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CONCENTRATIONS AND HEALTH RISK ASSESSMENT OF HEAVY METALS IN WATER, SEDIMENT, AND FISH SPECIES FROM OSE RIVER, ONDO STATE, NIGERIA

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ABSTRACT

Indiscriminate discharge of untreated sewage, effluent and agricultural runoff in most developing countries had been a major problem that has led to increase in deposition of heavy metal in the aquatic system especially the essential metals at a level higher than most of the non-essential ones. Concentrations of chromium (Cr), iron (Fe), nickel (Ni) and zinc (Zn) were investigated in water, sediments and fish of Ose River, Ondo State, Nigeria to determine the potential health risk associated with consumption of fish from the river. A total of 116 fishes were collected between June and November (2019) and analyzed for heavy metal using Atomic Absorption Spectrophotometer. Results show high concentrations of these metals above tolerable limits set by Nigeria Industrial Standards (NIS) and World Health Organization/Federal Environmental Protection Agency for water and sediment respectively except for Ni. The Concentrations of Cr in examined organs (Gill, muscle and liver) exceeded WHO consumption safety limits of 0.15mg/kg. Concentrations of Ni and Zn in all the organs also exceeded permissible limits except the gill and liver of Heterbranchus longifilis respectively. Fe was the only metal below safe limit except in liver of all the fish samples. A positive correlation was seen between metals' concentration in the sediment and fish species. The human health risk assessments show that all Estimated Daily Intake values were lower than Recommended Dietary Allowance except for Cr among fishers. Target Risk and Hazard Index values reveal high risk of both carcinogenic and non-carcinogenic effect respectively, for both the general public and fishers.

Keywords: Bio-accumulation, Essential metals, Health risk assessment, Hazard index, Cancer risk, Estimated Daily Intake

INTRODUCTION

Heavy metals especially the essential ones are known to be indispensable to the growth and development of human and animal body systems but which could be detrimental at higher doses. These metals are required in animal diets and are essential for protein synthesis (Rengel, 1999). Examples of essential metals are copper (Cu), nickel (Ni), cobalt (Co), manganese (Mn), molybdenum (Mo), iron (Fe), zinc (Zn), etc. Iron (Fe), for instance, is one of the most abundant elements in the Earth's crust and about 4 grams in the adult human body, mostly in the haemoglobin and myoglobin, for the transportation of oxygen by blood and storage of oxygen in muscles, respectively (Micronutrient Information Center, MIC, 2022). However, excessive intake of iron has been reported to cause cellular damage to the heart, liver and pancrease leading to shock, liver failure, coma, and adult respiratory distress syndrome (Abbaspour *et al*., 2014). Similarly, Zn is one of the most widely distributed essential metals in the human body with the highest concentration in the prostate. It is required for the function of over 300 enzymes and 1000 transcription factors (Cherasse and Urade, 2017). However, at high concentrations, Zn can suppress the absorption of Cu and Fe. Plants ' absorption of other essential metals can also be inhibited by excessive Zn in the soil (Hamzah *et al*., 2022).

Although Ni has not been confirmed to be an essential metal in humans, its biological role as an enzyme (urease) has been established in some plants, eubacteria, and fungi (Alloway, 2013). Zambelli and Ciurli (2013) argued that Ni affects human health through infections by Ni-dependent bacteria. The tolerable upper intake level was set at 1mg/kg. Several researchers have reported the presence of some essential metals in different environmental matrices at concentrations higher than the non-essential ones such as lead (Pb) and Arsenic (Yılmaz *et al*., 2010; Qui *et al*., 2011; Akpanyung *et al*., 2014; El-Moselhy *et al*., 2014; Varsha *et al*., 2017; Liu *et al*., 2018; Edogbo *et al*., 2020). However, little attention is paid to the possible health risks associated with them.

Ose River is a major perennial river in South-West Nigeria (Ololade and Ajayi 2009; Odedeyi and Fagbenro, 2010; Ekhator *et al*., 2015) that contributes significantly to global freshwater catches. In addition, it serves as a primary source of irrigation and drinking water for some communities from the Dam built across it (Talabi, 2018). Ose River originated from the Apata hills, Ikere, Ekiti State and flows through Ose Local Government area in Ondo State, thereafter, it empties into the Benin River (one of the four major rivers that drain into the Atlantic Bight of Benin). It lies between longitudes 5°20'E to 6°10'E and latitudes 6°20'N to 8°00'N (Odedeyi and Fagbenro, 2010). Several streams and creeks drain into the river with lots of human activities such as construction, automobile car wash, open dump sites

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(including electronic wastes) and farming along its banks (Ololade and Ajayi 2009). Thus, it is imperative to assess the level of some biologically essential heavy metals classified by Gupta (2018) in water, sediment and fish and to find out the potential health risks associated with the consumption of fish from freshwater rivers. The objective of this study was to quantify the concentration of Fe, Ni,Cr and Zn in water, sediments, and organs of *Heterobranchus longifilis, Clarias angularis*, *Parachanna obscura*, *Sarotherodon galilaeus*, *Clarias gariepinus,* and *Oreochromis niloticus* from Ose River and to determine the potential health risk associated with the consumption of these fishes.

