

# Chemometric Analysis for Heavy Metal Pollution Assessment of Beach Sediments in Balongan, Indramayu

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## Article Info

Received: Sept 13, 2025  
Revised: Dec 27, 2025  
Accepted: Feb 01, 2026  
Online: May 31, 2026

### Citation:

Bakti, A. B., & Azis, M. Y. (2026). Chemometric Analysis for Heavy Metal Pollution Assessment of Beach Sediments in Balongan, Indramayu. *Jurnal Kimia Valensi*, 12(1), 1-13.

### Doi:

[10.15408/jkv.v12i1.46477](https://doi.org/10.15408/jkv.v12i1.46477)

## Abstract

Balongan is a coastal area whose waters are vulnerable to contamination heavy metals due to anthropogenic activities, particularly oil refinery operations. This study aimed to (1) characterize the physicochemical properties and quantify heavy metal concentrations in sediments from Balongan; (2) identify the contamination level, sources, spatial distribution, and ecological risk; and (3) analyze inter-parameter relationships, site similarities, and dominant factors controlling contamination pattern variability using a chemometric approach. Sampling was conducted in May 2025, and heavy metal analysis was performed using F-AAS. The results showed that the concentrations of Pb, Cu, Zn, and Cr remained within safe limits, whereas Cd and Ni exceeded the threshold values. Cd was predominantly attributed to anthropogenic sources ( $CF > 6$ ), whereas Ni was mainly attributed to natural processes ( $CF < 1$ ). Although still below threshold values, Pb, Cu, Zn, and Ni concentrations were higher at sites closer to the oil refinery, suggesting influences from oil-related activities. Elevated Cd and Ni concentrations were the primary contributors to sediment toxicity, with PERI values ranging from moderate to severe. The clay fraction and organic matter showed positive correlations with heavy metals. Thus, the dominant factors controlling the variability of contamination patterns are the similarity of pollution sources and sediment physicochemical properties.

**Keywords:** Balongan Indramayu; beach sediment; chemometric analysis; heavy metals; pollution assessment

## 1. INTRODUCTION

The sea is both an identity and a key to prosperity for maritime nations. As an archipelagic country with three-quarters of its territory consisting of seas, Indonesia possesses enormous marine resource potential<sup>1</sup>. According to the Ministry of Marine Affairs and Fisheries' 2021 report, the estimated potential value of Indonesia's ocean economy reached IDR 19,371 trillion<sup>2</sup>. Indramayu has successfully utilized its marine and coastal resources. In 2023, it became the largest producer of capture fisheries and aquaculture in West Java, contributing 34.63% of the province's total fisheries production<sup>3</sup>. The Balongan area of Indramayu hosts an oil refinery industrial zone operated by PT. Pertamina RU VI. Oil processing activities and tanker traffic pose risks of marine pollution, particularly from oil spills and liquid waste discharges. In 2008, an oil spill incident

involving the Arendal tanker caused pollution along the Indramayu coast<sup>4</sup>. Crude oil contains heavy metals such as Cd, Pb, Mn, Ni, and V<sup>5</sup>. Oily sludge, a by-product of refinery processes, contains Pb, Cu, Ni, V, Zn, and Cr<sup>6</sup>.

Heavy metals are hazardous inorganic pollutants that are difficult to degrade and persistent in the environment. Transition elements with a density greater than 5 g/cm<sup>3</sup> are classified as heavy metals<sup>7</sup>. Pb is toxic because it can increase the production of reactive oxygen species (ROS), which damage protein structures, cell membranes, and lipids. Cd is more toxic and carcinogenic, causing alterations in the structure and function of various organs by disrupting the biosynthesis of DNA, RNA, and proteins.<sup>7</sup> Similarly, Ni is carcinogenic because it regulates RNA expression. Cr also exerts multiple adverse effects on the human immune system by inhibiting lymphocyte

proliferation. Cu and Zn are essential micronutrients. However, excessive exposure to Cu can lead to Wilson's disease and neurobehavioral disorders resembling mental illness<sup>8</sup>. In contrast, excessive Zn exposure may cause headaches, nausea, vomiting, diarrhea, and abdominal discomfort<sup>9</sup>. The threshold limits for heavy metals Pb, Cd, Cu, Zn, Ni, and Cr in sediments are 50, 1.5, 65, 200, 21, and 80 mg/kg dry weight, respectively<sup>10</sup>.

Sediments are commonly used as environmental media to indicate water quality. They are capable of accumulating pollutants through complex adsorption mechanisms<sup>11</sup>. Within sediments, pollutants may undergo transformation or recycling and can also re-interact with the overlying water or with benthic organisms living within the sediments<sup>12</sup>. Previous studies conducted in the Balongan Indah coastal area of Indramayu have reported varying levels of heavy metal contamination in marine sediments. Fitriani (2023) reported Pb concentrations ranging from 6.33 to 10.43 ppm, which remain within an acceptable threshold limit<sup>13</sup>. In contrast, Sihombing (2023) found that Cd concentrations ranged from 2.68 to 4.18 mg/kg, exceeding the threshold limit of 1.5 mg/kg, suggesting potential contamination. Meanwhile, Cr concentrations were relatively low, ranging from 0.09 to 0.75 mg/kg, and were classified as within safe limits<sup>14</sup>. Research on heavy metal contamination in sediments of Balongan, Indramayu, remains limited and fragmented. Most existing studies primarily focus on determining concentration levels and comparing them with the threshold limit.

However, environmental quality monitoring generally produces complex, non-linear, multivariate, and cumulative data. A chemometric approach is considered the most effective and efficient method. Chemometrics is a multivariate analysis that simultaneously processes all observed variables with high accuracy, making it more effective and allowing the extraction of additional information, such as relationships among variables<sup>15</sup>. Principal Component Analysis (PCA) is the most widely used chemometric technique, with the advantage of reducing original variables and generating new ones that can be interpreted more simply<sup>16</sup>. A study by Pedersen et al. (2015) used PCA to identify pollution sources and to examine relationships between pollutants and sediment characteristics in a harbor area. The results showed that polycyclic aromatic hydrocarbons (PAHs) were positively correlated with sediment organic matter at domestic wastewater disposal sites, indicating their pollution source. In contrast, polychlorinated biphenyls (PCBs) showed no correlation with organic matter, suggesting multiple pollution sources<sup>17</sup>.

