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Some Physicochemical Parameters and Heavy Metals Concentration in Water and Organs of *Oreochromis niloticus* (Linnaeus, 1758) in Ajiwa Reservoir, Katsina State

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Abstract
Ajiwa Reservoir serves as a vital drinking water source and protein source to the nearby communities and Katsina metropolitan. Fishes are a great dependable source of protein and assessment to determine their safety or otherwise is very crucial. In this work, four vital organs (gills, gonads, liver and muscles) collected from Ajiwa reservoir in Katsina state were analyzed for some heavy metals (Cobalt, Copper, Manganese, Nickel, and Lead). This preliminary investigation was carried out between September to December (2023) to expose additional information on the concentration of heavy metals in both water and tissues of *Oreochromis niloticus,* four sampling stations were randomly chosen for the survey along the bank of the reservoir. Samples were collected fortnightly throughout the research. The mean physicochemical parameters of the Ajiwa reservoir were pH (7.10±0.48), Temperature (22.93±4.02), Transparency (12.61±2.46), Conductivity (113.97±10.63), DO (6.78±1.09), and BOD (3.52±0.88). Atomic Absorption Spectrometry (AAS) instrument was used to determine the heavy metal concentrations in the samples and their levels compared with the World Health Organization (WHO, 2020) specified maximum levels. The results showed the concentration of heavy metals in the water to be (mg/L) Co (0.45±0.23), Cu (0.05±0.03), Mn (0.06±0.09), Ni (0.10±0.07), and Pb (0.40±0.18). Co, Mn, Ni and Pb were higher than the set standards limits of WHO (2023). The results indicate that the concentration of heavy metals in the analyzed fish tissues are in (mg/L) Co (1.03±0.16), Cu (0.13±0.05), Mn (0.10±0.19), Ni (0.18±0.22) and Pb (0.21±0.66). The concentration of Co, Ni and Pb were above the set standards of WHO (2020). Co, Mn and Pb concentrations mostly in the liver and gills were higher than the maximum permissible limit recommended by standard bodies.

Keywords: Ecotoxicology, Freshwater Ecosystem, Trace Elements, Fish, Atomic Absorption Spectrophotometer (AAS), Pollutants

Introduction
Water resources boost advancement in terms of the socioeconomic activities that are essential for humanity at large. These resources are used mostly for domestic, agricultural, and industrial activities. According to Shehab et al. (2014). In the twenty-first century, providing clean drinking water to the world's rising population is one of the most difficult challenges humans are dealing with. Biotic and abiotic variables that influence water quality (Siegel, 2014). Heavy metals are exclusive naturally occurring elements that persevere in the environment for a long time and may not be eco-friendly (Ogbeide & Henry, 2024). Heavy metals are naturally found in the Earth's crust. They enter water bodies from diverse sources (Babayemi et al., 2017). Major sources of heavy metal in the ecosystem could be natural or anthropogenic activities. The naturally occurring metals already exist in nature and became part of the environment by weathering, metal-bearing rocks, and volcanic eruptions, while anthropogenic sources of heavy metal include various industries, mining, and agricultural practices (Ikponmwen et al., 2023). Human activities are the major sources of heavy metals contaminating rivers (Babayemi et al., 2016), activities such as farming, surface runoffs from manufacturing areas, effluents from industrial (Zhou et al., 2020), and mining activities contribute to the levels of heavy metals in the water bodies (Gheorghe et al., 2017). Another source of heavy metals is inappropriate waste management or disposal (Babayemi et al., 2017). The discharge of wastes containing toxic heavy metals into water bodies may substantially affect fish and other aquatic organisms, endangering public health

Heavy metals can bioaccumulate over a long time and the concentrations become apparent and measurable (Garg et al., 2022). Through food chains and trophic levels, heavy metal bioaccumulation within target organs or tissues of organisms can ultimately threaten human health. Aquatic animals can accumulate and bio-magnify heavy metals up to concentrations that are tenths and even thousands of times higher than their concentrations in the environment through the aquatic food web (Huang et al., 2021).

