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Assessment of the Bioavailability of Zinc (Zn), Lead (Pb), Nickel (Ni), and Cadmium (Cd) in the Sediments of the Sungai Buloh Estuaries, Selangor

Aqilah Mukhtar[1]*, Mohd Nazri M. Yunus[2], Nur Amiera Kamarudin[2,3], Syaizwan Zahmir Zulkifli[2] and Ahmad Ismail[2]

[1]Department of Earth Sciences and Environment, Faculty of Science and Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia
[2]Department of Biology, Faculty of Science, Universiti Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia
[3]International Institute of Aquaculture and Aquatic Sciences, Lot 960, Jalan Kemang 6, 71050 Port Dickson, Negeri Sembilan, Malaysia

ABSTRACT

A study was conducted on sediment samples from Kuala Sungai Buloh, located within the cockle cultivation area of Selangor. The research focused on assessing the presence of selected heavy metals, including zinc (Zn), lead (Pb), nickel (Ni), and cadmium (Cd). The total concentration of heavy metals within the sediment matrix was determined through aqua regia digestion. Meanwhile, the speciation of these metals was identified using sequential extraction technique (SET). The mean concentrations of Zn, Pb, Ni, and Cd were 130.41 ± 25.61, 29.22 ± 7.81, 13.08 ± 4.96, and 0.166 ± 0.074 µg/g, respectively ($p < 0.05$). The fractionation distribution of Zn, Pb, and Ni followed the trend [residual] > [oxidisable-organic] > [acid-reducible] > [exchangeable]. For Cd, the trend was [residual] > [acid-reducible] > [oxidisable-organic] > [exchangeable]. Based on the geoaccumulation index (I_{geo}) assessment, sediments from Kuala Sungai Buloh were categorised as unpolluted to moderately contaminated at selected sampling points for Zn and Pb. The Contamination Factor (CF) suggested moderate contamination levels for Zn and Pb and low contamination levels for Ni and Cd. Additionally, the Pollution Load Index (PLI) classified the area as non-polluted since all values were less than 1. These heavy metals in the river and estuaries of Sungai Buloh are attributed to natural processes, as well as moderate industrial, agricultural, and domestic activities. Understanding heavy metals is crucial for assessing ecotoxic effects in aquatic

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E-mail addresses:
aqilahmukhtar@ukm.edu.my (Aqilah Mukhtar)
mohdnazriyunus96@gmail.com (Mohd Nazri M. Yunus)
nuramiera2510@gmail.com (Nur Amiera Kamarudin)
eazwan@gmail.com (Syaizwan Zahmir Zulkifli)
ismailmt56@yahoo.com.my (Ahmad Ismail)
* Corresponding author

environments. This knowledge is crucial for safeguarding ecosystems and advancing Sustainable Development Goal 14 (SDG14), emphasising the conservation of life below water.

Keywords: Contamination, heavy metal, sediments, sequential extraction technique, sustainability

INTRODUCTION

Heavy metal pollution is a major global issue, especially in aquatic ecosystems, due to its harmful effects on both the environment and human health. Heavy metals originate from two primary sources: natural phenomena and human activities. Natural processes such as rock weathering, soil erosion, and volcanic activity contribute to the occurrence of heavy metals in the environment. However, human activities significantly exacerbate heavy metal pollution through mining, industrial processes, improper disposal of industrial waste, urban runoff, sewage disposal and more. Heavy metals released into the environment can dissolve in water or accumulate in sediments. Heavy metals often settle into the lower layers of water bodies, making them more prevalent in sediments than in the water column (Bhuyan et al., 2023). As a result, pollutants such as heavy metals tend to accumulate in sediments (Aydın et al., 2023). Sediments are crucial in transporting heavy metals and often act as key reservoirs for these pollutants. In addition, geographical variation also plays a significant role in influencing heavy metals in sediments. A previous study conducted along the west coast of Peninsular Malaysia reported that the concentrations of heavy metals in surface sediments ranged from 66 to 443 µg/g for Zn, 2 to 54 µg/g for Pb, 11 to 30 µg/g for Ni, and 0.5 to 1.7 µg/g for Cd (Buhari, 2020). These findings suggest that higher concentrations of heavy metals were primarily associated with sampling sites in close proximity to anthropogenic sources, such as domestic discharges, industrial activities, shipping operations, and other pollution inputs.

Heavy metals are not biodegradable. Once they enter the aquatic ecosystem, they can spread throughout the water body, accumulate in sediments, or be absorbed by aquatic organisms (Frémion et al., 2016). Metals that settle in sediments are associated with bioaccumulation processes. When pollutants accumulate, they contaminate the food chain and cause significant ecotoxic stress on both humans and aquatic organisms (Okereafor et al., 2020; Wang et al., 2022). Exposure to heavy metals can lead to their buildup in human bones and fat tissue, resulting in nutritional deficiencies and a weakened immune system (Aziz et al., 2023). Additionally, certain heavy metals, such as cadmium and lead, have been associated with intrauterine growth retardation (Zinia et al., 2023). Therefore, assessing the health of aquatic ecosystems is crucial for managing pollution caused by human activities.

Most studies concentrate on the total metal content in sediment, which does not adequately address the metal's mobility, bioavailability, and toxicity (Abollino et al., 2011; Swati & Hait, 2017; Abubakar et al., 2018). Thus, evaluating the speciation of heavy metals is crucial for understanding their potential mobility and bioavailability, as

the bioavailable fraction, despite being small, plays a significant role in determining the overall metal content and its environmental impact (Swati & Hait, 2017; Abubakar et al., 2018). Heavy metal speciation can be determined using various techniques such as Sequential Extraction Techniques (SET) (Khan et al., 2020; Awad et al., 2021; Golui et al., 2021), Laser-Induced Breakdown Spectroscopy (LIBS) (Yoon et al., 2021; De Morais et al., 2022), Diffusive Gradients in Thin Films (DGT) (Yuan et al., 2020; Gu, 2021; Liang et al., 2023; Liu et al., 2023), and X-ray Absorption Spectroscopy (XAS) (Ye et al., 2022). Among these methods, SET is the most widely used due to its affordability, simplicity, and ability to provide insights into metal bioavailability, mobility, and environmental risks, which are critical for monitoring efforts.

The sequential extraction technique involves multiple steps using various chemical reagents and conditions (Domingues & e Silva, 1990). The SET procedure comprises four fractions: exchangeable, acid-reducible, oxidisable-organic, and residual. This technique differentiates between lithogenic metals and those of natural origin, offering insights into the main binding sites, the intensity of metal interactions with particulates, and the phase associations of trace elements in sediments. Understanding these aspects is crucial for deciphering the mobilisation of heavy metals and the potential risks they pose. In addition to these analytical approaches, alternative methods have also been explored to address heavy metal contamination. Phytoremediation using specific plants, such as water hyacinth (*Eichhornia crassipes*), lettuce (*Pistia stratiotes*), and duckweed (*Lemna minor*), offers a sustainable solution for mitigating heavy metal contamination (Ali et al., 2020). Phytoremediation not only addresses the contamination but also enhances the ecological health of the affected areas. Integrating such methods with sediment analysis and treatment strategies could enhance the overall effectiveness of pollution management.

