# **Geochemical rorms and risk assessment of some heavy metals in Aden coasts sediments, Yemen**

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# **Abstract**

 The objective of the present study is to investigate risk assessment and geochemical forms of Pb, Mn, Zn and Cu in Aden Coasts sediments as well as the content of sediment texture, organic carbon, and carbonates. Sequential extraction technique was applied to estimate the metals in five different fractions; exchangeable, bound to carbonate, bound to oxides, bound to organic matter and residual. The results indicated that the metals bound to non-residual fraction (anthropogenic input) represent 72.8 % of the total extractable fractions for Pb, 79.7 % for Mn, 87.4 % for Zn, and 87.3 % for Cu. The five fractions of the metals in the present study could be arranged in the following sequence:

 Residual > Exchangeable > Oxide form > Organic form > Carbonate for Pb; Residual > Oxides form > Organic form > Carbonate > Exchangeable for Mn or Zn; Organic form > Residual > Oxide form > Carbonate > Exchangeable for Cu.

The risk assessment of the metals showed medium risk for Pb at all sampling sites except Sira zone which revealed very high risk to aquatic environment. Fractionation pattern of Zn and Cu showed low risk, while Mn showed low (most sites) to medium risk (Sira and Caltex) to aquatic environment. In general, the risk assessment of the metals in the study area decreased as follows: Pb >Mn>Zn> Cu.

**Key words:** Sequential extraction; Heavy metals; Geochemical; Risk assessment; Aden coasts

#### **Introduction**

 It is now widely accepted that the role of aquatic sediments as a sink or as a source of pollutants cannot fully be assessed by measuring the total metal concentrations. In addition, determination of the total elements does not give an accurate estimate of the likely environmental impact. Instead, it is desirable to have information on the potential availability of metals (whether toxic or essential) to biota under various environmental conditions. Since the mobility of trace metals, as well as their bioavailability and related ecotoxicity to plants, critically depends upon the chemical form in which a metal is present in the sediment, a considerable interest exists in trace element speciation  $[2 \& 6]$ .

 Chemical speciation can be defined as the identification and quantification of the different species, forms or phases present in a material, or the description of these. However, the determination of specific chemical species or binding forms is difficult and often hardly possible [10]. Therefore, determinations of broader forms, e.g. so-called mobile or carbonate bound forms, depending on operationally defined procedures, can be a good compromise to give information on environmental contamination risks. As a result of this practicality, single and sequential extraction schemes have been designed for the determination of binding forms of trace metals in sediments and increasingly used over the last 10 years [6].

 A large number of sequential extraction methods have been reported, many of which are variants on the Tessier procedure [19] in which the exchangeable metals and those nominally associated with carbonate, Fe–Mn oxides, organic material and silicate residues were extracted with different reagents [18]. Although the reagents used in sequential extraction procedures may be insufficiently specific to dissolve exclusively the ''target'' phases, and results obtained can vary widely when

**Geochemical rorms and risk assessment** ……Shaif M. K.S., Samir M. Nasr, Mohamed A. Okbah different extraction schemes and experimental conditions are used, useful information has been gained from such studies [4]. To assess the environmental impact of sediments pollution, chemical partitioning of trace metals, among the various geochemical phases, is more useful than the measurement of the total content. The metal speciation will give more information about the potential release of contaminants, migration and toxicity of the metals as well as the risk assessment of these metals [20].

 The main objectives of the current study are: (1) Identification of geochemical form of Pb, Mn, Zn and Cu sediment samples collected from Aden coasts applying sequential extraction technique, (2) determination of sediment texture, organic carbon, and carbonates, (3) to estimate the potential availability of metals contained in grain size fractions of the sediments, by using Risk Assessment Code (RAC) of the metals in the sediments to give a clear indication of sediment reactivity, which assesses the risk connected with the presence of metals in aquatic environment.

