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COMPARATIVE ASSESSMENT OF SELECTED HEAVY METAL LOAD IN THREE TILAPIINE SPECIES INHABITING OSINMO RESERVOIR, SOUTHWESTERN NIGERIA

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ABSTRACT. The levels of selected heavy metal in the water and fillets of *Tilapia zillii* Gervais, *Sarotherodon galilaeus* Trewavas and *Oreochromis niloticus* Lineaus in Osinmo Reservoir were assessed and compared with established regulatory limits of WHO and FEPA with a view to providing information on the water and the fish fillet heavy metal load as well as the fillet bio-accumulative potential as a likely indicator for human fish consumption safety. Water samples and fish specimens were collected monthly from Osinmo Reservoir, Ejigbo, Southwestern Nigeria for a period of 6 months. The descaled-dried fish fillet and water samples were digested and analysed for lead, chromium, iron, zinc and cadmium using Atomic Absorption Spectrophotometer (A.A.S.). The data obtained were subjected to descriptive and inferential statistics using SPSS 21. The results showed that zinc concentration which was the highest in the water samples and ranged between 144 $\mu g\ l^{-1}$ and 288 $\mu g\ l^{-1}$ was also the highest in the fillets of all the cichlid species. However, the concentrations were within the mandatory regulatory limits. The result of the study also revealed significant differences ($P < 0.05$) in heavy metal loads of the fish fillets between the cichlid fish species from Osinmo Reservoir. Irrespective of the month of sampling, the heavy metal levels in fish fillets samples were found to increase in the order: *O. niloticus* > *T. zillii* > *S. galilaeus.* However, the levels of lead (11.00–26.00 $\mu g\ l^{-1}$ and 15.00–31.00 μgL^{-1}) and cadmium (6.33–13.00 $\mu g\ l^{-1}$ and 2.33–12.33 $\mu g\ l^{-1}$) which was the least in both water and fish fillet respectively, were above the recommended regulatory acceptable limit of WHO and FEPA. The study concluded that the elevated levels of lead and cadmium in the water and the fish fillet samples assayed indicated that the two elements negatively impacted the fish fillet quality, thereby raising human health consumption safety issues.

Introduction

The steady increase in the industrial revolution has led to the increase in the pollution of the aquatic environment especially with heavy metals which are indestructible and most of which have toxic effects on organisms (MacFarlane, Burchett, 2000). Apart from the industrial revolution, human activities have been associated with increase in the concentration of metals in many of the natural water systems which have raised concerns regarding metal bio-accumulation and human health hazards (Ekpo *et al*., 2013). Documented sources of how heavy metals enter aquatic systems from natural sources and other anthropogenic activities include industrial or domestic sewage that contain sediments with huge quantities of inorganic anions and heavy metals (ECDG, 2002; Ekpo *et al*., 2013), leaching from landfills/dumpsites, atmospheric deposits, and run-offs from agricultural land and drainage water which have been reported to contain pesticides and fertilizers (Edem *et al*., 2008).

Heavy metal pollutants which compare with other types of aquatic pollution have been reported to be less visible, but its effects on the ecosystem and humans are intensive and very extensive due to their toxicity and their ability to accumulate in the aquatic organisms (Svobodova *et al*., 2004). Heavy metals when present in the water accumulate in tissues and organs of aquatic organism to a level that might affect their physiological state especially fish and thereby incorporated into food chain (Akan *et al*., 2012; Ekpo *et al*., 2013).

Fish are nutritious and essential food items which provide high-quality protein, vitamins, minerals and omega-3 fatty acids (which have been associated with health benefits due to their cardioprotective effects) (Wim *et al*., 2007; Gamal, Shamery, 2010). Despite the many health benefits associated with fish in a diet, there are also some health risks related to fish consumption, mainly due to potential adverse effects of heavy metal contamination (Mansour, Sidky, 2002).

The accumulation of heavy metals within fish varies with route of uptake, type of heavy metal, species of fish concerned, physiology and metabolism (Olatunji, Osibanjo, 2012; Amita *et al*., 2012). The exposure of fish and other aquatic organism to heavy metals in aquatic environment has also been shown to be a function of the concentration of the dissolved metals in the water bodies. Multiple factors including season, physical and chemical properties of water have also been reported to play a significant role in metal accumulation in different fish tissues (Hayat *et al*., 2007). The accumulation of these contaminant residues may ultimately reach concentrations hundreds or thousands of times above those measured in the water, sediment and food (Goodwin *et al*., 2003; Osman *et al*., 2007). For this reason, monitoring fish tissue contamination serves as an early warning indicator of related water quality problems (Barak, Mason, 1990; Mansour, Sidky, 2002), enables detection of toxic chemicals in fish that may be harmful to consumers, and ensure appropriate action to protect public health and the environment (Adefemi *et al*., 2008).

Fish is an important component of the human diet in Ejigbo and its environs, most of which are sourced for in Osinmo Reservoir, Ejigbo, Southwestern, Nigeria. Several studies have been done on Osinmo reservoir (Adewumi *et al*., 2014; Adedeji *et al*., 2015), but heavy metals bio-accumulation potential in the organ and tissue of the tilapiine species in Osinmo reservoir have not been reported. This study, therefore, intends to determine the concentration of heavy metals in the water and fillets of the tilapiine species from Osinmo reservoir and establish the fish-human consumption safety.

Materials and methods

The Study Area

The study was carried out on Osinmo Reservoir, Ejigbo, Southwestern, Nigeria. The reservoir was a man lake that was created by the impoundment of Ataro River in 2005 primarily to supply potable water. It also provides a number of ancillary benefits among which is the production of fish to the surrounding communities. The reservoir basin extends in length from Latitude 07°52.8' N to 07°53.2' N and in width from Longitude 004°21.2' E to 004°21.7' E (Figure 1). The reservoir has a surface area of about 0.78 km^2 with a mean depth of 3.2 meters (Komolafe, Arawomo, 2008).