Kuala Sungai Buloh has been the site of a cockle farming project initiated by the Department of Fisheries Malaysia (DOFM) since 2008. However, human activities along the Sungai Buloh River have released various pollutants, including heavy metals from industrial, boating activities, and agricultural and domestic sources. Cockle farming can be significantly influenced by sediment contamination as cockles are benthic organisms that inhabit and feed in close association with the sediment. Blood cockles and filter feeders are particularly susceptible to accumulating heavy metals and other pollutants. These contaminants can be absorbed through dissolved metals passing through their gills or ingested with food particles during dietary intake (Rainbow, 2018). Bivalves are known to bioaccumulate heavy metals in their tissues, which can lead to toxic effects such as reduced metabolic activity and impaired physiological functions (Pavón et al., 2022). The bioaccumulation in blood cockles is closely linked to their feeding behaviour and environmental conditions, making them valuable bioindicators of heavy metal pollution in aquatic ecosystems. Sediment contamination also impacts the broader ecosystem by altering habitat quality, disrupting benthic community balance, and interfering with nutrient cycling.

Therefore, this study aims to i) assess the levels of selected heavy metals, including Zn, Pb, Ni, and Cd, in the sediment of Kuala Sungai Buloh from vertical profile, ii) measure the heavy metal speciation on the surface sediment, and iii) evaluate the contamination of these heavy metals using pollution indices, such as the Index of Geoaccumulation (I_{geo}), Contamination Factor (CF), and Pollution Load Index (PLI). By integrating vertical profiling, surface sediment speciation, and pollution indices, this study comprehensively assesses the spatial distribution and environmental risks of heavy metals. The findings will contribute valuable data for monitoring pollutants released into the environment, enhancing understanding of heavy metal interactions in marine ecosystems. This is essential for protecting marine environments from harmful impacts and supports Sustainable Development Goal 14 (SDG 14), which focuses on conserving and sustainably using the oceans, seas, and marine resources.

MATERIAL AND METHOD

Study Area

The study was conducted in Kuala Sungai Buloh, Selangor (3.255880°N, 101.303401°E), located on the west coast of Peninsular Malaysia. Manufacturing industries and using fertilisers and pesticides in agricultural activities, fisheries, tourism, and domestic activities significantly contribute to metal loading in Sungai Buloh. Seven sampling stations (A, B, C, D, E, F, and G) were selected for this study (Figure 1).

Sampling

Throughout the sampling process, in-situ parameters, including temperature, salinity, dissolved oxygen (DO), and pH, were recorded using a YSI meter Model 556 MPS at a depth of approximately one meter from surface water.

Sediment collection involved the use of a 1-meter-long PVC tube with caps at each sampling point. The PVC tube was inserted until it reached the bottom sediment layer, then capped and removed. The sediment inside the PVC tube was removed using another PVC tube equipped with a piston mechanism. Subsequently, the sediments were sectioned using a plastic knife into layers of approximately 3 cm thickness from the sediment surface. A study by Mohamed et al. (2008) in Sabak Bernam, near the current study, reported that the sedimentation rate is an average of 0.2 cm/year; thus, each 3 cm section of sediment describes 15 years of sediment load. Each sediment sample was labelled with the sampling plot and its specific depth level and then placed into polyethene bags.

Sediment samples were immediately placed in an icebox for transport to the Ecology Laboratory, Department of Biology, Universiti Putra Malaysia. Upon arrival, they were frozen at -10°C to preserve them until further analysis.

Figure 1. Sampling points of sediment in Sungai Buloh estuaries, Selangor

Preparation of Samples

The sediment samples underwent thawing at room temperature for a minimum of 6 hours. The samples were then dried in an oven at 80°C for 72 hours or until a constant dry weight was achieved. After drying, the samples were ground using a mortar and pestle to obtain a fine powder. The powdered samples were then sieved through a 63 µm stainless steel sieve to ensure a consistent particle size. The prepared samples were kept in sealed plastic bags with labels to prevent contamination prior to further laboratory procedures, such as aqua regia and sequential extraction techniques.

Experimental Analysis

Total Heavy Metals in Sediment by Aqua Regia Method

The total metal content in the sediment was determined through aqua regia digestion (Ismail & Ramli, 1997; Zulkifli et al., 2010). Digestion was performed on sediment samples categorised by plot and depth level. A sample weighing approximately 1.0 g was placed in a digestion tube. A 10 mL of a mixed acid solution, prepared in a 4:1 ratio of concentrated

nitric acid (HNO_3, 65%, AnalaR grade, R&M Chemicals) to perchloric acid ($HClO_4$, 70%, AnalaR grade, R&M Chemicals), was added to the sample. The digestion tubes were then placed in a digestion block and heated to ensure complete digestion of the samples. The temperature was initially set to 40°C for the first hour, followed by an increase to 140°C for the next three hours. Once digestion was finished, the tubes were allowed to cool to room temperature. The volume was adjusted to 40 ml by adding double-distilled water, and the samples were subsequently filtered using Whatman® No. 1 filter paper (12.5 cm diameter and 11 µm pore size). The filtered samples were analysed using an Atomic Absorption Spectrophotometer (AAS) (Perkin-Elmer Model AAnalyst 800). The limit of detection (LOD) for Zn, Pb, Ni and Cd were 0.01 µg/g, 0.02 µg/g, 0.05 µg/g, and 0.001 µg/g, respectively.

Speciation of Heavy Metal in Sediment using Sequential Extraction Technique (SET)

Surface sediment from each sampling point was analysed for heavy metal speciation using the sequential extraction technique (SET), which was adapted from the methods of Badri and Aston (1983), Naji et al. (2010) and Abubakar et al. (2018).

A 10 g sediment sample was added to a conical flask to determine the exchangeable fraction. About 15 ml of 1.0 M ammonium acetate (NH_4CH_3COO) solution was then introduced, adjusted to pH 7.0. The mixture was subjected to continuous shaking at 100 rpm for 3 hours at room temperature. Subsequently, the mixture was filtered using Whatman filter paper, and the obtained supernatant was immediately analysed.

For the acid-reducible fraction, the residue from the first fraction was mixed with 50 ml of 0.25M hydroxylammonium chloride ($NH_2OH{\cdot}HCl$), acidified to pH 2.0 with hydrochloric acid (HCl), and shaken for another 3 hours at 100 rpm at room temperature.

The residue from the preceding step was transferred to a conical flask to determine the oxidisable organic fraction. This residue was oxidised by adding 15 ml of 35% hydrogen peroxide (H_2O_2, R&M Chemicals) and placing it in a 90°C water bath for at least 4 hours until the sediment was completely dry. After cooling to room temperature, the residual sediment was treated with 50 mL of 1.0 M ammonium acetate (NH_4CH_3COO) solution, adjusted to pH 2.0 using hydrochloric acid (HCl). The mixture was then agitated at 100 rpm for 3 hours at room temperature.

