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A baseline study of heavy metals composition in the marine sediment of Pulau Layang-Layang, Malaysia

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Abstract: This study establishes comprehensive benchmark data regarding the heavy metal composition in surface marine sediments of Pulau Layang-Layang, Malaysia. Analysis of sediment samples utilizing scanning electron microscopy (SEM) revealed a predominance of biogenic carbonate materials. Total metal concentrations determined through inductively coupled plasma optical emission spectroscopy (ICP-OES) demonstrated a decreasing order of mean values: Ca (384,286 mg/kg) > Mg (10,733 mg/kg) > Na (9,700 mg/kg) > Sr (5,317 mg/kg) > K (474.14 mg/kg) > Fe (343.39 mg/kg) > Al (88.0 mg/kg) > Mn (15.60 mg/kg) > Zn (6.56 mg/kg) > As (3.53 mg/kg) > Cr (3.41 mg/kg) > Ni (1.22 mg/kg). Multivariate statistical analyses, including Pearson correlation, Hierarchical Cluster Analysis (HCA), and Principal Component Analysis (PCA), identified natural sources as the primary contributors to the metal content in the sediments. The heavy metal concentrations remained consistently below the effects range limits (ERL/ERM) established by National Oceanic and Atmospheric Administration's Screening Quick Reference Tables (SQuiRTs), indicating no risk of adverse biological effects. The sediment quality guidelines further suggested that the marine ecosystem maintains its health, with all sampling points exhibiting safe heavy metal levels. Consequently, this baseline study serves as a crucial reference for future monitoring and comparisons of heavy metal contents with other Bornean sedimentary islands or along Malaysia's coastline.

Keywords: Heavy metals, Pearson Correlation Matrix, Hierarchical Cluster Analysis, sediment quality guideline, baseline study

INTRODUCTION

Pulau Layang-Layang plays a pivotal role as both a significant breeding ground and a vital source of sustenance for diverse marine species, contributing substantially to the preservation of rich marine biodiversity and the overall health of the ecosystem in the region. Metals are continuously introduced into the marine environment through natural and anthropogenic processes from surrounding areas. Natural environmental pollution by heavy metals has been a global problem due to their persistence, toxicity, and non-degradable characteristics, which enable them to bioaccumulate and cause severe contamination risks when introduced into the marine environment (Jafarabadi *et al*., 2017; Bantan *et al*., 2020; Algül & Beyhan, 2020; Ling *et al*., 2023a). These heavy metals could also smother the surrounding coral reefs and adversely impact the coral ecosystem, marine life, and benthic organisms (Satpathy *et al*., 2012).

Possible anthropogenic sources such as oil spills, semi-treated sewage, plastic debris, harbor operations, local fisheries disposal, building activities along the seashore, landfilling, and dredged materials found near the island may cause heavy metal buildup along the coastal regions of Pulau Layang-Layang. Despite the potential anthropogenic inputs, the island is surrounded by seawater that could rapidly dilute and disperse any pollutants through ocean currents, thus mitigating the potential for adverse environmental effects (Schlosser *et al*., 1995). The dynamic nature of ocean currents also aids in preventing the accumulation of metals in localized areas, further reducing the risk of metal contamination in marine sediments. Overall, there appears to be minimal anthropogenic influence on heavy metal concentrations in the surface sediments surrounding the island. Instead, the metal concentrations are predominantly contributed by natural processes, and the heavy metal contents determined are considered natural background values of the study area.

Generally, natural sources of marine sediments include the accumulation of lithogenic, biogenic, hydrogenic, and other compounds. Lithogenic constituents typically originate from the weathering of rocks on land or the seafloor, as well as from volcanic eruptions (Li & Schoonmaker, 2003). Biogenic constituents are composed of skeletal fragments or tests of planktic and benthic organisms, along with biogenic apatite, a mineral associated with the remains of

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marine life. The hydrogenous fraction of marine sediment is formed through inorganic precipitation from seawater due to chemical reactions. Such natural processes can lead to elevated metal levels in marine sediments, which may pose risks to marine ecosystems. Therefore, monitoring heavy metal concentrations is crucial for detecting deviations from baseline levels, allowing researchers to distinguish between natural variability and potential anthropogenic influences in the study area.

The naturally sorbed constituents in marine sediments, whether heavy metals, trace metals, or metalloids, may still pollute marine resources or disrupt the marine life cycle (Adams *et al*., 1992; Chakraborty *et al*., 2014; Salem *et al*., 2014). Although human activities have a minimal impact on the total metal contents of this island, the SQuirTs set by NOAA (National Oceanic and Atmospheric Administration) is a significant guideline used to understand and predict the bioavailability and ecotoxicity levels of these heavy metals when exposed to marine species or potentially deteriorating the marine ecology (Li *et al*., 2015). This is because metal contaminants adsorbed onto sediments can be easily remobilized and re-enter the marine water system as secondary pollutants (Ling *et al*., 2023b). Therefore, heavy metal concentrations in sediments are usually higher than in water, and sediments could also serve as a source of heavy metals to the overlying water, causing significant damage to the ecological status of the marine system (Algül & Beyhan, 2020). The direct transfer of metals from sediments to marine organisms has assumed national and global prominence. Therefore, the use of interpretative tools, such as quality guidelines and indexes, provides significant guidance for decision-making and management strategies regarding the study area.

