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Levels of Heavy Metals in Water and Nile Tilapia fish (*Oreochromis niloticus*) of Eleyele Lake in Ibadan, Nigeria

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Abstract

Surface waters are susceptible to hazardous pollution which may migrate with devastating health challenges through the food chain to humans. This study evaluates the levels of heavy metals in water and Nile tilapia of Lake Eleyele. The physicochemical properties of water samples were determined using standardized methods, while heavy metals in both water and fish samples were determined using atomic absorption spectrophotometer (AAS). Water pH, electrical conductivity, total dissolved solids, total suspended solids, and dissolved oxygen were within WHO and USEPA limits for potable water. Cu (0.028 – 0.112 mgL^{-1}), Pb (0.002 – 0.009 mgL^{-1}), and Zn (1.46 – 3.79 mgL^{-1}) were below the maximum permissible limits by both WHO and USEPA, while cadmium, chromium, and nickel were not detected in the water samples. Cu (2.56 -3.54 mg/kg), Zn (22.93 – 32.13 mg/kg), and Ni (0.39 – 0.48 mg/kg), are within acceptable levels, while Cd and Cr were also not detected in the fish samples. However, Pb (1.71 – 2.30 mg/kg) was higher in the fish samples than the threshold limit (0.3 mg/kg) by FAO and WHO. In accordance, Pb with bioconcentration factor (BCF: 402) was more highly bio-accumulated in the fishes than Cu (BCF: 54.4) and Ni (BCF: 10.97). The results recommend the river water for utility purposes but raised concerns about the consumption of Nile tilapia fishes in the lake.

Keywords: Heavy metal, Nile tilapia, AAS, Bioconcentration factor, Eleyele Lake.

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INTRODUCTION

Pollution of heavy metals in aquatic ecosystems resultantly from anthropogenic activities is growing at an alarming rate and has become an important worldwide problem in recent times (Abdel-Baki, *et al*., 2011). Heavy metal pollution of water and aquatic organisms brought by urbanization and industrialization poses a great threat to their drinkability and consumption and this has recently become a global issue. In general, the term "heavy metals" refers to a collection of metals and metalloids with an atomic density larger than 4 g/cm^3 or five times or more, greater than water. Heavy metals are unbiodegradable and persist in the environment for a quite long time even when the source has been eliminated (Javed and Usmani, 2017). Toxic heavy metals that are potentially hazardous in their combined or elemental forms include Arsenic (As), chromium (Cr), cadmium (Cd), lead (Pb), mercury (Hg), nickel (Ni), copper, and zinc with varying toxicity level and associated health effects (Kinuthia, *et al*., 2020). Consequently, the metals can be deposited, assimilated, and incorporated into environmental matrices such as air, water, soil, sediment, and aquatic and terrestrial biota. Therefore, heavy metals are considered the most dangerous in toxicological studies.

Consumers of fish and other wildlife are at risk of health problems due to heavy metals in aquatic settings and aquatic biota. Agricultural practices, industrial or domestic sewage sludge (Adekola and Saidu, 2005), runoff, leaching from landfills and dumpsites, and atmospheric deposits are just a few examples of the various natural and human-made ways that heavy metals can enter aquatic ecosystems and cause water pollution, which in turn contaminate fish (Fortsner and Wittman, 1981). Despite the fact that fish need to absorb vital metals from their diet, water, or sediment for normal metabolism or physiological function (Kalay and Canli, 2000). Lead, cadmium, and other heavy metals like mercury serve no biological purpose or benefit to the living body (Robert, 1991) and can be highly toxic. Fish at the top of the aquatic food chain can accumulate heavy metals through the food chain and water, which can then be consumed by humans and cause both acute and chronic disorders. Depending on the method of exposure - dietary and/or aqueous exposure - metals are distributed differently across the various tissues (Ekeanyawu, *et al*., 2015).

Research had proven that the amount of heavy metals that accumulate in fish tissue is primarily influenced by the amount of metals present in the water and the length of exposure, as well as other environmental parameters including temperature, pH, salinity, and hardness (Authman, 2008). In addition to frequently being at the top of the aquatic food chain, fish and other aquatic animals are also known for being excellent accumulators of both organic and inorganic contaminants and for having the capacity to bioconcentrate heavy metals in significant concentrations in the tissues (Ekeanyawu, *et al*., 2015). It contributes significantly to human nutrition and serves as a healthy, affordable food source for people. Therefore, they need to be thoroughly checked to make sure that excessive concentrations of dangerous heavy metals are not ingested by humans (Adeniyi and Yusuf, 2003).