In the resistant fraction, the residue is digested according to the procedure outlined for total digestion using the aqua-regia method. All collected supernatants were stored at 4°C before metal analysis.

The residue from each extraction phase was rinsed with 20 mL of distilled water, filtered, and weighed before proceeding to the subsequent extraction fraction. The concentrations of heavy metals in all filtrate samples were then determined using an atomic absorption spectrophotometer (Perkin-Elmer Model Analyst 800).

Quality Control

Acid washing was performed on all glassware and equipment to eliminate contamination. Blank samples were processed concurrently with each experimental batch to verify sample purity. The accuracy of the analytical procedure was validated using Certified Reference Materials (CRM) for marine sediment (PACS-2), with recoveries ranging from 80.48% to 113.85%, as detailed in Table 1. The data obtained from the analysis were converted to and expressed on a µg/g dry weight basis. These results were then compared and adjusted using CRM values to determine the final results.

Table 1
The CRM value and the percentage of recoveries

Metals	Certified Reference Value (ug/g)	Measured Value (ug/g)	Percentage of Recovery (%)
Zn	49.900	43.737	87.65
Pb	11.700	11.397	97.41
Ni	39.500	31.790	80.48
Cd	2.110	1.849	87.65

Environmental Indices

Metal pollution levels in the surface sediment of Kuala Sungai Buloh were evaluated using three environmental indices: the Geoaccumulation Index (I_{geo}), Contamination Factor (CF), and Pollution Load Index (PLI).

a) The Geoaccumulation Index (I_{geo}), developed by Muller (1969), was employed to assess metal contamination in the sediment. This index compares current metal concentrations to pre-industrial baseline levels (Equation 1).

$$I_{geo} = \log_2 (C_n / 1.5B_n) \quad [1]$$

In the equation, Cn represents the measured concentration of element n in the sediment sample, while Bn signifies the geochemical background concentration of element n in average shale, as established by Turekian and Wedepohl in 1961. Table 2 presents the seven distinct categories of I_{geo}, along with the corresponding levels of sediment pollution proposed by Muller (1969).

b) The Contamination Factor (CF) is an index used to evaluate sediment contamination and assess environmental pollution from specific toxic substances. The CF is determined by dividing the sediment concentration of each heavy metal by its corresponding background value, as defined by Turekian and Wedepohl (1961) in Equation 2.

$$CF = \frac{(\text{heavy metal concentration in sediment})}{(\text{background value of metal})} \quad [2]$$

Table 2
Classification for the geoaccumulation index (I_{geo})

I_{geo} value	I_{geo} class	Sediment quality
<0	0	Unpolluted
0–1	1	Unpolluted to moderately polluted
1–2	2	Moderately polluted
2–3	3	Moderately polluted to strongly polluted
3–4	4	Strongly polluted
4–5	5	Strongly polluted to extremely polluted
>5	6	Extremely polluted

In the equation, the criteria for assessing CF are defined as follows: CF < 1 indicates low contamination, 1 < CF < 3 indicates moderate contamination, 3 < CF < 6 indicates considerable contamination, and CF > 6 indicates very high contamination. The background concentrations of metals used in this assessment were derived from global average shale values reported by Turekian and Wedepohl (1961).

c) The Pollution Load Index (PLI) comprehensively evaluates heavy metal contamination at potentially polluted locations. The PLI indicates the cumulative toxicity of a sample, incorporating the impacts of various heavy metals. The PLI was determined using Equation 3, proposed by Tomlinson et al. (1980).

$$PLI = (CF_1 \times CF_2 \times CF_3 \ldots \times CF_n)^{1/n} \quad [3]$$

In the equation, CF refers to contamination factors, and n is the number of metals. A PLI less than 1 signifies no pollution, while a value greater than 1 indicates pollution.

Data Analysis

Heavy metal concentration data was analysed using IBM SPSS Statistics version 21 and Microsoft Office Excel 2021 (Microsoft Corporation, USA). One-way ANOVA followed by Tukey's post-hoc test was utilised to determine differences in heavy metal concentrations among sampling points.

RESULTS AND DISCUSSION

Water Quality of Study Areas

The water parameters of Kuala Sungai Buloh are recorded in Table 3. The water temperature ranged from 28.40°C to 30.76°C. Sampling points A to F exhibited consistent temperatures, except for sampling point G, which recorded the lowest temperature. Factors contributing to the variation in temperature values include weather and climate conditions, sampling time, and the locations of the sampling points. In this study, the high temperatures were due to

sunny weather during the sampling activities, and sampling points A, B, C, D, E, and F were in open estuaries. In contrast, sampling point G was in the Sungai Buloh riverine, which was surrounded by palm oil trees. Temperature influences various water parameters, including dissolved oxygen (DO), pH, and nutrient levels. Elevated temperatures reduce oxygen solubility in water while increasing microbial metabolism, resulting in higher oxygen consumption (Chapra et al., 2021). This process leads to increased carbon dioxide (CO_2) release, subsequently lowering pH levels. The resulting low pH caused by elevated CO_2 can impose additional stress on aquatic organisms, impacting their physiological functions and overall ecosystem health (Peytoureau et al., 2023). Furthermore, elevated temperatures enhance microbial enzymatic activity, accelerating nutrient cycling, decomposition rates, and nutrient release (Elena, 2019).

Table 3
The in-situ parameters of water in all sampling points of Sungai Buloh

Sampling point	Parameter			
	Temperature (°C)	Salinity (mg/l)	Dissolve oxygen (mg/l)	pH
A	30.36	28.22	7.07	6.22
B	30.36	26.03	7.39	6.71
C	29.25	26.80	8.77	8.22
D	30.40	41.40	6.59	8.06
E	30.62	41.09	9.41	6.52
F	30.76	39.76	11.64	6.50
G	28.40	0.44	3.40	5.75
Average	30.02	29.11	7.75	6.85

The highest salinity was recorded at sampling points D and E, with values of 41.40 and 41.09 mg/l, respectively, while the lowest value was recorded at sampling point G (0.44 mg/l). Variations in salinity are influenced by river runoff, where fresh water from rivers mixes with seawater, and ocean currents distribute water masses with varying levels of salinity. Varying salinity levels can influence organisms' distribution and population size within a specific region.

The analysed water samples exhibited dissolved oxygen (DO) levels ranging from 3.40 to 11.64 mg/L. These values comply with the Malaysian Marine Water Quality Standard (MMWQS) Class 1 for sampling points A to F, except for sampling point G, which falls into Class 3. Industrial activities, oil palm plantations, and domestic activities characterise sampling point G. Dissolved oxygen levels are influenced by the photosynthetic production of oxygen by phytoplankton and aquatic plants, which increases during daylight hours. Conversely, biological respiration and bacterial decomposition of organic matter consume oxygen, leading to decreased DO levels. This effect is particularly pronounced in areas with high organic loads, such as sampling point G.

The pH levels ranged from 5.75 at sampling point G to 8.22 at sampling point C. Results falling between 6.5 and 9.0 are within the standard range of the MMWQS. A pH below 6.5 indicates poor water conditions, often due to industrial emissions from chemical manufacturing, paper production, and metal processing upstream of Sungai Buloh. These discharges can introduce acidic substances that directly lower the pH of the receiving water. Significant pH fluctuations can disrupt biodiversity, food webs, and ecosystem dynamics.