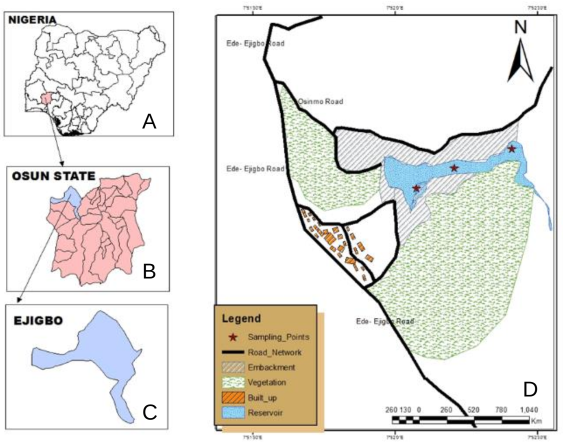

Figure 1. (A) Map of Nigeria showing Osun State, (B) Map of Osun State showing Ejigbo Local Government, (C) Map of Ejigbo Local Government (D) Osinmo reservoir. The map of the study area showing the sampling points

Sample Collection

Water sampling. Surface water samples were collected monthly for a period of 6 month (July–December 2015) from three points in the study area: the transition, open water and lacustrine area of the reservoir which was designated as Station A, B and C respectively (Figure 1). Two litres polyethene bottles washed with 5% nitric acid rinsed with distilled water and dried in an oven were used for the collection of water samples. At each sampling point, the bottles were rinsed three times with the reservoir water before the collection of the water sample. The water samples were then transported to the laboratory where they were filtered with Whatman filter paper No 42. The filtrate were then acidified with 2 ml concentrated HNO_3 per litre of filtered water to minimize precipitation and adsorption to container's wall. The water samples were then stored for digestion and heavy metal analysis.

Fish sampling. The Tilapia species used for this study were caught from Osinmo Reservoir with the help of local fishermen fishing on the Reservoir Area. Specimen of the fish species collected monthly for a period of 6 months between July to December 2015 and identified using identification keys prepared by (Adesulu, Sydenham, 2007) were those of *Oreochromis niloticus*, *Sarotherodon galilaeus* and *Tilapia zillii*. The fish specimens were transported to the Fish Culture Laboratory, Department of Zoology, Obafemi Awolowo University, Ile-Ife, Nigeria in an ice chest for further processing.

Fish Preparation. In the laboratory, the specimens were cleaned by rinsing with distilled water to remove debris, plankton and other external adherents. Morphometric measurements of the fish specimens were taken by metric ruler (in centimetres) for the total length and standard length while the weight of the fish specimens were measured using a Metler balance (Model P1210) and values recorded (to the nearest gram). The fishes were then descaled and the fillets of the fish specimen were dissected out, weighed and dried using Gallenkamp hotbox oven (Model DHG-9030A) at a temperature 60 °C for 24 hours. After drying, the samples were pulverised in clean ceramic mortar and pestle and kept in a well labelled universal specimen bottle prior to digestion for heavy metal analysis.

Heavy metal determination. Water sample and 2 g of pulverized fish sample were digested following the method of Ademoroti (1996). The levels of lead, zinc, chromium, iron and cadmium of digested samples were then read using an ALPHA 4 ChemTech Analytical (serial number 4200) Atomic Absorption Spectrophotometer (AAS) (Buck Model 205).

Statistical Data Analysis. The data obtained were subjected to one-way analysis of variance (ANOVA) and significant differences accepted at $P \leq 0.05$ (Zar, 2001). Where significant differences were recorded, the mean values were separated using post-hoc Tukey's (HSD) test. Descriptive statistics for all collected data were also obtained using SPSS software package Version 21.

Results

Heavy Metals Concentrations of the Water Samples Collected from Osinmo Reservoir

The mean concentrations of some heavy metals assayed in the water samples collected from Osinmo Reservoir is shown in Table 1. Analysis showed that zinc had significantly higher concentration out of the heavy metals assayed, followed by iron and chromium respectively (Table 1).

Table 1. Post-hoc multiple comparison (Duncan) of the selected heavy metal mean concentration (µg l^{-1}) in the water sample collected from the sampled stations

Heavy metals	Station A	Station B	Station C
Lead	16.50 ± 0.99^a	21.17 ± 1.51^b	13.83 ± 0.87^a
Chromium	20.17 ± 2.55^a	33.17 ± 2.77^b	20.00 ± 2.25^a
Zinc	254.17 ± 9.35^b	197.00 ± 15.77^a	204.67 ± 9.39^a
Iron	39.33 ± 2.63^a	66.83 ± 2.50^b	38.00 ± 3.93^a
Cadmium	19.17 ± 0.95^a	20.50 ± 2.22^a	24.33 ± 2.44^a

Row means with the same superscript are not significantly different ($P > 0.05$) from each other.

Zinc concentration in Station A was found to be significantly higher ($P < 0.05$) than that in Stations B and C. The highest mean concentration of Pb, Fe and Cr were recorded in water samples collected from Station B, while Station C had the lowest concentration of Cd, Pb, and Fe. In station A, Zn had the highest concentration followed by Fe, Cr, and Cd respectively. The concentrations of Pb, Cr, and Fe were significantly higher ($P < 0.05$) in Station B than at the other two sampled Stations (Table 1). Although there was variations in the Cd levels between the three Stations, there was no significant difference ($P < 0.05$) between the three Stations (Table 1). Irrespective of the month of study, Zn had the highest concentration out of all the elements assayed followed by Fe, Cr, Cd, and Pb in that order. In Station A, the order of heavy metal concentration was Zn > Fe > Cr > Cd > Pb, while in Station B, the order of heavy metal concentration was Zn > Fe > Cr > Pb > Cd. For Station C, the order of heavy metal concentration in the water samples analysed was Zn > Fe > Cd > Cr > Pb (Table 1).

Monthly mean variation of the heavy metal concentration in the water sample collected from Osinmo Reservoir

Lead (Pb). The monthly variation in the Pb^+ concentration in the water samples collected from the different sampled stations during the period of study (Figure 2) revealed that the level of the element in station A ranged between 13.00 µg l^{-1} (July 2015) and 20.00 µg l^{-1} (November 2015). In Station B, the concentration varied between 16.00 µg l^{-1} (July 2015) and 26.00 µg l^{-1} (December 2015). In Station C however, lead concentration monitored ranged between 11.00 µg l^{-1} (September 2015) and 16.00 µg l^{-1} (August and November 2015). Comparative monthly variation in the water mean Pb^+ concentration between the sampled stations as shown in Figure 2 revealed a bimodal pattern of Pb concentration with peaks in August and November (2015) in Stations B and C during the period of study. In Station A, however, a peak concentration which occurred in August 2015 was followed by a drop in the element concentration in September which was subsequently followed by a steady increase in concentration up to December 2015.

