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# Influence of the dry and rainy period on trace metal enrichments in sediments of the southern coast of the state of Pernambuco, Brazil

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**Research Article**

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# Abstract

Marine sediment contamination by trace metals is an important environmental concern due to their persistence and potential ecological impacts. This study evaluated the environmentally available concentrations of trace metals in surface sediments from six beaches along the southern coast of Pernambuco, northeastern Brazil, during the dry and rainy seasons. Sediments were characterized by granulometry, organic matter, decarbonation, FTIR, TGA, and ICP OES after partial extraction with 0.1 mol $L^{-1}$ HCl. High carbonate contents and variable organic matter were observed, while FTIR and TGA confirmed carbonate and silicate minerals associated with metal retention. Ba, Co, Cr, Fe, Mn, Ni, and Zn were detected, whereas Cd and Cu were below quantification limits and Pb was detected only at one site. In general, metal concentrations were higher during the rainy season, indicating seasonal influences on metal availability. All concentrations remained below the limits established by Brazilian and international sediment quality guidelines. Contamination and ecological risk indices classified all sampling sites as presenting low environmental risk. These results provide important baseline data for environmental monitoring and management of tropical coastal ecosystems.


# 1. Introduction

The coast of Pernambuco stretches for about 187 km, of which 156 km are sandy beaches embedded in an environmental context that includes reef systems, estuaries, and diverse coastal ecosystems (Short and Klein, 2016). This natural configuration not only sustains essential ecological processes but also forms the basis of the region's tourist appeal. However, one of the most recurrent environmental problems in coastal marine areas worldwide is trace metal pollution (Kuang et al. 2024). In this regard, Li et al. (2017) emphasizes that contamination by these elements in estuaries and coastal zones has received increasing attention, as it can cause significant impacts on human health, living resources, and marine ecosystems.

From a geological perspective, the sedimentary basins of the southern coast stand out, where the presence of sandstones, conglomerates, shales, and siltstones (Lima Filho 1998) directly influences weathering processes and the formation of sedimentary deposits. These coastal sediments result from the interaction between erosion, transport, and chemical reactions involving minerals and organic matter (Rajput and Thakur, 2016). The high surface area of the fine fraction, associated with the presence of humic substances, favors metal retention, turning deposits into important reservoirs of these elements (Chatterjee et al. 2007; Gonçalves et al. 2022). As highlighted by Clark (2001), metal concentrations in sediments function as environmental "fingerprints," reflecting both natural geochemical evolution and the impacts of human activities.

In the context of trace metals, their persistence in different chemical forms and degrees of mobility (Vicente et al. 2024) is concerning due to their toxicity, lack of biodegradability, and high bioavailability. These factors facilitate their transfer into food chains and compromise the quality of water resources and human health (Jiang et al. 2024). Although rock weathering is the main natural source, excessive

input from urban and port expansion, industrial activities, maritime transport, and the use of agricultural inputs has raised concentrations to critical levels for soils and coastal ecosystems (Paul et al. 2024; Wang et al. 2025; Cho et al. 2026).

Given the environmental relevance of these concentrations, the assessment of sediment contamination has prioritized the use of partial extractions with weak acids, aimed at identifying the exchangeable fraction (Ali et al. 2024). This fraction includes metals weakly retained by electrostatic interactions, susceptible to release through ion exchange or coprecipitation with carbonates (Tavares 2013). Due to their chemical instability, these elements exhibit greater mobility and risk of remobilization into the water column, as well as high availability for biological uptake (Reis et al. 2020). Thus, they represent a significant threat both to aquatic organisms and to human health (Linnik et al. 2023).

In this regard, studies such as that of Bevilacqua et al. (2009) employed selective extraction protocols to distinguish the distribution of metals (Cd, Pb, Cu, Cr, Ni, and Zn) between labile and non-labile fractions. The results revealed significant concentrations of heavy metals in the Pirapora reservoir, highlighting a scenario of considerable contamination with high potential for bioavailability. This finding contributes decisively to the understanding of the dynamics of metallic pollution in the Tietê River, by demonstrating that not only the presence of contaminants must be considered, but also their capacity for mobilization and interaction with aquatic organisms.

Similarly, Pereira et al. (2017) applied extraction with diluted hydrochloric acid to sediments from the São Francisco River, located in Northeastern Brazil, with the aim of assessing the capacity of benthic organisms to assimilate pollutants present in the aquatic environment. The relevance of this approach lies in the extractor's ability to release metals adsorbed onto aluminosilicates and dissolve alkaline or amphoteric oxides, thereby allowing a more accurate estimation of the fraction that effectively enters the trophic chain.

The effectiveness of acid extraction was likewise demonstrated by Cotta et al. (2006), in their investigation of the Betari River region, located within the Alto Ribeira Tourist State Park (PETAR) in São Paulo. In this study, a 0.1 mol $L^{-1}$ HCl solution was employed to isolate bioavailable metals (Al, Cd, Cr, Cu, Fe, Mn, Ni, Pb, and Zn). The choice of this method is grounded in the premise that environmental toxicity is dictated by the chemical lability of the metal, rather than solely by its total content in the matrix. Thus, the simulation of environmental conditions through partial extraction becomes a superior predictive tool for assessing the actual ecological risk in aquatic ecosystems.

Despite methodological advances, a systematic gap can be observed in the literature dedicated to the Pernambuco coast. Although there are studies focused on total decomposition, such as those by Santos et al. (2025) on sediments from the Sirinhaém River and Oliveira et al. (2026) on the Ipojuca River, scientific production specifically directed at the coastal ecosystem of Pernambuco remains scarce. This absence is particularly evident when considering the need for systematized studies in the six main sites of the region (Boa Viagem, Pina, Piedade, Enseada dos Corais, Carneiros, and Serrambi), which undermines a comprehensive understanding of trace metal contamination processes.

Moreover, few studies have employed partial extractions to estimate the metallic fraction with the greatest likelihood of transfer to the biota, a crucial aspect for assessing impacts on the food chain. After all, the total content of metals does not necessarily correspond to the fraction that is effectively bioavailable. In this sense, the use of acid extractors serves the purpose of quantifying potentially available metals, reflecting not only overall concentrations but, above all, the environmentally relevant fraction—namely, the one that truly represents a risk to ecosystems and can be absorbed by organisms (Cotta et al. 2023).

From this perspective, the present research has as its central objective the determination and quantification of trace metals in coastal sediments of Pernambuco, while also seeking to elucidate the mechanisms that favor their transport and mobility. In doing so, it aims to fill the existing gap in academic investigations focused on the region, providing support for a broader understanding of the dynamics of contaminants in coastal sedimentary environments. It is therefore emphasized that continuous monitoring is critically important, not only to identify current sources of contamination but also to anticipate future risks, particularly in light of the scarcity of systematic analyses dedicated to the interaction between metals and local aquatic ecosystems.

## 2. Experimental

# 2.1. Samples

Surface sediments were collected from beaches located on the southern coast of the state of Pernambuco, Brazil (Fig. 1), encompassing different environmental and hydrodynamic conditions. The sampling points were distributed across six locations: Pina (P1), Boa Viagem (P2), Piedade (P3), Enseada dos Corais (P4), Toquinho (P5), and Carneiros (P6). The beaches of Pina, Boa Viagem, and Piedade correspond to urbanized areas of the Recife Metropolitan Region, while Enseada dos Corais, Toquinho, and Carneiros have a greater tourist influence and a lower degree of urbanization.

The collections were carried out during two distinct seasonal periods, corresponding to the dry season (September 2022) and the rainy season (March 2023), with one sample obtained per point in each seasonal period (n = 1). In each location, sediments were manually collected in the intertidal zone using previously decontaminated plastic spatulas, scraping the 0–2 cm surface sandy layer over an approximate area of 10 × 5 cm. Part of the collected mass was subsequently shared among the different analyses performed in the study.