# **Materials and Methods**

# **1. Study Area: Description of the study area and Sampling Properties**

 Geographically, Aden is located at the southern part of Yemeni coast in Gulf of Aden with the coordination  $12^{\circ}$  28' -  $12^{\circ}$  57' N and  $44^{\circ}$  27' -  $45^{\circ}$  07' E (Table 1 & Figure 1). It is located at the south west tip of Yemen and the Arab peninsula. Mid-way between Europe and Far East, Aden lies on major world trading route through the Suez Canal. It is one of the largest natural harbors in the world with an area of about 70 km<sup>2</sup> of sheltered water surrounded by Jebel Shamsan, khoremakser, and the shore which extends to the hills of Little Aden. Aden has a series of communities stretching around a well protected bay; these include Crater, Ma'alla, Tawahi, Khormaksar, Shikh-Othman, Al-Mansura, Dar Saad, and Little Aden. The port of Aden consists of the inner harbor, the oil harbor serving Aden refinery and the anchorage and approach channels of the outer harbor.



**Figure 1: Sampling Sites of Study area**

<b>Zones</b>	<b>Sites</b>	<b>Location of Sites</b>		Location	<b>Properties</b>
		Latitude	Longitude		
	1	12°45'04"N	44°49′37″	Fuqum	Sewage effluents (near from Bab
I			E		Al-Mandab)
	$\overline{c}$	12°45'06"N	44°53'23"E	Little Aden	Refinery plant, (discharging)
	$\overline{3}$	$12^{\circ}45'40''N$	44°53'53"E	Little Aden	wastes: industrial and domestic)
	$\overline{4}$	$12^{\circ}46'01''N$	44°53′32″	Khawr Bir	Industrial facilities (near from oil
			E	Ahmed	harbor)
	5	12°49'32"N	44°56'25"E	Al Hiswah	desalination Power and plants,
$\mathbf{I}$	6	12°44'48"N	44°55'44"E	<b>Bandar Tawahi</b>	runoff
	7	$12^{\circ}50'51''N$	44°59'37"E	Caltex (surface)	Oil spill from the oil pipes
	$\overline{8}$	$12^{\circ}48'15''N$	45°01'32"E	Labour Island	
III	9	12°49'19"N	$45^{\circ}$	Lab. Island	used the store old and damaged
			00'43"E		ships and boats
	10	12°48'01"N	$45^{\circ}$	Lab. Island	
			00'43"E		
	11	$12^{\circ}48'10''N$	44°59'56"E	Malla Harbor	
	12	12°48'01"N	45°00'34"E	Malla Harbor	Industrial and municipal waste
	13	12°48'10"N	45°00'27"E	Terminal	water
				Container	$^{\prime\prime}$
	14	12°48'00"N	44°59'40"E	Terminal	$^{\prime\prime}$
				Container	$^{\prime\prime}$
	15	12°47'24"N	45°00'00"E	<b>Fishing Harbor</b>	$^{\dagger}$
	16	12°47'29"N	44°58'57"E	Tawahi	
IV	17	12°46'04"N	44°59'22"E	Cornish Q.	
				Mohur	Area open used Tourism Potential
	18	12°45'39"N	44°59'54"E	Cornish Q.	
				Mohur	
	19	$12^{\circ}45'25''N$	45°00'15"E	Dunafah Island	
$\mathbf{V}$	20	12°46'39"N	45°02'48"E	Sira Island	Sewage effluents
	21	12°49'55"N	45°02'57"E	Sahel Abyan	11

**Table 1: Description of the Study Area**

# **2. Sample Collection:**

 Twenty one sediment samples were collected from different selected sites from Aden coasts, Yemen, during 2004 (Table 1 & Figure 1). The study area was divided into five zones as follows: zone I located in western area of the port and representing four sites (site 1 to site 4); zone II located in the north of the port (polluted area, industrial waste) from site 5 to site 7; Zone III is the higher polluted area, more human activity, representing nine sites from site 8 to site 16, zone IV representing three sites (site 17 to site 19), in additions two sites 20 and 21, and Zone V located in the eastern area of the port. The sediments were collected using Grab collector placed into sealed polyethylene bags, carried to the laboratory in an ice box and stored at 4 C˚ in the dark until analysis.

