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Evaluation of Nickel, Copper, and Cadmium Contamination in Sediments from The Waters of Mare Island, North Maluku

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ABSTRACT

Particularly in the waters of Tinangkung, Central Sulawesi, the management of seaweed through marine Mare Island has been appointed as a conservation area and tourist park due to its seagrass, coral reef, and mangrove ecosystems. However, intensive nickel mining activities in Maluku have the potential to contaminate the marine environment. Heavy metals resulting from these activities are toxic and harmful to both human and marine biota health. Monitoring of the marine environment is important through sediment quality assessment. Therefore, this study aims to evaluate contamination of Ni, Cu and Cd in sediments from the waters of Mare Island, using verified testing methods. The sediment samples were wet-digested using aqua regia on a hotplate and analyzed using flame atomic absorption spectrophotometer (FAAS). The testing method was verified according to AOAC guidelines, covering linearity, LOD, LOQ, recovery, and repeatability. The results show that the concentrations of Ni, Cu, and Cd in the waters of Mare Island ranged from 23.30–29.30, <0.51–5.21 and 6.20–7.94 mg/kg dw, respectively. The Cu concentration is still within safe limits, while Ni and Cd concentrations are categorized as hazardous, posing a risk to marine biota. Based on Contamination Factor, Enrichment Factor, Pollution Load Index, and Potential Ecological Risk Index values, the waters of Mare Island are classified as polluted, originating from anthropogenic activities.

Keywords: *Heavy metals, Assessment of contamination, Mare island, Sediment, Verification of method.*

ABSTRAK

Pulau Mare telah ditetapkan sebagai kawasan konservasi dan taman wisata karena ekosistem lamun, terumbu karang, dan mangrovenya. Namun, aktivitas penambangan nikel yang intensif di Maluku berpotensi mencemari lingkungan laut. Logam berat yang dihasilkan dari aktivitas tersebut bersifat toksik dan berbahaya bagi kesehatan manusia maupun biota laut. Pemantauan lingkungan laut penting dilakukan melalui penilaian kualitas sedimen. Oleh karena itu, penelitian ini bertujuan untuk mengevaluasi pencemaran Ni, Cu, dan Cd pada sedimen dari perairan Pulau Mare dengan menggunakan metode pengujian yang telah diverifikasi. Sampel sedimen didestruksi secara basah menggunakan aqua regia pada penangas listrik dan dianalisis menggunakan spektrofotometer serapan atom (SSA). Metode pengujian diverifikasi berdasarkan pedoman AOAC, mencakup linearitas, LOD, LOQ, *recovery*, dan *repeatability*. Hasil penelitian menunjukkan bahwa konsentrasi Ni, Cu, dan Cd di perairan Pulau Mare masing-masing berkisar antara 23,30–29,30; <0,51–5,21; dan 6,20–7,94 mg/kg dw. Konsentrasi Cu masih berada dalam batas aman, sedangkan konsentrasi Ni dan Cd tergolong berbahaya sehingga berpotensi menimbulkan risiko terhadap biota laut. Berdasarkan nilai *Contamination Factor*, *Enrichment Factor*, *Pollution Load Index*, dan *Potential Ecological Risk Index*, perairan Pulau Mare diklasifikasikan sebagai tercemar yang berasal dari aktivitas antropogenik.

Kata kunci: Logam Berat, Penilaian Pencemaran, Pulau Mare, Sedimen, Verifikasi Metode

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1. Introduction

Mare Island is a small volcanic island located in the City of Tidore Islands, North Maluku. The population on Mare Island is distributed across two villages, namely Marikofo with 509 people and Maregam with 461 people (Sukowati et al., 2023). Mare Island is home to seagrass, coral reef, and mangrove ecosystems, which serve as natural habitats for marine biota. The island's natural beauty and biodiversity have led to the waters of Mare Island as a conservation area and tourist park, based on the Ministry of Maritime Affairs and Fisheries Decree No. 66 of 2020 (Ministry of Marine Affairs and Fisheries of the Republic of Indonesia, 2020). Anthropogenic activities, such as coastal settlements, ship ports, Diesel Power Plants, as well as mining and industrial activities operating around Mare Island, have the potential to cause pollution. Therefore, proper and wise environmental management is crucial to maintain the sustainability of Mare Island's conservation area.

In the assessment of environmental pollution, the testing method to be used for identifying pollutants must first be confirmed to ensure that it can effectively be applied to real laboratory conditions. Performance evaluation, limitations, and factors affecting the standard method can be conducted through method verification. This confirms several aspects such as performance selectivity, specificity, linearity, working range, accuracy, precision, recovery, uncertainty, detection limit, quantification limit, robustness, method stability, and its comparison with other methods (Peris-Vicente et al., 2015).