Eleyele lake is a clean, enclosed river that has a significant impact on the lives of many people in Ibadan. It is a source of commercial fishery. The communities in its vicinity use it as a source of drinking water and for pleasure. Human activities including industrialization, farming methods, deforestation, and sewage discharge have an impact on it. These could cause the Eleyele lake to absorb various types of contaminants that are heavy metal-enriched through a variety of methods. The river is said to receive water from the dam site, residential wastewater from nearby homes, and effluent discharged from the cassava processing factory. This lake connects a number of other rivers, including the Nihort River, the Eleyele Dam, and towns like Nihort and Idishin, among others, although it has no outflow. This study is being conducted to ascertain the present level of the concentrations of heavy metals, such as copper (Cu), lead (Pb), nickel (Ni), chromium (Cr), and zinc (Zn), in water and their accumulations in the edible tissue of the tilapia fish during the dry season in the river. In this investigation, additional water characteristics of the river water, including temperature, dissolved oxygen, pH, conductivity, total dissolved solids, and suspended particles, were also determined. Rivers, lakes, and other bodies of water are now frequently contaminated with heavy metals.

MATERIALS AND METHODS

Description of Study Area

Eleyele lake is situated in the North-west of Ibadan, Oyo State, Nigeria at an altitude of 125 m above sea level and between7°25′00′ and 7°26′30′N latitudes and 3°51′00′ and 3°52′30′E longitudes (Figure 1). It covers some parts of Ijokodo, Apete, Awotan, Ologun-eru, Agbaje, Idi-Osan, Polytechnic of Ibadan, and Eleyele area with the rural fishing communities mostly dominated by Ilaje and Yorubas. Similarly, it connects many other rivers like Nihort river, Eleyele lake and neighboring communities. According to Akinyemi *et al*., (2014) the Eleyele lake receives sewage from households in the catchment, effluent from the cassava processing factory, and water from the dam site. Additionally, a variety of human activities in the region, including fishing, vehicle washing, block making, textile washing, auto repair, boat trafficking, water tanker loading, farming, and agricultural operations, expose the lake to pollution of all kinds.

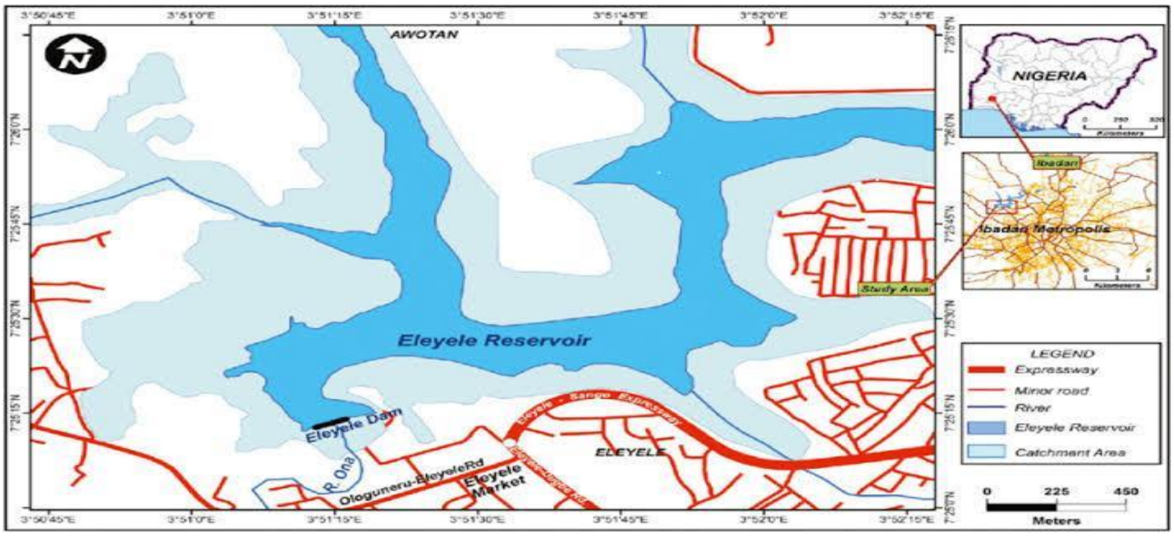

Figure 1: Map showing the study area of Lake Eleyele

Sampling Procedure

In order to identify the Lake Eleyele's point source of pollution, a preliminary field assessment of the study region was carried out using a canoe. During the sampling period, the sampling sites were utilized. By taking into account the relative sources of pollution, three sampling locations in the lake were chosen. Different types of contaminants from various sources of pollution have been received at each chosen sample site. The three locations are chosen because the three sources dominate the lake's pollution. The spot where people enter the lake at a high point is the first site and this is the area from which locals and others collect water for drinking and other domestic purposes. The second sampling site is where all of the canoe passengers disembark, and the third place is where the trash and waste are dumped by the fishermen. The samples were taken in the month of March, 2022 which signaled the period of the dry season in the region.