After collection, the sediments were lyophilized in a freeze dryer (L101, LIOBRÁS, Brazil) for approximately 24 hours, followed by homogenization, quartering, and grinding in a ball mill (8000D, MIL, Brazil), following the subsequent procedural steps: 30 s for system acclimation and 2 min and 30 s for grinding. This grinding procedure was performed twice to obtain approximately 7 grams of total sediment. After completing this phase, the samples were preserved in plastic containers and kept in a desiccator until the extraction process.

## 2.2. Characterization of the sediments

The sediment samples were subjected to physicochemical characterization through thermogravimetric analysis, evaluation of total organic matter content, molecular absorption spectrophotometry in the infrared spectrum using Fourier Transform (FTIR), and granulometric analysis.

The decarbonation of the sediments followed a standardized protocol described by Campos et al. (2017): approximately 2 g of sample were weighed and dried at 60 ºC on a balance (MG214Ai, Bel Engineering, Italy), then treated with 1 mol $L^{-1}$ HCl for at least 10 hours to remove $CaCO_3$. The reaction was tested with concentrated HCl and repeated until no bubbling occurred. Then, the samples were diluted in distilled-deionized water, centrifuged, and washed five times to eliminate acid residues, then dried again at 60 ºC and weighed.

To determine the organic matter content, the Empresa Brasileira de Pesquisa Agropecuária (EMBRAPA 2017) protocol for soils was used, but since the matrix is sediment, specific adaptations were applied for this study. Initially, the mass of the porcelain crucibles was weighed, then they were tared, and 2 g of sediment were added to each. The samples were heated in a muffle furnace (F-3000 10P – EDG, Tecnal, Brazil) at 600 ºC for 6 h. After this process, each set (crucible + sediment) was weighed again, in triplicate. The calculation was done using the following Eq. (1):

$$OM\ (\%) = \frac{(M - (T - C) * 100)}{M}$$

1

Where: M is the initial weight of the sample, C the mass of the crucible, and T the weight after heating.

For the FTIR analyses, infrared spectra were obtained using an FTIR spectrometer (IFS66, Bruker, Germany), using the Fourier transform technique. The powder samples were previously mixed with KBr in a 2:200 mg ratio and pressed into pellet form. Measurements were performed in the spectral range of 4000 to 500 $cm^{-1}$.

Thermogravimetric analyses were conducted on a thermal analyzer (STA 6000, PerkinElmer, USA), by heating the samples from room temperature to 1000°C, at a heating rate of 20°C $min^{-1}$. Approximately 8 mg of sediment were used in each assay, under an oxidizing atmosphere (synthetic air).

To determine the size of the sedimentary grains, granulometric analysis was performed using a laser scattering particle size distribution analyzer (LA-950, HORIBA, Japan). This technique is based on measuring particle dimensions through the angle and intensity of scattered light. Initially, the samples were suspended in a liquid medium (distilled water) to prevent agglomeration. Subsequently, the LA-950 software implemented mathematical models to transform the light scattering pattern into a particle size distribution curve.

## 2.3. Metal extraction and determination

All solutions were prepared using analytical grade reagents and distilled-deionized water (resistivity of 18.2 MΩ cm), obtained by a Milli-Q® purification system (Millipore, Bedford, MA, USA). 37% w $w^{-1}$ HCl (Merck KGaA, Darmstadt, Germany) was used for the extraction step. Decontamination of glassware, plastic bottles and other materials was carried out with a 10% w $w^{-1}$ $HNO_3$ solution for at least 24 h, followed by rinsing with distilled-deionized water.

Approximately 1.0 g of sediment was weighed for each collection point using an analytical balance (AUY220, Shimadzu, Brazil). Next, 25 mL of 0.1 mol $L^{-1}$ HCl extraction solution was added to each sample, which were then incubated in a shaker (TE-4200, Tecnal, Brazil) at 200 rpm, at room temperature, for 2 h, according to the methodology described by Cotta et al. (2006). The same procedure was applied to the certified marine sediment reference material MESS-3 (NRC-CNRC, Ontario, Canada). After extraction, the extracts were filtered through PTFE membranes (SFRC-2545, Filtrilo, Brazil) with a porosity of 0.22 µm to separate the solid phase, and subsequently transferred to 50 mL Falcon tubes. The filtrates were then stored under refrigeration at − 4°C until the time of analysis.

The determination of Ba, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, and Zn in the samples was performed using an inductively coupled plasma optical emission spectrometer (ICP OES) (iCAP PRO XP, Thermo Fisher Scientific, USA). Table S1 of the Supplementary Material shows the instrumental parameters used for the spectrometer.

## 2.4. QA/QC

Quality assurance and quality control (QA/QC) procedures included the evaluation of linearity, limits of detection (LOD), limits of quantification (LOQ), precision and method performance. Calibration curves presented coefficients of determination ($r^2$) higher than 0.995 for all analytes. LOD and LOQ values ranged from 0.0004–0.05 mg $L^{-1}$ and 0.0012–0.16 mg $L^{-1}$, respectively. Repeatability, expressed as relative standard deviation (RSD, n = 10), remained below 10% for all analytes. Method performance was assessed using certified reference material (CRM). However, because the proposed procedure employed partial extraction with 0.1 mol $L^{-1}$ HCl rather than total digestion, the obtained concentrations are not directly comparable to the certified values. Therefore, CRM recoveries were used as an indicator of analytical performance and extraction efficiency of the labile fraction rather than as a direct measure of method accuracy. This approach is consistent with studies focused on environmentally available metal fractions in sediments (Bevilacqua et al. 2009; Cotta et al. 2006). Complete validation data are provided in Table S2.

## 2.5. Data processing and statistical analysis

The correlation between the variables was assessed using Pearson's correlation matrix, with the aim of identifying linear relationships between metal content and sediment granulometry. Correlation coefficients (r) were calculated considering a significance level of 95% ($p < 0.05$). The data were

previously autoscaled to minimize magnitude effects between the variables. The interpretation of the coefficients followed the conventional classification regarding the intensity of the correlation (weak, moderate and strong), considering the direction (positive or negative) of the associations. All statistical calculations were performed using Statistica software (version 10, StatSoft Inc., USA).

The degree of sediment contamination was assessed using the Geoaccumulation Index (Igeo), as proposed by Müller (1969), using as geochemical background values the average concentrations of the continental crust described by Wedepohl (1995). The adopted background values (Bn, in μg $g^{-1}$) were: Ba (425), Cd (0.098), Pb (17), Co (25), Cu (14.3), Cr (92), Fe (47,200), Mn (850), Ni (47) and Zn (67). The Igeo was calculated according to Eq. (2):

$$Igeo = log_2 \left( \frac{Cn}{1{,}5 \times Bn} \right)$$

2

Where: Cn = concentration of the element in the sample (μg $g^{-1}$); Bn = geochemical background value of the element in the continental crust and; 1.5 = correction factor for natural lithological variations.

The ecological risk assessment was carried out using the Potential Ecological Risk Index (Er), according to the methodology proposed by Hakanson (1980), using the same geochemical background values (Bn). The toxicological response factors (Tr) adopted were: Cd (30), Pb (5), Cu (5), Cr (2) and Zn (1). The index was calculated according to Eq. (3):

$$Er = Tr \times \left( \frac{Cn}{Bn} \right)$$

3

Where: Tr = toxicological response factor of the element; Cn = concentration of the metal in the sample and Bn = geochemical background value.

In addition, the Global Potential Ecological Risk Index (PERI) was determined, obtained by summing the individual Er values following Eq. (4):

$$PERI = \sum Er$$

4

## 3. Results and Discussion

# 3.1. Physicochemical and Mineralogical Aspects of Estuarine Sediments

The analysis of carbonate contents (%) at the different sampling sites revealed seasonal variations between the dry and rainy periods (Fig. 2).

The presence of carbonates in sediments exerts a significant influence on metal mobility, acting through processes such as adsorption onto carbonate surfaces, co-precipitation with minerals, and cation exchange within crystalline structures such as aragonite and calcite (Edahbi et al. 2018). Furthermore, the formation of complexes between carbonates and metals can alter their bioavailability, directly impacting their uptake by organisms and, consequently, their toxicity in the sedimentary environment (Devi et al. 2019).