The standard method commonly used for the determination of total heavy metals in sediments is the

US EPA 3050 B method. This method is used for quantifying total heavy metals. Heavy metal fractionation is frequently performed on sediment samples, which are essential for understanding the fate and mobility of heavy metals in the environment and for identifying pollution sources. Different pollution sources may result in distinct heavy metal fractions. The Tessier and the Community Bureau of Reference (BCR) methods are commonly applied for heavy metal fractionation in sediments. Sequential extraction procedures yield several geochemical fractions: (1) the exchangeable fraction, which is water-soluble or weakly bound and associated with carbonate minerals; (2) the reducible fraction, bound to Fe and Mn oxides; (3) the oxidizable fraction, associated with organic matter and sulfide minerals; and the residual fraction, bound to silicate minerals<sup>18</sup>.

Sediments are highly complex dynamic systems influenced by hydrodynamic factors (such as storms, submarine landslides, and bioturbation by benthic organisms), physicochemical processes (including sorption and redox reactions), and microbial transformations<sup>12</sup>. Consequently, sediments exhibit site-specific characteristics. The physical properties of sediments include porosity and texture. Sediments with low porosity have a lower capacity to retain water compared to those with high porosity<sup>19</sup>. Furthermore, sediments with finer textures or higher clay fractions tend to exhibit greater accumulation of heavy metals<sup>20</sup>. In addition to physical properties, several chemical characteristics of sediments include pH, organic matter, and carbonate content<sup>17</sup>. pH strongly influences the solubility of heavy metals<sup>21</sup>, while organic matter and carbonates have the capacity to adsorb heavy metals and promote their deposition within sediments<sup>22,23</sup>.

Based on the above description, it is necessary to conduct a study to evaluate heavy metal pollution in beach sediments from the Balongan coast of Indramayu using a chemometric approach. Therefore, this research aims to (1) determine the concentrations of Pb, Cd, Cu, Zn, Ni, and Cr in beach sediments from Balongan, Indramayu; (2) identify the levels, sources, distribution, and risks of their contamination; and (3) analyze the interrelationships among test parameters, locations similarity, and dominant factors through a chemometric approach.

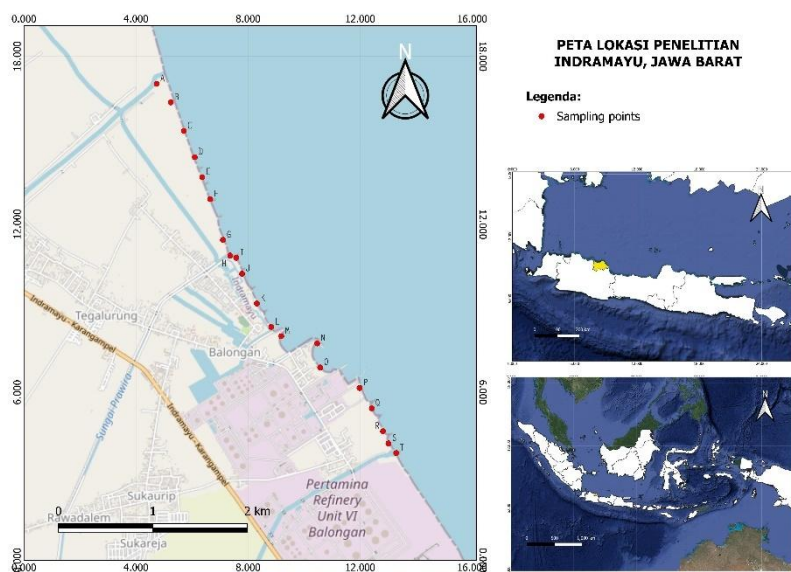
## 2. RESEARCH METHODS

In this study, the total heavy metal content was analyzed by Flame-AAS using the US EPA Method 3050 B<sup>24</sup>. Heavy metal fractionation was adopted from Yap et al. (2002)<sup>25</sup>. In contrast, physicochemical characterization of sediments included measurements of porosity<sup>26</sup>, texture<sup>27</sup>, organic matter<sup>28</sup>, carbonate<sup>29</sup> and pH<sup>30</sup>.

The materials used in this study included Lead(II) nitrate,  $Pb(NO_3)_2$  ( $\geq 99.95\%$ , Merck, Germany); Cadmium(II) acetate dihydrate,  $Cd(CH_3COO)_2 \cdot 2H_2O$  ( $\geq 99.0\%$ , Merck, Germany); Copper(II) sulfate pentahydrate,  $CuSO_4 \cdot 5H_2O$  (99.0–100.5%, Merck, Germany); Zinc(II) chloride,  $ZnCl_2$  ( $\geq 98\%$ , Merck, Germany); Nickel(II) chloride hexahydrate,  $NiCl_2 \cdot 6H_2O$  ( $\geq 98\%$ , Merck, Germany); Hexavalent chromium, Cr(VI) (1000 mg/L, Merck, Germany); Multi-element ICP Standard XVI (100 mg/L Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, Zn; Merck, Germany); Nitric acid,  $HNO_3$  (65%, Merck, Germany); Hydrochloric acid,  $HCl$  (37%, Merck, Germany); Hydrogen peroxide,  $H_2O_2$  (30%, Merck, Germany); Filter paper (No. 42, Whatman, UK); Ammonium acetate,  $CH_3COONH_4$  ( $\geq 98\%$ , Merck, Germany); Hydroxylamine hydrochloride,  $NH_2OH \cdot HCl$  (99%, Loba Chemie, India); Sodium hexametaphosphate,  $[NaPO_3]_6$  (68%, Loba Chemie, India); Sodium hydroxide,  $NaOH$  ( $\geq 98\%$ , Merck, Germany); Calcium chloride dihydrate,  $CaCl_2 \cdot 2H_2O$  ( $\geq 99\%$ , Merck, Germany); potassium hydrogen phthalate ( $>99\%$ , Merck, Germany); phenolphthalein indicator solution (0.375%, Merck, Germany) and buffer solutions (pH 4, 7, and 10, Merck, Germany).

The instruments used in this study included an analytical balance (Mettler Toledo ME 204), oven (Mettler UNB 400), hotplate (Thermolyne MIRAK), AAS (Agilent 280FS AA), rotator (iRoll PR35), centrifuge (Centurion Scientific Pro-PRP S), furnace (Ney Vulcan D-130), and pH meter (Mettler Toledo SevenCompact S220).

Sediment samples were collected from the Balongan coast, Indramayu, West Java (**Figure 1**). Sampling was conducted in May 2025. The sampling sites were determined using a purposive sampling method, based on specific considerations such as proximity to pollution sources, namely industrial activities, residential areas, and marine tourism. A total of 20 sampling points were established around the identified pollution sources. Sampling was carried out during low tide in the highest tidal zone using a stainless-steel scoop for grab sampling. Surface sediments (0–5 cm), approximately 200 g, were collected along a 10 m transect containing five  $1 \times 1$  m quadrats. Sediments from each quadrat were composited to obtain a 1 kg sample. The composite samples were placed in containers and stored under cold conditions for transport to the laboratory<sup>31–33</sup>.



