International Journal of Applied Chemical and Biological Sciences (2023), 4(3), 1-6
ISSN- 2582-788X

RESEARCH PAPER

Article Identifier: https://identifier.visnav.in/1.0001/ijacbs-23g-27001/

Analysis of Organochlorine Pesticides Residue in African-lung fish (*Protopterus aethiopicus*) from Odogwu Farming Community of Ibaji Local Government Area of Kogi State

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Received on: 6 April 2023 *Published on: 30 June 2023*

ABSTRACT

Pesticides are widely used worldwide to control weeds and insects and to increase food production. However, the residues left behind by these pesticides can have adverse effects on both the environment and human beings. This study presents the results of quantitative analysis of fifteen organochlorine pesticide (OCP) residues in two catfish samples, A and B, obtained from waterlogged rice farms in Odogwu community, Ibaji. The analysis employed the Quick Easy Cheap Effective Rugged and Safe (QuechERS) method, followed by gas chromatography mass spectrophotometer (GC-MS) analysis. In sample A, eight different organochlorine residues were identified, and in sample B, nine organochlorine residues were identified. The concentrations of the residues in both samples ranged from 0.01 µg/mL of Atrazine to 7.67 µg/mL of Lindane (δ-BHC, delta-BHC). These two compounds were found to have the lowest and highest levels, respectively, in both samples. The mean levels of OCPs in the fish were generally higher than the recommended Maximum Residue Levels (MRLs) set by the World Health Organization (WHO) for food items. For instance, γ-BHC (Lindane) and dieldrin (δ-BHC) with WHO MRLs of 6.0 µg/kg were found in this study at levels of 7.67 µg/kg and 3.37 µg/kg, respectively. However, the levels of 1,1,1-trichloro-2,2-bis(p-chlorophenyl) ethane (DDT) in the fish samples were generally lower than the WHO MRLs of 5.00 µg/kg. The results obtained for Lindane (γ-BHC) were higher than the

Maximum Residue Level (MRL) set by the United States (USA). Additionally, β-BHC levels exceeded the USA MRL recommended limit of 0.03 µg/mL, while α-BHC levels were within the USA MRL of 0.05 µg/mL.

Keywords: Pesticides, persistent organic pollutant, organochlorine pesticides, Benzene Hydrochloride (BHC).

1. INTRODUCTION

The use of pesticides to control weeds, insects, and other pests has brought numerous benefits, including increased food production and a reduction in insect-borne diseases. However, it also raises concerns about potential adverse effects on the environment, particularly water quality [1].

The invasion of pests in farm crops has led farmers to seek methods of combating this issue. Initially, they tried different farming methods like crop rotation and the use of natural substances such as ash and canola oil. Eventually, the discovery of chemical pesticides became a significant breakthrough. However, it was found that all these methods were only effective for a limited period before side effects emerged, including those associated with chemical pesticides.

While chemical pesticides have undoubtedly contributed to agricultural production by protecting crops and increasing yields, the presence of pesticide residues on non-targeted substances and in the environment has raised concerns about potential negative impacts that may outweigh the overall benefits [2].

One group of chemical pesticides is organochlorine pesticides (OCPs), which are organic compounds containing carbon and chlorine. They have been extensively used worldwide but are now considered environmental contaminants and persistent organic pollutants (POPs). OCPs have long half-lives ranging from months to decades, resisting degradation by various means (Darko and Acquaah, 2007) [3].

OCPs are known to be toxic to humans, animals, and aquatic organisms, posing ecotoxicological and public health risks (Adebisi et al., 2020 [4] Exposure to OCPs at low concentrations can disrupt sex hormones, leading to abnormal sexual development, sex ratios, and mating behaviors in animals. Symptoms of pesticide exposure include headaches, vomiting, skin rashes, respiratory problems, and convulsions. Prolonged pesticide exposure can even lead to cancer. Furthermore, OCP residues can harm unborn children's DNA and the endocrine system, as well as damage nerves and brain cells (Williams et al., 2013) [5].

The World Health Organization (WHO) categorizes pesticides as either class I (extremely hazardous) or class II (slightly toxic) based on their toxicity, as they are known to be poisonous, hazardous, and toxic to humans [6].

Numerous researchers have investigated organochlorine pesticide residues in various food crops, soils, sediments, water, and aquatic animals using different techniques and instruments. However, this research will focus solely on analyzing organochlorine pesticide residues in catfish samples from the local government area of Kogi State.

