

EVALUATION OF SOME HEAVY METALS (LEAD, NICKEL, CADMIUM, AND CHROMIUM) IN FRESH AND SMOKED *Clarias gariepinus* AND *Heterotis niloticus* IN HADEJIA FISH MARKET, JIGAWA STATE, NIGERIA

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ABSTRACT

The evaluation of heavy metal concentrations, specifically lead (Pb), cadmium (Cd), nickel (Ni), and chromium (Cr), in fresh and smoked fish species *Clarias gariepinus* and *Heterotis niloticus* was conducted to assess potential health risks associated with fish consumption. A total of 36 fish samples, consisting of 18 each of the two species, were procured from Hadejia fish market and subsequently brought to the laboratory for analysis. The concentrations of the heavy metals were determined using standard analytical techniques following digestion of the samples. Results indicated that smoked *Clarias gariepinus* exhibited Pb levels of 0.19 mg/kg, Cd at 0.18 mg/kg, Ni at 0.16 mg/kg, and Cr at 0.18 mg/kg. In comparison, the smoked *Heterotis niloticus* displayed lower concentrations with Pb at 0.13 mg/kg, Cd at 0.12 mg/kg, Ni at 0.14 mg/kg, and Cr at 0.16 mg/kg. Notably, fresh *Clarias gariepinus* samples showed the highest Pb concentration in the gills (0.06 mg/kg), while fresh *Heterotis niloticus* exhibited the highest Ni levels in the gills at 0.16 mg/kg. These results highlight the presence of heavy metals in both fresh and smoked fish, underscoring the importance of regular monitoring for food safety and public health, particularly in regions where fish consumption is prevalent. The findings call for further investigation into the sources of these contaminants in aquatic environments.

Keywords: Toxicity, Fish preservation, Freshwater fish, Fish market and Contaminants.

INTRODUCTION

Naturally occurring elements with high atomic weights and densities at least five times higher than water are known as heavy metals. According to Islam *et al.* (2020) and Biswas *et al.*, (2021), heavy metal contamination poses a serious threat to the aquatic environment and aquatic life, especially fish. Heavy metals in aquatic habitats are primarily caused by a variety of anthropogenic activities, including industrial and agricultural discharges, household waste, landfill erosion, and various natural processes (Ezemonye *et al.*, 2019, Mahmuda *et al.*, 2020). These activities have detrimental effects on ecosystems and human health (Choudhury *et al.*, 2022, Hassan *et al.*, 2022). Due to their bioaccumulation and bio-magnifications in aquatic animals, such as fish, non-biodegradable heavy metals are extremely toxic. Even trace amounts of these metals can cause significant harm to aquatic animals and consumers (Abdel-Baki *et al.*, 2011, Nargis *et al.*, 2021, Taslima *et al.*, 2022). The eating contaminated fish, heavy metals build up in important human organs like the liver, kidney, and bones, causing a number of neurotoxic and carcinogenic effects (Duruibe *et al.*, 2007). While copper (Cu) was linked to a number of liver and kidney complications (Mustafa *et al.*, 2009), metals like chromium (Cr) and nickel (Ni) caused a number of pulmonary abnormalities (Forti *et al.*, 2011). Additionally, chromium (Cr) poisoning damaged human DNA and led to the development of cancer cells (Mishra *et al.*, 2019).

Presence of heavy metals (Lead, Nickle, Cadmium and chromium) in fish contaminates the food chain and has an impact on both terrestrial and aquatic ecosystems, posing

serious health hazards to both humans and animals. Furthermore, incorrect handling and storage of fish might result in bacterial growth, which can deteriorate them and pose health risks. By examining the amount of bacteria and heavy metal contamination in fish sold at the Hadejia market, this study hopes to provide light on the possible dangers and repercussions for customers, the environment, and the economy.

