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Assessment of Some Heavy Metals and Pesticide Residues in Soil, Sediment, *Clarias gariepinus* and *Oryza sativa* Cultivated in Some Selected Agricultural Sites of Bauchi State, Nigeria

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ABSTRACT

Living organisms especially human beings get exposed to harmful substances such as heavy metals through the plants and animals we consume. Human sources of food like *Oryza sativa* and *Clarias gariepinus* get contaminated by the soil as a result of pesticides used by farmers. This study aimed to determine heavy metals levels and pesticide residue in soil, sediment, *Oryza sativa,* and *Clarias gariepinus* in three locations namely, Gadau, Jama'are, and Zabi (Giade LGA) in Bauchi State Nigeria. Samples of Soil, sediments, *Oryza sativa,* and *Clarias gariepinus* were collected at three different locations namely Gadau, Zabi, and Jamaare, to assess the level of some heavy metals and pesticide residues. The heavy metals present were detected in this order Zn>As>Pd>Cd>Cr>Ni in soil, sediment, *Oryza sativa,* and *Clarias gariepinus* respectively. The detected heavy metals were higher than the WHO/FAO recommended maximum tolerance values in three locations in this study. Among the active residues observed Abamectin and Dichlovos were more active compared to others with a 1- >5ppm range. The presence of these harmful substances in the environment is of serious concern to human health. This study recommends strict regulations on the use of pesticides for food crops and fish in the study area.

INTRODUCTION

In line with the increase in the world's food production especially in aquaculture (Kalbassi et al., 2013). There is a need to study environmental factors such as soil and how it affect plants and their diversity in relation to fish production (Fattahi & Ildoromi, 2011; Siddika & Parveen; Talabi et al., 2023). According to a comprehensive analysis by Troell et al. (2023), aquaculture has the potential to make significant contributions to achieving the Sustainable Development Goals. Rocha et al. (2022) provide a global overview of aquaculture food production and how it rises year in and year out. It is also important to sample and study the water quality of locations that are used for irrigation (Homayoonnezhad, 2023) Studies have shown the potential of using machine learning models to predict the quality of irrigation water (Dimple et al., 2022). Soil, the upper layer of the earth's crust is an admixture of numerous solid, liquid, and gassy substances having both living and non-living matter similar to mineral patches, decaying organic matter, microbes along with water and air contained in the spaces between the particles. Formation of soil is a unique and slow process starting from weathering which is the breakdown of large rocks known as bedrocks into small mineral particles (Karuma et al., 2021). This leads to soil development called pedogenesis, modification of mineral solid matter through relations between climatic, topographic, and biological factors (Karuma et al., 2021; Itkin et al., 2022). Therefore, soil is an important natural resource, formed over the centuries that supports the biodiversity of plants and provides a dwelling place for macroscopic and microscopic life forms apart from other known ecological functions (van Gestel et al., 2021; Yang et al., 2021).

Heavy metals are usually defined as metals with fairly high densities, atomic numbers, or atomic weights. The criteria used, and whether metalloids are included, depends on the context and author (Pourret et al., 2021; Jadaa & Mohammed, 2023). Heavy metals are life-threatening because they tend to bioaccumulate. Bioaccumulation refers to an increase in the absolute concentration of chemicals found in a living organism over a long or specified period of time, in relation to the chemical concentration found in that environment. Chemical compounds tend to accumulate in most living things any time they're taken into their systems and stored more than they're metabolized (Mitra et al., 2022).

