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## Heavy metals in mangrove sediment of mengkabong lagoon, sabah: Multivariate and enrichment approaches

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### ABSTRACT

The intertidal mangrove environment of Mengkabong lagoon is important as it supports the local fishing activities, nursery grounds for many fish and shellfish species, and as well as being central for ecotourism activities. The study showed that in general, the physicochemical parameters (pH, salinity and electrical conductivity), granulometric fractions, organic matter, base cations (Na, K, Ca and Mg) and heavy metals (Fe, Cu, Pb, Zn and Al) showed an increasing loadings at high tide compared to low tide. Multivariate statistical techniques, principal components analysis (PCA) and cluster analysis (CA), were employed to better interpret information about the sediment and its controlling factors. The PCA results revealed six controlling factors at high tide while seven at low tide. In CA there are two distinct clusters were identified at high and low tide. The calculation of geoaccumulation index ( $I_{geo}$ ) suggests the Mengkabong mangrove sediments could be classified as having background concentrations of Al, Cu, Fe, and Zn as unpolluted with regard to Pb.

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### KEYWORDS

Mangrove sediment;  
Tide;  
Multivariate analysis;  
Geoaccumulation index.

### INTRODUCTION

Mangrove sediments have been extensively studied all around the world (India, Australia, Brazil, Malaysia, United Arab Emirates, China, Thailand etc) and some of these assessed the pollution status of the sediments. For example, a study done by Kehrig et al.<sup>[1]</sup> in Jequia mangrove forest, Brazil concluded that the mangrove forest has been polluted with heavy metals by the

anthropogenic sources surrounding the estuary, while study done by Ramanathan et al.<sup>[2]</sup> concluded the concentration of heavy metals in Pichavaram mangrove forest, India were generally below the levels found in polluted and unpolluted estuaries and mangroves.

Heavy metal cycling is a serious problem addressed in mangrove environments<sup>[3,4]</sup>. The high concentrations of heavy metals are derived from anthropogenic inputs from industrial activities around the estuary such as dis-

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carded automobiles, batteries, tires, waste water disposal etc<sup>[5]</sup>. For an example, Shriadah<sup>[5]</sup> found high concentrations of lead due to the inputs from oil spills from discarded automobiles in addition to petrol combustion while study done by Bloom and Ayling<sup>[6]</sup> in the Derwent Estuary in revealed high concentration of zinc and lead due to a zinc refining company situated near the estuary. Sediments act as sinks and sources of contaminants in aquatic systems because of their variable physical and chemical properties<sup>[3,4,7]</sup>. Pekey<sup>[4]</sup> demonstrated that heavy metals tend to be trapped in the aquatic environment and accumulate in sediments.

A multivariate statistical approach allows the researchers to manipulate more variables<sup>[8]</sup>. Principal Components Analysis (PCA) and cluster analysis (CA) were the statistics methods used in the interpretation. PCA and CA often used together to check the results and provide grouping of each variable. PCA and CA are explanatory tools in multivariate statistical analysis to discover and interpret relationships between variables<sup>[9,10,11]</sup>. According to Facchinelli et al.<sup>[10]</sup>, FA and CA are often used together to check the results and provide grouping of each variable. Yongming et al.<sup>[9]</sup> explained that PCA is widely used to reduce data and to extract a small number of factors depending on the correlation matrix, whereas CA is performed to further classify elements of different sources on the basis of their similarities chemical properties. Hierarchical cluster analysis using dendograms identifies relatively homogeneous groups of variables in separate clusters and combines clusters until only one is left.

Mengkabong lagoon formerly earmarked as the Mengkabong Forest Reserva, but released by the Forestry Department to become a state land. The Mengkabong mangroves, Tuaran District, experienced a 15% decrease from 1991 to 2000. In 1991 the mangroves covered 12.6km<sup>2</sup> while in 2000 it was 10.7km<sup>2</sup>. Most of the mangroves have been lost due to the spread of rural development such as housing, aquaculture projects and surrounded by an industrial zone, Kota Kinabalu Industrial Park (KKIP)<sup>[12]</sup>. There is practically no detailed information on the environmental study of Mengkabong mangroves. Therefore, it was decided to conduct a study of Mengkabong lagoon mangrove sediment and its pollution status.

