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Trace element concentrations in fine sediment and linkages to non-point pollution source: Lower Johor river basin

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Abstract. Johor Strait is an economically important freshwater system in the southern portion of Peninsular Malaysia. In past decades, Johor has been experiencing rapid developments especially in industrialisation, urbanisation and agricultural activities which have impacted the quality of Johor river. This study focused on identifying the intensity and degree of sediment contamination by trace elements from different anthropogenic sources using the multiple Risk Indexes. Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) was used to detect trace element concentrations from nine sampling stations. The overall ranges for metals are 0.35-4.25, 505.86-1864.56, below detection limit (BDL)-5.37, 0.02-0.07, 0.02-0.17, 0.59-2.05, BDL-5.35, 247.07-1010.23, 0.71-9.62, 1.08-5.68 and 10.87-21.15 mg/kg for Ag, Al, As, Cd, Co, Cr, Cu, Fe, Mn, Ni, and Zn respectively. The mean concentrations of trace elements follow the order: Al > Fe > Zn > Cu > Mn > Ni > Ag > Cr > As > Co > Cd. In this study, high concentrations of most elements (Al, As, Cd, Co, Fe, Mn, and Ni) were recorded at SS5 as the station is located near the Kota Tinggi city. Comparison with the sediment quality guidelines (SQGs) portrayed that concentrations of As, Cd, Cr, Cu, Ni, and Zn were below the Threshold Effects Level (TEL), Severity Effects Level (SEL), Probable Effects level (PEL) values in all sampling stations. The Pollution Load Index (PLI) that ranged between 0.151 and 0.389 (PL1 < 1) indicates that the Johor river sediments are free of trace element contamination. Potential Ecological Risk Index (RI), and Potential Ecological Risk Factor (E_r) were in the range of 3.018-11.823 (RI < 150) and 0.103-7.141 (E_r <40) respectively, which indicate that trace elements in Johor river pose no adverse effects on aquatic biota. The Pearson's correlation matrix showed a good positive correlation between Al and As (0.546), Co (0.595), Fe (0.440), Mn (0.770), and Ni (0.496), representing similar sources of pollution. The cluster analysis indicates that Al, Mn, As, Ni, Fe, Cd, and Co originated from natural processes while Cr, C, Ag, and Zn are mainly from anthropogenic sources. Suggesting that man-made activities are accelerating sedimentation rate and washing down the pollutants together to the adjacent water bodies. Tracing the origin of the elements and planning for target mitigation to reduce further deterioration to the receiving river system could be the next mode of action.

1. Introduction

Trace elements are the most common environmental contaminants in aquatic environments, and their impacts on plant and animal life have led to serious concerns [1]. Trace elements may originate from natural processes (i.e. weathering of rock) and anthropogenic activities (i.e. domestic and industrial wastes discharge, atmospheric pollutants, agricultural activities, emission from vehicles and urban development) [2, 3]. Rivers play an important role in transporting trace elements from one point to another (including from diffused sources) which eventually end up being stored in sediment.[4]. Since sediments act as both carriers and sinks for pollutants, they can be used as an indicator of trace element pollution in aquatic environments [2, 5]. The assessment of sediment elemental contamination may reveal the history of the pollution and aids in detecting the impacts of anthropogenic activities on freshwater environments [6, 7].

Sediment quality guidelines (SQGs) have been developed to assess the biological effects of contaminants in sediments [2]. Threshold effects level (TEL), severity effects level (SEL), and probable effects level (PEL) are the most commonly used guidelines to provide an acceptable threshold concentration level for sediment-bound pollutants in order to protect aquatic biodiversity [2], [8]. Sediment pollution indicators such as the potential ecological risk (RI), potential ecological risk factor (E_r) and pollution load index (PLI) applied in the present study have been used in previous studies worldwide [2, 9]. Cluster analysis is a multivariate statistical approach that is used to identify the interrelationship between variables and to classify the contaminants from different sources based on their similarity [4, 10].

