Assessment of zinc and cadmium contamination using metal chemical fractions in sediments of Lake Edku, Egypt

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Abstract

To evaluate the contamination and bioavailability of zinc and cadmium in sediments of LakeEdku,the distribution of total and different chemical forms of zn and Cd has been studied using sequential extraction technique. Total Organic Matter (TOM), pH, particle size and carbonate contents were also studied. The I_{geo} results showed thatsamples can be characterized as ‘‘unpolluted’’ class for Zn while moderately to highly polluted for Cd.This may be due to industrial activities as well as agriculture drainage water discharged into the Lake. Comparison with the sediment quality guidelines showed that Zn and Cdlevelsare less than the PEL limitvalue.Data of chemical analysis of the sediments show that the lowest portion of Cd and Zn were associated with exchangeable fraction reflecting the lower degree of bioavailability. The highest content of Cd was associated withoxide fraction may be due to the fact that it can form stable complexes with Fe and Mn oxides.The highest content of Zn was associated withresidual fraction, indicating thatZn was associatedstrongly with crystalline structure of the minerals and has high stability and low ability of transfer under natural conditions.

Keywords: Zinc, Cadmium, Chemical fractionation, Lake Edku

1. Introduction

Northern lakes of Egypt are important wetland reserves for the maintenance of biodiversity in Egypt (Ramdani et al., 2001). Lake Edku, the smallest Northern lakes is a brackish and a shallow eutrophic lake lies to the west of Rosetta branch approximately 35 km east of Alexandria. The dimensions of the Lake are 17 km length and 11 km width at its widest part with an average water depth around 0.65 m. The total surface area is 62.787 million m^2. It is connected to the sea via a narrow artificial inlet (Boughaz El-Maadiya) (Masoud et al., 2005). In the meantime, the Lake was subjected to a gradual shrinkage during the past few decades due to land reclamation and transformation of significant parts of the Lake to fish farms. The lake can be divided into three ill-defined basins; eastern, central and western. It receives huge amounts of agriculture drain water from four main drains, namely, Edku, Bousaly, El-Khairy and Berseek, which open into the eastern basin of the lake (Okbahand El-Gohary, 2002).The influent waters from these drains enter the lake at a rate that varies from 1.5 to 4.2 million m^3/d(Mahmoud, 2008).So the Lake is subjected to drainage water enriched with chemical fertilizers, domestics and industrial effluentsand thereby rising metal contents accumulated in sediments.

Metals are of considerable environmental concern due to their toxicity, non-biodegradable properties, and accumulative behaviors .Metal contamination of sediments is often estimated by determining the totalmetals content, but it may not giveclear pictureof the impact and danger for living organisms (ŁebkowskaandKlimiuk, 1992; Dembska et al., 2001). Sequential extraction techniques will give a more complete estimation of actual environmental impact of heavy metals. In addition, the procedure can differentiate between metals originating from anthropogenic sources and metals of geochemical origin successfully.

The objective of this study was therefore to determinethe concentrations and spatial distributions of zinc and cadmium in sedimentcollected from LakeEdku. Additionally, the bioavailability of metalsin sediment was assessed by differentiating their chemicalforms.

2. Materials and Methods

2.1 Sampling

Sediment samples were collected in March 2015 from 11 sites distributed along LakeEdku(Fig. 1). A grab sampler was used to collect the sediment samples. Samples are kept in labeled polyethylene plastic bags. In laboratory, sediment samples were air-dried, ground and stored at room temperature.

2.2 Analytical methods

Total metal concentrations were determined according to Ajay and Van Loon (1989). Metals were determined using theAAS/flame mode (Shimadzu AA-6800).Chemical fractionation of zn and Cd was made based on Tessier et al. (1979) ; Engler et al. (1977); Pickering (1981) and modified by Rifaat et al. (1992), where fraction 1 (F_1), ion- exchange