# **3. Quality Control Studies:**

 Quality control studies for the total concentrations of metals were done using a reference material (HISS-1, National Research of Council of Canada). It was analyzed in duplicate in six batches; the accuracy of the results ranged between 90-110%, while precision was agreed within 10%. Table (2) illustrates the concentrations of different metals in the reference materials. The variation coefficients were 1.3-6 % for Fe; 2.5-8.2 % for Mn; 2.6-8.9 % for Zn; 2.8-11.3 % for Cu; **Geochemical rorms and risk assessment** ……Shaif M. K.S., Samir M. Nasr, Mohamed A. Okbah 3.1-8.7 % for Pb; 2.4-9.4 for Cr; 3.9-9.3 % for Co and ND-6.3 % for Ni. The comparison of the sum of the fractions with the results from the total concentration (a separate strong acid digestion) was performed to study the quality control for sequential extractions procedure; the percentage recovery of the metals was resulted to be within  $\pm 20$  %.



#### **Table 2: The concentrations for different metals (mg/kg) in the reference materials (HISS-1, National Research of Council of Canada)**

# **4. Determination of Carbonate, Organic Carbon and Organic Matter:**

Sediment samples were prepared for grain size analysis by air drying. Samples were then homogenized smoothly, weighed, and placed in glass jars. Each individual sample was treated separately for grain size distribution. It was determined by wet sieving for sand fraction and by pipette technique for silt and clay fractions [9]. Total carbonate  $(TCO<sub>3</sub>)$  content was determined, using the method described by Dean [3]. Organic carbon (OC) and total organic matter (OM) of the investigated sediments were determined, using wet oxidation method as mentioned by El-Wakeel and Riely [5].

# **5. Sequential Extraction Procedure:**

 For the estimation of some heavy metals, using the sequential extraction method, 12 selected samples were used from sediments of Aden coasts (see Table 1). The metal speciation in the sediment was done by sequential extraction scheme proposed by Tessier *et al.,* [19]. The procedure (Figure 2) is one of the most thoroughly researched and widely used procedures to evaluate the possible chemical associations of metals in sediments and soils. The residual fraction was determined after being digested with acid mixture  $(HNO<sub>3</sub>, HClO<sub>4</sub>, HF)$  ratio 3:2:1) [13].

# **Results and Discussions**

 The study highlights at the content of Pb Mn, Zn and Cu in the sediments from Aden Coasts. Heavy metal distributions in marine deposits are influenced by sediment texture, organic carbon, and carbonates [16].

# **1. The Physical and Chemical Characteristics:**

 The particle size distribution, carbonate, and organic carbon contents in the sediments of the study area are given in Table (3) and Figures (3-7).

# **1.1. Grain Size Distribution:**

 Grain size distribution was performed on 21 sediments collected from Aden Coasts. The textures of sediments have been shown in Table (3) and Figures (3-5).

The investigated sediments can be classified into three types; the first type, which is characterized **by sandy clay loam**, the presented clay more than 20%, observed at zone IV, the second type of the sediments is characterized **by sandy loam**, the percent of clay is lower than 20% observed at zones (II, III, V), and the third type of the sediments is characterized **by loamy sand**; the percent of sand in the sediment is more than 80%. The relative percentage of clay, silt and sand in the Aden Coast sediments were in the range (15-86%) for sand, (1-62%) for silt and (0-25%) for clay (see Figurers 3-5).

# **1.2. Organic Carbon and Organic Matter in Sediments:**

 The organic carbon and organic matter content of the sediment was of the order of the range of 0.32-2.97% and 0.66-5.04 %, respectively. The high values of OC and OM content at site 15 (Fishing harbor) is due to discharge of sewage and human activities from surrounding areas, while the minimum concentration value was found at site 19 (Dunafah island) (Table 3 and Figure 6).

