Indian Journal of Geo-Marine Sciences
Vol. 45(8), August 2016, pp. 1017-1023

Heavy metal contamination and risk assessment in the riverine sediment

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Received 18 September *2014; revised 29 December 2014*

Intensity of pollution was evaluated using enrichment factor (EF), geo-accumulation index (Igeo), pollution index (I_{POLL}), m – ERM – Q and RF for (As, Cd, Cu, Mn, Ni, Pb and Zn) in sediments collected from Siahrud river, Iran. Chemical sequential extractions were employed for determination of the anthropogenic portions of metals. Heavy metal contents in the sediments follow the order of Al>Ca>Mn> Zn > Ni > Cu >Pb> As > Cd. Anthropogenic portion of the metals was determined through three-step chemical sequential extraction. The results of partition studies revealed the proportion of anthropogenic metals to their original bulk concentrations are Pb(85%) > Zn(84%) > Cd(62%) > As(47%) >Mn(35%) > Ni(34%) > Cu(2l %). Mean enrichment factor (EF) values obtained for various metals were between moderate enrichment and extremely high enrichment. Based on the classification of metals contamination, all sediment samples have a 74% probability of toxicity. Newly developed pollution index (RF) showes very good perfomance to determine the degree of contamination. Interestingly, the results of chemical partitioning tallies well with cluster analysis. Metals with highest anthropogenic portions are grouped together at a high similarity coefficient. Presence of organic matter in this group may be indicative of organic pollution source.

[**Keywords:** Anthropogenic metals; Sediment; Contamination; Aquatic; River]

Introduction

Heavy metal contamination in Marine ecosystems has increased[1-7]. Since sediments have been in contact for a long time with over lying water, therefore they have been able to absorb pollution on to their surface[8]. Studies show that marine sediments from coastal areas near centers of large industrial are contaminated by heavy metals and other pollutants[9,10]. Sediments are a sink for heavy metals, but when environmental conditions change sediments can act as a source for contaminants in aquatic environments[11].Estuarine sediment is recognized as a major source of ecosystem health stress. Thus, assessment of sediment contamination in estuaries is important problem[12,13]. Heavy metals distribution between water and sediments depends on different factors such as the nature of sediment particles and the environmental condition[14,15].

In recent years different environmental quality indicators and metal assessment indices applied to aquatic environments have been developed[16]. Metal assessment indices can be classified in three types: (1) contamination indices: which compare the contaminants with clean and/or polluted stations measured in the study area[17]; (2) enrichment indices: which compare the results of the contaminants with different baseline or background levels[18]; and (3) ecological risk indices: which compare the results for the contaminants with Sediment Quality Guidelines[19,20]. Heavy metals are present in different soluble fractions in sediments and speciation of particulate metals have different impacts on the environment[21,22].Therefore, researchers have recently followed different sequential digestion and chemical partitioning methods to evaluate the fractionation of metals in sediments[23].

Present investigation was carried out in Siahrudriver (36°26.855' N, 52°56.708' E), that is located in southeast of the Caspian Sea basin, north of Iran. The aim of this study was to determine sediment contamination and ecological risk assessments in riverine ecosystem. Subsequently, various indices were used to show the intensity of pollution in this area. Also, the chemical partitioning of these elements makes it possible to know the mobility of heavy metals in the sediments of Siahrudriver.

Materials and Methods

In the present study, the surficial sediments of Siahrudriverwere collected by a Peterson grab at 10 sampling stations. The area of Siahrud with an area of over 10,070 hectares is located in Mazandaran province in Qaemshahr city in the north of Iran. The length of this river is 51km. Sampling stations were selected using a land use map to incorporate various activities (Fig. 1). Locations of sampling stations in

Siahrud riveralong with the water depths and distances is given in Table 1.

The collected sediment samples were sealed plastic bags and stored at 4°c until their arrival at the laboratory. Grain size fractions less than 63 µm were chosen for chemical analysis.

Total metal contents were determined by digesting the samples with a mixture of HNO3& HClO4[24].The concentration of heavy metals (As, Cd, Cu, Mn, Ni, Pb and Zn) in sediment samples were determined by inductively coupled plasma atomic emission spectrometry (ICP-AES).Loss on Ignition (LOI) were determined by combusting the samples for 4 hours at 450°C in a muffle furnace.To obtain bio accessibility values,a solution of pH=5 was prepared using NaOH and HOAc.The chemical partitioning of metals were carried out in three sequential steps: Fraction II (reducible): acetic acid 25% v/v–0.1 M hydroxylamine hydrochloride, pH & 2, occasional agitation.