**Figure 1.** Map of the sampling locations

Porosity<sup>26</sup> was calculated from the bulk density and particle density values of the sediment, as shown in Equation (1).

$$\phi (\% \text{ v/v}) = \left( 1 - \frac{\rho_{db}}{\rho_p} \right) \times 100 \quad (1)$$

Bulk density ( $\rho_{db}$ ) was determined by weighing a pre-calibrated bottle of known volume filled with dry sediment. Particle density ( $\rho_p$ ) was measured using a pycnometer of known weight ( $W_1$ ). The pycnometer

containing dry sediment was weighed ( $W_2$ ), then filled with distilled water to the top and weighed again ( $W_3$ ). If the pycnometer volume was not predetermined, it was filled only with distilled water at the same temperature and weighed ( $W_4$ ).  $\rho_p$  was calculated according to Equation (2).

$$\rho_p \left( \frac{g}{mL} \right) = \frac{(W_2 - W_1) \times \rho_{air}}{[(W_4 - W_1) - (W_3 - W_2)]} \quad (2)$$

Sediment texture<sup>26</sup> was determined using a dry sieving and pipette method. Dry sediment was sieved using 10-mesh (2 mm) and 200-mesh (0.074 mm) sieves. The sand fraction was defined as the material passing through the 10-mesh sieve but retained on the 200-mesh sieve, while the silt and clay fractions were those passing through the 200-mesh sieve. The pipette method was applied to identify the clay fraction, approximately 4 g of dry sediment was dispersed in 40 mL of dispersing solution ( $[\text{NaPO}_3]_6$  0.05% w/v and NaOH 0.01 M), then stirred at 55 rpm overnight. The suspension was allowed to settle for 2 h, after which a 2.5 mL aliquot was withdrawn at a depth of 2.5 cm below the water surface. The aliquot was dried in an oven at 105 °C until constant weight, and the residue was weighed. The measured mass was corrected for the solid content of the dispersing solution<sup>27</sup>.

Sediment pH<sup>30</sup> was measured by weighing 10 g of dry sediment, then separately mixing it with 10 mL of distilled water and 0.01 M CaCl<sub>2</sub> solution. Each suspension was stirred for one hour, after which the pH was measured using a calibrated pH meter. Organic matter content<sup>28</sup> was determined using the Loss-on-Ignition (LOI) method. Approximately 2 g of dry sediment was heated at 500 °C for 6 hours. The loss of sediment mass after ignition was assumed to represent the organic matter content. Carbonate content in sediment<sup>29</sup> was determined by acid digestion followed by titration. 0.1 g of dry sediment was reacted with 20 mL of 0.1 M HCl and allowed to stand for 48 hours. The reacted solution was then titrated with standardized NaOH using potassium hydrogen phthalate (KHP) as the primary standard. Phenolphthalein was used as the endpoint indicator. Carbonate content, expressed as CaCO<sub>3</sub>, was calculated using Equation (3).

$$\text{carbonate (\% w/w)} = (V_0 - V_1) \times C_{\text{NaOH}} \times \frac{F}{w} \times 100 \quad (3)$$

$C_{\text{NaOH}}$  is the concentration of standard NaOH solution (M);  $V_0$  and  $V_1$  are the consumed NaOH solution in blank and formal experiments, respectively (ml);  $w$  is sample mass (g) and  $F = 0.05004$ .

Total heavy metals (Pb, Cd, Cu, Zn, Cr, and Ni) were analyzed following SNI 8910:2021<sup>34</sup>. A 0.5 g portion of sediment (<0.15 mm) was digested with 5 mL of HNO<sub>3</sub> (1:1, v/v) and heated at 95 °C for 10 min. Subsequently, 2.5 mL of concentrated HNO<sub>3</sub> was added and heated at 95 °C for 2 h. Afterward, 1 mL of distilled water and 1.5 mL of H<sub>2</sub>O<sub>2</sub> (30% w/v) were added and heated again at 95 °C for 2 h. The digestion continued with the addition of 5 mL of concentrated HCl, heated at 95 °C for 15 min. Finally, the digest was filtered through Whatman No. 42 filter paper, and the filtrate was collected in a 50 mL volumetric flask.

Heavy metal non-resistant fractionation<sup>25</sup> was carried out through sequential extraction as follows:

(1) Exchangeable/acid-soluble (EFLE): 5 g of sample was shaken for 3 h in 25 mL of 1 M CH<sub>3</sub>COONH<sub>4</sub> at pH 7 and room temperature; (2) Acid-reducible: the residue from step (1) was shaken for 3 h in 25 mL of 0.25 M NH<sub>2</sub>OH·HCl at pH 2 and room temperature; (3) Oxidisable-organic: the residue from the previous step was oxidized with 30% H<sub>2</sub>O<sub>2</sub> in a water bath at 90–95 °C. After cooling, the residue was shaken for 3 h in 25 mL of 1 M CH<sub>3</sub>COONH<sub>4</sub> at pH 2 and room temperature. The residue from each fraction was weighed before proceeding to the next extraction step. The mathematical sum of the EFLE, acid-reducible, and oxidisable-organic fractions represented the non-resistant components.

The Contamination Factor (CF) and the Potential Ecological Risk Index (PERI) were applied to evaluate the level, sources, and risks of heavy metal pollution in sediments<sup>35</sup>. CF and PERI was calculated according to Equations (4) and (5).

$$CF = C_n / B_n \quad (4)$$

$C_n$  represents the concentration of heavy metal 'n' in sediments, while  $B_n$  represents its background concentration. CF is classified into four categories: low contamination ( $CF < 1$ ), moderate ( $1 \leq CF \leq 3$ ), considerable ( $3 \leq CF < 6$ ), and very high ( $CF \geq 6$ ). The background concentrations ( $B_n$ ) used were: Pb = 20 ppm, Cd = 0.3 ppm, Cu = 45 ppm, Zn = 95 ppm, Ni = 68 ppm, and Cr = 90 ppm<sup>36</sup>.

$$PERI = \sum_{i=1}^n E_r^i = \sum_{i=1}^n T_r^i \times C_f^i \quad (5)$$

$C_f^i$  denotes the contamination factor of heavy metal  $i$ , while  $T_r^i$  represents the toxic response factor of heavy metal  $i$ , reflecting its toxicity level and the sensitivity of bio-organisms to the metal. The toxic response factors ( $T_r^i$ ) were 5, 30, 5, 1, 2, and 6 for Pb, Cd, Cu, Zn, Cr, and Ni, respectively<sup>37</sup>. PERI was categorized into four ecological risk levels: low ( $PERI \leq 150$ ), moderate ( $150 < PERI < 300$ ), considerable ( $300 \leq PERI < 600$ ), and high ( $PERI \geq 600$ ).

