

TIME DEPOSITION OF COPPER, MANGANESE AND NICKEL AT PERAI RIVER, PENANG

Yii Mei-Wo, Wilfred Paulus and Zal U'yun Wan Mahmood

Malaysian Nuclear Agency,
43000 Kajang, Malaysia

Correspondence author: yii@nuclearmalaysia.gov.my

ABSTRACT

Perai Industrial Area is one of the densest industrial and heavily populated zones in Malaysia. Concentration levels of copper, manganese, and nickel were investigated in the Perai River in this area. Sediment core was collected within the river, about 4-5 km away from the estuary, one each during the rainy and dry period using a KC™ Kajak corer. Concentrations of elements were performed using the Energy Dispersive X-ray Fluorescence Spectrometer (EDXRF); meanwhile, the sediment deposition rates which were determined through the Lead-210 dating technique were taken from previous the publication. Using continental crust values and iron metal concentration as normalization values, enrichment factors for elements were calculated. The results showed copper showing moderate enrichment in sediment during the dry period in the study area. Meanwhile, no enrichment for manganese and nickel was found in that area. Estimated sedimentation rates suggested the contaminants were deposited quickly and happened during the last two to four years prior to this study.

INTRODUCTION

Environmental issues in nature are difficult and complex processes to understand. Interactions occur both within the biotic and the abiotic environment and between themselves (Humbird, 2008). Nearly 40% of the world's population live within 100 km of the coastal areas, where people getting benefit from the use of coastal and marine resources and create employment that is linked with coastal and maritime activities, as well as from the coastal recreational opportunities (Burke et al., 2001; United Nation, 2017). Development of the coastal zone, exploitation of its natural resources, and its use as a receptacle for societal wastes promote sediment contamination and endanger living marine resources (Cochran and Masque, 2004). Remediation of the marine environment is not a standalone process but must take into consideration the ecological interdependence of the oceans, coastal areas, and the freshwater systems associated with them (Jejeff, 1999).

Pollutants released from terrestrial will be transported to rivers and eventually ended up in the marine ecosystem. Inorganic pollutants of environmental concern, such as elementals, that are released in the river water potential be accumulated in the aquatic food chain and sediments (Islam et al., 2017; Li et al., 2017), and promote adverse effects on the aquatic life (Elias et al., 2012; Yi et al., 2011) and also to the human being consuming them. The major sources of inorganic pollution originated from anthropogenic sources such as agriculture activities, animal waste, domestic, industrial, mining and petroleum activities, as well as industrial emissions (Pandey & Singh, 2017; Shazili et al., 2006; Zhu et al., 2011).

Sediment is a product created from the combination of all complex materials contributed by rivers, coastal erosion, biological and chemical processes, as well as anthropogenic waste. Usually, weathering is one of the major sources of sediments; however, settlement development and rapid economic growth can also drive the release of waste materials into the coastal area and accumulate into the sediment (Joseph et al., 2018). The deposition rate of contaminants in sediment can be estimated by means and one approach is by using the natural radionuclide profiles such as of ^{210}Pb ($T_{1/2} = 22.20 \pm 0.22$ years) in a sediment core, where the sedimentation rates can be estimated by employing different mathematical models (Krishnaswami & Lal, 1978; Sanchez-Cabeza & Ruiz-Fernández, 2012).

Energy-dispersive X-ray fluorescence (EDXRF) spectrometry is a non-destructive analytical nuclear technique used to obtain simultaneous elemental information from different types of materials. It is employed in a wide range of industries and applications such as in cement production, cosmetics, food and environmental, forensics, glass production, healthcare products, iron, mining and mineral beneficiation, petroleum and petrochemicals, pharmaceuticals, polymers and related industries, steel, and non-ferrous metals. The basic concept of the spectrometer contains a radiation source, a sample, and a detection system. In EDXRF spectrometers, the X-ray tube acts as a source that directly irradiates a sample, and the fluorescence coming from the sample is measured with an energy dispersive detector. This detector can measure the different energies of the characteristic radiation coming out from the sample. The detector can separate the radiation from the sample into the radiation from the different elements present in the sample. This separation phenomenon is known as dispersion.