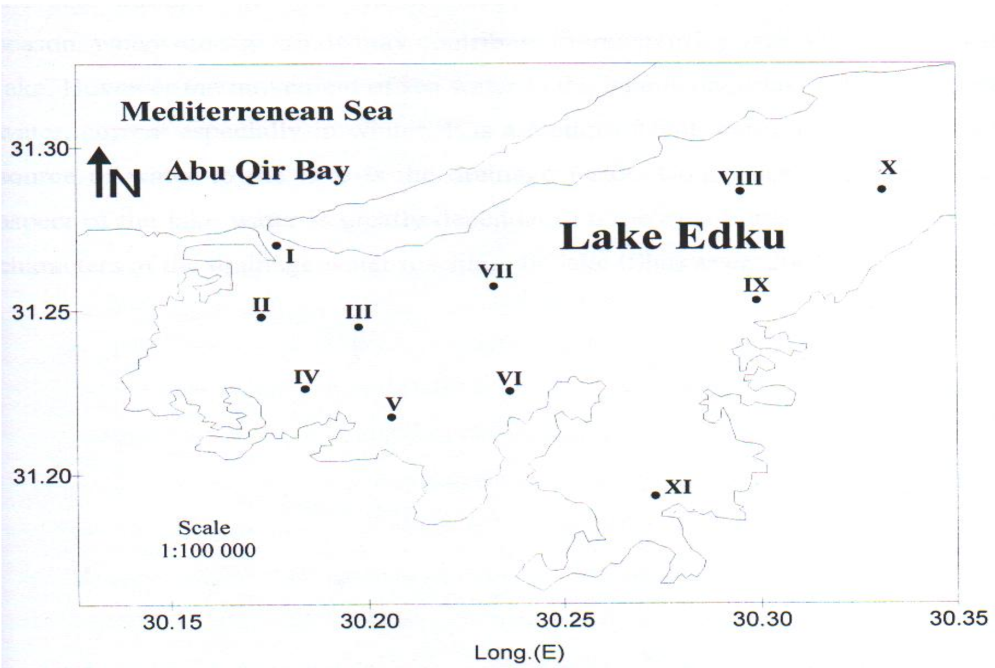

Fig . 1. Locations of sampling sites in Lake Edku

sites and surface adsorption by using (1M ammonium acetate); fraction 2 (F_2) precipitated CO_3^{2-} and copercipitated amorphous hydrous oxides (acetic acid and 1N hydroxyl amine hydrochloride); fraction 3 (F_3) copercpitated crystalline hydrous oxides (1N hydrochloric acid); fraction 4 (F_4), co- ordinate to organic matter; (1:1 nitric and perchloric acid) and fraction 5 (F_5); occluded i.e. crystalline silicate and lattice compounds (HNO_3, HF and $HClO_4$).Organic matter was determined according to Gaudette et al.(1974), and the carbonate content was determined as described by Molnia (1974).Grain size analysis was determined according to Folk (1974).

2.3Quality assurance and quality control

Duplicate samples analysis, blanks, and standard reference materials (IAEA-356) are used to assess the quality assurance and quality control. Analytical results of the quality control samples show good agreement with the certified values with recoveries of 92.63% and 97.04% for Zn and Cd respectively, whereas the precision was agreed within 10%. Quality control for the sequential extractions procedure was performed by comparing the sum of fractions with total metal content. The recovery of the sequential extraction procedure was calculated as follows:

$$\text{Recovery \%} = \left(\frac{\sum \text{sequential extracti on procedure}}{\text{single digestion with strong acids}}\right) \text{x100}$$

The recovery % of the metals was found to be within ± 20%.

2.4 Assessment method of sediment pollution

The geoaccumulation index (I_{geo}) values were calculated to estimate the pollution levels in each sample point,according to the following mathematical formula:

$$I_{geo} = \log_2 C_n / 1.5\ B_n$$

whereC_n and B_n are measured concentration and geochemical background concentration of the element, respectively (Anagnostou et al., 1997; Loska et al., 1997; Abrahimand Parker, 2008; Christophoridis et al., 2009). The average earth crust content was used as background where,Cd (0.2) and Zn (70) (Hamilton,2000;Kabata-PendiasandMukherjee, 2007).According to the I_{geo}value each metal can be classified as follow: (I_{geo}< 1) unpolluted, (1<I_{geo}< 2)very low polluted, (2<I_{geo}< 3)low polluted, (3 <I_{geo}< 4)moderately polluted, (4<I_{geo}< 5) highly polluted and (I_{geo}> 5)very highly polluted.Individual contamination factor (CF) is also calculated to evaluate the pollution of environmental single substances (TurekianandWedepohl, 1961; Loska et al., 1997) based on the following formula:

$$CF = M_x/M_b$$

whereM_x is the concentration of the target metal and M_b is the concentration of the metal in the selected reference background.CF is classified into four categories as follows: (CF< 1 low contamination factor; 1 <CF < 3 moderate contamination factor; 3 <CF < 6 considerable contamination factor; CF >6 very high contamination factor).

