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# Potential Sources and Contamination Levels of Pb and Ni in Surface Sediment of Lampung Bay, Indonesia

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Sediment in Lampung Bay has been reported to contain heavy metals, but information on their sources and the level of contamination is limited. This study aims to estimate the sources of lead (Pb) and nickel (Ni) and the extent of their contamination. Five locations were chosen to collect sediment samples, and heavy metal concentrations were determined by geochemical partitioning using the Sequential Extraction Procedure Bureau Commune de Reference of the European Commission (SEP BCR) method. The results showed that the total Pb concentration ranged within 12.48–24.46 mg/kg, dominated by the residual fraction (9.62–23.74 mg/kg) compared with the non-residual fraction (0.21–0.55 mg/kg). The total Ni concentration ranged within 7.48–13.70 mg/kg, also dominated by the residual fraction (2.95–7.96 mg/kg) compared with the non-residual fraction (2.54–5.75 mg/kg). These results have not exceeded the quality standards set by the Australian and New Zealand Environment and Conservation Council (ANZECC). The dominance of the residual fraction in both metals suggests that natural sources are the primary origin. The level of contamination was assessed using the enrichment factor (EF), geo-accumulation index (Igeo), contamination factor (CF), and pollutant load index (PLI). In general, the sediments of Lampung Bay have been contaminated and enriched by Pb and Ni. However, the enrichment remains harmless to organisms and the aquatic environment. The SEP BCR method can be used to determine the partitioning of heavy metals in marine sediments and estimate the sources of natural or anthropogenic inputs, making it a valuable tool for initial monitoring related to assessing the sources and extent of heavy metal contamination in marine sediments.

**Keywords:** contamination level, heavy metal, Lampung Bay, SEP BCR, surface sediment.

## Introduction

It is believed that heavy metals in the aquatic environment derive from natural processes and anthropogenic activities (Gautam et al., 2016). They are part of various mineral rocks in the Earth's crust (Briffa et al., 2020). In addition, they are released through metal-containing waste of mining, industry, ports, and land use activities (Schmidt et al., 2022). All of those are subsequently transported through rivers into marine environment, deposited and accumulated in sediment. Therefore, sediments can be used for long-term monitoring of possible changes of the environmental status.

The onshore of Lampung Bay is a developed area in which resort areas, mining, ports, and industry are established, and hence densely populated settlements and the change of land use (Kamil et al., 2021). These activities have affected the ecological equilibrium, such as reducing the environmental quality of Lampung Bay (Kamil et al., 2021). It has been reported that the concentration of lead (Pb) in sediment has exceeded the standard shown in the Australian and New Zealand Environment and Conservation Council (ANZECC) (2000), and also in organisms such as *Perna viridis* L. (Safitri et al., 2018) and *Anadara granosa* Linn (Rahmah et al., 2019). However, information about metal mobility and bioavailability is limited. This could further understand such as metal sources of origin, availability in organisms, and the impact of biota-environment interactions. Geochemical partitioning is a method that has been used to determine the sources of heavy metals, together with their mobility and bioavailability (Liang et al., 2018). The Australian and New Zealand Environment and Conservation Council (SEP BCR) multistage extraction method employs four bonded fractions to assess the sources of heavy metals: the acid-soluble fraction (F1), the reduced fraction (F2), the oxidized fraction (F3), and the residual fraction (F4) (Passos et al., 2010). Furthermore, F1, F2, and F3 are categorized as labile non-residual fractions that have been shown to be effective in predicting metal bioavailability and speciation (Pearson, 2017), while F4 is a resistant fraction strongly associated with minerals, very stable and uneasily absorbed by biota (Kouassi et al., 2019). Several studies have successfully applied this strategy, including those involving soil samples from Erciyes University Campus, Turkey (Tokaloğlu et al., 2010), soil and regolith in the Assaluyeh region, southwest Iran

(Alimohammad et al., 2012), surface sediments from the Chinese continental shelf (Yanli et al., 2018), surface sediment from North Sumatera, Indonesia (Yolanda et al., 2019), and sediment from the Gohar Rood River, Iran (Ashayeri et al., 2023). Therefore, using the same approach, this study focused on examining the potential sources and levels of Pb and Ni contamination in the sediments of Lampung Bay.