Perai Industrial Estate (Fig. 1) is one of the major industrial parks in the Penang state of Malaysia and is adjacent to the Juru River located at the east of the area and the Perai River to the northwest. Perai Industrial Area is densely populated and comprises multiple heavy industries. Associate population growth in the surroundings of the Perai industrial area, industrial and household discharges, terrestrial materials, agriculture, and sewage emission (Shazili et al., 2006) were suspected to be released into the nearby waters including into the Perai River. Zainudin (2010) reported that the Juru River is one of the most polluted rivers in the country. Pollutants history in the Juru River had been widely studied (Buhari & Ismail, 2016; Idriss and Ahmad, 2013; Lim and Kiu, 1995; Wood et al., 1993; Shazili et al., 2006) before, but a limited study was conducted on the Perai River. So, a study on the Perai River will provide useful information about the metal pollution history in that area.

This study is aimed to estimate the enrichment of elements copper (Cu), manganese (Mn) and nickel (Ni) and their deposition timeframe in the sediment cores collected during two different seasons from the Perai River, Penang that might have been promoted by the economic development and population growth in the Perai Industrial area.

MATERIALS AND METHODS

Sample collection and preparation

By using a KCTM Kajak Core Sampler completed with the 8 cm diameter PVC tube, one sediment core was collected each during the rainy and dry seasons between 2017 and 2018 at the Perai River, which is located about 4 - 5 km away from the estuary (Fig. 1 and Table 1). Each core was extruded from the tube bottom and sliced into 2 cm thick sections at the sampling site and samples were placed

into HDPE plastic bag and refrigerated until further analysis. Back in the laboratory, sediments were weighted and small portions of samples were taken for particle analysis, re-weighted, and dried in an oven at 80°C until constant weight (IAEA, 2003). The dried samples were finely ground using a Rocklabs® grinder mill to obtain homogeneous powders of less than 200 mesh (74 µm) size. All data in this study was reported on a dry weight basis.

Table 1. Sampling location details

Date	Station	Coordinate	Core length (cm)
Nov 2017(rainy)	SP 06, Perai River	5.3992°N	56
July 2018 (dry)	SP 09, Perai River	100.3927°E	62

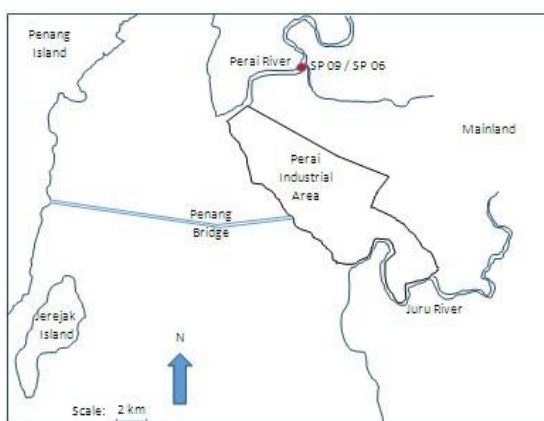


Figure 1. Sampling location

Particle size analysis

Particle size distribution in the wet sediment sample was analysed with a Microtrac™ X100 laser diffraction (Fig. 2) in the laboratory. Using the Udden–Wentworth scale, particles were characterized into a fraction of clay (< 4 µm), silt (4–63 µm), and sand (> 63 µm) (Blair and McPherson, 1999; Blott and Pye, 2012; Krumbein and Aberdeen, 1937).



Figure 2. Particle Size Analyzer

Loss on Ignition

A rough estimation of organic matter content (% OM) in soil/sediment samples was based on the Loss on Ignition (LOI) analysis (Fig. 3a). This method does not involve the use of any chemicals but

is only performed using a muffle furnace (Fig. 3b). LOI calculates the % OM by comparing the weight of a sample before and after the soil/sediment has been ignited. Organic matter is oxidized to carbon dioxide and ash at temperatures between ~200 and 500 °C. Weight losses associated with water and carbon dioxide evolutions are easily quantified using equations (1) and (2) below by recording sample weights before and after controlled heating (ignition at 550 and 1000 °C) and, in turn, maybe correlated to water content, organic matter, and carbonate content (Nelson and Sommers, 1982). Using two equations below, percentage of organic carbons and carbonate were calculated for each section of the sediment core.

