

SEASONAL VARIATION IN CONCENTRATION OF HEAVY METALS IN TROPICAL RIVER SEDIMENT

(Variasi Bermusim dalam Kepekatan Logam Berat di Dalam Sedimen Sungai Tropikal)

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Abstract

Heavy metal contamination has a more significant impact than microbial and organic contaminations as the elements could remain in the aqueous and particulate phases for an extended period. Setiu River, Terengganu, is one of the crucial rivers utilised as sites for aquaculture by the local communities. Generally, the concentrations of lead (Pb), zinc (Zn), copper (Cu), and arsenic (As) were influenced by human activities around river systems. Consequently, the present study investigated the seasonal concentration variations of Pb, Zn, Cu, and As in the surficial sediment of the river. The sediment samples were collected between January and July 2017, corresponding to the wet and dry seasons, from 60 different sampling locations along the river. The samples were subjected to Teflon bomb closed digestion procedures with mixed acid before the concentrations of the metals were determined with inductively coupled plasma mass spectrometry (ICP-MS). Resultantly, the mean concentrations of Pb, Zn, Cu, and As were 19.9 ± 9.89 , 68.4 ± 31.8 , 1.30 ± 0.66 , and 3.05 ± 0.53 $\mu\text{g/g}$ dry weight (wt.), respectively, in the samples gathered in the wet season (January). Alternatively, the samples collected during the dry season (July) contained 16.7 ± 15.1 , 68.9 ± 48.5 , 14.7 ± 16.9 , and 2.46 ± 2.78 $\mu\text{g/g}$ dry wt. mean concentrations of Pb, Zn, Cu, and As, respectively. The sample sediment quality was assessed based on the geoaccumulation index (I_{geo}). Based on the I_{geo} , the area was considered uncontaminated to moderately contaminated as the average values acquired were within Classes 0 and 2. Nevertheless, the pollution load index (PLI) revealed elevated concentrations of the metals, thus indicating anthropogenic sources, particularly in the aquaculture area. Accordingly, the Setiu river was assumed to be slightly polluted as the PLI values were 1.25 ± 0.34 (wet season) and 1.00 ± 1.00 (dry season).

Keywords: heavy metals, tropical river, pollution, index of geoaccumulation, pollution load index

Abstrak

Pencemaran logam berat mempunyai kesan yang lebih signifikan berbanding dengan pencemaran mikrob atau organik

kerana unsur-unsur ini dapat dikitar dalam fasa berair dan partikulat untuk jangka masa yang panjang. Sungai Setiu di Terengganu adalah salah satu sungai yang penting sebagai tempat akuakultur bagi masyarakat setempat. Secara amnya, kepekatan plumbum (Pb), zink (Zn), kuprum (Cu), dan arsenic (As) dipengaruhi oleh aktiviti manusia di sekitar system sungai. Oleh itu, kajian ini dilakukan untuk menyelidik variasi bermusim dalam kepekatan logam Pb, Zn, Cu dan As dalam sedimen permukaan sungai. Sampel sedimen dikumpulkan dari 60 lokasi persampelan yang berlainan di sepanjang sungai, di antara Januari dan Julai 2017, selaras dengan musim hujan dan musim kering. Setelah kaedah penghadaman tertutup bom Teflon dengan asid campuran, kepekatan logam ditentukan menggunakan spektrometri jisim plasma gandingan aruhan (ICPMS). Dari keputusan yang diperolehi, kepekatan purata Pb, Zn, Cu dan As adalah 19.9 ± 9.89 $\mu\text{g/g}$ berat kering, 68.4 ± 31.8 $\mu\text{g/g}$ berat kering, 1.30 ± 0.66 $\mu\text{g/g}$ berat kering dan 3.05 ± 0.53 $\mu\text{g/g}$ berat kering. untuk musim hujan (Januari), manakala, 16.7 ± 15.1 $\mu\text{g/g}$ berat kering; 68.9 ± 48.5 $\mu\text{g/g}$ berat kering, 14.7 ± 16.9 $\mu\text{g/g}$ berat kering dan 2.46 ± 2.78 $\mu\text{g/g}$ berat kering untuk musim kering (Julai). Penilaian kualiti sedimen menggunakan indeks geoakumulasi (I_{geo}) menunjukkan bahawa kawasan tersebut masih boleh dianggap tidak tercemar hingga sederhana tercemar kerana nilai purata I_{geo} dikategorikan dalam Kelas 0 hingga 2. Sementara itu, indeks beban pencemaran (PLI) mendedahkan peningkatan kepekatan logam yang dikaji, menunjukkan sumber antropogenik, terutama di kawasan akuakultur. Sungai Setiu boleh dianggap sedikit tercemar kerana nilai PLI lebih dari 1; 1.25 ± 0.34 (musim hujan); 1.00 ± 1.00 (musim kering). Secara amnya, kepekatan Pb, Zn, Cu dan As telah menunjukkan bahawa logam tersebut dipengaruhi oleh aktiviti manusia di sekitar sistem sungai tersebut.

