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Determination of Pesticide Residues in Sediment, Water and Fish from Some Selected Agricultural Sites in Bauchi State, Nigeria

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ABSTRACT

Pesticide residues play a crucial role in the health of human through bio-accumulation through food chain or food web. The study was conducted to determine the pesticide residues (abamectin, aldrin, chlorpyrifos, cyhalotrin, cypermethrin, dichlorvos, glyphosate, heptachlor, Imadachlorpid) present in sediment, fish and river water samples from some agricultural sites in Bauchi state. Samples were extracted and analyzed by High Performance Liquid Chromatography (HPLC). Fish samples were analyzed based on organs (gills, flesh, liver, and kidney) with a pesticide residue value ranged from 0.06±0.08ppm to 0.16±0.24ppm in Gadau site, 0.04±0.01ppm to 2.5±3.37ppm in Giade site and 0.04±0.00ppm to 0.06±0.03ppm in Jama'are site. The sediment samples 0.09±0.00ppm to 13.70±13.46ppm, 0.97±0.00ppm to 24.15±31.28ppm and 0.04±0.00ppm to 6.93±13.38ppm at Gadau, Giade and Jama'are sites respectively. Meanwhile the river water sample had a ranged value of pesticide residues as 0.15±0.15ppm to 1.04±1.50ppm, 0.11±0.00ppm to 3.34±4.29ppm and 0.17±0.16ppm to 8.20±7.38ppm from Gadau, Giade and Jama'are sites. Jama'are samples were less contaminated with the organophosphorus pesticide residues and the other corresponding sampling sites. Dichlorvos and Heptachlor were found to be higher than the permissible limits of 0.1 mg/kg and 0.01mg/kg in all the samples that were determined except where not detected. Also farmers should be educated on a proper usage of the pesticides they use and the implications of using too much of it to the environment..

Keywords: Pesticides, Sediment, Fish, Water, Agriculture.

INTRODUCTION

Agriculture comprises crop and livestock production, aquaculture, fisheries and forestry for food and non-food products. Pesticides are toxic substances or a mixture of substances that are naturally or chemically synthesized. These are widely used for controlling harmful weeds (herbicides), fungi (fungicides), bacteria (bactericides), and insect infestations (insecticides) in the agricultural field (Abdel-Khalek, *et al.,* 2018). Also, these can be used on some disease carriers and pests (e.g., ticks, rodents, mosquitoes, and lice) in the entire ecosystem (Kim, Kabir, and Jahan, 2017).

Agricultural fields are the largest consumer, which represent about 85% of the global production of pesticides. Furthermore, these can help reduces and prevent insect infestation outbreaks, fungi, and bacteria in moisturized areas (carpets, refrigerators, and cupboards, etc.) (Gilden, Huffling, and Sattler, 2010).

Pesticides are top list of environmental intoxicants that endanger nature and poses a serious threat to human health and agricultural production (Alengebawy *et al.,* 2021). Fish are rich sources of proteins and lipids, and their health is very important for Human, Therefore, the protection of aquatic ecosystem and water quality will be possible only with the sagacious and quantified applications of pesticides (Pallavi *et al.,* 2016). It is recommended that the extensive use of these

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pesticides should be reduced near water bodies and the applications should be sagacious.

Pesticide poisoning is a global public health concern, with almost 300,000 deaths every year worldwide. Pesticide exposure is inevitable; there are multiple methods in which people are exposed to pesticides (Sabarwal, Kumar and Singh 2018).

This study is aimed at assessing the level of pesticide residues present in sediment, fish, and water of the selected agricultural sites.

MATERIALS AND METHODS

Study Area

Bauchi state is a state in the north-east 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. The targeted sampling areas were Giade, Gadau and Jama'are local government areas of Bauchi state which are located within:

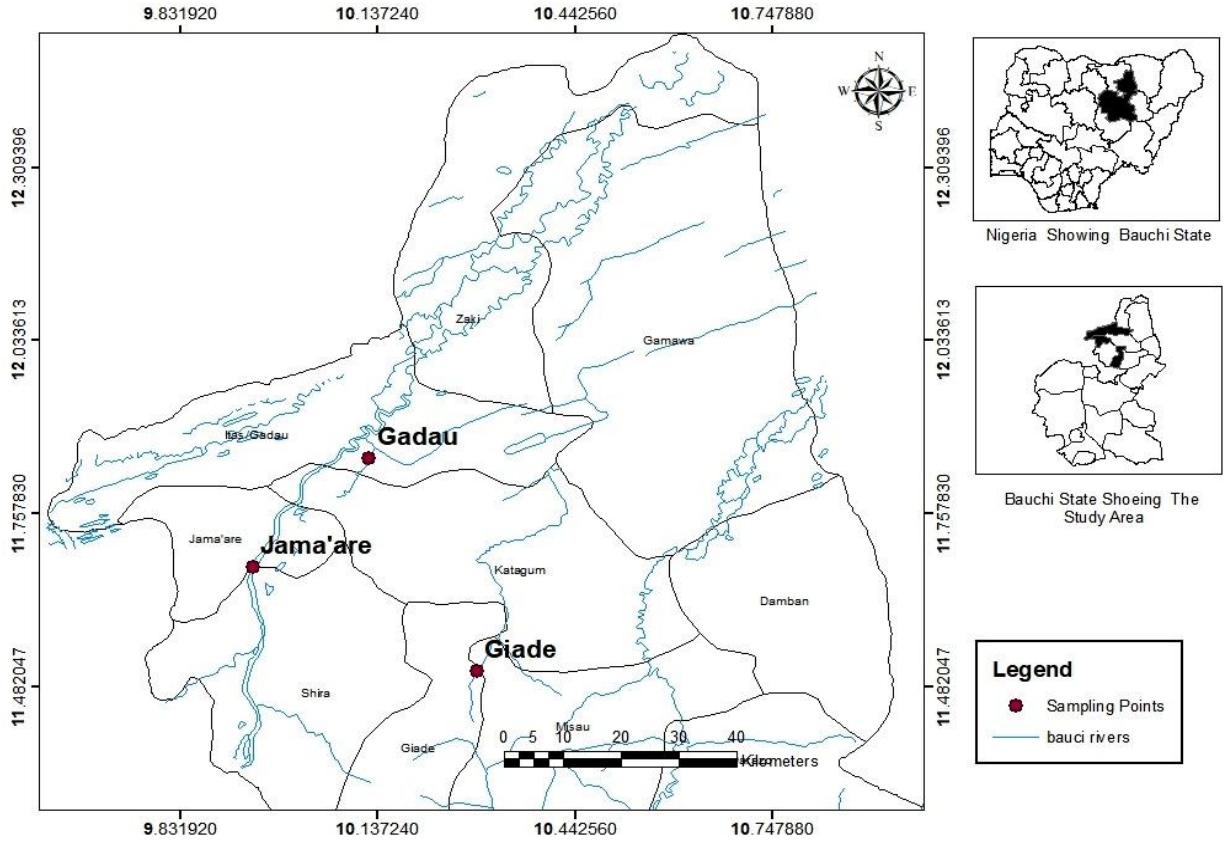

Figure 1: Map showing the sampling location.

Sample Collection

The samples comprises of sediment, fish, and river water from each of the 3 agricultural farm sites. The samples were collected in a sample rubber, bottles and polyethylene bags and transported to the laboratory. The fish samples were preserved inside an ice and later frozen to avoid early spoilage while the river water was acidified using Nitric acid.

Sample Preparation

Sediment: the sediment was prepared by oven drying and then homogenized. Then

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transferred and stored in a dried clean container.

Water: the acidified river water was subjected to necessary analysis to determine the presents of heavy metals residues. Fish: the collected preserved samples were dissected into different portions (organs) and washed thoroughly, then rinsed with distilled water. It was then allowed to oven dry at 105℃ for 4hours, then grinded into powder (homogeneous) and then placed in a well labeled clean dried container for analysis.

Pesticide Residue Determination

Sample extraction (Fish and Sediment)

Five grams each of the samples were added into 150 mL of boiling water (85±5°C) for 30 min and then filtered via filter paper (Whatman, 110 mm diameter, no. 6) in a Buchner funnel. The filtrate was transferred to a 250-mL separatory funnel containing 100 mL dichloromethane. The separatory funnel was vigorously shaken for 5 min and then kept until the organic layer separated from the aqueous layer. The organic layer was collected filtered with anhydrous sodium sulfate and then evaporated on a rotary vacuum evaporator. The concentrated extract was dissolved in acetonitrile (5 mL) and then transferred to a 15-mL Teflon centrifuge tube containing C18 (100 mg) and PSA (200 mg). The tubes were vortex mixed for 0.5 min and centrifuged at 4,500 rpm for 5 min. The supernatant was collected and kept for HPLC/GCMS analysis. (Sung-Woo *et al.,* 2014*)*