Nickel contamination has been widely reported across various waters in North Maluku and surrounding regions. Previous studies indicate that Ni levels in Bacan and Morotai Islands generally fall within the lightly contaminated range, with concentrations typically below 53 ppm (Marasabessy et al., 2010; Edward, 2015). In contrast, several locations in Southeast Sulawesi, such as Kendari Bay and Lasolo Bay, show substantially higher Ni accumulation, reaching up to 436 ppm and posing ecological risks (Ahmad, 2013). Other areas, including Muna Island, Kabaena, and Buton, also exhibit elevated but more variable levels, ranging from 3.774 to 155.877 ppm (Ahmad, 2009). The most severe contamination has been documented in Fatufia, Morowali, where Ni concentrations exceed 700 ppm, indicating intense anthropogenic influence (Lestari et al., 2024).

Beyond nickel, several studies have also reported the presence of copper and cadmium contamination in North Maluku and surrounding waters. Moderate levels of Cu and Cd have been observed in Morotai, where concentrations ranged from 12.393 to 74.842 ppm and 0.01 to 0.736 ppm, respectively (Edward, 2015). Similar patterns appear in the waters around Ternate City, although the overall concentrations are comparatively lower (Edward et al., 2020). In contrast, sediments in the waters of Bacan Island contain Cu at higher concentrations, while Cd levels are lower than those reported for Morotai and Ternate (Marasabessy et al., 2010). Evidence of Cd contamination has also been documented in the waters of Tulehu and along the Ambon Bay coastline, with Tulehu exhibiting markedly higher values, reaching up to 1.25 ppm (Natsir et al., 2019; Sukaryono & Dewa, 2018). Meanwhile, Cu enrichment has been recorded at Bastiong Port in Ternate City, although at comparatively lower concentrations relative to other reported sites (Nurhamiddin & Ibrahim, 2018).

Heavy metals such as Ni, Cu, and Cd are inorganic pollutants that are hazardous, non-degradable, and can persist in the environment for a long time (Kiran et al., 2022). In the body, heavy metals can increase free radicals, including Reactive Oxygen Species (ROS), which lead to cancer, genetic mutations, and tissue damage (Mitra et al., 2022). In nature, the concentrations of Ni, Cu, and Cd in sedimentary rocks are 68 ppm, 45 ppm, and 0.3 ppm, respectively (Turekian & Wedepohl, 1961). The presence of these metals in coastal and marine environments can increase due to anthropogenic activities. Anthropogenic sources include agricultural activities, such as the use of fertilizers, manure, and pesticides containing heavy metals, metallurgy activities like mining, smelting, metal finishing, power plants, transportation, electronic products, and waste disposal (Bradl, 2005).

Sediment is a commonly used indicator of marine pollution. Sediment quality is an excellent indicator of marine pollution levels because pollutants accumulate in sediments through complex physical and chemical adsorption mechanisms (Amadi et al., 2018). In sediments, pollutants can undergo

transformation, be stored, or recycled, and interact with the overlying water and/or the biota living in and on the sediments. Sediment is a dynamic and highly complex system influenced by hydrodynamic factors (storms, underwater landslides, bioturbation by resident organisms), physicochemical processes (sorption, redox reactions), and microbial transformations (Chiaia-Hernández et al., 2022). Therefore, sediment quality evaluation is crucial for identifying the level of pollution and potential risks to marine ecosystems and the health of biota.

Based on the above explanation, research is needed to evaluate the contamination of Ni, Cu, and Cd in sediments of the waters of Mare Island using a verified testing method. The objectives of this study are (1) to verify the testing method for heavy metals of Ni, Cu, and Cd in sediments, (2) to determine the concentrations of Ni, Cu, and Cd in sediments from the waters of Mare Island, North Maluku, and (3) to identify the levels, sources, distribution, and pollution risks of these heavy metals.