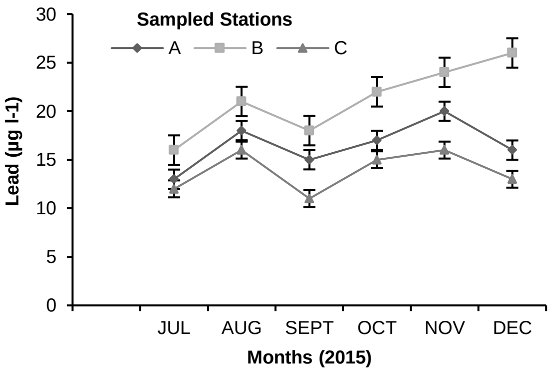

Figure 2. The monthly mean variation of lead ion concentration (± SEM) in the water samples collected from Osinmo reservoir

Cadmium (Cd). The mean cadmium concentration in water sample collected from Osinmo Reservoir during the study period ranged between 15.00 µg l^{-1} (Station B, July and September 2015,) and 31.00 µg l^{-1} (Station C, November 2015) (Figure 3). The highest level of Cd ion (31.00 µg l^{-1}) and lowest level of Cd ion (15.00 µg l^{-1}) was recorded in Station C in the month of September and October 2015, respectively (Figure 3). A bimodal pattern of Cd^+ concentration was recorded in all the sampled stations during the period of study. In Stations A and B, peak concentrations of Cd^+ were recorded during the months of August and October 2015, while the least concentration was recorded in September 2015. In Station C however, peak concentrations of the element occurred in September and

November 2015. The lowest Cd concentration was recorded in October 2015 (Figure 3).

Zinc (Zn). The zinc ion levels in the water samples from Osinmo Reservoir during the period of the study had the highest value of 288.00 µg l^{-1} (Station A, December 2015) and of 144.0 µg l^{-1} in Station B, (October 2015) (Figure 4). Analyses showed similar monthly pattern of variation in zinc concentration in Stations A and B (Figure 4). The variation in the two Stations showed a steady increase between July and September 2015, followed by a decline in the month of October 2015 subsequently with a steady increase till December 2015. In Station C, a peak concentration (224.00 µg l^{-1}) in August, 2015, was followed by a decline in concentration until the month of October, 2015, before an increase which culminated in the highest concentration (241.00 µg l^{-1}) of the element in the water samples during the month of December, 2015.

Chromium (Cr). The chromium ion concentrations in the water samples collected from Osinmo Reservoir during the period of study ranged between 9.00 µg l^{-1} in Station A (October 2015) and 48.00 µg l^{-1} in Station B, (December, 2015) (Figure 5). Monthly comparative analyses of the concentration of the elements between the sampled stations showed variations between the stations. In Station C, a steady increase occurred in the concentration of the element between July and December 2015. In Station A, however, the increase in concentration between July and August 2015 was followed by a decline in concentration up till October 2015, before a peak concentration (26.00 µg l^{-1}) was recorded in November 2015. In Station B, the slight increase in concentration of Cr^{2+} between July and August 2015 was followed by a declining concentration between August and September 2015. Subsequently, a steady increase in concentration occurred before a peak concentration (48.00 µg l^{-1}) was recorded in December 2015.

Iron (Fe). The mean iron concentration in water samples collected from Osinmo Reservoir during the period of study ranged between 25.00 µg l^{-1} (Station C, December 2015) and 73.00 µg l^{-1} (Station C, October 2015) (Figure 6). Comparative analyses of the Fe levels between the sampled Stations showed that in Station A, the peak levels of Fe occurred in the months of July and September, 2015, while the peak concentration of the element (73.00 µg l^{-1}) was recorded in October 2015 in Station B. However in Station C, the peak concentrations (51.00 µg l^{-1}) of Fe occurred during the month of July and October 2015. Analyses also revealed that the lowest concentration of Fe (25.00 µg l^{-1}) in all the sampled stations occurred during the month of December 2015 (Figure 6).

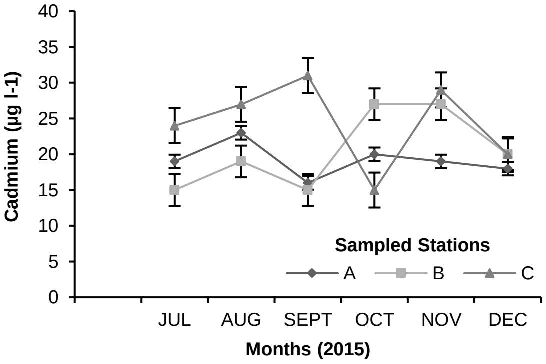

Figure 3. The monthly mean variation of cadmium ion concentration (± SEM) in the water samples collected from Osinmo reservoir

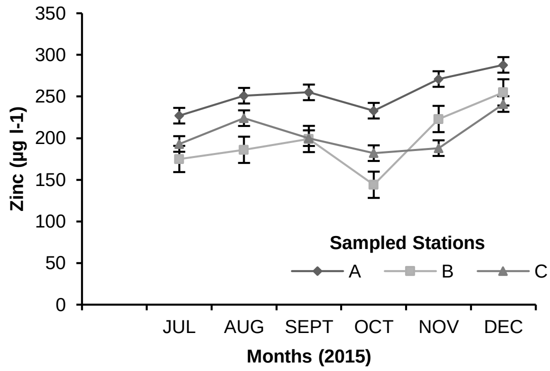

Figure 4. The monthly mean variation of zinc ion concentration (± SEM) in the water samples collected from Osinmo reservoir

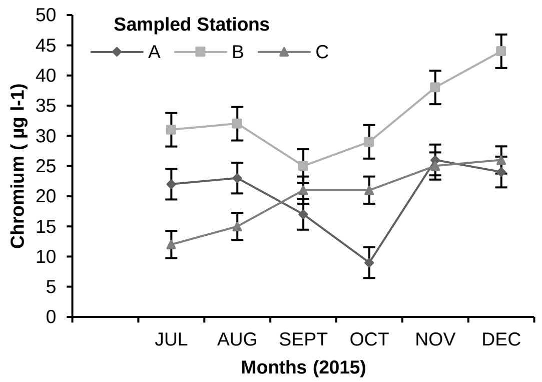

Figure 5. The monthly mean variation of chromium ion concentration (± SEM) in the water samples collected from Osinmo reservoir

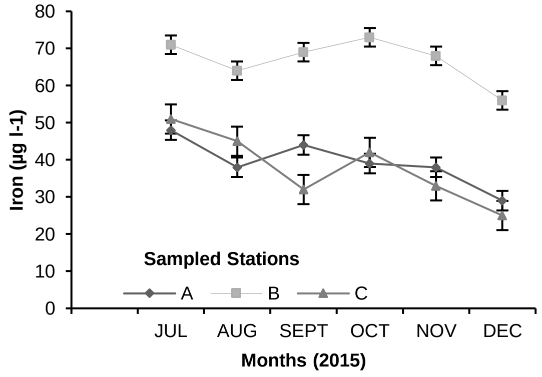

Figure 6. The monthly mean variation of iron ion concentration (± SEM) in the water samples collected from Osinmo reservoir