The traditional smoking process and handling practices used by vendors at Hadejia Fish Market may compromise the safety and quality of smoked fish, potentially leading to contamination with pathogenic microorganisms and heavy metals, which can cause serious health problems, including gastrointestinal infections, heavy metal poisoning, and long-term health effects, such as cancer and neurological damage. A thorough assessment of the safety and quality of smoked and fresh fish sold at Hadejia Fish Market in Jigawa State, Nigeria, is necessary due to the high consumption of smoked fish and the potential for bacterial contamination and heavy metal pollution during the traditional smoking process.

MATERIAL AND METHODS

Samples collection, handling and preservation

Four samples with three replicates each of two different fresh and smoked-dried fish species (*Clarias gariepinus* and *Heterotis niloticus*) weighing between 500g and 1.5kg were purchased from the Hadejia Fish Market in Hadejia Local Government Area, Jigawa State, Nigeria, making a total of 36 samples. Samples were collected in newly purchased, sterile polythene bags, properly labelled, and transported to the Department of Fisheries and Aquaculture Laboratory in insulated containers with

ice blocks to maintain a temperature of approximately 4°C for the heavy metal analysis. Upon the arrival at the laboratory, the fish samples underwent a series of preparatory procedures, including weighing, measuring, and meticulous preparation for subsequent analysis. The fish samples were weighed preserved using 2-3 drops of nitric acid and refrigerated at 4°C before analysis.

Determination of heavy metals

The total concentrations of Lead (Pb), Cadmium (Cd), Chromium (Cr), and Nickel (Ni) in each sample were measured following standard laboratory procedures (APHA, 2012). The concentrations were read using an Atomic Absorption Spectrophotometer (Agilent Technologies, 200 Series AA 240FS AA Model) (Agilent Technologies, 2014). The individual metal concentrations were expressed on a dry weight basis (mg/kg), and the data generated were analysed statistically

Preparation of Materials for analysis

The working tables were washed with ethanol to disinfect them. All the wares were washed and air-dried, after which they were sterilized in a hot-air oven at 160°C for 1 hour. Culture media such as nutrient agar were prepared according to the manufacturer's specifications, and the distilled water used for serial dilution was sterilized in an autoclave at 121°C for 15 minutes before use (APHA, 2012)

The agar plates were organised by first sterilizing the Petri dishes. This was done by placing the Petri dishes in Petri dish cans and sterilizing them in a hot-air oven at 160°C for 1 hour. The sterilized plates were then left in the Petri dish cans until required. The wire loop was sterilized by flaming it until red-hot using a spirit lamp. The agar was prepared by dissolving 0.6g of agar in 100ml of distilled water, and then sterilizing the mixture using a microwave at 121°C for 15 minutes, after which it was allowed to cool to 45°C.

Digestion Procedure

For smoked-dried fish samples, each sample was homogenized separately in a clean mortar and weighed accurately into a porcelain crucible. Before ashing, 1 ml of concentrated HNO₃ was added to the samples and allowed to pre-digest overnight to minimize loss of volatile metals. The samples were then charred on an electric hot plate and subsequently ashed in a muffle furnace at 550°C for 4 hours. The resulting white ash was dissolved in 5 ml of 1:1 HCl, and the volume was made up to 50 ml in a standard flask (Okoye, 1991).

A blank solution for the biotic samples was prepared by diluting 1 ml concentrated HNO₃ with 5 ml of 1:1 HCl and making up the solution to 50 ml with distilled water. For fresh fish samples, analysis focused on the liver and gills, following established recommendations that these organs serve as reliable indicators of internal and external contamination, respectively (Yousuf *et al.*, 2000; Adams *et al.*, 2010). Liver (Internal Contamination): The liver is a central organ responsible for detoxification, metabolism, and nutrient storage (Hinton, 1993). Because of its detoxification role, it tends to accumulate heavy metals and other toxic substances entering the fish through ingestion or absorption (Banni *et al.*, 2011). Gills (External Contamination): The gills are responsible for respiration and are in constant contact with the surrounding water (Evans, 1987). They readily accumulate contaminants, making them effective proxies for assessing external exposure (Karlsson *et al.*, 2010). Using both liver and gills provides a comprehensive understanding of contamination patterns within the fish and helps evaluate relationships between bioaccumulation pathways and metal uptake mechanisms (Van der Oost *et al.*, 2003).