All data processing and analysis were performed using Microsoft Excel 2016 (version 2411) and Minitab (version 21.1). The sampling location map was generated using QGIS (version 3.40.1), while diagrams and graphical visualizations were prepared with Origin (version 8.5.1).

### 3. RESULTS AND DISCUSSION

#### Physicochemical Characteristics of Sediments

The porosity of beach sediments from the Balongan coast, Indramayu, ranged from  $34.9 \pm 0.0\%$  to  $53.2 \pm 3.5\%$  (v/v), with an average of  $44.7\%$  (v/v). These values are comparable to the porosity of natural beach sand, which is approximately  $40\%$ <sup>38</sup>. Porosity

plays an important role in the sorption and diffusion of pollutants at the sediment–water interface. Coarse sediments with lower porosity generally exhibit greater desorption capacity compared to fine sediments. In other words, in coarse-grained sediments, pollutants are more easily released back into the water column following even slight environmental changes<sup>39</sup>. Porosity differed significantly among sampling sites (one-way ANOVA,  $p < 0.05$ ). In addition to grain size, porosity is strongly influenced by the degree of grain size uniformity<sup>40</sup>.

The sediment texture of the Balongan coast, Indramayu, was dominated by the sand fraction, ranging from  $65.8 \pm 0.84\%$  to  $99.8 \pm 0.04\%$  (w/w) with a median of  $95.8\%$  (w/w). The clay fraction ranged from  $0.06 \pm 0.015\%$  to  $0.22 \pm 0.009\%$  (w/w) with an average of  $0.13\%$  (w/w), while the silt fraction ranged from  $0.09 \pm 0.031\%$  to  $34.01 \pm 0.835\%$  (w/w) with a median of  $4.04\%$  (w/w). Sediments at all sites were predominantly sandy, except at stations M and N, which were classified as sandy loam. Fine-grained sediments (silt and clay) generally possess larger surface areas and higher cation exchange capacities than coarse-grained sediments such as sand, thereby enhancing the retention of heavy metals within the sediment matrix<sup>20</sup>. Sediment texture differed significantly among sampling sites (Kruskal–Wallis and one-way ANOVA,  $p < 0.05$ ). These differences are primarily influenced by current velocity, with coarser sediments typically found in high-energy environments, whereas finer particles tend to accumulate in areas with weaker currents<sup>41</sup>.

The pH of beach sediments from the Balongan coast, Indramayu, ranged from 7.8 to 8.5, with an average of 8.2. Sediment samples were suspended in a 0.01 M CaCl<sub>2</sub> solution, as this medium contains ions that more closely resemble the conditions of sediment porewater, thereby providing more accurate pH measurements and minimizing dilution effects<sup>30</sup>. pH strongly influences the solubility of heavy metals; alkaline conditions generally promote adsorption and precipitation processes, whereas acidic conditions reduce the ability of sediments to retain heavy metals. Sediment pH varied significantly among sampling sites (one-way ANOVA,  $p < 0.05$ ). These variations are influenced by depth (aerobic/anaerobic decomposition of organic matter), temperature (which controls the decomposition rate of organic matter), and CaCO<sub>3</sub> content<sup>42</sup>.

The organic matter content in beach sediments from the Balongan coast, Indramayu, ranged from  $0.8 \pm 0.07\%$  to  $8.3 \pm 0.71\%$  w/w, with an average of  $4.9\%$  w/w. Organic matter contents below 5% generally indicate good environmental quality, whereas values above 10% are associated with degraded conditions. Organic matter consists mainly of carbon<sup>43</sup>. Organic

matter plays an important role in the accumulation of heavy metals in sediments due to its high binding capacity, which facilitates metal sequestration and deposition<sup>22</sup>. Organic matter content varied significantly among sampling sites (one-way ANOVA,  $p < 0.05$ ). Variations in organic matter are influenced by inputs of domestic wastewater and sedimentation rates<sup>22</sup>.

The carbonate content in sediments from the Balongan coast, Indramayu, ranged from  $2.8 \pm 0.3$  to  $8.7 \pm 0.4\%$  w/w, with an average of  $5.9\%$  w/w. This value is slightly higher than the carbonate content reported for sediments from the Cilacap coast, which averages  $4.21\%$ . The contrasting oceanographic conditions between the northern and southern waters of Java Island influence this difference. The shallow northern waters, with lower wave energy, facilitate the retention of carbonate material along the coast<sup>44</sup>. Carbonate content varied significantly among sampling sites (one-way ANOVA,  $p < 0.05$ ). Variations in sediment carbonate content are controlled by multiple environmental and climatic factors, including temperature, pH, precipitation, biological productivity, microbial communities, and hydrological conditions<sup>29</sup>.

