition materials, and electronic components that contain lead in solders and circuits.

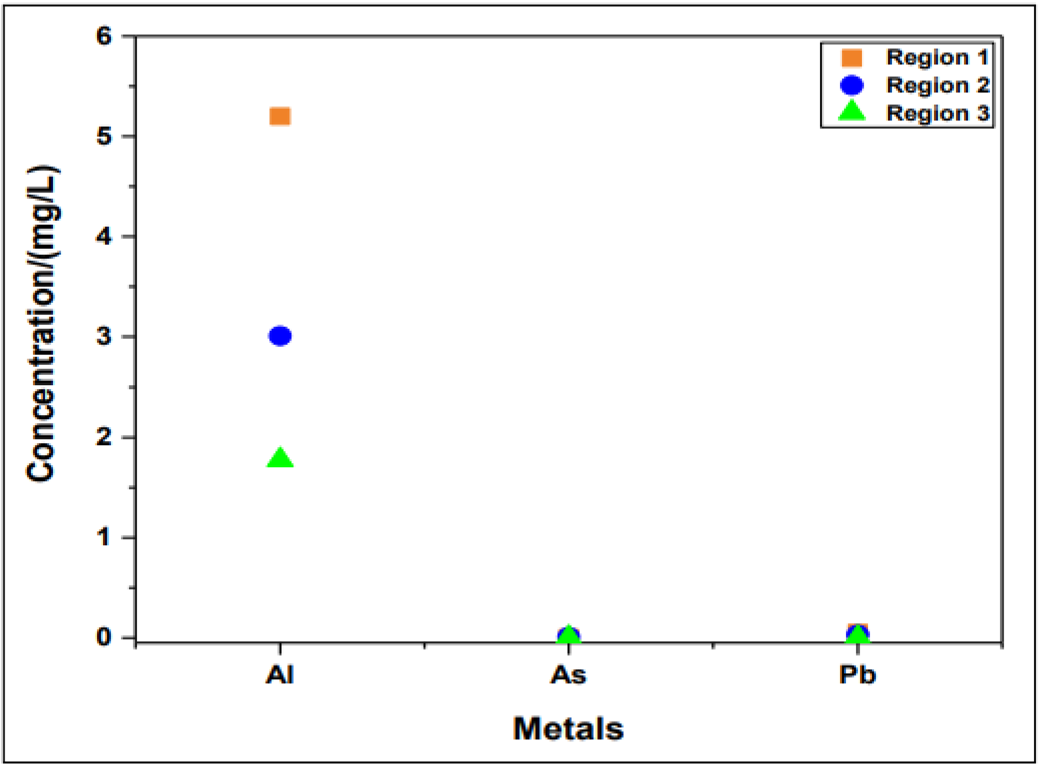

Figure 6. Al, As and Pb results for the regions.

Lead concentrations exceeded 0.2 mg/L in regions R1 and R2 for groundwater, however, for release purposes it meets legislation [44–49]. Various human activities, such as the disposal of lead-containing paint waste, lead batteries, and piping, are related to higher concentrations of lead in leachate [50]. For dissolved aluminum, all regions exhibit concentrations above 0.2 mg/L for groundwater [43].

Lange et al [26] investigated the technical feasibility of treating landfill leachate through an advanced oxidative process (AOP) using Fenton's reagent. These experiments were conducted at the Belo Horizonte Landfill. The results indicated that the levels of metals such as Al (0.22 mg/L) and As (0.11 mg/L) for the raw leachate were lower compared to those found in the current study.

Hussain et al. [51] conducted a study to assess the environmental impacts of leachate produced by municipal solid waste landfills on groundwater in India. The physicochemical properties and the presence of metals in the leachate were analyzed. Additionally, leach levels from these landfills were compared to standards established by the Environmental Protection Agency in 1986 in order to assess their compliance with environmental regulations. It was observed that the concentration of lead and arsenic were respectively 0.6 mg/L and 1.86 mg/L, the concentration of these metals exceeds the typical values laid down in Indian norms for the disposal of wastewater in surface water bodies. It is important to highlight that heavy metals of concern to human health, such as lead, cadmium, mercury and arsenic, are present in all leachate samples from the respective study [52].

