
Speciation of heavy metals in marine sediments from Damietta Harbor, Egypt

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Abstract

The chemical speciation of heavy metals (Fe, Mn, Zn and Cu) in twenty one sediment samples collected from Damietta harbor and the adjacent Mediterranean Sea area to evaluate the possible contamination of the sediments by trace metals. Sequential extraction techniques were performed to study the different geochemical forms of these metals. The elevated concentrations are associated with the oxides form for both Fe and Mn. While for Zn the organic form is the major and for Cu the higher concentrations are associated with the residual form. Risk assessment code (RAC) was calculated for the metals to assess its availability in solution. The results revealed that, Fe gives 100% no risk, while for Mn and Zn, RAC is 62% and 67% of low risk respectively. Finally for Cu it gives 48% Medium risk and 52% low risk. The recovery of the sequential extraction for each metal was calculated and the sum of the five fractions is in good agreement with the total content, which implies that the method used is reliable and repeatable.

Keywords: Speciation, Heavy metals, Sequential extraction, Marine sediments, Nile delta.

1. Introduction

Sequential extraction of elements from sediment is a common analytical tool and widely applied technique in geochemical exploration and environmental geochemistry (Sutherland and Tack, 2004). Speciation of heavy metals largely determines their mobility and bioavailability in sediments (Zhou *et al.*, 2010 and Katherine & Christine, 2003).

Sequential extraction procedures are designed to isolate specific fractions from sediment (or other materials) by successively attacking the sample with chemical reagents selected for their ability to react with different components of the matrix and release associated trace metals, then analyzing the resulting supernatant (Sobczynski and Siepak, 2001). Many of the techniques used are based modified sequential extraction protocols developed for use in soil chemistry and trace metal analysis (Gibbs, 1973; Gupta and Chen, 1975; Tessier *et al.*, 1979; Gibson and Farmer 1986; Miller *et al.*, 1986; Oughton *et al.*, 1992; Morera *et al.*, 2001; Hong *et al.*, 2002; Banerjee, 2003; Kryc *et al.*, 2003; Tüzen 2003).

In geoenvironmental studies of risk assessment, chemical partitioning among the various geochemical phases is more useful than measurements of total heavy metals contents. Heavy metal speciation in environmental media using sequential extraction techniques offers a more realistic estimate of actual

environmental impact (Gumgum and Ozturk, 2001; Dold, 2003).

The aims of this study are: (i) to determine the metal pollution levels in marine sediments from Damietta harbor of Egypt; (ii) to determine and compare the chemical speciation of heavy metals to evaluate relative mobility and bioavailability.

2. Materials and methods

2.1. Study area

Damietta harbor (A port) is a marine harbor lying just west of Damietta city on the coast of Nile Delta, Egypt. A port was constructed in 1982 for about 10 km west to Damietta outlet of the Nile River (Figure 1), for different harbor activities it is necessary to make an environmental assessment and monitoring study for the pollutants occurring in this critical area as a result of human activities a heavy metal concentrations assessment.

2.2. Sampling locations description

Surface grab sampler sediments were collected at twenty one stations in Damietta harbor and the area in front of it (Figure 1), their locations were determined using a Geographical Positioning System (GPS); sites were chosen to cover areas which are known to be

affected by land-based activities. Samples from 1 to 13 were bottom samples. The first three samples were collected from the off shore area at depth of 100 m to be used as reference samples because they were far from the sources of pollution. The other 10 collected from the navigation channel and the harbor basin at depths ranging from 8m to 15m. The samples from 14 to 21 were collected from the land surface in the harbor surrounding the water basin. The samples were kept frozen until analysis.

2.3. Sequential extraction method

The sequential method used for the speciation of heavy metals (Figure 2) was done by (Tessier A., et al., 1979). One gram sediment samples (dry weight) were conducted in centrifuge tubes (poly propylene, 50 ml) to minimize losses of solid material.

The concentrations of trace metals in each fraction were measured using A.A.S. (Shimadzu, model 6800). The concentrations of trace metals were determined and measured in $\mu\text{g/l}$ (ppm).