Sample extraction (WATER)

Ten (10) mL of each samples were added into a 250-mL separatory funnel containing 100 mL dichloromethane. The separatory funnel was vigorously shaken for 5 min and then kept until the organic layer separated from the aqueous layer. The organic layer was collected filtered with anhydrous sodium sulfate and then evaporated on a rotary vacuum evaporator. The concentrated extract was dissolved in acetonitrile (5 mL) and then transferred to a 15-mL Teflon centrifuge tube containing C18 (100 mg) and PSA (200 mg). The tubes were vortex mixed for 0.5 min and centrifuged at 4,500 rpm for 5 min. The supernatant was collected and kept for HPLC/GCMS analysis. (Sung-Woo *et al.,* 2014*)*

The mobile phase consisted of acetonitrile and water (95:5 v/v) with 1% acetic acid. The flow rate was 1.0 mL min^{-1} and UV detection was made at 245 nm.

The HPLC equipped with a solvent-delivery system, an auto-injector fitted with a 20 µL loop, an online degasification system, a column thermostat oven and an ultraviolet/visible (UV/VIS) with photodiode array detector.

Analytical curves for abamectin were obtained using six (6) different concentration levels. Standard solutions were diluted with mobile phase and determinations were made in triplicate. Sample solution were reconstituted to a 2 mL with acetonitrile. UV detection was performed at 243, 245 and 247 nm.Flow rate was maintained at 1.0 mL/min. 50 µL was injected and run for about 12 minutes. Abamectin concentrations in the samples were extrapolated from the standard chromatogram based on their retention time matching

RESULTS

Table 1, 2 and 3 shows the pesticide residues in samples from Gadau, Giade and Jama'are sites respectively.

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Table 1: Concentrations (ppm) of Pesticide Residues (PRs) in samples from Gadau

Pesticides	F/Gills	F/Liver	F/Flesh	F/Kidney	Sediment	Water
Abamectin	0.164±0.24	ND	ND	0.06±0.08	8.04±10.43	1.04±1.50
Aldrin	ND	ND	ND	ND	ND	ND
Chlorpyrifos	ND	ND	ND	ND	13.7±13.46	0.154 ±0.151
Cyhalotrin	ND	ND	ND	ND	ND	ND
Cypermetrin	ND	ND	ND	ND	1.4 ±0.00	0.27±0.00
Dichlorvos	ND	ND	ND	ND	2.89±1.27	0.278±0.34
Glyposate	ND	ND	ND	ND	1.67±0.00	ND
Heptachlor	ND	ND	ND	ND	0.09±0.00	0.27±0.00
Imdaclorpid	ND	ND	ND	ND	ND	0.18±0.00

ND= Not Detected SD= Standard Deviation

Table 2: Concentrations (ppm) of Pesticide Residues (PRs) in samples from Giade

Pesticides	F/Gills	F/Liver	F/Flesh	F/Kdny	Sediment	Water
Abamectin	0.494±0.71	0.26±0.04	0.04±0.01	0.18±0.26	24.15±31.28	3.34±4.29
Aldrin	ND	ND	ND	ND	ND	0.11±0.00
Chlorpyrifos	0.18±0.00	ND	ND	2.5±3.37	ND	0.28±0.10
Cyhalotrin	ND	ND	ND	ND	ND	0.47±0.00
Cypermetrin	ND	ND	ND	ND	ND	0.15±0.01
Dichlorvos	ND	ND	ND	2.5±3.27	2.11±0.48	2.39±1.11
Glyposate	ND	ND	ND	ND	1.76±0.00	0.46±0.14
Heptachlor	ND	ND	ND	ND	ND	ND
Imdaclorpid	ND	ND	ND	ND	0.97±0.00	1.39±0.71

ND= Not Detected SD= Standard Deviation

Table 3: Mean Concentrations (ppm) of Pesticide Residues (PRs) in samples from Jama'are

