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Monitoring of Polychlorinated biphenyls in Surface Water Using QuEChERS Extraction Method with Gas Chromatography-micro Electron Capture Detector

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Abstract

Surface water samples were collected to measure polychlorinated biphenyls (PCBs) at two sites: Shubra-Khait and Kafer-El-Zayet districts. Gas chromatography-micro-electron capture detector (GC-ECD) was used to evaluate water samples after they were extracted using the QuEChERS method. Eleven PCB congeners were identified, and the findings demonstrated that surface water was contaminated with low concentrations of PCBs. The lowest, highest and the average of PCB concentrations at Kafer-El-Zayet, were 0.15, 5.21, and 1.124 µg L^{-1}, and at Shubra-Khait, they were 0.28, 3.58, and 0.929 µg L^{-1}. Even though it was only found in trace amounts, its cumulative nature allowed it to find its way into subterranean water, which is thought to be one of the main sources of environmental contamination. This could have an adverse effect on both human and environmental health.

Keyword: PCBs, surface water, water contamination, QuEChERS, GC-µECD

1. Introduction

Water is the main source for all forms of life and plays an important role in human life. Water covers 71% of the Earth's surface. The Nile has historically been considered the longest river in the world (Liu et al., 2009). Surface is the main natural source of fresh water in Egypt. The river provides 97% of all renewable water resources, about 70% of water used in agriculture (Abdel-Satar et al., 2017).

It gets a variety of pollutants, with industrial effluents being the main source of pollutants. Drinking or use of water polluted with pollutes residues might be has health impact on human and should not exceed international allowable limits (Radwan et al., 2019).

The governorates of El-Beheira and El-Ghrbia are located close to the Rashid Branch of the Nile. The El-Ghrbia governorate's Shubrakhit city located west the Nile, is well-known for a variety of industries, the most prominent of which are derived from fishing and agriculture as well as the textile, chemical, carpet, and electrical sectors. While the governorate of Kafr El-Zayat El-Beheira located east the Nile is host to numerous industrial facilities, chemical plants, pesticides, fertilizers, paper, oils, and soaps are just a few of the many firms and companies that operate there. Abo-Shady et al., 2017 and Mankoula et al., 2021.

Industrial and municipal wastewaters are considered the main source of water contamination is due to irresponsible or negligent practices (Schwarzenbach and Hofstetter, 2010). Leaching of PCBs through agricultural soil might be causing contamination of the aquatic systems (Jadwiga et al., 2012). Organic pollutants are globally distributed chemicals which has persistent, accumulation and toxicity Properties that affected environmental and human health (Di Bella et al., 2006). The contamination by these chemicals can reached long and far distance due to its semi-volatile properties which could carried by air to areas were never used there, and it has

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bioaccumulation and biomagnification potential (Jallow et al., 2017). PCBs is one of the most persistent organic pollutants in the ecosystem, were used in many industries, for example, in dielectric and in fluids which used for cooling capacitors and transformers (Nasr et al., 2009). Before their persistence and toxicity were investigated and determined, polychlorinated biphenyls, or PCBs, were widely used as additives in heat exchange fluids, parts for transformers and large capacitors, carbon-free copy paper, paints, and plastics. PCBs are composed of 209 congeners, the physicochemical properties and toxicity of which depend on the number and position of chlorine atoms in the molecule. (Shang et al., 2016 and Cui et al., 2020). PCBs can enter the human body through a variety of pathways. The most common one is by the consumption of contaminated foods, such as fish, seafood, and dairy products. Other pathways include dust ingestion and skin contact, which are less common, and breathing of contaminated indoor and outdoor air. These contaminants, which come from building supplies (flame-retardants, plasticizers, paints, caulking compounds, sealants, fluorescent light ballasts, etc.) and electrical equipment, have been the subject of numerous research on occupational and environmental exposure. The highest levels of PCB contamination were found in e-waste recycling facilities, indicating the necessity of implementing remediation measures in such polluted places to protect local residents' and workers' health. Moreover, workers in PCB-contaminated environments showed a strong association between PCB exposure and elevated blood PCB concentrations. Furthermore, bioaccumulation of PCBs has been linked to decreased fertility and detrimental effects on the reproductive system that can be passed on to progeny, according to recent research. More research will soon be required to determine the true consequences of low-concentration PCB exposure for extended periods of time spent in particular indoor environments and workplaces. (Montano et al., 2022). Polychlorinated biphenyl compound (PCBs) mainly contained two phenyl rings attached with 1 to 10 chlorine atoms (Andersson et al., 1999). PCBs has human health and environmental impact due to its ability to bioaccumulation and biomagnifying effects in the food chain (Di Bella et al., 2006). It has been demonstrated that prolonged exposure to these chemicals can impair reproductive function and have a variety of negative effects on the immunological, endocrine, and neurological systems. They might also be the cause of cancer. This means that there are still some safety issues due to their persistence and the fact that they build up in the food chain, particularly in animal fat. Therefore, Stockholm Convention have been banning use of polychlorinated biphenyls (PCBs) compounds because of its physiological toxicity and ecological magnification.

