Speciation the heavy metals; Fe, Mn, Cu, Zn, Ni, Cd and Pb in the Sediment of Lake Burullus (Ramsar site), Egypt

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ABSTRACT

This study was carried out to investigate the speciation and distribution of the heavy metals; Fe, Mn, Cu, Zn, Ni, Cd and Pb in the sediments of Lake Burullus, Egypt. The mean metal concentrations of the sediments followed the order of Fe >Mn> Zn > Cu > Ni >Pb> Cd.

The evaluated contamination with these metals was revealed moderate to strong ecological risk. According to applying index of the potential ecological risk index (RI), risk assessment code (RAC) and modified risk assessment code (mRAC); the risk levels showed the following order Cd = Pb>Mn> Ni > Zn > Cu > Fe.

Considering the metals speciation, high levels were found in the residual fraction for Fe, Cu, Zn and Ni; while were in Fe-Mn hydroxide fraction for Cd and Pb, and in carbonate fraction for Mn.

All metals showed enrichment during summer at stations of the southeastern parts of the lake due to the heavy discharge of contaminants from shakhloba, kotichener and Hoksa drains. Therefore, monitoring of contaminants discharge and determination of heavy metal and other pollutants in the water and sediments and its fractionation at Lake Burullus should be introduced for future and management strategies.

Key words: Lake Burullus, sediments, heavy metals, speciation.

INTRODUCTION

Heavy metal contamination is an environmental problem today inthe world due to their toxicity, widespread sources and accumulativebehaviors (Li *et al*., 2014; Xu *et al*., 2014; Gu *et al*., 2015; Liu *et al*., 2015).Measurements of total concentration of heavy metals provide inadequate information to allow us to assess the bioavailability and toxicity of the metal because heavy metals are distributed over the various chemical compounds and its re-mobilization ability. Therefore, the use of sequential extractions furnishes detailed information about the origin, mode of occurrence, biological and physicochemical availabilities, mobilization, and transport of heavy metals will be known (Tessier *et al*., 1979).Previous studies found that the translocation ability is indicated bythe acid-soluble fractions, and the bioavailability of heavy metals is thesum of acid-soluble fractions, reducible fractions and oxidizable fractions, while the residual fractions are not available (Ma *et al*., 2016). Shokr *et al*. (2016) confirmed that the sediments adjacent to lake represent a hazard to human life in the area. This could be caused byinfiltration of irrigation water through the studied area. In this context, El-Amier *et al*. (2017)

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stated that the lake sediments work asimportant sources of different toxic pollutants such as heavy metals, which in turn accumulate in aquatic organisms through food chains.

Accordingly, this work designed to determinethe different physical and chemical forms of heavy metals and their bioavailability by using sequential extraction in Lake Burullus sediments.

MATERIALS AND METHODS

Study area

LakeBurullus is one of Egypt's most important wetlands offering a wide range of ecosystem services and valuable biodiversity, the wetland was declared as a natural protectorate in 1998 under the Egyptian protectorate law 102/1983. It is also one of Egypt four Wetlands of international importance (Ramsar sites). Lake Burullus is the second largest of the Egyptian northern lakes along the Mediterranean coast, It is located in the central part of the northern shoreline of the Nile Delta between longitudes 30º 30'–31º 10' E and latitudes 31º 35'–31º 21' N, 70 km west of Damietta branch. The lake is shallow, with a depth varies between 40 cm near the shores and 200 cm near the sea outlet. The lake surface area is 410 km2. The brackish lake salinity levels range from 2.1% in the west to 17.2% in the north, as it's separated from the Mediterranean Sea by a strip of land covered with sandbars and sand dunes of different width and height, with one connection through an opening outlet in its northeastern corner known as Boughaz. The lake is located in Kafr El Sheikh Governorate, Egypt (Kassas, 2002; Shaltout and Khalil, 2005; Basiony, 2014; Darwish, 2017).

Sampling

Twelve sediment samples were collected from different stations atLake Burullusin summer, 2015 (Fig. 1).Sediment samples were collected using Van Veen Grab coated with polyethylene sampler according to Amini Ranjibar (1998). The description of sampling stations is shown in Table (1).All samples were transferred into an ice box to the Chemical laboratory, Baltim Research Station for Aquatic Resources, National Institute of Oceanography and Fisheries (NIOF) for more analysis.

Table (1): The latitudes and longitudes of sampling location inside Lake Burullus.

Station No.	Location	Latitude (N)	Longitude (E)
1- Br1	Burullus East	31˚ 33` 29.9˝	31˚ 04` 25.3˝
2- Br2	Boughaz	31˚ 43` 26.0˝	30˚ 59` 03.0˝
3- Br3	Belak	31˚ 32` 58.0˝	30˚ 58` 22.0˝
4- Br4	Front No. 7	31˚ 28` 02.0˝	30˚ 56` 38.0˝
5- Br5	El-Zanka	31˚ 28` 52.0˝	30˚ 48` 37.0˝
6- Br6	Tawila	31˚ 27` 13.0˝	30˚ 45` 18.0˝
7- Br7	Shakhloba	31˚ 24` 25.0˝	30˚ 45` 38.0˝
8- Br8	Mastroh	31˚ 29` 09.0˝	30˚ 45` 24.4˝
9- Br9	AbuAmer	31˚ 25` 41.0˝	30˚ 40` 40.2˝
10- Br10	The westmiddleBerka	31˚ 24` 17.0˝	30˚ 37` 03.0˝
11- Br11	Hoksa	31˚ 23`15.5˝	30˚36` 15.3˝
12- Br12	Brinbal	31˚ 24` 06.3˝	30˚ 35` 00.4˝

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Fig. (1): Map showing sampling stations in Lake Burullus.