Table 2. Length-weight profile of the sampled cichlid fish specimens from Osinmo reservoir during the study period

Family	Fish species	Total number examined	Total length (cm)	Mean total length (cm)	Standard length (cm)	Mean standard length ± SEM (cm)	Weight (g)	Mean weight ± SEM (g)
	Oreochromis niloticus	30	16.00–28.00	19.85 ± 2.50	13.00–22.70	15.97 ± 2.04	94.99–421.08	166.90 ± 74.23
Cichlidae	*Tilapia zillii*	30	11.20–26.10	18.14 ± 3.71	9.20–21.50	13.88 ± 3.00	83.80–373.72	151.31 ± 72.97
	Sarotherodon galilaeus	30	10.30–25.50	16.23 ± 3.45	8.30–20.80	13.00 ± 2.78	68.29–355.13	128.05 ± 56.50

Fish Composition and Morphometrics

Thirty (30) specimens each of three fish species: *Oreochromis niloticus*, *Tilapia zillii* and *Sarotherodon galilaeus* belonging to the family *Cichlidae* were assessed for the fillet heavy metal concentration during the period of study. The total length, standard length and the weight of the specimens used for the study is shown in Table 2. In all cases, sub-adults and adult specimens of the three species were analysed for the heavy metal content.

Heavy Metals Concentrations in the Fillet of the Fish Specimens

The concentration of the heavy metals assayed in the fillet of the three cichlid species studied is shown in Table 3. Irrespective of the species, Zn has the highest concentration in the fish fillet while Cd concentration was the least. In the species, the order of concentration was Zn > Fe > Cr > Pb > Cd. Generally, the levels of the assayed heavy metals were higher in *O. niloticus* while the least concentration occurred in *S. galilaeus*. The only exception was Fe where the least concentration was recorded in the fillet of *T. zillii*. Comparative analyses between the cichlid species showed that the highest Zn concentration was recorded in the fillet of *O. niloticus*. Statistical analyses revealed that the concentration of Zn was significantly higher ($P < 0.05$) in the fillet of *O. niloticus* than in the fillet of *T. zillii* and *S. galilaeus* whose concentration were however found not to be significantly different ($P > 0.05$) from each other.

Table 3. Post-hoc multiple comparison of the assayed heavy metals concentration (µg g^{-1}) in the fillets of the fish specimens in mean and ± SEM

Heavy metals	*O. niloticus*	*T. zillii*	*S. galilaeus*
Lead	12.61 ± 0.81^b	10.00 ± 0.81^a	9.94 ± 0.81^a
Chromium	18.44 ± 1.63^a	18.22 ± 1.63^a	11.67 ± 1.63^b
Zinc	190.06 ± 4.20^b	175.22 ± 4.20^a	168.22 ± 4.20^a
Iron	36.67 ± 2.41^a	14.56 ± 2.41^b	31.00 ± 2.41^a
Cadmium	8.33 ± 0.46^b	6.83 ± 0.46^a	5.94 ± 0.46^a

Row means with the same superscript are not significantly different ($P < 0.05$) from each other.

The concentration of Fe which was also highest in the fillet of *O. niloticus* was not significantly different ($P > 0.05$) from those of *S. galilaeus* but was significantly higher ($P < 0.05$) than the concentration of the element in the fillet of *T. zillii*. The levels of Cr in the fillet of *O. niloticus* and *T. zillii* were not significantly different ($P > 0.05$) from each other but the levels were found to be significantly higher ($P < 0.05$) than in the fillet of *S. galilaeus.* Relatively trace amounts of Pb and Cd recorded in the fillet of *T. zillii* and *S. galilaeus* were found not to be significantly different ($P > 0.05$) from each other. However, the levels of the two elements in the two species were found to be significantly lower ($P < 0.05$) than in the fillet of *O. niloticus* (Table 3).

Monthly Variation in the Fillet Heavy Metals Concentration

Lead (Pb). The monthly variation in the Lead concentration of the fillet of the cichlid species examined during the period of study is shown in Figure 7. Irrespective of the month of sampling, similar pattern of concentration of the element was recorded in *O. niloticus* and *T. zillii* specimens caught. However, the level of Pb was higher in *O. niloticus* for each corresponding month. In *O. niloticus* and *T. zillii* fillet, higher concentrations of Pb (15.33 ± 5.03 and 13.00 ± 3.00 µg g^{-1} respectively) were recorded during the month of November 2015. In *S. galilaeus*, the lowest fillet Pb concentration (6.33 ± 3.21 µg g^{-1}) was recorded during the month of July (2015), while the highest level (12.33 ± 4.51 µg g^{-1}) was recorded in December (2015). In *S. galilaeus*, the level of Pb rose steadily in the fish fillet sample between July and September (2015) before a decline in October (2015) which was subsequently followed by a steady increase until December 2015 (Figure 7).

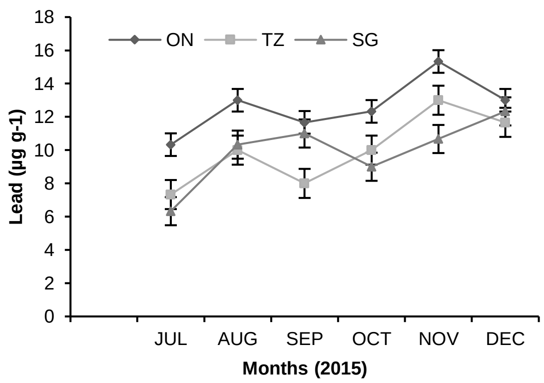

Figure 7. Monthly variations in the lead concentration (±SEM) of the fillet of the fish species caught during the study period (ON – *Oreochromis niloticus,* TZ – *Tilapia zillii,* SG – *Sarotherodon galilaeus*)

Chromium (Cr). The level of chromium in the fillet of the three tilapiine species studied is shown in Figure 8. Analyses showed that although the level of the element varied between the species, between July and August 2015, *T. zillii* had the highest elemental fillet level (28.67 ± 10.60 µg g^{-1}) while *S. galilaeus* had the least (6.33 ± 3.21 µg g^{-1}). In *O. niloticus* and *S. galilaeus*, the levels of Cr in the fillet of the fishes were relatively stable between August and October 2015, before a steady increase was recorded between October and December 2015. In *T. zillii*, however, the lowest Cr concentration (12.33 ± 3.51 μg g^{-1}) was recorded in September 2015 while the highest concentration (28.67 ± 10.60 μg g^{-1}) occurred in the fillet in November, 2015.