MATERIALS AND METHODS

Sample collection

Water, sediments, and fish samples were collected from Ose River, Ondo State, Nigeria (Fig. 1) from June - November 2019. A total of 116 fish samples (comprises of 6 species) were procured from local fishermen at the landing site during the six months sampling period (Table 1). The fish samples were kept in an ice-pack igloo cooler to prevent spoilage and transported to the Animal and Environmental Biology laboratory, Adekunle Ajasin University, Akungba-Akoko for analysis (Olatunji-Ojo *et al*., 2019; Olayinka-Olagunju *et al*., 2021). The samples were kept in the refrigerator at 4°C until analysis.

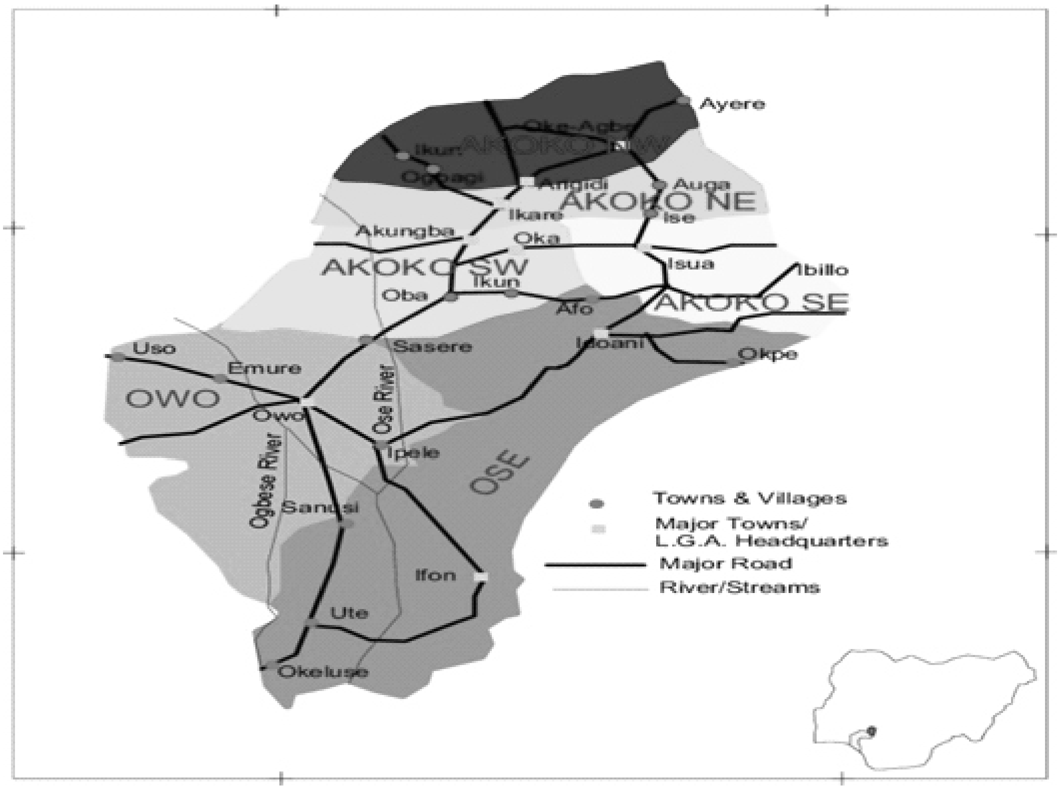

Fig. 1: Map showing Ose River (Falowo, 2017)

Table 1: Ecological characteristics and morphometrics of selected fish species.

Fish species	Habitat	Feeding habit	No of samples	Length (cm)	Weight (g)
Parachanna obscura	Benthic	Carnivorous	35	20.5-39.7 (28.76)	67-602 (226.95)
Clarias angularis	Benthic	Carnivorous/ herbivore	13	20.5-30.0 (24.46)	67-195 (111.57)
Clarias gariepinus	Benthic	Carnivorous	9	21.9-41 (33.91)	71-464 (293)
Heterobranchus longifilis	Benthic	Carnivorous/ herbivorous	10	23.3-41.5 (32.86)	111-525 (298.14)
Sarotherodon galilaeus	Pelagic	Detritivorous, herbivorous (alguivorous) and omnivorous	15	12-22.2 (17.27)	35-228 (112.22)
Oreochromis niloticus	Pelagic	omnivorous	34	15-28.7 (18.11)	51-385 (121.37)

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Laboratory Analysis

Sediment and Water Analysis

The sediments were air-dried, ground, and sieved in a 2mm mesh sieve to obtain a fine homogeneous powder in the laboratory. Digestion was done using the methods described by Yi *et al*. (2011). From the fine sediment powder, 2g were weighed and transferred into test tubes. Furthermore, 10 mL of nitric acid (HNO_3) was added to the test tubes containing the sediment samples and was allowed to stay overnight at room temperature in the fume cupboard. The mixture was digested and allowed to boil in a water bath set at 100°C for 2 h. After cooling, the digests were filtered and poured into 25 mL volumetric flasks up to the mark by adding 1% nitric acid. The metal concentrations were determined from the digest using an Atomic Absorption Spectrophotometer.