Collection and Preservation of Water and Fish samples

Water and fish samples were collected from the three selected sample points (SPs) of the lake Eleyele. Three replicate surface water samples (50 cm depth) were collected from three different points of the lake using 1-Litre polyethylene sampling bottle during the sampling period. Before sampling, the sample containers were washed by soaking them in detergent for 24 hours, subsequently rinsed with tap water, and then rinsed with 5% nitric acid and lastly with distilled water. The water samples in the plastic bottle were immediately filtered through Whiteman-541 filter paper and preserved with 1M Conc. HNO_3 was taken to the laboratory and stored using an ice box at 4°C prior to analysis. The three replicated samples for each site were composited for all analytical procedures conducted in the laboratory. Meanwhile, one hundred samples of Tilapia fish were caught and collected at each sampling site with the help of fishermen (Figure 2). After collection, the samples were immediately dissected at the river bank and only the edible tissue (fillet) was transferred to plastic bags and kept in an ice box at 4°C and then transported to the laboratory for analysis. A Composite of the three sampled fish in each sampling site was prepared by taking the edible tissue (fillet) to obtain a representative sample. The fish samples were oven dried at 105°C until they reached a constant weight. Each dried sample was then ground into a fine powder using a porcelain mortar and pestle, and thereafter all powdered tissues labelled were kept in desiccators prior to further chemical analysis.

Fig 2: Tilapia Fish Samples from Eleyele river

Digestion of Fish Samples

The powdered fish samples were thoroughly homogenized and digested using concentrated nitric acid and hydrogen peroxide (2:1) v/v according to FAO methods (Daziel and Baker, 1983). A dried and powdered fish sample of 0.5g was weighed and transferred into a 50 mL round bottled flask and the mixture of 10 mL of concentrated HNO_3 (65%) and 10 mL of H_2O_2 (30%) was added. The flask was covered with a watch glass and left aside until the initial vigorous reactions occur. Then, the samples were heated on a Heating Mantle to 130°C until dissolution inside a fume hood to reduce the volume to 3-4 mL in the digestion flask. After that, the samples were allowed to cool, filtered, and diluted to 50 mL in a volumetric flask with deionized water.

Physicochemical and Heavy metal Analyses

The physicochemical parameters were analysed using standard procedures. Temperature and dissolved oxygen of the water samples were determined in situ in the three sampling sites using CO-411 ELMETRON, pH with Wagtech pH meter, while conductivity and total dissolved solid were determined by EC/TDS. The suspended solid of the water was determined in the laboratory by Hack Spectrophotometer. Nitric acid-preserved water samples meant for trace metals analysis were digested according to standard procedure (APHA, 2005). Analyses of heavy metals concentrations (Cd, Cu, Pb, Ni, Cr, and Zn) in water and fish samples were carried out by graphite furnace atomic absorption spectrophotometer (GFAAS) and flame atomic absorption spectrophotometer (FAAS). The 210/211 VGP Atomic Absorption Spectrophotometer equipped with a graphite furnace and ASC-990 autosampler for GFAAS and ASC-900 autosampler for flame was used for determinations. Modifiers [$Mg(NO_3)_2$ and $NH_4H_2PO_4$] were used for GFAAS to eliminate matrix interferences. All measurements of samples and blank were carried out in triplicates.

Bioconcentration factor

Bioconcentration factor (BCF) is the accumulation of the contaminant by aquatic organisms through non-dietary uptake routes, e.g., from a soluble phase. It is a measure of fish's ability to accumulate metals from water. BCF is the ratio of metal concentrations in fish (biota) to that in water can be evaluated as shown (Suheryanto and Ismarti, 2018).

$$BCFb/w = \frac{Concentration\ of\ heavy\ metals\ in\ the\ biota\ (\frac{mg}{Kg})}{Concentration\ of\ heavy\ metals\ in\ the\ water\ (\frac{mg}{L})}$$

Statistical analysis

Statistical analyses was done using SPSS version 22. The mean values of the data were subjected to analysis of variance (ANOVA) with 5% ($p < 0.05$) level of significance.