Zinc (Zn). The monthly variation in the level of zinc in the fillet of the studied cichlid species is shown in Figure 9.

Analyses showed the concentration of the element in the three species varied within a narrow amplitude. The highest zinc concentration (220.33 ± 20.50 and 205.00 ± 42.79 μg g^{-1}) in the fillet of *O. niloticus* and *S. galilaeus* specimens respectively was recorded in October 2015 while in *T. zillii*, the highest concentration (180.00 ± 5.00 μg g^{-1}) of the element occurred in September 2015. The least zinc concentration (121.33 ± 16.01 μg g^{-1}) was recorded in December 2015 in *S. galilaeus*, 169.67 ± 1.53 μg g^{-1} *T. zillii* in November 2015 and 180.00 ± 16.22 μg g^{-1} in *O. niloticus* in September 2015 (Figure 9).

Iron (Fe). The highest fillet Fe concentration (44.00 ± 18.03 μg g^{-1}) was recorded in *O. niloticus* irrespective of the month of sampling while the least concentration (13.00 ± 3.61 μg g^{-1}) was recorded in *T. zillii*. The concentration of Fe in the fillet of *T. zillii* between July and December 2015 was relatively stable (Figure 10). However, in *O. niloticus* the fish fillet Fe concentration was found to increase steadily between July and September 2015 when peak concentration (44.00 ± 18.03 μg g^{-1}) was recorded before declining until December 2015. In *S. galilaeus* specimens, however, the highest Fe concentration (39.67 ± 9.61 μg g^{-1}) in the species was recorded in September 2015, while the least concentration (24.00 ± 12.29 μg g^{-1}) of the element in the fillet of the fish was recorded in October 2015.

Cadmium (Cd). The monthly concentration of Cd in the fillet of the three studied cichlid species varied widely (Figure 11). In *T. zillii*, the concentration of Cd increased steadily between July and September 2015 when the peak concentration (11.33 ± 1.15 μg g^{-1}) was recorded before declining too (2.33 ± 1.53 μg g^{-1}) in December 2015, when the least concentration was recorded. The concentration of Cd in *S. galilaeus* which increased in the fish fillet between July and August 2015, declined until October 2015 when the least concentration (3.67 ± 1.15 μg g^{-1}) was recorded. Subsequently, the level of the element increased in the fish fillet until the maximum concentration (9.00 ± 2.65 μg g^{-1}) was recorded in December 2015. The peak Cd concentration was recorded in July 2015. However, the concentration of Cd in *O. niloticus* declined between August and September 2015. Subsequently, a high concentration (11.33 ± 1.15 μg g^{-1}) which was recorded in October 2015 was followed by a steep decline in concentration of the element until December 2015 when the least concentration (4.00 ± 1.00 μg g^{-1}) was recorded.

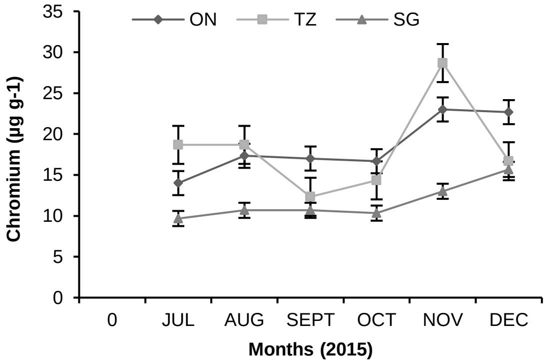

Figure 8. Monthly variations in the chromium concentration (± SEM) of the fillet of the fish species Caught during the study period (ON – *Oreochromis niloticus,* TZ – *Tilapia zillii,* SG – *Sarotherodon galilaeus*)

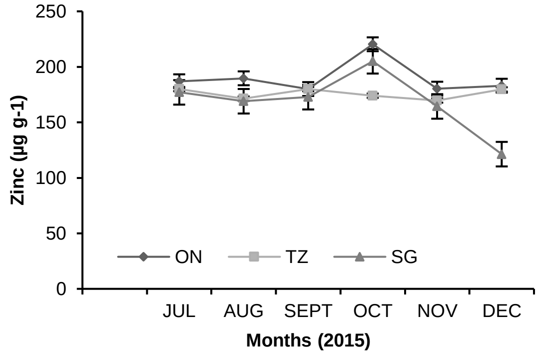

Figure 9. Monthly variations in the zinc concentration (± SEM) of the fillet of the fish species caught during the study period (ON – *Oreochromis niloticus,* TZ – *Tilapia zillii,* SG – *Sarotherodon galilaeus*)

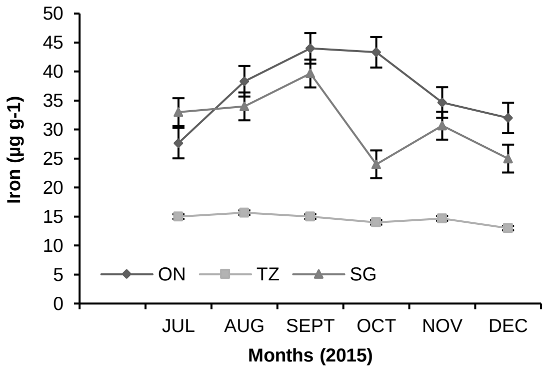

Figure 10. Monthly variations in the iron concentration (± SEM) of the fillet of the fish species caught during the study period (ON – *Oreochromis niloticus,* TZ – *Tilapia zillii,* SG – *Sarotherodon galilaeus*)

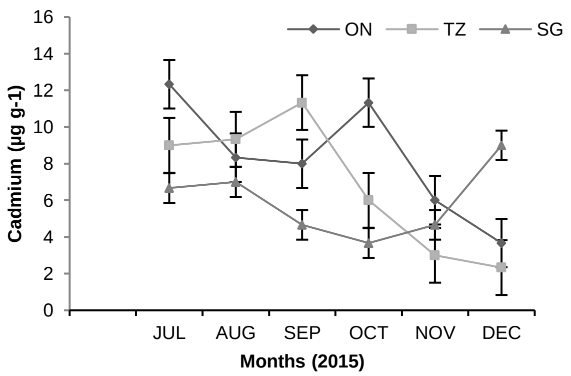

Figure 11. Monthly variations in the cadmium concentration (± SEM) of the fillet of the fish species caught during the study period (ON – *Oreochromis niloticus,* TZ – *Tilapia zillii,* SG – *Sarotherodon galilaeus*)