2. Materials and methods

2.1. Verification of Analysis Method

The verification of the heavy metal testing method using AAS was conducted according to the EURACHEM (Magnusson & Ornemark, 2014) and AOAC (Latimer, 2023) guidelines, including linearity, limit of detection (LOD), recovery, and repeatability. A standard series was prepared with different concentrations, and the acceptance limit for the linearity (r^2) of the AAS calibration curve was > 0.990. The LOD and LOQ were determined by measuring 6-15 blank samples or reagent blanks, or samples with very low analyte concentration. Recovery was performed by spiking the samples with low, medium, and high concentrations of standard analyte. Recovery was carried out by measuring 6-15 sample replicates. The acceptance criterion for recovery for analyte concentrations ranging from 0.1-10 ppm is 90-110%. Repeatability was tested by measuring 6-15 sample replicates, with the acceptance limit being RSD ≤ 0.5 CV Horwitz.

$$LOD = 3 \times SD' \quad (1)$$

$$LOQ = 10 \times SD' \quad (2)$$

SD' is the standard deviation of the measurements (SD) divided by the square root of the number of measurements ($\sqrt{n}$).

Accuracy can be expressed as the percent recovery, which can be calculated using equation (3). Meanwhile, precision repeatability is calculated using equation (4).

$$\%Recovery = \frac{C(spike) - C(sample)}{C(standard)} \times 100 \quad (3)$$

$$RSD = {SD}/{x} \quad (4)$$

C is the analyte concentration, and x is the average analyte concentration.

2.2. Heavy Metal Concentrations

Sediment samples were collected in August 2021 from Marekofo Village (Station 1) and Maregam Village (Station 2), Mare Island, North Maluku (0°33' - 0°35' N and 127°22' - 127°24' E). The sampling stations consisted of eight sampling points in Marekofo Village (1.1–1.8) and three sampling points in Maregam Village (2.1–2.3). **Figure 1** shows the map of the Mare Island research region, North Maluku. The sediment was collected using a sediment core made of PVC pipe,

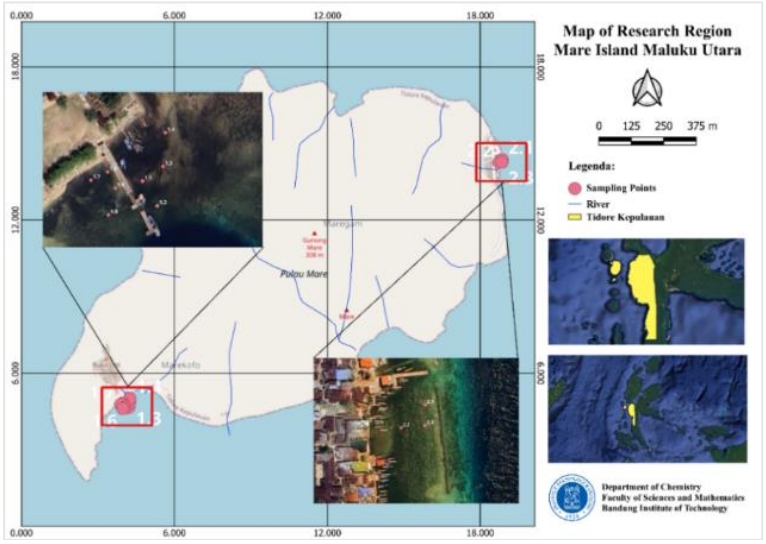

Figure 1 Location of sediment sampling on Mare Island

approximately 20 cm in length and 9 cm in diameter. At each sampling point, the corer was manually pushed into the seabed to obtain the upper 0–10 cm layer. Each sampling point was represented by a single core sample, resulting in a total of 11 individual sediment samples (8 samples from Station 1 and 3 samples from Station 2).

The sediment samples were placed in ziplock plastic bags and transported to the laboratory under cold conditions in a cooler box. The sediment samples were dried under sunlight. The dried sediment samples were then sieved using a 200mesh sieve. The sediment samples were wet-digested for heavy metal testing of Ni, Cu, and Cd according to standard procedures using a hotplate and aqua regia (Food and Agriculture Organization, 2023). A 0.5 g of dried sediment was placed into a 50 mL beaker. Then, 12 mL of aqua regia (HCl: HNO_3 3:1 v/v) was added to the beaker. The sample was digested with an electric heater (Thermolyne MIRAK) for 3 hours at a temperature of 110°C. During the digestion process, the beaker was covered with a watch glass. The sample was filtered using Whatman No. 42 filter paper. The sample was then diluted to 50 mL with deionized water in a volumetric flask. The beaker and filter paper were thoroughly rinsed with deionized water to ensure that no contaminants remained during sample processing. The testing was performed using flame atomic absorption spectrophotometer (Agilent 280FS AA).

The heavy metal concentrations (mg/kg) in the sediments are presented as the mean of replicates (n=3) ± standard deviation. The sediment sampling locations are mapped using QGIS version 3.40.1, while the diagrams are created using Origin version 8.5.1. Statistical analysis, like One-Way ANOVA, was performed to determine significant differences in heavy metal concentrations at each sediment sampling point, using Minitab version 21.1.