Heavy metals have the capacity to degrade water, air, and soil quality, and thereafter lead to health issues in animals, plants, and people when they become highly concentrated as a result of various industrial activities (Namla et al., 2022; Zhao et al., 2022; Fatima & Singh, 2023). The common major sources of heavy metals here include smelting, mining, industrial wastes, motor oil, vehicle emissions, dyes, painting and pigments, illegal deposition of waste from construction, etc (Guo et al., 2012; Wang et al., 2019). Others include the burning of waste openly in rural areas, food contamination, contamination of ventilation by the environment or lead–acid batteries, treated timber, and electronic waste recycling yard (Asif et al., 2020; Bradl, 2005). Some aging water infrastructure is used for supply and little plastics floating in the earth's oceans (Howell et al., 2012). Of recent example of heavy metal health risk and contamination includes the disease occurrence in Minamata, in Japan between 1932 to 1968 with an ongoing lawsuit as of 2016 (Amasawa et al., 2016; Liu et al., 2022; Osae et al., 2023).

METHODS

Description of the Study Area

The study areas are located at Gadau (N 11º50'48.804" and E 10º7'25.65588"), Zabi (Giade Local Government) (N 11º30'28.78812" and E10º17'27.10212") and Jama'are (N 11º40'14.76516" and E 9º56'18.88836") towns in Bauchi State, Nigeria (figure 1). Bauchi state is a state in the northeast geopolitical zone of Nigeria, boarded by Kano and Jigawa to the north, Taraba and Plateau to the south, Gombe and Yobe to the east, and Kaduna to the west. A Sudan savanna vegetation type that can be considered less uniform with short grasses (Nodza et al., 2013).