The objectives of this study are to determine the heavy metal concentrations in marine sediments and identify their potential sources. This study is important as it offers readers valuable scientific insights into the current status of sediment quality in Pulau Layang-Layang, serving as a preliminary assessment for sustainable management and future development of the area. In summary, this study highlights the importance of effectively monitoring and managing the potential loading factors (natural sources) contributing to metal pollution in the local region. The results obtained will serve as baseline data for comparison with future monitoring studies of heavy metal compositions in marine sediments.

MATERIALS AND METHODS

Description of study region

Pulau Layang-Layang forms a segment of the fragmented Spratly Islands within the Dangerous Ground Province adjacent to the North-West Borneo Trough (Hamilton, 1979; Hutchison & Vijayan, 2010). The island lies approximately 300 km from the Sabah coast and comprises a carbonate platform resulting from the karstic weathering of underlying carbonate formations along the cuesta and encircling coral reefs (Neal *et al*., 2018). The Quaternary to recent surficial marine sediments consist predominantly of clastic materials, coral fragments, skeletal remains of marine organisms, and oceanic detritus throughout the lagoon and adjacent reef systems (Hutchison & Vijayan, 2010; Waheed *et al*., 2015). The study area encompassing Pulau Layang-Layang is demarcated by coordinates 7°21’45” N to 7°23’10” N latitude and 113°47’06” E to 113°51’23” E longitude (Figure 1).

The weathered constituents of dredged igneous and metamorphic rocks discovered near the cuesta axes plunging in the Spratly Islands accumulate and contribute to the marine sediments of Pulau Layang-Layang (Hutchison & Vijayan, 2010). As these rocks deteriorate and undergo dredging, their components and mineral content are transported and deposited within the marine environment, thereby affecting the sediment composition in the surrounding island. For example, the weathering of rhyolite and basalts dredged from Reed Bank influences the heavy metal concentrations in these sediments (Hutchison, 2010). The geological framework substantially affects the metal content in sediments, potentially causing adverse biological effects on marine ecosystems. Consequently, this research is essential as it establishes geochemical baseline data regarding the present status of heavy metals in marine sediments.

Sediment sampling

All apparatus used for this study were washed thoroughly using Merck Extran® MA2 soap and rinsed with tap water, then with deionized water. The glassware and sampling tools were soaked overnight in 10% HNO_3 acid before rinsing with Milli Q® quality water. The marine sediment samples were collected using pre-cleaned stainless steel sampling tools, including a hand scoop in the shallow region, such as the coastal areas along the island, and a Grab sampler in deeper regions such as the lagoon, as shown in Figure 2. The stainless-steel material is highly resistant to corrosion and generally has lower reactivity with environmental samples. The sediment collected was kept in sealed ziplock bags and stored at 4°C inside a cooler box until further experimental analysis. A total of 23 sampling points were selected for the study across the lagoon and coastal areas, which served as control natural sediment conditions and established baseline values for the study area.

Experimental and statistical analysis

Scanning electron microscopy (SEM) was conducted to identify the mineralogy and mineral structures using a JEOL JSM-35 6100 microscope and Link AN 10/855 analyser (ASTM, 2018). Inductively coupled plasma optical emission spectrometry (ICP-OES) analysis was performed using an aqua regia digestion method to measure total metal concentrations in mg/kg using a PerkinElmer Optima 5300DV spectrometer (US EPA, 2014). The aqua regia

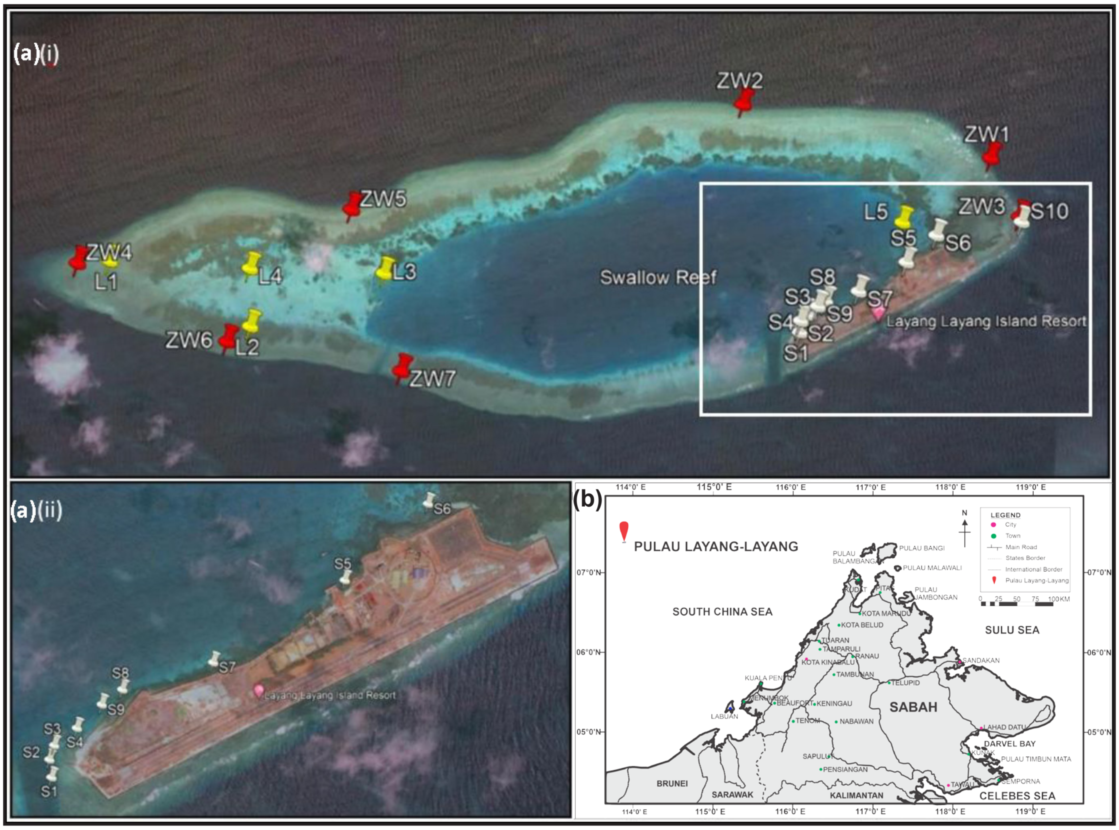

Figure 1: (a) (i-ii) Google satellite images for the sampling sites around Pulau Layang-Layang and (b) location of the island from Sabah, Malaysia. Source: Google Earth satellite (accessed on March, 2023).