RESULTS AND DISCUSSION

Physicochemical Tests

The results of the physicochemical parameters and their comparison with standards are presented in Table 1. The lowest temperature was recorded at SP I (21.38° C), while the highest was observed at SP III (22.53° C). The temperatures recorded were within the permissible range of 20-30° C by WHO and USEPA. According to WHO and USEPA standards, these values were acceptable for aquatic life and utility purposes. The pH of the lake water falls in the alkaline range and is also within the WHO range (Rehman *et al.,* 2008). There were slight differences in the pH of the water samples with the lowest value at SP I (8.64) and the highest at SP III (8.75). This difference in pH could be attributed to the influence of anthropogenic activities taking place around the lake Akinyemi *et al.*, (2014). pH affects the speciation and bioavailability of aqueous metal ions (Ayoade and Nathaniel, 2018). The pH values recorded in this study were higher than those reported by Akinyemi *et al.*, (2014) from other locations of the water. and within regulatory standards and guidelines for potable drinking water. The degree of mineral salts, filtrate residues, and dissolved salts constitutes electrical conductivity (EC) in water. This parameter is also directly related to the total dissolved solids in the water (Rehman *et al*., 2008; Adegbe *et al*., 2021). The highest EC was recorded at SP II (835.83 µS/cm) while the lowest value (820.50 µS/cm) was recorded at SP III. The EC of the water samples in all the SPs falls below acceptable limits. Ayoade and Nathaniel (2018) regarded solids in water as undesirable because they lower the portability of the water. TDS is an indicator of contamination such as dissolved salts present in water (Ojelabi *et al*., 2018). The minimum value of TDS was recorded at SP II (491.83 mg/L) and a maximum of 501.17 mg/L was recorded at SP I. The maximum value of total suspended solids (TSS) was observed at SP I

(17.67 mg/L) and a minimum of 9.67 mg/L was recorded at SP II. TDS and TSS values fall within the acceptable limit by WHO and USEPA.

Dissolved oxygen (DO) is an important marker of the stability of an aquatic ecosystem (Adegbe *et al*., 2021). The maximum DO was obtained at SP III (8.43 mg/L) and the minimum at SP III (5.20 mg/L). The values also fall within acceptable limits except for the value at SP III (5.20 mg/L), which falls slightly below the acceptable limit (6 mg/L) for aquatic life survival. The values of physicochemical properties reported were higher than the values recorded by Akinyemi *et al*., (2014). There were no significant differences (P<0.05) in the physicochemical parameters of the three sampling locations of the lake except for the dissolved oxygen.

Table 1: Physicochemical parameters of three samples from Eleyele Lake and their comparison with standard limits

Physicochemical Parameters	SP I	SP II	SP III	WHO	USEPA
Temperature (°C)	21.38±0.38 ^{a}	22.18±0.62^a	22.53±0.33^a	20 -30	25
DO (mg/L)	6.80±0.70^b	8.43±0.68^a	5.20±0.08^c	6	-
pH	6.34±0.02^a	6.75±0.01^a	6.73±0.03^a	6.5 – 8.5	-
EC (µS/cm)	835.83±6.03^a	820.50±1.32^a	832.33±0.57^a	1000	1000
TDS (mg/L)	501.17±3.51^a	491.83±0.76^b	499.00±0.50^a	600	500
TSS (mg/L)	17.67±0.58^a	9.67±0.58^c	14.67±0.28^b	1000	-

Mean values with different letters within the same row differ significantly (p < 0.05)
Key: SP= Sample Point; DO= Dissolved Oxygen; EC = Electrical Conductivity; TDS = Total Dissolved Solids; TSS= Total Suspended Solids; WHO= World Health Organization standard (Onyegeme-Okerenta *et al*., 2016), USEPA = United States Environmental Protection Agency standards (Rahman, *et al*., 2021).