Recently, Johor is experiencing rapid development especially in industrialisation, urbanisation and agricultural activities which have impacted the water quality of Johor river – the main river in southern Peninsular Malaysia [11]. High impact developments along the Johor river can contribute significantly to trace element contamination in river sediments. Trace elements are serious environmental contaminants due to their toxicity, persistence, abundance, non-degradable nature and potential bioaccumulation in aquatic biota [2, 3, 12]. Therefore, there is a critical need to continuously monitor the levels of trace element contamination along the Johor river.

The objectives of the present study are (i) to determine trace element concentrations in the sediments along the Johor river using the Inductively Coupled Plasma-Mass Spectrometry (ICP-MS); and (ii) to determine the degree of contamination in sediments using pollution indexes (iii) to identify the inter-relationship between trace elements using cluster analysis (CA).

2. Methodology

2.1. Study area

Johor river (main river of the state of Johor) is located in the southeast of Peninsular Malaysia-. The river with a length of approximately 122.7 km originates from mount Gemuruh (at elevation of 109 m) [11]. The river flows in the north-south direction and then south-west before it empties at Johor Strait. The Johor river basin is located between the latitude 1°27′00″N to 1°49’00″N and longitude 103°42′00″E to 104°01′00″E. Sayong river and Linggiu river are the two main tributaries located in northern portion of the Johor river [13]. The mean annual rainfall around the Johor river basin is approximately 2,500mm with temperature ranging from 21ºC to 32ºC. Major land-uses along the Johor River are urbanization, oil palm cultivation and mangrove [11]. Water quality of the Johor river has deteriorated with increasing levels of pollutants, originating from different land-use activities.

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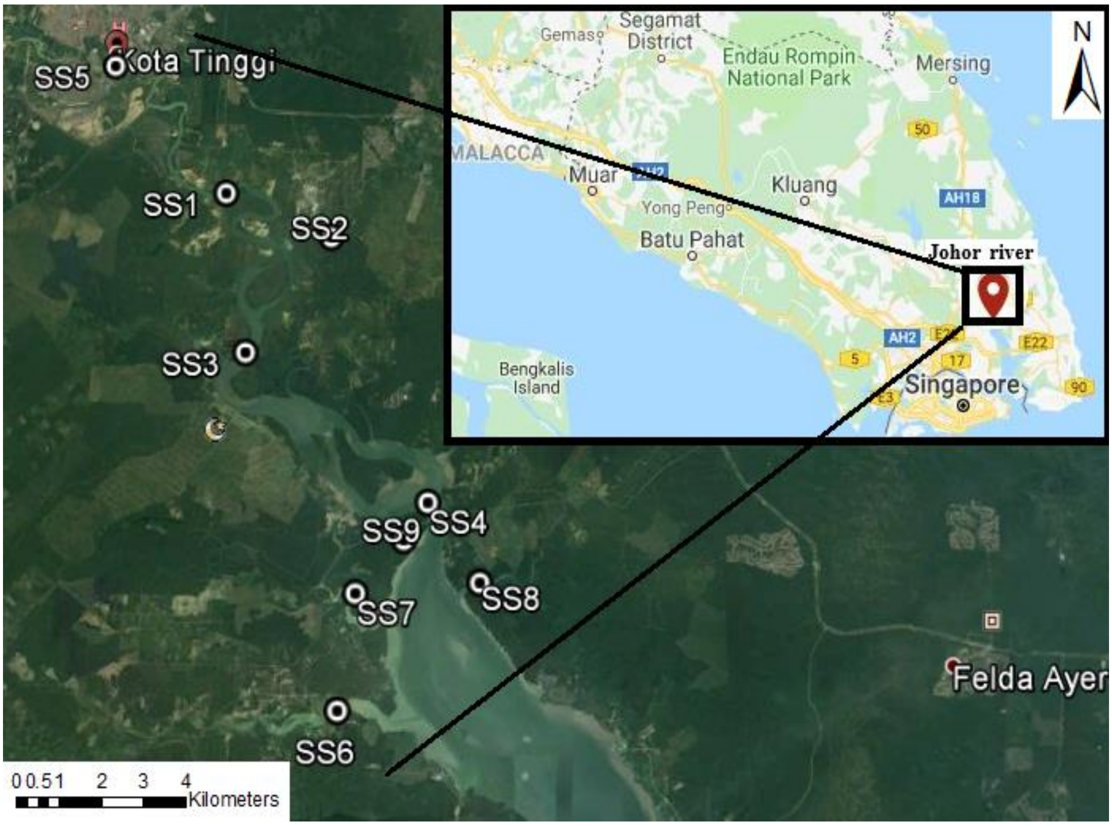

Figure 1. Sampling points of Johor river.