In their 2015 study, Riguetti [53] and collaborators investigated the concentrations of several metals, including lead, in leachate samples collected at the Dourados landfill, Mato Grosso do Sul/Brazil. Samples were collected from two reservoirs, one subjected to aerobic treatment and the other to anaerobic treatment. Over the course of four collections, carried out between 2012 and 2013, covering both dry and rainy periods, data for analysis were obtained.

In most analyses, it was found that the lead concentration was higher during the first and second collections, both for the lagoon with aerobic treatment and for the one with anaerobic treatment. These results suggest that the increase in the volume of water in the landfill had an impact on the availability and mobility of these elements. The highest values were recorded in the second collection:

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0.587 mg/L in the anaerobic lagoon and 1.280 mg/L in the aerobic lagoon. In this context, the dilution of the leachate did not result in a reduction in concentration, suggesting that the availability and mobility of lead were higher than those of manganese and zinc.

Similar results were observed by Oliveira and Santana [54], who reported an increase in the concentration of lead in water contaminated by landfills during periods of rain, indicating a greater mobility of this element during this period.

Figure 7 presents the results of the concentrations of some metals, such as chromium (Cr), copper (Cu), iron (Fe), manganese (Mn) and zinc (Zn), identified in the three regions analyzed. Chromium is also one of the metals present in solid waste leachate, some sources may contain chromium as cleaning products contain chromium compounds, another example is leather materials treated with chromium compounds such as shoes and clothing.

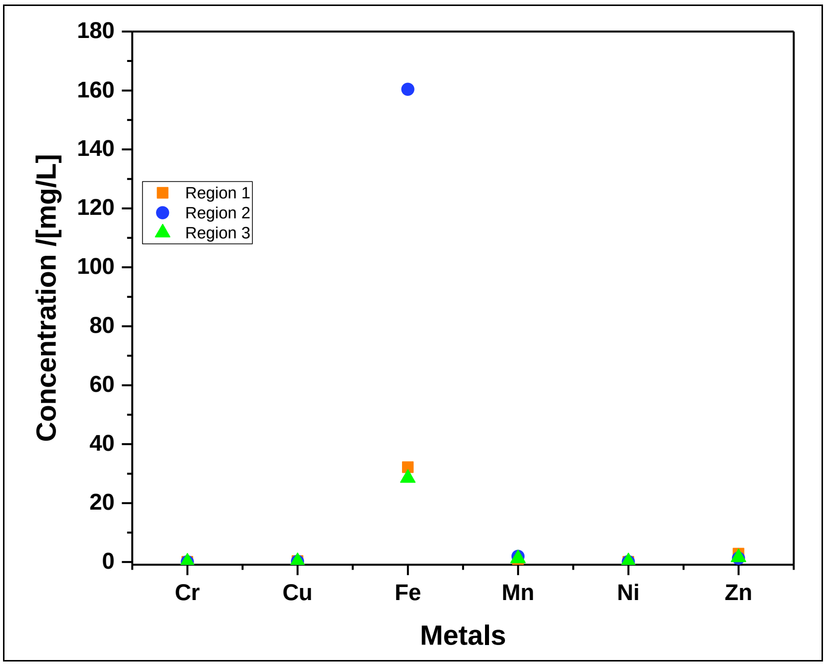

Figure 7. Results of Cr, Cu, Fe, Mn, Ni and Zn for the regions.

Regarding copper, its presence in leachate can be attributed to different sources, such as electrical cables, circuit boards and other electronic components can contain copper, copper tubes used in plumbing systems and pipe connections can contribute to the presence of copper in the leachate. The presence of iron in municipal solid waste leachate is common and can occur due to the decomposition of a variety of iron-containing materials, as well as natural and industrial processes. Iron is one of the most abundant elements in the Earth's crust and is present in many everyday products and materials.