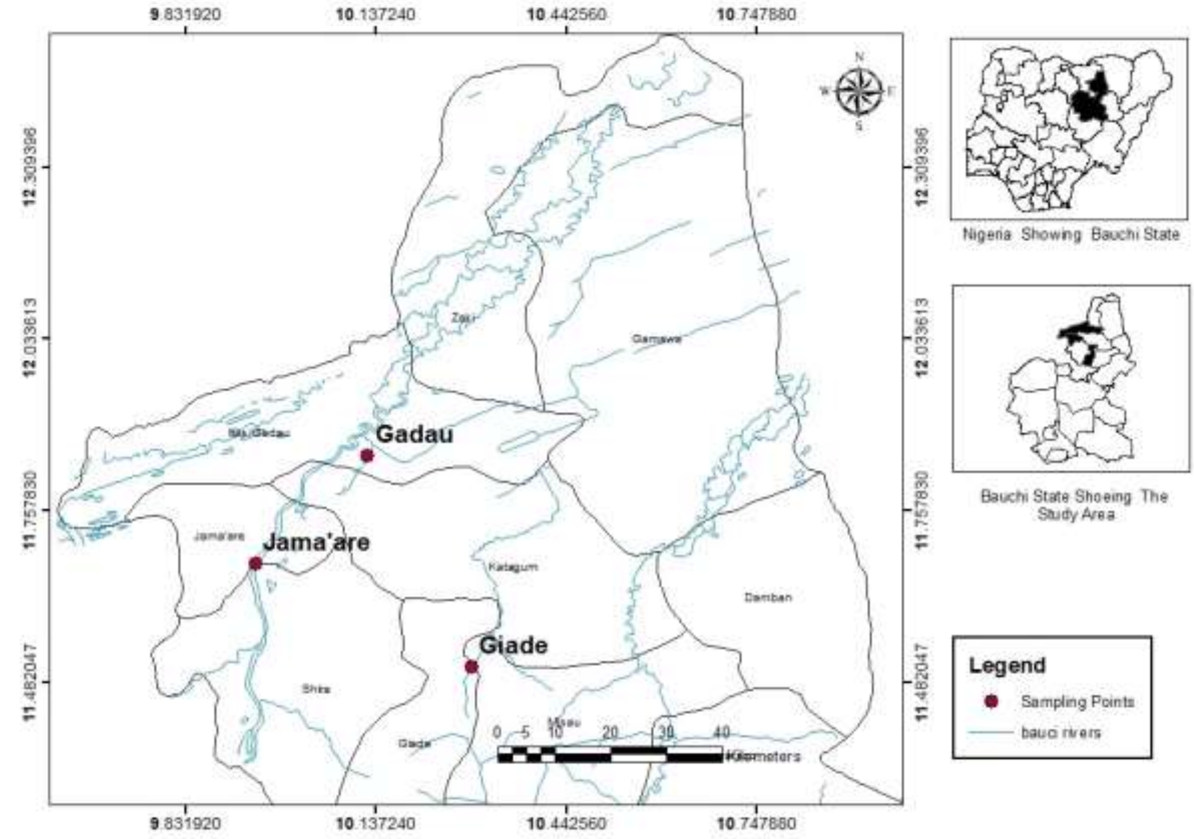

Figure 1: Map showing the sampling location

Sample Collection and Preparation for Heavy Metal Analysis

In each study area, soil and sediment samples were collected from several locations along the rivers. At least five replicates of each sample were collected, leveled, bagged, and stored properly for further analysis. After collection, the soil and sediment samples were dried (50°C for 24 hours), measured then ground to small powder using a pestle and mortar, and stored for further analysis. *Oryza sativa* samples were collected, using the same sampling pattern, from the same location where the soil and sediment samples were collected. *Oryza sativa s*amples were carefully collected by hand, and cross-contamination was avoided. The *Oryza sativa* samples were collected from at least five locations in each sampling site in the month of September 2023. They were subsequently dried at 70–80°C until a constant weight was obtained (Tiwari et al., 2011).

Collection and Preservation of *Clarias gariepinus* Samples

A sum of 45 samples of *Clarias gariepinus* were purchased from fishermen at the rivers in Gadau, Jama'are, and Zabi towns in the month of September 2023. The samples were then placed into a box containing ice blocks immediately after purchasing them from the fishermen, in order to prevent the specimen's tissue decay. These *Clarias gariepinus* samples were transported immediately to the Chemistry Laboratory of Yobe's State University, where the samples were then preserved in a freezer while awaiting dissection and preparation of the body parts for analysis of heavy metals.

Determination of Heavy Metal Concentrations in Soil Sediment and *Oryza sativa* Samples

The concentrations of heavy metals were detected in both soil, sediment and *Oryza sativa* using an atomic absorption spectrophotometer (iCE-3000 series, -ermo Scientific, USA). To ensure maximum sensitivity, an air-acetylene flame was used during the instrument operation. About 0.3 g of ground soil, sediment, and *Oryza sativa* were digested using a microwave digestion system (Berghof Speedwave, Germany) with 5 ml of 70% HNO3 and 2 ml of 30% H2O2. After digestion, Milli- Q water was then added to the digested samples to make a final volume of 25 mL. The chemicals used for this particular analysis were purchased from Merck, Germany, and are of analytical grade. All the digested samples were then filtered using a 0.45 μm filter syringe. Before analysis, all the consumables were soaked in diluted HNO3 for 24 h and finally rinsed with distilled water. The limits used for the detection of Cd, Zn, Pb, Cr, Ni, Co, and As were 0.07, 0.6, 0.10, 0.8, 0.8, 0.07, and 0.09 ng/l, respectively. A certified reference material by (Sigma Aldrich, USA) was used to ensure the precision of the applied method (Hasan et al., 2022).

Digestion and Metal Analysis

The frozen *Clarias gariepinus* samples were left to thaw at room temperature (20–27 °C). Parts of the flesh, gills kidney, and liver were collected from the fresh samples and oven-dried at 120 °C to constant weight. The dried samples were then ground to powder with the aid of a laboratory mortar. One gram of the grounded sample (for the flesh, gills, kidney, and liver each) was digested with 40% nitric acid. Cooled digested samples were then washed with deionized water, filtered, and made to the desired volume. The digested tissue parts were then analyzed for cadmium, arsenic, lead, chromium, zinc, and nickel concentrations using an atomic absorption spectrophotometer (Akaninyene et al., 2016).