$$\% \text{ Organic Matter} = \frac{(\text{Weight Post } 105^{\circ}\text{C Dry sample}) - (\text{Weight Post } 550^{\circ}\text{C Ash})}{(\text{Weight Post } 105^{\circ}\text{C Dry sample})} \times 100 \quad (1)$$

$$\% \text{ CaCO}_3 = \frac{(\text{Weight Post } 550^{\circ}\text{C Ash}) - (\text{Weight Post } 1000^{\circ}\text{C Ash})}{(\text{Weight Post } 105^{\circ}\text{C Dry sample})} \times 2.274 \times 100 \quad (2)$$

where $2.274 = 100.088 / 44.009 = \text{molecular weight CaCO}_3 / \text{molecular weight of CO}_2$.



Figure 3a. LOI (from left to right) at 105 °C, 550 °C and 1000 °C)



Figure 3b. Furnace for LOI

Trace elements analysis

Elemental analysis was performed using Shimadzu™ EDX-7000 EDXRF (Fig. 4), by applying an in-house loose powder method. Approximately 1.0 – 2.0 g fine ground sample aliquots were placed into a small Teflon ring cap cup, covered with a 6 µm polypropylene Mylar® to be analysed using a Rhodium x-ray tube. The reference material SRM 2709a was used as quality control material and calibration of the method. The results of samples were accepted when the reference material achieved the 95% confidence level as stated in the material certificate.

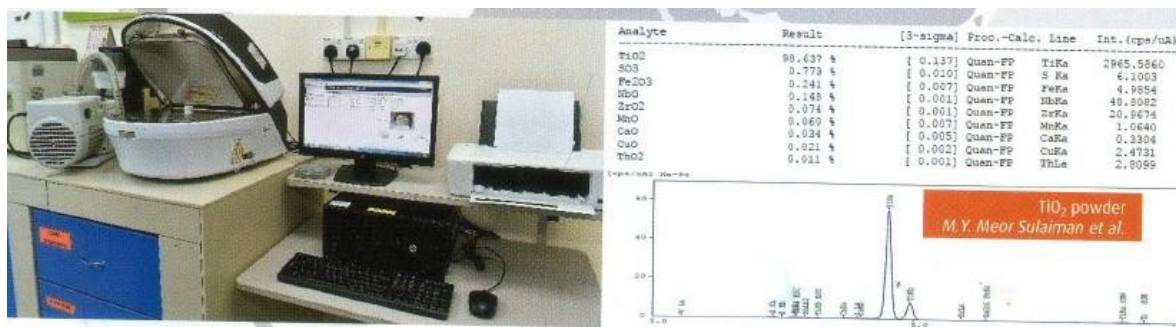


Figure 4. Energy Dispersive X-ray Fluorescence Spectrometer

Enrichment factor (EF)

In order to evaluate potential contamination by trace elements, the enrichment factor (EF) was calculated based on the Eq. (3) below (Elias et al., 2018):

$$EF = \frac{(M/R)_{measure}}{(M/R)_{CC}} \quad (3)$$

where $(M/R)_{measure}$ is the concentration ratio of the element of interest (M) to the reference element (R) where $(M/R)_{measure}$ is the concentration ratio of the element of interest (M) to a reference element (R) measured in the sample, and $(M/R)_{CC}$ is the concentration ratio in the continental crust (Wedepohl, 1995). Iron (Fe) was used as reference element to normalize the trace element concentrations to compensate for variations in particle size and mineralogy (Loring & Rantala, 1992). Deely & Fergusson (1994) reported that this is due to iron distribution not being related to other elements. Iron usually has a relatively high natural concentration (Abraham & Parker, 2008), and therefore this element is not expected to be substantially enriched from anthropogenic sources in estuarine sediment (Niencheski et al., 1994). If the element found in the sediment originated predominantly from the lithogenous materials, the EF value will be less than 2.0, whereas EFs are much greater than 2.0 indicating that the element is of the anthropogenic origin (Szefer et al., 1996). EF values were classified according to Ayari et al. (2016) as shown in Table 2.