### Levels, Sources, and Ecological Risks of Heavy Metal Pollution

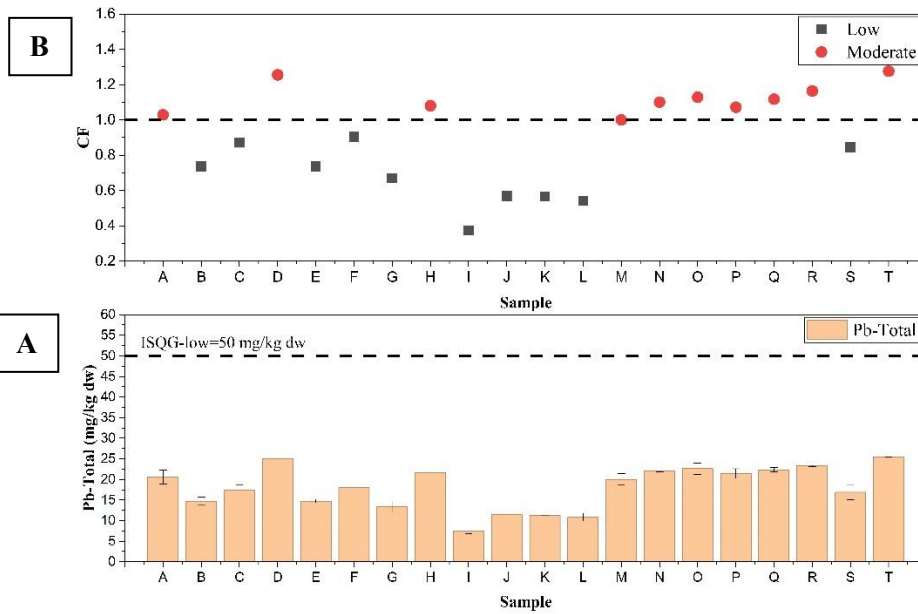
Lead (Pb) concentrations in beach sediments from Balongan, Indramayu, ranged from  $7.49 \pm 0.71$  to  $25.52 \pm 0.05$  mg/kg dw, with an average of  $18.04$  mg/kg dw (**Figure 2(A)**). These values are below the ISQG-low threshold of  $50$  mg/kg dw, indicating that the sediments are still within a safe range. Pb concentrations differed significantly among sampling sites (one-way ANOVA,  $p < 0.05$ ). Although overall Pb levels remain below the guideline value, sites located near the oil refinery exhibited relatively higher concentrations compared to other locations. The CF values (**Figure 2(B)**) indicate enrichment relative to natural background conditions, which may be associated with oil-related activities, including oil refinery operations and maritime transportation such as fishing vessels and oil tankers. Heavy metals, including Pb, are known to be present in crude oil, conventional octane boosters, and oil sludge<sup>5 6</sup>.

The concentration of Zn in beach sediments from the Balongan coast, Indramayu, ranged from  $62.44 \pm 6.85$  to  $119.32 \pm 14.97$  mg/kg dw, with an average of  $86.4$  mg/kg dw (**Figure 3(A)**). This concentration is still lower than the ISQG low value of  $200$  mg/kg dw, indicating that the condition remains safe. Zn concentrations differed significantly among sampling sites (one-way ANOVA,  $p < 0.05$ ). Similar to Pb, although the overall total Zn concentrations were still below the threshold, sites located close to the oil refinery showed relatively higher levels compared to

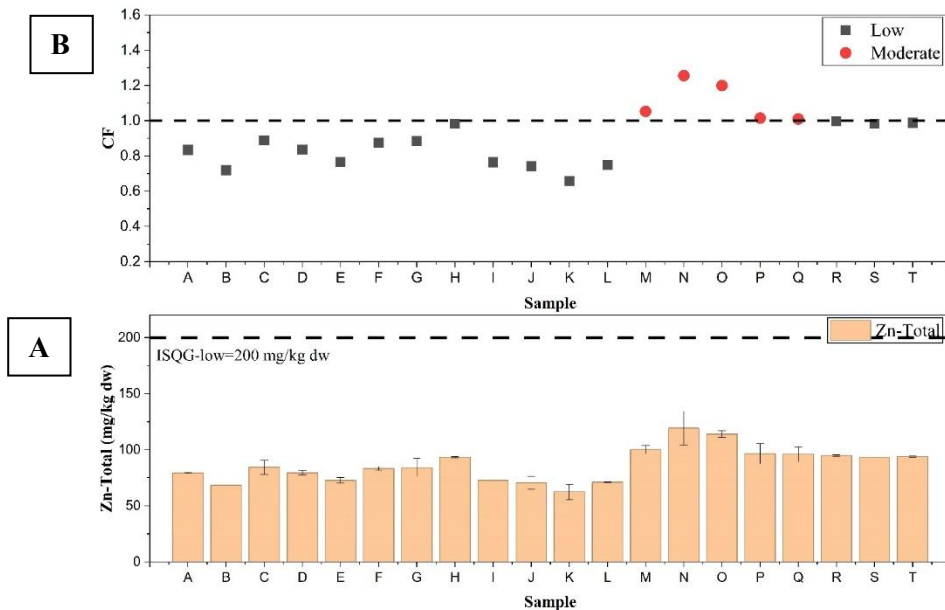
other sites. The CF values (**Figure 3(B)**) indicated that these sites experienced enrichment from their natural background, which may be associated with oil-related activities, including oil refinery operations and maritime transportation such as fishing vessels and oil tankers. Oil spills and oil sludge contain Zn<sup>6,45</sup>.

The concentration of Cu in beach sediments from the Balongan coast, Indramayu, ranged from 2.14±0.00 to 8.95±0.25 mg/kg dw, with an average of 6.53 mg/kg dw (**Figure 4(A)**). This level is still lower than the ISQG low value of 65 mg/kg dw, indicating that the condition remains safe. Cu concentrations differed significantly among sampling sites (one-way ANOVA,  $p < 0.05$ ). This finding is consistent with

sediment conditions in Karangsong waters, Indramayu, which also remain within a safe range<sup>46,47</sup>. The concentration of Cr in beach sediments from the Balongan coast, Indramayu, ranged from 14.85±0.09 to 28.81±0.42 mg/kg dw, with a median of 18.09 mg/kg dw (**Figure 4(B)**). This level is still lower than the ISQG low value of 80 mg/kg dw, suggesting that it does not pose adverse effects on aquatic organisms. Cr concentrations differed significantly among sampling sites (Kruskal-Wallis test,  $p < 0.05$ ). This finding is consistent with sediment conditions in sediment from the Balongan Indah beach, which is also classified as within safe limits<sup>14</sup>.



**Figure 2.** Lead (Pb) concentrations (A) and their CF (B) in sediments at each sampling site



**Figure 3.** Zinc (Zn) concentrations (A) and their CF (B) in sediments at each sampling site

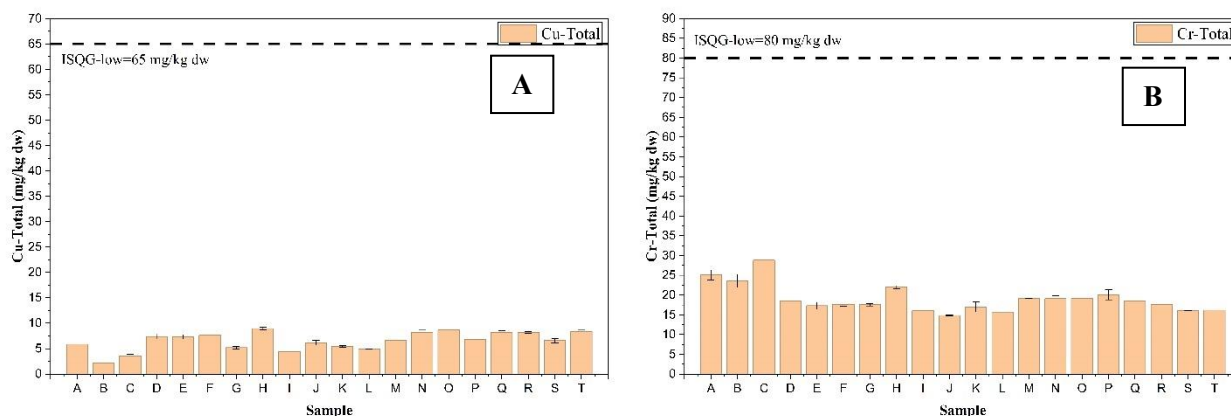


Figure 4. Copper (Cu) (A) and Chromium (Cr) (B) concentrations in sediments at each sampling site