## Methods

### Time and place of research

Sampling was carried out in Lampung Bay at five locations from the head to the outlet of the bay (Fig. 1), and, bathymetrically, the water depth varies from shallow to 30 m. These stations represented deposition environments possibly affected by river mouths (TL 1), close to the head of the bay (TL 2), and possibly mixing processes of the bay (TL 3 to TL 5).

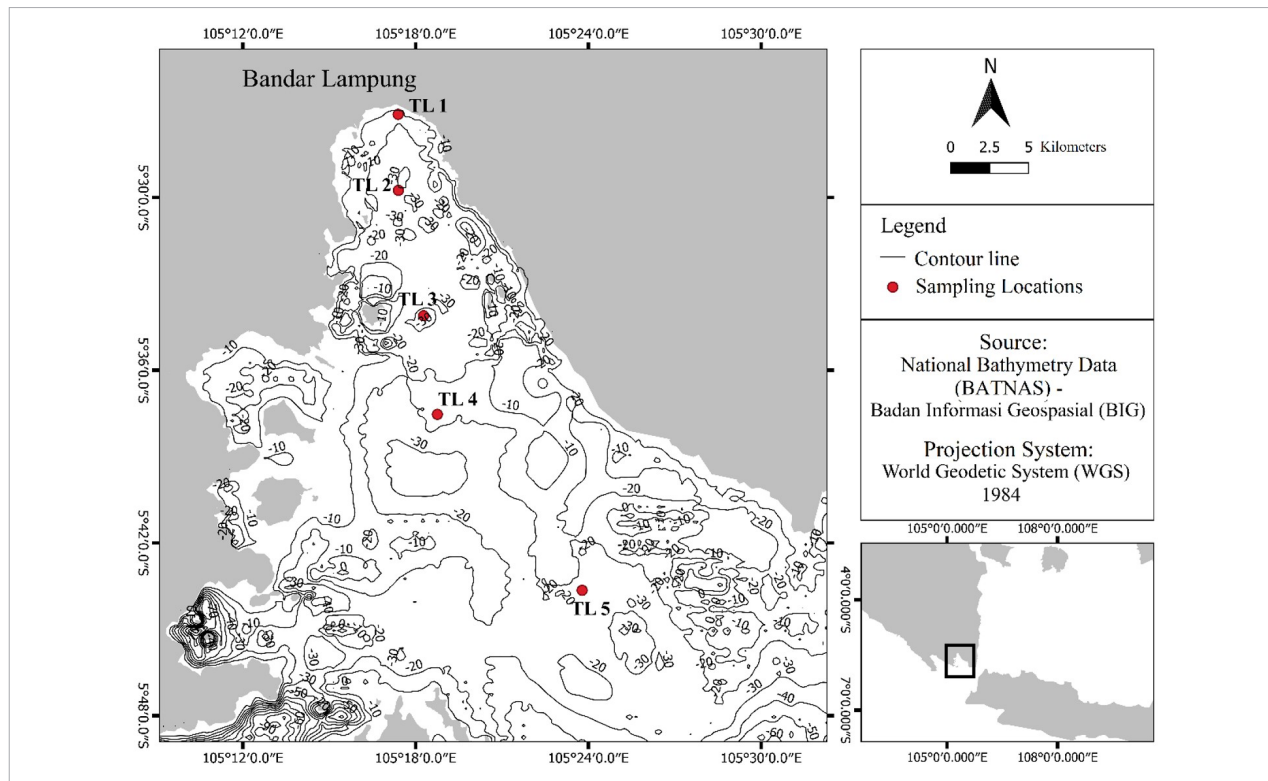
### Geochemical partitioning analysis of heavy metals

The geochemical partitioning of heavy metals was performed using the SEP BCR multistage extraction method, which was verified using the BCR-701 sediment-certified reference material (Passos et al., 2010). The method employed four phases of geochemical fractionation (Kouassi et al., 2019). Sequential extraction used 1 g of a dried sediment sample based on a constant weight at 60°C.

