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Distribution of Ni and Zn in the Surface Sediments Collected from Drainages and Intertidal Areas in Selangor

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ABSTRACT

Surface sediments were collected from 11 sampling sites in selected intertidals and drainages of Selangor. The sediment samples were analysed for Ni and Zn. The metal concentrations ranged from 15.1 to 121 µg/g dry weight for Ni and 50.2 to 336 µg/g dry weight for Zn. The highest total (Ni and Zn) concentrations in sediments were found at an industrial site in Serdang. The Ni and Zn ranges resulting from this study were wider and higher than those reported previously in Malaysia. Generally, the 'oxidisable-organic' fraction contributed the largest percentage of metals among the other three anthropogenic-related fractions. This study shows that the non-resistant fraction dominated the total Zn based on sequential extraction technique. Some sites had higher percentage (>50%) of non-resistant fraction of Ni and Zn, indicating anthropogenic sources of these metals. Therefore, it is suggested that continuous monitoring of the study areas be implemented especially at industrial areas in Serdang. Perhaps, the industrial waste must be treated before draining to the waterways.

Keywords: Heavy metal, surface sediments, drainages, Selangor, Malaysia

INTRODUCTION

Many anthropogenic activities such as shipping, industry, agriculture and urbanization are based on the west coast of Peninsular Malaysia (Abdullah *et al.*, 1999) and mainly concentrated in the state of Selangor. Industrialization in Selangor has prompted the economic development as well as population expansion. From an ecotoxicological point of view, this is very interesting to know if those industrial activities impacted our natural resources in the coastal area. This study focused on 2 essential elements which are Ni and Zn (Boyle and Robinson, 1988; Astorga España *et al.*, 2007). Though essential, excessive occurrence of these two metals will cause toxicity to organisms in the environment. The toxic responses to Ni and Zn involve interference with Fe metabolism in the organism which causes anemic effects (Magee and Matrone, 1960; Stokes, 1988). Therefore, any risk assessment of the potential effects of Ni and Zn on organisms must take into account local environmental conditions. Previously, Yap *et al.* (2002a, 2002b, 2003b) has reported the concentrations of Cd, Cu, Pb and Zn in sediments collected from the offshore and intertidal area of the west coast of Peninsular Malaysia. However, studies on the Ni and Zn levels in the area of Selangor is still lacking in the literature. In order to estimate such a possible environmental problem, the background concentrations of heavy metals in the sediment samples collected from the aquatic ecosystems should be known. Therefore, studies monitoring heavy metal pollution are very significant and important.

The use of sediments is advantageous to assess human impacts on the aquatic environment. This is because, sediments play a major role in the transport and storage of metals and are also frequently used to identify sources of pollutants spatially and temporally and to locate the main sinks for heavy metals and the heavy metals that are persistent in the marine environment (Yap *et al.*, 2002b). Takarina (2004)

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† Abbreviations: dw=dry weight; *et al.*= and all; Jln = Jalan; Peng= Pengkalan; Sg= Sungai; Tjg= Tanjung; Tmn= Taman; DDW= Double Deionized Water.

reported that analysis of the speciation of the various heavy metals allowed for identification of potential pollution sources that would have otherwise been missed if only total metal content was known.

Since there is no current information in the concentrations and speciation of Ni and Zn in the surface sediments of Selangor, the objective of this study was to provide such information which mainly focused on the surface sediment samples collected from 11 sampling sites in Selangor including intertidal areas and drainages to which metal industrial effluents are deposited.

MATERIALS AND METHODS

Sampling of surface sediments was conducted in 6 intertidal sites and 5 urban drainages or rivers, in Selangor. The top 3 to 5 cm of surface sediments were collected from each site on 25th April 2005 (*Fig. 1*). The longitude, latitude and site descriptions for each sampling sites are given in Table 1. Each sediment sample was placed in an acid-washed polyethylene bag and deep frozen prior to analysis and brought back to the laboratory (Yap *et al.*, 2002a).

In the laboratory, the surface sediment samples were dried at 60ºC for at least 16 hrs until a constant dry weight. Then the samples were sieved through a 63μm stainless steel sieve and shaken vigorously to produce homogeneity.