Table 1— General features and description of sampling sites of Siahrud river

Position	St. No.	N	E	Depth (cm)	(m) From Upstream
Upstream	St1	53°1′	36°27′	70	3.5
	St2	52°59′	36°27′	60	7
	St3	52°54′	36°27′	50	19
	St4	52°54′	36°28′	90	26
	St5	52°54′	36°29′	50	27
	St6	52°54′	36°32′	80	32
	St7	52°55′	36°32′	70	36
	St8	52°56′	36°37′	70	41
	St9	52°56′	36°43′	100	67
Downstream	St10	52°58′	36°46′	200	76

Fraction III (oxidizable): 30% H2O2 ‘‘extraction with 1 M ammonium acetate’’, pH & 2, 85 ± 2_C, 3 h, intermittent agitation.

Furthermore, fourth fraction (within lattice) was determined by subtracting total metal content from the sum of the contents in the three previous fractions[22,23,25].

The accuracy of the Chemical Analysis weredetermined using CRM 320 (sediment reference material). Replicate analysis of this CRM showed good accuracy, with recovery rates for heavy metals between 97 and 101%. A standard reference material (CRM-60l) was used to verify the accuracy of the chemical partitioning method.

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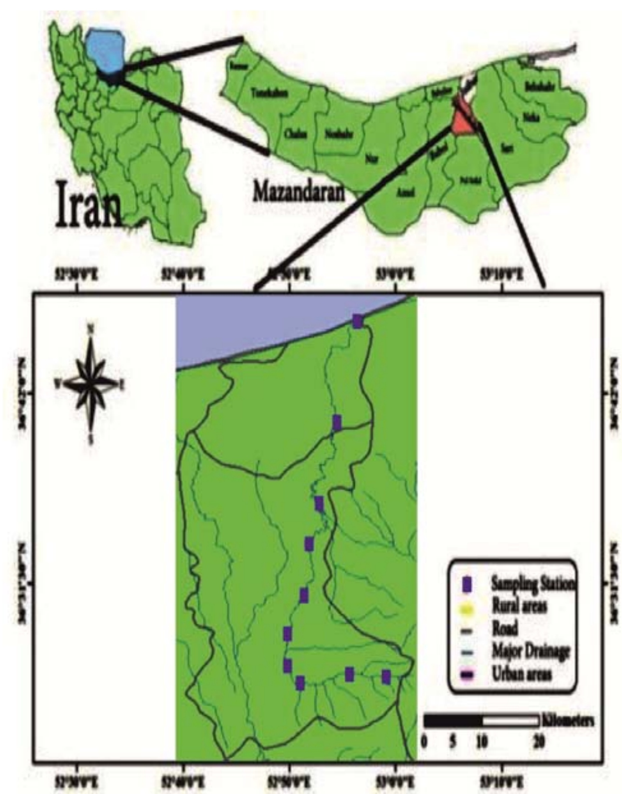

Fig. 1— Location of the study area showing sampling points

Results and Discussion

The concentration of studied metals along with mean earth crust values are given in Table 2.

As shown in table 2, there are a significant difference between mean concentration of Al and the correspondent earth crust value. Concentration of Al decreases from upstream to downstream that may be indicative of increase of non-lithogenous materials. Results revealed that the concentration of Ni ranges from 522 to 1470 mg/kg with a mean value of 886 mg/kg. The higher concentration of Ni could be attributed to the input of oil materials into the river.

Table 2— Elemental concentration of surficial sediments of Siahrud river

St. No.	As	Cd	Cu	Mn	Ni	Pb	Zn	Ca	Al	OM
				mg/kg					%	
1	112	5	56	362	611	36	132	2.1	2.2	4.2
2	117	7	57	370	670	37	125	2.2	2.2	4.7
3	170	8	52	430	770	40	130	2.0	2.1	7.2
4	160	11	60	440	917	150	520	1.8	1.9	7.9
5	230	10	62	430	913	175	912	1.6	1.8	9.3
6	310	10	66	475	850	160	720	1.6	1.7	9.9
7	320	9	71	520	522	110	510	1.5	1.7	12.3
8	330	9	58	520	1100	132	1100	1.7	1.6	13.2
9	320	8	44	493	1470	87	987	1.6	1.7	10.6
10	175	9	48	512	1040	67	188	2.9	1.9	8.9
Min	112	5	44	362	522	36	125	1.5	1.6	4.2
Max	330	11	71	520	1470	175	1100	2.9	2.2	13.2
Mean	224	9	57	455	886	99	532	1.9	1.9	8.9
SD	88	1.7	8	59	275	54	382	0.4	0.2	2.9
earth crust	13	0.3	50	950	80	14	75	4.1	8.2	-