The purpose of this study was: (1) to determine the physicochemical parameters (pH, electrical conductivity

and salinity), granulometric fractions, organic matter, heavy metals (Al, Cu, Fe, Pb and Zn) and base cations (Ca, Mg, Na and K) at high and low tide; (2) to identify the controlling factors by using factor analysis (FA) and cluster analysis (CA) at high and low tide and (3) to gauge the degree of anthropogenic influence on heavy metals concentration in mangrove sediment using geo-accumulation index ( $I_{geo}$ ).

## MATERIALS AND METHODS

### Study area

This study was conducted in Mengkabong mangrove forest, Tuaran District, West Coast of Sabah which is 40 km away from Kota Kinabalu. The total of study area spreads over from latitude 06°06'N to 06°11'N and longitude 116°08'E to 116°13'E (Figure 1). The Mengkabong mangrove forest consists of two shallow spurs, with the southern spur forming the administrative boundary between Tuaran and Kota Kinabalu Districts. This spur ends in Salut Bay which is entirely surrounded by an industrial zone, Kota Kinabalu Industrial Park. The southern spur of the estuary has been significantly degraded already and there is little left to protect. The northern spur is much larger and more irregular. There are still abundant and high quality mangroves remaining around the estuary<sup>[13,14]</sup>.

### Soil sampling and analysis

The sampling strategy was to study the spatial variability and tidal effects on a number of parameters. Mangrove sediments were sampled randomly and taken

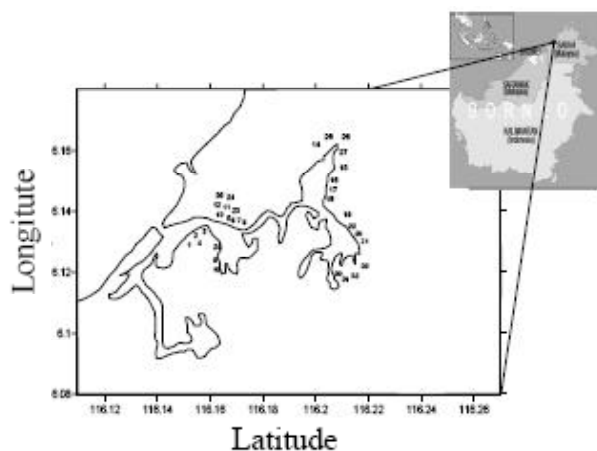


Figure 1: Sampling locations of Mengkabong and mangrove surface sediment sampling sites (n=33)

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**TABLE 1: A brief account of the methods used in this study**

Parameter	Method	Reference
pH	Glass electrode	Mettler toledo Delta 320 manual
EC	Glass electrode	Eutech/Oakton ECTestr™ 11 plus manual
Salinity	Glass electrode	Eutech/Oakton salt testr™ 11 plus manual
Organic matter	Loss on ignition (LOI)	Heiri et al. <sup>[17]</sup> , Radojevic & Bashkin <sup>[16]</sup>
Granulometric analysis	Pipette method	Radojevic & Bashkin <sup>[16]</sup>
Total heavy metals	AAS	APHA <sup>[18]</sup>
Base cations	AAS	APHA <sup>[18]</sup>