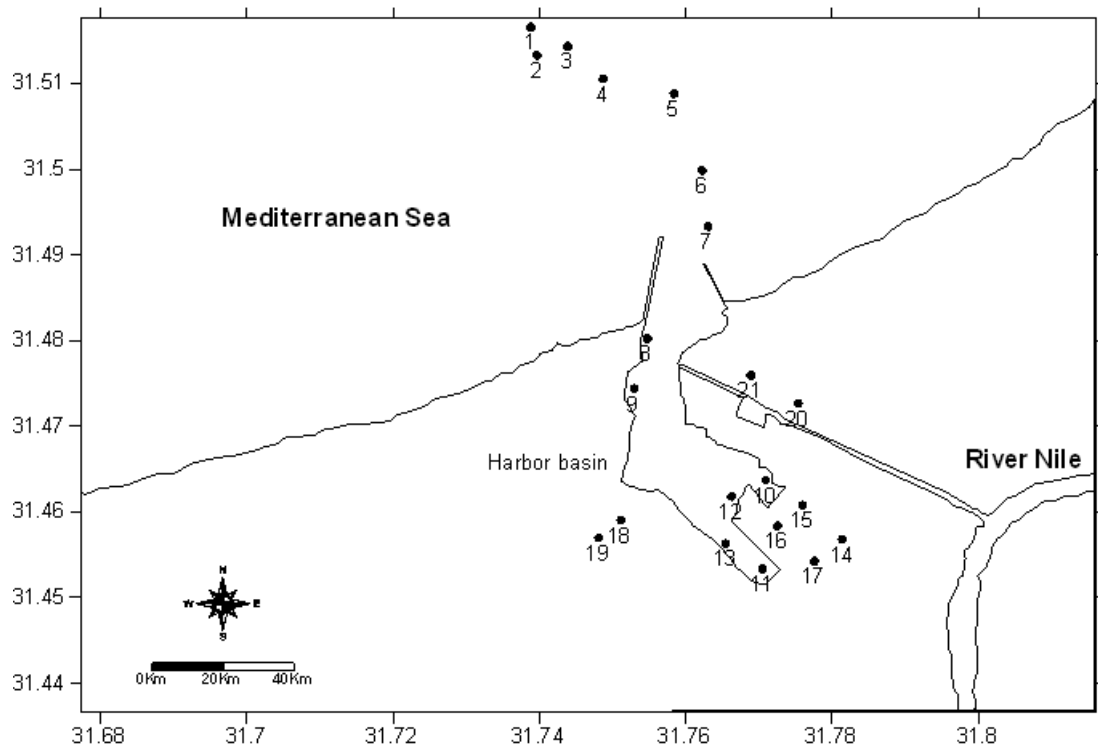


Figure 1. Map for the study area and the samples locations, Damietta harbor-Egypt.