Heavy Metals Analysis Procedure

Metal concentrations in all digested samples were determined using a Perkin-Elmer Model 306 Atomic Absorption Spectrophotometer (AAS). Each metal concentration was measured against appropriate blanks and standard solutions for accuracy. Final concentrations were expressed on a dry weight basis (mg/kg).

Data Analysis

The Statistical Package for Social Scientists (SPSS) version 16.0 was used for data analysis. Data obtained for bacterial load and heavy metal accumulation were subjected to inferential statistics (mean) and visual analysis (bar charts). Heavy metals were analysed using Analysis of Variance (ANOVA) and means were separated by Fisher's LSD at $P \leq 0.05$ significant level.

RESULTS

The results for the bacterial count of fresh and smoked *C. gariepinus* and *H. niloticus* are indicated in Table 1. Both fresh and smoked samples showed significantly different ($p \leq 0.05$) bacterial counts, with fresh samples exhibiting higher values than smoked ones in both species. For *C. gariepinus*, the fresh sample recorded 1.72×10^4 CFU/g, while the smoked sample recorded 2.31×10^4 CFU/g.

Table 1: Bacterial Count of Fresh and Smoked Fish Species

Species	Parameters	Fresh	Smoked	Recommended limits	
				(fresh)	(smoked)
<i>C. gariepinus</i>	TBC	$1.72 \times 10^4 \pm 0.20^a$	$1.27 \times 10^4 \pm 0.16^b$	(50 $\times 10^5$) CFU/g	(5.0 $\times 10^5$) CFU/g
<i>H. niloticus</i>	TBC	$2.31 \times 10^4 \pm 0.39^a$	$1.78 \times 10^4 \pm 0.44^b$	(50 $\times 10^5$) CFU/g	(5.0 $\times 10^5$) CFU/g

Mean values are significantly different ($p \leq 0.05$)

Key-TBC= total bacteria count



The concentrations of selected heavy metals (Lead, Cadmium, Nickel, and Chromium) were determined in the gills, liver, and smoked tissues of *Clarias gariepinus* and *Heterotis niloticus* and presented in table 2. The results were compared with the permissible limits established by FAO, WHO, and FEPA. Lead (Pb) concentrations ranged from 0.06 ± 0.00 mg/kg in the liver to 0.19 ± 0.02 mg/kg in the smoked tissue. Although the smoked sample recorded the highest concentration, all values were below the permissible limit of 0.30 mg/kg, indicating that lead contamination in the fish does not pose an immediate health risk. Cadmium (Cd) concentrations were low in all tissues, varying from 0.01 ± 0.00 mg/kg in both gills and liver to 0.03 ± 0.02 mg/kg in the smoked tissue. These values were below the recommended limit of 0.05 mg/kg, suggesting minimal cadmium accumulation. Nickel (Ni) concentrations ranged from 0.16 ± 0.05 mg/kg in the smoked tissue to 0.27 ± 0.41 mg/kg in the gills. No significant differences ($P > 0.05$) were observed among the tissues. All concentrations were below the FEPA permissible limit of 0.50 mg/kg. Chromium (Cr) concentrations varied between 0.05 ± 0.02 mg/kg in the liver and 0.18 ± 0.03 mg/kg in the smoked tissue. The concentrations in the gills and smoked tissues exceeded the permissible limit of 0.10 mg/kg, indicating possible chromium contamination and accumulation in these tissues.

Overall, the order of heavy metal accumulation in *Clarias gariepinus* was Chromium > Nickel > Lead > Cadmium. Except for chromium, all metals were within the acceptable limits recommended by regulatory agencies.