Overall, the water quality in the estuary of Sungai Buloh is generally good, except at sampling point G, where discharges from industries, oil palm plantations, and domestic activities contribute to the alteration of water quality. Monitoring and managing water's physicochemical parameters are crucial for protecting aquatic biodiversity and ensuring the sustainability of aquatic resources.

Mean Metal Concentrations of Sediment Cores

Table 4 presents the mean Zn, Pb, Ni, and Cd concentrations at varying depths within the sediment core samples. The mean concentration of metals in sediments that are being studied is generally in order of [Zn] > [Pb] > [Ni] > [Cd]. Zn has the highest concentration with a mean of 130.20 ± 5.02 µg/g, followed by Pb (29.24 ± 1.31) µg/g, Ni (13.23 ± 0.63) µg/g, and Cd (0.16 ± 0.01) µg/g ($p < 0.05$). Compared to the average shale values of heavy metals, Zn and Pb exceed the average shale values, clearly indicating the enrichment of these metals in the core. In contrast, the mean values of Ni and Cd are below the average shale values.

The mean concentration of Zn rises towards the top of the sediment core, whereas Pb, Ni, and Cd showed minimal variation throughout the vertical sediment core. Numerous researchers have employed sediment cores to investigate the behaviour of metals, including in Malaysian marinas (Ashraf et al., 2018; Khalid et al., 2019; Vane et al., 2020; Pradit et al., 2022). The concentration of Zn at the bottom of the sediment core is lower than the mean, while the highest Zn concentration was detected at a depth of 19 to 21 cm at a concentration of 140.60 µg/g. For Pb, the highest concentration detected was 31.43 µg/g at a depth of 19 to 21 cm, and the lowest was 27.52 µg/g at a depth of 22 to 24 cm. Similarly, for Ni, the highest and lowest concentrations were detected at depths of 19 to 21 cm and 22 to 24 cm, with concentrations of 14.22 µg/g and 12.27 µg/g, respectively. The similar trend of Zn, Pb and Ni suggests that metals were highly distributed within the 15 years from 1825 to 1913 in this area. However, Cd concentrations in the sediment core of Sungai Buloh remained consistent, with 0.15 µg/g between depths of 1 to 21 cm and 0.17 µg/g between depths of 22 to 36 cm. In contrast, the vertical sediment core study by Lasumin et al. (2022) at Sungai Buloh reported the highest concentrations of Zn, Pb, and Cd as 80.18 µg/g, 23.24 µg/g, and 0.19 µg/g, respectively. The highest concentrations were observed at depths of 9–12 cm for Zn and Pb and 0–3 cm for Cd.

In fact, the metals accumulated in the sediment core show a historical input in previous years in the study area. The difference in metal values is recorded due to metals that are produced naturally or also from a variety of human activities. It should be emphasised that the high Zn concentration may be attributed to the use of fertiliser in the past few years, when agriculture, such as oil palm cultivation, is actively carried out in this area. For instance, elevated levels of heavy metals, including Cu and Zn, have been detected in soil from Felda Jengka 8, Pahang, attributed to the use of fertilisers in oil palm plantations (Manan et al., 2018).

Table 4
The mean concentrations of heavy metals in the sediment core of Sungai Buloh (µg/g)

Depth (cm)	Years	Zn	Pb	Ni	Cd
1–3	2003–2018	131.31	28.23	13.13	0.15
4–6	1988–2003	128.46	27.14	12.50	0.14
7–9	1973–1988	131.99	28.69	13.30	0.15
10–12	1958–1973	133.70	29.25	13.98	0.15
13–15	1943–1958	127.30	28.36	12.32	0.15
16–18	1928–1943	134.83	29.89	13.61	0.15
19–21	1913–1828	140.60	31.43	14.22	0.15
22–24	1898–1913	123.78	27.52	12.27	0.18
25–27	1883–1898	130.88	29.28	13.50	0.17
28–30	1868–1883	131.45	30.04	13.63	0.17
31–33	1853–1868	123.61	30.34	12.97	0.17
34–36	1838–1853	124.50	30.75	13.36	0.16
Mean		130.20	29.24	13.23	0.16
Std		5.02	1.31	0.63	0.01

Vertical Heavy Metal Profiles of Sediment Core

Zinc (Zn)

Figure 2 shows the profiles of Zn in the vertical sediment cores collected from seven sampling points. At sampling point A, the Zn concentration was 109.53 µg/g in surface sediment (1–3 cm depth) and showed small variation between depths of 4–27 cm, with a mean concentration of 126.02 µg/g. A relatively high Zn concentration of 161.87 µg/g was observed in the layer at a 28–30 cm depth. At sampling point B, the Zn concentration decreased in the bottom sediment core at a depth of 22–36 cm, with a higher concentration recorded at 19–21 cm, and the lowest concentration found at a depth of 13 to 15 cm with concentrations of 146.16 and 125.82 µg/g, respectively. This pattern is similar to the Zn profile recorded at sampling point E. At sampling point C,

a high Zn concentration of 131.6 µg/g was recorded in the upper sediment; then, the values decreased until a depth of 13 to 15 cm. Variation in Zn concentration occurred in the middle and lower layers of the sediment core. At sampling point D, the deeper the sediment, the higher the Zn concentration, with the highest concentration of 121.67 µg/g recorded at a depth of 31–36 cm. Sampling point F showed the highest Zn concentration at the sediment surface, 138.55 µg/g, with large variations along the vertical core. Unlike the other sampling points, at sampling point G, the Zn concentration decreased from 1–18 cm depth, but at 19–21 cm, it recorded the highest concentration of 208.1 µg/g. The pattern of Zn concentrations observed in the sediment core from Sungai Buloh was comparable to findings reported by Baharudin et al. (2021) in their study of the Strait of Malacca, which covered areas in Johor, Selangor, and Penang. The presence of Zn in the Sungai Buloh sediment is primarily attributed to water runoff from palm oil plantations and effluents from nearby manufacturing industries.