**TABLE 2: Physico-chemical properties, organic matter, granumetric fraction, heavy metals and base cations**

Parameter		Min	Max	Mean	SD
pH	HT	5.4	7.6	6.5	0.4
	LT	4.5	7.2	6.1	0.6
Sa (%)	HT	1.5	7.4	4.5	2.2
	LT	0.1	1.5	0.5	0.4
EC(mS/cm)	HT	2.1	11.4	6.8	2.6
	LT	0.4	4.8	2.6	1.0
OM (%)	HT	6.4	11.7	9.0	1.3
	LT	1.4	5.2	2.5	0.8
Sand (%)	HT	91.9	95.1	93.6	0.8
	LT	91.7	97.3	95	1.7
Silt (%)	HT	2.5	5.2	3.6	0.8
	LT	0.4	6.4	2.7	1.8
Clay (%)	HT	2.2	4.2	2.8	0.4
	LT	1.3	2.9	2.4	0.4
Na (g kg <sup>-1</sup> )	HT	14.2	83.5	47.5	962.8
	LT	2.5	92.7	41.6	34.6
K (g kg <sup>-1</sup> )	HT	5.7	16.0	9.4	2.5
	LT	2.4	10.6	7.8	1.8
Mg (g kg <sup>-1</sup> )	HT	2.0	9.2	5.3	1.8
	LT	1.0	7.6	3.8	1.9
Ca (g kg <sup>-1</sup> )	HT	2.6	52.9	21.3	41.7
	LT	1.5	47.7	16.2	15.1
Fe (g kg <sup>-1</sup> )	HT	3.4	14.2	7.7	2.7
	LT	1.4	18.4	6.8	4.0
Cu (mg kg <sup>-1</sup> )	HT	4.1	49.0	28.0	14.0
	LT	2.1	44.0	19.0	13.0
Zn (mg kg <sup>-1</sup> )	HT	24.0	93.0	57.0	17.0
	LT	12.0	73.0	41.0	17.0
Pb (mg kg <sup>-1</sup> )	HT	24.0	69.0	52.0	11.0
	LT	34.0	47.0	41.0	3.0
Al (g kg <sup>-1</sup> )	HT	4.4	3.5	14.8	8.2
	LT	2.4	2.4	9.5	6.0

in triplicates with an auger at 33 stations from March 2006 to November 2006 (Figure 1) at low and high tide. The exact position of each sampling site was recorded using Global Positioning System (GPS). The sampling was done based to the accessibility to the

mangrove forest. Mangrove surface sediments were chosen for this study as this layer controls the exchange of metals between sediments and water<sup>[15]</sup>.

Sampling bottles and the laboratory apparatus were acid soaked in diluted nitric acid before the analysis. After acid soaking, they were rinsed thoroughly first with tapwater and then with distilled water to ensure any traces of cleaning reagent were removed. Finally, they were dried and stored in a clean place. The sediments were kept cool in an icebox during transportation to the laboratory. The physicochemical measurements of the surface sediment were made as soon as possible in the Environmental Science Postgraduate Laboratory, School Science and Technology, Universiti Malaysia Sabah. The physicochemical parameters (pH, electrical conductivity and salinity) were measured on 1:2 soil to water ratio extracts as soon as the samples reached the laboratory<sup>[16]</sup>. The pH, electrical conductivity and salinity electrodes were calibrated before the measurements were taken.

For other analyses, the surface sediments were air-dried, and after homogenization using pestle and mortar, passed through a 2-mm mesh screen and stored in polyethylene bags. Methods and references for various analyses are given in TABLE 1. For the determination of heavy metals, the samples were digested using aqua regia. Approximately 2g of each sample was digested with 15mL of aqua-regia (1:3 HCl: HNO<sub>3</sub>) in a Teflon bomb for 2h at 120°C. After cooling, the digested samples were filtered and kept in plastic bottles before the analysis. Radojevic and Bashkin<sup>[16]</sup> stated that aqua regia is adequate for extraction of total metals in soil sample and is widely used in most soil analyses.

For base cations (Na, K, Ca and Mg), the method used in this study is the measurement of exchangeable cations using ammonium acetate. Heavy metals and base cations were analyzed using AAS with air/acetylene (Cu, Fe, Pb, Zn, Na, K, Ca and Mg) and nitrous oxide-acetylene (Al) at specific wavelengths (Atomic Absorption Spectrometer Perkin Elmer 4100).