Kata kunci: logam berat, sungai tropikam pencemaran, indeks geoakumulasi, indeks beban pencemaran

Introduction

Environmental contaminants from human and natural activities are the most severe problems confronting communities today. Industrial and economic growth and the production of various compounds and chemicals pose serious environmental and human health risks [1–3]. Moreover, heavy metal contaminants from human activities accumulate in the environment, most notably in river sediments [4–6]. Nonetheless, river sediments are complex and heterogeneous media. The roles of river sediments as a sink or source of contaminants has yet to be understood entirely, while numerous biogeochemical processes determine the fate of trace metals [7, 8].

The Setiu River is located in the Setiu district in the northern region of Terengganu, on the eastern coast of Peninsular Malaysia. The river possesses a catchment area of 188 km² and is approximately 52 km long. The northeast monsoon or the wet season strongly influences the river between November and March, while the southwest monsoon or the dry season occurs from May to September. Consequently, the river is vital to the surrounding communities, and it supplies freshwater to the Setiu Wetland as it flows southeast towards the wetland [9, 10]. Furthermore, water for irrigation and freshwater aquaculture is obtained from the Setiu River [11]. Intake points are also constructed along the riverbanks to extract raw water, providing water for domestic use [11].

Metal contaminants might have a more significant impact compared to organic and microbial contaminations, as metals could cycle between the aqueous and particulate phases over an extended time [12, 13]. Consequently, the current study aimed to determine the concentrations of heavy metals in the surficial sediment of the Setiu River system. The data would be crucial in highlighting the potential threat of the metals to humans and ecosystems. Furthermore, the present investigation aspired to evaluate the seasonal changes in the distribution of selected heavy metals and the Setiu River sediment pollution status.

Materials and Methods

Sample collection and sample analysis

For each season, wet (January) and dry (July), 60 bottom sediment samples were collected by employing Ponar Grab along the Setiu River (see Figure 1). Foreign objects that were not sediment were discarded in the field. The acquired samples were kept in plastic bags, preserved at low temperatures, and transported to the laboratory. Subsequently, the sediment samples were transferred to Petri dishes and oven-dried at 60 °C for several days to complete dryness. The dry samples were then ground into a fine powder with pestle and mortar and stored in polyethylene vials before subjecting the samples to the digestion process.

The heavy metals present in the samples were analysed by employing the Teflon bomb closed digestion method [14–16]. A total of 0.05 g of sediment samples were transferred to Teflon beakers. Subsequently, 1.5 mL of HNO_3 , HCl , and HF (3:3:1) was added to each beaker. Following that, the samples were digested in the oven

for eight hours at 100 °C. The digested samples were transferred into 10 mL test tubes, and deionised water was added until the volume was 10 mL. Finally, the lead (Pb), zinc (Zn), copper (Cu), and arsenic (As) contents were determined and identified with inductively coupled plasma mass spectrometry (ICP-MS) [14–16].