2.5Sediment quality guidelines (SQGs)

The sediment quality guidelines (SQGs) have an important role in predicting the risk of contaminated sediments with heavy metals on the living organisms.(MacDonald et al., 2000; Farkas et al., 2007; Varol 2011).They are also used to classify and arrange contaminated areas for further investigations (MacDonald et al. 2000). Two terms are used to express the degree of environmental risk: TEL «threshold effect level» and PEL «probable effect level». Below TEL, adverse biological effects are not expected to occur, while adverse effects can happen repeatedly and affect living organisms above PEL.(CCME, 1995; Smith et al., 1996; MacDonald et al., 2000).

3. Results and discussion

3.1 Sediment characterization

Table (1) shows that, the sediments of Lake Edku are varied in the sand fraction from 3.53% at station (III) to95.45% at station (XI) , while the maximum value of pan (silt and clay %) was 96.47% at station (III) and minimum value of 4.55% at station (XI).Organic matter affects the aquatic environment by forming complex compounds. It also serves as

Table 1: Properties of Lake Edku sediment

Stations	Grain size analysis		Sediment type	TOM %	$CaCO_3$ %	pH
	Sand %	Pan %				
I	41.26	58.73	Sandy mud	3.00	27.73	7.43
II	20.57	79.43	Sandy mud	8.00	24.11	8.10
III	3.53	96.47	mud	15.25	20.09	8.54
IV	22.98	77.02	Sandy mud	8.46	19.23	8.71
V	21.59	78.41	Sandy mud	9.00	25.62	8.71
VI	45.83	54.17	Sandy mud	13.00	31.82	8.20
VII	49.63	50.37	Sandy mud	13.62	19.78	7.70
VIII	49.62	50.38	Sandy mud	7.64	16.27	8.20
IX	35.63	64.37	Sandy mud	12.00	18.66	8.10
X	31.32	68.68	Sandy mud	6.81	27.25	8.10
XI	95.45	4.55	sand	2.00	35.24	8.10
Maximum	95.45	96.47		15.25	35.24	8.71
Minimum	3.53	4.55		2.00	16.27	7.43
Average	37.95	62.05		8.98	24.16	-
S.D	23.83	23.83		4.22	5.99	0.39

source of food for several animal groups. As shown in Table (1) the organic matter in the analyzed sediment samples ranged from 2 % at station (XI; El Barsik drain) to 15.25% at station (III;El Nagaa) with an average value of 8.98%, which is higher than that reported by Mahmoud, 2008. In general, the high level of total organic matter was found in the eastern region of the Lake, because there was a continuous in flow of wastewater, from main three drains namely Edku, Bousily and Berzik.

pH plays an important roles that control the availability and uptake of metals in aquatic environment.When the pH is about 6.5 to 7, the metal availability is relatively low (KashemandSingh,2001).Adsorption and precipitation are enhanced with increasing pH value. Decreasing the pH value leads to dissolve hydroxide and carbonates minerals, weak the association of metal and hinder the retention of metals by sediments (Zhang et al., 2014). This may be attributed to the competition between H^+ and metal ions for sorption at negative sites in the sediments (Shafie et al., 2014).The sediment samples show an alkaline pH ranged from 7.43 to 8.70, reflecting a relatively safer environment.

The carbonate content is expressed as calcium carbonate equivalent. Calcium carbonate is attributed to the abundance of mollusks and calcareous tube worms. Table (1) shows the distribution of calcium carbonate in sediment samples. Carbonate content ranged from 35.24% at station XI to 16.27 % at station VIII. High values of carbonate at some stations may be due to the extraction of CO_2 by phytoplankton and aquatic plants, which enhance carbonate precipitation. Moreover,biogenic precipitation of organite by aquatic organisms and precipitation of $CaCO_3$ from water rich in calcium during photosynthesis which increases pH value(Hasan, 2006). The carbonate contents and distribution affected the grain size distribution of the Lake sediment, because it is dominated by mollusks shells fragments.