## RESULTS

### Descriptive statistics

The descriptive statistics of physico-chemical properties (pH, salinity, electrical conductivity) granulometric fraction, organic matter, heavy metals (Fe, Cu, Zn, Pb

and Al) and base cations (Na, K, Mg, and Ca) low and high tide are given in TABLE 2.

**Principle components analysis (PCA)**

PCA was applied to discover and interpret relationships between variables at high and low tide. The results showed a different trend at high tide and at low tide. TABLES 3 and 4 display the factor loadings with a Varimax rotation as well as the eigenvalues, percentile of variance and cumulative percentage at high and low tide. In reference to the eigenvalues, six factors at high tide and seven factors at low tide were extracted as they have eigenvalues greater than 1 (TABLES 3 and 4).

At high tide, factor one accounted for 22% of total variance and is mainly characterized by high levels of salinity, electrical conductivity and clay fraction. Factor two accounted for 13% of the total variance with sand loadings. Factor three consists of Cu and K with total variance of 11%. Factor four characterized by 9% of total variance with high loadings of Fe, Ca and Al. Factor five with 8% of total variance, contains high loading of Na and pH. Factor six with 7% of total variance is characterized by high loading of Pb.

At low tide, factor one accounted for 24% of total variance and is mainly characterized by high levels of silt, Al and Zn. Factor two accounted for 13% of the total variance with Mg, Ca and Pb loadings. Factor three consists of Cu and clay with a total variance of 10%. Factor four is characterized by 9% of total variance with high loadings of salinity and K. Factor five, with 8% of total variance, contains high loading of pH while factor six, with 7% of total variance, is characterized by high loading Fe. Factor seven has 6% of total variance with Na loading.

**Cluster analysis (CA)**

Cluster analysis was performed on the data set using average linkage between groups (Rescaled Distance Cluster). Although not substantially different from PCA, CA can be used as a substitute method to confirm the results of PCA. The results are illustrated in the dendrograms on Hierarchical Cluster Analysis (Figure 2 and Figure 3). At high tide (Figure 2), two distinct clusters can be identified. Cluster one contains salinity, electrical conductivity, clay, organic matter, Cu, Pb and K. Cluster two contains pH, silt, sand, Na, Zn, Al, Fe, Mg and Ca. At low tide (Figure 3), two distinct clusters

**TABLE 3: Rotated component matrix of mengkabong mangrove forest at high tide**

Variable	Component					
	1	2	3	4	5	6
pH	-0.03	-0.12	-0.16	-0.11	0.86	-0.08
Sa	0.90	0.09	-0.09	0.08	0.01	-0.14
EC	0.87	0.16	-0.16	-0.04	-0.14	-0.09
OM	0.45	0.29	0.34	-0.40	-0.03	0.15
Clay	0.65	-0.19	0.24	-0.25	-0.35	0.20
Silt	-0.37	-0.84	-0.10	0.10	0.20	-0.07
Sand	0.04	0.96	-0.03	0.03	-0.02	-0.03
Al	-0.38	0.13	-0.57	0.64	0.04	0.17
Cu	0.01	0.09	0.56	0.02	0.19	0.38
Fe	0.01	-0.20	0.26	0.65	-0.14	0.30
Pb	-0.08	0.04	-0.07	0.09	0.01	0.88
Zn	-0.54	-0.24	-0.44	0.05	-0.46	-0.13
Mg	-0.29	0.45	0.37	0.05	-0.26	0.11
Ca	-0.02	0.09	-0.04	0.81	-0.06	-0.05
Na	-0.15	-0.11	0.09	-0.12	0.64	0.11
K	-0.21	0.06	0.75	0.20	-0.17	-0.23
Initial Eigenvalue	3.52	2.11	1.71	1.48	1.31	1.06
Percent of variance	21.98	13.20	10.67	9.26	8.21	6.64
Cumulative Percent	21.98	35.18	45.84	55.11	63.32	69.96