#### **Geochemical rorms and risk assessment** ……Shaif M. K.S., Samir M. Nasr, Mohamed A. Okbah

# **1.3. Total Carbonate:**

 The concentration of total carbonate in the sediment samples of the investigated area is ranged between 5.67% at site 12 (Sahel Abyan) and 78.67% at site 10 (Dunafah island) (Table 3 and Figure 7). Okbah *et al.,* [12] showed that the mineralogical analysis of Red Sea sediments are well crystallized mineral matrices. They found that carbonate minerals were represented by calcite and Mg-Calcite. The silicates were included quartz and feldspars and koalinit was, also, detected at the sediments.

#### **2. Geochemical Forms of Heavy Metals:**

 The results application of sequential extraction of heavy metals (Pb Mn, Zn, Cu) in five different chemical phases (exchangeable F1, bound to carbonate F2, bound to Fe-Mn oxides F3, bound to organic matter F4 and residual F5) are presented in Figure (8) and Tables (4 to 7).

#### **2.1. Geochemical of Lead (Pb):**

 Lead in the environment may be derived from either natural or anthropogenic sources. Natural sources of atmospheric lead include geological weathering and volcanic emissions and emission from sea spray. Lead and its compounds may enter the environment at any point from the inputs of industrial effluents, sewage sludge, domestic wastes, pigments, petrol (gasoline) additives, steel products and the combustion of fossil fuels [14].

 The data obtained in the present study of Lead speciation revealed that the Pb concentration in the residual fraction represents from 38 % (35.92  $\mu$ g/g) at zone III to 71 % (83.88  $\mu$ g/g) at zone IV (Figure 8 and Table 4). This metal was mostly concentrated at the residual fraction at sites (1, 4 and 19) about (71%), while at sites (14,15,20) the relative percentage values in this fraction were 27- 34%. It is assumed that Pb in these sediments (Main Port and Sira Island) is a great extent derived from anthropogenic inputs rather than geochemical background. The next important phase of Pb was the non-residual fraction (labile form). The large amount of Pb was recorded in the exchangeable fraction. The study showed that the Pb concentration in the exchangeable fraction was very high, compared with those reported in the other metals, this fraction represents from 10 % to 26% of the total Pb concentration. The metal is present in the exchangeable and carbonate fractions are considered to be weekly bound and may equilibrate with the aqueous phase, thus becoming more bioavailable [7]. This result indicates that an abundant amount of Pb discharging into this sea water was associated with the sediment matrices.

 The data obtained about the fracture of lead (Pb) revealed that they were extracted similar in exchangeable fraction in the all sites (10-13%), except zone V (26%). This indicates that lead is more distributed in the study area. Measurable concentration of Pb was obtained in the exchangeable fraction (F1) of the sequential extraction procedure for all samples indicating that lead is anthropogenically source into sediments.

 The binding behavior of Pb bound to the sediment fractions showed high positive correlation of Pb concentration in the carbonate phase with Zn and Cu, and significant relationships between Pb bound to oxide with Zn-oxide; while the Pb bound to organic form was correlated with Mn, Zn and Cu (Figure 9).

#### **2.2. Geochemical of Manganese (Mn):**

 This metal (**Mn**) was mostly concentrated at the residual fraction more than (60%), while at sites 7, 15, 20 and 21 the relative percentage values in this fraction were lower than 55% (Table 5 and Figure 8). It is assumed that Mn in these sediments are to a great extent derived from natural sources rather than man-mode sources. The next important phase of Mn was the soluble fraction.

 The results indicate that the Mn concentration associated with residual fraction (73%) (natural sources) is much higher than bound to non-residual fraction (man-made sources) in the sediments from Aden coasts. Caltex and mine port showed high content, they may be due to the anthropogenic origin. The metal presented in the exchangeable and carbonate fractions are considered to be weekly bound and may equilibrate with the aqueous phase, thus becoming more bioavailable [7]. Caplat *et al.,* [1] found that manganese is bound to the non residual and the **Geochemical rorms and risk assessment** ……Shaif M. K.S., Samir M. Nasr, Mohamed A. Okbah residual fractions of the sediment. This element is mainly present in the acid-soluble fraction (carbonate form) and shows significant affinity for the residual fraction (up to 50%).