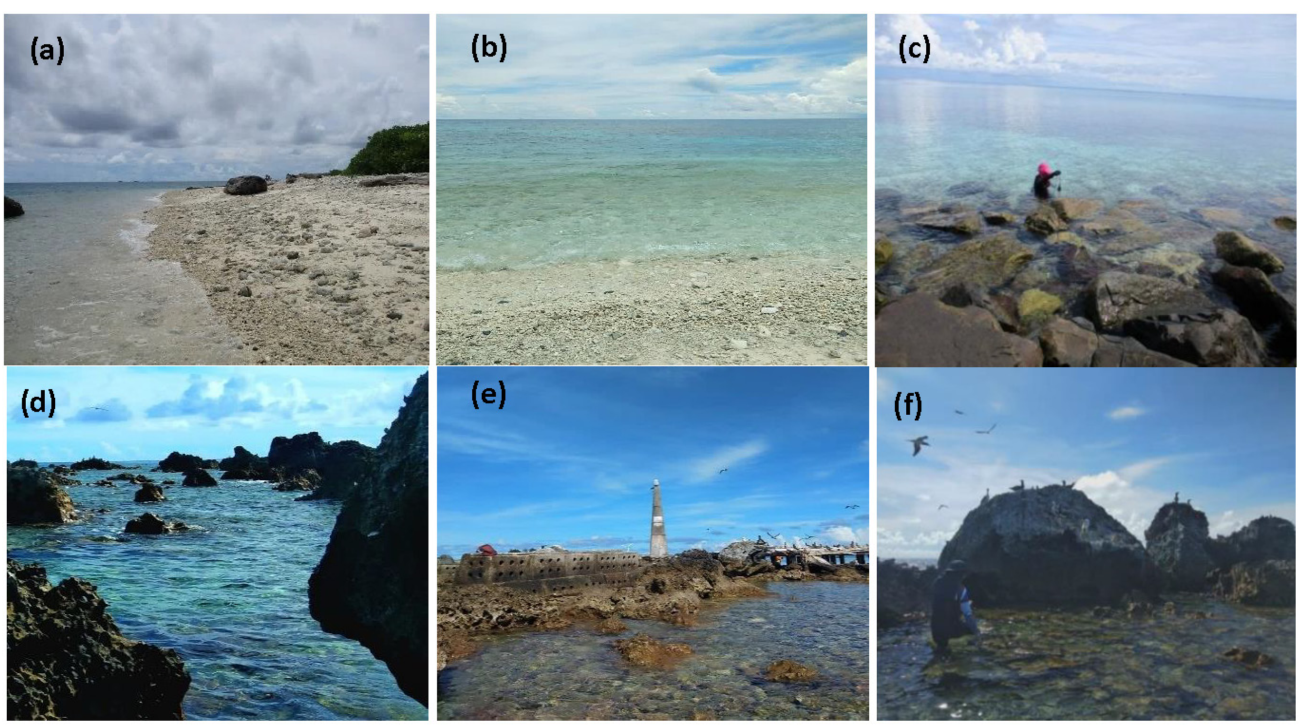

Figure 2: Sampling for surface marine sediment near the coast (a-c) and around the lagoon (d-f) regions of Pulau Layang-Layang, Malaysia.

mixture was prepared using a 3:1 ratio of HCl (35-37%) to HNO_3 (65%). The metal contents in sediments were then compared with the effects range low (ERL) and effects range median (ERM) limits based on the Sediment Quality Reference Tables (SQuiRTs) by NOAA (Buchman, 2008). The sediment quality guidelines were used to evaluate the levels of heavy metal contamination and their potential adverse biological effects in the marine sediments of Pulau Layang-Layang, Malaysia.

All metals detected were evaluated using a Pearson correlation matrix and presented in a dendrogram from hierarchical cluster analysis (HCA) based on Ward's linkage method via IBM SPSS Statistics 28.0 software (Ward, 1963). Principal component analysis (PCA) was employed using the varimax rotation method with Kaiser normalisation to extract principal components with eigenvalues greater than 1 (Kaiser, 1960). PCA and HCA were used to interpret the potential sources of contamination for improved management of heavy metals. To eliminate bias in the analysis, all data obtained were log-transformed and standardised using z-scores.

Experimental quality control

All tools were regularly inspected for any signs of wear, corrosion, or damage to reduce the risk of contamination before use. The instrument used to carry out ICP-OES analysis was calibrated using a standard dilution stock solution of 100 mg/L in compliance with the International Certified Reference Materials (CRM). Five reagent blanks were analyzed to measure the metal carry-over before conducting the ICP-OES analysis, and all sediment samples were analyzed in triplicates to increase the accuracy and precision of the laboratory data obtained. The calculations for standard reference materials (SRM NIST-2702) analysis were also performed with 80-120% accuracy to determine the suitability of the acid digestion method. The mean percentage recoveries were determined for both major elements Al (86%), Ca (118%), Fe (102%), K (92%), Mg (112%), and Na (80%), as well as trace elements As (82%), Cr (82%), Mn (98%), Ni (80%), Sr (118%), and Zn (91%).