Metals analyses:
Analyses of total heavy metals in sediments

Analyses of total metals in sediments were done as described by Oregioni and Aston (1984). Seven heavy metals (Fe, Mn, Cu, Zn, Ni, Cd and Pb) were measured in the digestionextract using Atomic Absorption Spectrophotometer (ASS, GBC, model 932), and results were expressed as µg/g.

Speciation

The sequential method used for the speciation of heavy metals was carried out according to Tessier *et al*. (1979). One gram sedimentsamples (dry weight) were conducted in centrifuge tubes (50 ml polypropylene) was added to minimize losses of solid material. The metal fractionations had been preceded in the following fractions:

I) Exchangeable:

The sediment was extracted at room temperature for 1h with 10 ml sodium acetate solution (1M) adjusted to pH 8.2 with continuous agitation for 6h.

II) Bound to carbonates:

The residue from (I) was leached at room temperature with 10 ml sodium acetate (1M) adjusted to pH 5 with acetic acid. Continuous agitation was maintained for about 6h in order to complete extraction.

III) Bound to Fe-Mn oxides:

The residue from (II) was extracted with 20 ml of 0.04M Hydroxyl-amine hydrochloride (NH$_2$OH. HCL) in 25% (v\v) acetic acid. The later experiment was performed at 96±3 ℃ with occasional agitation for about 6h to complete dissolution of the free iron-oxides.

IV) Bound to organic matter:

To the residue from (III), added 3 ml of 0.02M HNO_3 and 5 ml of 30% H_2O_2 adjusted to pH 2 with HNO_3, and the mixture was heated to 85 ± 2°C for 2h with occasional agitation. A second 3 ml aliquot of 30% H_2O_2 (pH 2 with HNO_3) was added and the sample was heated again to 85$\pm$2 °C for 3h with intermittent agitation. After cooling, 5 ml of 3.2M ammonium acetate in 20% (v\v) HNO3was added and the sample was diluted to 20 ml and agitated continuously for 30 min. The addition of ammonium acetate is designed to prevent adsorption of extracted metals on the oxidized sediment.

V) Residual:

The residue from (IV) was digested with a mixture of HNO_3, $HCLO_4$ and HF acids with a ratio of 3:2:1 in previously cleaned and dried Teflon beaker, then evaporated to near dryness at 80 °C. After complete digestion, the residue was transferred to a 25 ml volumetric flask with 0.1M HCL.

Between each successive extraction, separation was affected by centrifuging at 6000 rpm for 10 min. the supernatant was removed with a pipette and analyzed for heavy metals, whereas the residue was washed with 8 ml of deionized water, after centrifugation for 10 min. Finally, the concentrations of heavy metals in each fraction were measured using AAS (GBC, model 932) at National Institute of Oceanography and Fisheries, Red Seabranch, Hurghada.

Heavy metals indices in the sediments of Lake Burullus

The potential ecological risk index (RI) was widely used toquantitatively assess the ecological risk induced by heavy metals (Hokanson, 1980; Chen *et al*., 2014). It was calculated by the following formula:

$$RI = \sum_{i=1}^{n} \frac{Ti \times Ci}{Cb}$$

Where Ti is the toxic-response factor for a given metal (Zn = Mn = 1,Pb = Cu = Ni = 5and Cd = 30) (Hokanson, 1980;Chen *et al*., 2014; Cao *et al*., 2015); Ci represents the concentration of heavy metals in the sample; and Cb represents the background value ofheavy metals in sediments of Lake Burullus. The potential ecologicalrisk index wasdivided into: low ecological risk (RI<150), moderateecological risk (150<RI<300), strongecological risk (300<RI<600) and extreme ecological risk (RI>600) (Hokanson, 1980).

Assessment of mobility and availability of heavy metals

The mobility and availability of heavy metals were assessed using the RAC and mRAC indices. The RAC is defined as follows (Perin *et al*.,1985):

$$RAC = \frac{F1 + F2}{Ct} \times 100$$

Where F1 and F2 are the concentrations of exchangeable (EXC) and carbonate-bound (CA) metal fractions, respectively, and Ct is the sum of the contents of the five metal fractions. The sum of the EXC and CA metal fractions represents the amount of loosely bound heavy metals (LOS). The risk level classifications of the RAC was divided into: No risk (<1), low risk(1−10), medium risk (11−30), high risk (31−50),very high risk (>50).

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The mRAC considers the aggregation and toxicity of the metals and is defined as follows (Saeedi and Jamshidi-Zanjani, 2015):

$$mRAC = \frac{\sum_{i=1}^{n} TriRACi}{\sum_{i=1}^{n} Tri}$$

Where RAC is the risk assessment code and Tr is a toxic factor of themetal according to defined values (Hokanson, 1980). Categories of the modified risk assessment code (mRAC) were divided into: <1 nopotential adverse effect, $1 \leq mRAC > 10$ low potential adverse effect, $11 \leq mRAC < 30$ medium potential adverse effect,$31 \leq mRAC < 50$ high potential adverse effect and ≥ 50 very high potential adverse effect.