There are many modifications in QuEChERS method depending on the analyte and the matrix by using various combinations (amount, type, ratio) of solvents, sorbents, or salts to be suitable to determine a lot of chemical groups as PCBs (Han et al., 2016). Extraction of polychlorinated biphenyls (PCBs) from the water samples by using QuEChERS method before analysis using the Gas Chromatography has been modified by (Mathews and Sichilongo, 2016). Gas chromatography combined with micro electron capture detector (GC-μECD) or with mass spectrometry (GC-MS) was used for analysis and determination of PCBs (Kuzukiran and and Filazi 2016). The aim of this search was to monitor the traces levels of PCBs in the aquatic environment from surface water at Rosetta Branch at Kafer-El-Zayet and Shubra-Khait districts.

2. MATERIAL AND METHODS

2.1. Collection of samples

Brown glass bottles containing 2.5 L of water samples were filled with water at depth 50 cm below the water surface from 4 sites along river Nile during different seasons from two sites in Kafer-El Zayet and two sites in Shubra-Khait regions. Totally ninety-six water samples were taken over a year. Twelve samples were taken every season at different locations., then the bottles were transported to the laboratory and filtered with Whatman filter paper, to evaluate the pollutants.

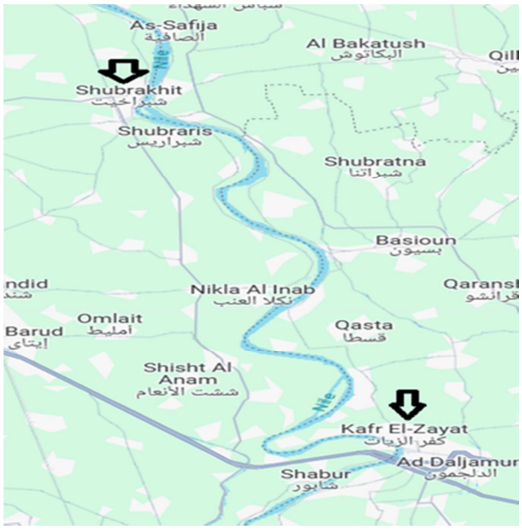


Figure 1: Sample sites of Kafer-El Zayet and Shubra-Khait regions.

2.2. Chemicals and Reagents:

Acetonitrile (HPLC grade) were acquired from Sigma Aldrich, ethyl acetate, acetone and n-hexane (HPLC grade) were purchased from SDS. Anhydrous Magnesium sulphate (MgSO4) and sodium chloride (NaCl). Primary secondary amine (PSA) was purchased from Agilent Technologies (Wilmington, DE, USA). PCBs congeners (PCB 28, PCB 52, PCB 44, PCB 70, PCB 152, PCB 101, PCB 118, PCB 105, PCB 138, PCB 180 and PCB 192) (Dr S. Ehrenstorfer, Wesel, Germany) with purity ranged from 97-99.5%, were used in preparation of stock solution in hexane which stored in a freezer at -20 ºC. Working solution of PCBs for sample fortification and calibration curve were prepared by diluting stock solutions in n-hexane.