Total Metal Concentration

Direct aqua-regia method was used for the analyses of total Ni and Zn concentrations in sediment samples (Yap *et al.*, 2002a). About one gram of each dried sample was weighed and digested in a combination of concentrated nitric acid (AnalaR grade, BDH 69%) and perchloric acid (AnalaR grade, BDH 60%) in the ratio of 4:1, first at low temperature (40 ºC) for 1 h and then the temperature was increased to 140ºC for at least 3 h. Double distilled water (DDW) was used to dilute the digested samples to 40 ml and the samples then filtered through Whatman No.1 filter paper and the filtrate stored until metal determination (Yap *et al.*, 2002b).

Speciations of Ni and Zn of Sediments Samples

Geochemical fractions of Ni and Zn in the sediments were obtained by using the modified sequential extraction technique (Badri and Aston, 1983; Yap *et al.*, 2002a). The four fractions considered, the extraction procedures and the conditions employed were:-

i. Easy, freely, leachable or exchangeable (EFLE): About 10 g of sample was continuously shaken for 3 hrs with 50 ml 1.0 M ammonium acetate (NH_4CH_3COO), pH 7.0 at room temperature.
ii. 'Acid-reducible': The residue was continuously shaken for 3 hrs with 50 ml

TABLE 1
Longitude, latitude and descriptions of sampling sites for surface sediment samples in some locations of Selangor

No	Locations	Longitude	Latitude	Site description
1.	JP Metal, Serdang	05º20.072' N	100º26.080' E	Drainage at the industrial area
2.	Subang Utama Industry	03º02.665' N	101º32.512' E	An industrial area
3.	Jln Renggam Urban	03º03.683' N	101º31.173' E	A riverside near the Fire Station
4.	Tmn Rashna Urban	03º03.684' N	101º30.347' E	A river beside a residential area
5.	Sultan Suleiman Urban	03º01.151' N	101º22.421' E	A drainage beside a residential area
6.	Peng Nelayan Intertidal abundant mangroves.	03º01.120' N	101º22.453' E	A jetty with fishing activities and
7.	Tjg Harapan Intertidal around.	03º005.96' N	101º21.637' E	A rocky beach with shipping activities
8.	Sg Kapar Intertidal	03º00.141' N	101º21.823' E	A riverside near a main highway
9.	Sg Janggut Intertidal	03º08.161' N	101º22.511' E	A small river near the dam and agriculture area
10.	Pantai Jeram Intertidal Bakar' Stalls nearby.	03º10.403' N	101º18.819' E	An estuary with fishing activities, 'Ikan
11.	Sungai Buloh Intertidal	03º15.467' N	101º18.245' E	A fishing village with '*dried prawn*' industry area