Statistical analysis

The means and standard deviations were tested for significance (*P≤0.05*) using ANOVA analysis. All statistical analyses were done using the computer program of SPSS Inc. (2007, version 16.0 for Windows XP).

RESULT AND DISCUSSION

Iron (Fe)

Total Fe in sediments ranged from 23133 at Burullus Eaststation to 39752µg/g at front no.7 station with a mean of 32457 µg/g (Table 2).Results indicated that Fe levels were within those (560- 34610 µg/g) findings by Akan *et al*. (2012), Basiony (2014), El-Alfy (2015) and Darwish (2017), and lower than those observed by Saeed and Shaker (2008), Chen *et al*. (2009) and Masoud *et al*. (2011). It's obvious that the maximum values of iron in the sediment of Lake Burullus were observed nearby drains which are more than the EPA (2002) limit and (WHO, 2004), where the guideline value is 15 µg/g.The increment in Fe concentration during summer season in sediments may be attributed to different industrial wastes as paints factories, agricultural and domestic effluents.

Results of Fe speciation (µg/g) in sediment samples collected from Lake Burullus are presented in Table (2) and Figure (2). Fe-exchangeable fraction (F1) in sediment samples ranged between ND –587µg/g (represented 0.0 to 1.61%);Fe-carbonate fraction (F2) was not recorded in all stations (0.0%); Fe-Mn oxides fraction (F3) ranged between 1857 – 14869µg/g (7.2 to 42.47%); Fe-organic matter fraction (F4) ranged between ND– 13323µg/g (0.0 to 33.32%); whileFe-residual fraction (F5) was ranged between 18305 and24955µg/g (61.18 - 79.78%).

With regard to the effect of Burullus stations on Fe-speciation during summer, the most polluted stations with F5, F3, F4, and F1 were front no.7, Boughaz, Abu Amer, and lake west,respectively, while F2 was not found in Lake Burullus sediments. In general, Fe-speciation in sediments samples in Burullus stations was taken the following order: F5> F3>F4> F1and F2 was didn't detect. The speciation of Fe in the present study is in agreement with those reported by Okbah *et al.* (2005)who found thatresidual F5 (820.44 ppm, 32.74%) > Organic form F4 (818.26 ppm, 32.65%) >Fe-Mn oxides F3 (765.21 ppm, 31.47%) > Carbonate F2 (23.94 ppm, 0.98%) >Exchangeable F1 (3.86 ppm, 0.16%).Saleh (2006) reported that sediment samples followed the order of F3 (2056.89 ppm, 81.51%) > F5 (449.68 ppm, 17.82%) > F4 (11.80 ppm, 0.47%) > F2 (3.47 ppm, 0.14%)> F1 (1.74ppm, 0.07%). The speciation of Fe in the present study is disagreement with those reported by El-Gharabawy (2009), the average values and the percentage of Fe concentration in the five fractions in Damietta Harbor were followed the order:

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F3 (2244.34 ppm, 81.26%) > F5 (465.04 ppm, 16.84%) > F4 (46.13 ppm, 1.67%) > F1 (3.94 ppm, 0.14%) > F2 (2.50 ppm, 0.09%) for bottom samples.

Table 2: Concentrations of total iron and its speciation form(µg/g) in sedimentsof Lake Burullus.

Station	Total	Exchangeable	Carbonate	Hydroxide	Organic	Residual
Burullus East	23133	138	ND	3909	520	18369
Boughaz	36121	ND	ND	14869	570	19638
Belak	33125	102	ND	4715	5246	23023
Front No.7	39752	58	ND	5571	10278	24955
Zanaka	38251	198	ND	11161	4775	22603
Tawela	29231	113	ND	3329	4302	21916
Shakhloba	27140	126	ND	4149	ND	23279
Mastroh	31254	ND	ND	5114	7449	18305
Abu Amer	38890	ND	ND	4769	13323	21823
West Berka	37125	587	ND	3783	8218	23719
Hoksa	25248	545	ND	1857	ND	23373
Brinbal	30215	390	ND	4090	5940	20351
Min.	23133	ND	ND	1857	ND	18300
Max.	39752	587	ND	14869	13323	24955
Mean±SD	32457±562	188±207	ND	5610±367	5052±431	21779±2158

Manganese (Mn)

In aquatic environment, manganese toxicity is slight to moderate and is influenced by several factors such as water hardness, salinity and pH.In sediments, total Mn ranged from 2020 at Boughazto 9152µg/g at front no.7 with mean value of 4153 µg/g (Table 3). These results are higher than(414.80 µg/g)reported by Darwish (2017) in Lake Burullus. The increase level may be due to the drainage of agriculture, industrial and sewage wastewater from drains as well as, its high clay content during summer.