The presence of manganese in municipal solid waste (MSW) leachate can be attributed to several consumer products, such as batteries, paints and coatings, which may contain manganese in their formulations. Nickel is another metal that can be present in MSW leachate, although generally in lower concentrations compared to other metals such as iron, copper or manganese. Its sources include discarded electronic products, such as cell phones, computers and household appliances, which may contain nickel in their circuit boards and other parts. Additionally, some rechargeable batteries, such as lithium-ion batteries used in electronic devices, may contain nickel.

Zinc can also be present in MSW leachate, being found in electronic products, paints, coatings and in some batteries, such as lithium-ion batteries, which may contain zinc. These metals, when present in leachate, pose environmental concerns due to their toxicity and potential impacts on public health and the environment. Therefore, it is crucial to implement appropriate treatment measures to minimize the risks associated with the presence of these metals and protect water quality and the ecosystem.

It is evident that for this research the dissolved iron content exceeded the maximum permissible limits for groundwater (0.3 mg/L) according to the standard and for the release standard (15 mg/L) in all three regions [43,44,49]. According to Lindamulla et al. [55], in their research on the quality of leachate from waste disposal sites in tropical climate zones, the presence of iron (Fe) is generally attributed to the disposal of metallic waste and tin-based products, along with other heavy metals. The presence of iron in municipal solid waste leachate is common and can be attributed to several factors, such as the presence of discarded metallic materials, such as metal packaging, kitchen utensils and metal parts, which are discarded daily as municipal solid waste. Corrosion of these materials over time can release iron into the leachate [55].

Manganese concentrations were observed to be high in the R2 and R3 regions. Chromium exceeded the Maximum Allowable Value (MPV) established for groundwater in all three regions, while zinc and nickel presented low concentrations, not representing a significant risk of contamination to groundwater or surface water.

Hussein et al [56] focused their study on evaluating heavy metal contamination in leachate and surface soils in different landfills in Malaysia. Elevated levels of Cr and As were observed in raw leachate in the landfills of Sungai Udang and Ladang Tanah Merah cities, which were reduced to the standard limit after treatment. Concentrations of cadmium (Cd), copper (Cu), nickel (Ni), zinc (Zn), manganese (Mn), iron (Fe) and lead (Pb) were also observed in the leachate from both landfills, however they were within stipulated standards. The increase in arsenic (As) and chromium (Cr) levels is probably related to agricultural activities and industrial processes.

Additionally, wood waste from construction, demolition, utility poles, highway barriers, landscape structures, and wood products factories can also contribute to high metal contents, especially As, Cr, and Cu, due to leaching of these metals. from wood generally treated with preservatives containing chromated copper arsenate, the most serious risk occurs when the treated wood is burned. However, both parameters are often neglected in most pollution tracking, monitoring and control programs, despite the widespread environmental threat that these pollutants pose [57].

Fu et al. [58] investigated heavy metal pollution in the surface sediments of the Jialu River in China. The average values of heavy metals in the sediments were as follows: Cr (60.80 mg/kg), Cu (39.22 mg/kg), Ni (42.44 mg/kg) and Zn (107.58 mg/kg). Comparing these concentration levels with other basins in China, it was observed that the Zn level is 1.5 times higher in the Jialu River than in the Huaihe River Basin, also located in China. However, the levels of other heavy metals are similar between the Jialu River and the Huaihe River basin. Compared to rivers and lakes in other regions of China and other countries, metal levels in the Jialu River are considerably lower. Specifically, the levels of Cu, Hg, Ni, Pb and Zn in the Jialu River are at least three times lower than in other parts of the world.