Extraction by Gas Chromatography

The procedure of extraction of the samples by gas chromatography is the following. A 200g sample of fish was chopped and homogenized. A quantity of 15 g of the aliquot was put in one glass and mixed with 50 ml of dichloromethane (DCM) and stake in a centrifuge for 2 minutes. The sulfate of the anhydrous sodium (50 g) was added to the mixture and was put again in a centrifuge for one minute. The mixture was allowed to rest for 2 minutes then it was filtered through a funnel (Büchner) with 9 cm and was then filtered again through a Wattman paper and of the anhydrous sodium sulfate. The solvents evaporated to dry in a rotary evaporator (35°C - 40°C). The dried residual was taken and one added 5 ml of cyclohexane there. In a phial of 2 ml containing 50 μl of solution internal stallion of 20 mg/l, 1 ml of this solution has been added to reach the final of 2 ml in a volume of cyclohexane (Aïkpo et al., 2017).

Data Analysis

Data obtained from this research was analyzed using analysis of variance (ANOVA) and the means were separated with the Tukey posthoc method where there is a significant difference. These analyses were done using Minitab version 16.

RESULTS AND DISCUSSION

Heavy Metals

Cadmium

The mean cadmium values in (ppm) recorded in *Clarias gariepinus* body parts which include flesh, gills, kidney, and liver *Oryza sativa*, soil, and riverside sediment are presented in Table 1 The liver recorded the highest value (1.86) while the gills recorded 0.8 within *Clarias gariepinus* body parts. *Oryza sativa* had 1.09, the soil recorded 6.28 while the riverside sediment had the overall highest value (7.45). Among the three locations sampled, Zabi had the highest value (3.47) while Gadau had the lowest 1.57. These values are significantly different at p (0.05).

Arsenic

The mean values as presented in Table 1 of arsenic in (ppm) recorded in the samples indicated that kidney of *Clarias gariepinus* had the highest value (3.60) compared to the flesh, liver and gills while the gills recorded the least value (-1.63). The value recorded by *Oryza sativa* was 1.09 while the soil and the sediment recorded 7.06 and 17.77 respectively. Here Jama'are had 7.67 which was the highest value compared to 2.0 and 0.35 recorded for Zabi and Gadau respectively. The difference between these values is statistically significant at the 0.05 level.

Lead

The lead mean values in (ppm) observed in *Clarias gariepinus* body parts indicated that the value of 2.07 recorded in the kidney was higher than what was recorded in the flesh (0.71), liver (0.60) and gills (-0.96) which was the least. *Oryza sativa* had 1.68 compared to 10.29 and 5.99 recorded by the sediment and the soil respectively (table 1). The location Jama'are with a value of 5.13 was the highest among the three locations while the remaining two recorded 1.72 (Zabi) and 0.96 (Gadau). A statistically significant difference was observed between these values ($p < 0.05$).

Chromium

The heavy metal chromium recorded the following values in (ppm) in Table 1. The body parts of *Clarias gariepinus* had the following values in descending order; kidney (1.30), liver (1.13), and flesh (0.57). When *Oryza sativa* recorded value was compared to that of the soil and sediment, the sediment came on the top with 10.02 followed by 7.71 recorded by the soil while *Oryza sativa* had the least (1.62). These values are significantly different at p (0.05). The three locations differ significantly ($p < 0.05$) where Zabi recorded 4.32 compared to 2.97 and 0.96 recorded by Jama'are and Gadau respectively. The difference between these values is statistically significant at the 0.05 level.

Zinc

The mean zinc values recorded in (ppm) indicated that the liver of *Clarias gariepinus* had the highest value (26.37) compared to the 16.52, 15.77, and 12.10 recorded flesh, kidney, and gills respectively (table 1). The comparison between soil, sediment, and *Oryza sativa* indicated that 65.85 recorded by the soil was the highest as against what was recorded by sediment and *Oryza sativa* with the values 61.09 and 1.87 respectively. Similarly, Zabi recorded the highest value (41.50) among the locations compared to 23.47 and 10.23 observed in Gadau and Jama'are respectively. A statistically significant difference was observed between these values ($p < 0.05$).

