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Metal Characterization and Risk Assessment for Cu and Pb in Surface Sediments of Sungai Puloh Mangrove, Malaysia

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Abstract- The speciation profile of Cu, and Pb, as well as the total organic carbon (TOC) and pH were determined in 42 surface sediment samples from Sungai Puloh intertidal mangrove ecosystem The four stage sequential extraction technique (SET) (exchangeable, acid–reducible, oxidizable-organics, and residual fractions) was employed to investigate the heavy metals distribution pattern among phases. The concentrations of Cu and Pb in the samples were determined using an air-acetylene flame atomic absorption Spectrophotometer and presented in µg/g dry weight basis. The Risk Assessment Criteria Code (RAC) was applied to estimate the metals bioavailability and implication to food chain. The result revealed that the total mean metal concentrations for Cu and Pb were (52.70±45.16 and 77.18±38.46) µg/g, respectively. Chemical speciation of Cu (87.76% on average) existed in the residual fraction. The non-residual fraction of Pb (43.91%) showed Pb is at the borderline for remobilization. Based on the RAC, Cu poses no risk, while Pb poses low risk for the possibility to enter into the food chain. The relationship obtained for TOC, pH, and non-resistant fractions indicated that TOC has more significant effect on the distribution of elements among phases than pH. Further research is encouraged to clearly understand the effect of organic carbon in Pb remobilization and its resultant availability in the food chain of mangrove ecosystem.

***Index Terms:* Bioavailability, Metal Speciation, Risk Assessment, Sungai Puloh, TOC**

I. INTRODUCTION

Mangrove ecosystems are among the most productive coastal ecosystems in the world, providing essential ecological functions that connect terrestrial and marine environments. They support biodiversity, enhance carbon sequestration, and provide important ecosystem services to coastal communities [1], [2]. Mangroves also play a vital role in shoreline stabilization, sediment trapping, and coastal protection against erosion, storm surges, and sea-level rise [1], [3].

The intertidal mudflats of mangrove ecosystems support diverse aquatic and terrestrial organisms, including fish, crustaceans, molluscs, birds, and reptiles, many of which constitute important food resources and contribute to local livelihoods.

However, mangrove ecosystems are increasingly threatened by anthropogenic activities associated with rapid urbanization, industrialization, agriculture, aquaculture, and coastal development. These activities introduce a variety of contaminants into the environment, among which heavy metals are of particular concern due to their persistence, toxicity, and tendency to accumulate in sediments [2], [1]. Heavy metals released into aquatic systems may be absorbed by plankton, adsorbed onto suspended particles, and subsequently deposited in bottom sediments, which act as major sinks for metal contaminants [4], [1].

Although sediments can immobilize heavy metals, changes in environmental conditions such as pH, redox potential, salinity, and organic matter content may remobilize sediment-bound metals, thereby increasing their bioavailability and potential transfer through aquatic food webs [4], [5]. Consequently, understanding the chemical forms or speciation of metals in sediments is essential for assessing their mobility, bioavailability, ecological risk, and potential impacts on aquatic organisms and human health [5].

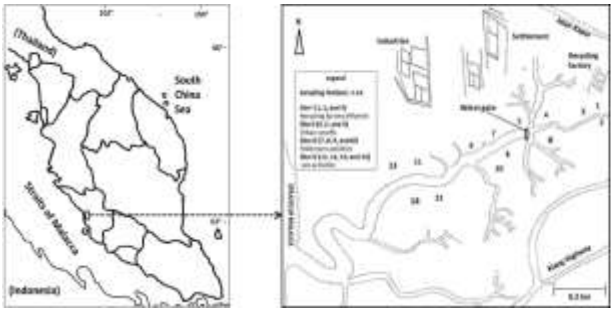
In this study, a modified sequential extraction technique (SET), based on the procedures of Tessier et al. [6], was applied to fractionate Cu and Pb into four operationally defined phases: exchangeable, acid-reducible, oxidizable-organic, and residual fractions. This approach was used to distinguish metals originating from anthropogenic sources from those associated with natural geological backgrounds and to evaluate their potential bioavailability under varying environmental conditions. The objectives of this study were to: (1) quantify the speciation of Cu and Pb in the intertidal sediments of the Sungai Puloh mangrove ecosystem; (2) identify the metal exhibiting a higher proportion of anthropogenic contribution; and (3) assess the potential bioavailability and ecological implications of these metals within the mangrove food web.