MATERIALS AND METHOD

Study Area

Ajiwa reservoir is located at Batagarawa Local Government Area of Katsina State on latitude and longitude 12°54'69" - 12°57'58" N and 7°42'53" - 7°47'50" E. It is in the Sudan savannah zone of Nigeria with two distinct seasons (wet and dry). The wet season period on average lasts from May to October and the dry season from November to April. The main purpose of the reservoir is irrigation and water supply to the people of Katsina, Batagarawa, Mashi, and Mani local government areas. The reservoir was constructed in 1973 and commissioned in 1975. Its major source of water is the river Tagwai. It has an original height of 12m but after being rehabilitated in 1998 the height is now 14.7m, the original reservoir crest length was 880m, but after being rehabilitated reservoir crest length is now 1491.8m. It also has a surface area of 607.0ha. The storage capacity of the water is about 22,730,000m^3. The reservoir serves as the source of livelihood for the nearby communities such as Ajiwa, Masabo, Tsagero, Kadaji, Kwatami, Maje, and Gajeren giwa villages (Usman and Adakole, 2017).

Sample Collection

Four sampling points labeled Station A, B, C and D were selected along the bank of the reservoir for the study. Samples were collected between 8-10 am fortnightly for a period of four months (September to December 2023).

Water samples were collected in bottles (1000ml) at four different stations. At each of the sampling stations, each bottle was rinsed at least twice with the water to be sampled. Water was collected at the surface level by a dipping plastic sampling bottle sliding over the surface of the water with their mouth against the water current to permit undisturbed passage of the water into the bottle, the bottles were then labeled appropriately, and taken to the laboratory for further analysis.

Digestion of Water Sample

100 ml of the water sample was measured with a measuring cylinder and 5 ml of concentrated hydrochloric acid was added to it. This solution was placed in a conical flask and was heated on the hot plate for two hours at 105^{0}C to 25ml. It was then transferred into a 100ml volumetric flask and distilled water was added to fill up the mark where it was filtered and transferred into the pre-cleaned sampled bottle and taken for further (ASS) analysis (Adebayo, 2017; Pandiyan *et al.,* 2020).

Collection and Digestion of Fish Samples

About 8-12 fish of medium size were procured immediately after arrival at the reservoir from local fishermen between the hours of 7-8 am. The fish samples were washed with distilled water, gills, liver, and muscles were removed and dried for 24 hours to constant weight in an oven at 105^{0}C. After drying the samples in the oven, the samples were milled with a mortar and pestle. They were put in a dry labeled crucible and stored until digestion. This involved digesting 2 g of the grounded samples with 5 ml of HNO_3 and 2 ml of HCL and was heated on a hot plate for 30 minutes at 85^{0}C. After completing the digestion, the residue was allowed to cool and filtered into a 50ml volumetric flask. Distilled water was added to it to fill up the mark. The filtrate was transferred into a pre-cleaned sampled bottle and stored at a cool temperature until is taken for further (AAS) analysis (Yi *et al.,* 2017).

Heavy Metals Analysis

The level of heavy metals (Cobalt, Copper, Manganese, Nickel, and Lead) concentration in water and fish was determined using an Atomic Absorption Spectrophotometer (Buck Scientific Model 230) at the Central Laboratory, Umaru Musa Yaradua University, Katsina State.

Data Analyses

Data was entered on the spreadsheet and analyzed with the Statistical Package for Social Science (SPSS) version 20.0. The data was summarized using descriptive statistics and was later analyzed using One-Way Analysis of variance (ANOVA). ANOVA was used to analyze the concentration of heavy metals in both water and fish tissues (*O. niloticus*) of Ajiwa Reservoir. The results of the data analyses were presented in tables and figures in the results section. Statistical Significance was set at $P < 0.05$ confidence level.

RESULT AND DISCUSSION

Physicochemical parameters of water indicate possible chemical reactions in water and influence the distribution of uptake of metals, for effective production of fish and other aquatic organisms, the balance of physicochemical parameters in water is indispensable. The variation in the measurement of physicochemical parameters found in this study could be attributed to the differences in location and anthropogenic activities in and around the reservoir.

The water temperature observed in this study fluctuated within months in all four sampling stations and it is conducive and could support both fisheries and aquatic life. The mean temperature of Ajiwa reservoir was 22.93 which is within NESREA's (2011) set standard of (25°C) as being most suitable for the existence and development of aquatic life, similar findings were reported by Mohammed (2023) on Zobe Dam. Usman (2016), in his study also reported the temperature of the Ajiwa reservoir to be within the set standard limits of regulatory bodies.

Water pH, DO, BOD, and transparency were observed to be lower in October and higher in September with pH being higher in November and Transparency higher in December. Temperature was observed to be lower in December and higher in October.