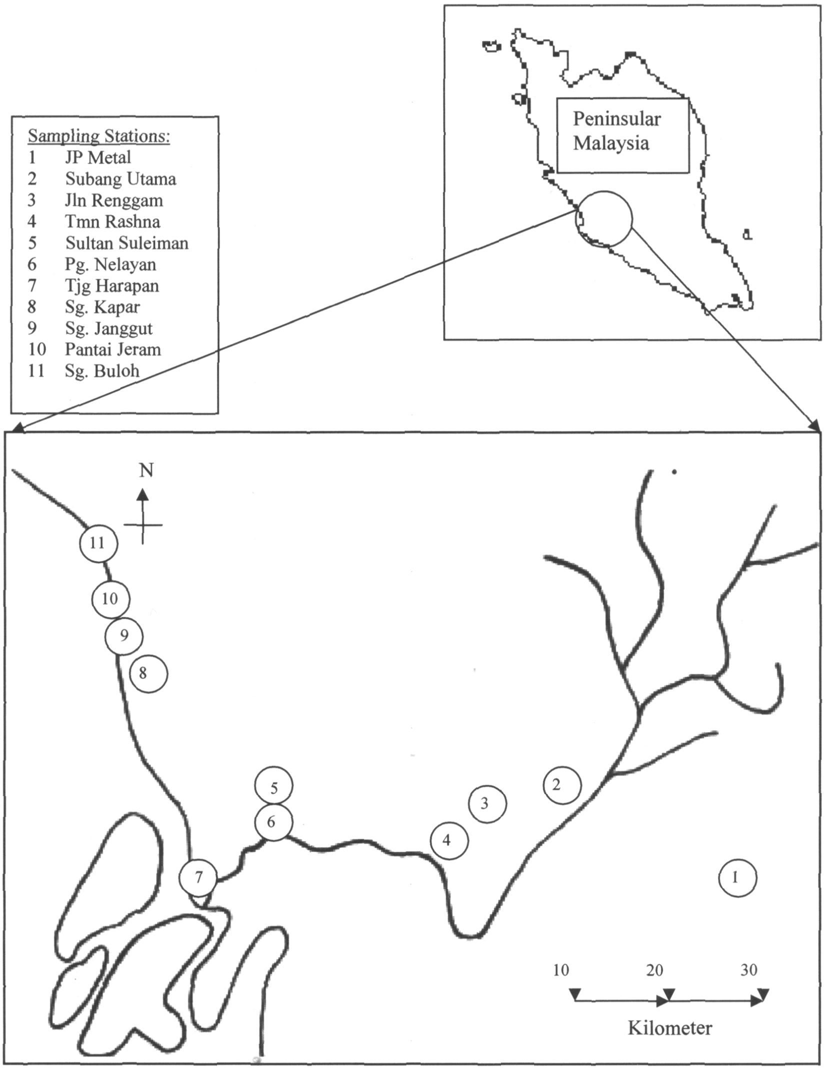

Fig. 1: Map showing sampling sites of surface sediment samples from some places in Selangor

0.25 M hydroxylammonium chloride ($NH_2OH.HCL$) acidified to pH 2 with HCl, at room temperature.

iii. 'Oxidisable-organic': The residue was first oxidized with 30% H_2O_2 in a water bath at 90-95°C. After cooling, the metal released from the organic complexes was continuously shaken for 3 h with 1.0 M ammonium acetate (NH_4CH_3COO) acidified to pH 2.0 with HCL, at room temperature.

iv. 'Resistant': The residue from (iii) was digested in a combination of concentrated nitric acid (69%) and perchloric acid (60%) as in the direct aqua-regia method.

The residue used for each fraction was weighed before the next fractionation was carried out. The residue was washed with 20 mL DDW. It was then filtered through Whatman No. 1 filter paper and the filtrate was stored until metal determination. For each fraction of the sequential extraction procedure, a blank was employed using the same procedure to ensure that the samples were free of contaminants.

Analysis of Ni, and Zn

The prepared samples were determined for Ni and Zn by using an atomic absorption spectrophotometer (AAS) Perkin Elmer Model

A Analyst 800 and the data were presented in µg/g of sample dry weight (dw).

Quality control samples of known concentrations made from standard solutions for each metal were routinely run through during the period of metal analysis. To avoid possible contamination, all glassware and equipment used were acid-washed. The metal percentages of recoveries were between 90-110%. The quality of the method used was checked with a Certified Reference Material (CRM) for Soil (International Atomic Energy Agency, Soil-5, Vienna, Austria). The agreement between the analytical results for the reference material and its certified values for each metal was satisfactory with recoveries of Zn: 87.8% and Ni: 124.6% as shown in Table 2.

In order to check the accuracy of this method, the sum of all extraction steps for each metal was compared with that found by using the direct digestion with the aqua-regia method. Our method was acceptable since satisfactory recoveries (90-105%) for Ni and Zn were found in the analytical results by using the SET when compared to those of the direct aqua-regia method and they correlated significantly ($P < 0.05$) with each other.

Data Analysis

The data recorded from the analysis of heavy metal were statistically analyzed using the Statistical Analysis System (SAS) for Windows, version 6.12. Microsoft Excel was used for Spearman' correlation analysis to ascertain the strength of the correlation coefficients among the samples. The analyzed data obtained were depicted as graphs using Kaleida Graphs, version 3.08, November 1996.