2. METHODOLOGY

2.1. Fish Sample Collection and Identification

The fish samples used in this analysis were obtained from waterlogged rice farms in Odogwu community, Ibaji area of Kogi State, Nigeria. The fish samples were identified by the Fisheries Department. The selected fish species for this study is the African lungfish (*Protopterus*

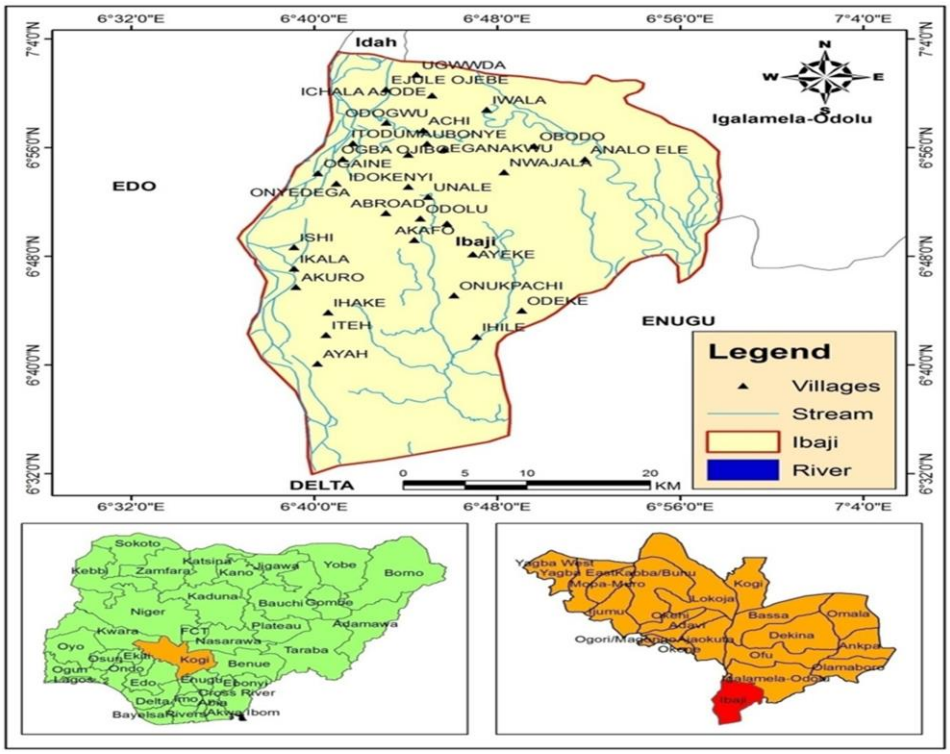

Figure 1. Study area (Igbaji local government) [7].

aethiopicus). This species is common in the study areas but is gradually facing extinction due to the presumed effects of pesticides.

2.2. Sample Preparation and Extraction

The analysis of pesticide residues in solid samples is challenging due to the presence of interfering compounds in the sample matrix. Thus, sample pre-treatment, including sieving, grinding, and sometimes drying, is necessary to obtain reliable results. In recent decades, several modern techniques have been proposed to reduce prolonged sample handling and toxic waste and to maximize analyte recovery while minimizing interferences by employing appropriate extraction and clean-up procedures [8].

2.3. Extraction of Pesticide Residues in Fish Samples

The Quick Easy Cheap Effective Rugged and Safe (QuechERS) method of extraction, as described by [9], was used. Five grams of blended fish samples were weighed in 50cm3 centrifuge tubes using an analytical balance. Then, 15 cm3 of acetonitrile containing 1% glacial acetic acid (v/v) was added to each sample using a solvent dispenser. The tube was tightly capped and shaken for 1 minute to ensure thorough contact between the solvent and the sample matrix. Next, 6 g of anhydrous MgSO4, 1.5 g NaCl, 1.5g sodium citrate tribasic dihydrate ($C_6H_5Na_3O_7 \cdot 2H_2O$), and 0.75 g sodium hydrogen citrate ($C_6H_6Na_2O_7 \cdot 1.5H_2O$) were added to each sample. The sample mixture was shaken for 5 minutes on a mechanical shaker at 300 rpm to enhance sample throughput. Subsequently, the sample mixture was centrifuged for 5 minutes, and a 5 cm3 aliquot of the extract was taken into a 10 cm3 test tube. From this aliquot, 1 cm3 was placed into a 10 cm3 tube and diluted to 10 cm3 with water. The extract was then transferred from the 10 mL flask into an autosampler vial for further cleanup.

2.4. Sample Cleanup

Table 1. GC-MS results of Catfish obtained from an Ibaji River.

Compounds	Concentration, ug/mL Samples	
	A	B
Etridiazole	N.D.	N.D.
Chloroneb	N.D.	N.D.
α-BHC	N.D.	0.01
Simazine	N.D.	N.D.
Atrazine	0.01	0.01
β-BHC	0.04	0.02
γ-BHC	N.D.	3.34
δ-BHC	7.67	5.01
Chlorothalonil	0.73	1.01
Alachlor	N.D.	N.D.
Aldrin	N.D.	N.D.
Dacthal	2.49	1.2
Heptachlor epoxide	3.85	7.08
γ-Chlordane	2.27	1.08
trans-Nonachlor	6.66	N.D.