**TABLE 4: Rotated component matrix of mengkabong mangrove forest at low tide**

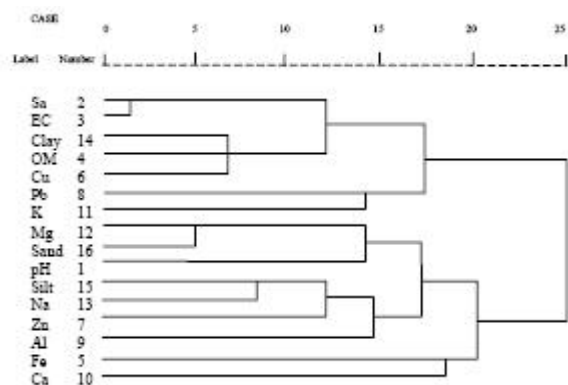
Variable	Component						
	1	2	3	4	5	6	7
pH	-0.09	0.32	0.01	0.03	0.80	-0.16	0.16
Sa	-0.17	0.01	-0.03	0.73	0.04	-0.37	0.04
EC	0.28	0.24	0.01	-0.12	-0.75	-0.14	0.13
OM	-0.04	0.45	-0.42	0.33	-0.15	0.33	-0.11
Clay	-0.52	-0.06	0.61	0.11	0.10	-0.32	-0.08
Silt	0.91	-0.18	-0.08	0.06	-0.26	0.04	0.03
Sand	0.89	0.21	-0.05	-0.09	0.26	0.03	-0.02
Al	0.78	0.20	-0.18	-0.29	0.22	0.11	-0.12
Cu	-0.07	0.06	0.84	0.10	-0.09	0.17	0.01
Fe	0.05	0.03	0.05	-0.03	-0.01	0.87	0.06
Pb	0.33	0.58	0.23	-0.34	0.13	0.15	-0.10
Zn	0.57	-0.12	-0.45	0.25	-0.26	0.03	-0.22
Mg	-0.16	0.83	0.13	0.09	0.02	-0.23	0.05
Ca	-0.20	0.63	-0.19	0.04	0.20	0.21	-0.03
Na	-0.05	-0.01	0.02	0.06	0.02	0.06	0.97
K	0.24	0.07	0.19	0.81	0.11	0.32	0.05
Initial Eigenvalue	3.89	2.04	1.63	1.49	1.27	1.11	1
Percent of variance	24.26	12.73	10.21	9.34	7.92	7	6.28
Cumulative Percent	24.26	36.99	47.20	56.53	64.45	71.45	77.72

were observed, Cluster one contains silt, Al, Fe, K, Zn, electrical conductivity and salinity; Cluster two con-

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**TABLE 5 : Geoaccumulation indexes[19] of heavy metals concentration in sediment of Mengkabong mangrove forest**

Geoaccumulation index	Pollution Intensity	Heavy metals ( $I_{geo}$ class for Mengkabong lagoon sediment)
0	Background concentration	Al, Cu, Fe, Zn
0-1	Unpolluted	Pb
1-2	Moderately to unpolluted	
2-3	Moderately polluted	
3-4	Moderately to highly polluted	
4-5	Highly polluted	
>5	Very highly polluted	



**Figure 2: Dendrogram showing hierarchical cluster analysis at high tide**

tains Ca, Mg, organic matter, clay, sand, pH, Pb, Cu and Na.