For *H. niloticus*, Lead (Pb) concentrations ranged from 0.08 ± 0.04 mg/kg in the liver to 0.13 ± 0.00 mg/kg in the smoked tissue (Table 3). These concentrations were below the permissible limit of 0.30 mg/kg, indicating acceptable

levels of lead contamination. Cadmium (Cd) concentrations were 0.01 ± 0.00 mg/kg in the liver, 0.02 ± 0.00 mg/kg in the gills, and 0.12 ± 0.00 mg/kg in the smoked tissue. The smoked tissue recorded a value above the permissible limit of 0.05 mg/kg, suggesting potential health concerns associated with the consumption of smoked *Heterotis niloticus*. Nickel (Ni) concentrations ranged from 0.06 ± 0.04 mg/kg in the liver to 0.16 ± 0.02 mg/kg in the gills. All values were below the permissible limit of 0.50 mg/kg and therefore considered safe for human consumption. Chromium (Cr) concentrations varied from 0.08 ± 0.04 mg/kg in the liver to 0.16 ± 0.66 mg/kg in the smoked tissue. The concentrations in the gills and smoked tissues exceeded the permissible limit of 0.10 mg/kg, indicating chromium accumulation in these organs. The overall trend of heavy metal accumulation in *Heterotis niloticus* was Chromium > Nickel > Lead > Cadmium. Most metals were within acceptable limits except cadmium in the smoked tissue and chromium in the gills and smoked tissue.

Comparative Assessment

A comparison between the two fish species showed that *Heterotis niloticus* accumulated higher concentrations of lead and cadmium, particularly in the smoked tissue, while *Clarias gariepinus* recorded higher nickel concentrations. Chromium concentrations exceeded permissible limits in both species, especially in smoked samples, suggesting environmental contamination and possible concentration during processing. The findings indicate that the studied fish species are generally safe for human consumption with respect to lead, cadmium, and nickel. However, the elevated chromium levels in both species and cadmium concentration in smoked *Heterotis niloticus* warrant continuous monitoring of the aquatic environment and fish processing methods to protect public health.

Table 2: Heavy Metal Concentration in *Clarias gariepinus*

Parameter	Gills (mg/kg)	Liver (mg/kg)	Smoked (mg/kg)	Standard Limit	Reference
Lead	0.061 ± 0.05^b	0.06^b	0.19 ± 0.02^a	0.30 mg/kg	FAO/FEPA (2012)
Cadmium	0.01 ± 0.00^b	0.01 ± 0.02^b	0.03 ± 0.02^a	0.05 mg/kg	WHO/FEPA (2012)
Nickel	0.27 ± 0.41^a	0.20 ± 0.02^a	0.16 ± 0.05^a	0.50 mg/kg	FEPA (2012)
Chromium	0.13 ± 0.08^a	0.05 ± 0.02^b	0.18 ± 0.03^a	0.10 mg/kg	WHO/FEPA (2012)

Table 3: Heavy Metal Concentration in *Heterotis niloticus*

Parameter	Gills (mg/kg)	Liver (mg/kg)	Smoked (mg/kg)	Standard Limit	Reference
Lead	0.11 ± 0.05^b	0.08 ± 0.04^{bc}	0.13 ± 0.0^a	0.30 mg/kg	FAO/FEPA (2012)
Cadmium	0.02 ± 0.00^a	0.01 ± 0.00^b	0.12 ± 0.00^a	0.05 mg/kg	WHO/FEPA (2012)
Nickel	0.16 ± 0.02^a	0.06 ± 0.04^b	0.14 ± 0.03^a	0.50 mg/kg	FEPA (2012)
Chromium	0.14 ± 0.08^a	0.08 ± 0.04^b	0.16 ± 0.66^a	0.10 mg/kg	WHO/FEPA (2012)

DISCUSSION

The concentrations of Pb, Cd, Ni, and Cr in the fresh fish samples of gills, liver, and smoked samples of *C. gariepinus* varied, reflecting tissue specific uptake and the impact of processing. Fish bioaccumulate metals via gill absorption, dietary intake, and dermal contact, while smoking can further modify metal content through concentration or deposition (Asuquo *et al.*, 2014; Wangboje and Okhwarobo, 2024).