Table 2. The indication status of Enrichment Factor (Ayari et al., 2016)

Enrichment factor (EF) value	Enrichment status
< 2	no enrichment
between 2 to < 3	minor enrichment
between 3 to < 5	moderate enrichment
between 5 to < 10	moderately severe enrichment
between 10 to < 25	severe enrichment
between 25 to < 50	very severe enrichment
more than 50	extremely severe enrichment

Statistical Analysis of data

MicrosoftTM Office Excel 2013 analysis tools, the Pearson's correlation function and Regression data will be used to analyse data in this study to express the correlation relationship among variables. Pearson's analysis was used to assess the strength and direction of the linear relationships between

pairs of variables while the Regression's analysis could determine the significance of the relationships. Correlation between variables would be expressed as significant correlated ($P < 0.05$ or $P < 0.10$) if their relationships are found to be significance. Non-quantitative terminology such as weak, moderate or strong correlations will not be used to discuss the correlations in this article.

RESULTS AND DISCUSSION

Particle size analysis

The sediments in all cores were mostly composed of fine-grained particles (Fig. 5). In most core sections, the percentage of silt was higher than the clay (almost double), and the clay was higher than that of sand. Overall, the silt contents were around 35–75 %, the clay contents between 15–65 % whilst the sand contents were around 0–35 % in both the cores. Clay contents showed decreasing pattern from the top towards the bottom of the core of SP 09 which could be due to the re-suspension of the fine particle when the river water flows through the sampling area. Meanwhile, the silt content showed a slight increase from the upper section of the core towards the bottom for core SP 09. On the other hand, the sand contents were low at the top sections of the core SP 09 and became higher from 10 cm onwards. The movement of sand might be related to erosion due to nearby river activities and also the volume and speed of water flow in the river. On the other hand, the particulate profile for SP 06 was unique where the clay and silt contents in the core were almost constant from the top towards the bottom which might indicate mixing of the core due to massive water flow and turbulence during the rainy season.

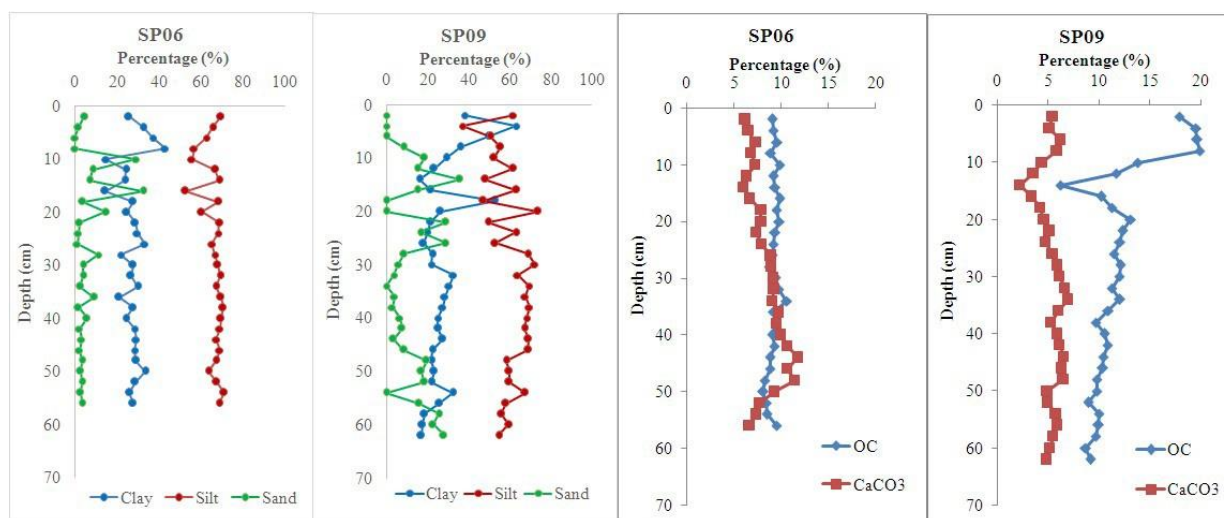


Figure 5. Particle size distribution (left) and Loss on Ignition (right) in sediment cores at study area