The binding behavior of Mn bound to the sediment fractions showed a high positive correlation of Mn in the organic form with Zn, Cu and Pb in the same fraction  $(r = 0.92, 0.86, \text{ and } 0.85, \text{)}$ respectively) (Figure 9).

# **2.3. Geochemical of Zinc (Zn):**

 The low concentration of Zn was obtained in the exchangeable fraction (F1) for all samples (except sites 14 and 15) indicating that zinc is not anthropogenically source into the sediments, but the relatively high concentration in sites 14 and 15 at zone III (main port) can be an indicator for that. The most of zinc content was recorded in the residual form in the five zones (52-95%) (Table 6 and Figure 8). The percentage of Zn associated with the residual fractions is in the order: zone IV (95% or155.15 μg/g) > zone V (77.9% or 62.30 μg/g) > zone I (70 % or 29.89 μg/g) > zone II (55% or 22.88  $\mu$ g/g) > zone III (52 % or 99.18  $\mu$ g/g). The result indicates that the more polluted area with Zn in the study area were recorded at zone II (Al-Hiswah and Caltex) and zone III (main harbor), they may be possible due to the disposal of untreated sewage wastes from domestic drainage of city, harbor activity, as well as power and electrical site.

# **2.4. Geochemical of Copper (Cu):**

 The data obtained revealed that the high concentration of copper associated with organic fraction was recorded at zone III (38.62  $\mu$ g/g or 77.7% of the total copper) (Table 7 and Figure 8), this area is characterized by relatively high organic matter percentage about 3 %, probably due to the intervention of the sources of pollution and informal settlements  $[8 \& 11]$ . This indicates that Cu occurrs in the forms of stable organic complexes and metal sulfides. This behavior can be explained by the well-known high affinity of Cu to humic substances, which are chemically very active in complexion with such metal [6]. On the other hand, most of the Cu was presented in the residual fraction (60–72%) in all zones except zone III (17%). The high Cu is content associated with the residual fraction while thelow concentration in the exchangeable (ND-0.24%) as well as carbonate fractions (2.25-6.2%) indicated the low bioavailability of Cu in Gulf of Aden.

# **3. Risk Assessment Code (RAC):**

 According to the Risk Assessment Code (RAC), the metals in the sediments are bound with different strengths to the fractions. The RAC assesses the availability of metals in solution by applying a scale of the relative percentage in the exchangeable and carbonate fractions (% $\Sigma$  F1  $+F2$ ). This classification is 1-10% (low risk), 11-30% (medium risk), 31-50% (high risk) and >50% (very huge risk) [15 &17]. The Risk Assessment Code (RAC) for Pb, Mn, Cu and Zn in the study area are given in Table (8). The data revealed that RAC are within very high risk for Pb at site (20) Sira island and medium risk at another sites. Zn and Cu had low risk. Mn was in low risk range at most of the sites, except Sira and Caltex which revealed medium risk for aquatic environment.

# **Conclusion**

 The Sequential Extraction Method has been used to study the geochemical form of Pb, Mn, Zn and Cu in Aden Coasts sediments. The present speciation of heavy metals revealed that the nonresidual fractions (anthropogenically sources) represent 72.8 % of the total extractable fractions for Pb, 79.7 % for Mn, 87.4 % for Zn, and 87.3 % for Cu. The study revealed that the high levels of metals in the investigated area are due to natural phenomena, as well as man-made sources. The risk assessment of trace metals in the study area can be arranged as follows: Pb > Mn > Zn > Cu. In general, the spatial distribution of heavy metals in Aden Coast sediments was controlled by the association of the metals with carbonate, fine particles (silt-clay) and organic matter content.

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<sup>a</sup> Samples using the sequential extraction method for the estimation of heavy metals; <sup>b</sup>TCO<sub>3</sub>:Total Carbonate; <sup>c</sup>OC: Organic Carbon; <sup>d</sup>OM: Organic Matter.