RESULTS AND DISCUSSION

Total Metal Concentrations

Based on the 11 sampling sites in Selangor (*Fig. 2*), the total metal concentrations based on direct aqua-regia method ranged from 15.1 to 121 µg/g dw for Ni and 50.2 to 336 µg/g dw for Zn. From *Fig. 2*, JP Metal site in Serdang was found to have the highest concentrations of Ni and Zn. On the other hand, Tanjung Harapan recorded the lowest concentrations of both the metals.

Both metal concentrations in the sediments obtained in this study were higher when compared to values from other regional and Malaysian studies (Table 3). This indicated

TABLE 2
A comparison of the measured results (µg/g dry weight) of the CRM for soil with its certified concentrations for Ni and Zn

Metal	Certified value (C)	Measured value (M)	Percentage of recovery (M/C)
Zn	368	323.24	87.8
Ni	1.3	1.62	124.6

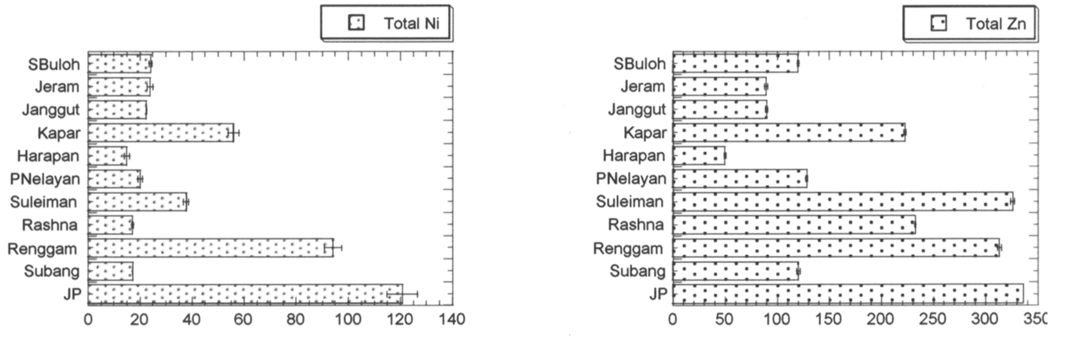

Fig. 2: Total concentrations (mean mg/g ± SE dry weight) of Ni and Zn in surface sediments collected from Tanjung Harapan to Sungai Buloh, based on direct aqua-regia method

TABLE 3
Comparison of the study data with concentrations (μg/g dry weight) of Ni and Zn reported from this region and Malaysia

Location	Ni	Zn	References
Regional studies			
Manila Bay, Philipines	10-19.0	60-329	Prudente *et al.* (1994)
Pitchavaram, India	21-58	25-60	Kurokawa & Tatsukawa (1990)
Cochin estuary, India	-	1266	Balachandran *et al.* (2005)
Central Java Coast, Indonesia	17.8 – 36.1	84 – 259	Takarina *et al.* (2004)
Singapore estuary	-	100 – 550	Sin *et al.* (1991)
Singapore coral reefs	-	58 – 95	Flammang *et al.* (1997)
Jurujuba Sound, Brazil	-	158	Baptista *et al.* (2000)
Java Sea, Indonesia	-	33 – 192	Evaraarts (1989)
Mai Po, Hong Kong	65.3 – 66.0	277.2 – 321.2	Ong Che (1999)
Deep bay, Hong Kong	30	240	Tam and Wong (2000)
South China Sea	-	12.5 – 49.9	Shazili *et al.* (1987)
Malaysian Studies			
Port Klang, Selangor	-	11.0 – 66	Ismail *et al.* (1989)
West coast Peninsular Malaysia	-	50 – 1400	Ismail *et al.* (1993)
Langat River -	71.0 – 374		Sarmani (1989)
Sepang Besar River	-	4.0 – 550	Ismail and Rosniza (1997)
Urban Lake of Kelana Jaya	-	34.3 – 529	Ismail *et al.* (2004)
Offshore of west coast Malaysia	-	4.00 – 79.05	Yap *et al.* (2003)
Intertidal of west coast Malaysia	-	3.12 – 306.20	Yap *et al.* (2003)
Sediments in Selangor (11 sites)	15.1 to 121	50.2 to 336	This study

anthropogenic sources from the effluents of the metal factory in the vicinity.