RESULT AND DISCUSSION

Mineral composition

Mineralogical studies are conducted to reveal the predominant minerals in marine sediments, as they play a crucial role in facilitating heavy-metal mobility. The Scanning Electron Microscope (SEM) photomicrographs in Figure 3 demonstrate that the marine sediment derives from nearshore biogenically produced carbonate sand, primarily comprising calcareous remains of corals, benthic foraminifera, calcareous algae, mollusc shells, spicules, and other skeletal fragments of marine biota. Their tests and skeletal remains are typically cemented with sand grains (quartz), clay, or other materials, and crystalline CaCO3 occurs in the form of calcite or aragonite, depending on the species. Most carbonate minerals appear as clusters of calcium carbonate materials in the sediment samples. These carbonate minerals are essential in the marine environment as they facilitate heavy-metal adsorption, thus forming stable or insoluble compounds (metal carbonates) that reduce metal bioavailability and prevent contamination (Ling *et al*., 2023a).

Environmental factors, such as changes in ocean currents, seafloor temperature, and pressure, frequently influence the formation of silicate minerals containing Al and Mg (Wang *et al*., 2019). Clay content in aluminosilicate precipitates forms through mineral recrystallization in the alkaline environment of coastal shores (Ling *et al*., 2023a). Despite their low clay content, these minerals exhibit significant capacity to adsorb heavy metals through various

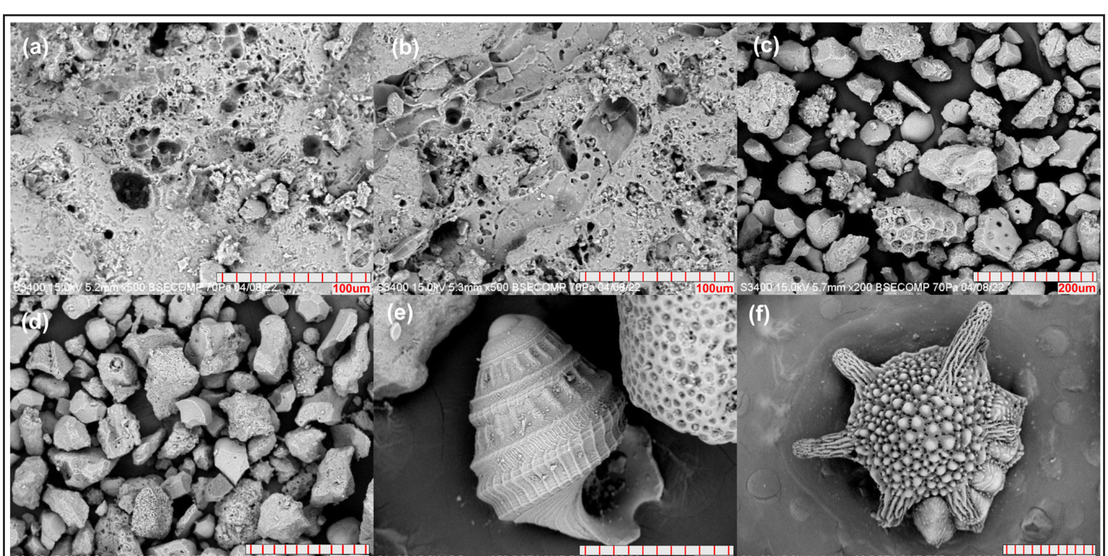

Figure 3: The SEM photomicrographs show that the marine sediment is composed mainly of (a-d) calcareous fragments of corals, benthic foraminifera, calcareous algae, mollusc shells, spicules and other skeletal remains of marine biota, including (e) gastropods and (f) foraminifera.

mechanisms, including ion exchange, direct bonding, and surface complexation with metal cations (Yuan *et al*., 2013; Ling *et al*., 2022). The layered arrangement of silica and alumina plates in clay provides an extensive surface area with numerous active sites, thereby facilitating heavy-metal adsorption from the surrounding environment. Moreover, clay minerals function as metal carriers that potentially limit metal mobility and availability in marine sediments (Ugwu & Igbokwe, 2019).

Total metal concentrations

The total metal concentrations of metals measured in the surficial marine sediments of Pulau Layang-Layang were presented in Table 1 for Al (88.0 mg/kg), As (3.53 mg/kg), Ca (384,286 mg/kg), Cr (3.41 mg/kg), Fe (343.39 mg/kg), K (474.14 mg/kg), Mg (10,733 mg/kg), Mn (15.60 mg/kg), Na (9,700 mg/kg), Ni (1.22 mg/kg), Sr (5,317 mg/kg), and Zn (6.56 mg/kg). The decreasing trend of total metal concentrations for this study was Ca > Mg > Na > Sr > K > Fe > Al > Mn > Zn > As > Cr > Ni. The metal concentrations were uniformly distributed in the surface sediments throughout the study region and were considered to be present at natural background levels. This geochemical study was thus carried out to determine baseline metal concentrations in the sediment as a reference for future studies.

Table 1: Total metal concentration in the marine sediments by ICP-OES analysis.