2.2 Sample collection and preparation

Sediments (0-2cm depth) from the Johor river were randomly sampled at nine sampling stations along the river. 3-5 samples were collected from each station. The exact locations of each station were recorded using a Global Positioning System (GPS) tracker (Model: Garmin Montana 650). The collected samples were sealed in clean zip-lock plastic bags and labelled before being transported to the laboratory for further analysis [14]. In the laboratory, the samples were air dried in an oven at 60^0C for 24 hours, homogenized using a pestle and mortar, then sieved through a 63μm mesh to obtain fine-grained sediments

2.3 Analytical method

0.5g of the sieved sample was treated with nitric acid, HNO_3, 30% hydrogen peroxide H_2O_2 and hydrochloric acid, HCI according according to USEPA (1996) 3050B method [15]. Then, the treated sample was digested in a microwave for 45 minutes and then cooled to room temperature over one hour. After the digestion, the trace element concentrations in the sediment sample were measured using the ICP-MS (Model Perkin Elmer NexION 320X).

2.4. Assessment of sediment contamination

Sediment quality guidelines (SQGs) and pollution indexes are effective tools in evaluating trace element contamination in sediment samples. Concentrations of elements from the study area were compared to an unpolluted reference material (geochemical background value). Since there were no background (pre-industrial) data for the study area, the average shale value (ASV) proposed by Turekian and Wadephol (1961) was used as the background element concentration for the sediment analysis [16].

2.4.1. Sediment quality guidelines (SQs). Concentrations of elements from the study site were compared with SQGs to evaluate the potential risk to the environment. SQGs have been widely used in sediment analysis to prescribe acceptable concentration ranges of trace elements in sediments in order to protect freshwater species [2]. In the present study, three quality guidelines such threshold effects level (TEL), severity effects level (SEL), and probable effects level (PEL) were used to evaluate the intensity of trace element pollution in the Johor river sediments. TEL, PEL, and SEL values are only available for As, Cd, Cr, Cu, Ni, and Zn [17, 18].

2.4.2. Pollution load index (PLI). Tomlinson et.al. (1980) proposed the PLI to measure severity of trace element pollution. The PLI for a single site is the n^{th} root of *n* number multiplying the factors (CF values) together [19], which is as follows:

$$PLI = \sqrt[n]{CF1 \ x \ CF2 \ x \ CF3 \ldots . xCFn} \tag{1}$$

$$CF = \frac{C^i_{sample}}{C^i_{ref}}, \tag{2}$$

where CF is the contamination factor, C^i_{sample} is the measured value of the heavy element in the sediment, and C^i_{ref} is the average shale concentration. Tomlinson et.al. (1980) was categorized PLI into three main classes; PLI = 0 indicates a perfect state of pollution; PLI = 1 points indicates only baseline levels of pollutants present and PLI >1would indicate progressive deterioration of sites [19].

2.4.3. Ecological risk index. Hakanson (1980) developed potential ecological risk index (RI) to assess the potential risks of trace elements to freshwater environment [20]. RI is calculated by the following formula:

$$RI = \sum_{i=1}^{n} E^i_r = \sum_{i=1}^{n} T^i_r \ x \ C^i_f \tag{3}$$

where C^i_f , E^i_r and T^i_r are the contamination factor, potential ecological risk factor and toxic response factor respectively, for the given element of *i.* The toxic response factors for Cd, As, Ni, Cu, Cr, Zn, Mn, Co are 30, 10, 5, 5, 2, 1, 1, 5, respectively [9, 20]. RI values were grouped into four classes such as RI < 150, low ecological risk; 150 ≤ RI < 300, moderate ecological risk; 300 ≤ RI < 600, considerable ecological risk; RI > 600, very high ecological risk. Hakanson (1980) classified potential ecological risk factor, E_r into 5 categories as shown in Table 1.