The overall concentrations of four geochemical fractions of Ni and Zn in the surface sediments are shown in Table 4. For the four geochemical fractions, the abundance of metal concentrations follow Zn> Ni. This disagrees with the fact that Ni is ranked 23rd [with an average concentration of 75 μg/g] while Zn as 24th [with an average concentration of 70 μg/g] most abundant element in the earth's crust (James, 1991). However, based on the present finding the concentrations of Zn is significantly higher than Ni, indicating anthropogenic input of Zn into the aquatic environment of the study sites.

Geochemical Fractions of Heavy Metal Concentrations in Sediments

Comparisons of the metal concentrations in the EFLE, acid-reducible, oxidisable-organic and resistant fractions among the sampling sites are shown in *Figs. 3* and *4*. The percentages of all fractions for each site are shown in Table 5.

Fig. 3 shows the concentrations of the Ni and Zn released in EFLE fraction and 'acid-reducible' fraction. The EFLE fraction contributed only a small portion (0–12.18%) of the total Ni and Zn in the sediments of the study areas. This clear pattern shows that JP Metal had the highest concentrations of EFLE Ni (3.59 μg/g dw) and EFLE Zn (59.96 μg/g dw). 'Acid-reducible' fraction contributed about 0.4 – 12.1% of the total concentrations of Ni and Zn in the sediments of all the sampling sites. A high concentration of Ni in the acid-reducible fraction was recorded in Jln Renggam (11.55 μg/g dw) and a high Zn concentrations (70.66 μg/g dw) was found at Sg. Kapar.

Fig. 4 shows the concentrations of the Ni and Zn released in 'oxidisable-organic' and 'resistant' fractions. 'Oxidisable-organic' fraction contributed 3.4 – 65.7% and covers the metals which are organically bound, and which are released when oxidised by, for example, peroxides. A clear pattern was shown by the Ni concentrations in which JP Metal site was found to have the highest 'oxidisable-organic' Ni with 51.70 μg/g dw. However, there was no clear pattern for Zn concentrations in the 'oxidisable-organic fraction'. The'resistant' fraction contributed the largest ranges (0.6 –73.5%) of Ni and Zn in the sediments of the study areas and