II. MATERIALS AND METHODS

Study area

The mangrove area of Sungai Puloh (SGP N 03° 04.786´, E 101° 23.903´) is located in the state of Selangor, west coast of Peninsular Malaysia. Sungai Puloh forms a small linked estuary with the Straits of Malacca. This mangrove area sustains a great diversity of macro benthic organisms. It stretches about 6.87km in length and is situated in approximately less than 5km off the major intercity road of Jalan Kapar.

There are several small and medium size industries (SMIs) operating in vicinity of the area. The industries involve various types such as scrap metal yards, recycling sectors (these factories handle a lot of scrap metals, polyethylene containers and various non- degradable materials), automobile workshops, oil palm mills, and aquaculture/agricultural activities. Most of their wastes are drained in this mangrove ecosystem. Also a handful of residential houses were observed in this area, while, fishing and cattle rearing are the main domestic activity close to the Puloh River.



Fig, I Sampling location for intertidal surface sediment of Sungai Puloh mangrove in west coast of peninsular Malaysia

Sampling

Surface sediment samples were collected in from 14 sampling stations along the intertidal mangrove area of Puloh River (Fig I). Three replicate samples of surface sediment from each station were collected using a clean plastic scoop. The upper 2cm of each sample was placed in a polyethylene bag with a stainless-steel spatula.

All the samples were kept in an iced chest, taken to the laboratory and frozen until further analysis. Surface sediment samples were dried by using air-circulating oven to a constant dry weight at 80°C. Later the samples were ground using a clean dry pestle and ceramic mortar and sieved through a 63µm stainless steel sieve and stored in acid –washed container for metal speciation.

Aqua Regia

The direct aqua regia method as described by [7] was employed for the pseudo-total heavy metal content analysis. About 1 g of each dried sample was weighed and digested in a mixture of concentrated nitric acid (HNO_3 AnalaR grade, BDH 69%) and perchloric acid (AnalaR grade 60%) in a ratio of 4:1. The samples were then placed in the digesting block, and the temperature was first set at a low temperature of 40 °C for 1 h and later increased to 140 °C for at least 3 h [7]– [9].

After digestion, the samples were diluted to a 40 mL volume with double-distilled water and filtered using Whatman No. 1 filter paper into acid-washed polyethylene sample bottles and stored for metal determination.

Sequential Extraction

Metal speciation was analyzed using the Sequential Extraction Technique (SET) (Table 1) as described by [10] and [11].

Stage 1: Easily Freely Leachable and Exchangeable (EFLE). About 10 g of sediment sample was placed into a clean Erlenmeyer flask, followed by the addition of 50 mL of 1.0 M ammonium acetate (NH_4CH_3COO, pH 7.0), and agitated continuously using an orbital shaker (Model GYROMAX 722) at a constant speed of 2,500 rpm for 3 h at room temperature. This first fraction liberates metals loosely bound to carbonates.

Stage 2: Acid-Reducible Fraction. The residue from Fraction 1 was dried, weighed, and transferred into a clean Erlenmeyer flask. Fifty millilitres of 0.2 M hydroxylammonium chloride ($NH_2OH{\cdot}HCl$), acidified to pH 2.0 with hydrochloric acid, was added, and the mixture was agitated under the same conditions as in Stage 1. This fraction releases metals associated with Fe-Mn oxides and hydroxides. Iron and manganese oxides bind trace metals because of their high scavenging affinity for heavy metals [12].

Stage 3: Oxidizable-Organic Fraction. The residue from Stage 2 was dried, weighed, and oxidized with 15 mL of H_2O_2 (35%, R&M Chemicals) in a water bath at 90–95 °C until dry. After cooling, 50 mL of 1.0 M ammonium acetate (NH_4CH_3COO), acidified to pH 2.0 with HCl, was added, and the mixture was continuously shaken for 3 h. This step liberates metals bound to organic complexes. Hydrogen peroxide acts as a powerful and versatile oxidizing agent capable of decomposing organic compounds and releasing metals associated with organic matter.