Figure 1. Sampling stations along Setiu River, Terengganu

Index of geoaccumulation (I_{geo})

The contamination of heavy metals in sediments was determined according to the geoaccumulation index (I_{geo}) [17, 18]. Moreover, the I_{geo} assessed the relationship between the geochemical background values of sediments and the measured metals

concentrations in the sediment samples. Table 1 lists the sediment contamination categories suggested by Haris et al. [19] and Williams and Antoine [20]. The I_{geo} values of the samples collected in the present investigation were calculated with Equation (1).

Table 1. Sediment contamination categories according to I_{geo} value

Class	I_{geo} value	Sediment Quality
0	$I_{geo} < 0$	Practically uncontaminated
1	$0 < I_{geo} < 1$	Uncontaminated to moderately contaminated
2	$1 < I_{geo} < 2$	Moderately contaminated
3	$2 < I_{geo} < 3$	Moderately to heavily contaminated
4	$3 < I_{geo} < 4$	Heavily contaminated
5	$4 < I_{geo} < 5$	Heavily to extremely contaminated
6	$I_{geo} > 5$	Extremely contaminated

$$\text{Index of geoaccumulation (Igeo)} = \text{Log}_2 \frac{C_n}{1.5B_n} \quad (\text{Eq. 1})$$

Where C_n is the concentration of the heavy metals in the sediment sample (n), B_n is the background value in the average shale of the metals (n), and 1.5 is the background matrix correction factor due to lithogenic effects.

Pollution Load Index

Pollution load index (PLI) value indicates the number of times the metal content in the sediment surpassed the mean background concentration, serving as an indicator of the overall level of heavy metal toxicity in a given sample. The PLI recommended by Tomlinson et al. [21] was employed to establish the mutual contamination status of the measured metals in the current study. The

PLI is calculated as a contamination factor (CF) for each metal in relation to the background value in the sediment sample [22, 23]. The CF value expresses the pollution by a single heavy metal in a sample (Table 2) [24]. The CF was defined by Equation 2, while Equation 3 was utilised to express the PLI. Table 3 demonstrates the contamination status according to the calculated PLI values [21].

Table 2. Contamination factor categories based on CF value

CF Value	Contamination Factor
$CF < 1$	Low contamination factor
$1 \leq CF \leq 3$	Moderate contamination factor
$3 \leq CF < 6$	Considerable contamination factor
$CF \geq 6$	Very high contamination factor

$$\text{Contamination Factor (CF)} = \frac{C_{\text{sample}}}{C_{\text{background}}} \quad (\text{Eq. 2})$$

Where C_{sample} is the metal concentration in the sediment sample and $C_{\text{background}}$ represents the background value of the metals.

$$\text{PLI} = (CF_1 \times CF_2 \times CF_3 \times \dots \times CF_n)^{1/n} \quad (\text{Eq. 3})$$

Where n is the number of metals examined.

Table 3. Contamination status based on PLI value

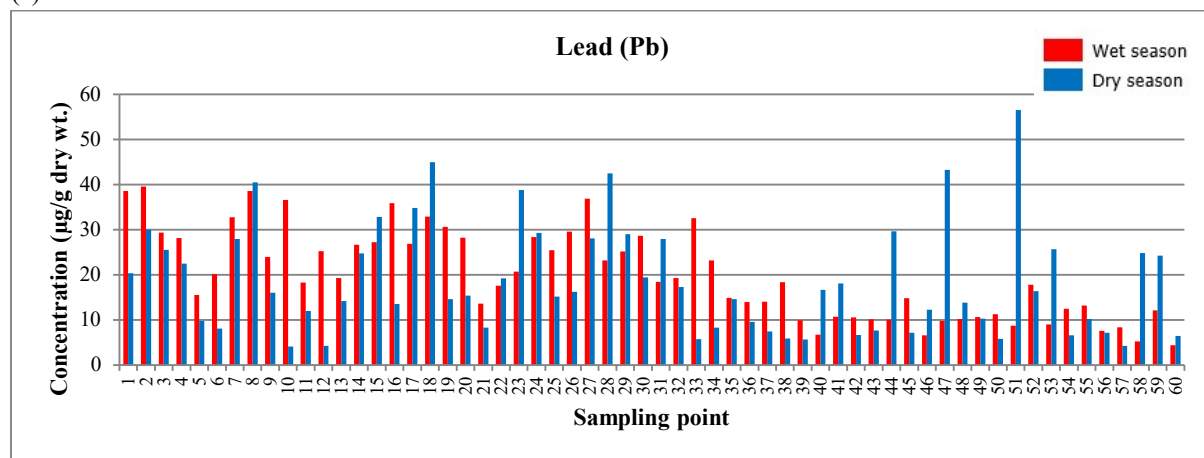
PLI Value	Contamination Status
$PLI < 1.0$	No heavy metal contamination occurs
$PLI > 1.0$	Heavy metal contamination occurs