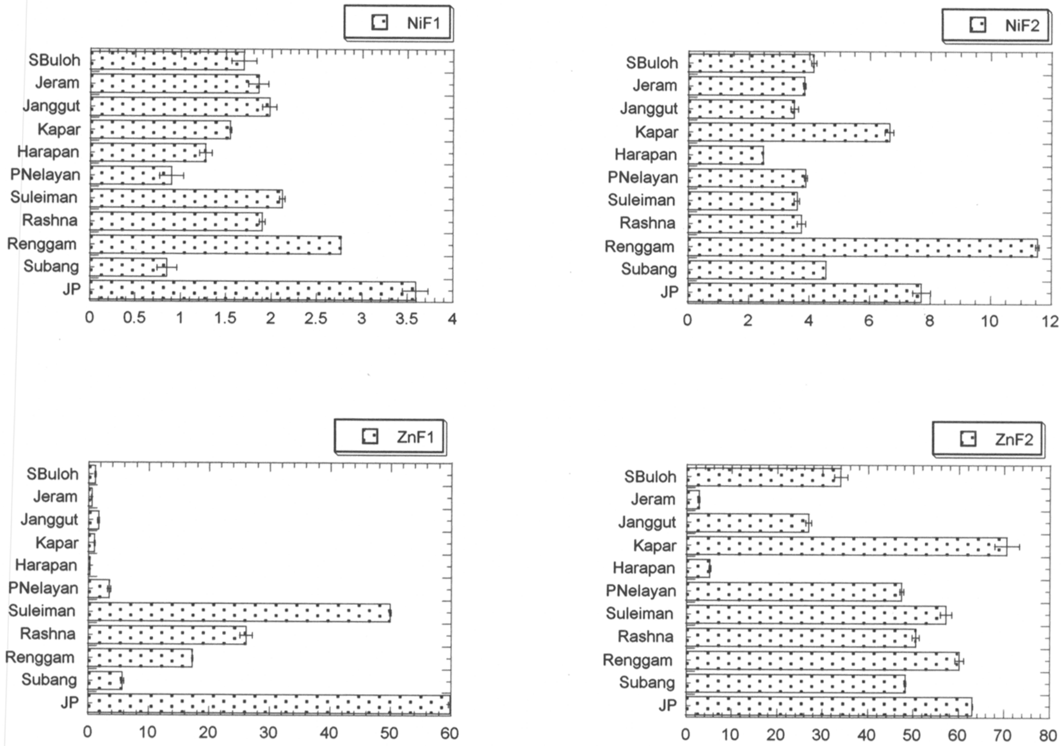

Fig. 3: Ni and Zn concentrations (mean mg/g ± SE dry weight) of the EFLE (F1) and acid-reducible (F2) fractions in the surface sediments collected from Tanjung Harapan to Sungai Buloh, based on sequential extraction technique

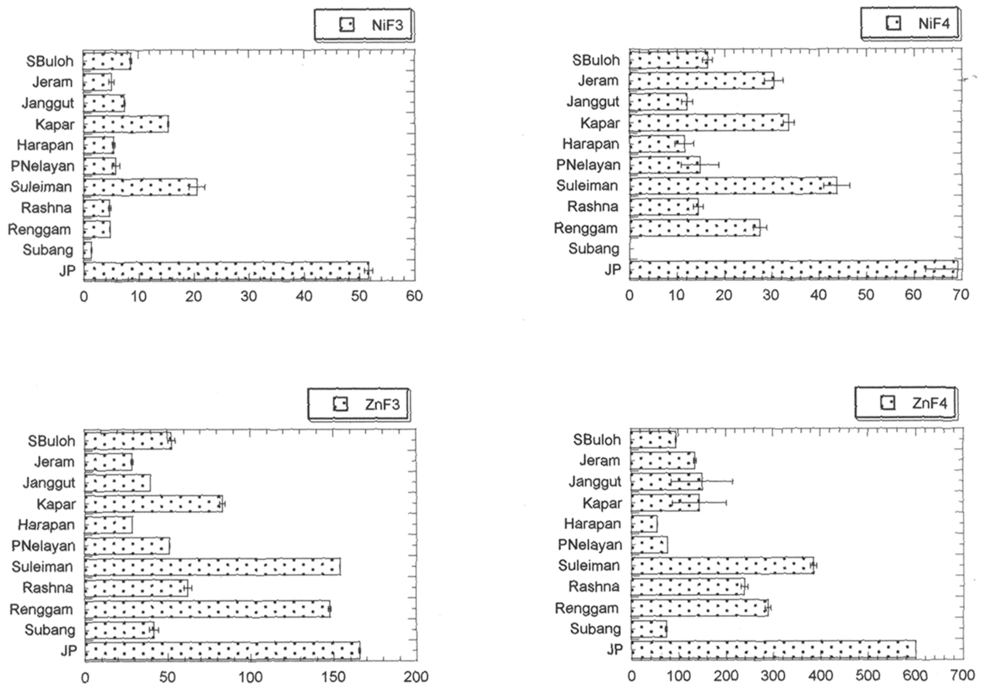

Fig. 4: Ni and Zn concentrations (mean mg/g ± SE dry weight) of the oxidisable-organic fraction (F3) and resistant (F4) fractions in the surface sediments collected from Tanjung Harapan to Sg Buloh, based on sequential extraction technique

TABLE 4
Overall concentrations (μg/g dry weight) of Ni and Zn in the surface sediments collected from urban drainages and intertidal areas of Selangor

		Minimum	Maximum	Mean	Std Error
Ni	Total	15.09	120.85	40.88	10.70
	F1	0.85	3.59	1.86	0.24
	F2	2.49	11.55	5.07	0.79
	F3	1.52	51.70	11.97	4.30
	F4	0.04	69.12	24.95	5.76
Zn	Total	59.89	344.39	197.62	30.85
	F1	0.26	59.96	15.21	6.45
	F2	2.75	70.66	42.41	6.85
	F3	28.68	165.73	77.83	15.88
	F4	29.68	793.71	165.99	67.31