2.3. Analytical procedures

2.3.1. Extraction of PCB's in Water Samples

A 50 mL polypropylene centrifuge tube was filled with 10 mL of water sample, and 15 mL of acetonitrile was then added. For one minute, the mixture was vortexed. Four grams of anhydrous MgSO4 and one gram of NaCl were added to the mixture, which was vortexed for one minute before being centrifuged at 5000 rpm for five minutes. A polypropylene centrifuge tube containing 200 mg PSA and 600 mg anhydrous MgSO4 was filled with two milliliters of the supernatant's upper layer. The mixture was vortexed for 30 seconds using a vortex. Next, the mixture was centrifuged at 5000 rpm for 3 minutes. After resolving the dry film with 0.5 ml of hexane, a 1 ml aliquot of the organic layer was transferred to a clean glass vial and concentrated under nitrogen gas. A micro electron capture detector (GC-μECD) was then used for analysis.

2.3.2. Instrumentation

Agilent Model 6890 equipped with a Ni63 -micro electron capture detector was used for analysis using DB-17 (30 m length x 0.32 mm) as analytical column, N2 used a flow rate of 4 ml/min as carrier gas, oven temperature was programed initial160 ℃ for 2 min, raised at 3 ℃/min. and then held at 230 ℃ for 2 min., then raised at 10 ℃ /min. to 260 ℃ and then held to 10 minutes, injector and detector temperatures were 300 ℃ and 320 ℃, respectively, total run time of 42 minutes, PAS-5 methyl silicone (30m x 0.32 mm i.d x 0.25 μm film thickness) was used to confirm the detected contaminants.

2.3.3. Quality control

The labs must demonstrate their capacity to produce method blank, precision, and recovery that are acceptable. According to Zhao et al. 2019, preliminary tests for precision and recovery were carried out, which included sample preparation and extraction. When measuring spiked samples, the level of S/N = 3:1 was used to define the limit of detection. The calibration curves linearity with five replicates covering a concentration series ranging from 0.4 to 40.0 μg L^{-1} with the correlation coefficient (R2) between 0.977 and 0.995. For the fortification sample, three levels (2, 10, and 20 μg L^{-1}) were used to calculate the percentage of recoveries, which is expressed as the ratio of the area of the standard to the area of the spiked sample. In accordance with the recovery percentage values and all data were adjusted. By comparing retention times to those of the real standard, compounds were found. to assess how matrix interferences in extracts affect the measurement of analytes and the detection of PCBs in water samples (commercial drinking water). Utilizing the calibration curve, eight duplicate blank samples spiked at concentrations of 2, 10, and 20 μg L-1 were injected in order to assess accuracy and precision. The recoveries of PCBs spiked in water samples ranged in 72.0–95.22%, and the relative standard deviation (RSD) was less than 20%. Based on a signal-to-noise ratio of 3, the limit of detection (LOD) varied from 0.2 to 1 μg L^{-1} and the 10 signal-to-noise ratios served as the base for the Limit of Quantification (LOQ) were 0.4 to 2 μg L^{-1}

3. Results and Discussion

PCBs can traverse international borders and remain in the environment for extended periods of a food chain, they bio magnify and accumulate in fatty tissues. Thirteen of the 209 distinct PCB kinds are regarded as POPs similar to dioxin. Their half-lives, which can range from 10 days to 1.5 years, are correlated with the level of chlorination and their persistence in the environment. (Montano et al., 2022). Although, the evaluation of polychlorinated biphenyls (PCBs) and their derivatives that arise from the contamination of all environmental media is intrinsically linked to their ubiquity and persistence. Among the most common and well-known POPs that are a burden on the environment and can be found in environmental media (such as water, air, and soil) as a result of various industrial operations are PCBs. Due to their bio-accumulative and persistent characteristics, as well as ongoing emissions, they remain in the environment even if their usage and manufacturing were outlawed decades ago. (Ngoubeyou et al., 2022). There are many symptoms, the most prevalent ones being rashes and skin disorders similar to acne in adults, along with neurobehavioral abnormalities in youngsters. Despite only being partially considered a carcinogen in humans, PCBs are known to cause cancer in animals. investigating PCB-exposed workers, the findings of

which suggested potential liver harm. Lastly, although though birth abnormalities are not known to be caused by PCBs, it has been observed that mothers exposed to PCBs gave birth to children that weighed a little less than average. Additionally, these babies displayed some aberrant answers on tests of short-term memory, immune system function, and motor skills.

3.1. Distribution pattern of PCBs

Concentration of eleven PCBs congeners is presented in (Table 1 and 2) and Fig. 2. The levels of different chlorinated congeners varied significantly, ranging from (ND - 5.21 µg L^{-1}) in kafr El Zayat region, while the range were (ND – 3.58 µg L^{-1}) in Shubra Khait region.