Results and Discussion

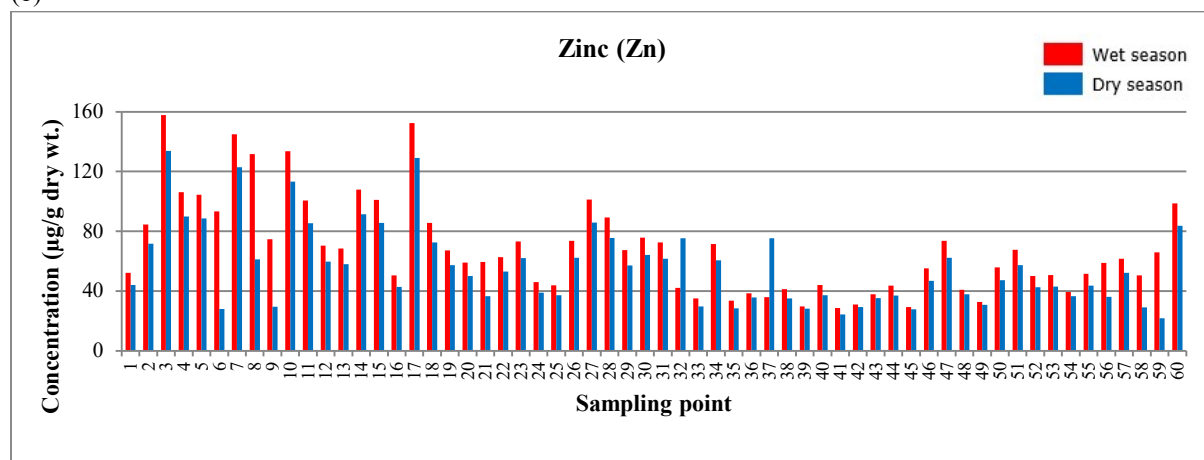
The distribution of Pb, Zn, Cu, and As concentrations in the Setiu River during the wet and dry seasons in 2017 are displayed in Figure 2. The concentrations of the studied metals were higher than the estimated average concentrations of shales and crust materials on Earth

[25]. Possible sources of contamination of the metals in the sampling area were likely anthropogenic activities, such as boating, aquaculture, agriculture, plantation, sand mining, and discharge of untreated waste into the river system.