Sample	Total Metal Concentration (mg/kg)											
	Al	As	Ca	Cr	Fe	K	Mg	Mn	Na	Ni	Sr	Zn
S1	98.79	3.95	380,729	4.68	436.04	261.08	11,314	16.71	4,920	2.22	4,400	4.56
S2	180.08	3.22	370,704	1.91	420.87	439.08	12,453	15.75	9,283	0.72	5,398	6.33
S3	132.59	3.34	393,337	2.11	271.01	382.68	12,198	15.06	8,363	1.20	4,970	5.27
S4	114.40	4.03	381,315	2.32	194.69	509.52	12,358	15.35	9,489	0.54	5,278	10.27
S5	201.79	3.47	384,613	3.05	1,622.84	427.22	8,310	9.58	8,266	1.73	5,099	12.66
S6	205.17	3.28	355,081	3.43	417.21	616.32	10,379	8.86	11,366	0.20	5,302	7.54
S7	221.49	2.23	349,282	1.22	1,311.05	541.55	8,486	10.05	9,543	0.42	5,374	5.66
S8	111.87	4.19	356,321	8.30	374.40	455.04	10,786	16.84	8,709	0.63	4,984	5.43
S9	109.52	4.48	374,440	5.25	310.55	561.74	11,861	16.11	12,478	0.72	5,812	5.04
S10	47.68	2.56	384,535	4.53	368.44	507.34	11,738	14.00	12,079	0.93	5,343	9.35
L01	40.55	2.69	354,587	1.80	157.60	446.79	11,512	9.72	8,465	2.49	4,742	7.40
L02	36.18	3.19	440,171	2.04	122.41	504.69	12,637	11.91	9,931	1.98	5,004	7.34
L03	63.82	3.53	412,874	5.67	151.39	383.74	11,397	7.81	7,543	2.37	5,607	9.71
L04	33.06	3.54	365,566	4.47	263.35	462.31	9,569	6.22	10,879	1.28	5,550	3.80
L05	37.16	3.27	385,016	4.65	141.88	453.41	5,083	7.18	4,542	0.23	4,486	4.58
L06	39.17	4.10	360,893	2.73	101.39	553.03	9,525	6.11	11,232	0.51	5,799	3.51
ZW01	57.78	3.53	361,404	2.13	140.37	354.13	9,525	16.92	7,907	1.61	5,603	5.13
ZW02	64.40	5.25	401,551	1.99	204.15	364.71	11,795	28.49	7,547	1.59	5,511	6.15
ZW03	61.65	2.95	382,689	6.61	173.54	328.24	10,771	17.55	7,553	2.12	5,824	9.23
ZW04	32.28	2.13	384,379	2.53	114.35	447.52	10,406	19.66	9,970	1.44	5,711	6.95
ZW05	81.96	5.32	397,130	1.73	133.46	704.01	11,881	35.72	15,573	1.23	5,484	5.92
ZW06	23.59	4.22	484,862	2.10	212.03	540.46	11,457	27.21	12,943	1.02	5,416	5.06
ZW07	29.02	2.81	377,106	3.18	255.00	660.63	11,410	25.94	14,508	0.86	5,600	3.93
Range	23.59-221.49	2.13-5.32	349,282-484,862	1.22-8.30	101.39-1,622.84	261.08-704.01	5,083-12,637	6.11-35.72	4,542-15,573	0.20-2.49	4,400-5,824	3.51-12.66
Mean ± SD	88.0 ± 62.11	3.53 ± 0.83	384,286 ± 30,243	3.41 ±1.81	343.39 ± 372.96	474.14 ± 106.47	10,733 ±1,730	15.60 ±7.72	9,700 ±2,708	1.22 ±0.70	5,317 ±400	6.56 ±2.35

SD = Standard Deviation

The high Ca and Sr were influenced by the amounts of carbonate content from the reef's platform, whereas the high K was influenced by the contents of illite and feldspar in the surface sediment (Cho *et al*., 1999). The high Ca, Mg, Sr, Na, and K compositions are also reflected in the highly saline and alkaline marine environments, whereby the predominant biominerals are mainly contributed by marine organisms such as the adjacent coral reefs, radiolaria, coccolithophores, foraminifera, bryozoans, and echinoids (Burdige, 2007). Dissolution of the biogenic oozes, made up of the calcareous (calcite and aragonite) and siliceous (amorphous silica) tests of marine organisms, upon burial also tends to release Ca, Mg, and Sr metals (Schlanger & Douglas, 1975; Ohde & Kitano, 1984; Ling *et al*., 2023a). Biogeochemical cycles of the carbonate phases, such as the substitution of major cations like Mg2+ and Mn2+ with Ca2+ (Sun *et al*., 2020), as well as the precipitation of high Mg content from the pore waters, and recrystallization of biogenic calcite (low Mg) to other forms of carbonates (high Mg), also contribute to the high contents of Ca, Mg, Sr, and Zn in the marine sediment (Berg *et al*., 2019).

Authigenic manganese and iron oxides were also found in the marine sediments. The elevated concentrations of metalliferous oxides (both Fe and Mn), whereby Fe2+ and Mn2+ are formed directly when the fluids from hydrothermal vents are in contact with oxygenated seawater (Burdige, 2007). Fe, Al, and Mn also attribute to the major components of marine hydrogenetic deposits formed by the precipitation of dissolved components from ambient seawater, which gradually settle and accumulate in bottom sediments (Kristiansen *et al*., 2002; Lusty *et al*., 2018). Hence, the integration of these elements, as seawater circulates through geological and chemical dynamics on the ocean floor, is vital for understanding their distribution patterns in marine ecosystems. Alternating layers of Fe-Mn metals are also commonly found in marine sediment with potential economic interests due to the high concentrations of trace metals like Ni, Cu, and Co, as a result of hydrogenetic, diagenetic, hydrothermal, and other redox cycling processes from different environments in the global ocean (Josso *et al*., 2017). These heavy metals, Fe and Mn, may also originate from the clastic materials and aluminosilicate minerals present in the clay within sediment (Ling *et al*., 2022).