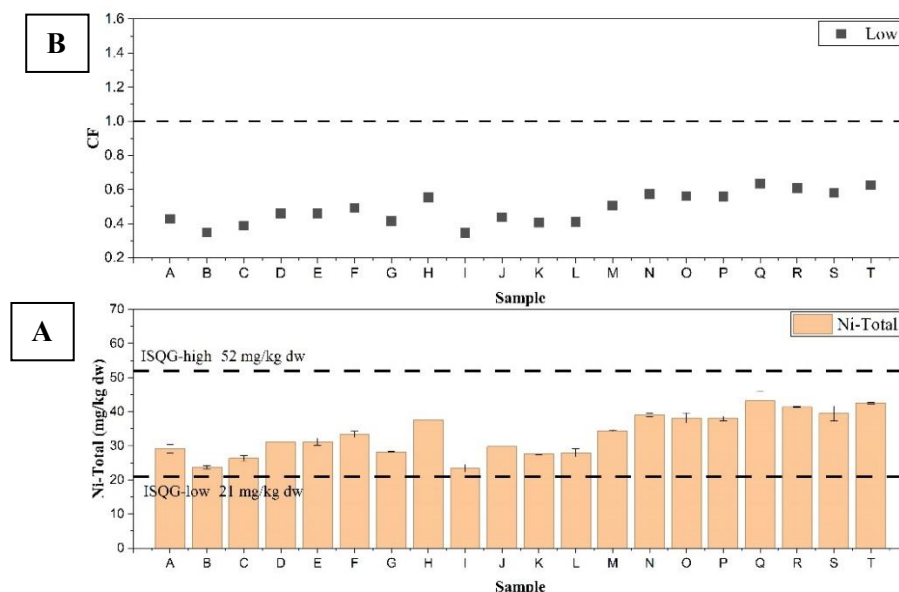


Figure 5. Nickel (Ni) concentrations (A) and their CF (B) in sediments at each sampling site

The concentration of Ni in beach sediments from the Balongan coast, Indramayu, ranged from  $23.44 \pm 1.07$  to  $43.16 \pm 2.75$  mg/kg dw, with an average of 33.28 mg/kg dw (Figure 5(A)). This level is higher than the ISQG low value of 21 mg/kg dw, but still lower than the ISQG high value of 52 mg/kg dw. In other words, this condition may cause adverse effects on aquatic organisms, although not significantly. Ni concentrations differed significantly among sampling sites (one-way ANOVA,  $p < 0.05$ ). Ni levels were found to exceed the quality guideline set by ANZECC, yet the CF values (Figure 5(B)) indicate that the presence of Ni is still within its natural background condition. This condition indicates that the input of Ni is predominantly derived from natural lithogenic sources rather than anthropogenic activities. This condition is presumed to originate from natural processes or from the weathering of volcanic rocks transported by rivers toward the estuary and coastal areas of Indramayu<sup>48</sup>.

The concentration of Cd in coastal sediments from Balongan, Indramayu, ranged from  $2.10 \pm 0.15$

to  $11.03 \pm 0.02$  mg/kg dw, with a median value of 3.99 mg/kg dw (Figure 6(A)). This concentration exceeded the ISQG-low value of 1.5 mg/kg dw but remained below the ISQG-high value of 10 mg/kg dw, except at site K. In other words, adverse effects on aquatic organisms may occur, although not significantly, except at site K. Cd concentrations at each sampling site differed significantly (Kruskal–Wallis test,  $p < 0.05$ ). The CF values (Figure 6(B)) indicated a very high enrichment of Cd compared to natural background levels. This condition is presumed to originate from anthropogenic activities unrelated solely to local industrial operations, such as oil refining. Still, it may also be exacerbated by runoff from agricultural activities and by emissions from fossil fuel combustion associated with shipping or waste burning<sup>49</sup>. Intensively used phosphate fertilizers are also suspected to be another source of contamination, as these fertilizers contain Cd as an impurity<sup>50</sup>.

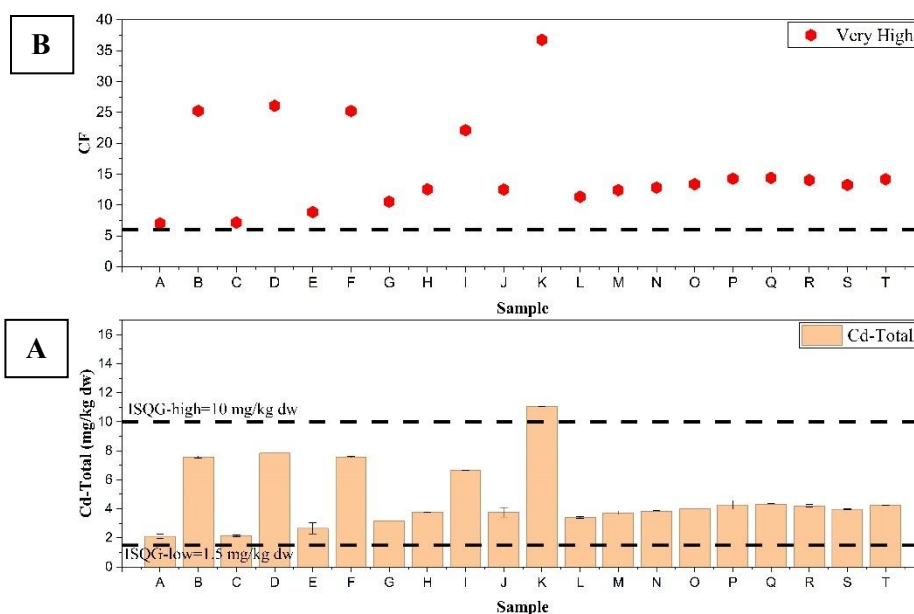


Figure 6. Cadmium (Cd) concentrations (A) and their CF (B) in sediments at each sampling site