Furthermore, the water samples were filtered using Whatman filter paper. 50 mL of the filtrate was acidified using HNO_3. Water digestion was done by adding 5 mL of HNO_3 to the filtrate, boiled at a temperature of 130°C until a light color change was observed (Yi *et al*., 2011). Additionally, more HNO_3 was added until the solution color was cleared. The solution was allowed to cool and then filtered using Whatman filter paper. The digested solution was washed with 0.1M of HNO_3 and poured into a 100 mL volumetric flask and allowed to reach the mark with deionized water.

Fish sample analysis ,

Fish samples were weighed and dissected and fish tissues (livers, gills, and muscle) were oven-dried at 105°C for 1 h. The tissues were collected in triplicates. 1 g of each tissue was weighed into a crucible, charred at low flame, and later ashes for 2 h in a muffle furnace, at a temperature 520°C. The ashes were then dissolved in 20cm^3 of 20% (V/V) Nitric acid, and filtered into 100cm^3 ammonium acetate (pH 7) was added. The samples were then agitated with a mechanical flask shaker (Griffins) for 2 hours. The contents were centrifuged and the supernatant was poured into a 100 cm^3 volumetric flask and made to mark volume with ammonium acetate solution. All digested samples were analyzed for heavy metals (Zn, Fe, and Ni) using the Atomic Absorption spectrophotometer (AAS Buck Scientific 210 VGP model).

1.2.3 Health risk assessment

1.2.3.1 Estimated Daily Intake (EDI)

EDI is an important ecological tool that describes how a person may be exposed to a certain pollutant per day/year. EDI was compared with Provisional Tolerable Daily Intake (PTDI) suggested by Joint Expert Committee on Food Additives (JECFA, 1993). The ingested dose was equal to the absorbed pollutant dose and cooking did not affect the pollutants (United States Environmental Protection Agency, USEPA, 1989; Chien *et al*., 2002). Using the formula of Ahmed *et al*. (2019):

$$\mathbf{EDI} = \frac{Con_m \times FIR}{Bwa}$$

Where Con_m is the average concentration of individual metal, FIR_g and FIR_f is the food ingestion rate for the general populace (36 g/person/day) and fishers (300 g/person/day) respectively, and Bwa is the average body weight (70 kg). The edible parts (muscle) were used for the calculation.

1.2.3.2 Determination of target hazard quotient (THQ)

THQ was used to estimate the risk of non-carcinogenic effects. When it was less than 1, the exposure level is less than the RfDo (Kortei *et al*., 2020). The model described by Chien *et al*. (2002) was used for estimating THQ by the following equation:

$$\mathbf{THQ} = \frac{EDI}{RFDo}$$

RfDo is the oral reference dose (Fe - 7.0 x 10^{-1}, Ni - 2.0 x 10^{-2}, Cr - 3.0 x 10^{-3}, Zn - 3.0 x 10^{-1})

Determination of Hazard Index (HI)

HI estimates the risk assessment of multiple contaminants/toxicants present in a fish. According to the method of Chien *et al*. (2002), HI is expressed as the arithmetic sum of the individual metal THQ value:

HI = THQ (toxicant 1) + THQ (toxicant 2) +…+ THQ (toxicant n)

Determination of Target Cancer Risk (TR)

TR is used to estimate the carcinogenic risk of examined/analyzed metals. It is simply expressed as:

TR= EDI x CSF

Where CSF is the Cancer Slope Factor; Ni - 9.0 x 10^{-1}, Cr - 0.5. $TR<10^{-6}$ means the result is negligible for carcinogenic (Ahmed *et al*., 2016), $TR>10^{-4}$ means risk unacceptable (USEPA, 2010), and $10^{-6}>TR<10^{-4}$ means risk is within the acceptable range (Showqi *et al*., 2018).

Statistical analysis

The mean and standard deviation were analysed using the Analysis of Variance (SPSS 24 statistical package) with a PostHoc test (Tukey) to determine the level of significance. Pearson correlation was used to determine the relationships between the mean concentration of metal in water and pelagic, sediment and benthic fish species respectively.

RESULTS

Concentrations of heavy metals in water and sediment

The average concentration of each metal in the water and sediment sample of Ose River is shown in Table 2. There is significant difference ($p<0.05$) in the average concentration of each metal with Zn having the highest concentration in both sediment and water.