Nickel

The mean nickel values recorded in (ppm) for the body parts of *Clarias gariepinus* indicated no significant difference between flesh, gills, kidney, and liver with the values 0.23, 068, 0.63, and 0.08 respectively (table 1). A significant difference was observed ($p < 0.05$) in the comparison between soil, sediment, and *Oryza sativa* indicating that soil (14.43) had the highest followed by sediment (11.73) while *Oryza sativa* (0.10) recorded the least. The values of the locations also differ significantly ($p<0.05$) where Gadau had the highest (5.10), followed by Zabi (3.82) while Jama'are recorded the least.

Table 1. Mean values of Cadmium, Arsenic, Lead, Chromium, Zinc and Nickel in ppm observed in *Clarias gariepinus* body parts, *Oryza sativa*, Sediments and soil in Gadau, Jama'are, and Zabi towns

	Cadmium (ppm)		Arsenic (ppm)		Lead (ppm)		Chromium (ppm)		Zinc (ppm)		Nickel (ppm)	
Sample	Mean	SE (±)	Mean	SE (±)	Mean	SE (±)	Mean	SE (±)	Mean	SE (±)	Mean	SE (±)
Clarias gariepinus flesh	0.99^{bc}	0.28	1.01^{bc}	0.45	0.71^{cd}	0.20	0.57^{b}	0.18	16.52^{bc}	2.73	0.23^{b}	0.07
Clarias gariepinus gills	0.80^{bc}	0.28	-1.63^{c}	0.45	-0.96^{d}	0.20	-0.63^{b}	0.18	12.10^{bc}	2.73	0.68^{b}	0.07
Clarias gariepinus kidney	1.36^{bc}	0.28	3.60^{bc}	0.45	2.07^{c}	0.20	1.30^{b}	0.18	15.77^{bc}	2.73	0.63^{b}	0.07
Clarias gariepinus liver	1.86^{b}	0.28	-0.0^{4c}	0.45	0.60^{cd}	0.20	1.13^{b}	0.18	26.37^{b}	2.73	0.08^{b}	0.07
Oryza sativa	1.09^{bc}	0.28	1.09^{bc}	0.45	1.68^{c}	0.20	1.62^{b}	0.18	1.87^{c}	2.73	0.10^{b}	0.07
Sediment	7.45^{a}	0.28	17.77^{a}	0.45	10.29^{a}	0.20	10.02^{a}	0.18	61.09^{a}	2.73	11.73^{a}	0.07
Soil	6.28^{a}	0.28	7.06^{b}	0.45	5.99^{b}	0.20	7.71^{a}	0.18	65.85^{a}	2.73	14.43^{a}	0.07
Location												
Gadau	1.57^{c}	0.16	0.35^{b}	0.12	0.96^{b}	0.30	0.96^{c}	0.31	23.47^{b}	2.89	5.10^{a}	0.78
Jama'are	2.60^{b}	0.16	7.67^{a}	0.12	5.13^{a}	0.30	2.97^{b}	0.31	10.23^{c}	2.89	1.59^{b}	0.78
Zabi	3.47^{a}	0.16	2.00^{b}	0.12	1.72^{b}	0.30	4.32^{a}	0.31	41.50^{a}	2.89	3.82^{ab}	0.78

Values sharing the same superscript in the same column are not significantly different ($p<0.05$).

Table 2. Pesticide Residues Mean Range Observed in *Clarias gariepinus* and *Oryza sativa* found in Gadau, Jama'are and Zabi towns

Location	Abamectin	Glyposate	Dichlovos	Chloripyrifos	Cypermetrin	Cyhalotrin	Imdaclorpid	Aldrin	Heptachlor
Gadau	+++	–	+	++	+	–	+	–	+
Jama'are	++	+	+	–	–	–	–	–	–
Zabi	+++	–	+	–	–	–	+	–	–

+++ (> 5 ppm), ++ (1 – 5 ppm), + (< 1 ppm) and – (0 ppm)

Pesticide Residues

The pesticide residues present in the soil of the three locations were presented in table (2) below. Abamectin was heavily present in the three locations (Gadau, Jama'are, and Zabi) but Gadau and Zabi recorded higher values. Dichlovos was the only other pesticide residue present in a mild form in the three locations. Chloripyrifos, Cypermetrin, and Heptachlor were only observed mildly in Gadau while Imdaclorpid was mildly observed in both Gadau and Zabi.