Data of Mn speciation in sediment samples (µg/g) obtained from Lake Burullus are presented in Table (3) and Figure (2). Mn-exchangeable fraction (F1) ranged between ND – 319µg/g (0.0 to 3.46%);Mn-carbonate (F2) ranged from 286 to 4485 µg/g (13.8% to 48.6%); Fe-Mn oxides (F3) showed a rangeof 137 –2690µg/g (6.6 to 29.2%);Mn-organic (F4) ranged between ND– 392µg/g (0.0 to 11.16%); andMn-residual (F5) varied from 369 to1649µg/g (10.6 - 79%).With regard to the effect of Burullus stations on Mn-speciation during summer, the most polluted stations with F2, F3, F5,F4 and F1 were at front no.7, Boughaz and Tawela, respectively. In general, Mn-speciation in sediment samples in Burullus stations was taken the following order: F2> F3>F5> F4> F1. The speciation of Mn in the present study wasdisagreed with those reported by (Saleh, 2006)who found that F5 (234.47 ppm, 71.07%) > F3 (45.12 ppm,13.68%) > F4 (25.63 ppm, 7.77%) > F2 (23.27 ppm,7.05%) > F1 (1.44 ppm, 0.44%). El Gharabawy (2009) reported that the average values and the percentage of Mn concentration in the five fractions atDamietta Harbor followed the order: F3 (454.71 ppm, 64.32%) > F5 (184.22 ppm, 26.06%) > F4 (60.53 ppm, 8.56%) > F2 (4.52 ppm, 0.64%) > F1 (2.92 ppm, 0.41%) for bottom sediment samples.

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Table 3: Concentrations of total manganese and its speciation form (µg/g) in sediments of Lake Burullus.

Station	Total	Exchangeable	Carbonate	Hydroxide	Organic	Residual
Burullus East	2050	64	756	297	0.5	996
Boughaz	2020	ND	286	137	ND	1649
Belak	3617	43	1868	723	105	960
Front No.7	9152	319	4485	2690	390	1344
Zanaka	5088	22	2405	1848	180	693
Tawela	3415	35	372	1890	392	823
Shakhloba	6350	111	3355	1732	137	1029
Mastroh	3430	ND	1436	1597	86	369
Abu Amer	3520	282	1306	1121	294	579
West Berka	5750	225	2480	1691	246	1200
Hoksa	2220	0	780	643	ND	852
Brinbal	3230	63	1173	1162	220	673
Min.	2020	ND	286	137	ND	369
Max.	9152	319	4485	2690	392	1649
Mean±SD	4153±210	97±114	1725±126	1294±750	171±142	931±351

Copper (Cu)

In sediments, total Cu ranged from 55at Boughazto 530 at AbuAmer station with a mean of 156 µg/g (Table 4).The present results are higher than those findings by El-Ghobashy*et al.* (2001); Akan *et al.* (2012); Basiony (2014); El-Alfy (2015) and Darwish(2017), they reported that the Cu concentration in sediment samples ranged from 3.93 to 234.99 µg/g.The maximum values in Lake Burullus were higher than the EU (2002) limit (140 µg/g), and also higher than the limit of EPA (2002) with a value of 25 µg/g.The higher Cu concentrations may be due to the use of fertilizers and other chemicals in agriculture.In addition, copper is widely used in electrical wiring, roofing, various alloys, pigments, cooking utensils, piping and in the chemical industry (WHO, 2004).

Results of Cu speciation (µg/g) are shown in Table (4) and Figure (2).Cu-exchangeable fraction ranged between 0.67 -4.21µg/g (0.46 to 6.87%) of sediments collected from Lake Burullus;Cu-carbonate fraction ranged from 0.17 to 23.55µg/g (0.03 to 38.48%); Fe-Mn oxides fraction was found in the range of 0.68 – 7.66µg/g (1.1 to 12.52%);Cu-organic matter fraction ranged between 14.8 – 80.9µg/g (23.76 to 132%); whileCu-residual fraction ranged between 29.8 -162.8µg/g (47.83 - 218%).according to the effect of Burullus stations on Cu-speciation, the most polluted stations with F5, F4, F2, F3and F1 were front no.7 followed by Boughaz station, respectively. In general, Cu-speciation in Burullus stations was taken the following order: F5> F4>F2> F3>F1. The speciation of Cu in the present study was incomparable with those reported by Saleh (2006),who reported that the average values and the percentage of Cu concentration in the five fractions were followed the order:F4 (11.44 ppm, 47.71%) > F5 (9.99 ppm, 41.65%) > F3 (1.79 ppm, 7.47%) > F2 (0.73 ppm, 3.04%) > F1 (0.03 ppm, 0.13%).Okbah*et al*. (2005)found that the mean values and percentage of Cu concentration in the five fractions were followed the order: F5 (189 ppm, 68.69%) > F4 (50.66 ppm, 18.41%) > F3(20.94 ppm, 7.61%) > F2 (8.77 ppm, 3.19%) > F1(5.79 ppm, 2.11%). While, El Gharabawy (2009) reported that the average

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values of Cu concentration in the five fractions were followed the order: F3 (454.71 ppm, 64.32%) > F5 (184.22 ppm, 26.06%) > F4 (60.53 ppm, 8.56%) > F2 (4.52 ppm, 0.64%) > F1 (2.92 ppm, 0.41%) for bottom sediment samples.

Table 4: Concentrations of total copper and its speciation form (µg/g) in sediments of Lake Burullus.