Relationship between Heavy Metal Concentration in Water and Fish Fillets

The relationship between concentration of lead in water and the fillet samples of the three studied species is shown in Figure 12. Analyses showed there was a very high positive correlation between the level of Pb in the water and fillet samples in *O. niloticus* ($r^2 = 0.8448$) and *T. zillii* ($r^2 = 0.8943$). However, there was a low correlation ($r^2 = 0.3255$) between the lead concentration in water and *S. galilaeus* fillet samples. The relationship between chromium concentration in water and fish fillet samples is shown in Figure 13. A very high positive correlation was recorded between water chromium levels and the element's concentration in the fillet of *O. niloticus* ($r^2 = 0.8822$) and *S. galilaeus* ($r^2 = 0.8849$). Comparatively, a low correlation ($r^2 = 0.2991$) was obtained between the water chromium level and the element fillet concentration in *T. zillii*. Analysis also showed a very low correlation between water Zn level and the fillet samples of *O. niloticus* ($r^2 = 0.4247$), *T. zillii* ($r^2 = 0.0645$) and *S. galilaeus* ($r^2 = 0.9395$) studied (Figure 14). Analysis showed a very low correlation between water Fe level and the fillet samples of *O. niloticus* ($r^2 = 0.0003$), *T. zillii* ($r^2 = 0.4478$) and *S. galilaeus* ($r^2 = 0.1255$) respectively (Figure 15). Also, very low correlation occurred between the water Cd level and the fillet elemental composition in the three species studied. Analysis showed the correlation coefficients recorded between Cd levels in water and fish fillet samples were: *O. niloticus* – $r^2 = 0.7520$; *T. zillii* – $r^2 = 0.0348$ and *S. galilaeus* – $r^2 = 0.1346$ (Figure 16).

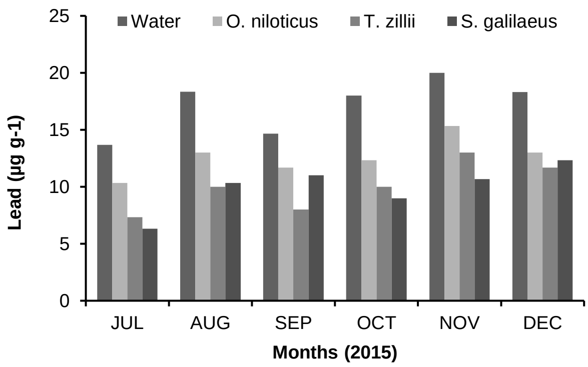

Figure 12. Relationship between Pb levels in the water and fillet samples of the fish specimens used

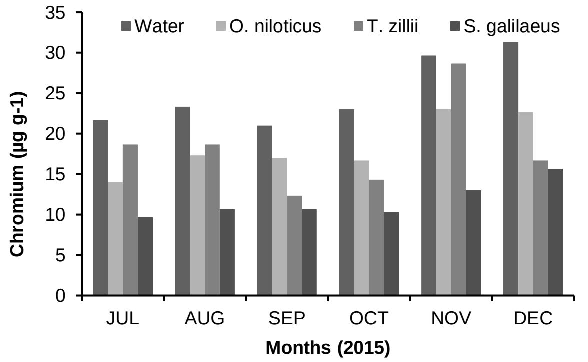

Figure 13. Relationship between Cr levels in the water and fillet samples of the fish specimens used

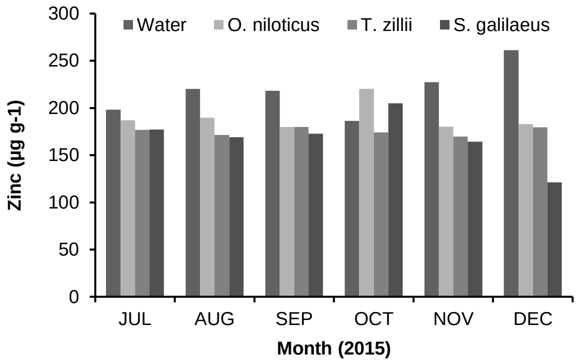

Figure 14. Relationship between Zn levels in the water and fillet samples of the fish specimens used

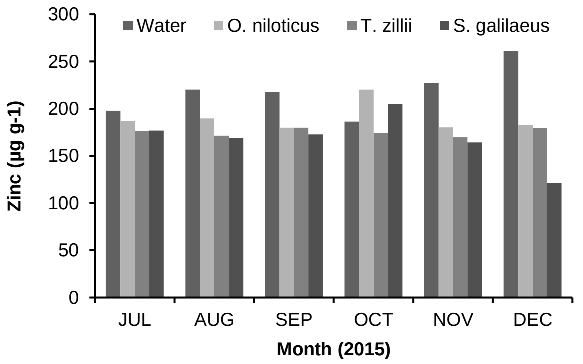

Figure 15. Relationship between Fe levels in the water and fillet samples of the fish specimens used

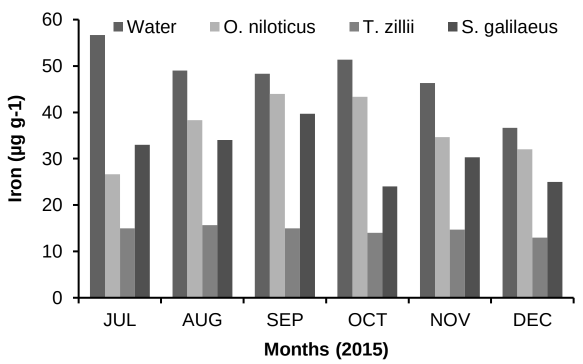

Figure 16. Relationship between Cd levels in the water and fillet samples of the fish specimens used

Discussion

Specific Variations in the Level of Selected Heavy Metals in Osinmo Water Samples

Lead concentration in Osinmo Reservoir during the period of study which recorded a monthly range value of 11.00–26.00 µg l^{-1} was similar to the result reported by Bolawa and Gbenle (2010) from Makoko River and around Carter Bridge, Lagos State, Nigeria. Lead concentrations which were significantly higher in station B could probably be attributed to increased anthropogenic activities observed during the period of study such as the proximity of the stations to the irrigation farming being practised in close proximity of the reservoir. The level of lead in the water samples collected from the reservoir, however, differed from the findings of Olatunji and Osinbanjo (2012) who reported a higher range of Lead values of (20.00–40.00 µg l^{-1}) in surface water of River Niger, Nigeria. Other studies of surface water from Ikpoba River, Nigeria by Oguzie, Izevbigie (2009) and Nairobi River, Kenya by Kithiia (2006), also reported higher mean Lead values of 35.00 µg l^{-1} and 100.00 µg l^{-1} respectively which were however higher than the level of Lead recorded in Osinmo Reservoir during the period of study. However, irrespective of the lower values of Lead recorded in the reservoir, the values were above the maximum acceptable limit of 10 µg l^{-1} for potable water (WHO, 2002; 2003). Goel (1997) reported that Lead concentration in natural water increases mainly through anthropogenic activities. Hence, the source of Lead into the Osinmo reservoir during the period of study will likely include depositions from soil erosion and run-offs from agricultural lands within the reservoir catchment areas (DWAF, 1996). Lower levels of lead recorded in water samples from station C was probably due to the minimal pollution activities close to the sampling point. The general decrease in Lead concentration during the period of study could also be due to dilution effect from water during the rainy period as well as absorption of the element by plants and sediments in the reservoir (Kithiia, 2006; Kar *et al*. 2008)