Also the higher concentration of Pb and Zn might be due to the discharge of textile industry that is located by the side of river. Higher concentration of arsenic is due to coal mining as well as forest area that are located upstream. Maximum concentration of Ca was found at the station 10(almost in estuarine zone). Since estuarine zones are biologically more active than rivers, this may justify for higher Ca contents at station 10. The concentration of Cd ranges from 5 to 11 mg/kg with a mean value of 9mg/kg. Also, the organic contents ranges from 4.2% to 13.2% with a mean value of 8.9%.

The association of heavy metals with different sedimentary phases has been assessed by chemical partitioning technique.

Results of chemical partitioning studies is presented in Table 3.

Percentiles of anthropogenic portion of heavy metal contentswhen compared to bulk concentrations in Siahrud river sedimentsshows the following pattern:

Pb(85%) > Zn(84%) > Cd(62%) > As(47%) > Mn(35%) > Ni(34%) > Cu(21 %).

Results of chemical partitioning of studied heavy metals show that all of them are to various degrees originated from anthropogenic sources.

Metal enrichment factor (EF) was used to determine the degree of contamination in each sediment sample[26]. The cumulative enrichment factor of all elements can be calculated using the following equation[27].

$$EF = ([(M_c)/(M_r)]_s)/([(M_c)/(M_r)]_b) \qquad (1)$$

Table 3— Specification of heavy metals inSiahrud river sediments

metals	Stations	Bioavailable	Fractional Steps			Anthropogenic portion(%)	lithogenous portion(%)
			step1	step2	step3		
Ni(mg/kg)	Mean	30	300	16	21	34	66
Mn(mg/kg)	Mean	121	153	7	5	35	65
Pb(mg/kg)	Mean	60	73	11	4	85	15
Cd(mg/kg)	Mean	4	5	0	0	62	38
Cu(mg/kg)	Mean	4	5	3	4	21	79
Zn(mg/kg)	Mean	355	444	16	28	84	16
As(mg/kg)	Mean	27	33	8	77	47	53

Table 4— Categories of sediment pollution based on the

calculated indexes	Ranges of Indexes	State of pollution
I_{geo} and I_{POLL}	I≥5	Extremely polluted
	4≤I<5	Highly–extremely polluted
	3≤I<4	Highly polluted
	2≤I<3	Moderately–highly polluted
	1≤I<2	Moderately polluted
	0≤I<1	moderately–Unpolluted
	I≤0	Unpolluted
EF	EF≤1	No enrichment
	1<EF≤3	Minor enrichment
	3<EF≤5	Moderate enrichment
	5<EF≤10	Moderately sever enrichment
	10<EF≤25	Sever enrichment
	25<EF≤50	Very sever enrichment
	EF>50	Extremely sever enrichment
m – ERM – Q	< 0.1	12% probability of toxicity
	0.11–0.5	30% probability of toxicity
	0.51–1.5	40% probability of toxicity
	>1.5	74% probability of toxicity
RF	RF<1	Low contamination
	1≤RF<3	Moderate contamination
	3≤RF<6	Considerable contamination
	6≤RF	Very high contamination

Where M_c is the concentration of metals, M_r is the concentration of reference elements, s is the studied sample, and b indicates the background. This method uses a normalization element such as Al or Fe[28]. Five contamination categorizes are recognized based on EF value[29,30](Table 4).

In order to evaluate the degree of contamination in each sediment sample, geo-accumulation index value was calculated using the following equation:

$$I_{geo} = \log_2\left(\frac{C_n}{1.5B_n}\right) \quad (2)$$

Where Cn is the content of metals in sediment samples, and Bn is the shale value for each element[28]. Muller's formula was modified by Karbassi et al. (2008) as follows:

$$I_{POLL} = \log_2\left(\frac{B_n}{L_p}\right) \quad (3)$$

Where Bn and Lp represent bulk concentration and lithogenous portions respectively.