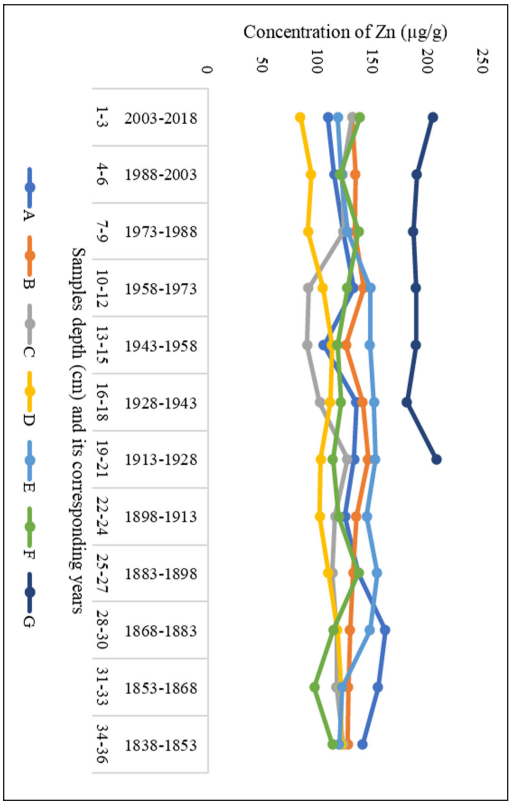

Figure 2. The concentration of Zn in sediment cores from Sungai Buloh

Lead (Pb)

For sampling points A and B, the Pb concentration in the sediment core increased from the 1-12 cm layer (Figure 3). The variation in Pb concentration is small in the middle to bottom layers for both sampling points, with Pb concentrations recorded at 31.89 and 28.68 µg/g, respectively, in the final layer. Generally, Pb concentration decreased toward the middle depth of sampling points C, F, and G. Pb increased relatively in the layer 13 to 21 cm before stabilising in the layers 22 to 30 cm and slightly increasing in the lower layer of sampling point C. Sampling point F showed small variation in Pb concentration, but the lowest Pb concentration was documented in the 31–33 cm layer at 21.73 µg/g. The Pb concentration at sampling point G was higher compared to other points, with the highest value of 51.71 µg/g in the surface sediment. Sampling points D and E exhibited a similar pattern, where Pb concentration increased with depth, reaching the highest concentrations

of 36.65 and 36.18 µg/g, respectively, in a final depth of 34 to 36 cm. The variation of Pb in the sediment core was also observed in the Matang Mangrove Forest Reserve (Mustapha et al., 2022), with Sungai Buloh recording higher Pb levels in the sediment compared to this area. The presence of Pb in the sediment is attributed to boating activities related to fisheries and tourism, where gasoline or diesel is used for boat engines. The large number of boats and potential gasoline leakage can significantly increase Pb levels in the water and sediment (Pazi et al., 2021; Mustapha et al., 2022).

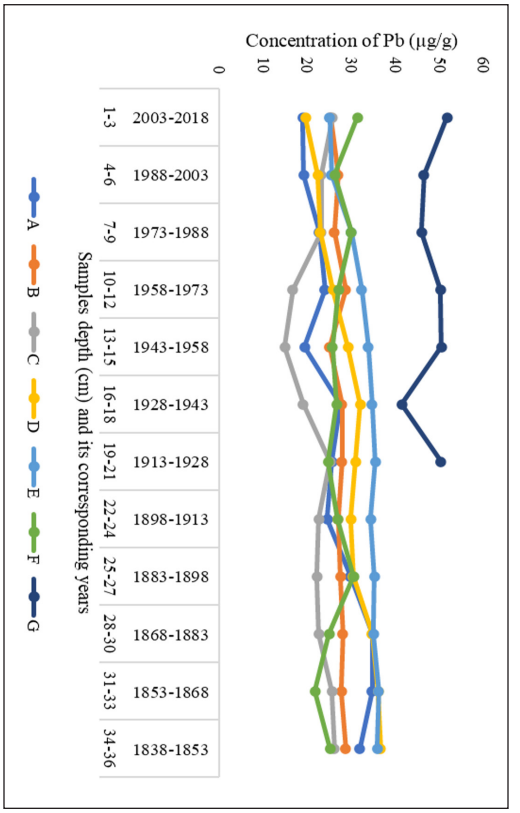

Figure 3. The concentration of Pb in sediment cores from Sungai Buloh

Nickle (Ni)

For sampling points A, D, and E, the Ni concentration followed a similar pattern: an increase in the sediment core from 1 to 18 cm followed by minor variation in the next layer (Figure 4). This Ni pattern is similar to the study by Omorinoye et al. (2019) in the Sadong River in Sarawak, where the concentration also increased with depth. Among these three points, sampling point E exhibited the highest Ni concentration, reaching 21.39 µg/g at the 25–27 cm depth layer. At sampling point B, the highest Ni concentration was 16.78 µg/g, detected in the 1–12 cm layer, after which it decreased, showing only small variations from 13–36 cm in depth. Sampling point C showed large variations in Ni concentration between 1–21 cm. The lowest concentration of 4.31 µg/g was recorded at 13–15 cm, then increased until 19–21 cm, followed by a slight decrease and stable values at 22–30 cm, then increased to 12.10 µg/g at 34 to 36 cm. At sampling point F, both the upper layer from 1 to 9 cm and the bottom layer from 25 to 36 cm exhibited significant variations in Ni concentration, while the middle core (10 to 27 cm) showed small variations. For sampling point G, the Ni concentration slightly decreased with depth, but at the final layer of 19–21 cm, it increased to 21.78 µg/g. The concentration of Zn in Sungai Buloh was comparable to that in Sadong River but significantly lower than in the Sungai Buloh in Selangor (Abubakar et al., 2018). Manufacturing activities, including those related to the chemical processes industry, contribute to the presence of Ni in the sediment of Sungai Buloh.

Cadmium (Cd)

At sampling sites A, F, and G, the Cd concentration in the sediment core consistently remained below 0.10 µg/g, with mean values of 0.05 µg/g, 0.09 µg/g, and 0.01 µg/g, respectively (Figure 5). At sampling site A, the Cd concentration increased with depth, whereas at site F, it increased towards the sediment surface. This pattern aligns with findings by Lasumin et al. (2022) in Sungai Buloh, which also reported an increase in Cd concentration towards the surface sediment. At sampling point B, the Cd concentration showed small variations from 1 to 24 cm, with the highest value of 0.26 µg/g recorded at the 19 to 21 cm layer, then decreased towards the bottom layer from 25 to 36 cm. For sampling point C, the Cd concentration decreased from 1 to 12 cm, then increased from 13 to 36 cm, with the highest value of 0.24 µg/g. A constant Cd concentration was observed throughout the sediment core at sampling point D. At sampling point E, variations in Cd concentration were recorded throughout the depth of the sediment core, with the highest