3.2.Total metal contents

The distribution pattern of total concentrations of zinc and Cadmium insediment from LakeEdku is given in Table (2) and Fig (2) and compared with TEL and PEL. Metal contents ranged between 67.02 and 236.18;3.07and 5.36 (µg/g dry wt) for Zn and Cd respectively. The highest level of Zn and Cd were recorded at station (V). Generally, the concentrations of Cd are higher than background value in all sampling sites. Lake Edku subjects to agriculture wastewaters(phosphatic fertilizers in crop farms) from drains (Barsik, Edku, Bosily and El Kaiary drains) which maycause rise in cadmium concentration, Masoud et al.(2005).

The calculated values of geoaccumulation index for the studied metals (Table 2), shows that all samplescan be classified as ‘‘unpolluted’’ class for Zn except station (V) which classified as very low polluted.WhileI_{geo} values for Cd describe sediments as moderately to highlypollute. According to the calculated data of the individual

Table 2: Total metal Content (µg/g dry wt),Geo-accumulation Index (I_{geo}) and contamination factor (CF)

stations	Total metal concentrations		I_{geo}		CF	
	Zn	Cd	Zn	Cd	Zn	Cd
I	90.79	4.49	-0.21	3.90	1.30	22.45
II	67.02	3.81	-0.65	3.67	0.96	19.05
III	185.77	3.07	0.82	3.36	2.65	15.35
IV	163.92	3.79	0.64	3.66	2.34	18.95
V	236.18	5.36	1.17	4.16	3.37	26.80
VI	114.05	3.34	0.12	3.48	1.63	16.70
VII	85.78	3.44	-0.29	3.52	1.23	17.20
VIII	171.77	4.06	0.71	3.76	2.45	20.30
IX	132.86	4.30	0.34	3.84	1.90	21.50
X	196.18	3.29	0.90	3.46	2.80	16.45
XI	106.11	3.81	0.02	3.67	1.52	19.05
Maximum	236.18	5.36	1.17	4.16	3.37	26.80
Minimum	67.02	3.07	-0.65	3.36	0.96	15.35
Average	140.948	3.89	0.325	3.678	2.014	19.436
S.D	53.510	0.65	0.573	0.231	0.761	3.270

contamination factor (CF), it can be concluded that sediments of Lake Edku are very highly contaminated with Cd (CF >6) and moderately contaminated withZn.

Sediment quality guidelines (SQGs) help in predicting the adverse biological impacts in sediments contaminated with metals. Figure (2) shows that Zn values are less than the PEL limit valuein all stations, while, surpassed TEL value in 55% of stations.For the Cd,SQGscomparison shows that all sediment samples have been found to excess the TEL limit value, on the other hand, station (V) has been found to excessCd-PEL limit value (5 ppm),inducing a risk for the aquatic system living organisms. Concentrations of the heavy metals detected in this study were compared with other published data in the northern five delta lagoons of Egypt(Table 3). It is clear that, concentration of Cd shows an increasing trend with time,so, Cd cannot be ignored for its higher concentrations. While, the levels of Zn were within the range measured in the lake.