Alves et al. [59] sought to quantify the levels of several metals, including Cd, Cr, Cu, Mn, Ni and Zn, in samples of surface water and sediments from the Rio Pardo, located in Brazil. Additionally, the study evaluated the risks to human health associated with exposure to these metals through oral ingestion and dermal absorption. The results revealed that the average levels of manganese and zinc in the water were the highest, regardless of the season, in Rio Pardo. According to authors Antweiler et al., 2012, manganese can be found in water supplies due to natural processes such as watershed erosion and the dissolution of manganese-containing minerals near the sediment-water interface. On the other hand, higher zinc levels in regions with large sugarcane production may be the result of the widespread use of fertilizers. Additionally, the burning of agricultural residues and the use of fungicides containing zinc can also be contributing factors to the presence of this metal [60].

In the study conducted by Kujara et al. [61], the levels of heavy metals in non-recirculated leachate collected at the Municipal Waste Deposit in the north of Porto Alegre, where waste of urban origin is deposited, were analyzed. The results revealed that the maximum and minimum levels of metals found were as follows: 0.01 to 0.015 mg/L for Cu; 0.01 to 0.3 mg/L for Cr and 0.07 to 20.5 mg/L for Mn. The concentrations of heavy metals reported in this study are similar to those detected in the present work.

It is important to highlight that the Mn concentration in the study by Kujara et al. [61] exceeded the maximum limit allowed by Resolution no. 357/2005 [49], values that were even higher than those observed in the present study, where only some samples presented Mn concentrations above the maximum limit allowed by the aforementioned resolution. The presence of manganese in municipal solid waste can occur due to several sources, including household items such as batteries, electronic products, paints and plastics, which may contain manganese in their compositions. When these products are discarded as municipal solid waste, manganese can be released into the environment.

In the study conducted by Oliveira & Jucá [62], the maximum and minimum concentrations of metals were recorded in leachate samples collected at the Muribeca Solid Waste Landfill, in the metropolitan region of Recife, Pernambuco. The results revealed a range of 0.2 to 2.9 mg/L for Cu, < 0.01 to 3.5 mg/L for Cr and < 0.01 to 35 mg/L for Mn. When comparing these data with those obtained in the present research, it is noted that the leachate generated in the collection routes presents lower maximum concentrations for the metals Cu, Cr, Mn, Ni and Zn while the maximum concentration for the metal Fe is higher.

Celere et al. [63] conducted research to examine the presence of metals in leachate from the Ribeirão Preto landfill, SP. They observed that, although the average manganese values were within the limits allowed by legislation, in 8.3% of the samples analyzed values were identified that exceeded the maximum allowed. According to the author, this metal is currently used in the manufacture of steel, batteries, matches and porcelain. The detection of high levels of this metal in the study may suggest the presence of these materials in the solid waste being deposited in the Dourado landfill.

Cort et al. [64] conducted an assessment of the levels of cadmium, lead, copper, chromium, manganese, mercury and zinc in raw and treated leachate from landfills in Francisco Beltrão and Nova Esperança do Sudoeste, PR. The average values found for the metals chromium, cadmium, copper, manganese and mercury in the leachate samples were within the maximum limits established by CONAMA [49]. However, the researchers noted that both manganese and lead presented values above those allowed only in the raw leachate from the two landfills evaluated. This finding contrasts with the results of this study, where high levels of these metals were observed both in the leachate during the initial phase of treatment (anaerobic lagoon I) and in the leachate collected in the last phase of treatment (maturation), during collections carried out in the months of October 2012 and February 2013.

Cintra [65] evaluated the influence of recirculating raw leachate and inoculated leachate (pre-treated in a UASB reactor) on the anaerobic digestion of municipal solid waste (MSW), and found values for Ni, Cr, Cu, Mn and Zn of 0.022, 0.022, 0.002, 0.026, 0.199, 0.134 and 0.178 mg/L, respectively.