Table 1. Classification of E_r [20].

E_r values	Risk Level
$E_r < 40$	Low ecological risk
$40 < E_r \leq 80$	Moderate ecological risk
$80 < E_r \leq 160$	Appreciable ecological risk
$160 < E_r \leq 320$	High ecological risk
$E_r \geq 160$	Serious ecological risk

2.5. Statistical analysis and cluster analysis (CA)

Basic statistical analysis such as range, mean, median and standard deviation along with Pearson's correlation analysis were carried out using SPSS software version 24.0 for windows. Multivariate statistical analysis such as CA was used to group the trace elements into different classes based on their similarities. CA has been widely used in sediment analysis to identify the sources of the pollution by interpreting the relationships between the trace elements [10]. The CA was represented in a dendrogram using the standard method of Ward's linkage with an interval of squared-Euclidean distances [4].

3. Result and discussion

3.1. Trace element concentrations

This study focused on frequent existing trace element in the study area namely Ag, Al, Cd, As, Co, Cr, Cu, Fe, Mn, Ni and Zn as shown in Table 2.

Table 2. Summary of element concentration in Johor river sediment (mg/kg).

	Ag	Al	As	Cd	Co	Cr	Cu	Fe	Mn	Ni	Zn
Min	0.35	505.86	BDL[e]	0.02	0.02	0.59	BDL[e]	247.07	0.71	1.08	10.87
Max	4.25	1864.59	5.37	0.07	0.17	2.05	5.38	1010.23	9.62	5.68	21.15
Mean	1.77	1119.49	0.96	0.05	0.08	1.09	3.07	505.19	3.03	1.94	14.71
Median	1.24	986.26	0.27	0.04	0.06	0.98	2.62	426.12	1.58	1.48	13.58
SD	1.39	489.76	1.80	0.02	0.05	0.45	1.27	261.19	2.86	1.44	3.65
ASV[a]	0.07	8.8	13	0.3	19	90	45	4.72	850	68	95
TEL[b]	NA[f]	NA[f]	5.9	0.6	NA[f]	37.3	35.7	NA[f]	NA[f]	36	123
PEL[c]	NA[f]	NA[f]	17	3.5	NA[f]	90	197	NA[f]	NA[f]	75	315
SEL[d]	NA[f]	NA[f]	33	10	NA[f]	110	110	NA[f]	NA[f]	16	820

[a] Average Shale Value [17].
[b] Threshold Effects Level [18].
[c] Probable Effects Level [18].
[d] Severe Effects Level [19].
[e] Below Detection Limit
[f] Not Available.

The order of mean trace element concentration in the sediment samples is Al > Fe > Zn > Cu > Mn > Ni > Ag > Cr > As > Co > Cd. Elements with the highest concentrations recorded in all sampling stations are Al and Fe. This may be due to the abundance of Al and Fe in the Earth's crust [21]. SS5 recorded the highest concentrations of most elements (Al, As, Cd, Co, Fe, Mn, and Ni). Since SS5 is located near the Kota Tinggi city, these elements may be derived from effluent discharges of urban and industrial origin. The concentrations of Ag and Cu was found to be higher in SS3. SS3 is dominated by *Nipah* mangrove which sediments can be a sink for various heavy elements [10]. The highest concentration of Zn was measured in SS1 where domestic and industrial wastewater could be the main sources of Zn [22]. Elements with the lowest concentrations are As, Cd, Co, and Cr in SS2; Al, Cu, Fe, Mn, and Ni in SS7; Ag in SS4; Zn in SS8. The concentrations of Ag, Al, and Fe in all sampling stations exceeded that of average shale values proposed by Turekian and Wadephol (1961) [16].

3.2. Evaluation of sediment contamination

Comparing with sediment quality guidelines (SQGs), the concentrations of the selected elements (As, Cd, Cr, Cu, Ni, and Zn) were below than the TEL, SEL, PEL values at all sampling stations. This showed that all these elements have no adverse effects on the freshwater species.