Lead levels peaked in smoked fish, followed by gills and liver. Lead accumulation during smoking may arise from moisture loss (concentration effect) and contamination from fuel wood or metallic racks (Bappayo and Sagagi, 2025). Comparable increases were reported by Edem *et al.* and Olayinka Olagunju. Although measured values were below international guideline limits, Pb is non-essential and cumulative; chronic exposure may cause neurotoxicity and renal impairment (WHO, 2021). Continuous consumption of smoked fish could thus pose long-term risk. Cadmium concentrations were low across tissues, below recommended limits. Low values suggest minimal environmental input; however, Cd accumulation was highest in smoked samples, confirming dehydration related concentration. As Cd is carcinogenic and linked to renal dysfunction, ongoing surveillance is necessary, particularly since other studies have reported Cd accidents in smoked fish from industrial zones.

Ni was highest in the fresh fish samples of gills, followed by the fresh fish samples of liver and smoked tissues. Gills, being the main site of ion exchange, rapidly accumulate metals from water (Authman *et al.*, 2015). The slightly lower Ni in smoked fish could result from volatilization of Ni compounds during heating (Kusemiju *et al.*, 2001). Although within permissible limits, long-term intake may provoke allergic dermatitis or hepatotoxicity (EFSA, 2020).

Chromium concentration peaked in smoked fish, likely due to contamination from wood smoke or metallic implements. Gills also showed notable accumulation, reflecting direct contact with water. The measured values fall within accepted limits, corroborating findings by Wangboje and Okhwarobo (2024). Nonetheless, prolonged exposure to hexavalent chromium is carcinogenic, hence caution is required.

Heavy metal patterns in *H. niloticus* mirrored those of *C. gariepinus*, with smoked samples generally exhibiting higher levels than the fresh fish samples of gills or liver tissues. Such consistency underscores the influence of smoking on metal enrichment. Lead levels were highest in smoked fish, followed by the fresh fish samples of gills and liver. The increments may stem from airborne deposition of Pb laden ash particles during smoking (Bappayo and Sagagi, 2025). Similar increases were recorded in smoked mullet by Edem *et al.* Although within guideline limits, cumulative exposure remains a concern, necessitating cleaner smoking environments.

Cadmium rose sharply in smoked samples compared with

the fresh fish samples of gills and liver. Values slightly exceeded recommended limits in some smoked products, suggesting possible contamination from fuel wood ash. Other authors similarly reported Cd accumulation in fish from effluent-impacted waters. Thus, while baseline aquatic contamination may be low, processing-related contamination can elevate Cd in final products.

The observed increase in cadmium concentrations in smoked fish samples compared to fresh gill and liver tissues suggests that post-harvest processing contributes significantly to metal contamination. This finding aligns with earlier studies indicating that smoking processes, particularly those involving contaminated fuel wood or ash, can introduce heavy metals into fish products (Adefemi *et al.*, 2008; Adeyeye, 1996). Although baseline cadmium levels in aquatic environments may remain low, the processing stage can elevate concentrations beyond permissible limits. This supports the assertion by FAO/WHO (2011) that food processing methods may act as secondary contamination sources, thereby increasing toxic metal exposure to consumers.

Ni was highest in the fresh fish samples of gills, followed by smoked samples and liver, consistent with prior studies noting that gills reflect ambient water conditions while smoked samples may show secondary accumulation. Ni values remained within commonly accepted safety thresholds, indicating low immediate health risk.