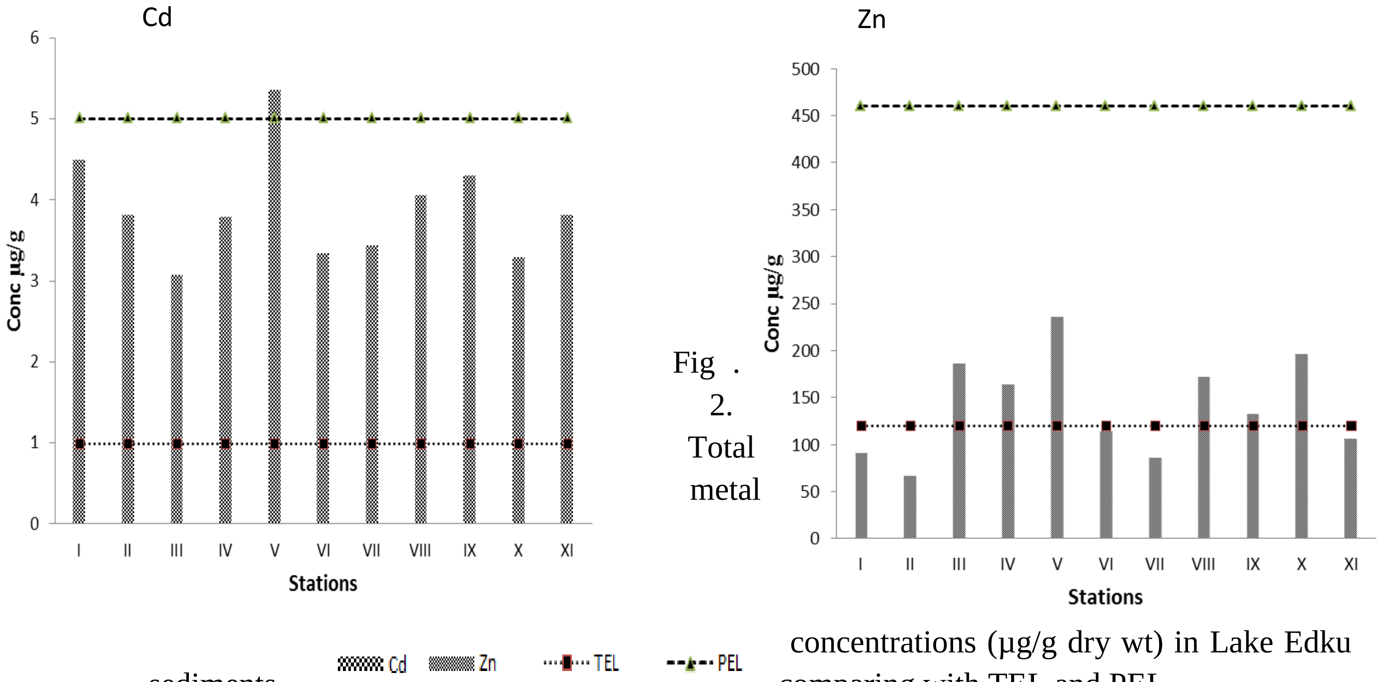

Fig . 2. Total metal concentrations (µg/g dry wt) in Lake Edku sediments comparing with TEL and PEL

From the previous results, more information about the origin of contaminants, their reaction pathways, bioavailability and possible remobilization are required.Samples were subjected to sequential chemical extractions to identify different fractions of metals. This indicatesvaluableinformationabout the geochemical mode of the metals retention and their environmental behavior.

Table 3 : Comparing the data recorded in the present study with the previous work in lagoons (µg/g dry wt).

Name of lakes	Metal concentration µg/ g dry wt		references
	Zn	Cd	
Edku	67.02 - 236.18	3.07-5. 36	Present study
Edku	35.9 - 194	0.157 – 2.939	El Zokm et al., 2015
Edku	650	-	El Said et al., 2014
Edku	276	2.11	Gu et al., 2013
Burallus	121	1.48	Gu et al., 2013
Burallus	267.91	10.4	Masoud et al., 2011
Burallus	317	1.47	Saeed& Shaker, 2008
Manzala	101	1.61	Gu et al., 2013
Bardawil	2.67 – 70.8	0.7 – 24.75	Abdo, 2005

3.3.Metals fractionation

Figure (3) shows the Results of chemical fractionationof Cd and Zn. It is clear that significant difference of the distribution patterns of Zn and Cd in sampling sites.