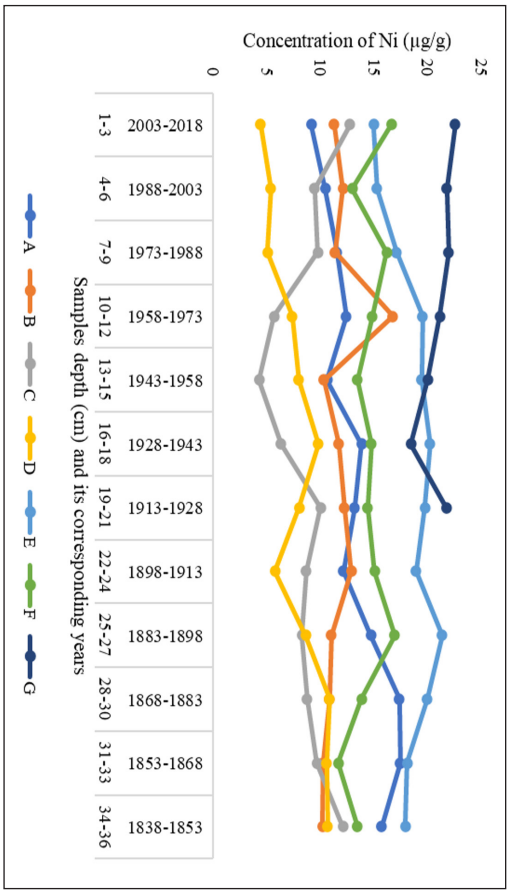

Figure 4. Concentration of Ni in sediment cores from Sungai Buloh

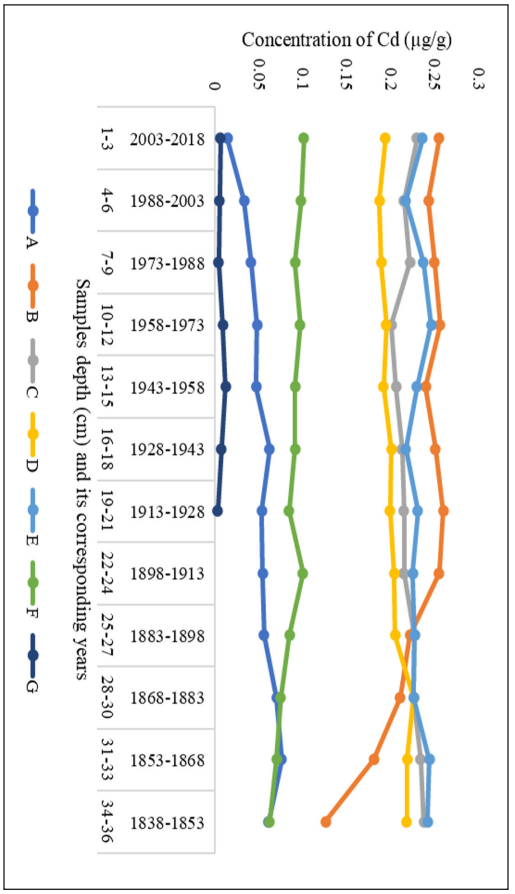

Figure 5. Concentration of Cd in sediment cores from Sungai Buloh

value of 0.25 µg/g at a depth between 10 to 12 cm. The Cd concentration at sampling site G is much lower due to the characteristics of the depositional environment, such as low water pH, which can increase Cd mobility (Zhang et al., 2023). As a result, a significantly lower concentration of Cd was detected at this site. However, the concentration of Cd observed in Sungai Buloh is significantly higher compared to the levels reported in previous studies conducted in Bagan Pasir, Perak; Sungai Buloh, Selangor; and Port Dickson, Negeri Sembilan (Lasumin et al., 2022). Human activities in the town within the study area, such as smelting manufacturing, release effluents into the drainage system, eventually accumulating in Sungai Buloh over time.

The Zn, Pb, and Ni levels were highest on average at sampling point G in the Sungai Buloh sediment cores. Results suggest that heavy metal pollution in the river originated from a combination of upstream contamination and effluents discharged from human activities in the Kuala Sungai Buloh area. This has led to an increased concentration of heavy metals at location G. As a result, heavy metals eventually accumulated in and adhered to the sediment. In contrast to the sampling point in the estuary, runoff carrying heavy metals mixed with seawater was affected by tides, reducing heavy metals' concentration before they finally accumulated in the sediment. In line with the analysed water parameters, the water quality at sampling point G is not particularly good, according to MMWQS. Heavy metals in Malaysian rivers are primarily attributed to anthropogenic and natural occurrences. For example, the anthropogenic activities include industrial waste, agricultural runoff, domestic effluents and landfill materials, as observed in Sungai Merbok in Kedah, Sungai Sembilang in Selangor, and Sepang Besar River in Selangor (Ateshan et al., 2020; Ibrahim et al., 2020; Krishnan et al., 2022).

This study's metal concentrations in sediment cores were compared to previous research in Malaysian marinas. The data revealed significantly higher heavy metal concentrations in the study area compared to sediment cores from coastal Sabah, Kong Kong estuaries in Johor, and the west coast of Peninsular Malaysia (Ashraf et al., 2018; Khalid et al., 2019; Lasumin et al., 2022). However, the mean concentrations of heavy metals from the seven sampling sites were lower than those reported in studies from Kaohsiung Harbor, Taiwan, and Dachan Bay, China (Chen et al., 2016; Yang et al., 2020).

To summarise, heavy metal concentrations exhibited significant spatial variability among the seven sediment cores, possibly due to different hydrodynamic mechanisms, including differences in heavy metal release sources and tidal water, varying sediment core characteristics, and homogeneous or inhomogeneous contaminants which are introduced into rivers and estuaries (Chen et al., 2016). However, Zn and Pb in the vertical sediment cores from sampling points B, D, and E showed a relatively stable pattern. This suggests that chemicals were released from the river and subsequently accumulated near the river mouth with minimal disturbance.

Bioavailability of Heavy Metals in Sediments Samples

Table 5 shows the concentration and distribution of heavy metals in surface sediment. The ranges for Zn, Pb, Ni, and Cd are 126.73 to 199.16, 31.40 to 52.99, 12.00 to 23.93 and 0.003 to 0.070 µg/g, respectively.

In this study, the Zn was mainly residual form, particularly in the surface sediments of sampling points A, B, C, D, and E. The residual proportion ranged from 41.42% to 59.34%. Meanwhile, sampling points F and G were dominated by acid-reducible fractions at 43.39% and 39.64%, respectively. The surface sediments of the study area documented Zn fractionation order of [residual] > [oxidisable-organic] > [acid-reducible] > [exchangeable]. Pb was mainly in the residual form, with a proportion of 41.61 to 63.60 % and an average residual component of 52.50%. The average percentage of Pb proportion of the exchangeable (F1), acid-reducible (F2), and oxidisable-organic fraction (F3) are 1.13, 14.0, and 33.0%, respectively. The fractionation order of Pb was [residual] > [oxidisable-organic] > [acid-reducible] > [exchangeable]. Ni was also mainly in residual form, ranging from 37.43% to 53.05%, averaging 47.06%. The exchangeable fraction has the lowest average of 4.26%. Meanwhile, acid-reducible and oxidisable-organic Ni ranged from 10.13% to 31.69% and 22.66% to 39.79%, respectively. To summarise, the Ni in surface sediments has fractionation in order of [residual] > [oxidisable-organic] > [acid-reducible] > [exchangeable]. Furthermore, Cd was primarily in the residual form, accounting for

Table 5
Concentration of heavy metal for sequential metal extraction (mean ± SD) µg/g and percentage (%)