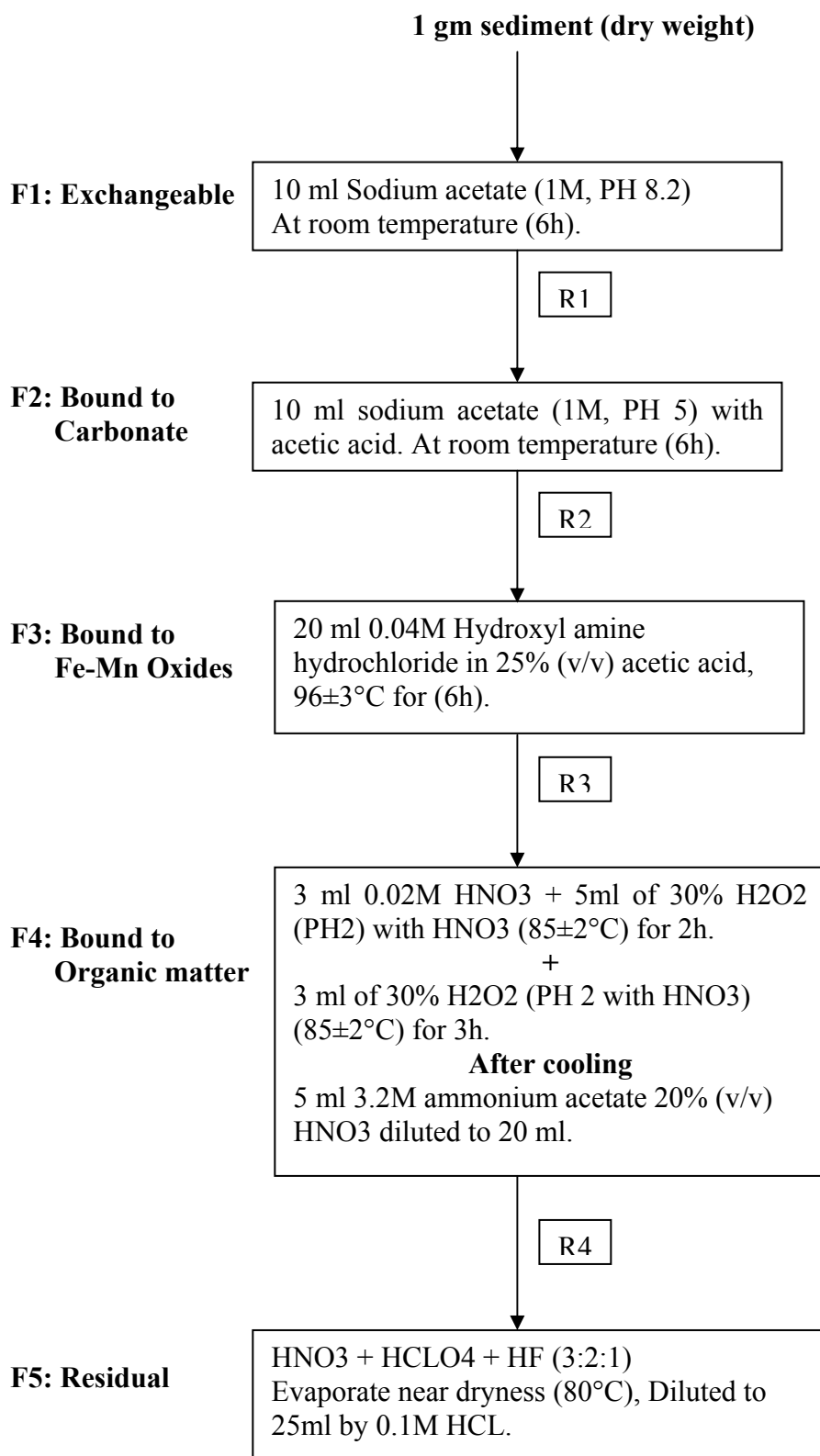


Figure 2. Schematic diagram shows the sequential extraction analytical procedures used on study of heavy metals in bottom sediments from Damietta harbor and the adjacent sea area, Egypt.

3. Results and discussion

3.1. Reproducibility and accuracy of the method

The analytical precision was tested by subjecting triplicate sediment samples to the previous procedure for the sequential extraction of heavy metals. The test results show good reproducibility and a precision (expressed as the % Coefficient of variation) of individual extractions varying from < 1 to 12%. Thus, the CV% calculated for all the metals fractions was within the range of precision. Where, the CV% calculated for the Fe fractions ranging from (0.39 – 3.75), Mn fractions ranging from (0.57 – 7.41), Zn fractions ranging from (0 – 7.93) and Cu fractions ranging from (0 – 11.77).

An internal check was performed on the results of the sequential extraction by comparing the total amount of metals extracted by different reagents during the sequential extraction procedure with the results of the total digestion. The recovery of the sequential extraction for each metal was calculated as follows;

Recovery = [(F1 + F2 + F3 + F4 + F5) / total digestion] x 100

Where: F1, F2, F3, F4 and F5 are Exchangeable, Carbonate, Fe-Mn oxides, Organic and Residual fractions respectively.

The results showed in (Table 1) indicated that the sums of the five fractions are in good agreement with the total digestion results with the satisfactory recoveries (70 – 126 %) and the method used is reliable and repeatable (Cuong and Obbard, 2006).

The recovery of all the metals were in the acceptable range where, the recovery of Fe ranging from (74.44% - 116.92%) while, the recovery of Mn ranging from (81.44% - 110.22%), the recovery of Zn ranging from (81.39% - 111.70%) and the recovery of Cu ranging from (89.35% – 122.91%).

3.2. Sequential extraction results

The concentrations of the selected elements in the five fractions are showed in (Figures 3, 4, 5 and 6). However the data obtained about the fractionation of Fe in both bottom and land samples revealed that Fe-Mn oxides fraction was extracted more than the other fractions in the non-residual. This indicates that, Fe is more distributed in oxides fractions. The Fe concentration bound to the exchangeable, carbonate and organic fractions were found to be much lesser content than the oxides fraction. The Fe extracted in the residual fraction was relatively high comparing to the last three fractions but still much lesser content than the oxides fraction. Generally, the average values and the percentage of Fe concentration in the five fractions were followed the order: Fe-Mn oxides F3 (2244.34 ppm, 81.26%) > Residual F5 (465.04 ppm, 16.84%) > Organic F4 (46.13 ppm, 1.67%) > Exchangeable F1

(3.94 ppm, 0.14%) > Carbonate F2 (2.50 ppm, 0.09%) for bottom samples. While, land samples followed the order of Fe-Mn oxides F3 (2056.89 ppm, 81.51%) > Residual F5 (449.68 ppm, 17.82%) > Organic F4 (11.80 ppm, 0.47%) > Carbonate F2 (3.47 ppm, 0.14%) > Exchangeable F1 (1.74 ppm, 0.07%). Thus, the average values and percentage of Fe concentrations in the five fractions of the bottom samples (F3 > F5 > F4 > F1 > F2) show slight difference from that of the land samples (F3 > F5 > F4 > F2 > F1). Iron is essentially bound to the residual fraction, suggesting relatively low mobility of the element (Caplat *et al.*, 2005).