Cadmium concentration in Osinmo Reservoir during the period of the study which has a mean monthly range 15.00 and 31.00 µg l^{-1} was higher than the values reported by Ekpo *et al*. (2013) in Ikpoba River (1.00 and 2.00 µg l^{-1}) in Benin City, Nigeria. The higher levels of cadmium in the water samples across all the stations of Osinmo reservoir could be attributed to various anthropogenic activities especially ongoing agricultural activities in the area. According to Modaihsh *et al*. (2004), fertilizers such as phosphate fertilizers and other types averagely contain 13.4 µg g^{-1} of cadmium which tends to accumulate in the soil when fertilizers are applied annually on farmlands. Some of the accumulated Cd probably get leached out of the soil and end up in the reservoir. The cadmium concentration which increased from July to September in sampling station C could, therefore, be attributed to such run-offs from the surrounding soils. The cadmium level in Osinmo Reservoir during the period of study was however found to be above the acceptable limit of 5.0 µg l^{-1} recommended by EPA for portable water (ATSDR, 1999) and 3.0 µg l^{-1} recommended by WHO (1984) and FEPA (2007).

Presence of zinc in water bodies such as Osinmo reservoir could be associated with human activities such as the use of chemicals and zinc-based fertilizers by farmers (Egila, Nimyel, 2002). Zinc ion concentration in water samples collected from Osinmo Reservoir during the period of study (with a monthly range value of 144.00–288.00 µg l^{-1}) was within the recommended acceptable limit for potable water of 300 µg l^{-1} (WHO, 2008). Zinc concentrations that were higher than what was recorded in Osinmo reservoir during the present study have been reported in other water bodies. Kar *et al.* (2008) and Agatha (2010) reported a higher mean zinc level of 78250 µg l^{-1} from Forcado River, Nigeria. Olatunji, Osinbajo (2012) also obtained higher mean zinc values in the range of (1980.00–4030.00 µg l^{-1}) in River Niger, North Central Nigeria. Although zinc is considered to be relatively non-toxic, especially if taken orally, zinc deficiency has been reported to cause anaemia and retardation of growth and development (McCluggage, 1991). Excess amount of the element has also been reported to cause system dysfunctions that result in impairment of growth and reproduction (INECAR, 2000; Nolan, 2003).

Iron concentration in Osinmo Reservoir during the period of study which recorded a monthly value of between 25.00 µg l^{-1} to 73.00 µg l^{-1} was within the 300 µg l^{-1} recommended permissible limits in drinking water (FAO, 1996; FEPA, 2003). During the period of study, the Fe concentration recorded in water samples from all stations were, however, smaller than a higher mean value of 380 µg l^{-1} recorded in water upstream of Ikpoba River (Oguzie, Izevbigie, 2009). Analyses which showed the iron concentration to significantly higher in station B, however, could be related to the nearness of the sampled station to an irrigation farming scheme being practised in close proximity of the reservoir during the period of study. The decrease observed in iron concentration between the months of October to December 2015 across the sampling stations, on the other hand, might be due to the advent of the dry season which probably led to higher uptake of element by the biota. Although the level of iron in water samples collected from Osinmo Reservoir was below the maximum acceptable limit of 300 µg l^{-1} for iron in portable water (WHO, 1984; FEPA, 2003), the concentration of the element in water from the reservoir was found to be higher than the concentration of other elements investigated except zinc. The high level of the element in the water samples could be due to the high degree of solubility of the ferrous and ferric forms of iron in water. This could be explained by the fact that iron being the most abundant in the environment could conceivably be bio-accumulated more than the other metals (Oronsaye *et al*., 2010).

Chromium concentrations in water samples from Osinmo Reservoir during the sampling period which ranged between 9.00 µg l^{-1} to 48.00 µg l^{-1}, were below the recommended acceptable limits for potable water which is 50 µg l^{-1} (FEPA, 2007; WHO, 2008). The Cr levels recorded were comparatively lower when compared to the levels of the element reported in other water bodies. A higher mean chromium level of 49.00 µg l^{-1} was reported by Oyhakilome *et al*. (2012) in Owena multipurpose dam water, Nigeria. Olatunji and Osinbanjo (2012) also reported much higher mean chromium levels of 1190 to 3160 µg l^{-1} in River Niger, Nigeria. During the period of study, however, the increase in chromium level between the months of October and December 2015 could be associated with the onset of the dry season which probably led to incremental concentration as the volume of water reduces in the reservoir.

Heavy Metal Levels in the Fish Fillet

The mean Lead concentration in the fillet of the tilapiine species analysed which were 12.61 ± 0.81 µg g^{-1} in *O. niloticus*, 10.00 ± 0.81 µg g^{-1} in *T. zillii* and 9.94 ± 0.81 µg g^{-1} in *S. galilaeus* were found to be above the 2.0 µg g^{-1} recommended acceptable limit for lead in fish products (FEPA, 2003). Microhabitat utilization, feeding habits, age, sex and fish species probably determined the accumulation pattern of the heavy metal in the fishes (Kotze, 1997). The results obtained during the study however closely agreed with Okoye (1999) who reported mean values of 9.00 µg g^{-1} for lead in fishes collected from the Lagos Lagoon. The values obtained during the period of study were higher when compared to the reported values of (0.395–0.62 µg g^{-1}) by Doherty *et al*. (2010) in fishes collected from Lagos Lagoon. Bolawa and Gbenle (2010) also reported higher mean values of the element which ranged between 31.00 and 65.00 µg g^{-1} in fishes collected from Makoko River and Carter River in Lagos, Nigeria. Farombi *et al*. (2007) on the other hand, reported the Lead values of between 0.73 to 4.12 µg g^{-1} in *C. gariepinus* collected from Ogun River while Obasohan *et al*. (2006) reported a mean range values of 0.10 to 0.83 µg g^{-1} in some fishes from Ogba River, Nigeria.