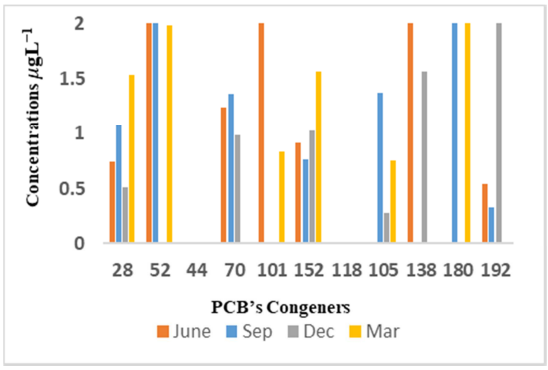
Table (1): Mean concentrations of PCBs (μgL^{-1}) in water samples from the river Nile, kafr El Zayat, Egypt

PCB's Congeners	Jun.	Sep.	Dec.	Mar.	Mean	Min	Max
28	1.05	1.57	0.98	2.14	1.44	0.98	2.14
52	ND	2.22	1.58	0.83	1.16	ND	2.22
44	ND	ND	1.02	ND	0.26	ND	1.02
70	5.21	0.25	0.88	1.63	1.99	0.25	5.21
101	2.81	1.69	0.64	ND	1.29	ND	2.81
152	0.99	0.15	0.5	1.32	0.74	0.15	1.32
118	ND	0.82	ND	ND	0.21	ND	0.82
105	2.48	ND	1.08	2.35	1.48	ND	2.48
138	ND	0.97	0.25	4.01	1.31	ND	4.01
180	1.89	ND	0.45	1.22	0.89	ND	1.89
192	2.54	ND	2.97	0.95	1.62	ND	2.97
Total	16.97	7.67	10.35	14.45	12.39	1.38	26.89

Table (2): Mean concentrations of PCBs (μgL^{-1}) in water samples from the River Nile, Shubra Khait, Egypt

PCB's Congeners	Jun.	Sep.	Dec.	Mar.	Mean	Min	Max
28	0.75	1.08	0.51	1.54	0.97	0.51	1.54
52	2.39	2.54	ND	1.98	1.73	ND	2.54
44	ND	ND	ND	ND	ND	ND	ND
70	1.24	1.36	0.99	ND	0.90	ND	1.36
101	3.58	ND	ND	0.84	1.11	ND	3.58
152	0.92	0.77	1.03	1.57	1.07	0.77	1.57
118	ND	ND	ND	ND	ND	ND	0.00
105	ND	1.37	0.28	0.76	0.60	ND	1.37
138	3.52	ND	1.57	ND	1.27	ND	3.52
180	ND	3.24	ND	3.44	1.67	ND	3.44
192	0.54	0.33	2.73	ND	0.90	ND	2.73
Total	12.94	10.69	7.11	10.13	10.22	1.28	21.65

Shubrakhit



Kafr El Zayat

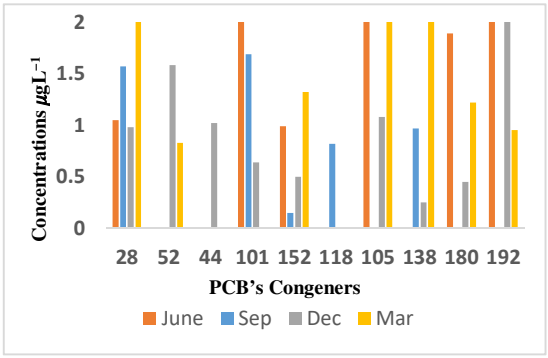


Figure 2: Concentrations of PCBs (µg L^{-1}) in collected water samples from shubrakhit and kafr El Zayat, Egypt

The data showed that the total highest concentrations of PCB's congeners in water samples were collected from Rosetta Branch of Kafer El Zayat region during June month were (16.97 µg L^{-1}) compared with the total lowest concentration of PCB's congeners in water samples were collected during September month were (7.67 µg L^{-1}). Also, the total highest concentration of PCB's congeners in water samples were collected from Shubra Khait region, El-Beheira governorate during June month were (12.94 µg L^{-1}) compared with the total lowest concentration of PCB's congeners in water samples were collected during December month (7.11 µg L^{-1}). Although, the results of Kafr El Zayat region showed higher contamination levels of PCBs than that detected in Shubra-Khait region (Fig.3) which were 12.39 (7.67 – 16.97) and 10.22 (7.11 – 12.94), respectively.