A column of about 15 cm (length) × 1 cm (internal diameter) was packed with approximately 10 g of activated silica gel prepared as a slurry in n-Hexane. About 10 g of anhydrous sodium sulfate was placed on top of the column to absorb any water in the sample or the solvent. The column was pre-eluted with 20 mL of n-Hexane without exposing the sodium sulfate layer to air. The reduced extract was then placed in the column and allowed to sink below the sodium sulfate layer. Elution was performed with 2 × 10 mL portions of the extracting solvent (DCM). The eluate was collected, dried with anhydrous sodium sulfate, and evaporated to dryness under a stream of analytical-grade nitrogen (99.999%).

2.5. Analysis of Pesticide Residues

Following the clean-up procedure, the samples were analyzed for pesticide residues using a gas chromatography-mass spectrophotometer (GC-MS). The oven was programmed to start at 50°C, then ramp at 20°C/min to 100°C, and finally at 20°C/min to 250°C. The total running time was 19 minutes, while the MS temperature was set at 280°C. Helium was used as the makeup gas at 10 mL.

3. RESULTS AND DISCUSSION

The results obtained from the GC-MS analysis after, and the extraction and clean-up of the two samples extract are tabulated in Table 4.1. Table 4.1 presents the results of quantitative analysis conducted on two samples of catfish obtained from water in rice farms at Odogwu community in Ibaji. Fifteen different organochlorine compounds were calibrated for the analysis. In sample A, eight (8) organochlorine compounds were quantified, while in sample B, nine organochlorine compounds were quantified. The concentrations of the residues in both samples ranged from 0.01 µg/mL of Atrazine to 7.67 µg/mL of δ-BHC (delta-BHC). These two compounds represented the least and highest quantified residues in both samples, respectively. These findings align with results obtained by [10, 11] on organochlorine and organophosphorus pesticide residues in fish samples from Lake Chad, Baga, northeastern Nigeria.

The mean levels of OCPs in the fish samples were, in most cases, higher than the recommended WHO Maximum Residue Levels (MRLs) for food items. For instance, γ-BHC (lindane) and dieldrin (δ-BHC), with WHO MRLs of 6.0 µg/kg [3], were found in this study at levels of 7.67 µg/kg and 3.37 µg/kg, respectively. However, the levels of DDT in the fish samples were generally lower than the WHO MRLs of 5.00 µg/kg [3].

The obtained values for chlordane, ranging from 1.28 µg/mL to 1.43 µg/mL, were higher than those reported by [11] for fish samples obtained from River Donga in Taraba state, which ranged from 0.003 µg/mL to 0.012 µg/mL. Similarly, β-BHC and α-BHC had higher values compared to the results reported by Emmanuel et al. in 2021. The levels of Lindane (γ-BHC) were also higher than the US Maximum Residue Level (MRL). Additionally, β-BHC exceeded the US MRL recommended limit of 0.03 µg/mL, while α-BHC remained within the US MRL of 0.05 µg/mL.

4. CONCLUSION

This study is limited to the quantitative analysis of pesticide residues in two catfish species. Based on the findings of this study, it can be concluded that chemical pesticides pose a significant danger to both humans and the environment. The presence of residual chemical pesticides in fish and other aquatic animals, which are subsequently consumed by humans, has been linked to a wide range of illnesses, including cancer and central nervous system (CNS) disorders.

Many of these chemical compounds have long half-lives and are not easily biodegradable into non-harmful substances. As a result, they persist in the soil for extended periods after application to crops and eventually wash down into rivers, streams, and lakes, contaminating the water and endangering the lives of aquatic animals and other organisms, including humans.

Based on the results of this study, it is strongly recommended that certain actions be taken. Farmers should receive re-education on the proper use of chemical pesticides, emphasizing adherence to recommended application guidelines, the use of protective gear, and the adoption of integrated pest management practices. Furthermore, relevant government agencies and regulatory bodies should consider banning the use of persistent chemical pesticides due to their long-lasting environmental effects and potential risks to human health. In addition, the adoption and promotion of biopesticides should be encouraged as an alternative to chemical pesticides, given their shorter half-lives and ability to biodegrade into non-harmful substances by microorganisms in the environment.

5. ACKNOWLEDGEMENT

NA

6. CONFLICT OF INTEREST

The authors have declared that there is no conflict of interest.

7. SOURCE/S OF FUNDING

NA

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