Station	Total	Exchangeable	Carbonate	Hydroxide	Organic	Residual
Burullus East	70	0.77	2.65	0.68	14.8	29.8
Boughaz	55	4.21	23.55	7.66	80.9	44.5
Belak	125	2.88	6.70	6.37	37.1	99.1
Front No.7	65	3.07	8.60	4.97	22.2	162.8
Zanaka	74	2.12	5.53	5.49	41.8	103.1
Tawela	135	0.67	0.87	5.23	43.5	82.5
Shakhloba	194	1.12	7.19	4.20	33.7	89.6
Mastroh	204	1.22	3.35	4.35	42.3	44.5
Abu Amer	530	1.35	0.17	5.06	46.4	73.9
West Berka	160	3.00	5.38	5.36	31.9	81.5
Hoksa	130	1.18	4.29	1.70	16.9	104.6
Brinbal	140	0.95	6.38	2.16	49.3	69.3
Min.	55	0.67	0.17	0.68	14.8	29.8
Max.	530	4.21	23.55	7.66	80.9	162.8
Mean±SD	156±127	1.90±1.1	6.2±6	4.40±2	38.3±17.5	82±35

Zinc (Zn)

Total Zn in sediments of Lake Burullus ranged from 351 at Tawela to 980 at Zanka with mean value of 563 µg/g (Table 5).The highest concentration of Zn may due to discharging from El-Gharbia drain which can explain the increase of Zn in Lake Burullus. This drain discharges industrial and agriculturalwastewater directly into the lake. The industrial wastes mostly derived from El-Mahalla El-Kobra factories. The distribution of zinc inbottom sediments shows an increase toward the north and northeastern stationsof the lake. The present results agree with El-Badry and El-Kammar(2018).

Data of Zn speciation (µg/g) in sediments samples of Lake Burullus are presented in Table (5) and Figure (2). Zn-exchangeable (F1) ranged between ND- 5.1µg/g (0.0 to 1.0%) of sedimentsamples; Zn-carbonate (F2) was in the range of 2.0-163 µg/g (0.6% to32.4%); Fe-Mn oxides (F3) varied between 33 - 705µg/g (6.56 to 71.3%); Zn-organic (F4) ranged from 5 to 278µg/g (1.1 to 39%); while Zn-residual (F5) showed a range of 231 – 369µg/g (23.4 - 78%). According to the effect of Burullus stations on Zn -speciation during summer, the most polluted stations with F3, F5, F4,F2 and F1 were Zanka, Belak, Brinbal, Burullus East and Mastrouh, respectively. Generally, Zn-speciation in sediments samples from Burullus stations was in the following order: F3> F5>F4> F2> F1. The results showed differedfrom those of El-Badry and El-Kammar (2018), who found that the average data of zinc speciation in lake Burullus sediments were in the following order of abundance: residual > organic > exchangeable > carbonate > Fe-Mn oxy-hydroxides.

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Table 5: Concentrations of total zinc and its speciation form (μg/g) in sediments of Lake Burullus.

Station	Total	Exchangeable	Carbonate	Hydroxide	Organic	Residual
Burullus East	510	1.7	163	33	47	258
Boughaz	734	3.6	114	164	207	236
Belak	460	4.9	22	53	24	369
Front No.7	454	3.0	21	83	37	312
Zanaka	980	3.1	13	705	37	231
Tawela	351	3.1	2	44	33	278
Shakhloba	940	4.4	26	586	42	303
Mastroh	480	5.1	7	200	24	261
Abu Amer	375	2.1	7	37	28	306
West Berka	354	4.3	14	39	34	274
Hoksa	428	0.0	33	120	5	282
Brinbal	695	0.2	26	115	278	291
Min.	351	0.0	2	33	5	231
Max.	980	5.1	163	705	278	369
Mean±SD	563±220	3.0±1.7	37±49	182±225	66±85	283±37

Nickel (Ni)

Table (6) shows the total concentration of Ni in Lake Burullussediments, which ranged from 50 at Mastroh to 195μg/g at Shakhloba, Zanka and front no. 7 stations with mean value of 139 μg/g. The results indicated that Nilevels were higher than those (40.91 and 84.82 μg/g) findings by Basiony (2014) and Darwish (2017), respectively. The present data of Ni exceeded the limits of WHO guideline value of 20 μg/g, indicating that the sediments of Lake Burullus were contaminated by nickel which may due to the effluents of the various industrial processes; i.e.chemical fertilizers and pesticides, domestic activities and dumping of municipal sewage into the drains of the lake.

Results of Ni speciation (μg/g) are shown in Table (6) and Figure (2). Ni-exchangeable (F1) varied between ND– 20.3μg/g (0.0 to 24.6%); Ni-carbonate (F2) was 5.1-28.9 μg/g (5.6%-14.4%); Fe-Mn oxides (F3) ranged from 3.2 to48.5μg/g (3.7 to 24.13%); Ni-organic (F4) was 7.9 – 30μg/g (9.29 to 23.14%); and Ni-residual (F5) ranged between 5.2 -114.1μg/g (9 – 55.6%).With regard to the effect of Burullus stations on Ni-speciation, the most polluted stations with F5, F3, F4,F2 and F1 were front no.7, Shakhloba, Abu Amer, and Burullus East, respectively. Generally, Ni-speciation in sediments of Burullus stations showed the following order: F5> F3>F4> F2> F1.

The speciation of Ni in the present study was agreed with those reported by Saleh(2006), who found that the average values of Ni in the five fractions followed the order: F5 (66.18 ppm, 66.65%) > F3 (16.35 ppm,16.49%) > F4 (13.27 ppm, 13.49%) > F2 (2.69 ppm,2.71%) > F1 (0.65 ppm, 0.66%).Okbah *et al*. (2005) reported that the average values of Ni in the five fractions were followed the order:F2 (19.49 ppm, 38%) > F4 (12.39 ppm, 24%) > F5 (11.94 ppm, 23%) > F3 (5.11 ppm, 10%) > F1 (2.47 ppm, 4.8%).

Table 6: Concentrations of total nickel and its speciation form (µg/g) in sediments of Lake Burullus.