The data obtained about the fractionation of Mn in both bottom and land samples revealed that Fe-Mn oxides fraction was extracted more than the other fractions in the non-residual indicating that Mn is more distributed in oxides fractions. The manganese concentration bound to the exchangeable, carbonate and organic fractions were found to be much lesser content than the oxides fraction. The manganese extracted in the residual fraction was relatively high comparing to the last three fractions but still lesser content than the oxides fraction. Generally, the average values and the percentage of Mn concentrations in the five fractions were followed the order: Fe-Mn oxides F3 (454.71 ppm, 64.32%) > residual F5 (184.22 ppm, 26.06%) > organic F4 (60.53 ppm, 8.56%) > carbonate F2 (4.52 ppm, 0.64%) > exchangeable F1 (2.92 ppm, 0.41%) for bottom samples. While, land samples followed the order of Fe-Mn oxides F3 (237.08 ppm, 45.59%) > residual F5 (228.43 ppm, 43.93%) > organic F4 (40.97 ppm, 7.88%) > carbonate F2 (11 ppm, 2.12%) > exchangeable F1 (2.51 ppm, 0.48%). Thus, the average values and percentage of Mn concentrations in the five fractions of the bottom samples (F3 > F5 > F4 > F2 > F1) are the same of the land samples (F3 > F5 > F4 > F2 > F1). Manganese is essentially bound to the residual fraction, suggesting relatively low mobility of the element (Caplat *et al.*, 2005).

Moreover, the data obtained about the fractionation of Zn in both bottom and land samples revealed that organic fraction was extracted more than the other fractions in the non-residual. Suggesting that, Zn is more distributed in organic fraction. The Zinc concentration bound to the exchangeable, carbonate and Fe-Mn oxides fractions were found to be much less content than the organic fraction. The Zinc extracted in the residual fraction was relatively high comparing to the last three fractions but still less content than the organic fraction. Generally, the average values and the percentage of Zn concentrations in the five fractions were followed the order: organic F4 (68.49 ppm, 44.81%) > residual F5 (45.31 ppm, 29.65%) > Fe-Mn oxides F3 (37.11 ppm, 24.28%) > carbonate F2 (1.89 ppm, 1.24%) > exchangeable F1 (0.04 ppm, 0.03%) for bottom samples. Also, Land samples followed the same order that, organic F4 (68.95 ppm, 45.67%) > residual F5 (60.59 ppm, 40.14%) > Fe-Mn oxides F3 (19.85

ppm, 13.15%) > carbonate F2 (1.56 ppm, 1.03%) > exchangeable F1 ("not detected" 0 ppm, 0%). Thus, the average values and percentage of Zn concentrations in the five fractions of the bottom samples (F4 > F5 > F3 > F2 > F1) are the same of the land samples (F4 > F5 > F3 > F2 > F1). Zinc is essentially bound to the residual fraction, suggesting relatively low mobility of the element (Caplat *et al.*, 2005).

The data obtained about the fractionation of Cu in both bottom and land samples revealed that, residual fraction was extracted more than the other fractions. This indicates that, Cu was strongly bound to the sediments and had strong association to the crystalline structures of sediments. In bottom sediment samples the organic fraction was extracted more than the other fractions in the non-residual fractions. While, in the land samples the Fe-Mn oxides fraction was extracted more than the other fractions in the non-residual fractions. Generally, the average values and the percentage of Cu concentrations in the five fractions were followed the order: residual F5 (32.32 ppm, 69.6%) > organic F4 (7.07 ppm, 15.22%) > Fe-Mn oxides F3 (4.81 ppm, 10.36%) > exchangeable F1 (2.24 ppm, 4.82%) > carbonate F2 (0 ppm, 0%) for bottom samples. While, land samples followed different order that, residual F5 (11.01 ppm, 53.55%) > Fe-Mn oxides F3 (3.31 ppm, 16.1%) > organic F4 (2.45 ppm, 11.92%) > carbonate F2 (1.99 ppm, 9.7%) > exchangeable F1 (1.8 ppm, 8.7%). Thus, the average values and percentage of Cu concentrations in the five fractions of the bottom samples (F5 > F4 > F3 > F1 > F2), while for the land samples (F5 > F3 > F4 > F2 > F1). Copper is essentially bound to the residual fraction, suggesting relatively low mobility of the element (Caplat *et al.*, 2005).