The results of PLI and RI were depicted in the Figure 2 and Figure 3, respectively. PLI values for all stations were less than 1 which indicate that Johor river sediments are free of trace element contamination. Figure 2 showed that the highest PL1 value was recorded at SS5 and lowest value at SS7. RI indicated that all stations are at low ecological risk as the RI values were less than 150 as shown in Figure 3. The results of Er values were summarized in Table 3. The order of E_r of trace elements in sediments of Johor river was Cd > As > Cu > Ni > Co > Zn > Cr > Mn. E_r results showed that the reported trace elements have low risk towards the freshwater environment. From the PLI, RI, and E_r results, it can be concluded that the Johor river sediments are free of trace elements pollution, however the continuous industrial and agricultural developments, and urbanisation may cause progressive increase in trace element pollution.

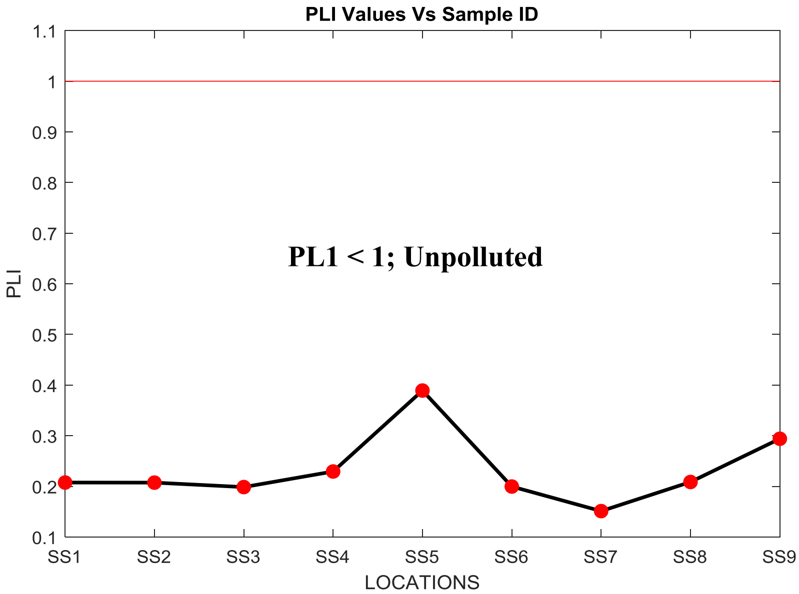

Figure 2. The line graph of pollution load index (PLI) at nine sampling stations.

Table 3. Evaluation of potential ecological risk factor (E_r) in Johor river sediments.

	As	Cd	Co	Cr	Cu	Mn	Ni	Zn	Risk grade
SS1	0.270	5.604	0.015	0.018	0.257	0.005	0.118	0.223	Low
SS2	-	2.473	0.006	0.013	0.212	0.001	0.103	0.209	Low
SS3	0.146	2.861	0.012	0.020	0.598	0.004	0.090	0.163	Low
SS4	0.619	4.358	0.027	0.027	0.400	0.005	0.085	0.159	Low
SS5	4.127	6.825	0.044	0.022	0.261	0.011	0.418	0.115	Low
SS6	0.481	5.010	0.018	0.022	0.321	0.002	0.144	0.131	Low
SS7	0.067	3.465	0.009	0.016	-	0.001	0.079	0.136	Low
SS8	0.079	4.409	0.016	0.045	-	0.001	0.138	0.114	Low
SS9	0.112	6.404	0.032	0.034	-	0.002	0.109	0.143	Low
Mean	1.767	7.141	0.518	0.222	1.007	0.103	0.628	0.239	Low