Station	Total	Exchangeable	Carbonate	Hydroxide	Organic	Residual
Burullus East	93	20.9	22.9	3.2	7.9	30.1
Boughaz	90	10.1	5.1	22.7	8.1	45.6
Belak	170	6.7	9.0	39.1	16.2	102.8
Front No.7	195	12.2	22.9	40.4	15.8	114.1
Zanaka	195	7.7	27.3	38.0	19.9	108.0
Tawela	180	15.3	20.4	37.8	27.6	84.5
Shakhloba	195	12.9	28.9	48.5	15.7	95.0
Mastroh	50	13.7	13.9	10.2	14.6	5.2
Abu Amer	120	0.0	7.3	27.2	30.0	65.1
West Berka	125	8.6	16.8	23.6	16.8	57.8
Hoksa	150	8.5	16.9	20.0	16.4	95.2
Brinbal	110	5.5	9.8	33.9	13.3	58.2
Min.	50	0.0	5.1	3.2	7.9	5.2
Max.	195	20.9	28.9	48.5	30.0	114.1
Mean±SD	139±48	10.2±5.3	16.7±7.9	28.7±13.3	16.8±6.5	71.7±33.8

Cadmium (Cd)

Table (7) shows the level of total concentration of Cd in sediments from Lake Burullus, which ranged from ND at Belak and Tawela to 2.6µg/g at Boughaz station with a mean of 0.66 µg/g.These results were similar to the findings given by El-Ghobashy *et al.* (2001), Akan *et al.* (2012), Basiony (2014), El-Alfy (2015) and Darwish (2017), who reported that Cd concentration in sediments samples ranged between0.012 and 47.8µg/g. In addition, Cd mean value(0.66 µg/g) in the present study was lower than the international permissible level (6 µg/g) reported by WHO (2004). The increase value of Cd in some stations is attributed to the enrichment of discharged water by cadmium, where it is present as an impurity in several products, including phosphate fertilizers and detergents (Greaney, 2005).

Results of Cd speciation (µg/g) in sediments are shown in Table (7) and Figure (2). Cd-exchangeable fraction ranged between ND -0.40µg/g (0.0 - 39.22%); Cd-carbonate fraction was ND -0.69 (0.0-30.8%); Fe-Mn oxides fraction ranged between ND -0.6µg/g (0.00 - 26.78%); Cd-organic fraction ranged fromNDto 0.56µg/g (0.0 - 70.8%); while Cd-residual fraction varied between ND and0.95µg/g (0.00 - 42.4%) of sediments obtained from Lake Burullus.According to the effect of Burullus stations on Cd-speciation, the most polluted stations with F5, F2, F3,F4 and F1 were Boughaz, Shakhloba and Abu Amer, respectively. In general, Cd-speciation in sediments samples at Burullus stations was taken the following order: F3 >F2> F4>F5 ≥F1. The speciation of Cd in the present study agreed with those reported by Saleh (2006) who recorded that the average values of Cd in the five fractions were followed the order: F5 (66.18 ppm, 66.65%) > F3 (16.35 ppm,16.49%) > F4 (13.27 ppm, 13.49%) > F2 (2.69 ppm,2.71%) > F1 (0.65 ppm, 0.66%). Okbah *et al.* (2005) found that the average values Cd in the five fractions followed the order: F2 (19.49 ppm, 38%) > F4 (12.39 ppm, 24%) > F5 (11.94 ppm, 23%) > F3 (5.11 ppm, 10%) > F1 (2.47 ppm, 4.8%).

Speciation the heavy metals; Fe, Mn, Cu, Zn, Ni, Cd and Pb in the Sediment of Lake Burullus (Ramsar site), Egypt

Table 7: Concentrations of total cadmium and its speciation form (µg/g) in sediments of Lake Burullus.

Station	Total	Exchangeable	Carbonate	Hydroxide	Organic	Residual
Burullus East	0.5	0.00	0.04	0.16	0.28	0.00
Boughaz	2.6	0.00	0.69	0.60	0.00	0.95
Belak	0	0.00	0.00	0.00	0.00	0.00
Front No.7	0.26	0.00	0.12	0.04	0.00	0.00
Zanaka	0.36	0.00	0.00	0.28	0.00	0.00
Tawela	0	0.00	0.00	0.00	0.00	0.00
Shakhloba	0.67	0.17	0.00	0.06	0.56	0.00
Mastroh	0.41	0.00	0.00	0.31	0.00	0.00
Abu Amer	1.1	0.40	0.08	0.54	0.00	0.00
West Berka	0.41	0.05	0.03	0.00	0.24	0.00
Hoksa	1.3	0.34	0.48	0.23	0.00	0.00
Brinbal	0.35	0.00	0.29	0.00	0.00	0.00
Min.	0.00	0.00	0.00	0.00	0.00	0.00
Max.	2.6	0.40	0.69	0.60	0.56	0.95
Mean±SD	0.66±0.7	0.08±0.14	0.14±0.23	0.18±0.21	0.09±0.18	0.08±0.27

Lead (Pb)

Total Pb in sediments of Lake Burullus ranged from ND at AbuAmer and West middle berka to 198µg/g at Boughaz station with mean value of 43 µg/g (Table 8). These result was in agreement with El-Badry and El-kammar (2017)and Darwish (2017), they found that high concentration of Pb was in Boughaz station.The distribution of lead in sediments increases to the north where agricultural activity and the human activities increased at El-Maksaba and Mastarouh villages, as well as northeastern portion of the lake near Boughaz where fishing activity using boat engine motor giving two high concentration spots. On the other hand, the western side of thelake showed declining in lead pollution level.