Whenever, based on the sampling season, the PCB concentrations in the surface water are in June > March > December > September sequence in kafr El Zayat region compared with the sequence of the PCB concentrations in the surface water in June > September > March > December. The USEPA's tolerable allowable level for PCBs in drinking water (0.5 μg/L) (Adeyinka et al., 2018) was exceeded by the concentration of PCBs in several of the water samples shown in Tables 1 and 2, suggesting a significant PCB pollution issue in the Nile River water and a substantial risk of harmful human health impacts.

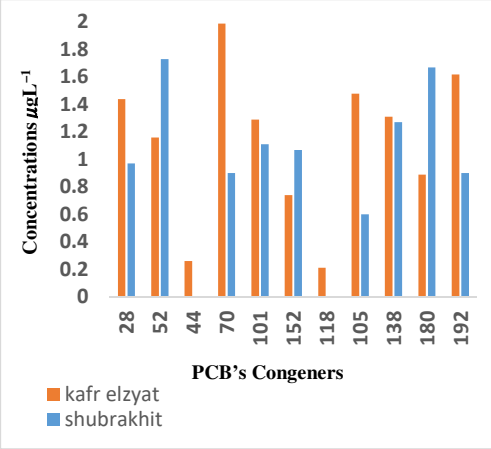


Figure 3: Concentrations of PCBs (μg L^{-1}) in collected water samples from Kafr El Zayat and Shubrakhit region along the River Nile, Egypt

Our results were agreed with (Megahed et al., 2015) who found that PCBs levels in the Nile River were ranged from 14 to 20 µg L-1 higher than those found in earlier investigations. The sampling sites had a different impact on PCB congener contamination profiles. The most common congener, accounting for more than 18% of all PCBs, was PCB-138. According to the PCB congener's levels in water, the primary PCB manufacture previously employed in Egypt consisted of highly chlorinated PCB technical mixes such Aroclor 1254. From the upper stream to the Nile River estuaries, there was a rising trend in PCB levels (Zahed et al., 2009). Studies revealed that four stations' coastal water and sediment in Shadegan Wetland Protected Area in the northwest of the Persian Gulf had PCB isomer groups (congener numbers 28, 52, 101, 118, 138, 153, and 180). The range of the total PCB contamination level in water was 8–375 ng L-1. The concentration of various congeners, Clophen A60 was detected, it might be the source of PCB in this area due to has been used extensively in Iranian electronic industry. while (Nasr et al., 2009) found that Bahr Shebin canal in Egypt has the highest concentration of total PCBs, which was found to be 67.89 ng L-1. Also, Stoll et al., 2020 determined 41 PCBs, the total PCB concentrations ranged from 2.17 to 5.29 ng L-1, above the threshold for river water quality standards in Brazil. While, Eze et al., 2023 presented the concentration of PCB congeners for the surface water and sediment ranged from 622.87–935.84 ng L-1 and 1621.03–2748.17 µg kg-1 respectively. The concentration of PCB congeners in the studied matrices revealed that PCB-118 had the highest concentration, while the lowest concentration was recorded in PCB-157. on other hand, Cui et al., 2020 determined the ΣPCBs concentrations in water, sediment, and SPM in the longest Yangtze River in Asia which ranged in 0.04–11 ng/L, 0.33–69 ng/g, and 0.72– 153 ng/L, respectively. The main pollutants were PCB17, 18, 28, 47, and 118 in the Yangtze River. The fSW value of PCB18 and PCB28 all exceeded 0.50, whereas the fSW value of PCB47 and PCB118 (except for PCB118 at Y-4) was less than 0.50. The toxic equivalent quantity (TEQ) range of PCBs in water and in sediment was ND–5.55 pg-TEQ/L and ND–2.51 pg-TEQ/g, respectively.