An increase in carbonate contents during the rainy season was observed at the Boa Viagem, Pina, and Toquinho sites, with variations of approximately + 2 to + 3 percentage points in Boa Viagem and Toquinho, and about + 1 percentage point in Pina. This behavior may be associated with the increased input of carbonate-forming ions transported by surface runoff during rainfall events. In contrast, the Carneiros site showed a decrease of approximately 2 percentage points during the rainy period, possibly related to the dilution of carbonate components or to local factors such as hydrodynamics and topography. At the Enseada and Piedade sites, variations were of low magnitude (around ± 1 percentage point), suggesting a lower seasonal influence in these environments.

These variations are consistent with the literature, which identifies factors such as rainfall regime, water composition, vegetation cover, and geomorphological characteristics as key determinants of carbonate dynamics in sediments (Morse and Mackenzie, 1990; Almeida et al. 2020). In the context of environmental monitoring, sediment analysis is recognized as an essential tool for understanding historical transformations in aquatic environments, as it effectively helps to fill gaps in long-term monitoring records (Frascareli 2021).

The data presented in Fig. 3 indicate variations in the organic matter content (OMC) of sediments between the dry and rainy seasons.

The quantity and quality of organic matter in soils and sediments are influenced by climatic factors such as precipitation and temperature, as well as biological activity (Miranda and Avelar, 2019). Seasonal variability in rainfall plays a fundamental role in this context, as it regulates sediment moisture and directly affects organic matter dynamics (Lima Rodrigues et al. 2022).

In the present study, a significant increase in organic matter content (OMC) was observed during the rainy season at the Boa Viagem, Enseadas, Pina, and Toquinho sites. The most pronounced increases were recorded at Pina, rising from approximately 8% to values between 16% and 17%, and at Toquinho, from about 1% to approximately 6%, indicating a strong seasonal influence at these locations.

At Boa Viagem and Enseadas, increases were also consistent, rising from approximately 5% to around 10–11% and from approximately 2% to about 6%, respectively. This pattern suggests a significant input

of organic matter, possibly associated with surface runoff, which transports organic material and nutrients of terrestrial origin into sedimentary environments, as discussed by Praise et al. (2020).

In contrast, the Carneiros and Piedade sites showed a reduction in OMC during the rainy season, with values decreasing from approximately 7.5% to about 4.5% at Carneiros and from around 7.5% to approximately 6.5% at Piedade. This behavior may be related to a dilution effect resulting from increased water volume, as well as hydrodynamic conditions that favor the remobilization and export of organic matter, as reported by Cortés Guzmán et al. (2022).

Additionally, during the rainy period, the Boa Viagem and Pina sites exhibited organic matter contents exceeding 10%, characterizing them as organic sediments according to the criteria established by Saraiva et al. (2009) and Dias et al. (2009). The remaining sites remained below this threshold and are therefore classified as predominantly mineral sediments, with lower organic matter accumulation.

These results highlight marked spatial heterogeneity and strong seasonal control over organic matter dynamics in the analyzed sediments, reflecting the interaction between environmental and anthropogenic factors such as land use, local hydrodynamics, and drainage patterns (Zeng et al. 2019; Connolly et al. 2021).

Furthermore, high organic matter contents, particularly at sites with values above 10%, favor metal complexation, contributing to their retention in sediments (Dias 2009). Thus, the characterization of organic matter constitutes an essential tool for understanding the distribution and behavior of metals in the environment, given its direct and indirect relationship with these elements (Mortensen 1963; Aiken et al. 2011).

The analysis of infrared spectra for sediment samples from Boa Viagem (Fig. 4) and Enseadas (Fig. 5) revealed similar characteristics of organic functional groups, allowing for a comparative discussion between these two main sites.

In the case of Boa Viagem (Fig. 4), bands were identified around 3500 $cm^{-1}$, 1500 $cm^{-1}$, and in the region of 1000–1100 $cm^{-1}$, present in both the dry and rainy periods, although with variations in peak intensity and definition. During the dry period, a greater definition of the band associated with hydroxyl (OH) groups around 3500 $cm^{-1}$ was observed, whereas in the rainy period there was a relative intensification of the band near 1500 $cm^{-1}$, suggesting a possible increase in the contribution of carbonate compounds or associated organic matter.

For the Enseadas sediments (Fig. 5), bands were also observed in the regions of approximately 3500 $cm^{-1}$ and 1000–1100 $cm^{-1}$ in both periods, as well as signals around 1500 $cm^{-1}$. However, unlike Boa Viagem, a higher relative intensity of bands in the 1000–1100 $cm^{-1}$ region was noted during the rainy period, indicating a possible increase in silicate minerals or a greater contribution of fine fractions transported during higher precipitation events (Baez et al. 2025).

Bands near 3500 $cm^{-1}$ are attributed to hydroxyl (OH) groups, commonly found in clay minerals and organic compounds containing alcohol functional groups. The region around 1500 $cm^{-1}$ is associated with C–O bond vibrations, often related to carbonate minerals such as calcite, while bands between 1000–1100 $cm^{-1}$ correspond to Si–O stretching vibrations, typical of clay minerals such as kaolinite and illite, as well as silicates and oxygenated compounds (Yisong et al. 2024).

For the remaining sampling sites (Pina, Piedade, Toquinho, and Carneiros), a similar spectral behavior was observed, with the presence of the same characteristic regions (3500, 1500, and 1000–1100 $cm^{-1}$), but with variations in the relative intensity of peaks between the dry and rainy periods (Figures S1–S4, Supplementary Material). These variations suggest differences in the relative contribution of organic matter, clay minerals, and carbonate components among environments and across seasons.

Overall, the results indicate that although the basic mineralogical composition of the sediments is similar among the sampled sites, there are seasonal modifications in band intensities, possibly associated with the differential input of organic and inorganic materials. This behavior is consistent with studies that use infrared spectroscopy for the characterization of sedimentary materials (Herbert et al. 1992; Barnett and Bailiff, 1997; Poulenard et al. 2009; Jiang et al. 2004; Ishikawa et al. 2023).

The obtained thermograms (Fig. 6) revealed similar thermal behaviors among the analyzed samples, with emphasis on the representative profiles from Boa Viagem during the dry and rainy periods.

The differential thermal analysis (DTA) curves indicated the occurrence of both endothermic and exothermic events under both seasonal conditions. It was observed that, during the rainy period, the endothermic peaks exhibited lower intensity compared to the dry period, possibly due to the influence of higher moisture content in the sediments. The thermogravimetric analysis (TGA) curves demonstrated mass loss as a function of increasing temperature.

In the rainy season samples, greater mass loss was observed at lower temperature ranges, mainly associated with the evaporation of adsorbed water. Under both seasonal conditions, significant mass losses were observed between 700°C and 800°C, indicating more intense thermal decomposition processes within this range.

The thermal events identified in the DTA curves reflect structural transformations in the materials, such as phase transitions and chemical reactions. The lower intensity of endothermic peaks in the rainy samples reinforces the influence of moisture on the thermal behavior of the sediments.

The mass losses observed between 700°C and 800°C are consistent with decarbonation processes, likely related to the decomposition of carbonate minerals such as calcite (Frost et al. 2007). In the dry period samples, the lower initial mass loss corroborates a lower moisture content, while thermal decomposition events occurred within temperature intervals similar to those observed in the rainy period.

For the remaining sampling sites (Enseada, Pina, Piedade, Toquinho, and Carneiros), the thermograms also exhibited thermal behavior similar to that described for Boa Viagem, with the occurrence of endothermic and exothermic events in the DTA curves and comparable mass loss profiles in the TGA curves (Figures S5-S9).