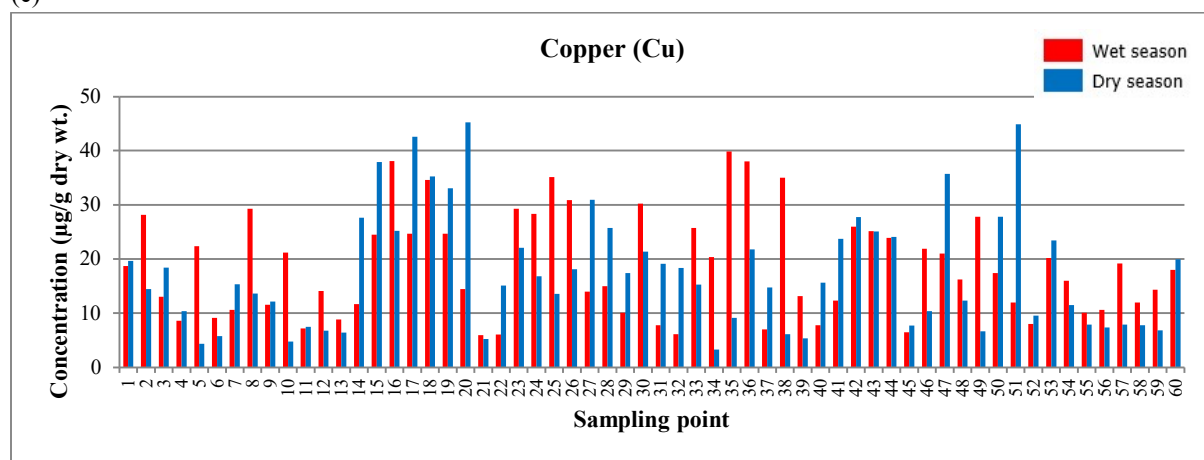
(a)



(b)



(c)



(d)

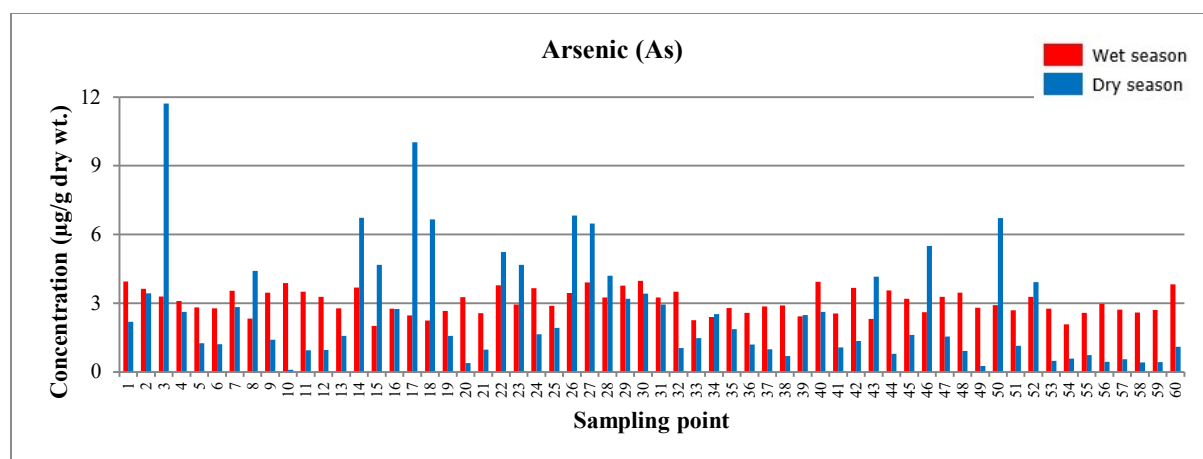


Figure 2. The distribution of (a) Pb, (b) Zn, (c) Cu, and (d) As concentration in the sediment samples from Setiu River during the wet and dry seasons

Lead has been known as poisonous since medieval times [26]. The sources of exposure to Pb vary from country to country, depending on historical and current uses [27]. Environmental Pb is from anthropogenic sources, such as lead-based paints, gasoline, and past and present mining and industrial activities [28]. Figure 2(a) displays the distribution of the concentration of Pb in the Setiu River sediment samples. The highest Pb concentration for the wet season was 39.5 µg/g dry wt., while the lowest was 4.38 µg/g dry wt. with an average of 19.9 ± 9.89 µg/g dry wt., which was considered to be slightly higher compared to the average background level of 17.0 µg/g dry wt.

The sediment with the lowest Pb concentration was obtained from station (St.) 60, while St. 2 contained the highest concentration of Pb. Nevertheless, during the dry season, the highest concentration of Pb concentration was 92.9 µg/g dry wt., recorded by the sample from St. 17, while the lowest concentration was in St. 60 at 1.58 µg/g dry wt. The average concentration for the dry season was 16.7 ± 15.1 µg/g dry wt., which was considered slightly lower than the average background level of 17.0 µg/g dry wt. [25].