2.3. Assessment of Heavy Metal Pollution

Contamination Factor (CF), Pollution Load Index (PLI), Potential Ecological Risk Index (PERI), and Enrichment Factor (EF) are used to evaluate the level, source, and risk of heavy metal pollution in sediments (Nugraha et al., 2022). The contamination factor is used to assess the level of heavy metal pollution. CF is calculated according to equation (5).

$$CF = {}^{Cn}/_{Bn} \qquad (5)$$

Cn is the concentration of heavy metal "n" in the sediment, and Bn is the concentration of heavy metal "n" in the natural environment. CF is divided into four categories of pollution level: low (CF<1), moderate (1≤CF≤3), substantial (3≤CF<6), and very high (CF≥6). The background concentrations (Bn) for the metals are Ni = 68 ppm, Cu = 45 ppm, and Cd = 0.3 ppm (Turekian & Wedepohl, 1961).

The Enrichment Factor (EF) is used to determine the source of heavy metal pollution. EF can be calculated according to equation (6).

$$EF = ({}^{Cn}/_{Cref})/({}^{Bn}/_{Bref}) \qquad (6)$$

Cn is the concentration of heavy metal "n" in the sediment, Cref is the reference metal concentration, which in this study is Fe due to its abundance in nature, Bn is the concentration of heavy metal "n" in the natural environment, and Bref is the concentration of the reference metal in nature (Fe = 47,200 ppm). EF can be divided into five categories of enrichment: minimal (1<EF<2), moderate (2≤EF<5), substantial (5≤EF<20), high (20≤EF<40), and very high (EF > 40). In simple terms, heavy metal inputs from anthropogenic sources have an EF value >1.5. When the EF value <1.5, it indicates that the heavy metal input is from natural weathering or natural processes (Qiu et al., 2018).

The Pollution Load Index (PLI) provides a simple measure to assess the level of contamination for various heavy metals and is calculated according to equation (7).

$$PLI = [CF1 \times CF2 \times CF3 \times \ldots \times CFn]^{1/n} \qquad (7)$$

CF is the contamination factor for each heavy metal; n is the number of heavy metals. A PLI value above 1 indicates heavy metal pollution, while a value below 1 indicates no heavy metal pollution.

Potential Ecological Risk Index (PERI) assesses the probability that harmful ecological effects may occur or have already occurred as a result of exposure to one or more pollutants. RI can be calculated according to equation (8).

$$PERI = \sum_{i=1}^{n} E_r^i = \sum_{i=1}^{n} (T_r^i \times C_f^i) \qquad (8)$$

C_f^i is the contamination factor for heavy metal "i"; T_r^i is the toxic response factor for heavy metal "i", which reflects the toxicity level and the sensitivity of bioorganisms to the heavy metal. The toxic response factors (T_r^i) for Cd, Cu, and Ni are 30, 5, and 6, respectively (Arviani et al., 2024). RI can be divided into four categories of ecological risk: low (RI < 150), moderate (150 ≤ RI < 300), substantial (300 ≤ RI < 600), and very high (RI ≥ 600).

Table 1. Comparison of LOD and LOQ between the two researches

Heavy Metal	This research (mg/kg dw)		Other reseacher (ppm) (Aquisman et al., 2019)	
	LOD	LOQ	LOD	LOQ
Ni	0.50	1.65	0.012	0.047
Cu	0.15	0.51	0.018	0.056
Cd	0.27	0.89	0.028	0.081

3. Results and Discussions

3.1. Verification of Analysis Method

The heavy metal testing method for Ni, Cu, and Cd in sediment matrices using FAAS was verified using a sediment sample from Marekofo Village, Mare Island, North Maluku. The method verification was conducted to confirm whether the standard method from the Food and Agriculture Organization (Food and Agriculture Organization, 2023) can or cannot be applied to the actual research conditions, including material conditions, equipment, and researcher competence. The performance evaluation of the method included linearity, LOD, LOQ, recovery, and repeatability.

Linearity is the ability of the method to provide a response, either directly or indirectly with the aid of mathematical transformation, that is proportional to the amount of analyte in the sample (Harmita, 2004). A testing method is said to have good linearity if it results in a coefficient of determination (r^2) value greater than 0.990 (Latimer, 2023). The results of the linearity test showed that the method for testing heavy metals Ni, Cu, and Cd in sediments using FAAS was confirmed to have good linearity. This was evidenced by the r^2 values for each heavy metal testing method, which were 0.9980 for Ni, 0.9991 for Cu, and 0.9975 for Cd.