Pb speciation (µg/g) in sediment samples is presented in Table (8) and Figure (2). Pb-exchangeable (F1) ranged between NDand 17.3µg/g (0.0 - 72.7%);Pb-carbonate (F2) was ND - 55.4 µg/g (0.0 - 26.94%); Fe-Mn oxides (F3) varied fromNDto 119.3µg/g (0.00 - 58%); Pb - organic (F4) showed a range ofND- 13µg/g (0.0 - 6.3%); and Pb-residual (F5) was ranged between NDand 29.2µg/g (0.00 - 38.37%).With regard to the effect of Burullus stations on Pb-speciation during summer, the most polluted stations with F3, F2, F5,F1 and F4 were Boughaz, Hoksa and Mastroh, respectively. In general, Pb-speciation in sediments samples in Burullus stations was found in the following order: F3> F2>F5> F1> F4. These resultswere slightly differed with El-Badry and El-kammar(2017), who reported that the average data of lead speciation in Lake Burullus sediments suggested thefollowing order of abundance; residual > exchangeable >carbonate > organic >Fe-Mn oxy-hydroxides.

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Table 7: Concentrations of total cadmium and its speciation form (µg/g) in sediments of Lake Burullus.

Station	Total	Exchangeable	Carbonate	Hydroxide	Organic	Residual
Burullus East	51.2	9.3	10.5	8.9	5.5	19.7
Boughaz	198	0.0	55.4	119.3	13.0	17.9
Belak	13	0.0	0.0	2.9	8.6	0.0
Front No.7	28.3	13.5	4.7	5.5	0.5	0.0
Zanaka	3.1	2.6	0.0	0.0	0.0	0.0
Tawela	1.3	0.0	1.1	0.0	0.0	0.0
Shakhloba	14.1	4.4	5.8	0.0	0.0	2.4
Mastroh	22.3	17.3	2.3	4.2	0.0	0.0
Abu Amer	0	0.0	0.0	0.0	0.0	0.0
West Berka	0	0.0	0.0	0.0	0.0	0.0
Hoksa	71.4	0.0	46.7	0.0	0.2	29.2
Brinbal	125	5.6	48.6	60.6	0.0	16.0
Min.	0.00	0.0	0.0	0.0	0.0	0.0
Max.	198	17.3	55.4	119.3	13.0	29.2
Mean±SD	43±61	4.4±6	14.6±21.8	16.7±36.5	2.3±4.4	7.1±11

RAC, mRAC and RI of heavy metals

As shown in Table (9), the RAC values were found in the range of ND–2.11 (low risk level), 11.59–54.46 (moderate to very high risk), 1.2–17.26 (low to moderate), 1.42–32.76 (low to high risk level), 5.63–51.53 (moderate to very high risk), ND–100 and ND–100 (low to very high risk) for Fe, Mn, Cu, Zn, Ni, Cd and Pb, respectively. The risk levels are ranked in the following order Cd = Pb>Mn>Ni>Zn>Cu>Fe. Form RAc, the most stations pose a relatively medium potential adverse effect except front no.7, Hoksa and Brinbal stations which are very high potential adverse effect. According to RI values, all stations are low in ecological risk except Abu Amer and Hoksa which have modrate ecological risk and Boghaz station has strong ecological risk.

The potential risk of heavy metals speciation in sediments is in part influenced by their total content, but chemical speciation in relation to the mobility of the heavy metals determines their potential risk of releasing into the water phase (Yang *et al*., 2014). The results showed that the variation of organic matter had a consistent trend in relation to RAC. Thus, we can speculate that organic matter content might be a major mediator of the ecological risk of heavy metals. It is evident that the metals in sediments are bound to different fractions with different strengths, thereby producing the inconsistent risks in an aquatic-sediment interface (Sundaray *et al*., 2011). In this study since up to 50 % of loosely bound heavy metals (exchangeable and carbonate) can be easily released into Lake Burullus water.

Speciation the heavy metals; Fe, Mn, Cu, Zn, Ni, Cd and Pb in the Sediment of Lake Burullus (Ramsar site), Egypt

Table (9): The risk assessment code(RAC),modified risk assessment code (mRAC) and the potential ecological risk index (RI) of mean values of heavy metals in the sediments of Lake Burullus during 2015.

station	RAC							mRAC	RI
	Fe	Mn	Cu	Zn	Ni	Cd	Pb		
Br1	0.60	38.80	7.02	32.76	51.53	8.33	36.73	19.76	85.91
Br2	0.00	13.80	17.26	16.23	16.59	30.80	26.95	28.15	332.85
Br3	0.31	51.66	6.30	5.69	9.03	0.00	0.00	3.34	40.02
Br4	0.14	52.06	5.79	5.26	17.09	75.00	75.21	59.97	69.95
Br5	0.51	47.14	4.84	1.63	17.42	0.00	100.00	14.18	76.03
Br6	0.38	11.59	1.16	1.42	19.23	0.00	100.00	13.21	37.72
Br7	0.46	54.46	6.12	3.16	20.80	21.52	80.95	26.71	125.69
Br8	0.00	41.17	4.77	2.43	47.92	0.00	82.35	15.50	84.41
Br9	0.00	44.33	1.20	2.39	5.63	47.06	0.00	31.96	192.73
Br10	1.62	46.30	6.59	5.01	20.55	25.00	0.00	20.36	79.97
Br11	2.11	34.29	4.25	7.50	16.18	78.10	61.37	60.08	181.97
Br12	1.27	37.56	5.72	3.69	12.68	100.00	41.44	71.39	102.55