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Table 2: Comparison of the average concentration of metals in water and sediment with the permissible limits

Sample	Heavy metal	Aver. Conc. (ppm)	Permissible limit (ppm)
Water	Cr	0.05 ± 0.05^{a}	0.05
	Fe	0.44 ± 0.65^{a}	0.30
	Ni	0.01 ± 0.00^{a}	0.02
	Zn	2.74 ± 1.11^{b}	3.00
Sediment	Cr	0.36 ± 0.35^{a}	0.1
	Fe	1.69 ± 2.22^{ab}	0.03
	Ni	0.02 ± 0.01^{a}	0.02
	Zn	3.29 ± 0.98^{b}	<1

Mean±SD with different superscripts in the same column per sample are significantly different ($p<0.05$). Permissible limits of Nigerian Industrial Standards (NIS, 2007) and WHO/FEPA (2003) were used for water and sediment respectively.

Mean Concentrations of essential metals in fish

Table 3 compares the concentrations of each metal in the gills, muscles, and liver of different fish species used for the study respectively. Metal concentrations in gills across all the fish species were low compared to other organs. Muscles of *C. angularis* recorded the highest value for Zn (83.08±47.41 mg/kg) while the livers of all the fish samples had the highest average metal concentration which was significantly different from other organs at $p<0.05$. From the study, *S. galilaeus* had the lowest concentration of metals in its muscle, followed by *C. gariepinus* while *C. angularis* recorded the highest value followed by *P. obscura*. The lowest concentration of metal, Ni (4.58±3.29 mg/kg), was recorded in *P. obscura* liver while the highest concentration, Fe (375.55±224.30 mg/kg), was recorded in *S. galilaeus*. Table 3 also reveals that *O. niloticus* had the highest metal concentrations in its liver followed by *S. galilaeus*.

Table 3: Comparison of average metal concentrations (mg/kg dry weight) in fish tissues with international standards.

Fish species	Tissues	Heavy metals (mg/kg dry weight)			
		Fe	Cr	Ni	Zn
P. obscura	Gill	51.30 ± 19.08^{a}	4.25 ± 0.93^{a}	6.02 ± 2.10^{a}	40.98 ± 10.86^{a}
	Muscle	65.73 ± 18.84^{ab}	4.01 ± 2.05^{a}	6.69 ± 4.08^{a}	43.33 ± 13.01^{ab}
	Liver	75.01 ± 18.19^{b}	8.09 ± 3.38^{b}	4.58 ± 3.29^{a}	60.18 ± 24.01^{b}
C. angularis	Gill	38.45 ± 5.43^{a}	1.96 ± 1.85^{a}	1.00 ± 1.58^{a}	73.26 ± 8.61^{a}
	Muscle	58.89 ± 15.65^{a}	5.79 ± 3.40^{ab}	3.88 ± 0.53^{a}	83.08 ± 47.41^{a}
	Liver	170.89 ± 36.50^{b}	13.58 ± 9.95^{b}	13.87 ± 6.77^{b}	156.03 ± 6.56^{b}
C. gariepinus	Gill	29.37 ± 12.82^{a}	1.30±0.00	4.70±0.00	65.61 ± 12.88^{a}
	Muscle	47.06 ± 21.49^{a}	6.18±3.61	3.24±0.00	42.96 ± 45.78^{a}
	Liver	191.77 ± 21.83^{b}	9.27±12.35	10.4±9.05	153.97 ± 6.17^{a}
H. longifilis	Gill	39.45 ± 3.36^{a}	5.05 ± 0.74^{b}	0.03±0.00	69.82 ± 0.96^{a}
	Muscle	44.08 ± 2.45^{a}	2.93 ± 053^{a}	3.73±0.68	49.55 ± 1.09^{b}
	Liver	130.59 ± 1.38^{b}	12.39 ± 0.66^{c}	5.49±1.50	14.43 ± 0.76^{c}
S. galilaeus	Gill	61.08 ± 5.91^{a}	11.29 ± 6.36^{a}	2.32 ± 0.02^{a}	40.97 ± 3.42^{a}
	Muscle	49.09 ± 19.83^{a}	6.47 ± 2.00^{a}	6.07 ± 1.48^{b}	35.63 ± 15.26^{a}
	Liver	375.55 ± 224.35^{b}	10.39 ± 2.27^{a}	14.43 ± 2.33^{c}	193.29 ± 124.58^{b}
O. niloticus	Gill	50.24 ± 17.92^{a}	8.03 ± 3.17^{a}	3.47 ± 2.20^{a}	66.36 ± 7.55^{a}
	Muscle	46.13 ± 9.01^{a}	5.64 ± 2.84^{a}	6.79 ± 10.34^{ab}	44.69 ± 10.64^{a}
	Liver	285.62 ± 217.40^{b}	35.17 ± 28.97^{b}	12.35 ± 6.60^{b}	273.22 ± 232.46^{b}
WHO/FAO (2003)	**Tissue**	**100**	**0.15**	**-**	**40**
FEPA (2003)	**Tissue**	**-**	**0.15**	**0.5**	**-**

Mean±SD with different superscripts in the same column per fish samples are significantly different ($p<0.05$).

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Health Risk Assessment

The EDI among the general populace and fishers is shown in Table 4. EDI ranged from 1.67x10^{-3} to 4.27x10^{-2} and 1.39x10^{-2} to 3.56x10^{-1} mg/kg/day in the general populace and fishers respectively. All values for each metal were below the PTDI. The THQs and TRs values are shown in Table 5 while Fig 2 shows the HI values of the general populace and fishers.