Variable solubility of different trace elements like As, Zn, Cr, and Ni under different redox conditions allows them to act as essential micronutrients, which support the normal growth of marine organisms through the coupled cycling of these elements from the seawater column into the organisms' bodies (Sweere *et al*., 2023). The element As is commonly found in marine environments but usually in trace amounts that do not significantly impact the ecosystem. In this study, the concentrations of arsenic detected are very low and do not pose a threat to marine life. The potential natural sources of these metals are diverse, such as from the dissolution of minerals in terrestrial environments, lithogenic crusts, or probably also transported from the Bornean Island.

The evaluation of heavy metal concentrations in Malaysian sediment lacks regulations of specific standards for sediment quality monitoring. Therefore, the use of sediment quality guidelines (SQGs) has become a significant tool to determine the toxicological relevance of contaminants associated with marine sediments and also delineate the range of biological effects on marine organisms (Buchman, 2008). Based on the SQG, metal concentrations below the ERL are not associated with adverse biological effects, whereas metal concentrations between the ERL and ERM may occasionally associate with adverse biological consequences. For concentrations above the ERM, they are associated with adverse biological effects (Romero-Murillo *et al*., 2023). The results obtained from the present study were compared to the Sediment Quality Reference Tables (SQuiRTs) established by the National Oceanic and Atmospheric Administration (NOAA), which showed that all heavy metal concentrations were below the TEL and are within natural background levels; hence, they do not pose a threat to marine life.

Pearson correlation matrix

A Pearson correlation matrix was employed to assess the relationships between metals, as shown in Table 2. The correlation matrix revealed positive correlations between Na and K (r=0.86), Al and Fe (r=0.74), Na and Sr (r=0.54), Mn and Mg (r=0.45), and Mn and Na (r=0.44). The analysis also demonstrated a negative correlation between Ni and K (r=-0.58), while other metals exhibited no correlation. The positive correlations of Na-K-Sr were attributed to saline minerals of soluble marine salts that contribute to the oceanic geochemical cycles (Culkin & Cox, 1966; Ankindinova *et al*., 2019; Zhang *et al*., 2020). Because Na and K are primary seawater ions, and Sr is a minor yet significant constituent, their correlation may indicate marine inputs or evaporite mineral presence.

The positive Mg-Mn correlation was linked to authigenic or clastic deposits, particularly clay materials present in marine sediments (Berg *et al*., 2019). Al and Fe demonstrate strong positive correlation because they constitute major lithogenous components of the upper continental crust. These elements are typically released through source rock weathering and subsequently transported to oceanic basins. Their strong correlation in this Pearson analysis also indicates their co-occurrence in sediment particles, where they frequently exist within aluminosilicate minerals or clay materials (Yi *et al*., 2021; Ling *et al*., 2023b). The negative K-Ni correlation suggested that elevated K concentrations might inhibit or decrease Ni metal adsorption onto sediment surfaces, thereby reducing Ni contamination in marine sediment. Metals showing no correlation suggested derivation from distinct sources.

Table 2: Pearson correlation matrix of the total metal concentrations.

	Al	Ca	Fe	K	Mg	Mn	Na	Sr	As	Cr	Ni	Zn
Al	1											
Ca	-0.38	1										
Fe	0.74**	-0.23	1									
K	0.04	0.02	-0.01	1								
Mg	-0.07	0.29	-0.34	0.07	1							
Mn	-0.20	0.39	-0.24	0.24	0.45*	1						
Na	-0.10	0.15	-0.10	0.86**	0.38	0.44*	1					
Sr	-0.12	-0.00	-0.15	0.29	0.21	0.23	0.54**	1				
As	-0.06	0.26	-0.22	0.10	0.23	0.48*	0.15	0.07	1			
Cr	-0.11	-0.16	-0.10	-0.26	-0.13	-0.21	-0.24	-0.03	0.05	1		
Ni	-0.32	0.25	-0.10	-0.58**	0.29	0.05	-0.33	-0.11	-0.05	0.04	1	
Zn	0.30	0.10	0.38	-0.17	0.12	-0.17	-0.16	0.01	-0.19	0.06	0.32	1

*Correlation is significant at the 0.05 level; **Correlation is significant at the 0.01 level; r is Pearson correlation coefficient

Hierarchical cluster analysis (HCA)

HCA serves as a crucial multivariate statistical technique used for classifying potential metal sources, based on visual summaries of clustering components. Figure 4 presents the dendrogram generated from standardized metal data with Z-scores, employing Ward's method and Euclidean squared distance. The clustering of sediment samples was categorized into two distinct groups (I and II), each further subdivided into two subgroups (A, B, C, and D). The HCA indicates that Group A comprises elements K, Na, and Sr that originated from the same source, likely sharing a common origin related to marine seawater salinity.