Zinc is utilised in various consumer, agricultural, infrastructure, and industrial products [29]. Most importantly, Zn is vital to life as the substance is essential in the biological processes of living organisms,

including humans. The chemical is crucial to cell division, protein synthesis, the immune system, and growth [30, 31]. A small part of Zn in soils, rocks, and sediments is constantly moved and transported throughout the environment via natural erosion processes [32].

Figure 2(b) displays the distribution of Zn concentration in the Setiu River sediment samples acquired during the wet and dry seasons of 2017. The highest concentration of Zn for the wet season was 158 µg/g dry wt., while the lowest concentration was 28.6 µg/g dry wt. The average concentration of Zn was 68.4 ± 31.8 µg/g dry wt., which was higher than the average background level at 52.0 µg/g dry wt. The lowest concentration of Zn was from St. 41, while the sample from St. 3 exhibited the highest concentration of Zn. The highest concentration of Zn was 323 µg/g dry wt. from St. 17, while the lowest was from St. 40 at 12.4 µg/g dry wt. during the dry season. The average concentration for the dry season was 65.9 ± 48.5 µg/g dry wt., which was above the average background level of 52.0 µg/g dry wt. [25].

Copper is an essential micronutrient required for the growth of plants and animals [33, 34]. The substance is usually present in drinking water distributed in Cu pipes and additives designed to control algal growth [35]. The highest Cu concentration in the wet season was obtained from St. 35 at 39.9 µg/g dry wt., while the lowest

concentration value was recorded by the sample collected from St. 21 at $5.94 \mu\text{g/g}$ dry wt. The average value was $18.7 \pm 9.48 \mu\text{g/g}$ dry wt., which was higher than the average background level of $14.3 \mu\text{g/g}$ dry wt. During the dry season, the highest concentration of Co was exhibited by the sample from St. 17 at $109 \mu\text{g/g}$ dry wt., while the lowest concentration was $1.05 \mu\text{g/g}$ dry wt. from St. 56. The average concentration value for the dry season was $14.7 \pm 16.9 \mu\text{g/g}$ dry wt., which was slightly higher than the average background level of $14.3 \mu\text{g/g}$ dry wt. [25].

Arsenic naturally occurs in the Earth's crust and is found in water, air, and land [36]. The inorganic form of the metal is highly toxic to organisms [37]. The chemical is employed in industrial activities, including as an alloying agent and in textile, glass, paper, wood preservatives, and metal adhesives processing [38]. The highest As concentration for the wet season was $3.98 \mu\text{g/g}$ dry wt., while the lowest concentration was $2.01 \mu\text{g/g}$ dry wt. The average value of $3.05 \pm 0.53 \mu\text{g/g}$ dry wt. was considered higher than the background average of $2.0 \mu\text{g/g}$ dry wt. The lowest concentration of As was in St. 15, while the highest concentration of As was recorded by St. 30. During the dry season, the maximum As concentration was documented in St. 17 at $15.8 \mu\text{g/g}$ dry wt. and the minimum concentration was demonstrated by the sample from St. 52 with a value of $0.04 \mu\text{g/g}$ dry wt. The average concentration during the

season was 2.45 ± 2.78 dry wt. and appeared to be above the average background concentration of $2.0 \mu\text{g/g}$ dry wt. [25].

Anthropogenic activities might significantly affect metal contaminations in the sediment of the Setiu River. Based on Figure 1, the concentration of metals is notably well distributed among most of the sampling points during the wet season. The observation might be due to the higher rainfall during the season [39] (November to March), with November being the wettest month of the year. Nevertheless, during the dry season, the concentration of metals was higher, mainly at St. 16 and 17 in July. The conditions might be due to heavy aquaculture and boating activities in the region. Theoretically, the dry season starts in May and ends in September, with June being the driest month [40].