Concentration of Heavy metals in water samples

The results of the analysis of heavy metals in water from the three sampling points were presented and compared with standard values in Table 2. The Cu, Pb and Zn were detected in all sample points. However, all the detected heavy metals occur below threshold levels according to the limits set by standard organizations (WHO and USEPA). Ni, Cd and Cr were not detected in all the water samples. These results are in agreement with the report by Akinyemi *et al*. (2014). There is no significant difference (P>0.05) between the concentrations of copper, lead and zinc from the sampling locations. Cu in Eleyele lake could be attributed to non-point sources of pollution, especially from agricultural fields where fertilizers and pesticides containing copper. Pb in Eleyele lake may be due to runoff form the highways into the river especially at sample point I, which is closer to the highways. Reduction in Pb at sample locations farther away from highways may be attributed to adsorption onto suspended particles and bio-accumulation (Campbell, 1995; Adekola and Saidu, 2005; Turner and Holmes, 2015).

Table 2: Heavy metals concentrations in Eleyele Lake water samples

SPs	Cu (mgL^{-1})	Pb (mgL^{-1})	Zn (mgL^{-1})	Ni (mgL^{-1})	Cr (mgL^{-1})	Cd (mgL^{-1})
SP I	0.030±0.002^a	0.007±0.002^a	2.65±0.32^a	ND	ND	ND
SP II	0.091±0.021^a	0.003±0.003^a	1.98±0.52^a	ND	ND	ND
SP III	0.050±0.020^a	0.004±0.001^a	2.77±1.02^a	ND	ND	ND
MPL*	1.5	0.05	5.0	0.1	0.05	0.005
MPL**	1.3	0.015	5.0	-	0.05	0.005

Mean values with different letters within the same column differ significantly (p < 0.05)
Key: SP = Sample Point; ND = Not Detected; MPL* = maximum permissible limit by WHO and MPL** = maximum permissible limit by USEPA (Ekramul Mahmud *et al*., 2016).

Heavy metal levels in Tilapia fish

The amount of heavy metals (Cu, Cd, Zn, Ni, Pb, and Cr) in tilapia fish from the three sampling points in Eleyele Lake were shown in Table 3. However, the heavy metal concentrations in the fishes were comparably higher than those of the water samples. This could be due to the bio-assimilation and bioaccumulation of heavy metals in the tissues of tilapia fish species in the water. On a dry basis, Cu ranged from 2.56 - 3.54 mg/kg, while Zn ranged from 22.93 – 32.13 mg/kg in the edible tissue of Nile Tilapia fish samples. The levels of Cu and Zn in the fishes were not significantly different ($P>0.05$) within the three sample points of the lake. This showed that there is no variation in the bioaccumulation of Cu and Zn in the edible tissue of Nile Tilapia fish species throughout the sampling points in the Eleyele lake. The concentrations were also below the acceptable maximum limits of 100 mg/kg and 30 mg/kg of both copper and zinc in the fish samples given by FAO/WHO (1989 & 1983) in Utete and Fregene (2020). Nickel is also an essential micronutrient required by red blood cells. However, above threshold levels, it may lead to unhealthy weight loss, skin irritation, as well as heart and liver damage. The toxicity of Ni to aquatic life has been shown to vary significantly with organism species, pH, and water hardness (Birge and Black, 1980). The level of Ni in the Tilapia fish samples ranged from 0.389 - 0.48 mg/kg. Accumulation of Ni in the tissue of the Tilapia fish samples statistically shows no significant difference ($P<0.05$) within the three sampling sites. Ni content in the fish samples was also below the acceptable maximum limit of 8.97 mg/Kg given by FAO/WHO (1989) in Younis *et al*., (2021). Lead is a non-essential and toxic element that can accumulate in fish tissues. The accumulation of Pb in the edible tissue of Tilapia fish collected from Eleyele lake ranged from 1.71 - 2.30 mg/kg. The high accumulation of Pb at SP I could be attributed to the closeness of the site to the highway, hence, more susceptible to pollutants released from automobile activities. The accumulation of Pb was significantly different ($P<0.05$) among the three SPs of the lake. This showed that the accumulation of Pb in fish tissue differs from one place of the lake to the other. Pb levels in the tissue of the Nile tilapia fish were higher compared to the FAO/WHO permissible limit of 0.3mg/Kg. Cd and Cr were undetected in the fish samples.