3.3. Risk Assessment Code (RAC)

According to the 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. This classification is tabulated in (Table 2) (Singh *et al.*, 2005). It is evident from the results of the fractionation studies that, the metals in the sediments are bound to different fractions with different strengths. The strength values can, therefore, give a clear indication of sediment reactivity, which in turn assess the risk connected with the presence of metals in an aquatic environment. This criterion of RAC as given below indicates that sediment which can release in exchangeable and carbonate fractions, lesser than 1% of the total metal will be considered safe for the

environment. On the contrary, sediment releasing in the same fraction more than 50% of the total metal has to be considered highly dangerous and can easily enter the food chain (Jain *et al.*, 2004).

Table 3 showed the risk assessment code (RAC) for the bottom samples sediments. The risk assessment code of Fe showed no risk at all the samples. The RAC average of Fe was (0.24%) suggesting no risk to the aquatic environment. The RAC of Mn showed no risk at most of the samples except samples (4, 5, 6, 7, 8 and 9), which showed very low risk. The risk assessment codes for these samples were (1.25%, 1.29%, 2.2%, 1.68% and 1.04%) respectively. The average risk assessment code of Mn was (0.97%) indicating no risk of Mn to the aquatic environment. The risk assessment code of Zn showed low risk at most of the samples except for samples (6, 7, 8, 9 and 13) that showed no risk. The risk assessment codes of these samples were (0.95%, 0.99%, 0.91%, 0.92% and 0.91%) respectively. The average risk assessment code of Zn was (1.64%) which showed very low risk to aquatic environment. The risk assessment code of Cu showed low risk in all the samples except for samples (1, 2 and 3) that showed medium risk. The risk assessment codes of these samples were (30.19%, 24.12% and 27.23%) respectively. The average risk assessment code of Cu was (9.66%) which showed low risk to aquatic environment. While, (Table 4) showed the risk assessment code (RAC) for the land samples sediments. The risk assessment code of Fe showed no risk at all the samples. The average risk assessment code of Fe was (0.21%) which showed no risk to the aquatic environment. The risk assessment code of Mn showed low risk at all the samples except sample (16), which showed no risk. The risk assessment code for this sample was (0.36%). The average risk assessment code of Mn was (2.62%) suggesting very low risk of Mn to the aquatic environment. The risk assessment code of Zn showed low risk at all the samples except for samples (14 and 16) that showed no risk. The risk assessment codes of these samples were (0.7% and 0.61%) respectively. The average risk assessment code of Zn was (1.03%) which showed very low risk to aquatic environment. The risk assessment code of Cu showed medium risk in all the samples except for sample (21) that showed low risk. The risk assessment code of this sample was (7.72). The average risk assessment code of Cu was (18.64%) which showed medium risk to aquatic environment.

In general, the risk assessment code of metals in the study area can be arranged as the following; Bottom Samples: Cu > Zn > Mn > Fe; Land Samples: Cu > Mn > Zn > Fe.

Table 1. Recovery of the sequential extraction and the total digestion.

Samples	Fe			Mn			Zn			Cu		
	Sum	Total	Recovery	Sum	Total	Recovery	Sum	Total	Recovery	Sum	Total	Recovery
1	1351.35	1215.43	111.18	444.60	481.37	92.36	59.60	53.35	111.70	6.98	7.28	95.94
2	1816.17	1970.77	92.16	405.79	438.69	92.50	63.43	67.15	94.47	8.47	8.26	102.57
3	1742.85	2341.37	74.44	352.95	373.43	94.52	60.90	66.38	91.74	7.75	8.68	89.35
4	3178.35	3393.20	93.67	686.95	843.46	81.44	168.57	161.49	104.38	32.02	30.11	106.34
5	3063.28	3577.08	85.64	872.26	914.80	95.35	167.40	163.55	102.36	39.04	39.57	98.66
6	2857.58	2494.22	114.57	703.92	757.66	92.91	190.94	179.05	106.64	27.96	28.32	98.71
7	3093.31	2645.76	116.92	740.82	811.53	91.29	179.69	170.51	105.39	61.21	59.76	102.41
8	3239.31	3440.70	94.15	1112.80	1193.29	93.25	204.60	192.09	106.51	73.34	71.19	103.02
9	2850.22	2456.44	116.03	793.02	817.03	97.06	193.51	198.29	97.59	68.03	69.45	97.95
10	2878.40	2909.51	98.93	733.50	731.52	100.27	106.16	99.23	106.99	56.79	46.90	121.08
11	3306.14	2991.84	110.51	827.91	751.17	110.22	199.87	192.73	103.71	66.88	68.15	98.13
12	3210.86	2996.13	107.17	695.75	691.40	100.63	182.61	186.43	97.95	76.67	69.33	110.59
13	3317.54	2903.17	114.27	786.83	727.80	108.11	209.69	196.11	106.92	78.49	75.94	103.35
14	2644.22	2795.66	94.58	543.98	498.10	109.21	105.90	96.42	109.83	16.98	15.73	107.93
15	2516.03	2578.55	97.58	547.58	550.67	99.44	102.46	93.35	109.76	23.00	21.40	107.48
16	2489.69	2514.31	99.02	506.45	540.10	93.77	179.80	189.40	94.93	20.62	18.63	110.67
17	2420.30	2422.29	99.92	484.13	472.39	102.48	141.35	140.34	100.72	18.75	17.01	110.24
18	2495.03	2533.67	98.48	513.15	536.17	95.71	146.73	180.28	81.39	19.67	18.54	106.10
19	2664.08	2665.51	99.95	559.18	560.38	99.79	186.14	188.66	98.66	23.76	21.24	111.87
20	2372.76	2396.02	99.03	468.98	442.37	106.02	183.68	189.76	96.80	20.09	16.34	122.91
21	2586.52	2632.12	98.27	516.41	592.91	87.10	161.52	168.88	95.64	21.56	23.67	91.09