In our study, chemical partitioning results are substituted for the mean crust and shale levels in the new pollution index ($RI_{Aquatic}$) [25].

The risk factor (RF), obtained by dividing the anthropogenic portion of metals concentration in the sediment sample by lithogenous portion of metals concentration in the sediment:

$$RF = \frac{A_p}{L_p} \quad (4)$$

The aquatic risk index ($RI_{Aquatic}$) can be defined as follows:

$$RI_{Aquatic} = \frac{\sum_{i=1}^{n} R_f^i}{n} \quad (5)$$

Where $RI_{Aquatic}$ is the aquatic risk index while RF and n are the risk factor of metals and the count of the heavy metal species, respectively. Contamination categories based Igeo and I_{POLL} and EF values are showen in (Table 4) [25].

The obtained mean enrichment factor (EF) values for various metals were between moderate enrichment and extremely high enrichment. Maximum mean EF value belongs toCd (Cd=128.1) indicating extremely high enrichment, and also the minimum mean EF value is seen for Ca (Ca=2.0) showing moderate enrichment (Table 5).

Table 5— Enrichment factors (EF) of metals for samples

St. No.	As	Cd	Cu	Mn	Ni	Pb	Zn	Ca
1	32.1	62.1	4.2	1.4	28.5	9.6	6.6	1.9
2	33.5	87	4.2	1.5	31.2	9.9	6.2	2
3	51.1	104.1	4.1	1.8	37.6	11.2	6.8	1.9
4	53.1	158.2	5.2	2	49.5	46.2	29.9	1.9
5	80.6	151.9	5.6	2.1	52	56.9	55.4	1.8
6	115	160.8	6.4	2.4	51.3	55.1	46.3	1.9
7	118.7	144.7	6.8	2.6	31.5	37.9	32.8	1.8
8	130.1	153.8	5.9	2.8	70.5	48.3	75.2	2.1
9	118.7	128.6	4.2	2.5	88.6	30	63.5	1.9
10	58.1	129.5	4.1	2.3	56.1	20.7	10.8	3.1
Mean EF	79.1	128.1	5.1	2.1	49.7	32.6	33.3	2

The degree of sediment contamination in Siahrud river was calculated using Muller's index (Table 6). Newly developed pollution index developed by Vaezi et al. (2015) was used to determine the degree of contamination (Table 7).

Table 6— Geo-accumulation Index of metals for samples

Elements	As	Cd	Cu	Mn	Ni	Pb	Zn
Mean	3.2	4.2	0	0	2.7	2.2	1.9

The values obtained from RF index are indicative of a broad range (from low to considerable contamination) for various studied elements. Based on I_{POLL} Pollution intensity of Zn and Pb in the sediments of Siahrudriver is 3.57 and 3.15, respectively that are indicative of high pollution intensity. Other studied metals fall within "no pollution" to "low pollution" intensities.

In order to consider the possible toxicity effects of the combined toxicants groups in different concentration, mean SQG quotients were calculated as the average of ratio between the samples and correspondent effective range median values (ERM) as follows[31,32]:

$$m - ERM - Q = \sum \frac{c_i / ERM_i}{n} \quad (6)$$

Where ci is the sediment concentration of compound i, ERMi is the respective Effect Range Median for compound i and n is the number of compound i.

The classification of toxicity probability of sediment samples according to mean ERM are shown in Table 4:

Based on the classification of metals contamination, all sediment samples have a 74% probability of toxicity (Table 8).

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The result of cluster analysis (CA) is shown in the form of a dendrogram, which indicates the degree of similarity amongst compounds[33,34,35].Similarities and significant relationshipsbetween the behavior and origin of the metals were established by cluster analysis using the weighted pair group method.

Dendrogram resulting from UPGMA analysis showed 3 major clusters (Fig. 2). Result of cluster analysis for Cd and Pb shows that these two elements are related together by high similarity coefficient. As LOI concentration in the environment is the index of organic source (both natural and anthropogenic ones) then As, Mn and Znalong with Pb and Cd might be partially derived/associated with organics.