These analyses are fundamental for understanding the thermal stability and composition of the sediments. The TGA technique, as highlighted by Blasio (2019), provides relevant information on thermal transformations, while Mukasyan (2017) emphasizes the importance of thermal analysis in investigating material properties as a function of temperature.

## 3.2. Concentration, sources, and seasonal influence of metals in sediments

The physicochemical characterization of the sediments revealed high carbonate content (37.4–46.7%), the presence of organic matter in different proportions among the sampled points, and the occurrence of functional groups associated with carbonate and silicate minerals identified by FTIR. These characteristics are recognized as important geochemical control factors in sedimentary environments, since they influence the adsorption, complexation, and retention processes of trace elements (Förstner and Wittmann, 1981; Salomons and Förstner, 1984; Alloway 2013). The concentrations of trace elements determined in the coastal sediments are presented in Table 1.

Table 1
– Mean concentration ± standard deviation (μg $g^{-1}$; n = 3) of trace elements in sediments collected during the dry and rainy seasons.

| Element | Mean concentration ± standard deviation (μg $g^{-1}$)[a] | | | | | |
|---|---|---|---|---|---|---|
| | **Boa Viagem** | **Carneiros** | **Piedade** | **Pina** | **Enseadas** | **Toquinho** |
| Dry seasons | | | | | | |
| Ba | 1.97 ± 0.07 | 1.08 ± 0.05 | 1.2 ± 0.1 | 1.30 ± 0.07 | 1.22 ± 0.01 | 1.21 ± 0.09 |
| Cd | < 0.02[b] | < 0.02[b] | < 0.02[b] | < 0.02[b] | < 0.02[b] | < 0.02[b] |
| Pb | < 0.42[b] | < 0.42[b] | < 0.42[b] | < 0.42[b] | < 0.42[b] | 0.99 ± 0.09 |
| Co | 57.8 ± 0.2 | 110 ± 3.2 | 19.1 ± 0.1 | 50.5 ± 0.8 | 52.2 ± 0.2 | 189.3 ± 8.1 |
| Cu | < 0.14[b] | < 0.14[b] | < 0.14[b] | < 0.14[b] | < 0.14[b] | < 0.14[b] |
| Cr | 0.06 ± 0.01 | 0.115 ± 0.002 | 0.06 ± 0.01 | 0.058 ± 0.004 | 3.458 ± 0.004 | 3.04 ± 0.05 |
| Fe | < 1.30[b] | < 1.30[b] | < 1.30[b] | < 1.30[b] | 75.6 ± 0.5 | 79.8 ± 2.7 |
| Mn | 13.1 ± 0.4 | 6.54 ± 0.03 | 22.2 ± 3.2 | 21.5 ± 0.8 | 30.22 ± 0.03 | 3.0 ± 0.1 |
| Ni | < 0.16[b] | < 0.16[b] | < 0.16[b] | < 0.16[b] | < 0.16[b] | 0.49 ± 0.04 |
| Zn | 0.255 ± 0.005 | 0.32 ± 0.01 | 0.238 ± 0.002 | 0.381 ± 0.002 | 1.78 ± 0.0 1 | 5.8 ± 0.5 |
| Rainy seasons | | | | | | |
| Ba | 2.15 ± 0.02 | 1.65 ± 0.03 | 1.02 ± 0.03 | 1.8 ± 0.1 | 1.67 ± 0.03 | 2.88 ± 0.08 |
| Cd | < 0.02[b] | < 0.02[b] | < 0.02[b] | < 0.02[b] | < 0.02[b] | < 0.02[b] |
| Pb | < 0.42[b] | < 0.42[b] | < 0.42[b] | < 0.42[b] | < 0.42[b] | < 0.42[b] |
| Co | 348.8 ± 2.5 | 289.7 ± 12.4 | 12.6 ± 0.3 | 17.8 ± 1.1 | 357.7 ± 42.4 | 257.4 ± 6.7 |
| Cu | < 0.14[b] | < 0.14[b] | < 0.14[b] | < 0.14[b] | < 0.14[b] | < 0.14[b] |
| Cr | 3.86 ± 0.04 | 3.98 ± 0.08 | 0.051 ± 0.003 | 0.07 ± 0.01 | 3.47 ± 0.14 | 3.65 ± 0.13 |
| Fe | 82.4 ± 2.0 | 178.6 ± 6.1 | < 1.30[b] | < 1.30[b] | 71.6 ± 8.1 | 300.8 ± 3.8 |

| Element | Mean concentration ± standard deviation (µg $g^{-1}$)[a] | | | | | |
|---|---|---|---|---|---|---|
| | Boa Viagem | Carneiros | Piedade | Pina | Enseadas | Toquinho |
| Mn | 5.6 ± 0.1 | 9.7 ± 0.2 | 8.3 ± 0.6 | 14.7 ± 1.0 | 5.06 ± 0.12 | 23.1 ± 0.6 |
| Ni | 0.85 ± 0.01 | 0.59 ± 0.01 | < 0.16[b] | < 0.16[b] | 0.81 ± 0.04 | 0.84 ± 0.03 |
| Zn | 3.58 ± 0.08 | 4.62 ± 0.01 | < 0.07[b] | 0.34 ± 0.01 | 3.57 ± 0.08 | 3.36 ± 0.02 |

[b]Below the limit of quantification (LOQ).

Ba levels ranged from 1.08 ± 0.05 to 1.97 ± 0.07 µg $g^{-1}$ during the dry season and from 1.02 ± 0.03 to 2.88 ± 0.08 µg $g^{-1}$ during the rainy season. The highest concentrations were observed in Boa Viagem and Toquinho during the rainy season. In coastal environments, Ba can be associated with carbonate minerals due to adsorption and isomorphic substitution processes in carbonate structures (Morse and Mackenzie, 1990).

Co showed the widest concentration range among the elements analyzed, varying from 19.1 ± 0.1 to 189.3 ± 8.1 µg $g^{-1}$ during the dry season and from 12.6 ± 0.3 to 357.7 ± 42.4 µg $g^{-1}$ during the rainy season. The highest values were observed in Enseadas (357.7 ± 42.4 µg $g^{-1}$), Boa Viagem (348.8 ± 2.5 µg $g^{-1}$), Carneiros (289.7 ± 12.4 µg $g^{-1}$) and Toquinho (257.4 ± 6.7 µg $g^{-1}$). The high spatial variability of this element may reflect differences in the mineralogical composition of the sediments, especially in the proportion of silicate minerals, whose characteristic bands were observed in FTIR spectra between 1000 and 1100 $cm^{-1}$ (Sparks 2003).

Cr concentrations ranged from 0.058 ± 0.004 to 3.458 ± 0.004 µg $g^{-1}$ during the dry season and from 0.051 ± 0.003 to 3.98 ± 0.08 µg $g^{-1}$ during the rainy season. The highest values were recorded in Carneiros, Enseadas, and Toquinho. The distribution of this element may be related to interaction with reactive sedimentary constituents, including organic matter and carbonate minerals, which have a high capacity for adsorbing trace metals (Kabata-Pendias 2010; Alloway 2013).

Fe showed concentrations below the quantification limit at several points during the dry period, being quantified mainly in Enseadas (75.6 ± 0.5 µg $g^{-1}$) and Toquinho (79.8 ± 2.7 µg $g^{-1}$). In the rainy period, concentrations of up to 300.8 ± 3.8 µg $g^{-1}$ were observed in Toquinho and 178.6 ± 6.1 µg $g^{-1}$ in Carneiros. Fe is frequently found associated with mineral oxides and hydroxides, which constitute important adsorption surfaces for trace elements in coastal sediments (Mali et al. 2024).

Mn concentrations ranged from 3.0 ± 0.1 to 30.22 ± 0.03 µg $g^{-1}$ during the dry season and from 5.06 ± 0.12 to 23.1 ± 0.6 µg $g^{-1}$ during the rainy season. The highest values were observed in Enseadas during the dry season and in Toquinho during the rainy season. The occurrence of this element in sediments is

frequently influenced by geochemical processes associated with metallic oxides and the redox dynamics of the sedimentary environment (Sharma et al. 2022).