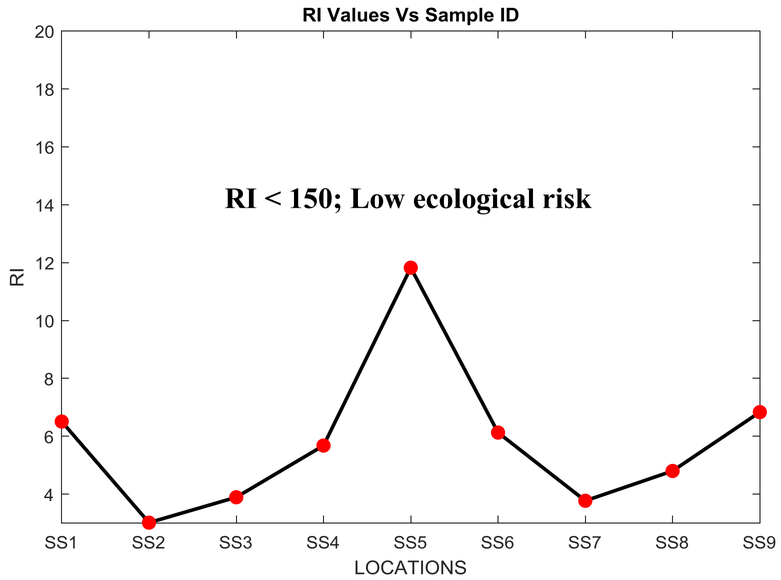

Figure 3. The line graph of potential ecological risk (RI) at nine sampling stations.

3.3. Statistical analysis and cluster analysis

Pearson's correlation coefficients among the trace elements were presented in Table 4. Pearson's correlation matrix was used in the present study to identify the interrelationship among the elements. The correlation analysis showed that a good positive correlation exits between Al and As ($r=0.546$, $p > 0.01$), Co ($r = 0.595$, $p > 0.01$), Fe ($r = 0.440$, $p > 0.01$), Mn ($r = 0.770$, $p > 0.01$) and Ni ($r = 0.496$, $p > 0.01$). This significant correlation with Al indicated that these elements may be originated from similar sources of pollution such weathering of rocks and lithogenic processes [23]. Co ($r = 0.629$, $p > 0.01$), Mn ($r = 0.749$, $p > 0.01$), and Ni ($r = 0.758$, $p > 0.01$) existed a good positive correlation with As, indicating that these elements tend to be accumulated together in the sediments. The positive correlations were also noticed between Cd and Co ($r = 0.701$, $p > 0.01$), between Co and Mn ($r = 0.670$, $p > 0.01$) and Ni ($r = 0.502$, $p > 0.01$), between Cr and Cu ($r = 0.609$, $p > 0.01$) , and between Mn and Ni ($r = 0.710$, $p > 0.01$). The positive correlation indicated by similar sources and redistribution of trace elements in the sediments by same physio-chemical processes [23].

Table 4. Pearson's correlation coefficient matrix.

	Ag	Al	As	Cd	Co	Cr	Cu	Fe	Mn	Ni	Zn
Ag	1.000										
Al	-0.181	1.000									
As	-0.202	**0.546**	1.000								
Cd	0.133	0.234	0.244	1.000							
Co	-0.092	**0.595**	**0.629**	**0.701**	1.000						
Cr	-0.001	0.015	-0.154	0.120	0.310	1.000					
Cu	0.172	-0.065	-0.219	0.047	0.075	**0.609**	1.000				
Fe	0.113	**0.440**	0.205	-0.013	0.293	0.185	-0.155	1.000			
Mn	-0.221	**0.770**	**0.749**	0.149	**0.670**	0.085	0.094	0.381	1.000		
Ni	-0.067	**0.496**	**0.758**	0.187	**0.502**	-0.030	0.055	0.397	**0.719**	1.000	
Zn	0.155	-0.082	-0.302	0.178	-0.104	-0.138	-0.065	0.101	-0.150	-0.037	1.000

The results of CA demonstrated two main clusters (Figure 4). Cluster 1 was mainly formed by Al, Mn, As, Ni, Fe, Cd, and Co, indicating that these elements could be originated from natural sources as Al and Fe are the most abundant elements in Earth's crust [21]. Cluster 2 was represented by Cr, Cu, Ag, and Zn. The elements in cluster 2 may be derived from wastewater discharges from urban, industrial and agricultural land uses.