Table 4: Comparison of Estimated Daily Intake (EDI) for General populace and Fishers with Provisional Tolerable Daily Intake (PTDI)

Fish species		**Fe**	**Cr**	**Ni**	**Zn**
P. obscura	General populace	3.38x10^{-2}	2.06x10^{-3}	3.44x10^{-3}	2.23x10^{-2}
	Fishers	2.82x10^{-1}	1.72x10^{-2}	2.87x10^{-2}	1.86x10^{-1}
C. angularis	General populace	3.03x10^{-2}	2.98x10^{-3}	2.00x10^{-3}	4.27x10^{-2}
	Fishers	2.52x10^{-1}	2.48x10^{-2}	1.66x10^{-2}	3.56x10^{-1}
C. gariepinus	General populace	2.42x10^{-2}	3.18x10^{-3}	1.67x10^{-3}	2.21x10^{-2}
	Fishers	2.02x10^{-1}	2.65x10^{-2}	1.39x10^{-2}	1.84x10^{-1}
H. longifilis	General populace	2.26x10^{-2}	1.51x10^{-3}	1.92x10^{-3}	2.55x10^{-2}
	Fishers	1.89x10^{-1}	1.26x10^{-2}	1.60x10^{-2}	2.12x10^{-1}
S. galilaeus	General populace	2.52x10^{-2}	3.33x10^{-3}	3.12x10^{-3}	1.83x10^{-2}
	Fishers	2.10x10^{-1}	2.77x10^{-2}	2.60x10^{-2}	1.53x10^{-1}
O. niloticus	General populace	2.37x10^{-2}	2.90x10^{-3}	3.49x10^{-3}	2.30x10^{-2}
	Fishers	1.98x10^{-1}	2.42x10^{-2}	2.91x10^{-2}	1.92x10^{-1}
PTDI		**8**†	**0.035***	**1**‡	**11**†

†Recommended Dietary Allowance (RDA), *Adequate Intake/day (AL), ‡Tolerable Upper Intake Level/day (UL). Source: DRI, (Bassey and Chukwu, 2019).

Table 5: Estimated THQs and TR of metals from consumption of different fish species from Ose River for both general populace (GP) and fishers (F)

Heavy metals	**Fish**	**THQ(GP)**	**THQ(F)**	**TR(GP)**	**TR(F)**
Fe	*P. obscura*	4.83 x 10^{-2}	4.02 x 10^{-1}	-	-
	C. angularis	4.33 x 10^{-2}	3.61 x 10^{-1}	-	-
	C. gariepinus	3.46 x 10^{-2}	2.88 x 10^{-1}	-	-
	H. longifilis	3.23 x 10^{-2}	2.70 x 10^{-1}	-	-
	S. galilaeus	3.61 x 10^{-2}	3.01 x 10^{-1}	-	-
	O. niloticus	3.39 x 10^{-2}	2.82 x 10^{-1}		
Ni	*P. obscura*	1.72 x 10^{-1}	1.43	3.13 x 10^{-3}	2.61 x 10^{-2}
	C. angularis	9.98 x 10^{-2}	8.31 x 10^{-1}	1.82 x 10^{-3}	1.51 x 10^{-2}
	C. gariepinus	8.33 x 10^{-2}	6.94 x 10^{-1}	1.52 x 10^{-3}	1.26 x 10^{-2}
	H. longifilis	9.59 x 10^{-2}	7.99 x 10^{-1}	1.75 x 10^{-3}	1.45 x 10^{-2}
	S. galilaeus	1.56 x 10^{-1}	1.30	2.84 x 10^{-3}	2.37 x 10^{-2}
	O. niloticus	1.75 x 10^{-1}	1.46	3.18 x 10^{-3}	2.65 x 10^{-2}
	P. obscura	7.43 x 10^{-2}	6.19 x 10^{-1}	-	-
	C. angularis	1.42 x 10^{-1}	1.19	-	-

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Zn	*C. gariepinus*	7.36×10^{-2}	6.14×10^{-1}	-	-
	H. longifilis	8.49×10^{-2}	7.08×10^{-1}	-	-
	S. galilaeus	6.11×10^{-2}	5.09×10^{-1}	-	-
	O. niloticus	7.66×10^{-2}	6.38×10^{-1}	-	-
Cr	*P. obscura*	0.69	5.73	1.03×10^{-3}	8.59×10^{-3}
	C. angularis	0.99	8.27	1.49×10^{-3}	1.24×10^{-2}
	C. gariepinus	1.06	8.83	1.59×10^{-3}	1.32×10^{-2}
	H. longifilis	0.50	4.19	7.5×10^{-4}	6.28×10^{-3}
	S. galilaeus	1.11	9.24	1.66×10^{-3}	1.39×10^{-2}
	O. niloticus	0.97	8.06	1.45×10^{-3}	1.21×10^{-2}