Ni was detected only in Toquinho during the dry season (0.49 ± 0.04 μg $g^{-1}$), while during the rainy season it showed concentrations between 0.59 ± 0.01 and 0.85 ± 0.01 μg $g^{-1}$ in Boa Viagem, Carneiros, Enseadas, and Toquinho. Zn showed concentrations between 0.238 ± 0.002 and 5.8 ± 0.5 μg $g^{-1}$ during the dry season and between 0.34 ± 0.01 and 4.62 ± 0.01 μg $g^{-1}$ during the rainy season. The retention of these elements may be favored by the presence of organic matter and carbonate minerals, widely recognized as important natural sorbents in sedimentary systems (Förstner and Wittmann, 1981; Alloway 2013).

Cd and Cu remained below their respective quantification limits in all samples analyzed. Pb was detected only in Toquinho during the dry period, with a concentration of 0.99 ± 0.09 μg $g^{-1}$, remaining below the quantification limit in the other points and periods.

The results indicate that the distribution of trace elements in the sediments is consistent with the physicochemical heterogeneity observed among the sampled points. The high carbonate content, the presence of organic matter, and the mineral constituents identified by FTIR and TGA represent important geochemical phases capable of influencing the adsorption, complexation, and retention processes of the elements in the sedimentary matrix.

## 3.3. Influence of particle size distribution on metals in sediments

Pearson correlation analysis was used to investigate the relationships between trace element concentrations and sediment granulometric fractions during the dry and rainy seasons (Tables S3 and S4). This approach allows for the evaluation of possible associations between the elements and the sedimentary constituents, contributing to the understanding of the factors that control their distribution in the studied coastal environments.

The predominance of positive correlations among Co, Cr, Fe, Ni and Zn further suggests that these elements are controlled by similar sedimentary processes or associated with common mineral phases. During the dry season, strong positive correlations were observed between Co and Cr (r = 1.0), Ni and Cr (r = 1.0), Zn and Cr (r = 1.0), and Fe and Ba (r = 0.8). Similar relationships persisted during the rainy season, particularly between Pb and Ni (r = 1.0), Pb and Zn (r = 1.0), Co and Ni (r = 0.9), and Co and Pb (r = 0.9). Such associations indicate that these elements may be retained within the same geochemical compartments of the sediment matrix.

The observed relationships are consistent with the physicochemical characteristics of the studied beaches. Carbonate contents ranged from 37.4 to 46.7%, indicating a substantial contribution of biogenic carbonate materials to sediment composition. Carbonate minerals are known to influence trace-element distribution through adsorption, co-precipitation and structural incorporation mechanisms,

acting as effective sinks for several metallic elements in marine sediments (Morse and Mackenzie, 1990).

In addition to carbonate phases, the FTIR spectra revealed absorption bands associated with silicate minerals, particularly in the 1000–1100 $cm^{-1}$ region corresponding to Si–O stretching vibrations. Silicate minerals can provide reactive surface sites capable of retaining trace elements through adsorption processes, contributing to the observed correlations among metals (Sparks 2003). The coexistence of carbonate and silicate phases therefore creates multiple geochemical compartments capable of influencing trace-element partitioning in these sediments.

The relatively weak relationship between trace elements and the silt fraction suggests that grain size alone does not adequately explain metal distribution in the investigated beaches. This finding differs from studies conducted in estuarine and riverine environments, where fine particles commonly act as the dominant carrier phase for trace metals due to their high specific surface area and elevated adsorption capacity (Förstner and Wittmann, 1981; Zhang and Liu, 2002). In contrast, the predominance of sandy sediments in the present study appears to shift the geochemical control toward sediment composition rather than particle-size refinement.

Furthermore, the occurrence of organic matter in the sediments may contribute to trace-element retention through complexation reactions involving oxygen-containing functional groups. Although the strength of this influence varied among sampling sites, the combined presence of organic matter, carbonate minerals and silicate phases likely enhances the overall capacity of the sediment matrix to retain metallic elements. Such interactions have been widely recognized as key factors controlling metal distribution and stability in coastal environments (Kabata-Pendias 2010; Alloway 2013).

Overall, the correlation patterns indicate that trace-element distribution in the studied beaches is governed by a combination of mineralogical and geochemical factors rather than by sediment texture alone. The predominance of sandy sediments, coupled with high carbonate contents and the occurrence of reactive mineral phases identified by FTIR and TGA, suggests that the sediment composition plays a central role in controlling the accumulation and distribution of trace elements along the investigated coastal areas.

# 3.4. Geochemical indices of environmental quality

The Geoaccumulation Index (Igeo) values for Cd, Cr, Pb, and Zn in the sediments during the dry and rainy seasons are presented in Table S5. In general, all sampled elements and points were classified as uncontaminated (Igeo < 0). Cd showed constant values (-2.88), while Pb and Zn also exhibited negative values, indicating concentrations within natural levels. For Cr, although still classified as uncontaminated, greater spatial variability was observed, with less negative values in locations such as Enseadas and Toquinho, especially during the rainy season, possibly associated with the mobilization of fine particles and continental input during rainfall events (Alloway 2013; Tuo et al. 2020).

The ecological risk factor (Er) values are presented in Table S6. All elements were classified as having low ecological risk (Er < 40). Cd showed constant values (Er = 6.12), while Cr, Pb, and Zn exhibited even lower values, indicating a negligible environmental contribution, even at points with relatively higher concentrations.

The Global Potential Ecological Risk Index (PERI) for sediments is presented in Table 2. Values ranged from 6.24 to 6.56 during the dry season and from 6.24 to 6.40 during the rainy season, classifying all areas as having low ecological risk (PERI < 150). The low variation between periods indicates a limited influence of seasonality on the overall risk.

Table 2
– Global Potential Ecological Risk Index (PERI) values and classification for sediments during the dry and rainy seasons.

| Local | Seasonal period | | Classification |
|---|---|---|---|
| | Dry | Rainy | |
| Boa viagem | 6.25 | 6.38 | Low risk |
| Carneiros | 6.25 | 6.40 | Low risk |
| Piedade | 6.24 | 6.24 | Low risk |
| Pina | 6.25 | 6.25 | Low risk |
| Enseadas | 6.34 | 6.37 | Low risk |
| Toquinho | 6.56 | 6.37 | Low risk |

The results indicate good environmental quality of the sediments, with levels predominantly controlled by natural sources. However, occasional variations, especially during the rainy season, reinforce the importance of continuous monitoring (Hakanson 1980; Kabata-Pendias 2010).

## 3.5. Comparison between measured concentrations and those established by national and international regulations

Comparing metal concentrations with national regulations and international sediment quality guidelines allows a broader assessment of sediment environmental quality (Table 3). In this study, concentrations were compared with the limits established by CONAMA Resolution No. 454/2012, the Canadian Sediment Quality Guidelines (CCME 2001), and the Effects Range criteria proposed by Long et al. (1995). For standardization purposes, TEL and ERL values were grouped as Ecotoxicological Threshold Values (ETV), representing concentrations below which adverse biological effects are rarely expected, whereas PEL and ERM values were grouped as Probable Effect Values (PEV), representing concentrations above which adverse effects are more likely to occur.

The concentrations of the analyzed metals were much lower than the limits established by Brazilian legislation (Level 1 and Level 2), indicating the absence of significant environmental risk from a regulatory standpoint. For Cd, the values remained below the detection limit (< 0.02 μg $g^{-1}$) in both periods, being well below both the CONAMA limits and the international ETV and PEV values, suggesting the absence of relevant contamination. A similar result was observed for Cu, whose concentrations were also below the detection limit (< 0.14 μg $g^{-1}$), indicating low anthropogenic contribution and reduced ecotoxicological potential.