Sum = F1 + F2 + F3 + F4 + F5, Total = Total metal concentration, & Recovery % = Sum / Total.

Table 2. Classification Risk Assessment Code (RAC) (Singh *et al.*, 2005).

No risk	< 1%
Low risk	1 – 10%
Medium risk	11 – 30%
High risk	31 – 50%
Very high risk	> 50%

Table 3. Risk assessment code (RAC) (F1+F2 percentage) in bottom samples.

Sample no.	Fe	Mn	Zn	Cu
1	0.33	0.14	3.40	30.19
2	0.23	0.17	3.22	24.12
3	0.23	0.19	3.98	27.23
4	0.26	1.25	1.22	6.19
5	0.14	1.29	1.06	6.62
6	0.22	2.20	0.95	7.46
7	0.30	1.68	0.99	3.89
8	0.16	1.04	0.91	3.45
9	0.61	2.29	0.92	3.27
10	0.16	0.89	1.71	3.94
11	0.17	0.66	1.07	3.40
12	0.17	0.22	1.00	2.94
13	0.15	0.55	0.91	2.81
Average	0.24	0.97	1.64	9.66

Table 4. Risk assessment code (RAC) (F1+F2 percentage) in Land samples.

Sample no.	Fe	Mn	Zn	Cu
14	0.23	2.84	0.70	22.60
15	0.35	2.87	1.02	17.63
16	0.18	0.36	0.61	19.22
17	0.18	2.35	1.33	21.38
18	0.17	3.14	1.28	20.09
19	0.15	2.48	1.01	18.99
20	0.18	4.04	1.07	21.47
21	0.20	2.88	1.23	7.72
Average	0.21	2.62	1.03	18.64

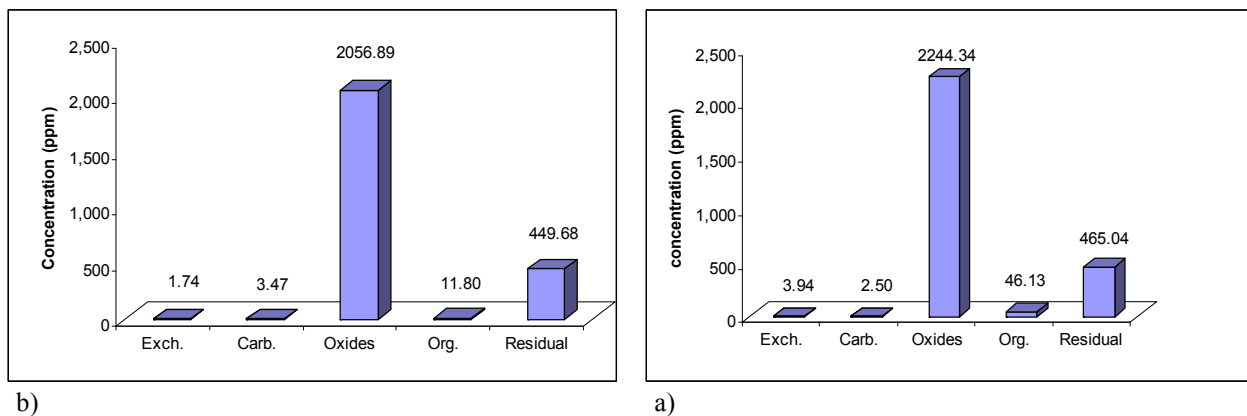


Figure 3. a) The average Concentration of Fe fractions (ppm) in bottom sediment samples.
 b) The average Concentration of Fe fractions (ppm) in Land sediment samples of Damietta harbor.