Table 3: Concentration of Heavy metals (mg/kg) in Nile Tilapia fishes from Eleyele Lake

Sampling Points	Cu	Pb	Zn	Ni	Cr	Cd
SP I	2.62±0.06[a]	2.15±0.15[a]	25.38±2.45[a]	0.41±0.021[a]	BDL	BDL
SP II	3.52±0.02[a]	1.95±0.24[b]	25.95±1.62[a]	0.48±0.005[a]	BDL	BDL
SP III	3.17±0.03[a]	1.92±0.22[b]	29.93±2.40[a]	0.43±0.011[a]	BDL	BDL
FAO/WHO	100*	0.3	30**	8.97*	0.05*	0.5

Mean values with different letters within the same column differ significantly ($p < 0.05$)
Key: Permissible maximum limit sources: FAO/WHO (2021) & FAO/WHO 1983** & 1989*

Bioconcentration factor of Heavy metals in Tilapia fish

The Bioconcentration factor (BCFb/w) of the tilapia fish in the Eleyele Lake is presented in Table 4. The results showed that all BCFb/w values of fish in water were greater than 1 which showed the transfer of metals detected in the water to the fish (Abdel-Baki, *et al*., 2011). This infers that heavy metals were bioaccumulated and biomagnified at different levels in the Tilapia fish species. A high category of bioaccumulation was recorded for Pb, moderate for Cu, and low for Zn. This shows that tilapia fish is a high accumulator of Pb in freshwater than other metals, and the result is in accordance with reports by Suheryanto and Ismarti, (2018) and Rizk *et al*., (2022).

Table 4: BCF values of the heavy metals of Tilapia fish in Lake Eleyele

Metals	Cu	Pb	Zn
Average fish Conc. (mg/Kg)	3.10	2.01	10.97
Average water conc. (mgL^{-1})	0.057	0.005	2.47
BCFb/w	54.4	402	10.97
	Moderate	High	Low

BCF: > 1000 = very high, 100 – 1000 = high, 30 – 100 = moderate, < 30 = low [14]; ND: Not Detected

Comparison of Heavy Metals Concentrations in this Study with Some Literature
The comparison of heavy metal concentrations in Nile Tilapia studied with reports in the literature is presented in Table 5. This showed that the bio-accumulation of heavy metals in Nile Tilapia fish tissue is not regular, as the fish is capable of various degrees of heavy metal bio-accumulation depending on its environment.

Table 5: Comparison of heavy metals (mg/kg) in Nile Tilapia fish tissue with reported values in present and previous studies in Nigeria

Locations	Cr	Cd	Cu	Ni	Pb	Zn	References
Eleyele Lake (Present study)	ND	ND	2.56-3.54	0.389-0.48	1.71-2.30	22.93-32.13	Present study
Eleyele Lake	NT	0.26-6.46	1.24-39.97	2.58-49.44	2.129-6.13	23.12-108.69	Utete & Fregene (2020)
Tunga kawo Lake	0.15±0.019	NT	0.13±0.015	NT	0.19±0.024	NT	Kinta, et al., 2021
River Benue	2.30	1.26	1.0	NT	2.04	3.0	Yarkwan & Apeh, (2015)
Akpoko Lake	2.055	1.23	0.6	NT	1.92	2.55	Yarkwan & Apeh, (2015)

"NT" means Not Taken, "ND" means Not Detected by the instrument

CONCLUSION
This study determined the concentration of heavy metals in water and the Nile tilapia fish from the Eleyele Lake. The physicochemical properties determined in water were within the acceptable limit of WHO and USEPA standards in all the SPs. The results revealed the level of heavy metals concentration in both water and Nile tilapia, *Oreochromis niloticus*, and only copper, lead and zinc were detected in water samples. The highest concentration of copper observed in water was 0.112mg/L, lead was 0.009mg/L and zinc was 3.79mg/L while chromium, nickel and cadmium were undetected in the water samples. The concentration of metals determined in water was in sequence Zn > Cu > Pb for the SPs. It is evident that they are below the permissible limit recommended for portable water. The level of heavy metal concentration revealed the presence of copper, nickel, lead and zinc in the Nile tilapia fish tissue while chromium and cadmium were undetected in the fish samples of the SPs. In the Nile tilapia, zinc has the highest concentration at the maximum value of 31.72 mg/kg, followed by copper at 3.54 mg/kg, then lead at 2.30 mg/kg and nickel at 0.485 mg/kg. Although, most of the heavy metals were below the maximum permissible limits of FAO/WHO in fish samples, however, Pb was found to be above the regulatory limit. It is concluded that the water is safe for drinking and domestic purpose whereas the Nile tilapia

fish of the Eleyele river is not safe for human consumption due to the elevated concentration of Pb above the FAO/WHO limit. Lead in fish may tend to bioaccumulate and biomagnify in the tissues of human beings, therefore posing a health risk. The pollution point sources of the river should be controlled to avoid waste disposal into the lake and continuous monitoring of the lake is encouraged.

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