The acid-soluble fraction stage (F1) is metal-exchangeable or associated with carbonates. This fraction was extracted by adding 40 mL of 0.11 mol/L acetic acid ( $\text{CH}_3\text{COOH}$ ) to 1 g of a dried sediment sample in a polyethylene centrifuge tube. The samples were stirred at  $30 \pm 10$  rpm for 16 hours at room temperature ( $22 \pm 5^\circ\text{C}$ ). The extract was separated from the solid phase by centrifuging at 3000 rpm for 20 minutes. The supernatant was placed in polyethylene tubes for heavy metal analysis.

The reduced fractional stage (F2) is a metal associated with Fe and Mn oxides. This fraction was extracted by adding 40 mL of 0.1 mol/L hydroxylamine hydrochloride ( $\text{NH}_2\text{OH}\cdot\text{HCl}$ ) (pH 1.5) to the residue of the acid-soluble fraction (F1). The samples were stirred at a speed of  $30 \pm 10$  rpm for 16 hours at room temperature ( $22 \pm 5^\circ\text{C}$ ). The extract was separated from the solid

Fig. 1. Map of sampling locations and bathymetry of Lampung Bay



phase by centrifugation at 3000 rpm for 20 minutes. The supernatant was placed in polyethylene tubes for heavy metal content measurement.

The oxidized fraction (F3) consists of metals with organic and sulfide properties. At this stage, the fraction (F2) residue was added to 10 mL of 8.8 mol hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), which was then left at room temperature for 1 hour. The sample was subsequently heated in a water bath at  $85 \pm 2^\circ\text{C}$  for 1 hour. After the solution volume was reduced by roughly 2–3 mL, 10 mL of 8.8 mol  $\text{H}_2\text{O}_2$  was added. The mixture was heated in the water bath for 1 hour at  $85 \pm 2^\circ\text{C}$ . After cooling 50 mL of ammonium acetate ( $\text{NH}_4\text{CH}_3\text{CO}_2$ ), the solution (pH 2, 1.0 mol/L) was added and stirred at  $30 \pm 10$  rpm for 16 hours at room temperature ( $22 \pm 2^\circ\text{C}$ ). The samples were centrifuged at 3000 rpm for 20 minutes, and the supernatant was decanted in the tube to determine the heavy metal content.

F4 is the fraction that bonds to the mineral crystal structure. This strong fraction was extracted from oxidized fraction (F3) residue by slowly mixing with 10 mL of nitric acid ( $\text{HNO}_3$ ) (1:1). The samples were refluxed

at  $95 \pm 5^\circ\text{C}$  for 10–15 minutes. After cooling, 5 mL of concentrated  $\text{HNO}_3$  was added to that solution sample, heated at  $95 \pm 5^\circ\text{C}$  for 30 minutes, and then left cooling at room temperature. The sample was re-heated at  $95 \pm 5^\circ\text{C}$  with 5 mL of concentrated  $\text{HNO}_3$  for 2 hours, or until a volume of 2–3 mL remained. After cooling, the sample solution was subsequently stirred and 2 mL of Aquabides was added, followed by the addition of 9 mL of  $\text{H}_2\text{O}_2$ . The samples were then heated at  $95 \pm 5^\circ\text{C}$  for 2 hours. Finally, 10 mL of hydrogen chloride (HCl) solution was added, heated at  $95 \pm 5^\circ\text{C}$  for 15 minutes, and cooled. The obtained samples were filtered using 0.45  $\mu\text{m}$  filter paper and diluted in distilled water to a volume of 50 mL. All these fractions were measured using a Flame Atomic Absorption Spectroscopy (FAAS) for Ni (detection limit: 0.1 mg/kg) and a Graphite Furnace Atomic Absorption Spectrometry GFAAS for Pb (detection limit: 0.04  $\mu\text{g}/\text{kg}$ ).