Conclusion

Lake Burullus is the second largest of the Egyptian northern lakes along the Mediterranean coast and also is a shallow lake. It receives a huge quantity of agricultural, industrial, municipal and domestic wastewater, in addition to human activity. The present study concluded that the content of Fe, Mn, Cu, Zn, Ni, Cd and Pb in southern lake sediments hade concentrations higher than those in the northern part, which indicated the influence of dumping wastewater from different sources into the lake. According to chemical and physical forms of the studied metals and the calculated potential ecological risk index(RI), risk assessment code (RAC) and modified risk assessment code (RAC), the bottom sediments of the Lake Burullus markedly showedmoderate ecological risk to strong ecological risk. The risk levels were ranked in the following order Cd=Pb>Mn>Ni>Zn>Cu>Fe. The spatial distribution of these metals in the Lake Burullus revealed that the agricultural wastewater and industrial wastewater brought by El-Gharbia, No.7,Shakhloba and No.11 drains were the main contributors of pollution. Speciation of the studied metals suggested that the residual fraction was the main carrier of Fe, Cu, Zn and Ni; the carbonate fraction was the main carrier of Mn; while the Hydroxides fraction was the main carrier of Cd and Pb.
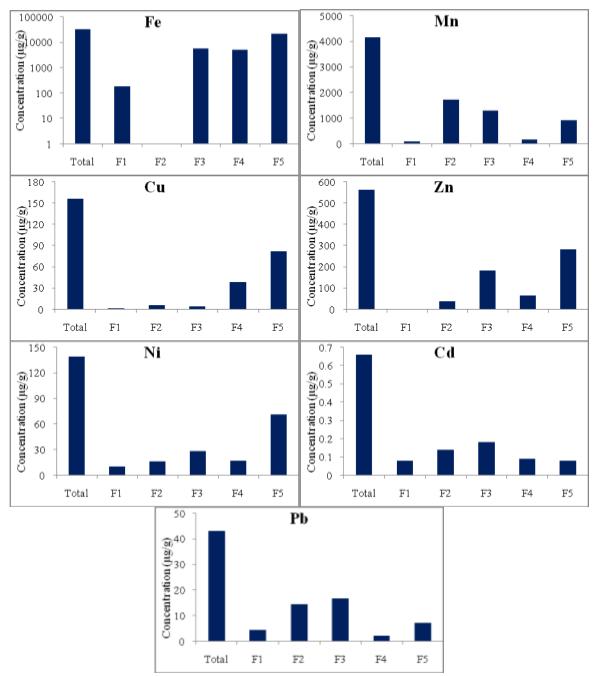
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Fig. (2): Concentration of total heavy metals and their speciation forms in sediments of Lake Burullus.

Speciation the heavy metals; **Fe, Mn, Cu, Zn, Ni, Cd and Pb in the Sediment of Lake Burullus (Ramsar site), Egypt**

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الصور الكيميائية والفيزيائية للعناصر الثقيلة ؛ الحديد،المنجنيز ،النحاس ، الزنك ،النيكل ،الكادميوم والرصاص فى رواسب القاع لبحيرة البرلس (رمسار)، مصر

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المستخلص

أجريت هذه الدراسة لتحديد الصور الكيميائية والفيزيائية للعناصر الثقيلة؛ الحديد، المنجنيز ، النحاس، الزنك، النيكل، الكادميوم، الرصاصفى رواسب القاعلبحيرة البرلس. حيث أوضحت النتائج أن متوسط تركيزالعناصر الثقيله فى الرواسبازداد تدريجيا على النحو التالى:الحديد > المنجنيز> الزنك> النحاس> النيكل> الرصاص> الكادميوم. ومن خلال دراسة الصور الفيزيائية والكيميائية لهذه العناصر أوضح كل من الحديد والنحاس والزنك والنيكل تركيزاتهم العالية داخل الشبكة البلورية للرواسب وهذ الارتفاع لا يمثل خطورة على البيئة؛بينما سجل المنجنيز أعلى تركيز له فى شكل كربونات وهذه الصورة خطرة على البيئة المحيطة؛ فى حين أن الكادميوم والرصاص سجلا أعلى تركيز فى صورة هيدروكسيدات. وبحساب مؤشرات الخطورة البيئية المختلفة (RI, RAC, mRAC) أوضحت رواسب القاع لبحيرة البرلس مستويات خطورة من متوسط حتى شديد الخطورة. وكان ترتيب العناصر طبقاً لمستوى الخطورة كالآتى: الكادميوم = الرصاص>المنجنيز>النيكل> الزنك> النحاس>الحديد. كما لاحظنا أن المحطات الجنوبية من البحيرة والمجاورة للمصارف هى الأكثر تركيزا والأكثر تلوثا بالعناصر الثقيلة بسبب الصرف الصناعى والصحى والزراعى. لذا توصى الدراسة باستمرار عملية رصد الملوثات وخاصة الصور الكيمائية والفيزيائية للعناصر الثقيلة وذلك للحفاظ على استراتيجية وإدارة بحيرة البرلس.