Group B comprises elements Mn, Mg, and Ca, which likely originate from carbonate materials within the marine environment. As falls into this category due to its geochemical association with manganese oxides and interaction with carbonate minerals. These elements share common environmental processes, including redox reactions and adsorption mechanisms, which influence their co-occurrence in marine sediments (Penrose & Woolson, 1974). Under oxic conditions, Mn oxides can adsorb and immobilize As but may release As into sediment pore waters during reducing conditions. The major ionic substitution or elemental uptake of Group A, concerning the calcite ratio in carbonate biogenic organisms from Group B, such as Sr/Ca, Na/Ca, and K/Ca ratios, provides insights into seawater chemistry that reflects charge balance and environmental changes, including sea-level fluctuations and CO2 partial pressure (Turchyn & DePaolo, 2019; Zhou *et al*., 2021). Seawater's overall charge balance maintains its chemical stability, where variations in ionic ratios affect charge equilibrium.

Group C comprises elements Al and Fe, which likely constitute major components of crustal rocks and are of authigenic or sedimentary nature containing oxides, while Group D comprises elements Zn, Cr, and Ni, which likely derive from aluminosilicate minerals associated with clays. The Fe/Al weight ratio distribution in global marine sediment composition can estimate whether average crustal rocks originated from continental crust (upper mantle) or oceanic crust (mid-oceanic ridge) based on the database (Hayes *et al*., 2021). In conclusion, Group I associates more closely with marine ecology deposits, such as carbonate minerals or marine salts within sediments, and constitutes major seawater chemistry components, whereas Group II likely derives from lithogenic origins or detrital materials accumulating in marine sediments.

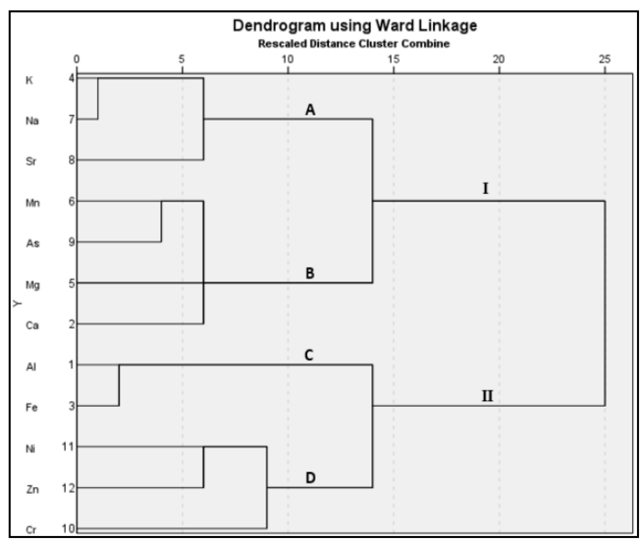

Figure 4: Dendrogram of HCA for the metal contents in marine sediments.

Principal component analysis (PCA)

In this study, PCA was conducted to further elucidate the common potential sources of heavy metals and determine their distribution patterns based on component group similarities. The analysis yielded five PCs generated using the Varimax rotation method, as shown in Figure 5, with eigenvalues exceeding 1, which explained 77.58% of the total variance in the dataset. With an eigenvalue of 3.14 and 26.20% of the variance, PC1 was strongly correlated with K, Na, and Sr. PC2, having an eigenvalue of 2.36 and 19.67% of variance, exhibited strong associations with Al and Fe. PC3 explained 13.49% of variance with an eigenvalue of 1.62, contributing to highly positive factor loadings for Mn and As, and moderately positive factor loadings for Ca and Mg. PC4 explained 9.61% of the variance with an eigenvalue of 1.15 and showed positive correlations with Ni and Zn. The final component, PC5, explained 8.62% of variance with an eigenvalue of 1.04 and demonstrated strong negative factor loadings for Cr.

The findings from these PCAs and their eigenvalues align closely with the results obtained from HCA and Pearson Correlation analyses, thus reinforcing the validity of the interpreted potential heavy metal sources discussed above. These multivariate statistical analyses emphasize the significance of loading factors for environmental assessment and management of heavy metals, as well as determining marine sediment quality in the local region. Since heavy metal behavior in marine sediments represents a dynamic process regulated by various physicochemical factors, identifying their potential sources is crucial for understanding the distribution and patterns of metal inputs in the study area.

Heavy metals comparison study

The baseline study of heavy metal composition in the marine sediments of Pulau Layang-Layang is crucial for establishing natural background metal levels in a relatively pristine environment. Comparing heavy metal compositions between this study and other local regions could assess the current status of heavy metals in marine sediments throughout Malaysia. Since Pulau Layang-Layang

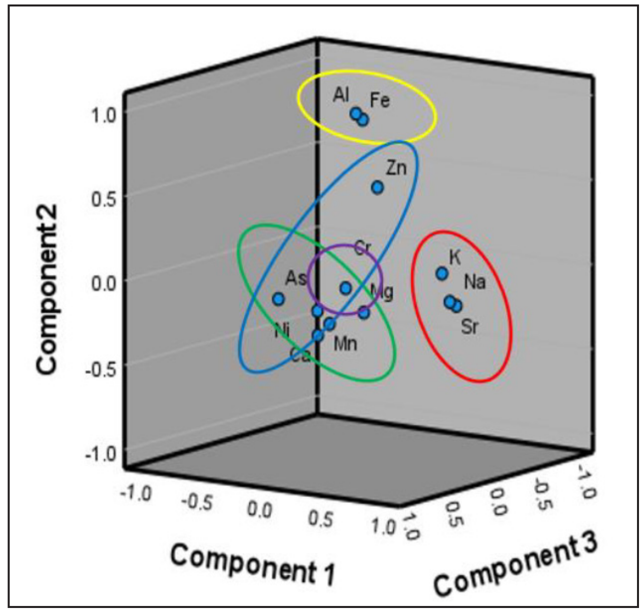

Figure 5: Three-dimensional scatter point load diagram based on all PCs generated using the Varimax rotation method with Kaiser normalization.