### XRD analysis

XRD (X-ray diffraction) analysis was used to characterize the crystal structure of sediment minerals. Sediment samples were dried and crushed using a mortar.

The samples were analyzed using an Empyrean Series 3 Panalytical XRD.

### Data analysis

The equation used to calculate the enrichment factor (EF) is (Dehno et al., 2022):

$$EF = \frac{(\text{metal/Al})_{\text{sediment}}}{(\text{metal/Al})_{\text{reference}}} \quad (1)$$

Metal/aluminium (Al) sediment is the ratio of concentrations (Pb and Ni) determined from the measured samples, and Al deriving from the closest representative areas. The metal/Al reference refers to the concentrations used as the background value of Pb (12.5 mg/kg), Ni (75 mg/kg) and Al (8230 mg/kg) (Taylor, 1964). EF value categories are  $EF \leq 1$  for no enrichment,  $1 < EF \leq 3$  for minor enrichment,  $3 < EF \leq 5$  for moderate enrichment,  $5 < EF \leq 10$  for moderately heavy enrichment,  $10 < EF \leq 25$  for heavy enrichment,  $25 < EF \leq 50$  for very heavy enrichment, and  $EF > 50$  for extraordinary enrichment (Ho et al., 2010).

The equation used to calculate the geo-accumulation index ( $I_{\text{geo}}$ ) is as follows (Jaworska and Klimek, 2021):

$$I_{\text{geo}} = \log_2 \left[ \frac{C_n}{1.5 B_n} \right] \quad (2)$$

where  $C_n$  is the measured heavy metal concentration,  $B_n$  is the background concentration. A constant of 1.5 is used to control the variation of  $B_n$  caused by the environment.  $I_{\text{geo}}$  categories are  $I_{\text{geo}} < 0$  for unpolluted,  $0 \leq I_{\text{geo}} < 1$  for unpolluted to moderately polluted,  $1 \leq I_{\text{geo}} < 2$  for moderately polluted,  $2 \leq I_{\text{geo}} < 3$  for moderately to strongly polluted,  $3 \leq I_{\text{geo}} < 4$  for strongly polluted,  $4 \leq I_{\text{geo}} < 5$  for strongly to very strongly polluted, and  $I_{\text{geo}} \geq 5$  for very strongly polluted (Ho et al., 2010).

The equation used to calculate the Contamination Factor (CF) is as follows (Zhao et al., 2021):

$$CF = \frac{C_n}{B_n} \quad (3)$$

CF categories are  $CF < 1$  for low CF,  $1 \leq CF < 3$  for medium CF,  $3 \leq CF < 6$  for moderately large CF, and  $CF \geq 6$  for very large CF (Khan et al., 2020).

The equation that can be used to calculate the pollutant load index (PLI) is as follows (Negrin et al., 2018):

$$PLI = \sqrt[n]{CF_1 \times CF_2 \times CF_3 \times \dots \times CF_n} \quad (4)$$

where  $n$  is the number of heavy metals analyzed. PLI values  $< 1$  indicate that the heavy metal load is close to the background value or has not been polluted, while PLI values  $> 1$  indicate that pollution has occurred (Negrin et al., 2021).

### Quality control and quality assurance

Sampling and sample handling were carried out using protocols of the laboratory, covering sampling and measuring metal concentrations. Sampling in the field was performed with caution to ensure that potential sources of contamination from equipment or vessels are avoided. All samples were stored in plastic-material made containers and kept in the refrigerator during transportation to the laboratory. The laboratory procedure of metals was also tested showing recovery rates ranging from 93% to 105%.