This pattern reflects the role of gills as primary interfaces with the aquatic environment, where dissolved metals are readily absorbed (Yilmaz, 2009). The relatively lower concentrations in smoked samples suggest limited additional contamination from processing compared to cadmium. Importantly, nickel levels remained within acceptable safety thresholds, indicating minimal immediate health risk. However, chronic exposure through bioaccumulation cannot be entirely ruled out (WHO, 2007).

Chromium levels were highest in smoked fish, followed by the fresh fish samples of gills and liver. Although concentrations were within acceptable limits, the potential for formation of more toxic chromium species during combustion necessitates further study. Chromium levels were highest in smoked fish samples, consistent with findings that link elevated chromium concentrations to contamination from smoking equipment and airborne particulates (Akoto *et al.*, 2014). The presence of chromium in smoked products raises concerns, particularly due to the potential formation of toxic hexavalent chromium (Cr^{6+}) during combustion processes (ATSDR, 2012). Although measured concentrations fell within permissible limits, the potential transformation into more toxic forms underscores the need for cautious interpretation and further investigation. The study revealed that *Heterotis niloticus* exhibited consistently higher concentrations of heavy metals compared to *Clarias gariepinus*. This difference can be attributed to ecological and feeding behaviors. *H. niloticus*, being a benthopelagic detritivore, feeds near



sediment surfaces where heavy metals tend to accumulate, thereby increasing its exposure (Bawuro *et al.*, 2018). In contrast, *C. gariepinus* is more pelagic and omnivorous, resulting in relatively lower sediment-associated contamination. This observation is consistent with previous studies that emphasize the influence of ecological niche and trophic level on metal bioaccumulation in aquatic organisms (Burger *et al.*, 2002). The findings highlight species-specific variations in contaminant uptake, which are critical in environmental monitoring and food safety assessments. Furthermore, the study underscores a significant trade-off between microbial safety and chemical contamination. While smoking effectively reduces microbial load and enhances shelf life, it simultaneously introduces or concentrates heavy metals. This dual effect presents a challenge for food safety management, necessitating improved processing techniques.

From a public health perspective, the coexistence of microbial and chemical hazards in fish products necessitates an integrated risk assessment approach. Even when heavy metal concentrations fall within recommended limits, continuous consumption may result in bioaccumulation and long-term health effects such as nephrotoxicity, carcinogenicity, and neurological disorders (Jarup, 2003).

In Nigeria, smoked fish constitutes a major dietary component, particularly in rural and riverine communities, thereby increasing the risk of chronic exposure (Orisakwe *et al.*, 2012). Additionally, improper cooking or handling can expose consumers to pathogenic microorganisms, leading to acute health issues. Environmentally, the presence of heavy metals in fish reflects broader ecosystem contamination arising from anthropogenic activities such as agricultural runoff, domestic waste discharge, and atmospheric deposition (Alloway, 2013). These findings highlight the need for sustainable environmental management practices to reduce pollutant input into aquatic systems.

Conclusion

The study concluded that fish sold in the Hadejia fish market, particularly *H. niloticus* and *C. gariepinus*, are contaminated with heavy metals. While the levels of some heavy metals like lead may be within safe limits, the concentration of cadmium in some smoked samples exceeded international safety standards, raising a significant health concern. Cadmium is a known toxicant that can accumulate in the human body, potentially causing damage to the kidneys, brain, and other vital organs (WHO, 2011; FAO/WHO, 2019).

Recommendations

Based on the findings, fish vendors and processors should adopt strict hygienic practices during handling, processing, and storage, while modern smoking technologies should be introduced to reduce contamination and improve product safety. Regulatory agencies should conduct regular monitoring of fish products and enforce proper waste management practices

to minimize environmental pollution and heavy metal accumulation in aquatic ecosystems. In addition, public awareness programs should educate consumers on the health risks associated with poorly processed fish and the importance of proper cooking before consumption, as traditional smoking methods alone may not guarantee fish safety.

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