Sampling point	Zn	Pb	Ni	Cd
A				
F1	1.31±0.23 (0.69)	NA	0.59±0.04 (3.40)	0.004±0.001 (4.48)
F2	48.88±2.26 (25.95)	1.82±0.92 (4.02)	2.00±0.14 (11.47)	0.010±0.002 (11.17)
F3	60.17±0.55 (60.17)	16.84±0.99 (37.15)	6.94±0.11 (39.79)	0.017±0.002 (17.95)
F4	78.04±0.56 (78.04)	26.66±3.02 (58.83)	7.91±0.13 (45.34)	0.061±0.009 (66.40)
Sum total	188.4	45.32	17.45	0.092
Digestion	144.01	31.14	15.5	0.063
Differences	44.39	14.18	1.95	0.029
B				
F1	0.57±0.17 (0.34)	NA	0.65±0.03 (3.44)	0.006±0.001 (7.15)
F2	25.16±0.95 (15.28)	1.09±0.3 (2.66)	1.96±0.07 (10.33)	0.006±0.001 (7.24)
F3	52.87±2.18 (32.1)	16.06±4.44 (39.14)	6.97±0.33 (36.82)	0.010±0.002 (12.97)
F4	86.09±6.10 (52.27)	23.88±5.39 (58.19)	9.36±0.82 (49.41)	0.056±0.006 (72.65)
Sum total	164.69	41.03	18.94	0.077
Digestion	130.75	27.8	11.05	0.199
Differences	33.94	13.23	7.89	0.122

Table 5 *(continue)*

Sampling point	Zn	Pb	Ni	Cd
C				
F1	0.67±0.09 (0.4)	0.44±0.27 (1.02)	0.74±0.04 (3.84)	0.004±0.002 (5.27)
F2	24.98±0.35 (14.92)	2.27±0.25 (5.22)	1.95±0.06 (10.13)	0.006±0.001 (7.8)
F3	49.76±0.75 (29.72)	18.67±1.61 (43.01)	6.35±0.33 (32.98)	0.011±0.002 (13.57)
F4	92.01±7.71 (54.96)	22.03±2.09 (50.76)	10.22±0.28 (53.05)	0.058±0.009 (73.37)
Sum total	167.41	43.41	19.25	0.079
Digestion	117.49	23.83	9.5	0.228
Differences	49.92	19.58	9.75	0.149
D				
F1	0.39±0.06 (0.25)	0.87 (1.96)	0.71±0.05 (4.26)	0.003±0.001 (3.42)
F2	20.61±0.93 (13.12)	2.22±0.20 (5.02)	1.80±0.15 (10.86)	0.008±0.002 (9.45)
F3	42.87±1.78 (27.29)	18.44±2.16 (41.68)	5.40±0.23 (32.54)	0.011±0.001 (13.2)
F4	93.20±24.93 (59.34)	22.71±4.37 (51.35)	8.68±0.77 (52.34)	0.060±0.007 (73.93)
Sum total	157.08	44.24	16.58	0.081
Digestion	114.65	33.55	9.31	0.214
Differences	42.43	10.69	7.27	0.133
E				
F1	1.75±0.09 (1.18)	NA	0.94±0.08 (6.19)	0.006 (7.5)
F2	31.90±8.80 (21.54)	0.89±0.65 (2.32)	1.84±0.23 (12.1)	0.015±0.003 (17.5)
F3	40.56±12.83 (27.39)	13.11±7.03 (34.09)	4.95±1.81 (32.59)	0.008±0.004 (9.11)
F4	73.89±8.05 (49.89)	24.45±6.68 (63.6)	7.46±1.67 (49.12)	0.055±0.013 (65.89)
Sum total	148.1	38.45	15.18	0.083
Digestion	137.78	35.33	19.28	0.233
Differences	10.32	3.12	4.1	0.15
F				
F1	1.86±0.18 (1.47)	NA	0.70±0.04 (5.8)	0.004±0.001 (7.8)
F2	55.01±11.27 (43.39)	10.35±7.28 (32.97)	2.87±1.13 (23.93)	0.015±0.006 (26.58)
F3	26.45±15.50 (20.86)	7.98±6.43 (25.42)	3.94±1.59 (32.83)	0.003±0.002 (5.07)
F4	43.45±11.19 (34.28)	13.07±4.06 (41.61)	4.49±0.73 (37.43)	0.035±0.010 (60.55)
Sum total	126.78	31.4	12.02	0.057
Digestion	116.36	25.86	14.21	0.078
Differences	10.41	5.54	2.19	0.021
G				
F1	4.31±0.28 (2.17)	0.21 (0.4)	0.70±0.07 (5.8)	NA
F2	78.95±3.95 (39.64)	24.32±1.37 (45.9)	7.58±0.48 (31.69)	0.031±0.003 (30.52)
F3	41.78±6.79 (20.98)	5.73±1.86 (10.82)	5.42±0.59 (22.66)	NA
F4	74.12±1.77 (37.21)	22.72±4.55 (42.88)	10.23±0.49 (42.85)	0.071±0.027 (69.48)
Sum total	199.16	52.99	23.93	0.103
Digestion	191.1	25.86	20.7	0.117
Differences	8.06	27.13	3.23	0.014

an average of 68.90% of the residual component. The proportion of exchangeable, acid-reducible, and oxidisable-organic fractions is 5.09%, 15.75% and 10.27%, respectively. Hence, the fractionation of Cd in surface sediments of the study areas is arranged in order of [residual] > [acid-reducible] > [oxidisable-organic] > [exchangeable].

The residual fraction of heavy metals exhibited the highest concentration among all fractions, suggesting that most heavy metals found in the surface sediment of Sungai Buloh are derived from natural sources (Figure 6). High metal concentrations in the residue fraction may be attributed to proximity to mangrove areas and geological weathering (Doabi et al., 2017; Swati & Hait, 2017). Additionally, metals in this fraction are deemed biologically unavailable because they are bound within the crystal lattice, rendering them inaccessible (El-Azim & El-Moselhy, 2005). Meanwhile, the first exchangeable fraction has the lowest average percentage of metal, followed by the acid-reducible and oxidisable-organic fractions. Together, these three fractions constitute the bioavailable fractions of anthropogenic origin. The high percentage of these fractions indicates significant anthropogenic activities, leading to substantial metal pollution in the water column. The percentage of Zn and Ni were significantly found to be high in these fractions at sampling points A, F, and G. Meanwhile, Pb was detected high at sampling points F and G, such as in Figure 6. This fraction poses the most significant potential risk to humans and aquatic organisms due to its easy availability with minimal changes in the sediment's physicochemical conditions. The activities that contributed to