Stage 4: Residual Fraction. The residue from Stage 3 was dried, weighed, and digested in concentrated nitric acid (HNO_3 AnalaR grade, BDH 69%) and perchloric acid ($HClO_4$ AnalaR grade, 60%) in a ratio of 4:1 following the direct aqua regia procedure. This fraction is neither readily leachable, acid-reducible, nor oxidizable. The metals are mainly embedded within the crystal lattice of the sediment matrix and are therefore not expected to be remobilized except under extremely harsh environmental conditions [13]. Consequently, they are considered to be predominantly of natural geological origin.

Table 1 Extrancts used in each extraction stages and various phases of sediment in the sequential extraction scheme

Extraction Stages	Extrctrants	Sediment target phase
Fraction 1	1.0 M NH_4CH_3COO, pH 7.0	Exchangeable
Fraction 2	0.2 M $NH_2OH.HCl$, pH 2.0	Acid- reducible
Fraction 3	H_2O_2 (35%), 1.0M NH_4CH_3COO, pH 2.0	Oxidizable - organics
Fraction 4	HNO_3 (69%), HClO4 (60%)	Residual

Metal Determination

The residue of each extraction stage was rinsed with 20 ml double distilled water (DDW). It is then filtered through Whatman No.1 filter paper into acid washed pill boxes and stored for metal determination (except for fraction 1 that was analyzed immediately because of its high sensitivity).

Samples were determined for heavy metal concentrations by using an air-acetylene flame atomic absorption Spectrophotometer (Analyst 800 model, by Perkin-Elmer), and the data are presented in µg/g dry weight basis.

Sediment pH

About 10 g of dried sediment sample was placed in a glass beaker. Then, 25 mL of distilled water was added, and the beaker was covered with plastic film. The mixture was shaken using an orbital shaker for 4 h at 175 rpm. Thereafter, the pH was measured using a digital electrode pH meter (WTW pH 330) [14].

Total Organic Carbon (TOC) in Sediment

About 1 g of dried sediment sample was thoroughly mixed with 1–2 mL of 1 M HCl to remove inorganic carbon and dried for approximately 10 h at 100–105 °C to eliminate traces of HCl. The TOC (%) was determined using a LECO CR-412 carbon analyzer [15].

Quality Control
To avoid contamination, all glassware was soaked in 10% HCl for at least 24 h, rinsed thoroughly with double-distilled water, and air-dried before use. To ensure the precision and accuracy of the analytical procedure, calibration curves were prepared using multi-level calibration standards.

Standard solutions for each metal were prepared from 1,000 mg/L BDH SpectrosoL® stock solutions. Certified Reference Material (CRM) (International Atomic Energy Agency, Soil-5, Vienna, Austria) was used to validate the analytical procedure. The analytical results obtained for the reference material showed satisfactory recoveries of approximately 97% and 98% for Cu and Pb, respectively (Table 2).

Furthermore, an internal quality assurance assessment was performed by comparing the sum of metals extracted in the sequential extraction procedure with the pseudo-total metal concentrations obtained by direct aqua regia digestion. The recovery percentages were satisfactory (Table 3).

The recovery of the sequential extraction was calculated thus:

Recovery (%) = [(Fraction 1+ Fraction 2 + Fraction 3 + Fraction 4) / Total digestion] X 100

Statistical analysis
The SPSS 20 software was used for all the statistical analyses and the Microsoft EXCEL for windows was employed for the graphs.

Table 2 A comparison of measured values and certified values (µg/g) of CRM for soil, and the percentage recovery (n=3).

Metal	Concentration (µg/g)		Recovery (%)
	Certified values	Measured values	
Cu	77.0 ± 4.70	75.0 ± 3.59	97
Pb	129 ± 26.60	126.9 ± 7.03	98

Table 3 Mean comparison of the sum of sequential extraction with the direct aqua regia digestion based on 42 different sediment samples taken from Sungai Puloh intertidal mangrove ecosystem (n=3)

Metal	Sum of extraction fractions (µg/g)	Direct aqua regia (µg/g)	Recovery (%)
Cu	52.70 ±45.16	46.89 ±43.79	112.39
Pb	77.18 ± 38.46	78.84±49.60	97.89

III. RESULTS AND DISCUSSION

Metal Speciation patterns
The spatial concentration distributions of Cu and Zn (in percent fractions) are summarized Fig 2a and b. Among these metals, the residual fraction of Cu (82.31%) was found to be higher, than that of residual fraction of Pb (56.09%) as shown in Table 4.