### Geo-accumulation index ( $I_{geo}$ )

The geoaccumulation index ( $I_{geo}$ ) introduced by Muller<sup>[19]</sup> was also used to assess metal pollution in sediments. Geoaccumulation index is expressed as follows:

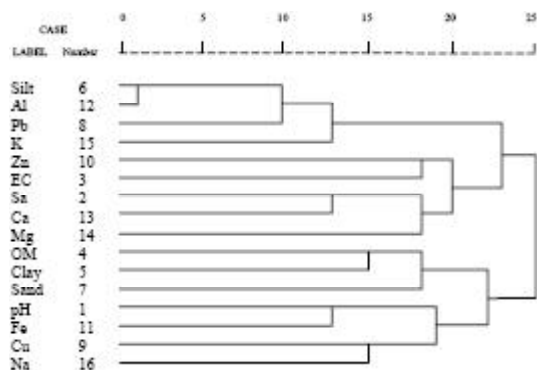
$$I_{geo} = \text{Log}_2 (C_n / 1.5B_n)$$

where  $C_n$  = measured concentration of heavy metal in the mangrove sediment,  $B_n$  = geochemical background value in average shale<sup>[20]</sup> of element n, 1.5 is the background matrix correction in factor due to lithogenic effects. The calculated values for the Mengkabong mangrove sediment are given in TABLE 5 and remain in either class 0 or class 1, which indicates that the investigated mangrove sediments in Mengkabong mangrove sediment are unpolluted.

## DISCUSSION

At high tide, factor one in PCA results contain sa-

linity, electrical conductivity and clay fraction. This corresponds to the associations in Cluster one results at high tide. Salinity and electrical conductivity clusters at the distance of one (Figure 2), shows that the contributions of seawater during high tide in Mengkabong. According to<sup>[21]</sup>, seawater contains 3.5% of salinity of which 90% is fully ionized ions. The fully ionized ions and high salinity at high tide explained the high load of salinity and electrical conductivity at high tide. The salinity and electrical conductivity then form another cluster with organic matter and clay fraction. These associations then formed another cluster with PCA results of factor one, factor six and factor three. While factor three consists of Cu and K, with total variance of 11% in agreement with CA results (Cluster one) where the association of Cu, Pb, Mg, K and sand. Tidal flooding can bring additional ions such as K, Mg and Na into the system. It enables ion exchange to occur, such as between K and Cu<sup>[22]</sup>. Similar findings were obtained between K and Cu by Grande et al.<sup>[23]</sup>, who observed a negative correlation with marine indicators such as K. The new condition induced by tidal clash causes precipitation of metals such as Cu. These associations are mainly related to anthropogenic inputs and reflect the complexing nature of clay. Pb most probably arises from indirect sources for instance atmospheric deposition<sup>[24]</sup>. Based on PCA (factor four, factor five and factor six) and CA (Cluster 2), Al, Ca, Fe and Zn were correlated in PCA and CA results. The CA results showed the relationship between Al, Ca, Fe and Zn at the distance of 12, 15 and 19. pH value in estuarine sediments is one of the important factor that regulates the concentration of dissolved metals in water and sediment<sup>[23,25]</sup>. The CA results showed associations between pH, Na silt and Mg. According to Hsue and Chen<sup>[25]</sup>, dissolved salts such as  $\text{Na}^+$  and  $\text{Mg}^{2+}$  played an important role in buffering the pH change. The process of tidal clash occurs in the Mengkabong mangrove forest. The influx of seawater from the high tide resulted in major inputs of selected cations which were then adsorbed by the sediment (clay, silt and sand). The association between Mg and sand is strongly controlled by biogenic carbonates and plays an important role as a dilutant material of the heavy metals in the samples<sup>[26]</sup>. Biogenic carbonates are the dominant source of  $\text{Ca}^{2+}$ , an abundant component in shallow marine sands<sup>[27]</sup>. Ca is an important components of marine biota and plays vital a role in the



**Figure 3: Dendrogram showing hierarchical cluster analysis at low tide**

marine biogeochemical cycle<sup>[28]</sup>.