The limit of detection (LOD) is the ability of the method to detect the lowest concentration of an analyte that can be distinguished from the blank (Magnusson & Ornemark, 2014). The LOD for this testing method for Ni, Cu, and Cd are 0.50, 0.15, and 0.27 mg/kg dw, respectively. The limit of quantification (LOQ) for this method is 1.65, 0.51, and 0.89 mg/kg dw. The LOQ is the analyte concentration that can be accurately quantified with a certain level of uncertainty (Magnusson & Ornemark, 2014). The LOQ for this method is considered acceptable because it is lower than the low threshold for heavy metals Ni, Cu, and Cd in sediments according to ANZECC & ARMCANZ, which are 21, 65, and 1.5 mg/kg dw, respectively (ANZECC & ARMCANZ, 2000).

The research conducted by Aquisman et al., (2019) using a similar protocol and sediment matrix to this research, employing a wet digestion method with aqua regia (HCl: HNO_3 3:1 v/v) and measurement using FAAS (Aquisman et al., 2019). A comparison of the LOD and LOQ between the two researches is shown in **Table 1**. The LOD and LOQ in the Aquisman et al., (2019) research is better, with lower detection limits. This difference is mainly due to the use of different AAS instruments and blank preparation (Konieczka, 2012). In this research, the AAS used was the Agilent 280FS, while the Perkin Elmer Optima 8300 series was used in the other research. Both instruments have different detection limits. Furthermore, in the Aquisman et al., (2019) research, the detection limit was obtained from the standard deviation of the calibration curve, whereas in this research, it was derived from the standard deviation of the reagent blank measurement.

Precision (repeatability) refers to the repetition of testing aimed to measure the variability of test results for the same test sample by a single analyst using the same testing method and equipment in the shortest possible time interval (Yamada et al., 2009). This testing method demonstrates good repeatability, as the RSD values for each metal measurement are less than 11% for 1≤x≤10 mg/kg, 7.3% for 10≤x≤100 mg/kg, and 5.3% for 10≤x≤100 mg/kg or in general, less than 0.5 CV Horwitz (Latimer, 2023). The RSD values and the CV Horwitz for Ni, Cu, and Cd are shown in **Table 2**. With good repeatability, this indicates that the testing method can produce consistent results, meaning that the instruments and protocols used in the testing are functioning stably.

Table 2. Repeatability and recovery results of the testing method

Heavy Metal	RSD (%) (n=10)	0,5*CV_H(%)	Recovery (%) (n=10)		
			Low	Middle	High
Ni	1.5	4.78	109±5.4	100±2.0	81±1.1
Cu	0.6	3.60	110±0.9	100±0.6	94±0.4
Cd	4.1	5.87	102±6.1	92±1.1	87±1.0

Table 3. Differences in recovery and precision between the two researches

Heavy Metal	This research		Other research (Aquisman et al., 2019)	
	RSD(%)	*Recovery*(%)	RSD(%)	*Recovery*(%)
Ni	1.5	81–109	6.1	90–96
Cu	0.6	94-110	4.0	92–102
Cd	4.1	87–102	4.9	92–101

Table 4. Heavy metal concentrations (mg/kg dw) of Ni, Cu, and Cd at each point

Sample	Ni	Cu	Cd
1.1	23.3±0.28	4.22±0.252	6.23±0.111
1.2	29.2±0.49	5.21±0.175	6.20±0.094
1.3	26.1±0.59	0.73±0.150	6.39±0.105
1.4	24.0±0.29	2.14±0.224	6.24±0.091
1.5	25.6±0.53	<0.51	7.30±0.100
1.6	27.0±0.60	<0.51	7.94±0.081
1.7	23.4±0.48	<0.51	7.04±0.062
1.8	28.5±0.46	<0.51	7.69±0.068
2.1	24.9±0.28	<0.51	6.59±0.070
2.2	29.3±0.46	<0.51	7.65±0.066
2.3	28.3±0.69	<0.51	7.87±0.058
Min	23.3±0.28	<0.51	6.20±0.094
Max	29.3±0.46	5.21±0.175	7.94±0.081
Average	26.3	3.08	7.01
Stdv	2.29	2.02	0.704

Accuracy refers to the degree of closeness of measurement results to the true concentration. Accuracy can be expressed as % recovery (Yamada et al., 2009). Samples are spiked with standard solutions to achieve low, medium, and high concentrations of analytes in the samples. The recovery measurement results for the testing methods of Ni, Cu, and Cd are shown in **Table 2**. This testing method demonstrates good recovery values, as it meets the acceptance criteria in the range of 80-110% for 0.1-10 ppm (Latimer, 2023). With good recovery, this not only indicates that the testing method has good accuracy but also that it has a good ability to reduce matrix interference effects. Despite the complex sediment matrix, this method selectively measures the analytes effectively. From these findings, it is confirmed that the testing method is accurate and selective.