Cr showed variation between 0.058 and 3.98 μg $g^{-1}$, values significantly lower than the national limits (81–370 μg $g^{-1}$) and also below the ETV values (52.3–81 μg $g^{-1}$), indicating a low probability of adverse effects on biota. Similarly, Zn showed concentrations ranging up to 5.80 μg $g^{-1}$, remaining well below both national and international reference values, which reinforces its low ecotoxicological relevance in the studied context.

Pb showed distinct behavior, with a point concentration of 0.99 μg $g^{-1}$ during the dry period at one of the sampled points, while in other cases it remained below the detection limit. Even so, these values are substantially lower than the CONAMA limits (46.7–218 μg $g^{-1}$) and the ETV values (30.2–46.7 μg $g^{-1}$), indicating that there is no evidence of ecological risk associated with this element.

The integrated comparison between national limits and international guideline values (ETV and PEV) shows that the analyzed sediments have good environmental quality, with metal concentrations well below the levels associated with adverse effects. These results suggest that the elements evaluated are predominantly controlled by natural sources and local sedimentary dynamics, with low influence from anthropogenic sources. However, the combined use of different normative and ecotoxicological approaches reinforces the robustness of the assessment and highlights the importance of continuous monitoring, especially in coastal environments subject to seasonal variations and anthropogenic pressures.

# Conclusions

This study achieved its main objective of determining and quantifying trace elements in coastal sediments from Pernambuco and investigating the factors influencing their distribution and mobility within the sediment matrix. The integration of physicochemical characterization, granulometric analysis, geochemical indices, and statistical approaches provided a comprehensive assessment of trace-element behavior in the investigated coastal environments. The analytical methodology demonstrated satisfactory performance for the determination of environmentally available metal fractions, generating reliable data for environmental assessment and monitoring. Moreover, the combined evaluation of sediment properties and trace-element distribution improved the understanding of the geochemical processes controlling metal retention and mobility in tropical coastal sediments. The findings provide valuable baseline information for a region where studies on trace elements in coastal sediments remain limited, contributing to the environmental characterization of the Pernambuco coastline. In addition, the study highlights the importance of continuous monitoring programs to support environmental

management, ecosystem conservation, and the prevention of potential environmental risks. Future research should include broader spatial and temporal sampling schemes, as well as complementary approaches such as sequential extraction procedures, bioaccessibility and bioavailability assessments, and advanced multivariate analyses. The integration of sediment geochemistry with biological indicators and water-quality parameters may further improve the understanding of contaminant dynamics and environmental impacts in coastal ecosystems.

# Declarations


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### Authors' Contributions

**Silvio J.J.C. do Nascimento -** Conceptualization, Data Curation, Formal Analysis, Investigation, Methodology, Validation, Visualization, Writing – original draft; **Iasmim M.S. de Miranda -** Data Curation, Formal Analysis, Investigation, Methodology, Validation, Writing – original draft; **Lucas F.C. da Silva -** Data Curation, Formal Analysis, Investigation, Methodology, Validation, Writing – original draft; **Ryan G.M.F. da Silva -** Data Curation, Formal Analysis, Investigation, Methodology, Validation, Writing – original draft; **Suelen N. dos Santos** - Resources, Validation, Writing – review and editing; **Suennia C.S. Sena** - Resources, Validation, Writing – review and editing; **Ana P.S. Paim** - Resources, Validation, Writing – review and editing; **Laís A. Souza** - Conceptualization, Data Curation, Methodology, Project Administration, Resources, Supervision, Validation, Writing – review and editing.

### Ethical Approval

This is not applicable

### Consent to Participate

This is not applicable

**Consent to Publish**

This is not applicable

**Competing Interests**

The authors have no competing interests to declare that are relevant to the content of this article.

**Data Availability Statement**

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

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## Table 3

Table 3 is available in the Supplementary Files section.

## Figures

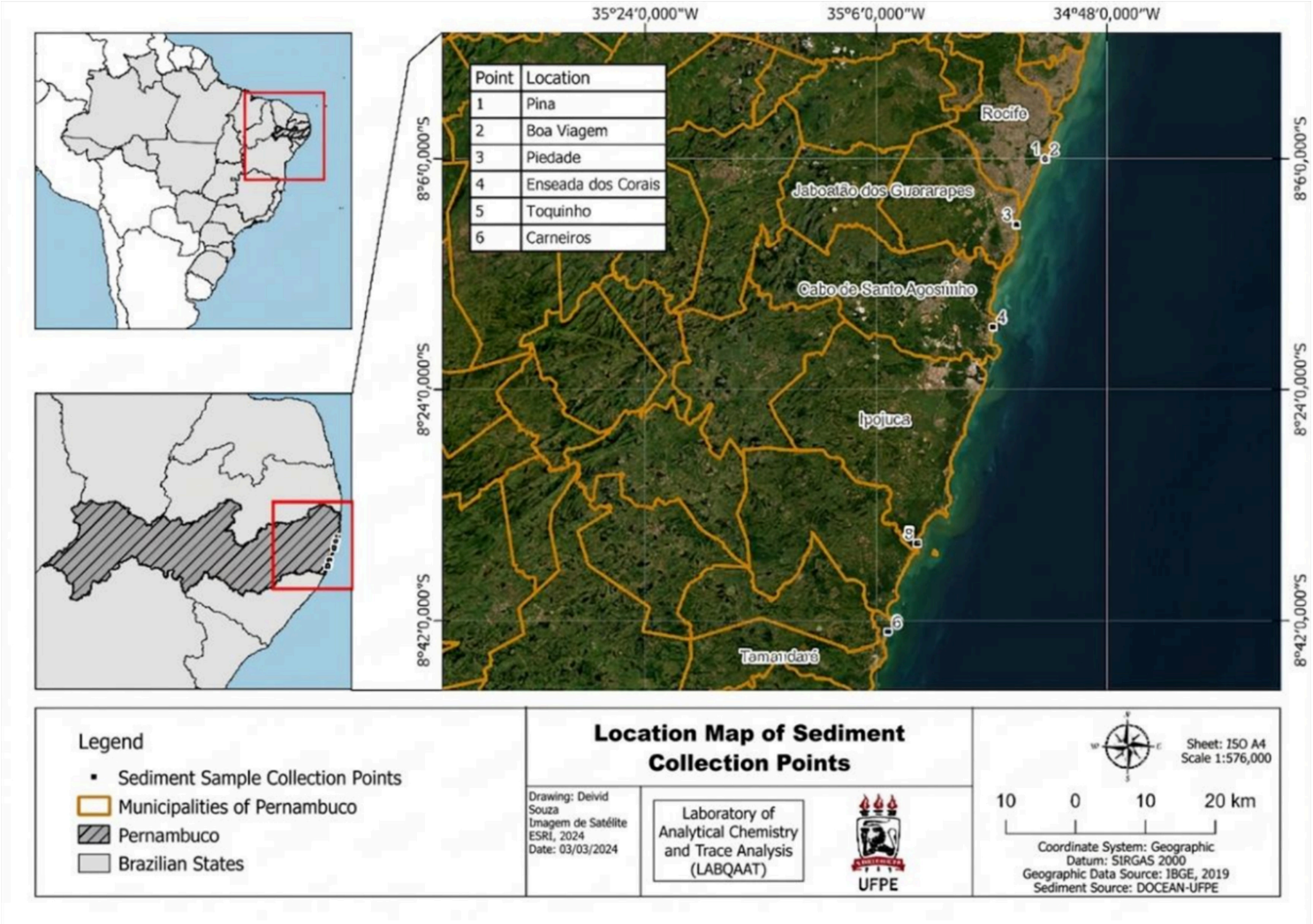



**Figure 1**

Location of the six sediment sampling points during the dry and rainy seasons.