Loss on Ignition

Typically, sedimentary organic matter contains between 40 and 60% of organic carbon. The difference in weight between the 550 and 1000°C ashes may (to a first approximation) be assumed to result from the loss of carbon dioxide during carbonate mineral break-down. However, loss on ignition techniques cannot indicate which carbonate minerals may be present within any given sample. As calcium carbonate is (typically) the dominant form of carbonate in most sediments, weight

losses at 1000°C may nonetheless be used to estimate calcium carbonate content. It should be noted that clay minerals may contain significant quantities of lattice-bound hydroxide (as much as five percent by weight) and these ions may be liberated (as water) at high temperatures. Calcium carbonate content estimates from weight losses at 1000 °C may therefore contain errors as great as five percent and (as such) data results are most appropriately reported as losses by weight percent (Weight % LOI, 1000 °C) (Nelson and Sommers, 1982).

Overall, looking at the two cores, the study found that the percentages of organic carbon decreased gradually from the surface sediment towards the bottom of the core of SP 09 but the organic carbon contents in the core of SP 06 were almost constant whilst carbonate contents were almost constant in both cores (Fig. 5). The organic carbon contents in the sediment core of SP 06 and SP 09 at the study site were between 8.1–10.6% and 6.1–19.9%, respectively. The organic contents for the cores in this study were comparable with the finding reported by Wan-Mahmood et al. (2016) of between 4.1–13.6 % at the Linggi River. Significant correlations ($p < 0.05$) were observed between the organic matter and the clay content in sediment cores collected during the dry seasons (SP 09, $r = 0.72$), whilst cores collected during the rainy seasons (SP 06, $r = 0.28$) shows no significant correlation. Water volume in the river and weathering during the two different seasons could be the factors behind these differences.

Metal profiles

Table 3 below summarized the concentration of the elements Cu, Fe, Mn, and Ni in the study area. Generally, concentrations for all elements (except iron) were below 100 µg/g for both cores except at the top portions of core SP 09 which has Cu concentration between 100 µg/g–200 µg/g. For element copper (Cu), higher concentrations were observed in the top portions of the sediment core suggesting the input of this element was coming from the surrounding. For core SP 06, the Cu concentrations were less than 20 µg/g throughout the core and were below 10 µg/g from 6 cm onwards. On the other hand, even though high concentrations of more than 100 µg/g were observed on the top 8 cm of core SP 09, the Cu concentrations were decreased sharply to below 10 µg/g from 14 cm onwards. Single-factor ANOVA analysis of Cu revealed that there was a significant difference between these two cores.

Looking at the element manganese (Mn), the concentrations appeared in two-digit numbers (Table 3) in both cores and were almost constant throughout the core with concentrations ranging between 20–65 µg/g. Single-factor ANOVA analysis for Mn revealed that there were no significant differences between these two cores. Lastly, for element nickel (Ni), concentrations found in core SP 06 generally were much lower when compared to core SP 09. In core SP 06, the concentrations were approximately 10 µg/g or less but the concentrations in core SP 09 were 3–5 times higher especially from 50 cm onwards. Single-factor ANOVA analysis of Ni revealed that there was a significant difference between these two cores.

Table 3. Concentrations and Enrichment Factor (EFs) ranges of potentially toxic elements (µg/g) in sediment cores from Perai River, Malaysia

Element	CC ¹	Location	Concentration Range	EF range ²
Cu	25	SP 06	0–17.8 (4.0)	0.0–0.8 (0.2)
		SP 09	1.7–176 (25.3)	0.1– 6.6 (1.0)
Fe	43200	SP 06	31450–62660 (45440)	-
		SP 09	26910–49570 (43080)	-

Mn	716	SP 06	19.0–54.0 (40.1)	0.0–0.1 (0.1)
		SP 09	24.1–63.4 (41.3)	0.0–0.1 (0.1)
Ni	56	SP 06	0.0–26.1 (7.7)	0.0–0.5 (0.1)
		SP 09	5.6–102 (27.9)	0.1–1.6 (0.5)

¹CC = Continental crust values as published by Wedepohl in 1995.