F1= EFLE, F2= acid-reducible, F3= oxidisable-organic and F4= resistant. Total= based direct aqua-regia method. (N = 11)

TABLE 5
Percentages of four geochemical fractions of Zn and Ni in the sediments collected from Selangor

No	Ni%	F1	F2	F3	F4
1.	JP	2.7	5.9	39.3	52.1
2.	Subang	12.1	65.7	21.7	0.6
3.	Renggam	6.0	24.8	10.5	58.8
4.	Rashna	7.7	15.1	19.4	57.8
5.	Suleiman	3.1	5.2	29.4	62.4
6.	PNelayan	4.0	16.5	23.6	55.9
7.	Harapan	6.2	12.0	26.8	55.0
8.	Kapar	2.7	11.6	26.9	58.8
9.	Janggut	7.9	14.1	29.7	48.4
10.	Jeram	4.5	9.3	12.7	73.5
11.	SBuloh	5.5	13.4	28.1	53.0
No	**Zn%**	**F1**	**F2**	**F3**	**F4**
1.	JP	5.5	5.8	15.3	73.3
2.	Subang	3.8	32.6	27.8	35.7
3.	Renggam	4.1	14.2	34.9	46.9
4.	Rashna	8.4	16.2	20.0	55.4
5.	Suleiman	9.3	10.6	28.7	51.4
6.	PNelayan	2.4	32.1	34.8	30.7
7.	Harapan	0.4	8.2	45.0	46.5
8.	Kapar	0.4	26.3	30.9	42.4
9.	Janggut	1.5	23.6	34.7	40.3
10.	Jeram	0.6	3.4	35.2	60.7
11.	SBuloh	0.9	25.9	39.9	33.3

Note: F1= EFLE, F2= acid-reducible, F3= oxidisable-organic and F4= resistant

presumably there were those strongly trapped within the silicate minerals (Badri and Aston, 1983). In this fraction, JP Metal site showed the highest Ni and Zn concentrations which were 69.1 μg/g dw and 343.0 μg/g dw, respectively.

The Spearman's rank correlation coefficients among the four SET fractions and total concentrations of each metal are shown in Tables 6 and 7. For Ni (Table 6), all the 15 pairwises were significantly correlated (R = 0.62-0.98,

TABLE 6
Spearman's correlation coefficients among the geochemical fractions of Ni in the sediments from some places in Selangor

	F1	F2	F3	F4	Sum	Total
F1	1.00	0.17^{ns}	0.39^{ns}	0.62	0.66	0.66
F2		1.00	0.05^{ns}	0.42^{ns}	0.46^{ns}	0.68
F3		1.00	0.70	0.74	0.61	
F4		1.00	0.98	0.86		
Sum			1.00	0.92		
Total			1.00			

Note: F1= EFLE, F2= acid-reducible, F3= oxidisable-organic, F4= resistant and ns=not significant (P>0.05). Total= based direct aqua-regia method. SUM= summation of all the 4 fractions based on sequential extraction technique.

TABLE 7
Spearman's correlation coefficients among the geochemical fractions of Zn in the sediments from some places in Selangor

F1	F2	F3	F4	Sum	Total	
F1	1.00	0.63	0.77	0.68	0.86	0.88
F2		1.00	0.90	0.68	0.86	0.88
F3			1.00	0.83	0.97	0.96
F4				1.00	0.86	0.84
Sum					1.00	0.99
Total						1.00

Note: F1= EFLE, F2= acid-reducible, F3= oxidisable-organic, F4= resistant and ns=not significant (P>0.05). Total= based direct aqua-regia method. SUM= summation of all the 4 fractions based on sequential extraction technique.

$P<0.05$) except for 5 pairwises (R = 0.05-0.46, $P>0.05$). For Zn (Table 7), all the pairwises were significantly correlated (R = 0.63-0.99, $P<0.05$).

Only small portions of Ni and Zn from the total concentrations in the sediments from all the sampling sites were contributed by the EFLE fraction. This fraction was the most available fraction since it could be released from the soil even at pH 7. The low EFLE fraction indicated that heavy metals in the sediments were not easily leached out by water. This fraction might be a model for "bioavailability" to sediment ingesting animals (Yap *et al.*, 2002a). However, the high concentrations of Ni and Zn found in the EFLE fractions at some sites indicated the potential harmful effects that can be posed directly to the living organisms.