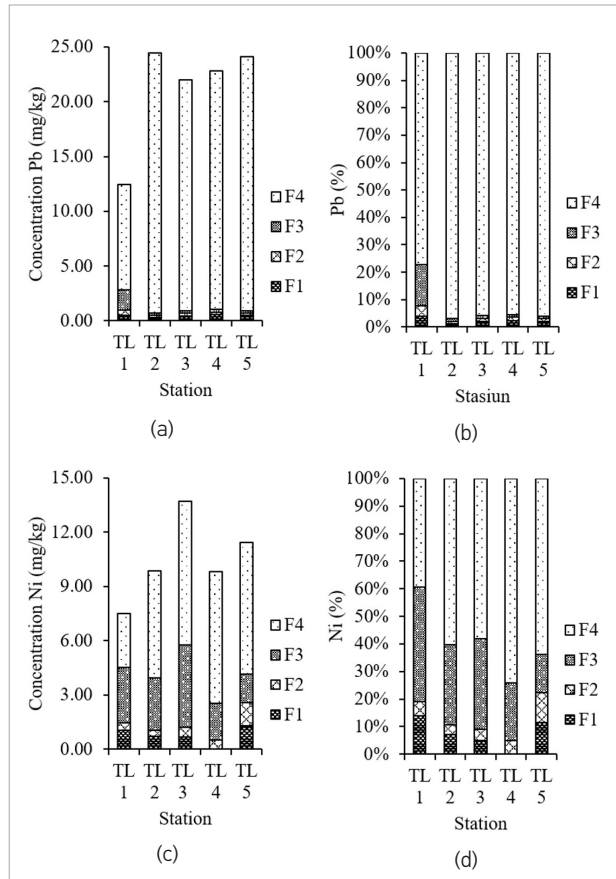
## Results and Discussion

### Sources of heavy metals

Pb concentrations ranged within 12.48–24.46 mg/kg and were mostly higher than those of Ni (7.48–13.70 mg/kg, Fig. 2). In comparison with the other stations, the lowest concentration of Pb was found in TL 1 station. Pb concentration in the sediment was generally dominated by F4, ranging within 9.62–23.74 mg/kg (77.12–97.02%). The non-residual fraction ( $F_1 + F_2 + F_3$ ) varied in the range of 0.21–0.55 mg/kg (2.98–22.28%). The highest non-residual fraction was found at TL 1 station located close to the river's mouth in which anthropogenic inputs occurred. Nonetheless, the predominantly non-residual fraction compared with the non-residual counterpart probably indicated more anthropogenic sources rather than natural sources. Pb from anthropogenic sources apparently has been detected at each location. Anthropogenic sources of Pb might derive from domestic and industrial waste such as crumb rubber, coal, industrial chemicals, and ships. The Ni fractions were also predominantly associated in F4 ranging within 2.95–7.96 mg/kg (39.42–74.09%), except the one observed in TL 1 (non-residual fraction of 60.58%). This indicates more anthropogenic sources, while the residual Ni fractions observed in TL 2 to TL 5 probably derived from natural processes.

The extent to which metals interact with sediment particles / suspended solids was determined by the

**Fig. 2.** Concentration Pb (mg/kg) (a), percentage geochemical partitioning Pb (%) (b), concentration Ni (mg/kg) (c), and percentage geochemical partitioning Ni (%) (d) of Pb in sediments from Lampung Bay in the acid-solubilized fraction (F1), the reduced fraction (F2), the oxidized fraction (F3), and the residual fraction (F4)



sediment geochemical fractions, i.e. the acid-soluble fraction (F1), the reduced fraction (F2), the oxidized fraction (F3), and the residual fraction (F4). The strong sequence of metal bonds in those materials is  $F1 < F2 < F3 < F4$  (Miranda et al., 2021). F1 is unstable, reactive,