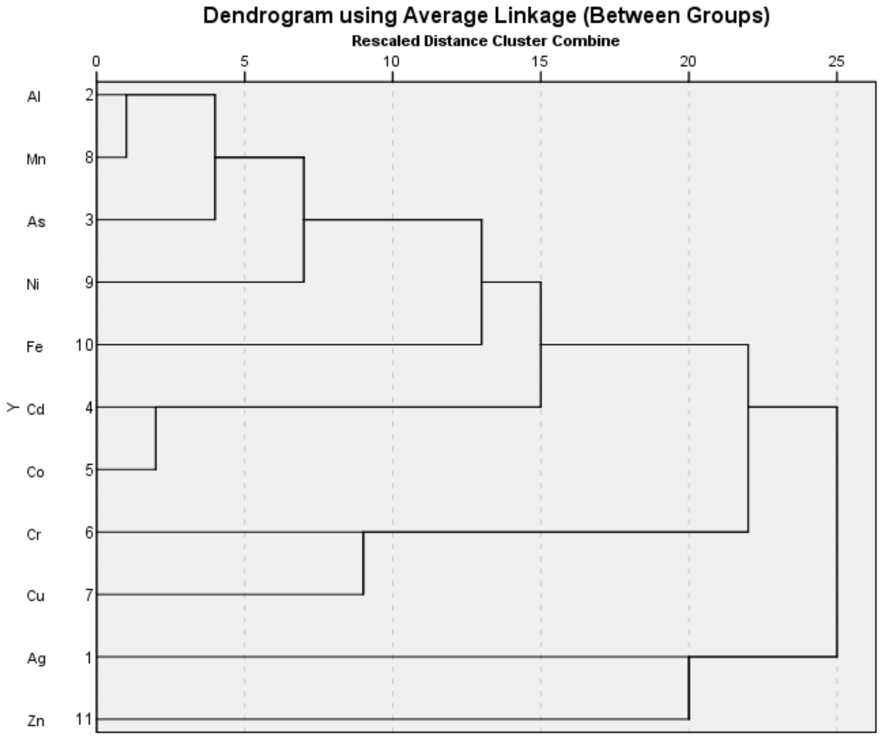

Figure 4. Dendrogram of cluster analysis for the trace elements in the sediments along Johor river

4. Conclusion

In this study, 11 elements (Ag, Al, As, Cd, Co, Cr, Cu, Fe, Mn, Ni, Zn) were analysed to assess the degree of trace element contamination in sediments of the Johor river. The decreasing order of trace elements concentrations in sediments is Al > Fe > Zn > Cu > Mn > Ni > Ag > Cr >As > Co > Cd. Based on comparison with sediment quality guidelines (SQGs), the concentrations of As, Cd, Cr, Cu, Ni, and Zn were below than TEL, PEL and SEL guideline values at all the sampling stations, indicating that these elements not likely to have adverse effects on aquatic biota. The results of PLI (ranged from 0.151-0.389, PLI < 1) showed that all the stations along the Johor river are uncontaminated by trace elements. RI values (ranged from 3.018-11.823, RI < 150) and E_r values (ranged from 0.101-7.141, E_r <140) indicated that trace metals present in concentrations that have no/little adverse effects on sediment-dwelling organisms. Pearson's correlation matrix showed a good positive correlation between Al and As (0.546), Co (0.595), Fe (0.440), Mn (0.770), and Ni (0.496), indicating these elements might be originated from similar sources of pollution. This correlation analysis was supported by cluster analysis (CA) which concluded that Al, Mn, As, Ni, Fe, Cd, and Co are mostly originated from natural processes and Cr, C, Ag, and Zn are mainly derived from anthropogenic sources. The overall results of the study demonstrated that Johor river sediments are free of element contamination, however continuous industry, agricultural, urban developments might increase the accumulation of trace elements in the sediments. This study recommended the application of multiple risk indexes with combination of statistical method for multiproxy water and sediment quality assessment and monitoring. This approach should be considered in preparing policies to ensure that all proposed economic development, expected to be environmentally damaging, is assessed prior to authorization and possible implementation.

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