Pesticides	F/Gills	F/Liver	F/Flesh	F/Kdny	Sediment	Water
Abamectin	0.046±0.02	0.04±0.00	0.06±0.03	0.06±0.03	2.2±1.60	0.29±0.09
Aldrin	ND	ND	ND	ND	ND	ND
Chlorpyrifos	ND	ND	ND	ND	ND	ND
Cyhalotrin	ND	ND	ND	ND	0.168±0.00	0.25±0.23
Cypermetrin	ND	ND	ND	ND	ND	ND
Dichlorvos	ND	ND	ND	ND	6.93±13.38	8.2±7.38
Glyposate	ND	ND	ND	ND	0.04±0.02	0.17±0.16
Heptachlor	ND	ND	ND	ND	ND	ND
Imdaclorpid	ND	ND	ND	ND	ND	ND

ND= Not Detected SD= Standard Deviation

DISCUSSION

Levels of Pesticide Residues (PRs)

Abamectin, Aldrin, chlorpyrifos, Cyhalotrin, Cypermetrin, Dichlorvos, Glyposate, Heptachlor and Imdachorpid were determined from the samples. Abamectin was found in all analyzed samples except in fish liver and flesh from gadau site. A highest concentration of abamectin was found in soil of Giade (33.25±41.96ppm). Aldrin was only found in water of Giade as 0.11±0.00ppm. The value was below the maximum residue limit of 0.2mg/kg which could be contributed due to the nature of the sample. This is contrary to the findings of (Abolagba *et al*., 2021) who recorded the following; aldrin, at 406.8 ± 0.12 µg/kg, endosulfan, measuring 315 ± 0.164 µg/kg and heptachlor epoxide, recorded at 156 ± 6.23 µg/kg.

Highest Chlorpyrifos concentration was observed in the sediment of Gadau site

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(13.7±13.46 ppm) as seen from table 1. All the obtained values of this OPP residue were above the maximum residue limit of 1.0mg/kg except that of Gadau water and Giade water& fish gills.

The mean concentration of Cyhalotrin in the water, sediment and fish parts from all agricultural sites were presented in the respective tables (1, 2, and 3). It was not detected in Gadau site, meanwhile found in Giade water (0.47±0.00 ppm) and also in Jama'are sediment and water samples (0.168±0.00 and 0.25±0.23 ppm). These values were said to be above the CODEX 2007 maximum residue limit of 0.02ppm for water. However (Patiri *et al.,* 2019) detected the following levels in Kisiju (2.26±0.51mg/kg) and Kilwa (0.28±0.40mg/kg).

Jama'are samples were said to be free from OPP Cypermetrin but present in some of Gadau and Giade samples with a range of 0.15±0.01 to 1.4±0.00 ppm. Dichlorvos is a predominant OPP used in domestic insect control in developing countries. Acute and prolonged exposure may lead to death, genotoxic, neurological, reproductive, carcinogenic, and immunological and other systematic effects (Okoroiwu and Iwara 2018). All the observed value of dichlorvos from all samples were higher than the maximum residue limit of 0.1mg/kg (Omeje *et al*., 2021) with a highest concentration in 8.2±7.38 in water of Jama'are and lowest in Gadau water with 0.278±0.34ppm.

Gadau sediment and water (0.09±0.00 ppm and 0.27±0.00 ppm respectively) were contaminated with higher concentration of heptachlor with a maximum residue limit of 0.01mg/kg. Heptachlor is an organochloride pesticide that was used to control pest, ants, maggots, termites, cutworms etc in agriculture and homes. Its sales was cancelled in 1987 by its sole US manufacturer due to its health hazard effects (ATSDR, 1993)

Imdaclorpid was not found in any of the Jama'are samples, while observed in Gadau (water 0.18±0.00 ppm) and Giade (sediment 0.97±0.00 ppm and water1.39±0.71 ppm). The mean concentration of Glyposate ranged from 0.04±0.02 ppm of Jama'are sediment to 1.76±0.00 ppm of Giade sediment. However (Peruzzo *et al.,* 2008) got a range from 0.10 to 0.70 ppm

CONCLUSION

From all the results of this study, Jama'are samples were less contaminated with the organophosphorus pesticide residues and the other corresponding sampling sites. Dichlorvos and Heptachlor were found to be higher than the permissible limits of 0.1 mg/kg and 0.01mg/kg in all the samples that were determined except where not detected.

Also farmers should be educated on a proper usage of the pesticides they use and the implications of using too much of it to the environment. Furthermore, they should be educated on how polluted water used for irrigation can be harmful to human health through food chain or web.

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