and weakly bound (exchangeable) to sediments associated with carbonate. This fraction is sensitive to environmental changes and easily desorbed or absorbed (Passos et al., 2010). F2 is the metal associated with oxides and unstable under a reducing environment in which metal could be released. F3 is associated with organic materials and metals could release back into the water column during oxidation (Giraldo, 2018). Metal associated F4 fraction is the strongest bond since they are part of the crystalline mineral matrix in which metals are transported and deposited into the sediment. Therefore, metals in the residual fraction F4 are possibly categorized as natural sources. In contrast, the non-residual fraction (F1, F2, and F3) contains metals removed from the solution in association with either inorganic or organic hosts. The metals in the solution are probably derived from land-based sources, in particular industrial, mining, fuel combustion from transportation. In the onshore of Lampung Bay, industrial companies dispose of waste through the watershed (Pratiwi et al., 2016). In addition, many activities potentially contribute to metal discharge on the bay, and they include domestic waste, harbors, shipping activities, and industries such as crumb rubber, coal, and chemical industries. It was clear that Pb was more residual, whereas Ni tended to be more non-residual, suggesting that Pb and Ni were different sources of origin.

The compositions of geochemical partition for Pb and Ni from various studies are compared in Table 1. The average percentage of the acid-soluble fraction (F1) of Pb in Lampung Bay was lower than that observed in Gohar Road, but higher than that found in Ambon Bay and Gulf of Gabes. The mean percentage of the residual fraction (F4) of Pb was higher compared with Ambon Bay, the Gulf of Gabes, and the Gohar Road River. The reduced fraction (F2) of Ni in Lampung Bay sediments was lower than in the other stations. This also suggests

**Table 1.** Average percentage of geochemical partitioning of Pb and Ni (%) in sediments in several studies

Location	F1		F2		F3		F4		Reference
	Pb	Ni	Pb	Ni	Pb	Ni	Pb	Ni	
Lampung Bay, Indonesia	2.4	7.5	1.6	5.7	3.8	27.6	92.3	59.1	Present study
Ambon Bay, Indonesia	nd	12.0	80.0	11.0	10.0	12.0	10.0	66.0	Lestari et al., 2019
Gulf of Gabes (Southern Mediterranean Sea)	1.0	1.4	45.0	16.2	0.7	23.5	53.2	58.8	Chifflet et al., 2019
Gohar Road River, Iran	5.1	13.1	23.7	12.0	14.9	6.5	56.3	68.5	Ashayeri et al., 2023

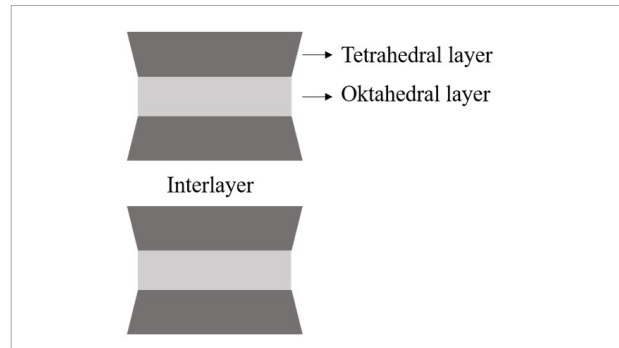
that differences in the crystal structure of sediment minerals and pollutant inputs were responsible for their variation in the marine environment concerned.

At low tide, pollutants such as heavy metals that entered and were near the head of the bay could be carried out of the bay, while at high tide, they could carry materials originating from Sunda Sunda and those at the mouth of the bay into the waters of Lampung Bay (Milasari et al., 2021). Based on the M2 residual current pattern near the bottom of the water and bathymetry, in the section near the head of the bay, there was a small basin that caused the formation of a counterclockwise eddy (Koropitan et al., 2004), so it was estimated that it could have influenced the entry of heavy metals into the Lampung Bay water environment. The presence of eddies and small basins was expected to have a long-term influence on the accumulation of heavy metals in the area.