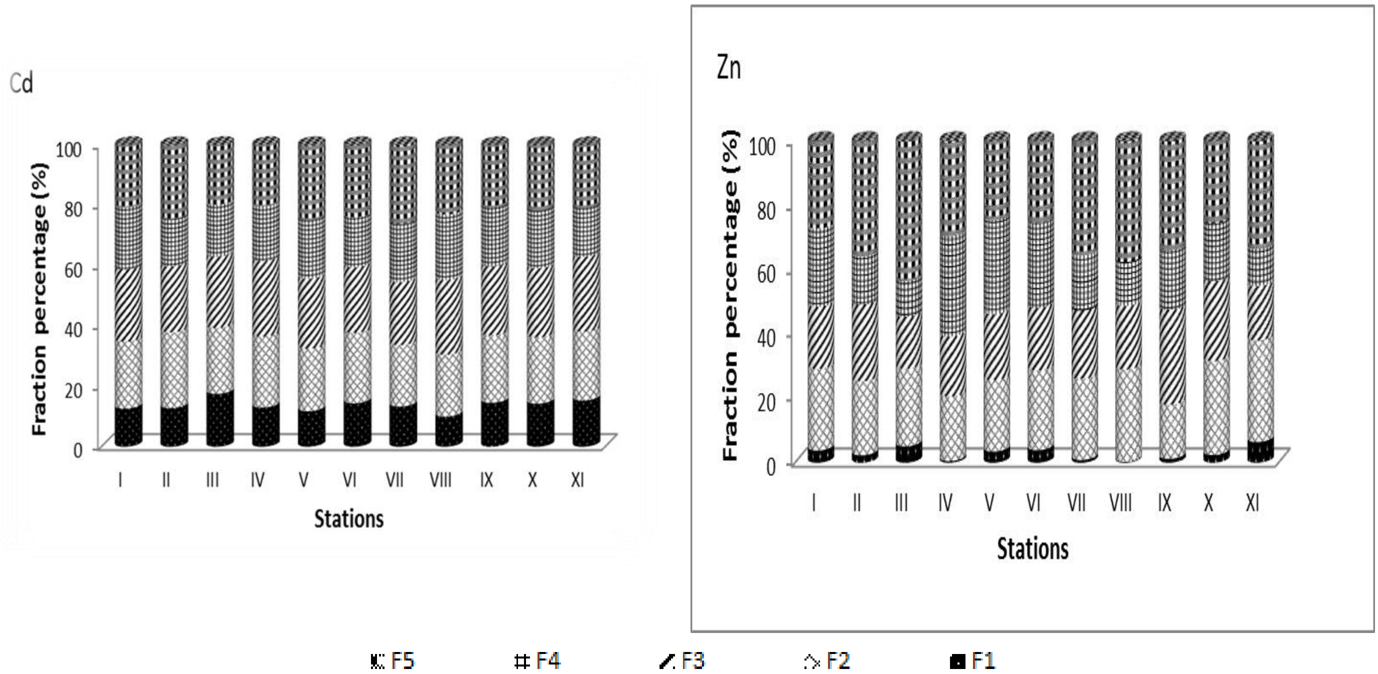

Fig . 3. Percentages of Cd and Zn in each fraction to the sum of fractions

In this study, the major portion of Zn is associated to residual fraction in most stations, ranging from 27.1% to 43.49% indicating that Zn isassociated strongly with crystalline structure of the minerals , it is stable under natural conditions and the ability of transfer is low (Nemati et al., 2009). Zinc has an appreciable portion associated with organic fraction (11.09 - 31.21%) and the content of this fraction increase in the central region (stations IV - VI) which have high content of organic matter (Table 1). Significant portions were measured in oxidizable and carbonate fractions (16.81%- 31.58% and 16.11%- 29.51% respectively). Moreover, a minor portion of Zn (less than 6.43%) is bound to exchangeable, because clay minerals, organisms and silicates can absorb Zn easily from aquatic environment.(wegbueetal.,2007). These results agree with the references (Pizarro et al., 2003 andXiaoling et al., 2016).

Relatively higher percentages averaging 23.17 % and 22.38 % of Cd were obtained in oxide and residual forms. It was reported that Fe and Mn oxides have a higher affinity of Cd than clay minerals (Alloway, 1995). High content of Cd in carbonate and bond to organic forms were reported (22.18 % and 18.52%) respectively. The ionic radius of Cd (0.97˚A) is as similar as Ca (0.99˚A), this lead to precipitate of Cd with carbonates and incorporate into the calcite lattice to give solid solutions of $Cd\alpha Ca1\alpha CO_3$. On the other hand, Cadmium form complexes with functional groups in natural organic matter (Karlsson, T., 2005). Also, high contribution of exchangeable fraction (9.97 % - 17.53%) revealing the anthropogenic source of Cd and high bioavailability. The significant levels of Cd may be attributed to agriculture wastewater where phosphate fertilizer that mostly contains Cd. In addition, rapid rise in petro- refineries and fertilizer manufacturing industries is the major source of Cd pollution in the lake.In contrast, the results recorded by Xiaolinget al.(2016) showed that the major portion of Cd was presented as exchangeable fraction of sediment samples collected from three adjacent regions of the yellow river, China due to the anthropogenic activities, mainly industrial.