The speciation profile also revealed Pb (37.38%) is more dominant in oxidizable organics fraction compared to Cu (Table 4). This suggests that the rate of bioavailability and remobilization is much higher for Pb than Cu. Therefore, the rate of chances of remobilization for these heavy metals in this intertidal mangrove ecosystem is Pb > Cu.

Copper Speciation
The total Cu concentration ranged from 23.27 to 139.05 µg/g. The exchangeable fraction for Cu ranged from 0.12 to 0.40 µg/g with a mean percentage of 0.67%. The acid-reducible fraction ranged from 0.07 to 0.18 µg/g with a mean percentage of 0.43%.

The oxidizable-organic fraction ranged from 1.47 to 53.73 µg/g with a mean percentage of 16.60%, whereas the residual fraction ranged from 15.94 to 85.85 µg/g with a mean percentage of 82.31%.

These results indicate that Cu speciation in the surface sediments of Sungai Puloh is dominated by the residual fraction (Table 4). This suggests that Cu in the study area is predominantly derived from lithogenic sources, including the weathering of igneous and metamorphic rocks as well as the decomposition of biota and detritus [16].

Consequently, a substantial proportion of Cu is unlikely to be bioavailable because the residual fraction represents metals embedded within the crystal lattice of sediment minerals and therefore cannot be readily leached, reduced, or oxidized except under extremely harsh environmental conditions [17].

The mathematical sum of the exchangeable, acid-reducible, and oxidizable-organic fractions constitutes the non-resistant (non-lithogenic) fraction [16].

Among these fractions, the exchangeable, acid-reducible, and oxidizable-organic phases contributed average values of 0.67%, 0.43%, and 16.60%, respectively. However, relatively high proportions of Cu occurred in the oxidizable-organic fraction at Stations 4 (42.22%), 5 (38.35%), and 6 (38.26%).

The elevated Cu concentrations in these stations may be associated with inputs of organic matter from domestic wastes [18], runoff from oil palm mills, cattle dung, aquaculture activities, and untreated wastes from small-scale industries [19]. Overall, Cu speciation in Sungai Puloh followed the order:

Residual > Oxidizable-Organic > Exchangeable > Acid-Reducible.

Lead Speciation

The total Pb concentration ranged from 40.86 to 146.60 µg/g. The exchangeable fraction ranged from 0.90 to 3.20 µg/g (mean = 2.84%), the acid-reducible fraction from 1.19 to 6.23 µg/g (mean = 3.60%), the oxidizable-organic fraction from 10.68 to 45.67 µg/g (mean = 37.39%), and the residual fraction from 17.79 to 93.32 µg/g (mean = 56.09%).

The fractionation profile indicates that less than 60% of Pb was associated with the residual fraction, whereas approximately 37% occurred in the oxidizable-organic fraction. This suggests that approximately 56% of Pb is relatively immobile, whereas about 40% may become remobilized under changing environmental conditions.

If oxidizing conditions prevail, oxidation of organic complexes and sulfides may release approximately 37% of the total Pb. The relatively low percentages of exchangeable and acid-reducible fractions (2–4%) indicate that changes in pH and redox potential are unlikely to cause substantial remobilization and bioavailability of Pb [20]. The Pb speciation followed the order:

Residual > Oxidizable-Organic > Acid-Reducible > Exchangeable.

Previous studies have reported that Pb contamination in Malaysian coastal sediments is largely associated with anthropogenic activities [21]. In the present study, anthropogenic Pb inputs are likely derived from illegal refuse dumping, boating activities, nearby recycling industries, vehicular emissions, coal-fired power plants, and atmospheric deposition [19].

Role of TOC and pH on Metal Partitioning and Bioavailability

The total organic carbon (TOC) and pH of the Sungai Puloh surface sediments ranged from 2.31% to 5.97% and 3.49 to 6.22, respectively. Metal partitioning between the solid and aqueous phases of sediments is strongly influenced by physicochemical factors such as pH, organic matter content, ionic strength, iron and manganese oxides, redox potential, and the nature of sorption surfaces [22].

Among these variables, pH, cation exchange capacity (CEC), clay content, and organic matter are considered the dominant factors controlling heavy metal mobility and bioavailability. At lower pH, hydrogen ions compete effectively with metal ions for negatively charged adsorption sites, thereby increasing metal mobility. Consequently, dissolved metal concentrations generally increase with decreasing pH, reaching maximum availability around pH 4.0 [23].