The sand fraction ranged within 3.00–3.81%, the silt fraction ranged within 75.95–78.73%, and the clay fraction ranged within 18.27–20.28%, so it was estimated that, in general, the silt fraction was more dominant than the sand and clay fractions (Kusumaningsih, 2024). The sediment fraction could affect the elemental bonds present, and the adsorption process was influenced by the specific surface area of the sediment; the larger the specific surface area of the adsorbent, the higher the amount of substance that could be adsorbed. The fine sediment fraction could adsorb more heavy metals compared with the sand fraction because the fine sediment fraction had a larger specific surface area and more organic matter that could aid in controlling heavy metal binding (Khan et al., 2020).

Clay minerals play a crucial role in heavy metal adsorption (Gupta and Bhattacharyya, 2009). According to the results of the XRD examination, the minerals identified in the sediments of Lampung Bay were chloritoid (46.5%) and montmorillonite (53.5%). Chloritoids and montmorillonite could adsorb heavy metals, lowering their concentration in water (Gupta and Bhattacharyya, 2009). F4 was a fraction attached to the mineral crystal structure and was located within the mineral crystal structure, making it difficult to desorb. When minerals flowed through areas containing organic matter and carbonates, these components adhered to the mineral interlayer (Fig. 3). F1, F2, and F3 are expected to represent metals from anthropogenic sources, while F4 is anticipated to represent metals from natural sources. This is because the metals in F4 are more tightly bound

**Fig. 3.** Tetrahedral, octahedral, and interlayer structure of montmorillonite



within the mineral crystal structure, making them less likely to be released. Therefore, source estimation can be conducted using these geochemical partitions.

### Pb and Ni contamination level

In general, Pb had a higher EF than Ni (Table 2). The EF of Pb ranged from 0.67 to 1.54. The EF category at TL 2 was  $Pb \leq 1$ , indicating that there was no enrichment. In contrast, at TL 1, TL 3, TL 4, and TL 5, there was a small enrichment ( $1 < EF \leq 3$ ). The EF of Ni ranged from 0.04 to 0.15 and was commonly classified in the  $EF \leq 1$  category, indicating that there was no enrichment or that any enrichment was negligible. Generally, the EF values of Pb and Ni were more enriched near the bay's center or mouth. However, a decrease in EF occurred at TL 2, which was consistent with the high concentration of total Pb and Ni that increased near the bay's mouth.

Igeo Pb ranged from -0.59 to 0.38. TL 1 had relatively low values, whereas the remaining stations were generally similar. Overall, the presence of Pb in the sediment was classified as unpolluted to moderately contaminated ( $0 \leq I_{geo} < 1$ ). Igeo Ni appeared to be lower

**Table 2.** Enrichment factor (EF), geo-accumulation index (Igeo), contamination factor (CF), and pollutant load index (PLI) values

Station	EF		Igeo		CF		PLI	
	Pb	Ni	Pb	Ni	Pb	Ni	Pb	Ni
TL 1	1.33	0.12	-0.59	-4.10	1.00	0.09	1.65	0.13
TL 2	0.67	0.04	0.38	-3.63	1.96	0.12		
TL 3	1.54	0.15	0.23	-3.16	1.76	0.17		
TL 4	1.42	0.10	0.28	-3.60	1.83	0.12		
TL 5	1.44	0.11	0.36	-3.41	1.93	0.14		

than Pb, with values ranging from  $-4.10$  to  $3.60$ . Similar to Ni, Igeo Pb was also lowest at TL 1. In general, Igeo Ni fell into the Igeo  $< 0$  category, indicating that it was not polluted. Igeo Pb and Ni levels generally increased at the bay's mouth. Pb was in the same group at TL 2 to TL 5 (not polluted to moderately polluted), while TL 1 remained not polluted. Furthermore, Ni remained unpolluted at all stations; however, the Igeo value rose as one approached the bay's mouth.