The mean pH of the Ajiwa reservoir is 7.10 which is neutral and falls within the range for most biological activities, the pH status of the Ajiwa reservoir is within the WHO and NESREA (2011) permissible limit (6-9) for the protection, survival and development of aquatic life. Abdulkarim and Ibrahim (2018) reported 6.5-8.5 as the mean pH in their work on phytoplanktonic species diversity concerning parameters of Ajiwa Reservoir, Katsina State Nigeria. The pH value recorded in this research is similar to the findings of (Tukur et al., 2023) in his work ecological observation of freshwater snails in the vicinity of an artificial lake in Bakolori Dam, (Ibrahim et al., 2023) on physicochemical condition of Jibia reservoir inhabited by a freshwater snail *Belamya crawshayi*, Usman (2016) in his work in Ajiwa reservoir and Mohammed (2023) on Zobe Dam. Islam et al., (2017) reported that the high use of agrochemicals for agricultural purposes can also bring a high amount of pH in water. The pH value obtained in this study is maximal for plant growth and best condition for aquatic organisms.

The mean Secchi disk transparency was 12.61. Lower secchi disk visibility was recorded in October this could be attributed to the increase in flooding which washed earth materials into the water body. This agreed with the findings of Usman (2016) who recorded similar results within that period.

The conductivity level of Ajiwa Reservoire was 113.97 µs/cm. It is below the 1000 µs/cm limit stipulated by NESREA, 2011 for the survival of aquatic organisms in freshwater.

Dauda & Abba (2018) a reported similar result in their study on the Ajiwa reservoir, Usman (2016) also reported similar results on Conductivity (147.55) while the study by Tukur et al., (2023) proved the result of these findings, where higher mean conductivity level of 113.97 µs/cm in Bakolori Dam in Zamfara State was recorded.

Dissolved oxygen obtained in this study (6.78±1.09) which was relatively high, suggested that the reservoir is well-oxygenated and ideal growth and survival of fish. Usman (2016) reported similar findings which state that the rainy season has a higher concentration of DO which is due to an increase in water movement which results in lower temperature and an increase in dissolved oxygen. The higher the temperature the lower the dissolved oxygen and the higher the temperature the lower the dissolved oxygen (Adakole, 2000). The findings of this study confirm the findings of Mohammed (2023) and Abba et al. (2018). The high DO value recorded in this study may be linked to wind during the wet season which causes turbulence of the water.

WHO and NESREA stipulated at least 6.0 mg/L and a limit of 8-10 mg/L respectively in surface waters. The BOD level obtained in this study is 3.52±0.88 mg/L which is within the standard level. However, it was observed that BOD was high in December (4.16±0.64) and lowest during October (2.70±0.77), this findings coincided with the findings of Usman (2016) who reported similar results in BOD which is high in the dry season and low during raining season due period of oxygen consumption by decomposers on the biodegradable materials. Maintaining water quality parameters within limits set by agencies like WHO and NESREA ensures safe water for humans and aquatic organisms.

The mean concentration of Cobalt obtained in this study was 0.45±0.23 which is below the WHO/NESREA 1.0 set standard. This indicates that the water samples have low concentrations of cobalt and are not a threat to the users of the water for both domestic and irrigation purposes. These findings correspond with 0.35±0.02 mg/L reported in water from the Sharada industrial area of Kano, and 0.19±0.01 mg/L in water samples of the Bompai industrial area of Kano reported by Hassan et al. (2022). These values were within the maximum permissible limit of cobalt (1.0 mg/L) in dam water as recommended by WHO/NESREA.

The mean in the concentration of Copper was 0.05±0.03 which is below the WHO/NESREA 1.2 set standard. Ahmad et al. (2020) reported that copper concentration in Ajiwa reservoir water (0.03±0.01 mg/L). These values were lower than the value obtained in this research, this will possibly be due to differences in the sampling site, increased anthropogenic activities, or differences in the period of sample collection. However, Mohammed (2023) reported high copper concentrations in Tsawa-Tsawa, Garhi, and Makera in Zobe Dam Zobe dam. This high value could be due to metal contamination by agriculture from the areas.

Manganese concentration was (0.06±0.09) mg/L, the concentration of Mn is within the standard limit set by WHO/NESREA of (0.4). Manganese is naturally occurring and essential for human life and other living organisms, but its presence in an excess level in water may cause changes in color and taste, which indeed affect the water acceptability by the consumers and are detrimental to the survival of aquatic organisms. Ahmad et al. (2020), reported in their study that the concentration of Mn was 0.21±0.01 mg/L and 0.18±0.01 mg/L in both the Ajiwa and Jibia Dam water respectively. These concentrations were almost the same in both water bodies and higher than the Mn concentration obtained in this study. In a similar study, 0.13±0.05 mg/L was reported for manganese concentration in a water sample of Ajiwa reservoir by Yaradua et al. (2018); these values were higher than the value obtained in this research, this could be due to the difference in the sampling site or difference in the period of sample collection.