3.1.2. Verification of Compliance with Legal Sanitary and Environmental Standards

The comparison of the results of metal analyzes of the solubilized extract of the organic fraction of collected urban solid waste was carried out considering the guidelines established by CONAMA Resolution nº 357/2005 [49], CONAMA Resolution nº 430/2011 [44] and CONAMA Resolution nº 396 /2008 [43]. Reference values were evaluated for different metals, such as aluminum (Al), arsenic (As), barium (Ba), boron (B), lead (Pb), copper (Cu), chromium (Cr), iron (Fe), manganese (Mn), nickel (Ni), zinc (Zn) and magnesium (Mg). The results obtained in the research for Region 1 were then compared with the limits established by these sanitary and environmental standards, as presented in Table 3.

CONAMA Resolution No. 396/2008 [43] deals with the classification, environmental guidelines and measures for the classification, prevention and control of groundwater pollution. According to this resolution, limits are established for the preponderant uses of water, such as human consumption, for the following inorganic parameters: Al (0.2 mg/L), As (0.01 mg/L), Ba (0. 7 mg/L), B (0.5 mg/L), Pb (0.01 mg/L), Cu (2 mg/L), Fe (0.3 mg/L), Mn (0.1 mg /L), Ni (0.02 mg/L), Na (200 mg/L) and Zn (5 mg/L).

CONAMA Resolution No. 430/2011 [44] established the standards for releasing effluents from polluting sources into receiving bodies. This standard defines limits for several parameters and their

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limits, including AS (0.5 mg/L), Ba (5.0 mg/L), B (5.0 mg/L), Pb (0.5 mg/L), Cu (1.0 mg/L), Cr (1.0 mg/L), Fe (15 mg/L), Mn (1.0 mg/L), Ni (2.0 mg/L), Zn (5.0 mg/L) and Mg (1.0 mg/L).

CONAMA Resolution No. 357/2005 [49] establishes water quality standards, determining the criteria for classification and, therefore, guiding the uses of the water resource at the sampling site. For this research, freshwater bodies classified as class II were considered, as defined in the resolution. Class II is adopted when the water course is unknown, following the guidelines of the resolution, which establishes a limit range for the release of effluents and parameters for inorganics for the following elements Al (0.1 mg/L), As (0.01 mg/L), Ba (0.7 mg/L), B (0.5 mg/L), Pb (0.01 mg/L), Cu (0.009 mg/L), Cr (0. 05 mg/L), Fe (0.3 mg/L), Mn (0.1 mg/L), Ni (0.025 mg/L) and Zn (0.18 mg/L).

After analysis and verification of compliance with Brazilian health and environmental legislation, it was found that the inorganic parameters (metals) investigated, such as iron, manganese and magnesium, did not comply with the standards established by CONAMA Resolution No. 430/2011 [44]. In CONAMA Resolution 375/2005 [49] , no inorganic parameter (metal) met the legal requirements. According to CONAMA Resolution 396/2008 [43], the metals aluminum, barium, boron, lead, iron, manganese, nickel and sodium exceeded the limits established by legislation for region 1 (Table 11). Furthermore, some parameters were not addressed in the mentioned laws, and the limit values are defined in only one legal standard.

Table 3. Assessment of compliance of Region 1 results with legal sanitary and environmental standards.

Metal Analysis (mg/L) (Region 1) CONAMA			
Metals	430/2011	357/2005	396/2008
Al	-	no	no
As	yes	no	yes
Ba	yes	no	no
B	yes	no	no
Pb	yes	no	no
Cu	yes	no	yes
Cr	yes	no	-
Fe	no	no	no
Mn	no	no	no
Ni	yes	no	no
Na	-	-	no
Zn	yes	no	yes
Mg	no	-	-

The results obtained for the solubilized extract of residues from region 2 revealed non-compliance with CONAMA Resolution nº 430/2011 [44] for the parameters of iron, manganese, and magnesium. In relation to CONAMA Resolution 375/2005 [49], only arsenic and barium were complying. Finally, according to CONAMA legislation 396/2008 [43], the metals arsenic, barium, copper, and zinc were within the standards established for region 2. The detailed results can be consulted in Table 4.

Table 4. Avaliação da conformidade dos resultados da Região 2 com as normas legais sanitárias e ambientais.