As for the 'acid-reducible' fraction, its contribution was the second lowest after the EFLE fraction in the sediment of the sampling sites. This fraction which may include metals associated with manganese and iron dioxides and hydroxides and possibly also with carbonates (Yap *et al.*, 2002b), had been proven to be sensitive to anthropogenic inputs (Modak *et al.*, 1992; Singh *et al.*, 2005). The low percentages of these fractions indicated that the affinities for this fraction in the sediment of the study areas were not high.

The 'oxidisable-organic' fraction usually contributed the highest percentage among the three anthropogenic fractions. The final fraction; the resistant fraction usually contributed the largest portion of the total concentrations of metals among the fractions. Metals in this form are not soluble under experimental conditions and may therefore be considered as being tightly bound and are highly associated with natural origins (Badri and Aston, 1983).

The high percentage in the nonresistant fraction of the total concentration of Ni in the sediment of Subang Utama (99.43%) was recorded while only 51.37% of the total Ni was accumulated in the nonresistant fraction of the

TABLE 8
Comparison of Ni and Zn concentrations (mg/g dry weight) in intertidal sediments with established Sediment Quality Criteria

Source		Ni	Zn	References
1. Hong Kong Sediment Quality Criteria.	Action level	40	200	Lau Wong and Rootham, 1993
2. Interim Sediment Quality Values (ISQVs) for Hong Kong.	ISQVs-low	40	200	Chapman *et al.*, 1999
3. Interim Sediment Quality Values (ISQVs) for Hong Kong.	ISQVs-high	NA	410	Chapman *et al.*, 1999
4. Interim freshwater sediment quality guidelines for Canada.	-	NA	123	CCME, 2002
Aquatic sediments in Selangor, Peninsular Malaysia (11 sites)		15.1 to 121	50.2 to 336	This study

sediment of Sg Janggut. The high percentage of Ni in the nonresistant fraction of Subang Utama may be due to the effluents of factories along the river bank of the Klang River. Sari and Cagatay (2001) reported that the high Ni values in the northwest of the Gulf of Saros were due to the industrial discharges delivered by the Meric River.

Nine out of the eleven stations of the study areas had high percentages of Zn in the nonresistant fraction. Peng. Nelayan, for example, showed a percentage of 70.2% from the total concentration of Zn in the sediment.

The clear pattern in the distribution of Ni and Zn in the various fractions at the 11 sampling sites could be because some (6 sites) were intertidal sediments while others (5 sites) were drainage sediments.

Since the Malaysian Interim Sediment Quality Guideline is not available, comparison of the present data with the established Sediment Quality Criteria found in the literature is presented in Table 8. The highest concentrations of Ni and Zn (at JP Metal) were found to be higher than the Action Levels of these metals established by the Hong Kong Sediment Quality Criteria (Lau Wong and Rootham, 1993) and the Interim Sediment Quality Values-low (ISQVs-low) for Hong Kong (Chapman *et al.*, 1999). In addition, comparisons with the Interim Freshwater Sediment Quality Guidelines for Canada (CCME, 2002), showed that there are 6 sampling sites (*Fig. 2*) with Zn concentrations higher than the established Zn value. All of the above comparisons indicated that these elevated metal concentrations are most likely resulting from contributions by nearby industrial activities especially at the JP Metal site.

CONCLUSIONS

The highest total concentrations of Ni and Zn in sediments were found at the JP Metal site. It was found to have the highest Ni concentrations with 120.85 μg/g dry weight which could potentially pollute the nearby river, Sg. Kuyoh. All the metal concentrations in the sediments obtained from this study were mostly higher when compared to values from previous studies. From the geochemical study of heavy metals, the results show that the non-resistant fraction dominated the total Zn. These metal fractions contributed more than 50% of the total Zn concentration in most of the study areas. It is suggested that a treatment plant should be established especially at JP Metal site in the Seri Serdang Industrial area.

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