\*ND: Not detected

# Table 5: The average values of Mn ( $\mu$ g/g) in the five fractions of Aden coasts sediments









# Table 7: The average values of Cu ( $\mu$ g/g) in the five fractions of Aden coasts sediments



# **Table 8: Risk Assessment Code (RAC)**





**Figure 2: Schematic diagram shows the sequential extraction analytical procedures used on study heavy metals in sediments from Aden Coasts, Yemen**





**Figure 3: The distribution of sand content (%) in Aden Coast sediments**



**Figure 4: The distribution of silt content (%) in Aden Coast sediments**<br> $\frac{43000}{44^\circ 50^\circ}$ 



**Figure 5: The distribution of clay content (%) in Aden Coast sediments**

Univ. Aden J. Nat. and Appl. Sc. Vol. 23 No.2– October 2019 392

**Geochemical rorms and risk assessment** ……Shaif M. K.S., Samir M. Nasr, Mohamed A. Okbah



**Figure 6: The distribution of organic matter content (%) in Aden Coast sediments** 



**Figure 7: The distribution of total carbonate content (%) in Aden Coast sediments** 



**Geochemical rorms and risk assessment** ……Shaif M. K.S., Samir M. Nasr, Mohamed A. Okbah





**Figurer 9: Significant correlations between any two heavy-metal binding fractions: Exchangeable (Exch); bound to carbonates (Carb); bound to oxides (Oxide); bound to organic matter (OM); (p <0.025 & p < 0.01)** 

### **Geochemical rorms and risk assessment** ……Shaif M. K.S., Samir M. Nasr, Mohamed A. Okbah

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# الصور الجيوكيميائية وتقييم خطورة بعض العناصر الثقيلة في رسوبيات سواحل

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# الملخص

 الهدف من هذه الدراسة هو تحديد الصور الجيوكيميائية وتقييم خطورة بعض العناصر الثقيلة )الرصاص ، المنجنيز، الخارصين، والنحاس( في رواسب سواحل مدينة عدن، وكذلك تحديد أنواع طبيعة الرسوبيات ونسب الكربونات والكربون العضوى بها. استخدمت طريقة الاستخلاص المتتابع لتقدير نسبة العناصر في خمسة أجزاء مختلفة تشمل: الجزء المتبادل؛ المرتبط بالكربونات؛ المرتبط بالأكاسيد؛ المرتبط بالمادة العضوية ؛ والجزء المتبقي. أظهرت النتائج أن نسبة العناصر المرتبطة بالجزء غير المتبقي )مصدرها صناعي - بفعل النشاطات البشرية) تمثل %72.8 للرصاص، %7.97 للمنجنيز ، % 87.4 للخار صين، و %87.3 للنحاس. وبينت الدراسة نسبة العناصر في الخمس الصور مرتبة على النحو التالي:

**الرصاص:** الصورة المتبقية > الصورة المتبادلة> الصورة المرتبطة باألكاسيد > الصورة العضوية > الصورة المرتبطة بالكربونات.

**المنجنيز أو الخارصين:** الصورة المتبقية>الصورة المرتبطة باألكاسيد > الصورة العضوية> الصورة المرتبطة بالكربونات> الصورة المتبادلة.

**النحاس:** الصورة العضوية > الصور المتبقية > الصورة المرتبطة باألكاسيد > الصورة المرتبطة بالكربونات > الصورة المتبادلة.

 ومن خالل دراسة تقييم خطورة العناصر الثقيلة على الكائنات الحية تبين بأن عنصر الرصاص تتراوح خطورته من المتوسط (في جميع المواقع) إلى العالي في جزيرة صيرة، الزنك والنحاس اقل خطورة، بينما المنجنيز اقل خطورة (في معظم المناطق) إلى متوسط الخطورة (منطقتي صبرة وكالتكس) على البيئة البحرية. وبشكل عام يمكن ترتيب خطورة العناصر في منطقة الدراسة على النحو التالي من األكثر خطورة إلى األقل: الرصاص > المنجنيز > الخارصين > النحاس.

**الكلمات ا لمفتاحية:** االستخالص المتتابع، العناصر الثقيلة ، الصور **ا** لجيوكيميائية ، تقييم الخطورة**،** سواحل عدن.