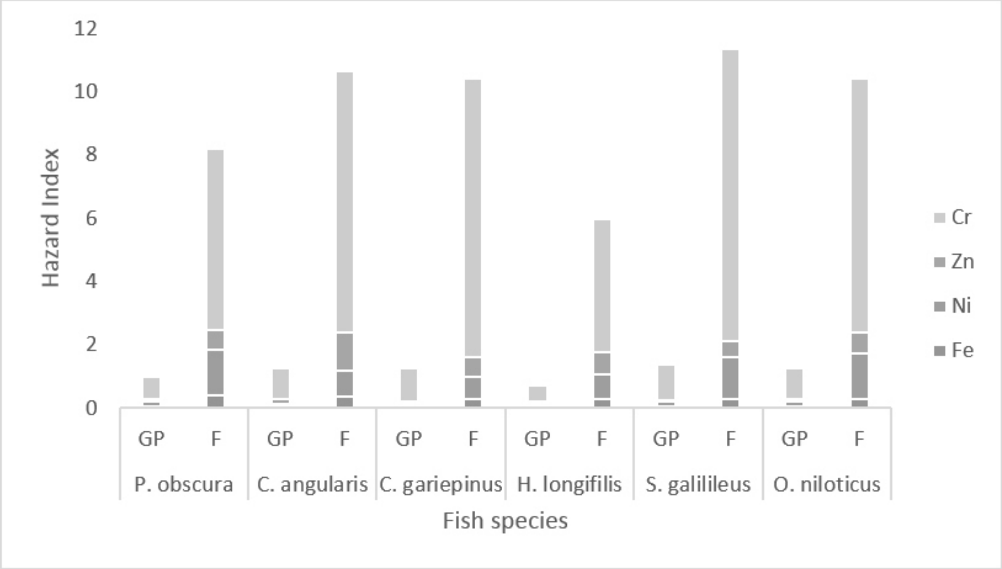

Fig 2: Estimated HI of fish species from simultaneous consumption of multiple metals from Ose River for both general populace (GP) and fishers (F)

Correlation coefficients

Table 6 shows the correlation between metal concentrations in water and pelagic fish species of Ose River. The result shows a positive correlation between Fe and Ni (0.99), Fe and Zn (0.87), and Zn and Ni (0.88). There is also strong positive correlation between Cr and the other three metals. There was a significant correlation between Fe and Ni at $p<0.05$. There was also a high positive correlation (0.93) between Fe and Ni in the sediment and benthic fish species (Table 7) which was a significant correlation at $p<0.05$. This suggests that these two metals tend to co-occur at similar levels in both sediment and fish samples. Fe and Zn show moderately positive correlation of 0.81 which is slightly weaker than Fe and Ni. However, there is a lower positive correlation between Ni, Zn, and Cr (0.58 and 0.56) when compared to others.

Table 6: Correlation coefficients of metal concentrations in water and pelagic fish species of Ose River

	Fe	Ni	Zn	Cr
Fe		0.9997	0.8747	0.9480
Ni			0.8849	0.9546
Zn				0.9835
Cr				

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Table 7: Correlation coefficients of metal concentrations in sediment and benthic fish species of Ose River

	Fe	Ni	Zn	Cr
Fe		0.9342	0.8083	0.7946
Ni			0.5789	0.5615
Zn				0.7861
Cr				

DISCUSSION

Concentrations of heavy metals in water and sediment

The mean concentrations of metals in the water and sediment of Ose River were in the same trend as Zn>Fe>Ni and are significantly different at $p<0.05$. This implies that metals in Ose River have the same distribution, abundance, and occurrence in water and sediment. The concentration of Fe in both water and sediment exceeded the standard stipulated by NIS (2007) and WHO/FEPA (2003). This could be an indication of high input of Fe from either natural or anthropogenic sources. The composition of Nigeria soil have also been reported to be very rich in Fe and may be a contributing factor to the abundance of Fe in most of the examined sediments in Nigeria rivers (Ezemonye *et al*., 2019; Olatunji-Ojo *et al*., 2019; Olayinka-Olagunju, 2021). This is similar to the findings of Imiuwa *et al*. (2014) who reported high concentrations of Fe (619.70 ± 95.70 to 1117 ± 322.40 ppm) in the sediment of Ikpoba River, Nigeria. Similarly, average concentrations of 110.53 ± 101.54 and 89.93 ± 88.36 ppm were reported for dry and wet seasons respectively for Fe in the sediment of Ase River, Nigeria (Iwegbue *et al*., 2007). An average of 12,461.25 and 18,407 mg/kg of Fe was analyzed from soil samples from an open dumpsite in Nigeria (Olayinka-Olagunju *et al*., 2019). These high values were, however, attributed to the dumping of electronic waste and other Fe-laden wastes. In comparison with other countries, Fe had the highest concentrations among the seven metals examined in Rivulet water, India (Javed and Usmani, 2014). Therefore, it is important to regulate the various sources of Fe contamination by enacting and enforcing environmental laws that will reduce its input into the environment. Nickel concentrations in water samples were below NIS (2007) standards for drinking water and within the tolerable range set by WHO/FEPA (2003) for sediment. Ezemonye *et al*. (2019) reported the occurrence of Ni in all examined samples throughout the sampling periods at a level slightly lower than WHO's limits for water.