Table 4 Mean percentages of four chemical speciation fractions for Cu and Pb in Sg. Puloh intertidal sediment

Metals	%EFLE	%Acid reducible	%Oxidizable organics	%Resistant
Cu	0.67	0.43	16.6	82.31
Pb	2.84	3.68	37.39	56.09

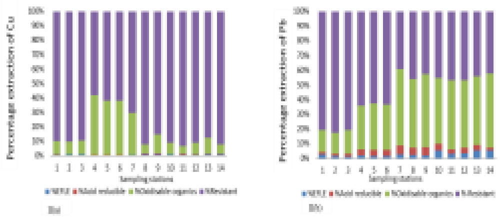


Fig 2 (a) and (b) Percentage extractions of Cu and Pb respectively in intertidal surface sediments of Sungai Puloh

High organic matter, particularly total organic carbon (TOC), promotes the immobilization of metals through binding with fulvic and humic acids. Some elements, such as Pb, do not readily form chloride complexes because of their strong affinity for organic matter [24].

Organic matter in sediments consists of carbon and nutrients in the form of carbohydrates, proteins, fats, and nucleic acids. Most sedimentary organic matter originates from plant and animal detritus, bacteria, plankton formed in situ, and both natural and anthropogenic sources. Sewage and effluents from processing plants, pulp mills, and fish farms are examples of anthropogenic organic wastes.

The discharge or release of contaminants into the environment increases the total carbon content in soils and sediments. Organic matter possesses several important characteristics, including the ability to form both water-soluble and insoluble complexes with metal ions and hydrous oxides, interact with clay minerals, bind sediment particles together, and adsorb or desorb naturally occurring and anthropogenic organic compounds [25].

Total organic carbon (TOC), often regarded as a measure of the amount of organic matter preserved within sediments, is an essential parameter in site characterization because it strongly influences the behaviour and fate of contaminants in sediments [25].

The relationships among metal phase distributions, TOC, and pH are presented in Table 5. In the exchangeable fraction, the phase distributions of Cu ($r = 0.625$, $p < 0.05$) and Pb ($r = 0.656$, $p < 0.05$) showed strong positive correlations with TOC.

Similarly, in the acid-reducible fraction, both Cu and Pb exhibited strong positive correlations with TOC, indicating that their distributions in this fraction are influenced by organic carbon content. In the oxidizable-organic fraction, Cu ($r = 0.587$, $p < 0.05$) and Pb ($r = 0.930$, $p < 0.01$) also showed significant positive correlations with TOC.

These findings suggest that TOC plays a major role in controlling the distribution of Cu and Pb in the oxidizable-organic fraction. This observation agrees with the findings of [26], who reported strong positive correlations between TOC and the concentrations of Pb, Cd, and Cu in Fraction C.

The relationships between pH and the phase distributions of all the investigated metals revealed no significant correlations in any of the extraction fractions. This suggests that TOC exerts a greater influence than pH on the distribution of metals among sediment phases. These findings are also consistent with those of [26], who concluded that pH had no obvious effect on the distribution of trace metals in natural sediment samples.

Table 5 Correlation of non-resistant fractions of metal speciation with TOC and pH in Sg. Puloh surface sediment

		Cu	Pb	TOC %	pH
Exchangeable fraction					
	Cu	1			
	Pb	0.775^{a}	1		

	TOC	0.625[b]	0.656[b]	1	
	pH	0.249	0.266	-253	1
Acid-reducible fraction					
	Cu	1			
	Pb	0.689[a]	1		
	TOC	0.839[a]	0.574[b]	1	
	pH	-0.302	-0.235	-0.253	
Oxidizable-organics					
	Cu	1			
	Pb	0.767[a]	1		
	TOC	0.587[b]	0.930[a]	1	
	pH	0.095	-0.204	-0.253	1

[a]correlation is significant at the 0.01 level; [b] correlation is significant at the 0.05 level

Risk Assessment

The Risk Assessment Code (RAC), as presented in Table 6, indicates that surface sediments releasing less than 1% of the total metal content in the exchangeable fraction (Fraction 1) are considered safe for the environment. In contrast, sediments releasing more than 50% of the total metal content in the same fraction are regarded as posing an extremely high environmental risk because the metals can be readily transferred through aquatic food chains [27].