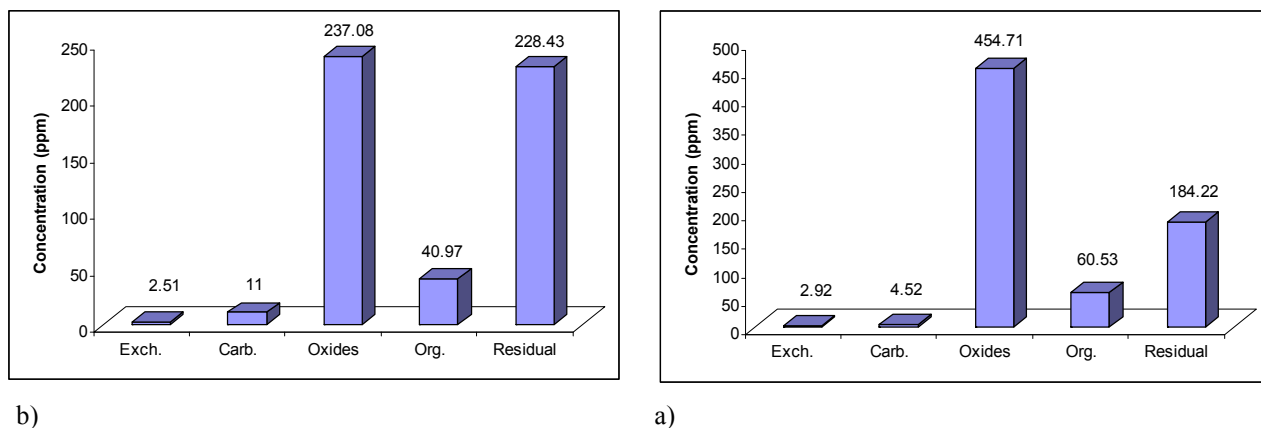


Figure 4. a) The average Concentration of Mn fractions (ppm) in bottom sediment samples.
 b) The average Concentration of Mn fractions (ppm) in Land sediment samples of Damietta harbor.

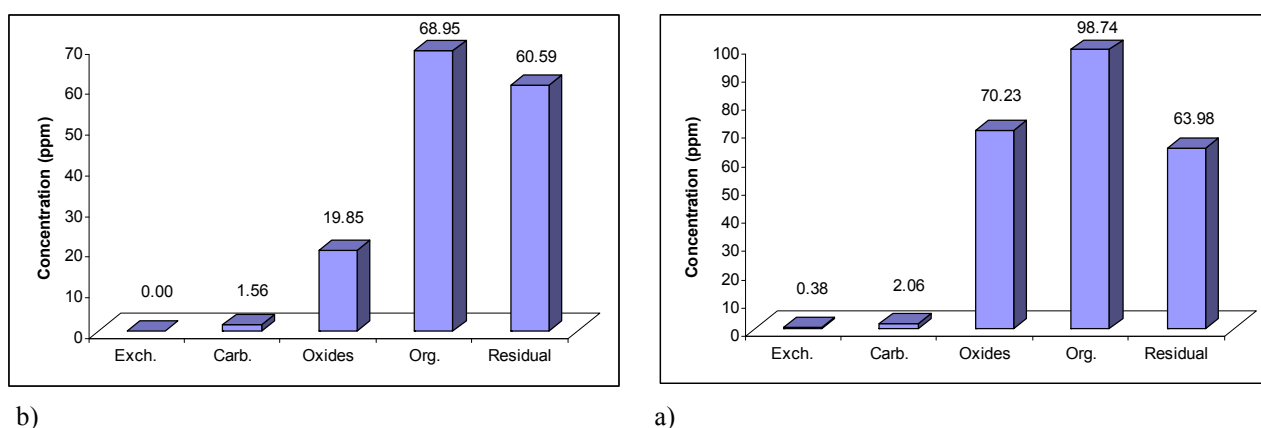


Figure 5. a) The average Concentration of Zn fractions (ppm) in bottom sediment samples.
 b) The average Concentration of Zn fractions (ppm) in Land sediment samples of Damietta harbor.