Zinc which is an essential microelement required for numerous aspects of cellular metabolism occurs naturally in organisms and in the earth crust. The mean zinc concentration in the fillet of the fish species assayed were 190.06 ± 4.20 µg g^{-1} in *O. niloticus*, 175.22 ± 4.20 µg g^{-1} in *T. zillii* and 168.22 ± 4.20 µg g^{-1} in *S. galilaeus*. These values were found to be below the recommended acceptable limit of 2000–13 000 µg g^{-1} (WHO, 2003). The order of zinc concentration in the fish species caught from Osinmo reservoir were *S. galilaeus* < *T. zillii* < *O. niloticus*. Fish has been reported to accumulate zinc from both the surrounding water and from their diet (Eisler, 1993). Although, zinc is an essential element, at high concentration it has been reported to be toxic to fish, causing mortality, growth retardation and reproductive impairment (Sorenson, 1991). Oluseye *et al*. (2012) reported a much lower mean values of zinc concentration (0.690 µg g^{-1}) in fish species caught in Dandaru Reservoir, Ibadan, when compared with those of the studied tilapiine species caught in Osinmo reservoir. Akan *et al*. (2012) also recorded a much lower mean zinc value in the range of 0.15 to 0.25 µg g^{-1} in fishes caught from River Benue in Adamawa, Nigeria when compared with the zinc levels in the studied cichlids during the period of study. However, a higher mean value of 158.30 µg g^{-1} was reported for fishes caught in Jakara River, Kano State (Ibrahim, Said, 2010). The concentration of zinc in *O. niloticus* which was significantly higher than in the other two cichlid fish species could probably be diet related. *O. niloticus* has been considered as a benthic omnivore feeds on benthic crustaceans and sessile molluscs and occasionally preying on other smaller fishes (Orban *et al*., 2008). The higher value of the element in the fish probably confirmed Ney and Van Hassel (1983) earlier observation that lead and zinc concentration were expected to be higher in benthic fishes.

The mean cadmium concentration in the fillet of the tilapiine fish species from Osinmo reservoir assayed which respectively were: 8.33 ± 0.46 µg g^{-1} in *O. niloticus*, 6.83 ± 0.46 µg g^{-1} in *T. zillii*, 5.94 ± 0.46 µg g^{-1} in *S. galilaeus* were above the recommended acceptable limit of 1.0 µg g^{-1} (Egila, Nimyel, 2002). Although cadmium occurs naturally in the environment, the accumulation of cadmium in fish samples could be as a result of agricultural activities such as land preparation, application of agrochemicals and other activities. According to Rashed (2001), Pb and Cd concentrations are known to increase in fish tissues collected in freshwater ecosystem impacted by agricultural activities. Thus, there is the possibility of these heavy metals emanating from the chemical fertilizers, weedicides and all forms of pesticides being utilized in the cultivation of farms within the reservoir catchment area. Comparatively, Ibok *et al*. (1989) reported lower cadmium values of 0.24 and 0.45 µg g^{-1} in *P. Obscrura* and *Hemichromis fasciatus* caught in some streams in Ikot Ekpene area of Nigeria. Abdulrahman and Tsafe (2004) on the other hand reported a slightly higher value of cadmium in fishes caught from Sokoto Rima River which was attributed to agrochemical usages. Kidwell *et al*. (1995) however observed that predatory fish species accumulated more mercury while the benthivores contained higher concentrations of cadmium and zinc. Bolawa and Gbenle (2010) reported a comparatively higher cadmium value of 23 to 90 µg g^{-1} from Makoko River and Carter Bridge River in Lagos, Nigeria, when compared with those of tilapiine species from Osinmo reservoir. However, Ekpo *et al*. (2013) reported a much lower mean cadmium value in the range of 1.00 to 2.00 µg g^{-1} in fishes caught from Ikpoba River, Benin City, Nigeria. The higher concentration of cadmium in fish fillets during this study especially in *O. niloticus* could be due to their ability to tolerate very high levels (14.8 mg l^{-1}) of waterborne cadmium (Garcia-Santos *et al*., 2006).

The mean iron concentration in the fillet of the three tilapiine fish species analysed during the period of study which were 36.67 ± 2.41 µg g^{-1} in *O. niloticus*, 14.56 ± 2.41 µg g^{-1} in *T. zillii* and 31.00 ± 2.41 µg g^{-1} in *S. galilaeus* respectively were below the 300 µg g^{-1} acceptable recommended limits for iron in food (FAO, 1996; FEPA, 2003). The levels of iron in the fillet of the three tilapiine species during the period of study agreed with values of between 51.32–107.54 µg g^{-1} reported by Obasohan (1997) in the fillet of fish species collected from Ogba River, Benin City, Nigeria. Although agricultural activities could have contributed to the concentration of iron in the fish fillet samples, the primary source of iron in the fillet could likely be the naturally occurring iron present in the soil (NRC, 1989). Also, the haemoglobin component of the blood which is iron-based is also probably a major contributor of the iron level in the fillet of the fishes (Camara *et al*., 2005).

Chromium is an essential trace element in human but in excesses, it could have lethal effect on fish and wildlife (Robertson *et al*., 1992). The mean chromium concentration in the fillet of the fish species analysed were 18.44 ± 1.63 µg g^{-1} in *O. niloticus*, 18.22 ± 1.63 µg g^{-1} in *T. zillii*, and 11.67 ± 1.63 µg g^{-1} in *S. galilaeus*. The values of the element recorded in the fillet of the various tilapiine species during the period of study were found to be within the acceptable limit of 50 µg g^{-1} recommended for fish and fish products (WHO, 1984; FEPA, 2007). However, the consumption of fish from the reservoir should, however, be done with caution as cumulative effects of the element might constitute health hazards to aquatic life as well as man who feeds on the fishes (Oronsaye, 2010). Ugwu *et al*. (2012) had earlier reported a much higher chromium level (56.00 µg g^{-1}) in muscles of *O. niloticus* obtained from River Usuma, Nigeria. Agricultural activities such as application of agro-chemical like fertilizers and pesticides utilized within the reservoir catchment basin could be fingered as the source of chromium in the fish sample.

Conclusion

The study concluded that the elevated levels of lead and cadmium in the water and the fish fillet samples assayed indicated that the two elements negatively impacted the fish fillet quality, thereby raising human health consumption safety issues.

Conflict of interest

None of the authors has any potential conflict of interest related to this manuscript.

Author contributions

V.F. Olaleye designed the experimental frame work and critically reviewed the manuscript for final submission

A.O. Olofinko: Data collection, analyses and interpretation were done by the author

H.A. Adewole: This author assisted in the analysis and interpretation of some of the data, and also draft the manuscript

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