3.4 .Statistical analysis

Correlation matrix is performed at confidence limit 95% and $p < 0.05$ (Table 4).It was carried out on the all determined variables in the Lake Edku sedimentsto describe the behavior and the association of extracted metals with the other parameters.A positive correlation betweenTOM% and pan % (r =0.531) revealed that concentratedorganic matter in fine grained. Previous researches indicate that particle size influences the distributions of TOM, where,OM associated strongly with fine sediment, while low OM associated with coarse sediment (Armid et al.,2014). Positively weak correlation (r = 0.251) was observed between total Zn and total Cd,reflecting different sources ofboth metals such as industrial and agricultural effluents from drains.Positively correlation (r = 0.493 and r = 0.334) observed between CO_3^{-2} and bond to carbonate fraction for Zn and Cd respectively.

4. Conclusion

This study aimed to determine total concentrations and different chemical forms of zinc and cadmium in sediment collected from Lake Edku, to assess the bioavailability and evaluate the ecological risk. The resultsshowed that the concentrations of Zinc and Cd in sediments of the Lake arehigher than the back ground levels. The I_{geo} values revealed an unpolluted status for Zn and moderately to highly pollute for Cd.According to the values of (CF), sediments of Lake Edku are very highly contaminated with Cd (CF >6) and moderately contaminated with Zn, suggesting that more anthropogenic Cd has been imported into the lake. The mean concentration of Cd lies between TEL and PEL (TEL < Cd concentration < PEL) which represent a range of concentrations within which adverse effects may occasionally occur for sensitive organisms, but only a slight risk may have taken place.The speciation of metals in the sediment samplesindicated that they are bound tofive fractions

Table 4:Correlation matrix of all determined variables in the Lake Edku sediments.

	F1_Zn%	F2_Zn%	F3_Zn%	F4_Zn%	F5_Zn%	F1_Cd%	F2_Cd%	F3_Cd%	F4_Cd%	F5_Cd%	T-Cd	T-Zn	TOM%	CO_3%	Sand %	Pan %	pH
F1_Zn%	1																
F2_Zn%	0.407	1.000															
F3_Zn%	-0.501	-0.457	1.000														
F4_Zn%	-0.225	-0.468	0.003	1.000													
F5_Zn%	-0.026	0.012	-0.162	-0.769	1.000												
F1_Cd%	**0.604**	-0.017	-0.139	-0.339	0.299	1.000											
F2_Cd%	0.189	-0.212	0.104	0.066	-0.057	0.265	1.000										
F3_Cd%	0.128	0.201	-0.436	0.035	0.057	-0.133	-0.015	1.000									
F4_Cd%	-0.546	-0.049	0.218	0.160	-0.114	-0.587	-0.745	0.219	1.000								
F5_Cd%	-0.269	0.071	0.139	0.101	-0.167	-0.476	-0.228	-0.684	0.001	1.000							
T-Cd	-0.069	-0.301	0.165	0.451	-0.400	-0.579	-0.238	0.234	0.437	0.148	1.000						
T-Zn	-0.037	-0.048	-0.083	0.234	-0.174	-0.053	-0.464	0.365	0.348	-0.182	0.251	1.000					
TOM%	-0.245	-0.489	0.122	-0.013	0.355	0.330	-0.178	-0.558	-0.081	0.237	-0.362	0.149	1.000				
CO_3%	**0.768**	0.493	-0.252	0.072	-0.515	0.276	0.334	-0.032	-0.518	0.007	-0.012	-0.222	-0.490	1.000			
Sand %	0.274	**0.595**	-0.161	-0.258	-0.098	-0.074	-0.073	0.174	-0.046	0.045	-0.001	-0.406	-0.531	0.525	1.000		
Pan %	-0.274	-0.595	0.161	0.258	0.098	0.074	0.073	-0.174	0.046	-0.045	0.001	0.406	0.531	-0.525	-1.000	1.000	
pH	0.001	-0.290	-0.207	0.258	0.032	0.091	0.076	0.333	-0.219	-0.147	0.085	**0.711**	0.297	-0.197	-0.423	0.423	1

Relation is significant at 0.05 levels.

with different strength. The Zn profile proved its affinity for the residual fractions, indicating that Zn has associated strongly with crystalline structure of the minerals and has high stability and low ability of transfer under natural conditions.Cd was bound mostly with oxide and residual forms. Highcontribution of exchangeablefraction reflects the anthropogenic source of Cd as a result of agriculture and industrial wastewater. This reflects the bioavailability of Cd.

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