²EF value in bold indicating of enrichment

Note: Values given in the parenthesis are the average values

When comparing the elemental concentrations found in this study with sediment studies elsewhere, such as the study conducted by Cheevaporn et al. (1994) (7–15 µg/g for Cu and 10–16 µg/g for Ni) at Bang Pakong River, Thailand; Kristina et al. (2014) (54–57 µg/g for Cu) at Butuanon River, Metro Cebu, Philippines; Elias et al. (2018) (1.5–66.2 µg/g for Cu and 1.8–29.7 µg/g for Ni) at Linggi River in Malaysia; Lim et al. (2013) 2.2–14.8 µg/g for Cu and 2.3–8.3 µg/g for Ni at Langat River in Malaysia, Idriss and Ahmad (2013) (5.4–16.8 µg/g for Ni) at Juru River; Abdus- Salam et al. (2019) (6.6–59.99 µg/g for Cu) at Perak River, Malaysia, and sediment cores from the Straits of Malacca by Saion et al. (2007) (Mean Cu, 17.5 µg/g; Mean Mn, 421.3 µg/g and mean Ni, 25.3 µg/g), all previous results showing that the values found in this study for Cu and Ni were higher than other areas while Mn values in this study were lower than the others. On the other hand, Ismail et al. (1993) reported that the background values for sediments on the west coast of Peninsular Malaysia, where 70% of sediments content Cu below 5 µg/g and 50 % of sediments content Mn between 3000 to 5000 µg/g.

Correlations between elemental with organic matter and particle size were calculated and summarized in Table 4. Only copper was found significantly correlated ($p < 0.05$) with organic matter content ($r = 0.92$ in SP 09) and clay content ($r = 0.67$ in SP 09) of the dry core, while manganese demonstrated significant correlation ($p < 0.05$) with the silt content ($r = 0.44$ in SP 06; $r = 0.40$ in SP 09) regardless of season effect. Meanwhile, other elements showed no significant correlations with the organic matter or particle size fractions. This could probably be due to the low concentrations of elements found in most sections of the sediment core.

Table 4. Summary of correlation analysis of trace element concentrations with the percentage of organic matter and particle size fractions in sediments from Perai River, Malaysia

Station	Element	Correlation coefficient (r)			
		Organic	Clay	Silt	Sand
SP 06 (rainy)	Cu	0.03	0.35	-0.07	-0.22
	Fe	-0.43	0.05	0.40	-0.27
	Mn	-0.34	0.03	0.44	-0.28
	Ni	0.31	-0.30	-0.58	0.57
SP 09 (dry)	Cu	0.92	0.67	-0.46	-0.31
	Fe	0.10	0.13	0.24	-0.33
	Mn	-0.59	-0.42	0.40	0.10
	Ni	-0.46	-0.31	0.05	0.27

Note: r values in Bold are significant at 95%, “-“ value indicates negative correlation.

The enrichment factors obtained in this study (Table 3 and Fig. 6) indicated that only copper was found moderately enriched in the dry season sediment core. At station SP 09, copper varied from moderate enrichment at the top portion of the core down to no enrichment at the lower core after 10 cm depth. This profile is very similar to the Zn enrichment profile found in the same core as reported

earlier in Yii et al. (*in press*). Meanwhile, copper concentrations in core SP 06 were also not enriched. On the other hand, no enrichment was observed by manganese and nickel in both sediment cores. A high concentration of copper found at the top portions of the core of SP 09 could be released as a result of natural weathering of soil, discharges from industries and sewage treatment plants, and also could be coming from intentionally applied activities into the water to kill algae (ATSDR, 2018).