Table 7— Anthropogenic and natural portions of metals and I_{POLL} and RF of surficial sediments

Elements	As	Cd	Cu	Mn	Ni	Pb	Zn
Mean concentration (mg/kg)	224	9	57	455	886	99	532
Anthropogenic portion %	47	62	21	35	34	85	84
lithogenous portion %	53	38	79	65	66	15	16
I_{POLL}	1.07	1.43	0.34	0.65	0.69	3.15	3.57
RF	0.89	1.63	0.27	0.54	0.52	5.67	5.25

Table 8— ERM quotient of the sediment samples of Siahrudriver

Station number	Ci / ERMi						m – ERM – Q
	AS	Cd	Cu	Ni	Pb	Zn	
1	1.6	0.5	0.2	11.8	0.2	0.3	2.4
2	1.7	0.7	0.2	12.9	0.2	0.3	2.7
3	2.4	0.8	0.2	14.8	0.2	0.3	3.1
4	2.3	1.1	0.2	17.6	0.7	1.3	3.9
5	3.3	1.0	0.2	17.6	0.8	2.2	4.2
6	4.4	1.0	0.2	16.3	0.7	1.8	4.1
7	4.6	0.9	0.3	10.0	0.5	1.2	2.9
8	4.7	0.9	0.2	21.2	0.6	2.7	5.1
9	4.6	0.8	0.2	28.3	0.4	2.4	6.1
10	2.5	0.9	0.2	20.0	0.3	0.5	4.1
ERL	8.2	1.2	34	21	47	150	-
ERM	70	9.6	270	52	220	410	-

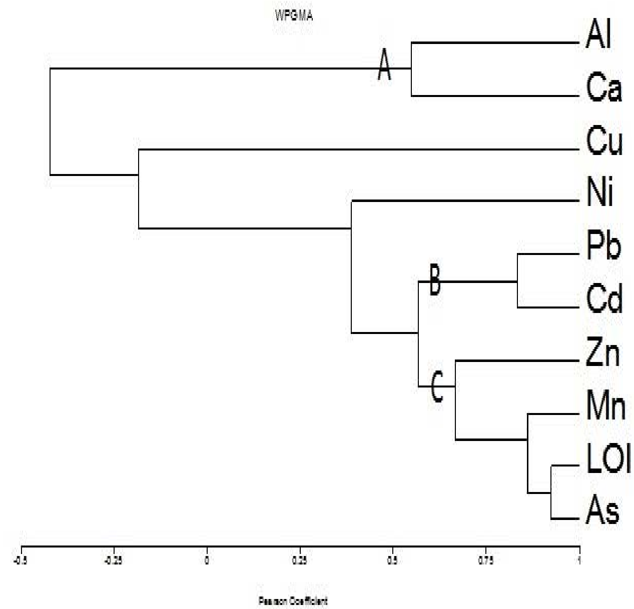

Fig. 2— Dendrogram showing clustering of metals and OM

Conclusion

In general, heavy metal concentration in the sediments are indicative of high pollution intensity of Ni, Mn, Pb, Cd, Cu, Zn and As in comparison with mean earth crust values. Also, the results of chemical partitioning studies revealed that Ni, Mn, Pb, Cd, Cu and Zn are derived from anthropogenic source to various degrees. Anthropogenic portion of heavy metal contents in Siahrud river sediments shows the following pattern: Pb(85%) > Zn(84%) > Cd(62%) > As(47%) >Mn(35%) > Ni(34%) > Cu(21 %).

The obtained mean enrichment factor (EF) values for various metals were between moderate enrichment and extremely high enrichment. Maximum mean

EF value belongs to Cd (Cd=128.1) indicating extremely high enrichment, and the minimum mean EF value is seen for Ca (Ca=2.0) showing moderate enrichment. The values obtained from Igeo and I_{POLL} and RF indices are indicative of a broad range (from unpolluted to strongly polluted) for various studied elements. Based on the classification of metals contamination, all sediment samples have a 74% probability of toxicity. Result of cluster analysis is in good agreement with chemical partitioning data in showing organic source of pollution. Higher concentrations of heavy metals alone cannot be indicative of anthropogenic sources in the specific area. Pollution index (I_{POLL}) and RF use background levels of heavy metals that are obtained from chemical studies. Though these index is rather superior to the previousindices that use shale or mean crust values, but the presence of pollution may not all alone be considered as a real threat to the environment. We propose researchers to focus on the bio-availability and bio-accessibility of trace metals. Such values should be used to revise the pollution intensity indices.

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