However, in a study conducted at Challawa River water of Kano State by Uzairu et al. (2014), a value of 0.28±0.05 mg/L was reported for manganese, indicating the manganese level in the water samples to be lower than the maximum permissible value. A range of values for manganese concentration was reported to be 0.01 – 3.45 mg/L in some water samples in Kano State (Sani *et al.,* 2016).

The mean concentration of Ni was found to be (0.10±0.07) mg/L. the concentrations of Ni are above the WHO/NESREA standard limit for freshwater (0.07). Nickel is usually needed for humans and other living organisms. This metal finds its way into the water by leaching through water pipes, and when taken above acceptable limits may lead to serious health challenges and possible death. In the research conducted by Yaradua et al. (2018), Ni was not detected in Ajiwa reservoir water. The variation between the finding of this current study and the one reported by Yaradua et al. (2018) could be due to the increased release of metals into the water body from agricultural fields, fertilizers, and pesticides, another reason might be fishing activities which can also contribute to the accumulation of heavy metals in water, as fish and other aquatic organism can absorb and accumulate heavy metals from the water and sediment. However, the result of this current study conforms with the findings of Ibrahim et al. (2018) who reported a value of 0.11±0.56 mg/L as the concentration of Nickel in the Jibia water sample, this is significantly greater than the acceptable concentration was 0.07 mg/L set by WHO/NESREA also, indicating that nickel is a threat in the water body. Furthermore, 0.21±0.02, 0.45±0.03, and 0.07±0.01 mg/L of Nickel were recorded in Sharada industrial area irrigation water, Bompai irrigation water, and Thomas irrigation dam of Danbatta, Kano respectively. These higher values obtained could be due to the discharge of industrial effluents in the mentioned dams. Ogunfowokan et al. (2013) observed 0.22 mg/L of nickel in the Asunle River at Obafemi Awolowo University in Ile-Ife, Osun State, which nevertheless remains greater than the permitted concentration of 0.07 mg/L. A further investigation executed by Ndede and Manohar (2014) in Kenya revealed a nickel concentration of 1.11 mg/L, which was greater than previously reported values. This could be related to the countless wastes tossed into the water source.

The mean concentration of lead (0.40±0.18) mg/L. It was observed that the concentration of lead in the water was above WHO and NESREA standard limit for freshwater (0.01 mg/L). The presence of lead at higher concentration in Ajiwa reservoir may be a result of household effluent, use of fertilizers for farming activities, dissolution of the metals from its natural sources in the water and discharge from water sources where industrial effluents end up in water bodies during raining season. Musa et al. (2020) reported Pb concentration of the Ajiwa reservoir to be 0.01±0.01 mg/L, while that of the Jibia reservoir to be 0.02±0.01 mg/L, This huge difference could probably be due to the differences in sampling time and contamination from either agricultural or domestic sources.

However, Ibrahim et al. (2018) reported the concentration of lead in the Jibia water sample to be 0.09±0.03 mg/L, which is significantly lower than the value obtained from this study. The mean high value recorded in this study may be due to the excessive influx of nutrients from water sources and farmlands where fertilizer is used to boost crop production, particularly around the reservoir. In another research conducted by Malami et al. (2014), it was reported that the level of lead in Challawa water of Kano State was 0.03±0.01 mg/L, which is lower than the value obtained in this research, In another study conducted by Sani *et al.* (2016), the lead concentration value range of 0.01 – 0.80 mg/L was reported in some selected water samples in Kano State. Research conducted by Moses (2018) showed the concentration of lead in the Bagega River of Zamfara as 0.39 ± 0.01 mg/L which is higher than what is obtained in the present work in the Ajiwa reservoir (0.40±0.18). This higher value could be attributed to the gold mining activities taking place in the environment. From another source, 0.81 mg/L lead concentration in Ibrahim Adamu Lake of Jigawa State (Sambo *et al.,* 2014). The amount of lead in the lake was higher than what was obtained in this research. This higher concentration could be due to some contaminants close to the lake. A significantly higher value of lead (5.94±0.74 mg/L) was reported by Imam (2012) in water analyzed along the Bompai-Jakara axis of Kano state. The high value could be due to the discharge of industrial effluents into the water. Mohammed (2023) also reported higher lead concentration values in Tsawa-Tsawa, Garhi and Makera in Zobe Dam. One of the primary environmental problems is the contamination of aquatic habitats by heavy metals. The reason for this is that heavy metals cannot be broken down; rather, they accumulate, incorporate, or combine into water, sediments, and aquatic life, which results in heavy metal pollution (Malik *et al.,* 2010). Results from this research revealed that the percentages of heavy metals in *O. niloticus* were higher than the concentrations in the surrounding water from which the fishes were sampled.