The recovery and precision results from this research differ from those conducted by Aquisman et al. (2019). **Table 3** shows a comparison of recovery and precision between the two studies. The differences in recovery and precision are primarily due to differences in the characteristics of the sediment samples used. Matrices with high salt content, such as marine sediment, can affect the sensitivity and accuracy of the testing by AAS. This is due to the high background noise and the attenuation of signal intensity (Zhou et al., 2024). Additionally, the homogeneity of the sediment samples can affect the consistency of the measurements. Hydrodynamic changes induced by water currents make river sediments more heterogeneous in terms of grain size (Xu et al., 2021). This causes one sub-sample to differ from another in terms of composition. Heavy metals tend to be found in the finer sediment fractions due to their high adsorption capacity (Liu et al., 2019).

3.2. Heavy Metal Concentrations

The sediment samples collected from the waters of Marekofo Village (station 1) and Maregam (station 2) were tested using the previously verified method. The results reveal contrasting distribution patterns of the three heavy metals. Nickel (Ni) and cadmium (Cd) were consistently detected at all sampling points, indicating their widespread occurrence. In contrast, copper (Cu) was only identified at several points within Station 1 (1.1–1.4), suggesting a more localized source of contamination. The concentrations measured at each sampling point are summarized in **Table 4**.

Nickel shows the most uniform distribution across the study area, with concentrations ranging from 23.3±0.28 to 29.3±0.46 mg/kg dw, with an average of 26.3±2.29 mg/kg dw. This concentration exceeds the ISQG-Low threshold, which is 21 mg/kg dw. In other words, under these conditions, the heavy metal of Ni can cause harmful effects on marine biota (McCready et al., 2006).

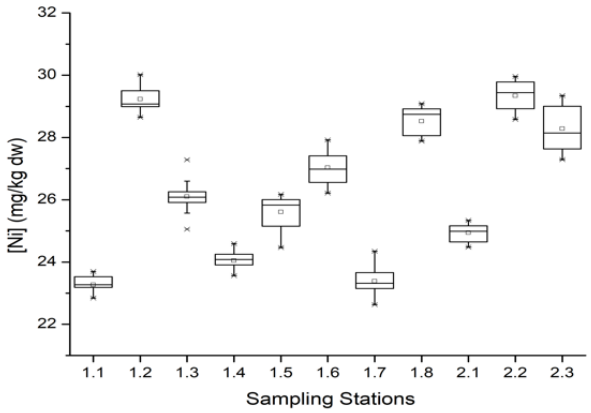

Figure 3. Ni concentration at each sampling point on Mare Island

Figure 2 shows the Ni concentration at each sampling point on Mare Island. Based on the One-Way ANOVA test, the concentrations of the heavy metal Ni at each sampling point differ significantly ($p<0.05$). The highest concentration of Ni was found at point 2.2, while the lowest was at point 1.1. These findings are lower compared to the concentration of Ni in the sediments of the coastal waters of Fatufia, Morowali, which is 1150.80 ± 393.948 µg/g (Lestari et al., 2024). This is because the Fatufia coast is closer to the source of nickel pollution, which is nickel mining activities. However, these findings are similar to the concentrations of heavy metal Ni in sediments from the waters of Morotai Island and Bacan, which are 20.605±14.243 ppm (Edward, 2015) and 21.871±14.277 ppm (Marasabessy et al., 2010), respectively. Pollution in the Maluku waters is not as severe, as nickel mining activities are less intensive than in Sulawesi. In 2020, 242 nickel mining companies were operating in Sulawesi, while only 46 companies were operating in Maluku Ministry of Energy and Mineral Resources, 2020).