Table 3: Comparison of the heavy metal concentrations (mg/kg) obtained from the present study with other coastal areas or small islands around Malaysia.

Area	Concentrations (mg/kg)					Reference
	As	Cr	Mn	Ni	Zn	
Pulau Layang-Layang	2.1-6.5	1.2-8.3	6.1-35.7	0.2-2.5	3.5-12.7	Present Study
Kudat Coast	-	4.1-14.0	31.0–69.0	0.7-8.2	3.5-9.3	[1]
Kota Belud Coast	-	8.2-110.0	100.0–540.0	9.1-230.0	19.0-51.0	[1]
Pulau Mantanani	-	5.0-14.0	13.0–26.0	0.1-6.0	8.0-39.0	[1]
Kudat – Pulau Jambongan	3.0-30.0	2.0-250.0	19.0-1,061.0	1.0-340.0	2.0-59.0	[2]
Pulau Jambongan -Tanjung Labian	3.0-15.0	13.0-57.0	61.0-630.0	5.0-58.0	7.0-62.0	[2]
Darvel	3.0-10.0	6.0-180.0	94.0-1,032.0	3.0-128.0	6.0-73.0	[2]
Sarawak Offshore	3.0-60.0	15.0-164.0	101.0-1,932.0	5.0-260.	7.0-85.0	[2]
East Coast Peninsular Malaysia Offshore	3.0-40.0	3.0-68.0	5.0-4,550.0	1.0-144.0	1.0-2,760.0	[2]
ERL	**8.2**	**81.0**	**-**	**20.9**	**150.0**	**[3]**
ERM	**70.0**	**370.0**	**-**	**51.6**	**410.0**	**[3]**

[1] Ling *et al.* (2023a), [2] Sulaiman & Sharaani (2020), [3] Buchman (2008);
ERL: Effects range low, ERM: Effects range median

remains largely unaffected by anthropogenic inputs, the study yields reliable natural background values for heavy metal concentrations, serving as a reference for evaluating human impact in other regions. From Table 3, the results indicate that the concentrations of all heavy metals (As, Cr, Mn, Ni, and Zn) in Pulau Layang-Layang are consistently lower than in other areas.

The comparison also identifies regions with elevated heavy metal concentrations, such as the highest As levels (ranging from 3.0 mg/kg to 60.0 mg/kg) found in Sarawak offshore, while Mn (ranging from 5.0 mg/kg to 4,550.0 mg/kg) and Zn (ranging from 1.0 mg/kg to 2,760.0 mg/kg) were detected along the East Coast of Peninsular Malaysia. Cr (ranging from 2.0 mg/kg to 250.0 mg/kg) and Ni (ranging from 1.0 mg/kg to 340.0 mg/kg) were found around Kudat to Pulau Jambongan. These elevated concentrations were predominantly between the ERL/ERM limits (for As and Cr) or exceeded them (for Ni and Zn), indicating potential risks of adverse biological effects requiring immediate intervention. Thus, this comparative study identifies environmental concerns warranting enhanced monitoring and investigation into metal enrichment sources to mitigate contamination risks.

The comparative analysis of heavy metals, specifically Cr, Mn, Ni, and Zn, in the marine sediments of Pulau Layang-Layang and Pulau Mantanani reveals significant geochemical similarities. Both islands comprise primarily carbonate materials and experience minimal anthropogenic influence, reflecting natural background levels of these heavy metals (Ling *et al*., 2023a; Ling *et al*., 2023b). These similarities may offer valuable insights into the environmental dynamics of these marine regions, attributable to comparable environmental conditions, sedimentary processes, or similar sources, such as geological background or biological activities. Understanding this relationship is essential for future sediment provenance studies, as it may elucidate the origins of depositional materials and historical geological processes. Additional research should examine the complete geochemical profile of Pulau Mantanani and other pristine small islands off Sabah's west coast. Such investigations would enable more comprehensive correlations and deeper understanding of the region's geochemical and environmental history with Pulau Layang-Layang.

CONCLUSION

As a concluding analysis, this study establishes a baseline for heavy metal compositions in the marine sediments of Pulau Layang-Layang and identifies their potential sources within pristine environments. The SEM analysis revealed the presence of biogenic carbonate materials, which serve a crucial role in sediment composition. The ICP-OES analysis demonstrated a decreasing order of mean metal concentrations: Ca>Mg>Na>Sr>K>Fe>Al>Mn>Zn>As>Cr>Ni. The minimal levels of these metals indicated natural background values, with multivariate statistical analyses, including Pearson correlation, HCA, and PCA, suggesting that these metals primarily originated from natural sources such as geological composition and marine biological processes. The heavy metal concentrations consistently remained below the ERL/ERM thresholds established by NOAA's SQuiRTs, and the sediment quality guidelines further confirmed a healthy marine ecosystem in Pulau Layang-Layang. Thus, these natural background values of heavy metals provide essential reference data for future monitoring and comparison of heavy metal concentrations with other Bornean sedimentary islands or along Malaysia's coastline. Establishing these baseline measurements is vital for detecting any variations caused by environmental changes or anthropogenic activities, thereby supporting the conservation and management of these marine ecosystems.

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AUTHORS CONTRIBUTION

The first author performed laboratory and statistical analysis based on the geochemical data. The first author also wrote the manuscript, and all other authors contributed significantly to its final draft. The corresponding author planned and designed the experiments and interpreted the data. All authors conducted the geological mapping and collected relevant samples.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest relevant to the content of this manuscript.

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