The concentration of Zn in water was 26% lower than NIS (2007) permissible limits but exceeded WHO/FEPA (2003) limits for sediment. This agrees with the findings of Ezemonye *et al.* (2019) and Maurya *et al.* (2019). The Concentrations of Cr in water samples were also within the Nigerian Industrial Standards (NIS, 2007) tolerable limits but exceeded WHO/FEPA (2003) limits for sediment. This correlates with the finding of Tesfamariam *et al*. (2016) but disagrees with Pradip *et al*. (2019) that reported a high concentration of Cr in river water from five sampling sites in India, above prescribed safe limits.The present concentration level of Cr in sediment may not affect the survival of most aquatic lives as reported by Besser *et al*. (2004). However, accumulating chronic concentration of Cr may induce a variety of adverse effects in aquatic lives.

Mean Concentrations of essential metals in fish

The lowest metal concentration in fish was recorded in Ni (*H. longifilis*), while the highest was observed in Zn (*C. angularis*). This result is similar to the findings of Izegaegbe and Oloye (2017) who reported a high concentration of Zn at a level higher than Ni in Ilushi River, Nigeria. In this study, the following trend was observed liver>gills>muscle and the trend corroborated the reports from previous studies within Nigeria that the liver of fish species had the highest concentrations of essential metals (Aladesanmi and Awotoye, 2014; Ahmed *et al*., 2015; Wolf and Wolfe, 2005). The trend observed may be associated with the fact that the liver plays a vital role in the metabolism of xenobiotic compounds (Islam *et al*., 2018).

Iron is known to be the fourth most abundant essential metal in nature. Its concentrations in the livers of fish samples in the present study exceeded the tolerable limit set by WHO/FAO (2003) except *P. obscura* but its concentrations in the muscle of all the fish species were below the consumption safety limits (Table 3). This agrees with the findings of El-Moselhy *et al*. (2014) and Olayinka-Olagunju *et al.*, (2021) that recorded a low concentration of Fe in the organs of fish samples. Iron is the most abundant metal in examined tissues and organs in the present study. This is consistent with several reports (Yılmaz *et al*., 2010; Ahmad and Sarah, 2015; Okorafor *et al*., 2015; Olayinka-Olagunju *et al*., 2021) and could be attributed to its abundance in nature and its usage as a component of fertilizer around the study location.

The concentration of Ni in all fish tissues exceeded both national and international standards of 0.5 mg/kg and 0.2 mg/kg, respectively except for the gill of *H. longifilis*. Ezemonye *et al.* (2019) also reported a high concentration of Ni in both shrimp and fish species of Benin River, Nigeria. This observation could be dangerous to health as a high intake of Ni has been reported to increase the incidence of lung and nasal cavity cancer (Okorafor *et al*., 2015). The present result agrees with the findings of Ahmed *et al*. (2015) who reported that Ni concentrations in most of the examined fish samples exceeded the WHO standard for food. Although acute poisoning of Ni is rare, prolonged chronic exposure to lower doses may result in asthma, dermatitis, lung fibrosis, and respiratory tract cancer (Kasprzak and Salnikow, 2007). The concentration

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of Zn in all fish tissues, except the liver of *H. longifilis* exceeded the WHO/FAO (2003) and FAO (2001) permissible limits. The lowest concentration of Zn in the sample muscle (35.63±15.26 mg/kg) was higher than the value (0.21±0.08 mg/kg) reported by Ololade and Ajayi, (2010) approximately ten (10) years ago from the same river. High concentrations of Zn in fish species have also been reported by several researchers (Akpanyung *et al*., 2014; Javed and Usmani, 2014; Ahmed *et al*., 2016; Varsha *et al*., 2017). This result however differs from the findings of Olayinka-Olagunju *et al*., (2021) who reported lower concentrations of Zn in fish organs from Ogbese River, Ondo State, Nigeria.

Cr concentrations in all the fish tissues exceeded different international standards of WHO/FAO (2003) and FEPA (2003). The lowest concentration, recorded in the gill of *C. angularis* is nine-fold higher than the tolerable limit of 0.15mg/kg while S. *galilaeus* has the highest concentration. This agrees with the findings of Edogbo *et al*. (2020) that reported high concentration of Cr in soil, vegetables, water and fish from Challawa area (Nigeria) including the river. This was, however, attributed to the discharge of untreated effluent from textile industries and excessive applications of fertilizer by farmers around the river. Although, Cr is an essential dietary mineral whose biologically usable form plays vital role in glucose metabolism but its occurrence in the environment at high doses is a major call for public health intervention. Öztürk *et al*. (2009), also reported high concentration of, Cr in fish samples from Avsar dam lake, Turkey, at a level higher than the tolerable limit.