The metal speciation results revealed that the percentage of metals in the exchangeable fraction ranged from 0.22–1.02% for Cu and 1.31–5.31% for Pb. The RAC was applied to evaluate which metals may pose potential ecological risks within the Sungai Puloh mangrove ecosystem.

Copper exhibited a very low proportion in the exchangeable fraction, with an average value of 0.67%. According to the RAC classification, Cu falls within the no-risk category, indicating that it is unlikely to be readily leached from the sediment or become bioavailable to aquatic organisms.

Lead exhibited an average exchangeable fraction of 2.84%, placing it within the low-risk category. This suggests that Pb has only a limited potential for remobilization and subsequent transfer into the aquatic food chain [27].

Table 6 Risk assessment

Risk assessment Code (RAC)	Criteria (%)
No risk	<1
Low risk	1-10
Medium risk	11-30
High risk	31- 50
Very high risk	>50
Metal	Exchangeable fraction (%)
Cu	0.22 – 1.02
Pb	1.31 – 5.31

In comparison with previous studies (Table 7), the Cu concentrations obtained in the present study are comparable to those reported by [33] and [39], but are higher than those reported by [28], [29], [30] [34]. In contrast, Pb concentrations were higher than those previously reported for Peninsular Malaysia, Hong Kong, and Singapore by [39], [31] and [35], respectively.

Table 7 Comparison of Cu, and, Pb (µg/g) in Sungai Puloh surface sediments with those from mangrove and coastal areas around the world.

Location	Cu	Pb	References
West coast of Peninsular Malaysia	<6.00	1.00-45.00	[28]
Bintulu coastal waters	7.00-13.00	11.00-36.00	[29]
Sebarang Perai, Malaysia	9.99-63.44	22.19-45.32	[30]
Hong Kong	2.60	31.20	[31]
Port Jackson Australia	102	443	[32]
Intertidal west coast of Peninsular Malaysia	0.40-315.00	0.96-69.81	[33]
East China Sea	19.24± 0.89	25.54± 0.41	[34]
Mangrove area of Singapore	7.06	12.3-30.9	[35]

Guanabara Bay, Brazil	2-18,840	2-193.4	[37]
Kaoshiung Habour, Taiwan	5-946	9.5-470	[38]
Dumai coast Indonesia	1.61-13.84	14.63-84.90	[36]
Akyatan Lagoon-Turkey	15.4-31.3	19.5-33.6	[41]
Peninsular Malaysia	1.63-150.81	7.97-93.11	[39]
Northern part of Peninsular Malaysia	4.79-32.91	15.85-61.56	[40]
Red Sea, Egypt	6.8-190.2	5.0-56	[42]
Shantou Bay, China	48.52	51.63	[43]
Sungai Puloh, Malaysia	23.27-139.05	40.86-146.6	Present study

However, when the present study is compared with reports from other regions of the world, the Cu concentrations are lower than those reported for Guanabara Bay, Brazil [37], Kaohsiung Harbour, Taiwan [38], and the Red Sea, Egypt [42]. Comparison of Pb concentrations with those reported for the Dumai coast, Indonesia [36] indicates that Pb concentrations in the present study are higher.

Conversely, the Pb concentration observed in the present study is lower than that reported for Port Jackson, Australia [32]. Overall, the concentrations of the investigated metals are comparable to those reported for Shantou Bay, China [43].

CONCLUSION

In the light of the findings copper was found to be more dominant than Pb in the residual fraction in this study area. Based on the speciation profile, the probability of bioavailability and remobilization of these metals in Sg. Puloh mangrove surface sediments follow the following pattern: Pb > Cu.

This study further revealed that the effect of TOC on the distribution and partitioning of heavy metal is more pronounced than that of sediment pH. Risk assessment showed that there is no possibility for Cu to enter into the food chain, while there is low possibility for Pb to enter into the food chain of Sg. Puloh mangrove ecosystem. Therefore, for the fact that Pb (which has no known beneficial value in biota) is implicated, all anthropogenic sources within the vicinity of the mangrove area should be intensely monitored to ensure Pb pose no risk to the food chain in this productive mangrove ecosystem.

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