This may either be because these fishes have bio-accumulated heavy metals over time from their diet; or because the heavy metals are lipophilic and as such reside and accumulate in fatty tissues (Ukachikwu, 2012). Furthermore, it was seen that heavy metals accumulated in the fish tissues at concentrations far above WHO, FEPA and FAO (2012) stipulated limits for heavy metals in fish. Heavy metals bio-accumulated at varying levels and were distinguishable in the different tissues. The trend of heavy metal accumulation in the tissues is in the following order; Liver > Gills > Muscle >Gonads. The differences in the levels of accumulation in the different tissues of a fish, according to Ukachikwu, (2012) may primarily be attributed to the differences in the physiological role of each organ. Furthermore, regulatory ability, behavior and feeding habits are other factors that might influence the differences in the rate of accumulation of metals in the organs. This study also revealed that the largest quantities of heavy metals were observed in the liver and gills, whereas the lowest were identified in the muscle tissues. This finding is in agreement with those of other studies regarding the differences in heavy metal accumulation in fish tissues by Abubakar et al. (2023), Ahmad et al. (2020), Musa (2020), and discovered on the gill reflects the concentration of heavy metals that are within the waters where the fish live (Yilmaz, 2007). The concentration of heavy metals in fish indicates the level of metal pollution of the water from which the fish were caught, not surprisingly, a higher concentration of Ukachikuw (2012). The liver and gill in fish are more commonly recommended as environmental indicators of water contamination than any other species (Karadede *et al.,* 2004). Though the liver may not be in direct contact with heavy metals in the water, a high concentration of heavy metals in the liver may reflect its major role in detoxification as well as storage (Ukachikwu, 2012).

The high concentration of heavy metals found in the gills could be attributed to the fact that the gills serve as respiratory organs in fishes and are in direct contact with the contaminated medium (water) through which metal ions are absorbed. Also, gills have the thinnest epithelium of all the tissues and metals can penetrate through the thin epithelia (Bebianno et al., 2004). In addition, the concentration of heavy metals discovered within the gills could be related to the element interacting with the mucus, which is incapable of being eliminated from between the lamellae before tissue is prepared for analysis. (Yilmaz, 2007). However it was observed in this study that there was a remarkably lower concentration of copper in the gills when compared to the liver of fish, this difference can be attributed to the lower binding affinity of copper on the gill surface (Chatterjee *et al.,* 2006) and is in conformity with the reports of Ukachikwu (2012) who stated that copper had a greater likelihood of entering the fish tissues rather than binding on the gills as it has a lower binding affinity metal. The concentration of heavy metals

Cobalt was found in the muscles of *O. niloticus* than the rest of the metals analyzed this is because Cobalt is an essential element to fish and other organisms, with its main role as an intrinsic part of Vitamin B_{12} or Cobalamin.

Table 1. The Mean Physicochemical Parameters of Ajiwa Reservoir, Katsina State. (Sept. – Dec., 2023)

Parameters	Mean±S.D	Range Values		NESREA/USEPA/WHO Limit
		Minimum	Maximum	
pH	7.10±0.48	6.04	8.020	6.5-8.5
Temperature (°C)	22.93±4.02	18.30	31.50	12-25
Transparency (cm)	12.61±2.46	7.50	17.30	5-100
Conductivity (µs/cm)	113.97±10.63	96.00	139.00	1000
DO (mg/L)	6.78±1.09	4.74	8.91	6.0
BOD (mg/L)	3.52±0.88	1.89	4.72	6.0

Table 2. The Mean Physicochemical Parameters of Ajiwa Reservoir with Sampling Stations