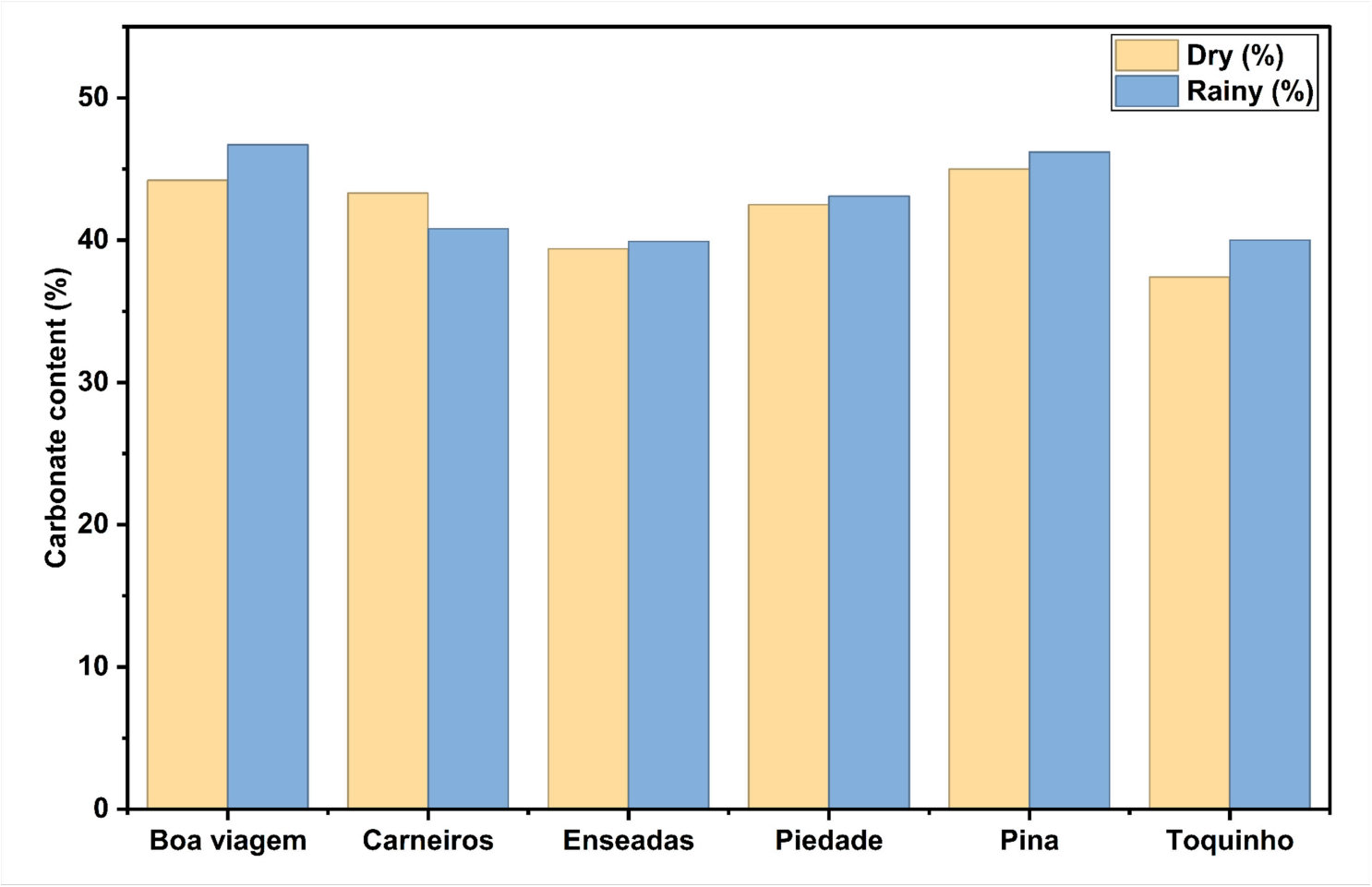



**Figure 2**

Carbonate content values of sediments during the dry and rainy periods (% w $w^{-1}$)

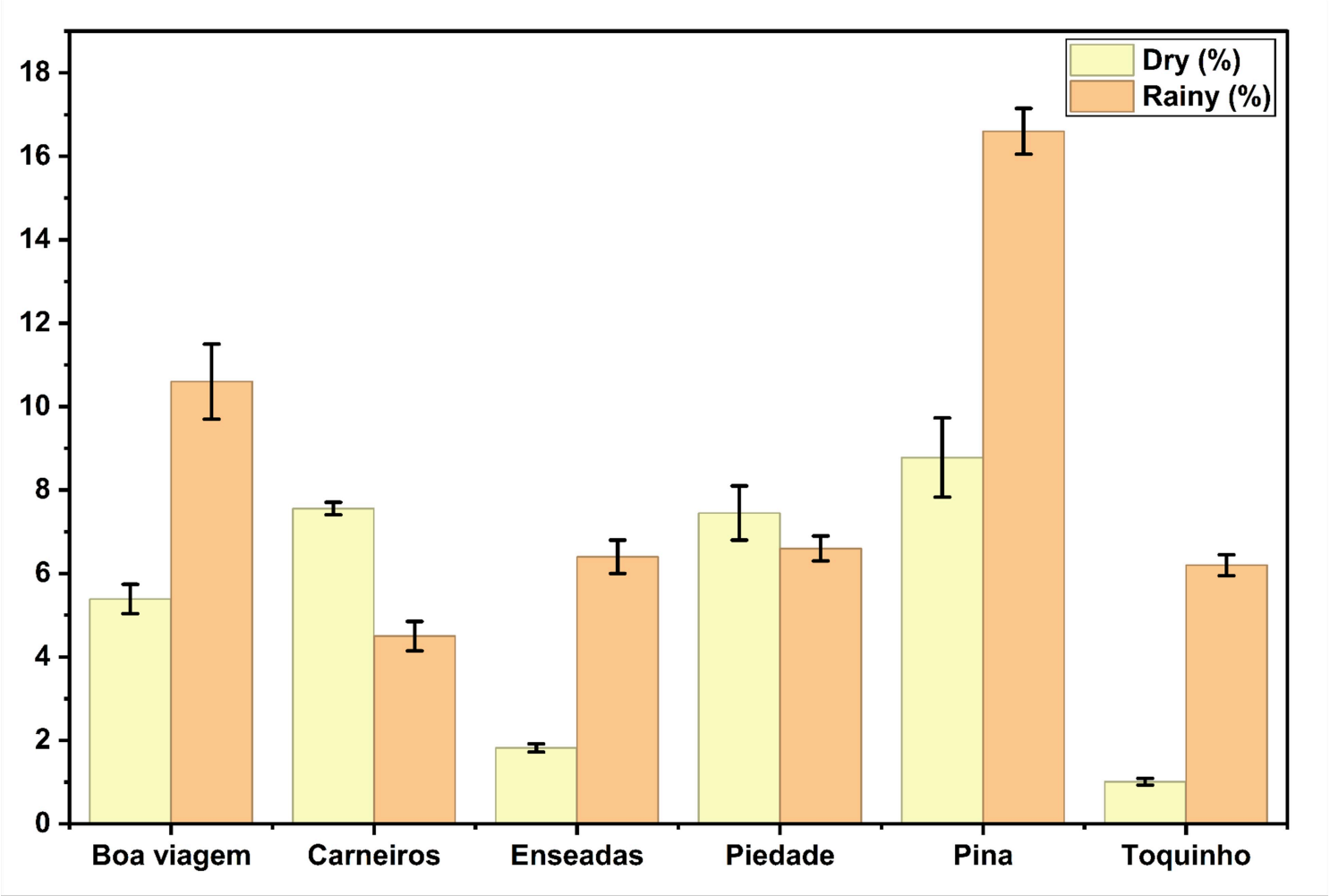



**Figure 3**

Organic Matter Content (OMC) values of sediments during the dry and rainy periods (% w $w^{-1}$) (Mean ± Standard Deviation, n = 3).