Copper was detected only at sampling points 1.1–1.4 at Station 1, revealing that its distribution is more spatially restricted than Ni. The concentrations of Cu in the sediments range from <0.51 to 5.21±0.175 mg/kg dw, with an average of 3.08±2.02 mg/kg dw. This concentration indicates that Cu is still at a safe level and does not pose harmful effects on marine organisms, as it is lower than the ISQG-Low threshold of 65 mg/kg dw (McCready et al., 2006). **Figure 3** shows the concentrations of Cu at each sampling point in the waters of Mare Island. Based on the One-Way ANOVA test, the concentrations of the heavy metal Cu at each sampling point differ significantly ($p<0.05$). This finding is lower than the concentration of Cu in the sediments of the waters of Morotai Island, which is 45.163±20.011 ppm (Edward, 2015). This finding is similar to the concentration of Cu in the sediments of Bastiong harbor, which is

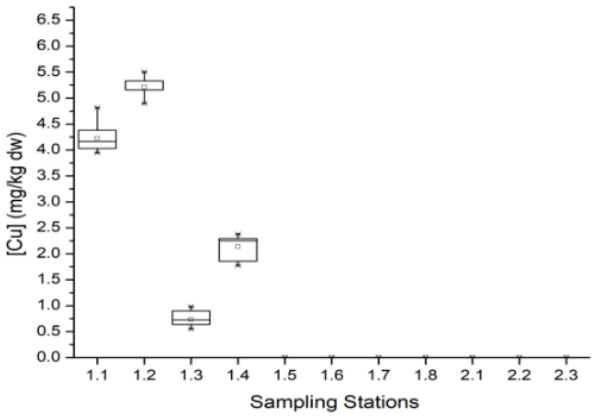

Figure 2. Cu concentration at each sampling point on Mare Island

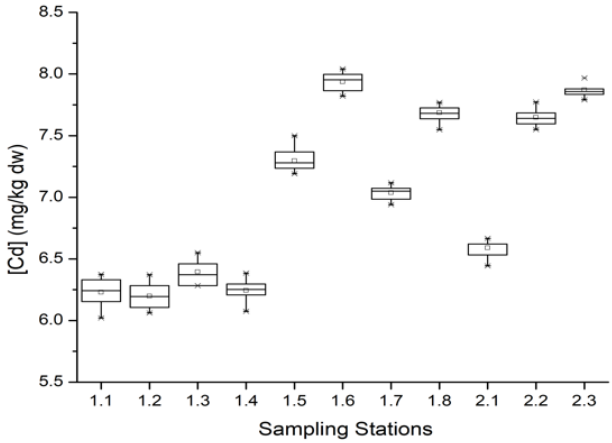

Figure 4. Cd concentration at each sampling point on Mare Island

3.857±0.216 ppm (Nurhamiddin & Ibrahim, 2018). The presence of Cu is suspected to be caused by the input of domestic waste containing Cu into the waters around the Marekofo coast. Additionally, Cu may be released from antifouling paints on ships that dock in the area (Lagerström et al., 2020).

Among the analyzed metals, Cd poses the greatest ecological concern. The concentrations of Cd in the sediments range from 6.20±0.094 to 7.94±0.081 mg/kg dw, with an average of 7.01±0.704 mg/kg dw. This concentration exceeds the ISQG-Low threshold, which is 1.5 mg/kg dw. Under these conditions, the heavy metal Cd can cause harmful effects on marine organisms, similar to Ni (McCready et al., 2006). **Figure 4** shows the concentrations of heavy metal Cd at each sampling point on Mare Island. Based on the One-Way ANOVA test, the concentrations of the heavy metal Cd at each sampling point differ significantly ($p<0.05$). These findings are higher than the concentration of Cd in the waters of Morotai Island and Bacan, which are 0.125±0.189 ppm (Edward, 2015) and 0.111±0.079 ppm (Marasabessy et al., 2010) respectively. These results are similar to the concentration of Cd in the waters of Tanjung Mas, North Semarang, which ranged from 2.382 to 7.121 mg/kg (Purba et al., 2014). The anthropogenic sources (human activities) of Cd in the environment include smelting and refining of Cu and Ni, fossil fuel combustion, and the use of phosphate fertilizers (Genchi et al., 2020). In addition to being located around nickel mining areas, Mare Island also has a Diesel Power Plant that could potentially be a source of Cd pollution.

3.3. Assessment of Heavy Metal Pollution

The assessment of the level, sources, and risks of heavy metal contamination (Ni, Cu, and Cd) is based on the values of CF, PLI, EF, and PERI. The CF values for Ni, Cu, and Cd in the waters of Mare Island range from 0.342–0.431, 0–0.116, and 20.66–26.45, respectively. The CF values for Ni and Cu indicate low contamination levels ($CF<1$) in the waters of Mare Island. A similar low contamination level for Ni was also found in the waters of Morotai Island with CF values ranging from 0.06 to 0.697 (Edward, 2015), and for Cu in the waters of Ternate City with CF values ranging from 0.015 to 0.899 (Edward et al., 2020). In contrast, the CF value for Cd indicates a very high level of contamination ($CF>6$) in the waters of Mare Island. In comparison, nearby areas such as Morotai Island, Ternate City, and Ambon Bay, with average CF values of 0.418, 0.653, and 0.037, respectively, show low contamination levels ($CF<1$) (Edward, 2015; Edward et al., 2020; Sukaryono & Dewa, 2018).