Health Risk Assessment

Health risk assessment is a significant tool for evaluating metals' potential toxic effects through daily intake. Table 4 reveals the EDI of the general populace and fishers. All EDI values of Fe for both the general populace and fishers were lower than the Recommended Dietary Allowance (RDA) of 8 mg/kg/day. This correlates with the findings of Ezemonye *et al*. (2019), who reported that the EDI value for Fe was below the reference dose for oral exposure. However, care must be taken when consuming Fe-laden fish as the excess amount of iron has been reported to cause a rapid increase in pulse rate and coagulation of blood in blood vessels, hypertension, and drowsiness (Bakare-Odunola, 2005; Lawrence, 2014; WHO, 2008). The THQs of Fe for the fishers were slightly below 1, thus indicating that the consumption rate of fish from Ose River should be controlled and the discharge rate or source of pollution should be monitored.

All EDI values for Ni were below the Tolerable Upper Intake Level/day (UL) of 1 mg/kg/day. Ezemonye *et al.* (2019) also reported low EDI for Ni. However, the THQs of Ni for *P. obscura*, *S. galileus*, and *O. niloticus* for the fishers were above 1 while others for the general populace and fishers were below 1. This shows that individual metal such as Ni can pose serious non-carcinogenic health effects through the consumption of the three fish species. It also has a high carcinogenic risk as TR values for both the general populace and fishers were higher than the USEPA limit of $1x10^{-4}$. A high intake of Ni has been implicated in increasing the incidence of nasal cavity and lung cancer (Varsha *et al.*, 2017). The TR results of all the five species examined by Ahmed *et al*. (2016) posed carcinogenic risks from Ni consumption. It is, therefore, important to monitor the usage and discharge of Ni into the environment. The values of Zn EDI were lower than the RDA value of 11 mg/kg/day. However, the THQ for fishers was above 1 from the consumption of *C. angularis*. Thus, showing that fishers are more vulnerable to health hazards associated with a high intake of Zn.

Estimated daily intake of Cr for both the general public and fishers in all the examined fish species were below Adequate Intake/day level (AL) of 0.035. However, the THQs for the fishers were above 1, thus, indicating that intake of Cr alone through consumption of fish species from Ose River may have non-carcinogenic health adverse effect throughout the life expectancy of 54.81 years. THQs of Cr in *Orechromis niloticus* from Azuabie and Okuluagu-Ama Creeks, Nigeria, were, however, below 1 (Ekweozor *et al*., 2017). The Carcinogenic risk of Cr in all examined species were greater than 10^{-4}, thus, indicating a high risk of carcinogenic effects for both general public and fishers.

The degree of toxicity of essential metals to humans depends on daily intake, and perennial intake of contaminated fish may likely induce adverse effects (Edogbo *et al*., 2020). Although the evaluation of non-carcinogenic health risks using THQ does not have a dose-response relationship of the examined metals (USEPA, 1989), however, humans can be adversely affected by the simultaneous consumption of multiple pollutants from contaminated food (Kwaansa-Ansah *et al*., 2019). Hence, the use of HI. All HI results for the fishers were greater than 1 with the range of 5.97 to 11.35 (Fig 2). Pinzon-Bedoya *et al*. (2020) reported that HI shows no evidence of potential health risk except for Cu and Hg in five of the most consumed fish species in Colombia. However, HI values were greater than 1 in shrimp and fish samples from Benin River, Nigeria (Ezemonye *et al*., 2019). Thus, indicating possible health risks from the consumption of shrimp and fish from the river. Ahmed *et al*. (2016) also reported that HI values of six commercially important fish species in Bangladesh show that children were approximately six (6) times more susceptible to non-carcinogenic health effects than adults. There is also strong positive correlation between each examined metal in the fish samples and their immediate environment. There is, therefore, a need to consciously control the usage and discharge of most heavy metals, including the essential ones, into the environment.

CONCLUSION

The present study examined concentrations of essential metals in water, sediments and fish tissues and organs. The results showed higher concentrations of all metals in water and sediments except for Ni. Also, the concentrations of these metals were higher in sediment than in water and exceeded international tolerable limits for sediment, except Ni. This could be because of its ability to serve as a sinking bed for most pollutants. The study also revealed that most of the examined metals exceeded consumption safety limits set by national and international agencies for the fish samples. There is strong

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positive correlation between each metal in the fish samples and their immediate environment. The TR and HI showed that there is potentially carcinogenic health risk for consumers especially fishers. Therefore, it is important to regulate the discharge of waste into the river body. Fishing and other activities such as farming along the river's course should also be monitored. Heavy effects of non-essential metals such as lead (Pb) and Arsenic (As) should be examined in future studies.

Declarations

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Competing interests

The authors declare that they have no competing interests.

Availability of data and material

The datasets used and analyzed during the current study are available from the corresponding author on reasonable request.

Code Availability

Not applicable

Authors contribution

OAM and JOO designed the study, experimented and, analysed the data; OPF, KEO and FFO experimented and collected the data. OAM wrote the draft of the manuscript while AAD proofread the manuscript. DOO supervised the research. All authors approved the manuscript for submission.

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