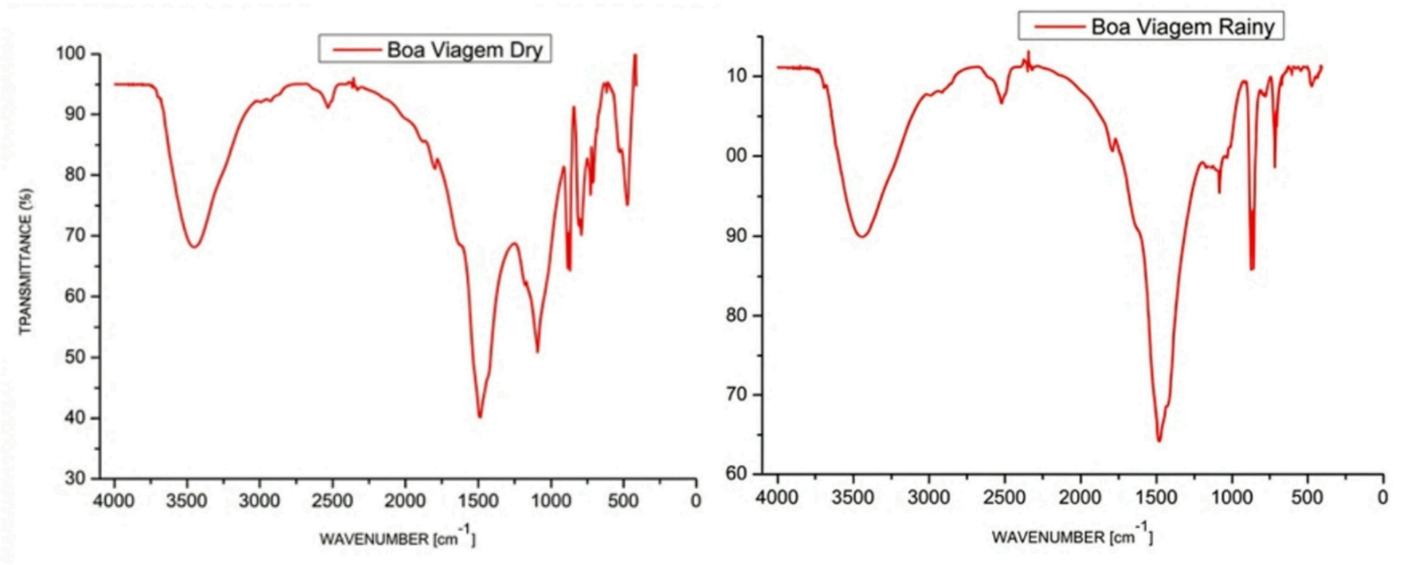



**Figure 4**

Infrared absorption spectra of sediment samples from Boa Viagem during the dry and rainy periods, respectively.

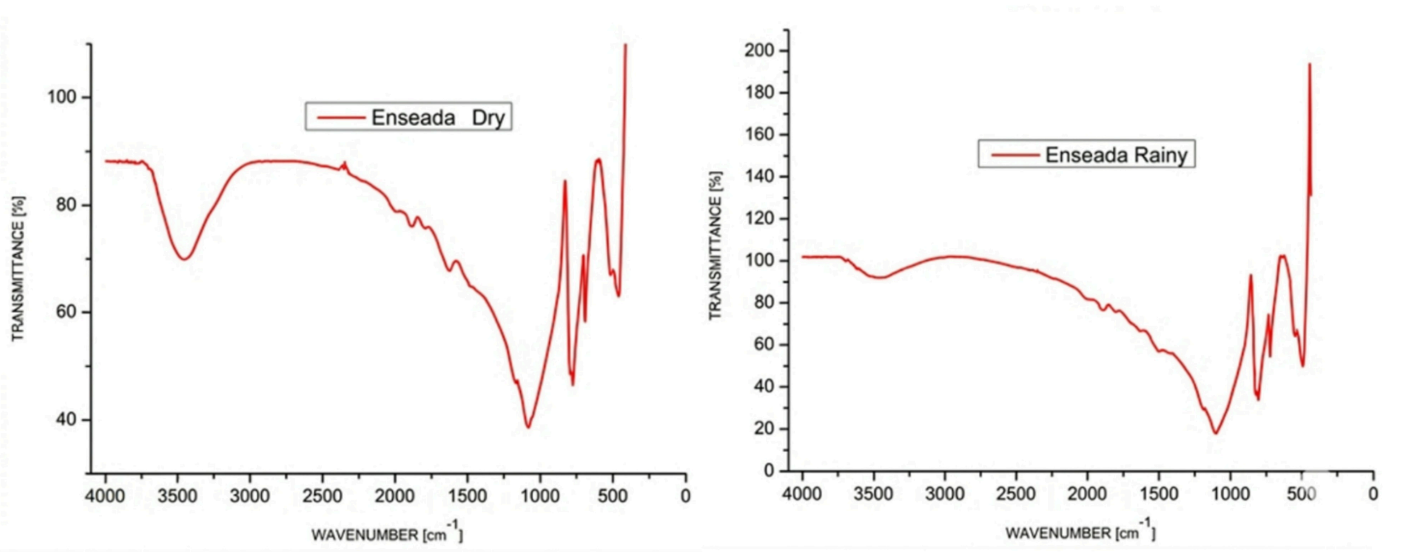



**Figure 5**

Infrared absorption spectra of sediment samples from Enseada during the dry and rainy periods, respectively.

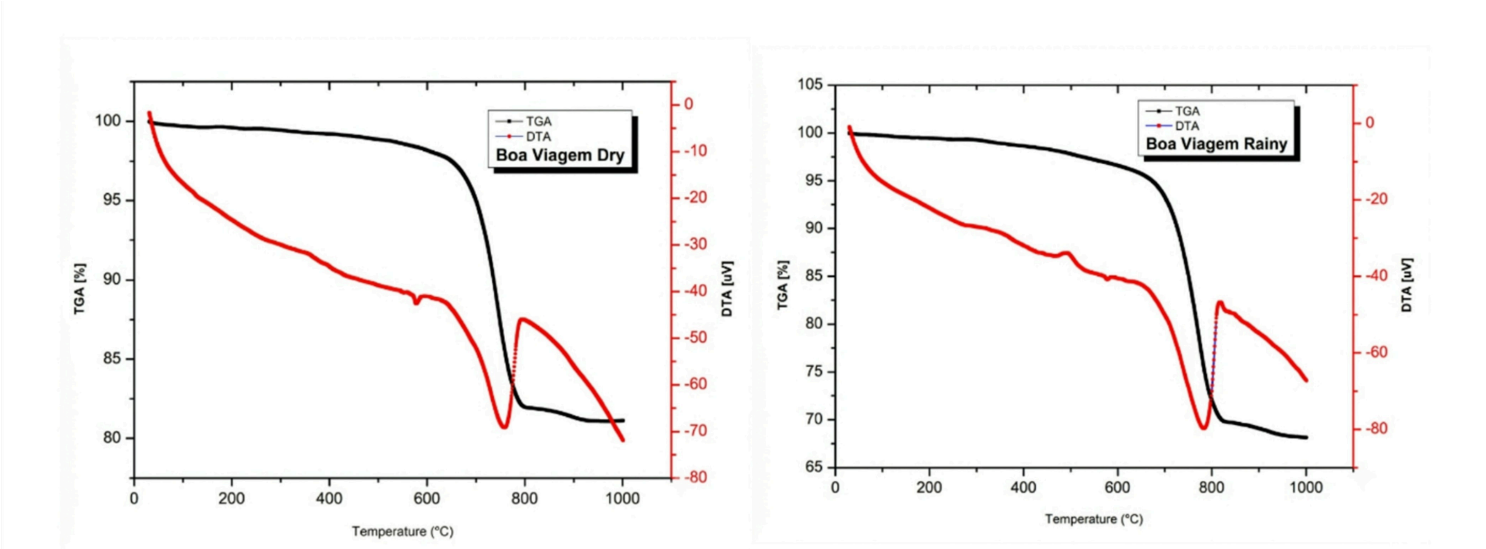



**Figure 6**

Thermograms of sediment samples from Boa Viagem during the dry and rainy periods.

## Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- Table3.docx
- SupplementaryMaterialESPR.docx