The CF Pb ranged from 1 to 1.96. The presence of Pb was classified as unpolluted to moderately polluted ( $1 \leq CF < 3$ ). CF Ni was lower than Pb, ranging from 0.09 to 0.17. CF Ni was commonly categorized as CF  $< 0$ , suggesting it was not polluted. CF Pb and Ni often followed the same pattern, increasing towards the bay's center or mouth. According to the Pb and Ni contamination levels analysis, the value increased towards the center or mouth of the bay.

The PLI value for Ni was 0.13, and for Pb, it was 1.65. Ni was included in the PLI  $< 1$  category, indicating that the sediments in Lampung Bay had not been contaminated by Ni. In contrast, Pb was categorized in the PLI  $> 1$  category, indicating that the sediments had been polluted by Pb.

Based on the level of contamination, Pb experienced enrichment to moderate contamination, while Ni did not. EF Pb generally had a value of  $< 2$ , but at TL 2, it was  $< 1$ . This indicated that the higher the enrichment, the closer one was to the bay's center. It was presumed that the reference metal concentration (Al) had an influence. Based on the analysis, Al concentrations ranged from 6158.04 to 24 083.85 mg/kg. At TL 2, the EF value was  $< 1$ . This was driven by the high Al concentration at the location, which was 24 083.85 mg/kg. Although there was an increase in Pb and Al concentrations at TL 2, it was still apparent that the enrichment was minimal. In line with the EF of Pb, the EF of Ni at TL 2 was lower than at other stations, despite the fact that Ni at TL 2 was higher than that at TL 1. This was also expected due to the influence of the normalization of the reference metal, Al, which had a comparatively higher concentration compared with at other stations.

The Igeo of Pb suggested that the Igeo value increased as one moved closer to the bay's center. The value  $< 1$  indicated that the area was not contaminated to moderately polluted. TL 1 had a value of  $< 0$ , expected due to the low total concentration of Pb compared with other stations. The difference in categories between Igeo and EF was attributed to the 1.5 factor in the Igeo

calculation, which permitted the inclusion of natural fluctuations in the concentration of certain substances in the environment and very small anthropogenic influences (Jaworska and Klimek, 2021). The Igeo of Ni was generally  $< 0$ , as the measured Ni concentration was lower than the background value used.

Generally, CF Pb had a value of  $\geq 1$ , indicating that it was unpolluted to moderately polluted. In contrast, CF Ni was generally  $< 1$ , implying it was unpolluted. This was expected because the Pb concentration in the measurements was higher than the background value (12.5 mg/kg), while the Ni concentration at each site remained below the background level. PLI was determined by the value of CF, and it was expected that as the value of CF rose, so would PLI.

The total concentration of Pb and Ni at TL 1 was still lower than at other stations, but the non-residual fraction was higher. The non-residual percentage could indicate high bioavailability and mobility. The degree of Pb and Ni pollution that occurred was still harmless for marine biota and the aquatic environment. However, TL 1 had a higher Pb non-residual fraction percentage than at other stations. The Ni non-residual fraction at TL 1 was also more dominant than the residual fraction, as was the case at TL 3. As a result, Pb and Ni at TL 1 and TL 3 were thought to have greater mobility and bioavailability than at other stations.

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## Conclusions

Low enrichment of Pb and Ni in the surface sediments of Lampung Bay was discovered. Pb had a higher percentage of the residual fraction than Ni, indicating that the input sources of Pb and Ni in Lampung Bay waters were still dominantly natural sources, primarily diagenesis processes associated with natural conditions. However, the station near the head of the bay and the mouth of the river (TL 1) already had a higher non-residual fraction compared with the stations in the middle or towards the mouth of the bay. Based on the total concentrations of Pb and Ni, they did not exceed the ANZECC (2000) quality standards. The Pb and Ni contamination was at a low to moderately polluted level, with a bioavailability level that was also low, indicating a minimal potential for contamination of biota. This study was limited to surface sediments, so further research on the accumulation rate by examining sediment cores is necessary.

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