Fish occupies various levels of food chain levels in aquatic environments hence the reason why they are fantastic indicators of heavy metals contamination levels (Rafeek et al., 2021; Karadede-Akin, 2007; Akaninyene et al., 2016; Bawuro et al., 2018). Taherizadeh et al. (2018) conducted a study in the Persian Gulf to assess the levels of heavy metals in edible fish and evaluate the potential health risks associated with their consumption. The accumulation of heavy metals in the body parts of *Clarias gariepinus* such as flesh, gills, kidney, and liver differ significantly ($p<0.05$) in this study. This is in conformity with what was reported by (Akaninyene et al., 2016; Rao and Padmaja, 2000; Sibomana et al., 2023) who reported different concentrations of heavy metals in different fish body parts. Cadmium and Zinc (1.86ppm and 26.37ppm) were higher in *Clarias gariepinus* liver while Arsenic, Lead, and Chromium (3.60ppm, 2.07ppm and 1.30ppm) were higher in *Clarias gariepinus* kidney, which is contrary to the report of (Indrajith et al., 2008). The above mentioned discrepancies could be a result of the rate of bioaccumulation of heavy metals in organisms like fish depending on its ability to digest such heavy metals, the concentration of heavy metals in the surrounding sediment and water bodies, and even the source of pollution. The overall heavy metal trend for this study was in the decreasing order of Zn>As>Pd>Cd>Cr>Ni. This trend is not in conformity of what was reported by (Eneji et al. 2011) who reported the trend Cr > Zn > Cd > Pd. Several authors reported different trends according to (Akaninyene et al., 2016).

The results obtained in a heavy metal assessment in *Oryza sativa* carried out by (Hasan et al., 2022) detected the following: Cd (0.98 ± 0.32 ppm–1.61± 0.79 ppm), Pb (ND–1.32 ppm), Cr (11.54 ± 4.09–23.67 ± 9.95 ppm), Co (8.54 ± 3.32–18.11 ± 5.09 ppm), Ni (ND–0.18 ppm), and As (0.031 ± 0.01–0.075 ± 0.03 ppm) in the crop samples of Savar, Gazipur, and Ashulia, respectively. This is contrary to what was observed in this study which recorded Cd (1.09 ppm ± 0.28), Pb (1.68 ppm ± 0.20), Cr (1.62ppm ± 0.18), Ni (0.10ppm ± 0.07), Ar (1.09 ppm ± 0.45), Zn (1.87 ppm ± 2.73).

Singh et al. (2015) analyzed ethion, dicofol, and chlorpyriphos in the river Deomoni from the region of Terai in West Bengal and revealed the mean pesticide residues of ethion, dicofol, and chlorpyriphos in the water sample as 0.0892 ± 0.0375 ppm, 0.0180 ± 0.0071ppm, and 0.0091 ± 0.0020ppm, respectively, in sediments 0.0513 ± 0.0085ppm, 0.0414 ± 0.0045ppm and 0.1271 ± 0.0122ppm and in fish muscles (Puntius sp.) 5.0371 ± 1.4236 ppm, 3.7700 ± 0.6391ppm and 2.9599 ± 0.4027ppm, respectively. This is almost similar to the values observed in the three locations in this study.

CONCLUSION

The detected heavy metals were higher than the WHO/FAO recommended maximum tolerance values in three locations in this study. Among the active residues observed Abamectin and Dichlovos were more active compared to others with a 1->5ppm range. The presence of these harmful substances in the environment is of serious concern to human health. This study recommends strict regulations on the use of pesticides for food crops and fish in the study area.

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