The bottom sediments are the most prominent heavy metals reservoirs in aquatic systems. The metals accumulated in the sediments threaten the entire aquatic ecosystem [41]. Heavy metal contamination was widely distributed in the Setiu River system. Table 4 lists the I_{geo} values of the sediment samples for two seasons, the wet and dry seasons of 2017. Resultantly, all studied metals were classified in Classes 0 to 2, indicating that the sediments were uncontaminated to moderately to heavily contaminated.

Table 4. The I_{geo} value based on the average concentration obtained from the sediment of Setiu river for wet and dry seasons

Metal	Min – Max I_{geo} Value		Average I_{geo} Value		I_{geo} Class	
	Wet Season	Dry Season	Wet Season	Dry Season	Wet Season	Dry Season
Pb	(-2.54) – 0.63	(-4.01) – 1.87	-0.55	-1.11	0–1	0–2
Zn	(-1.44) – 1.02	(-2.66) – 2.05	-0.33	-0.52	0–2	0–3
Cu	(-1.85) – 0.90	(-4.35) – 2.35	-0.40	-1.24	0–1	0–3
As	(-0.58) – 0.41	(-6.36) – 2.40	0.004	-1.26	0–1	0–3

* Class 0 = $I_{\text{geo}} < 0$ Practically uncontaminated; Class 1 = $0 < I_{\text{geo}} < 1$ Uncontaminated to moderately contaminated; Class 2 = $1 < I_{\text{geo}} < 2$ Moderately contaminated; Class 3 = $2 < I_{\text{geo}} < 3$ Moderately to heavily contaminated

The severity and variation of metal pollution at the 60 sampling points were assessed according to the PLI. Consequently, PLI was employed to determine the

contamination status of the measured metals in the present investigation. Resultantly, the CF values for As, Pb, Cu, and Zn in the wet and dry seasons in the studied

areas exceeded 1, as summarised in Table 5. The findings indicated a moderate CF, although the value for

Pb in the dry season was less than 1.

Table 5. The CF values of the metals in the sediment samples from Setiu River during the wet and dry seasons of 2017

Metal	Min – Max Value		CF Value		Contamination Factor	
	Wet Season	Dry Season	Wet Season	Dry Season	Wet Season	Dry Season
Pb	0.26–2.33	0.09–5.47	1.18	0.98	Moderate	Low
Zn	0.55–3.03	0.24–6.22	1.32	1.27	Moderate	Moderate
Cu	0.42–2.79	0.07–7.65	1.30	1.03	Moderate	Moderate
As	1.01–1.99	0.02–7.90	1.53	1.23	Moderate	Moderate

The PLI values obtained in the current study were more than 1, thus classifying Setiu River as slightly polluted due to heavy metals contamination (Table 6). Anthropogenic activities along the river contributed to the As, Pb, Cu, and Zn contamination. Fish loading and

offloading, cleaning, maintenance practices such as painting, antifouling applications, fuelling activities, and urban runoff could also be the sources of metal pollution. Moreover, the usage of leaded gasoline by fishers led to increased Pb concentration in the region.

Table 6. The PLI values obtained based on the concentration of metals in the sediment samples from Setiu River in the wet and dry seasons of 2017

Min – Max Value		PLI Value		Metal Contamination Status	
Wet Season	Dry Season	Wet Season	Dry Season	Wet Season	Dry Season
0.76–2.00	0.17–6.73	1.25±0.34	1.00±1.0	Polluted	Polluted

Conclusion

Generally, the concentrations of metals in Setiu River sediments were significantly influenced by anthropogenic activities in the surrounding areas. The present investigation proved that heavy metals contamination occurred in the Setiu river system based on the I_{geo} and PLI values. Furthermore, the levels of heavy metals in the samples obtained during the dry season (July) were higher than in the wet season (January). The observations might be due to the dilution of metals in the aquatic system through precipitation. Consequently, comprehensive studies that include other pollutants, such as hydrocarbon, persistent organic pollutants, and microplastics should be conducted to ensure good water quality for aquaculture purposes.

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