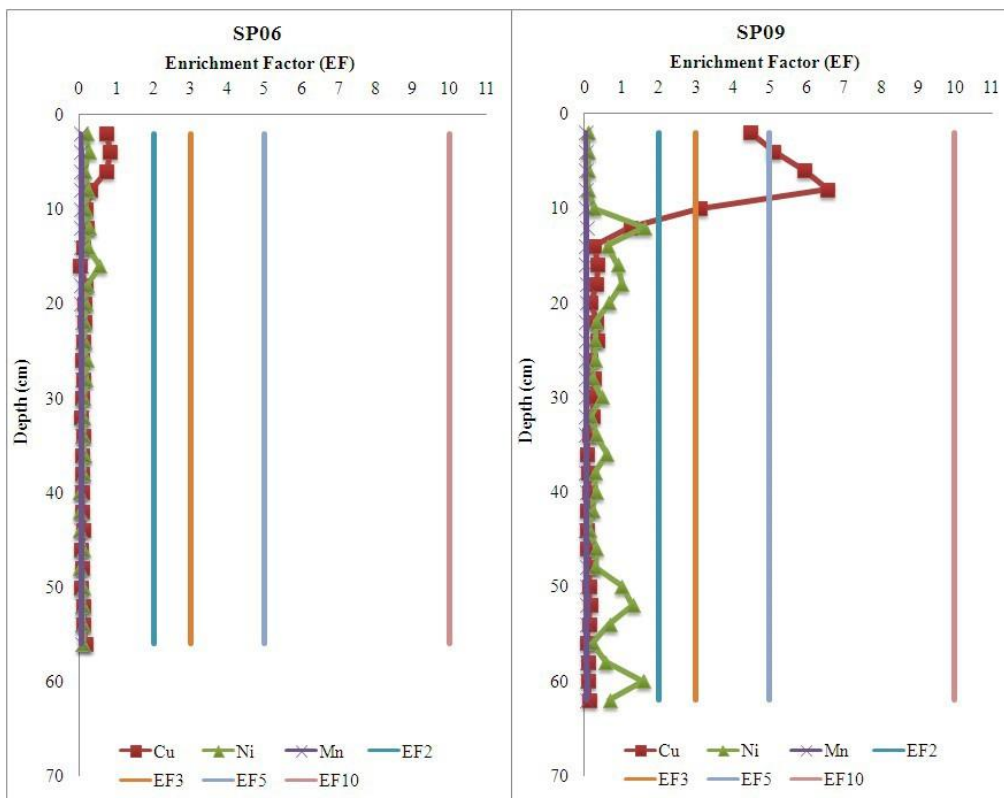


Figure 6. Elemental Enrichment Factor versus depth for SP 06 and SP 09.

Sediment accumulation rate

The mean sediment accumulation rates for these two cores were reported earlier to be 4.83 ± 0.86 cm/y in SP 06 and 1.79 ± 0.49 cm/y in SP 09 even though the proper age model could not be defined (Yii & Wan-Mahmood, 2020). Calculation efforts were made to approximate a preliminary mean sedimentation rate for each core and done by using the constant rate-constant sedimentation (CFCS) model (Krishnaswamy et al., 1971), which assumes a constant $^{210}\text{Pb}_{\text{ex}}$ flux to the sediment surface and a sediment accumulation rate (Sanchez-Cabeza & Ruiz-Fernández, 2012). Thus, from the logarithm of excess ^{210}Pb ($\text{Ln } ^{210}\text{Pb}_{\text{ex}}$) data plotted against the depth of sediment core; and the slope of the regression between both variables, the mean sediment accumulation rate was estimated. Based on the sediment accumulation rate value reported for SP 06 and SP 09 from Yii & Wan-Mahmood (2020), and assuming that the study area has alternate rainy and dry seasons in a year, the average sediment accumulation rates will be approximately 3.31 ± 0.99 cm/y and this accumulation rate suggesting that the copper contaminant was introduced into the sediment during the last 2–4 years.

CONCLUSIONS

The enrichment factors showed that the core sediments collected from the Perai River having moderately enriched copper at the top 10 cm portions in the dry season sediment core while no enrichment was found for copper in the rainy season core. Also, no enrichment was found for manganese and nickel in the study area during both seasons. The sediment accumulation rates with an average deposition rate of 3.31 ± 0.99 cm/y suggest that the copper was introduced into the sediment during the last 2–4 years. This result can provide vital information for future comparison and also will be useful to relevant authorities in preparing proactive actions to control future release into the rivers. In order to have a better understanding about the elemental deposition history at the study area, for future analysis, thin slice of sediment (0.5 cm – 1.0 cm instead of 2 cm that being practiced now) can be performed to get a better understanding of sediment accumulation rate. Various analytical techniques can be applied as a comparison as well.

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