3.2. Concentrations of PCBs in Nile River Water and Global Comparison.

Water samples collected from 4 sites of Nile River contained detectable concentrations of PCBs, indicating that PCBs are widespread in the surface water of Nile River especially near to industrial areas such as kafr El Zayat and Shubra-Khait. However, the obtained data reported that the PCBs levels in water samples collected from different 4 sites of Nile River were in a moderate level comparing to other data published from different areas and countries around the world (Table 3) and to other studies in Egypt. Our results were comparable with the data detected in different country around the world, in Pearl River Estuary and Liaodong Bay from China, the total concentrations of PCBs were 33.4 – 1060 ng/L and 5.51 - 40.28 ng/L (Zhang et al., 2002; Men et al., 2014) while total concentrations in Pakistan were 1.94-11.66 ng/L (Baqar et al., 2017) and in Brazil were 2.17 to 5.29 ng L-1 (Stoll et al., 2020). On other hand the total concentration of PCBs in previous studies in Egypt, (Nasr et al., 2009) determined the total PCBs concentration in Bahr Shebin canal 67.89 ng/L, El-Bagoria canal 6.036 ng/L and El-Sarsawia canal 7.478 ng/L in Menoufia governorate. Despite, (Megahed et al., 2015) found the total concentration of PCBs in Great Cairo were ranged 14 to 20 μg/L. while, (Nassar 2017) found the total concentration of PCBs in Beni-suef, Greater Cairo and Banha were 16.51 μg/L, 20.75 μg/L and 27.01 μg/L. also, Abdel-Wareth et al., 2024, found

the total PCBs in Al-Qaliobiah ranged 18.01-85.44 µg/L.

Table (3): Concentration of PCBs in the Nile River water compared with that recorded from other coastal areas.

	Locations	Year	ΣPCBs	References
Globally	Pearl River Estuary China	2000	33.4-1060 ng/L	Zhang et al., 2002
	Liaodong Bay China	2007	5.51-40.28 ng/L	Men et al., 2014
	River Ravi, Pakistan	2015-2016	1.94-11.66 ng/L	Baqar et al., 2017
	Brazil.	2020	2.17 to 5.29 ng/L ng L^{-1}	Stoll et al., 2020
Egypt	**El-Monofia Governorate:** Bahr Shebin canal El-Bagoria canal El-Sarsawia	2009	67.89 ng/L 6.036 ng/L 7.478 ng/L	Nasr et al., 2009
	Greater Cairo	2015	14 to 20 µg/L	Megahed et al., 2015
	Beni-suef Greater Cairo Banha	2017	16.51 µg/L 20.75 µg/L 27.01 µg/L	Nassar 2017
	Al-Qaliobiah	2024	18.01-85.44 µg/L	Abdel-Wareth et al., 2024
	Our study		7.67-16.97 µg/L Kafr El Zayat	
			7.11-12.94 µg/L Shubra-Khait	

4. Conclusion

Water is the main source for all forms of life and plays an important role in human life. Drinking or use of water polluted with pollutes residues might have health impact on human and should not exceed international allowable limits. Industrial and municipal wastewaters are considered the main source of water contamination is due to irresponsible or negligent practices. PCBs is one of the most persistent organic pollutants in the ecosystem, were used in many industries. PCBs has human health and environmental impact due to its ability to bioaccumulation and biomagnifying effects in the food chain. Water samples collected from 4 sites of Nile River contained detectable concentrations of PCBs, indicating that PCBs are widespread in the surface water of Nile River especially near to industrial areas such as kafer El Zayat and Shubra-Khait. Kafr El Zayat region showed higher contamination levels than detected in Shubra-Khait region.

The PCBs levels in water samples collected from different 4 sites of Nile River were in a moderate level comparing to other data published from different areas and countries around the world. Since PCBs were found in some samples, we advise setting up a monitoring program to learn more about the quality of bottled water over time and to help develop strategies for lowering the amount of organic contaminants.

(1.67 - 9.5), Mg, 11.51 (10.56 - 12.53), Mn, 0.86 (0.0 - 2.04), Ni, 1.83 (0.36 - 2.96), Pb, 0.23 (0.0 - 0.48) and Zn, 0.65 (0.43 - 0.88) µg/ml. The most frequent metal in all water samples was Mg which detected in levels higher than the maximum permissible levels was 11.97 (10.85 - 12.41) and 11.51 (10.56 - 12.53) from El-Beheira and El-Gharbia governorates, respectively.

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