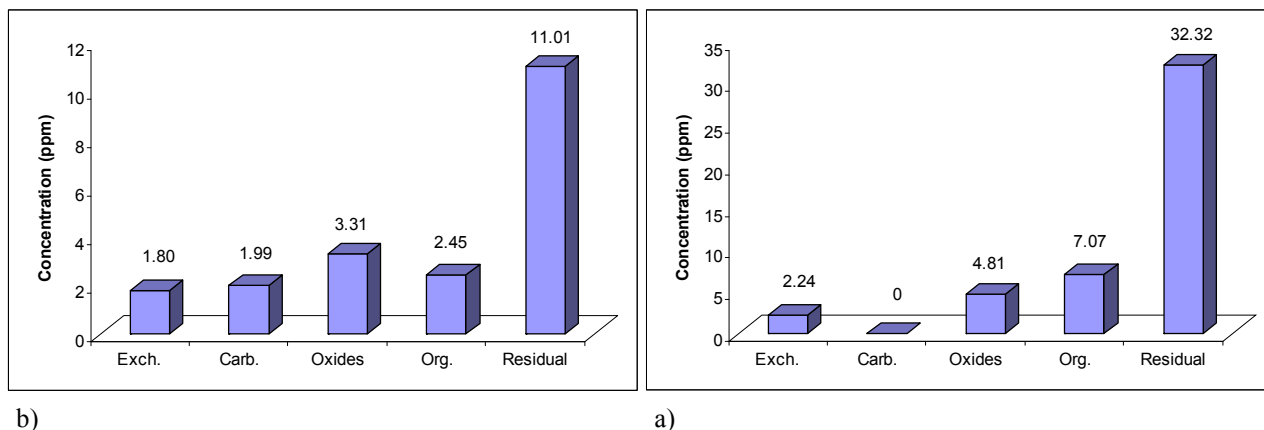


Figure 6. a) The average Concentration of Cu fractions (ppm) in bottom sediment samples.
b) The average Concentration of Cu fractions (ppm) in Land sediment samples of Damietta harbor.

4. Conclusion and Recommendations

The present study revealed that, Fe gives 100% no risk, Mn gives 62% low risk, while for Zn it was 67% low risk, finally for Cu it gives 48% Medium risk and 52% low risk. The present study tried to introduce some solutions and recommendations for improvement and future management of Damietta harbor environment, these are:

- It is recommended that a continuous monitoring program for Damietta harbor should be formulated and conducted to ensure that the heavy metals remain within the baseline levels established during the present study.
- Identifying environmental status, as well as, economic values of Damietta harbor.
- Review of site characteristics and evaluation of regulatory approaches.
- Progressive investigation, assessment and management of potential sediment contamination problems.
- Perform basic and continuous quantification of pollutants in the study area.
- Continuous evaluation of short and long term impacts of major pollutants on the harbor area.
- Further investigations in the future, are needed particularly to include sediment aging with the help of radioisotopes, which could help to identifying and recognize some more vital points.
- The results of this study could be used as a contribution to the knowledge and rational management of these regions in the future.
- The results also would serve as a baseline against which future anthropogenic effects can be assessed.

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الإستخلاص المتتابع للعناصر الثقيلة فى الرسوبيات البحرية بميناء دمياط - مصر

سوزان محمد الغرباوى - محمد على شطا - مى إبراهيم الجمال - محمود سالم إبراهيم - سوزان عمر دراز

تم دراسة طريقة الإستخلاص المتتابع الكيمياءى للعناصر الثقيلة (الحديد, المنجنيز, الزنك و النحاس) فى 21 عينة رسوبية تم تجمعها من ميناء دمياط و المنطقة البحرية المتصلة بها من البحر المتوسط بغرض تقييم احتمالية تلوث رسوبيات المنطقة بالعناصر الثقيلة.

و تم دراسة الصور الكيمائية المختلفة لتلك العناصر. من خلال تقييم وحساب قيم العناصر الثقيلة حيث وجد ان الحديد و المنجنيز يوجد فى الصورة المتأكسدة. و بالنسبة للزنك فقد كان سائدا فى الصورة المتحدة مع المواد العضوية, بينما النحاس كان متركزا فى الصورة المتبقية.

وبدراسة معامل كود الخطر (RAC) لتقييم مدى تحرر وانتشار العناصر الثقيلة بالمياه. فقد أوضحت النتائج ان عنصر الحديد اعطى نسبة 100% بدون خطر, وعنصر المنجنيز اعطى 62% خطر محدود, و الزنك اعطى 52% خطر محدود, واخيرا عنصر النحاس اعطى 48% خطر متوسط.

وبحساب قيمة الاسترداد لكل عنصر ومجموع التركيزات للخمس صور المختلفة, وجد انها جيدة التوافق مع التركيز الكلى وهذا يعكس دقة العمل وصحته.