The EF values for the metal Ni in the sediments of Mare Island range from 0.347 to 2.341, indicating a minimal ($1<EF<2$) to moderate ($2\leq EF<5$) enrichment level. The EF values for Cu range from 0 to 0.238, indicating minimal enrichment ($1<EF<2$). On the other hand, the EF values for Cd range from 23.65 to 155.77, indicating high enrichment ($20\leq EF<40$) to very high enrichment ($EF>40$). The obtained EF values for Cd suggest that this heavy metal is primarily sourced from anthropogenic sources ($EF>1.5$), while Cu is sourced from natural weathering or natural processes ($EF<1.5$). The EF value for Ni indicates that this metal has contributions from both anthropogenic and natural sources. **Table 5** shows the CF and EF values for each metal at every sampling point on Mare Island.

Table 5. CF, EF, PLI, and RI values for Ni, Cu, and Cd at each sampling point

Sample	Ni		Cu		Cd		PLI	RI
	CF	EF	CF	EF	CF	EF		
1.1	0.342	0.761	0.094	0.209	20.76	46.155	2.768	625.4
1.2	0.430	0.882	0.116	0.238	20.66	42.402	2.768	623.0
1.3	0.384	1.228	0.016	0.052	21.32	68.216	2.790	641.8
1.4	0.354	0.928	0.048	0.125	20.81	54.611	2.768	626.7
1.5	0.377	1.419	0.000	0.000	24.32	91.658	2.912	731.9
1.6	0.398	2.341	0.000	0.000	26.45	155.767	2.994	796.0
1.7	0.344	0.347	0.000	0.000	23.46	23.646	2.876	705.8
1.8	0.419	0.448	0.000	0.000	25.62	27.351	2.964	771.1
2.1	0.367	0.797	0.000	0.000	21.97	47.713	2.816	661.2
2.2	0.431	1.509	0.000	0.000	25.50	89.150	2.960	767.4
2.3	0.416	1.978	0.000	0.000	26.24	124.756	2.987	789.6
Min	0.342	0.347	0,000	0.000	20.66	23.646	2.768	623.0
Max	0.431	2.341	0.116	0.238	26.45	155.767	2.994	796.0
Average	0.342	1.149	0.094	0.156	20.76	70.129	2.873	703.6

The Pollution Load Index (PLI) and Ecological Risk Index (RI) values in the waters of Mare Island range from 2.768 to 2.994 and 623 to 796, respectively. The PLI values obtained for the waters of Mare Island indicate a polluted category (PLI>1). The RI values obtained indicate a very high ecological risk status (RI>600). This suggests a very high potential danger from Cd to the ecosystem in the waters of Mare Island. **Table 5** also presents the PLI and RI values for each sampling point on Mare Island.

4. Conclusion

The concentrations of Ni, Cu, and Cd in the sediments from the waters of Mare Island were tested using the standard FAO procedure, which involves aqua regia wet digestion and flame atomic absorption spectrometry (AAS) that has been verified. The verification results show that this method has good linearity, LOD, LOQ, recovery, and repeatability, meeting the acceptance criteria according to the AOAC guideline. The concentrations of Ni, Cu, and Cd in the waters of Mare Island, North Maluku, range from 23.3±0.28 to 29.3±0.46 mg/kg dw, <0.51 to 5.21±0.175 mg/kg dw, and 6.20±0.094 to 7.94±0.081 mg/kg dw, respectively. The concentration of Cu is still within safe limits as it is below the ISQG-Low threshold, whereas the concentrations of Ni and Cd are harmful to marine biota, as they exceed the ISQG-Low threshold. Based on the CF and EF values, the waters of Mare Island are categorized as contaminated, especially with heavy metal of Cd, which has a high contamination level that originated from anthropogenic activities. Meanwhile, heavy metal Ni contamination is at a low level, with sources from both natural and anthropogenic activities. Based on the PLI and RI values, sampling point 1.6 in the waters of Mare Island is the most polluted and poses a very high potential risk to marine biota. Therefore, the conservation area of Mare Island's seagrass beds is indicated to be contaminated with heavy metals.

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