Parameter	Station A	Station B	Station C	Station D	P-value

pH	6.93±0.47	7.19±0.43	7.39±0.42	6.89±0.51	0.112
Temperature (ºC)	22.600±3.8411	22.57±3.72	23.09±4.61	23.45±4.59	0.970
Transparency (cm)	11.9250±2.2537	13.54±2.86	12.97±2.77	12.00±1.97	0.511
Conductivity (µs/cm)	114.63±9.36	110.00±10.33	123.25±8.15	108.00±9.05	0.013
Dissolved Oxygen (mg/L)	7.03±1.49	6.35±0.36	6.78±0.87	6.97±1.35	0.606
BOD (mg/L)	3.58±1.02	3.18±0.83	3.66±0.48	3.67±1.15	0.663

Table 3: Mean Monthly Physicochemical Parameters of Ajiwa Reservoir (Sep. – Dec., 2023)

Parameter	September	October	November	December	P-value
pH	7.12±0.42	6.86±0.59	7.38±0.41	7.05±0.43	0.193
Temperature (ºC)	19.99±0.89	28.10±2.38	24.84±0.94	19.39±0.89	0.000
Transparency (cm)	13.90±1.28	9.51±1.81	13.12±2.29	13.90±1.28	0.000
Conductivity (µs/cm)	112.88±9.67	117.75±14.57	108.38±6.88	124.88±9.67	0.355
Dissolved Oxygen (mg/L)	7.50±1.01	5.95±0.81	6.17±0.40	7.50±1.01	0.001
BOD (mg/L)	4.16±0.64	2.71±0.78	3.06±0.32	4.16±0.64	0.000

Table 4: Heavy Metals Concentrations in Water of Ajiwa Reservoir, Katsina State. (Sept. – Dec., 2023)

Parameters	Mean±S.D	Range Values		NESREA/USEPA/WHO Limit
		Minimum	Maximum	
Co (mg/L)	0.45±0.23	0.10	0.79	0.3
Cu (mg/L)	0.05±0.03	0.013	0.15	2.0
Mn (mg/L)	0.06±0.09	0.06	0.45	0.5
Ni (mg/L)	0.10±0.07	0.03	0.26	0.02
Pb (mg/L)	0.40±0.18	0.10	0.87	0.01

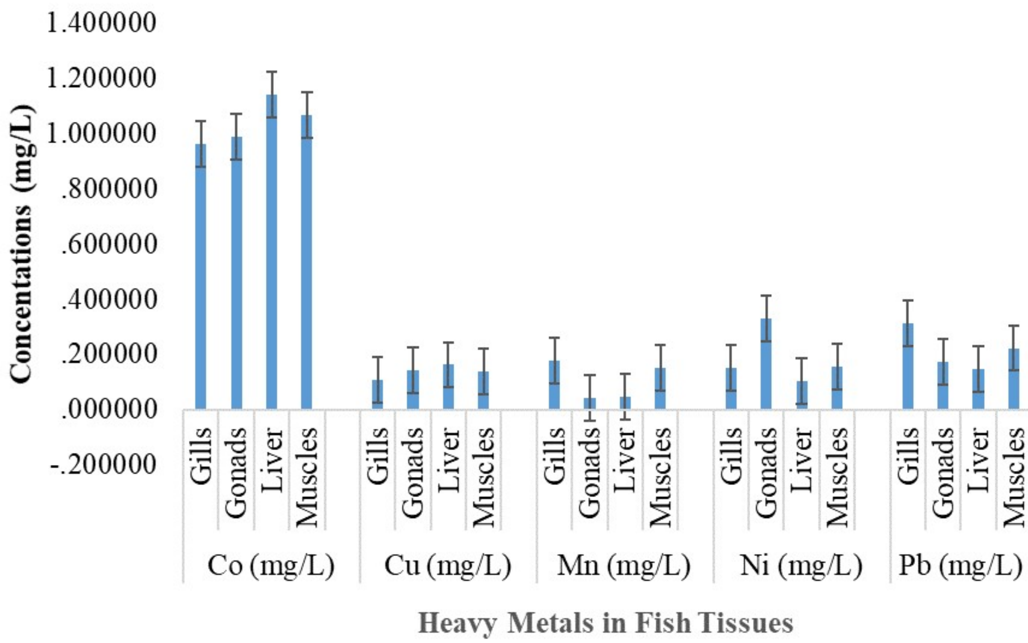

Figure 1: Mean Concentrations of Heavy Metals in Tissues of *Oreochromis niloticus* from Ajiwa Reservoir (Sept. – Dec., 2023).

CONCLUSION

The physicochemical parameters of the Ajiwa reservoir are within NESREA, WHO, and USEPA standards for aquatic life protection. However, heavy metals concentrations in water samples and fish tissues suggest a risk to aquatic organisms and human health.

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