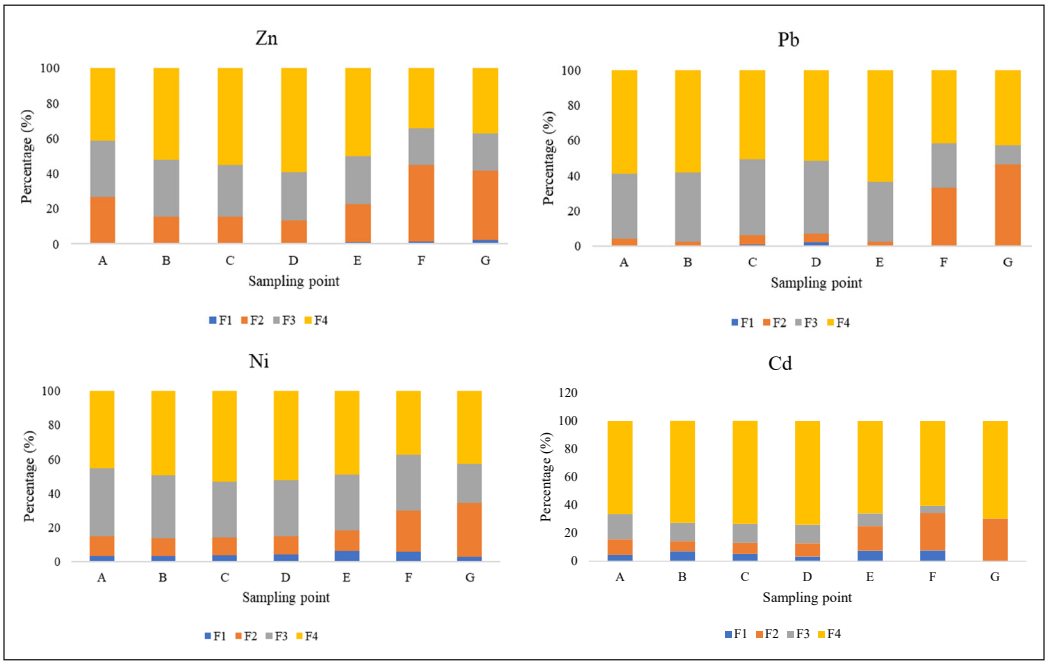

Figure 6. Sequential fractioning of heavy metals in surface sediment samples of Sungai Buloh

these pollutants in the Sungai Buloh area include industrialisation, agricultural activities involving the leaching of fertilisers and pesticides, manufacturing, workshops and other domestic activities.

Environmental Indices

This study uses selected indices such as I_{geo}, CF, and PLI to evaluate the extent of heavy metal contamination in the sediment. Table 6 indicates the I_{geo} of four heavy metals in sediment collected from seven sampling points. The range value of the I_{geo} index of Zn, Pb, Ni and Cd are from -0.23 to 0.45, -0.07 to 0.39, -2.98 to -2.19 and -2.74 to -1.51, respectively. Zn has the highest value of I_{geo}, which is found in sampling point G with the value of 0.45 (I_{geo} Class 1). Meanwhile, I_{geo} for Pb in each site except for sampling point F has values categories in Class 1, indicating that this element presents an ecological risk. In contrast, the I_{geo} values for Ni and Cd at all sites are below 0 (I_{geo} Class 0), indicating that these elements do not pose an ecological risk. Generally, all sampling points are categorised as unpolluted for each metal as the I_{geo} is lower than 1; however, Zn and Pb, which have I_{geo} values 0 to 1, indicate unpolluted to moderately contaminated in selected sampling points that have a chance of causing adverse effects towards the organisms.

Table 6
The I_{geo} index for the sediment from Sungai Buloh

Sampling Point	Zn	Pb	Ni	Cd
A	0.22	0.35	-2.63	-2.54
B	0.05	0.20	-2.77	-1.71
C	0.00	0.16	-2.83	-1.55
D	-0.07	0.37	-2.98	-1.61
E	0.00	0.30	-2.57	-1.51
F	-0.23	-0.07	-2.96	-2.74
G	0.45	0.39	-2.19	-2.03

The results obtained from the Contamination Factor (CF) followed a trend similar to that of I_{geo}. The CF for Zn, Pb, Ni, and Cd are within the ranges of 1.28 to 2.05, 1.43 to 1.97, 0.19 to 0.33, and 0.23 to 0.53, respectively. According to Table 7, Zn and Pb exhibit CF values between 1 and 3, indicating moderate contamination across sampling points A to G. Conversely, Ni and Cd, with CF values below 1 at all sampling points, are classified as low contamination. Sampling point G has the highest PLI value at 0.84, while sampling point F has the lowest at 0.53. PLI values indicated that all sampling points are unpolluted, as the PLI values are less than 1.

The Zn pollution in this area may originate from activities such as metal production, fossil fuel combustion, fertiliser use, and industrial, all of which can elevate Zn levels in

the environment. Pb is widely used as an additive in various applications, including lead pipes and paints, and contributes to pollution and gasoline leaks. Both Zn and Pb are found at moderate levels in the sediment of Sungai Buloh, resulting from a combination of natural occurrences and human activities. In contrast, Ni and Cd exhibit lower contamination levels, primarily due to natural occurrences leading to a low pollution status in Sungai Buloh. However, minimal human activities release Ni, typically used in chemical processing industries. Cd contamination, on the other hand, can be linked to mining and smelting activities, manufacturing processes, fertiliser application, and battery usage.

The findings from the study suggest that ongoing industrial, plantation, and domestic activities in the Sungai Buloh area, which result in the discharge of effluents containing heavy metals into water bodies, appear to be in compliance with the provisions outlined in Section 25 of the Environmental Quality Act 1974 (EQA 1974). This compliance reflects the effectiveness of the existing legal framework in controlling heavy metal pollution. However, the detection of moderate contamination underscores the necessity for continuous environmental monitoring to ensure that pollution levels remain within permissible limits and do not escalate over time.

Table 7
Contamination Factor (CF) and Pollution Load Index (PLI) of sediment from Sungai Buloh

Point	Zn	Pb	Ni	Cd	PLI
A	1.75	1.91	0.24	0.26	0.68
B	1.55	1.72	0.22	0.46	0.72
C	1.50	1.68	0.21	0.51	0.72
D	1.43	1.94	0.19	0.49	0.71
E	1.50	1.84	0.25	0.53	0.78
F	1.28	1.43	0.19	0.23	0.53
G	2.05	1.97	0.33	0.37	0.84

CONCLUSION

The distribution of selected heavy metals of Zn, Pb, Ni, and Cd in Sungai Buloh, Selangor, was assessed using sediment samples. The mean concentrations of these metals in the sediments, determined by aqua regia digestion, are as follows: Zn (130.406 ± 25.605 µg/g) > Pb (29.221 ± 7.811 µg/g) > Ni (13.083 ± 4.956 µg/g) > Cd (0.166 ± 0.074 µg/g). The sediment's fractionation of these heavy metals was evaluated using a sequential extraction technique. For Zn, Pb, and Ni, the distribution follows the trend: [residual] > [oxidisable-organic] > [acid-reducible] > [exchangeable]. For Cd, the trend is: [residual] > [acid-reducible] > [oxidisable-organic] > [exchangeable]. Environmental indices of the sediment, determined by the I_{geo} index, indicate that Kuala Sungai Buloh, Selangor, is unpolluted to

moderately contaminated at selected sampling points for Zn and Pb. Meanwhile, the CF shows moderate contamination for Zn and Pb and low contamination for Ni and Cd. The PLI indicates no significant heavy metal pollution in the area, as the PLI values are below 1. Manufacturing, industrial, plantation, and domestic activities, mainly natural occurrences, contribute to the presence and occurrence of these heavy metals in the rivers and estuaries of Sungai Buloh; however, they do not cause major pollution in the environment. Further research in Sungai Buloh is recommended, particularly focusing on cockles, as this area is commercially significant for their cultivation. This is essential to ensure the safety of both aquatic life and humans.

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