Metal Analysis (mg/L) (Region 2) CONAMA			
Metals	430/2011	357/2005	396/2008
Al	-	no	no
As	yes	yes	yes
Ba	yes	yes	yes
B	yes	no	no
Pb	yes	no	no
Cu	yes	no	yes
Cr	yes	no	-
Fe	no	no	no

Mn	no	no	no
Ni	yes	no	no
Na	-	-	no
Zn	yes	no	yes
Ca	-	-	-
Mg	no	-	-

During the analysis of the solubilized extract of organic residues from region 3, non-compliance was found in the inorganic parameters (metals) for iron, manganese, and magnesium, as established by CONAMA Resolution 430/2011 [44]. In relation to CONAMA Resolution 357/2005 [49], none of the metals analyzed were complying. Finally, according to CONAMA 396/2008 [43], only the metals copper and zinc were within the established limits. Complete details of the analysis can be found in Table 5.

Table 5. Assessment of compliance of Region 3 results with legal sanitary and environmental standards.

	Metal Analysis (mg/L) (Region 3) CONAMA		
Metais	430/2011	357/2005	396/2008
Al	-	no	no
As	-	-	-
Ba	yes	no	no
B	yes	no	no
Pb	-	-	-
Cu	no	no	yes
Cr	yes	no	-
Fe	no	no	no
Mn	no	no	no
Ni	yes	no	no
Na	-	-	no
Zn	yes	no	yes
Mg	no	-	-

3.1.3. Pearson Correlation

The correlation between the 11 leachate variables was analyzed and is represented in Figure 8.

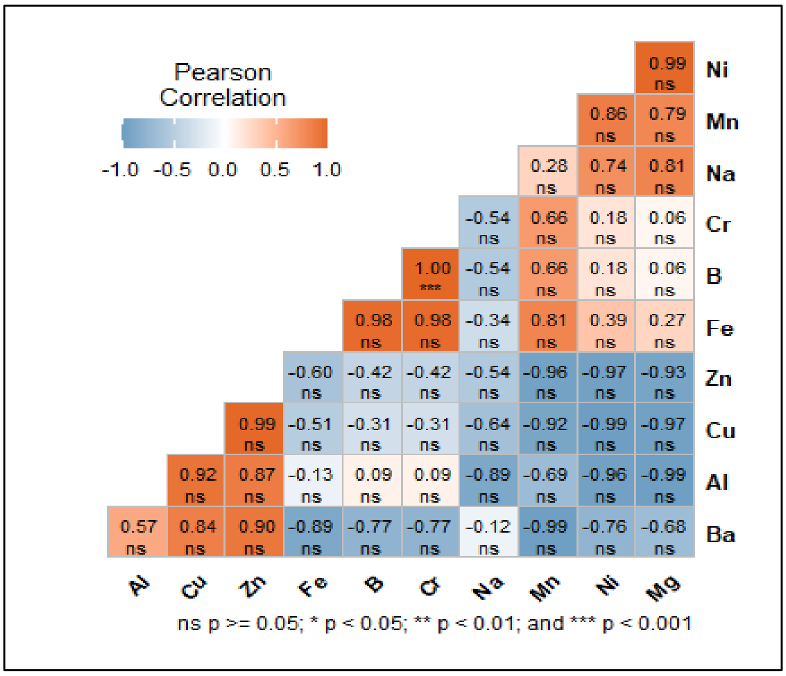

Figure 8. Correlation matrix of heavy metals in the study regions.

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The results from the selected R1, R2 and R3 regions indicate that the majority of parameters did not present a statistically significant correlation, suggesting a lack of association between several variables. However, a significant correlation was observed between chromium and boron, given that the correlation coefficient was equal to 1 (very strong) for a p-value lower than 0.001, which characterizes the correlation between the two variables. ble to be highly significant and with a probability of less than 0.1% chance that the observed correlation occurred by chance. Thus, there is sufficient evidence to state that boron and chromium concentrations are correlated.