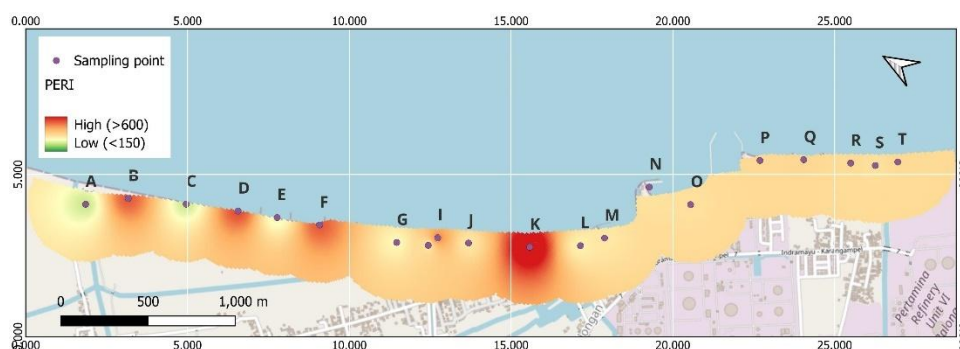


Figure 7. PERI values at each sampling site

From the above findings, adverse effects on biota may occur due to the relatively high concentrations of Cd and Ni. Based on the PERI values, coastal sediments from the Balongan coast, Indramayu, pose a potential ecological risk ranging from moderate ( $150 < PERI < 300$ ) to severe ( $PERI \geq 600$ ) (Figure 7). The elevated concentrations of Cd and Ni primarily influence this toxic condition. The order of toxicity of several metals to aquatic organisms is as follows:  $Hg^{2+} > Cd^{2+} > Ag^{+} > Ni^{2+} > Pb^{2+} > As^{3+} > Cr^{2+} > Sn^{2+} > Zn^{2+}$ <sup>51</sup>.

### Chemometric Analysis

The relationships among test parameters, location similarities, and dominant factors were examined using principal component analysis (PCA). The biplot diagram is presented in Figure 8, where the first two principal components explain 65.50% of the data variance. The results show that clay fraction and organic matter are positively correlated with heavy metals Pb, Zn, Ni, and Cu, indicating that the higher

the organic matter and clay fraction, the greater the capacity of sediments to accumulate these heavy metals. These results are consistent with numerous studies reporting significant correlations between fine sediment fractions and organic matter with heavy metals<sup>52-54</sup>. Fine fractions (clay and silt), the large specific surface area enhances the capacity to adsorb organic matter and heavy metals<sup>55</sup>. Regarding organic matter, carboxyl ( $-COOH$ ) and phenolic ( $-PhOH$ ) functional groups are the two most influential binding sites involved in metal complexation processes<sup>56</sup>. Distinct geochemical behaviors were observed for the heavy metals Cd and Cr. This finding is consistent with previous studies reporting that Cd<sup>57</sup> and Cr<sup>58</sup> show negative correlations with organic matter.<sup>49</sup>

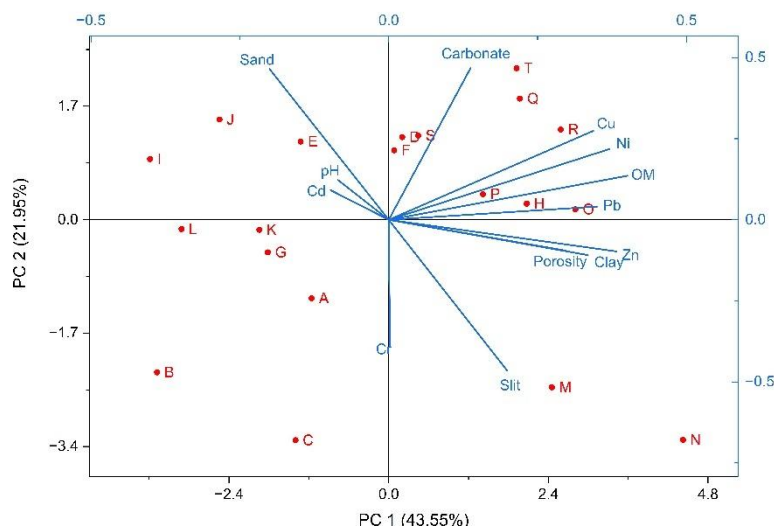


Figure 8. Biplot from PCA analysis

Table 1. Correlation coefficients between heavy metals and sediment physicochemical properties

	Porosity	Sand	Silt	Clay	pH	OM	Carbonate	Pb	Cu	Ni	Zn	Cr	Cd
<b>Porosity</b>	1.00												
<b>Sand</b>	-0.41	1.00											
<b>Slit</b>	0.41	-1.00*	1.00										
<b>Clay</b>	0.49*	-0.55*	0.55*	1.00									
<b>pH</b>	-0.41	0.00	-0.00	-0.08	1.00								
<b>OM</b>	0.58*	-0.30	0.30	0.76*	-0.06	1.00							
<b>Carbonate</b>	0.20	0.42	-0.42	0.02	0.05	0.42	1.00						
<b>Pb</b>	0.53*	-0.21	0.21	<b>0.62*</b>	-0.36	<b>0.76*</b>	0.35	1.00					
<b>Cu</b>	0.42	-0.03	0.03	<b>0.62*</b>	0.04	<b>0.93*</b>	0.54*	0.68*	1.00				
<b>Ni</b>	0.58*	-0.12	0.11	<b>0.51*</b>	-0.22	<b>0.91*</b>	0.58*	0.75*	0.85*	1.00			
<b>Zn</b>	0.57*	-0.56*	0.56*	<b>0.72*</b>	-0.12	<b>0.83*</b>	0.14	0.70*	0.64*	0.76*	1.00		
<b>Cr</b>	0.21	-0.28	0.28	-0.01	-0.52*	-0.24	-0.40	0.25	-0.33	-0.23	0.07	1.00	
<b>Cd</b>	0.07	0.21	-0.21	0.15	-0.06	-0.18	0.01	-0.20	-0.17	-0.26	-0.40	-0.24	1.00