At low tide, silt and Al clusters at the distance of 1. Then, this association clusters with Fe (factor six in PCA). Salinity and electrical conductivity clusters at the distance of 14 at low tide. This distance is far higher compared to the association of electrical conductivity and salinity at high tide (Figure 2 and Figure 3). This demonstrates the lower contributions of seawater at low tide compared to high tide. While at low tide, in PCA (TABLE 4), it shows the factor one accounted for 24% of total variance and is mainly characterized by high levels of silt, Al, Zn and EC. Cluster one (Figure 3) shows the silt and Al is associated at the distance of 1. Besides, there is also an association of Zn of factor one in PCA with Fe (factor six in PCA), K and salinity (factor four in PCA). Zhou et al.<sup>[28]</sup> stated that besides anthropogenic enrichment, heavy metals occur naturally in silt and clay-bearing minerals of terrestrial and marine geological deposits. The natural occurrence of heavy metals complicates the assessment of potentially contaminated estuarine sediments. The measurable concentrations of metals do not automatically infer anthropogenic enrichment in the estuary. Therefore, heavy metal enrichment assessment is conducting in detail. While association of  $K^+$  is explained as a function of ionic strength.  $K^+$  is a monovalent cation with low replacing power compared with divalent cations such as  $Fe^{2+}$ ,  $Zn^{2+}$  etc<sup>[29]</sup>. Cluster 2 at low tide (Figure 3) shows clusters of Ca, Mg, organic matter, clay fraction, sand fraction, pH, Pb, Cu and Na. These associations are consistent with PCA results with the loading factor of factor two, factor three, factor five and factor seven. PCA and CA results show associations between organic matter and pH, clay fraction, sand, Cu, Pb, Ca

and Mg. In coastal environments such as in mangroves, the associations between organic matter, granulometric fractions, heavy metals and base cations are become as functions of ionic strength of sediment solution and surface cation complexation<sup>[29]</sup>. The concept of cation exchange capacity at low tide implies that ions will be exchanged between wetlands, colloid surface and the surrounding water. Sediment organic matter has higher ion exchange capacity than sediment colloids and plays an important role in cation exchange capacity<sup>[30]</sup>. As elaborated by Hussien and Rabenhurst<sup>[29]</sup>, the replacing power of the cation exchange complex depends on its valence, ionic strength, its diameter in hydrated form and its concentration in water. This clarifies the loadings of marine indicator divalent cations ( $Mg^{2+}$ ,  $Ca^{2+}$ ) together with organic matter and  $Pb^{2+}$  due to the ionic strength. Moreover, it also observed that the dissolved salts such as  $Na^+$ ,  $Mg^{2+}$ ,  $Ca^{2+}$  etc. The role in buffering the pH change at low tide was also observed.

$I_{geo}$  has been applied to the set of sediment data to assess the sediment quality in Mengkabong mangrove forest. The  $I_{geo}$  index proposed by Muller<sup>[19]</sup> showed that all the heavy metals are in Class 0 and Class 1 (TABLE 5) at high and low tide. Buccolieri et al.<sup>[31]</sup> in the Gulf of Taranto, Italy, also reports similar findings with this study. This suggests that the mangrove sediment of Mengkabong is unpolluted. Soto-Jimenez and Paez-Osuna<sup>[32]</sup> demonstrated the input of metals into sediment that are located seawards to be low in the total concentration of most of the elements and this could be due to the mixing of enriched particulate material with relatively clean marine sediments.

## CONCLUSIONS

The application of multivariate analysis (PCA and CA) technique has been proved to be an effective tool for factors identification in Mengkabong mangrove sediment at high and low tide. PCA analysis extracted factors were six at high tide while there were seven at low tide. According to CA results, two clusters were formed at high and low tide. The salinity and electrical conductivity component loadings and clusters at high and low tide explained the tidal process where there is high contribution of seawater. The tidal clash causes the precipitation of metals. The relationships between organic matter, granulometric fractions, heavy metals, base cat-

## Current Research Paper

ions become as a function of ionic strength of sediment and surface complexation were observed. The associations between the parameters mainly related to anthropogenic inputs and reflect the complexing nature of clay. The  $I_{geo}$  calculations revealed that the Mengkabong mangrove sediment was in unpolluted condition.

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