Comparison between other elements resulted in a Pe-arson correlation coefficient above 0.9 (Cu and Al, Zn and Cu, B and Fe, Cr and Fe, and Mg and Ni) or below -0.9 (Mn and Ba, Ni and Al, Mg and Al, Mn and Cu, Ni and Cu, Mg and Cu, Mn and Zn, Ni and Zn, Mg and Zn), suggesting a strong relationship between the variables. However, a p-value greater than 0.05 indicates that these observed correlations are not statistically significant at the usual 95% confidence level, that is, there is a probability greater than 5% that the observed correlation may have occurred due to chance. The results presented in Table 6 demonstrate that the metals Aluminum (Al), Barium (Ba), Copper (Cu), Manganese (Mn), Nickel (Ni), Zinc (Zn) and Magnesium (Mg) are more adequately represented by the first main component (PC1), while the metals Boron (B), Chromium (Cr), Iron (Fe) and Sodium (Na) exhibit more expressive correlations in the second main component (PC2). The first principal component, PC1, represents 66.5% of the total variance. The first two principal components explain 100% of the variation in the data. Therefore, the PC1 and PC2 components are used to analyze the leachate parameters.

Table 5. Principal Components Correlation Matrix**.**

Variables	PC1	PC2
Al	**-0.821**	0.571
Ba	**-0.936**	-0.353
B	0.498	**0.867**
Cr	0.498	**0.867**
Cu	**-0.979**	0.205
Fe	0.674	**0.739**
Mn	**0.980**	0.199
Ni	**0.942**	-0.336
Na	0.466	**-0.885**
Zn	**-0.996**	0.090
Mg	**0.895**	-0.447

The biplot in Figure 9 shows the combined correlation between PC1 and PC2, showing that leachates from waste from regions R1, R2 and R3 have different characteristics. This suggests that the metal leaching process is characteristic for each region depending on the composition of solid waste. It is observed that the concentrations of aluminum, barium, copper and zinc increase in the direction of R1, while they decrease in the direction of R2 and R3. On the other hand, the concentrations of chromium, barium, iron and manganese increase towards R2, while the concentrations of sodium, nickel and magnesium increase towards R3.

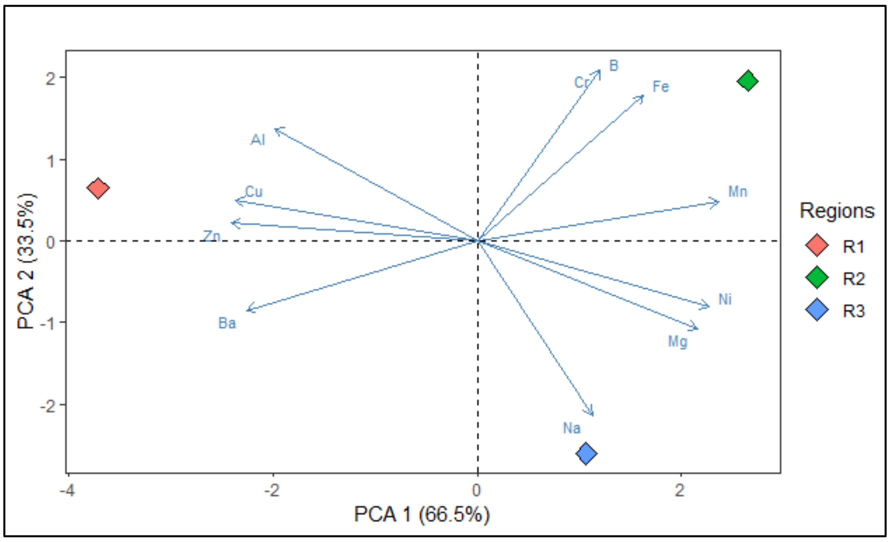

Figure 8. PCA results for the characterization of metals in leachate from the three regions.