2-tailed test of significance is used

\*: Correlation is significant at the 0.05 level

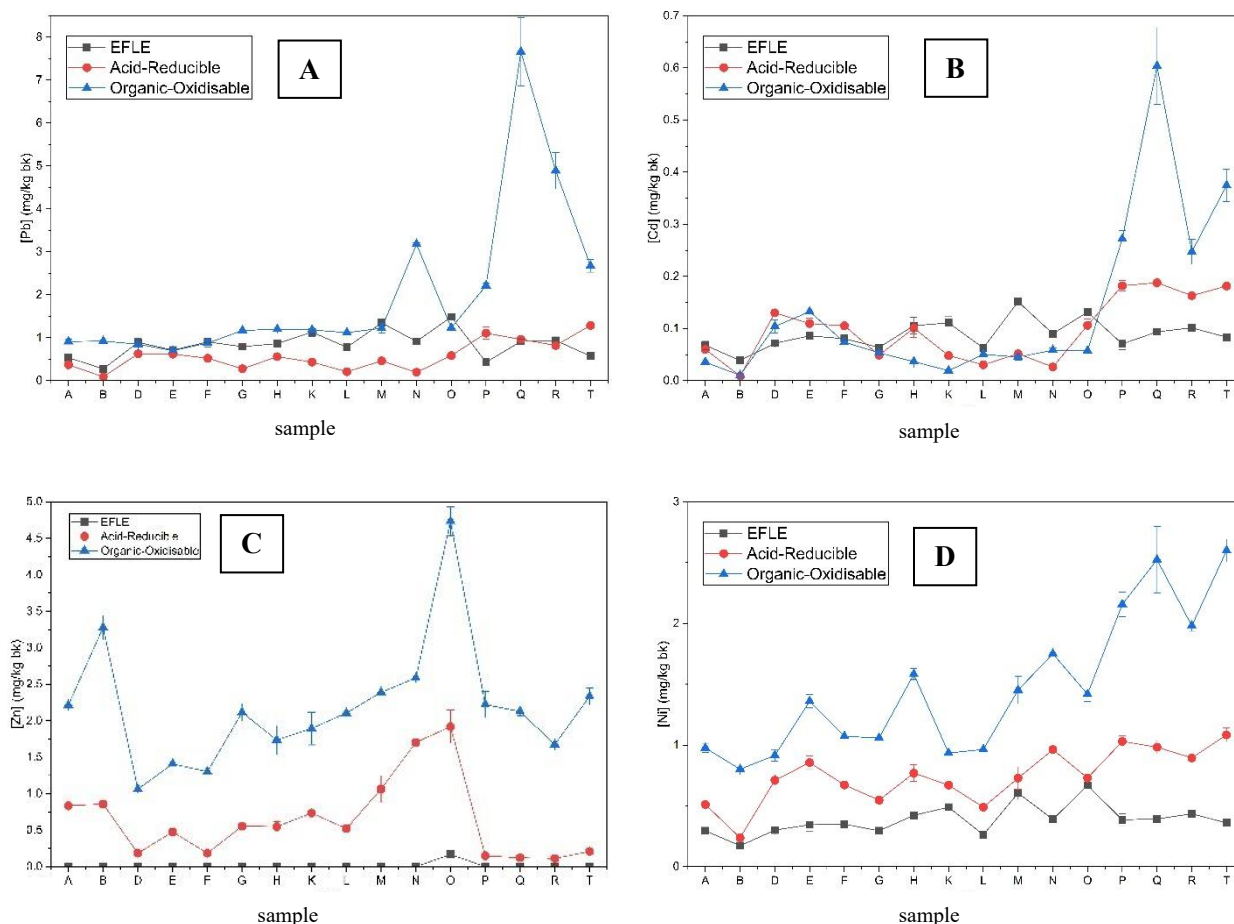
This finding is supported by the oxidisable-organic fractions of Pb, Zn and Ni (Figure 9), which are relatively higher compared to other nonresistant fractions. In contrast, Cd is not accumulated under the same conditions as the other metals. Cd tends to occur predominantly in a free or weakly bound form and is not dominantly associated with any specific sediment fraction. This behavior is attributed to Cd being one of the most mobile heavy metals in the environment. The retention of Cd in soils and sediments is mainly controlled by pH, followed by cation exchange capacity, oxide-hydroxides, clay minerals, and organic matter. Compared to other heavy metals, Cd generally exhibits thFine fractions (clay and silt), the large specific surface area enhancesFine fractions (clay and silt), the large specific surface area enhance weakest interaction with organic matter<sup>49</sup>.

This condition is similar to the findings of Arifin & Fadhlina (2009), who also reported high proportions of acid-reducible Cd in Jakarta Bay sediments. Cd tends to associate with Fe-Mn oxides and carbonates rather than with organic matter, which results in a higher mobility of Cd in sediments<sup>59</sup>.

Based on the biplot diagram presented in Figure 8, locations H, O, P, Q, R, S, and T are strongly associated with organic matter, carbonate, and heavy metals (Cu, Ni, Pb), indicating sites influenced by anthropogenic activities, such as oil refineries and maritime transport. Meanwhile, locations A, B, C, G, K, and L show minimal association with heavy metals, organic matter, and carbonate, indicating low contamination levels dominated by background or lithogenic inputs. Locations M and N are dominated by silt, clay, porosity, and Zn, reflecting fine-grained

sediments with higher metal retention capacity, where contamination patterns are mainly controlled by sediment physicochemical properties rather than direct point-source pollution. Thus, the dominant

factors influencing the variation in contamination patterns are the similarity of pollution sources and sediment physicochemical properties, particularly clay fraction and organic matter content.



**Figure 9.** Pb (A), Cd (B), Zn (C) and Ni (D) nonresistent fraction concentration at each sampling site

#### 4. CONCLUSIONS

Based on the assessment of heavy metal pollution in beach sediments from the Balongan coast, Indramayu, the concentrations of Cd and Ni exceeded the threshold limit (>ISQG-low). Other heavy metals (Pb, Zn, Cu, and Cr) remained at safe levels. According to the CF values, Cd is likely derived from anthropogenic sources (CF>6), whereas Ni originates mainly from natural lithogenic sources (CF<1). The elevated concentrations of Cd and Ni primarily influence the toxic condition, with PERI values ranging from moderate (150<PERI<300) to severe (PERI≥600). In general, higher concentrations of heavy metals were observed at sites near the oil refinery. Physicochemical properties, such as the clay fraction and organic matter, showed positive correlations with Pb, Cu, Zn, and Ni. Thus, the dominant factors influencing the variation in contamination patterns are the similarity of pollution

sources and sediment physicochemical properties, particularly clay fraction and organic matter content.

#### ACKNOWLEDGMENTS

This research is funded by Kementerian Transmigrasi Republik Indonesia in Tim Ekspedisi Patriot Program. The authors also thanks to Afidah Dzikra, Nur Hidayat, Liendy Heni Maria Maturbongs, Orlando Freziah de Araujo who have supported our research.

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