4. Conclusions

For Region 1, the results indicated that the levels of metal contamination in the analyzed samples do not comply with legal standards. This highlights the need for environmental and sanitary interventions to ensure the quality and safety of the environment and public health in the region.

In relation to Region 2, the data showed that although some metals comply with certain legal standards, there are still significant contamination issues. Non-compliance with CONAMA Resolution No. 430/2011 for iron, manganese and magnesium, and partial compliance with CONAMA Resolution 375/2005, highlight the need for corrective actions and continuous monitoring to ensure environmental safety and health public in the region.

The results from Region 3 showed significant non-compliance with established environmental standards, especially in relation to CONAMA resolutions nº 430/2011 and 375/2005, with the exception of copper and zinc according to CONAMA 396/2008. These findings highlight the urgent need for corrective measures and rigorous monitoring to mitigate metal contamination, ensuring the protection of the environment and public health in the region.

Regarding the Pearson correlation, the analysis of leachate from the three regions showed notable differences in the concentration of several metals, suggesting variations in the composition of municipal solid waste (MSW) and potential specific sources of contamination. R1 had the highest concentration of aluminum (Al) with 5,198 mg/L, in addition to relatively high concentrations of iron (Fe) with 32.2 mg/L and sodium (Na) with 748.0 mg/L, compared to the other regions. The high concentration of Al may be associated with the excessive use of Tetra Pak packaging and metallic waste in this region. R2 has the highest concentration of iron (Fe) at 160.4 mg/L and manganese (Mn) at 1,910 mg/L, in addition to a high concentration of sodium (Na) at 811.3 mg/L. The concentration of arsenic (As) in R2, at 0.0875 mg/L, is significantly higher than in R1 and R3, suggesting possible sources of contamination from electronic components, agricultural (fertilizers) and industrial products. R3 presents a very high concentration of sodium (Na) with 1047.8 mg/L and the highest concentration of nickel (Ni) with 0.15 mg/L among the three regions. Metals such as antimony (Sb), beryllium (Be), cadmium (Cd), cobalt (Co), lead (Pb), mercury (Hg), molybdenum (Mo), platinum (Pt) and selenium (Se) have concentrations below limits of quantification in all three regions.

The notably high concentrations of iron and sodium in R2 and R3, compared to R1, indicate a significant difference in the composition of MSW in these regions, where a percentage of 55.6% organic matter and 14.3% inert waste predominates. To deepen understanding of the sources and impacts of metal concentrations in leachate, it is suggested to investigate and characterize the specific sources that contribute to high concentrations of certain elements, especially where there are significant divergences between regions. It is essential to evaluate the use and disposal of packaging, electronics and industrial products that may be contributing to high levels of metals such as Al, Fe, As and Ni. Furthermore, the environmental impacts of metal concentrations in leachate must be studied, including soil and groundwater contamination, and the public health risks associated with exposure to heavy metals should be assessed, with a special focus on arsenic (As) and nickel (Ni), known for their harmful effects on health.

Supplementary Materials: The following supporting information can be downloaded at the website of this paper posted on Preprints.org.

Author Contributions: The individual contributions of all the co-authors are provided as follows: (J.C.M.) contributed with formal analysis and writing original draft preparation, investigation and methodology, (F.P.d.C.A.) contributed formal analysis, investigation and methodology, (D.O.P.) contributed with investigation and methodology, (J.F.H.F.) contributed with formal analysis and software analysis, (J.C.C.d.S) contributed with investigation and methodology, (F.F.S.A) contributed with investigation and methodology, (A.C.P.A.) contributed with formal analysis, (methodology and geographical information system, (N.M.M.) contributed with investigation, methodology and resources, (I.W.d.S.B.) contributed with investigation and chemical analysis, (A.O.M.) contributed with investigation and methodology, (L.P.B.) contributed with chemical analysis and resources, (J.A.R.P.) with investigation, methodology and resources, (N.T.M.) contributed with supervision, conceptualization, and data curation. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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