



Assessment of Pesticide Residue in Water from Farmlands around Mahanga Lake, Nigeria

*Modibbo U.U., Dowell B.F. and Umar, M.A.

Chemistry Department, Faculty of Physical Sciences, Modibbo Adama University Yola,

Adamawa State, Nigeria

*Corresponding Author: umarusmanmodibbo@gmail.com

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ABSTRACT

This study investigated the occurrence of organochlorine pesticide residues in water samples collected from points around Mahanga Lake, Bali Local Government Area, Taraba State, Nigeria. Pesticide residues, including lindane isomers (Alpha, Gamma, Delta), aldrin, endrin, heptachlor, heptachlor epoxide, endosulfan I/II, DDT, DDD, DDE, and methoxychlor, were analyzed using gas chromatography–mass spectrometry (GC-MS). Results revealed widespread contamination, with concentrations ranging from 0.01 to 0.66 mg/kg. Endosulfan I consistently recorded the highest values across all sites, while aldrin and lindane isomers were also prominent. The detection of DDT metabolites (DDD and DDE) alongside lower parent DDT concentrations reflects typical degradation pathways of legacy pesticides. Compared with WHO and USEPA guideline values for drinking water, the measured concentrations were several orders of magnitude higher, indicating severe contamination. These findings align with other studies that report pesticide residues in surface waters, but Mahanga Lake exhibited significantly higher levels, suggesting intense agricultural runoff and persistence of banned pesticides. The results highlight ecological and public health concerns, necessitating improved monitoring, regulation, and sustainable agricultural practices.

Keywords: Mahanga Lake, Organochlorine pesticides, GC-MS, Water contamination

INTRODUCTION

Pesticides are used in agriculture to protect crops against insects, fungi, weeds and other pests as well as to protect public health in controlling the vectors of tropical diseases like mosquitoes. They can also be used to prevent, destroy, repel or mitigate any pest and can either kill pests or render them ineffective [1]. Pesticides are used on fruits, vegetables, wheat, rice, olives, canola pressed into oil and on non-food crops such as cotton, grass and flowers. Pesticides applied to food crops in the field can leave potentially harmful residues [2].

Pesticides may contaminate water sources as a result of runoff, wastewater discharges, and return flow from agricultural and irrigated areas. They enter water through direct application to control aquatic weeds or indirectly through transportation from treated areas. Leakage and runoff from agricultural areas are the primary sources of conveyance to water sources [3]. The detection and quantification of pesticide in water sources together with their potential environmental health risks have been extensively documented in many areas of the world, such as in Argentina, Japan, the United States, India, the Czech Republic, China, and Malaysia. In spite of such significant research outputs, the potential human health risks associated with pesticides found in various water sources essentially remain unrevealed in most of these studies. Moreover, most of these studies focus less on water sources, such as treated drinking water, which is considered the safest drinking water source. As a result of the environmental dynamics and incessant use of a number of pesticides, their presence in the water environment as well as their end products are concerning, as they may trigger possible effects on aquatic ecosystems and human health, particularly via the ingestion of water [4].

Contamination occurs not only due to current use of agrochemicals but also due to leaching of persistent ingredients from soil. Pesticide contamination of surface water in a particular region depends on several factors, such as closeness of crop fields to surface water, characteristics of surrounding fields (soil, grassland, slope, and distance to water bodies), and climate conditions (temperature, humidity, wind, and precipitation). In consequence, pesticide residues are being reported as common organic contaminants worldwide in surface waters and other environmental matrices [5].

The deleterious health problems caused by toxicants are increasing due to their penetration and accumulation through the food chain, and their persistence in the ecosystem. Such contaminants can cause acute and chronic diseases in the human body, such as lung cancer, renal dysfunction, osteoporosis, and cardiac failure [6]. Mahanga Lake in Taraba State is a vital resource for drinking water, irrigation, fishing, and domestic use. Yet, no systematic study has assessed the extent of pesticide contamination in this lake, despite its importance to local communities and the likelihood of agricultural runoff from surrounding farmlands. This lack of data hampers effective risk assessment and management strategies.

Given the persistence and toxicity of organochlorine pesticides (OCPs), and their potential to bioaccumulate in aquatic ecosystems, it is essential to evaluate their presence in Mahanga Lake. Such an assessment will provide evidence for ecological protection, public health safeguarding, and policy enforcement regarding pesticide regulation. The present study investigates the occurrence and concentration of organochlorine pesticide residues in water samples collected from farmlands around Mahanga Lake, Nigeria.

MATERIALS AND METHODS

Study Area

Bali is a Local Government Area (LGA) in Taraba state, Nigeria. Bali LGA was created in 1976. The Local Government Area lies between latitude 7°46' N and 7°54' N of the equator and longitude 10°03' E and 11°00' E of the prime meridian. This falls within the dry guinea savannah with an estimated land area of 11,540 km². It has some mountains like Gazabu, Dakka, Maihula, Bagoni, among others. Based on the 2006 National Population Census, Bali had a population of about 211,024 persons [7].

Mahanga is a historic settlement located at outskirts of Bali town with a predominant population belonging to the Jibu tribe, traces of Fulani and Tiv farmers. The lake (Figure 1) plays a vital role in the economy of the people and their routine daily activities. It serves as a source of drinking water to the populace and their livestock especially during dry season. The fishermen in the community indulge in fishing in the lake. In addition to that, it also serves as a source of water for irrigation as they engage in dry season rice farming. And to the children is a place for swimming and washing of their clothes.

MATERIALS AND METHODS

The pH meter (Hanna Instruments, Woonsocket, USA) calibrated with standard buffer solutions at pH 4.0, 7.0, and 10.0 prior to measurement, Acetone or methanol solvent, analytical pesticide residues standard which was used as calibration curve to determine pesticide and Agilent 7890B Gas Chromatograph coupled with a 5977B Mass Selective Detector was used for analysis of pesticide residues in samples. All the chemicals and reagents used are of analytical grade.

Sampling and Sample Preparations

The water samples were collected by dipping plastic bottles below the water surface at farmlands around the lake. Total of four samples were collected from four sampling points i.e. North, South,

East and West. At each sampling locations, the sampling bottles were rinsed three times with the water before collection of the sample [8]. Water samples were measured in to 250 ml conical flask in volume of 50ml for digestion and extraction and was taken for analysis using GC-MS.

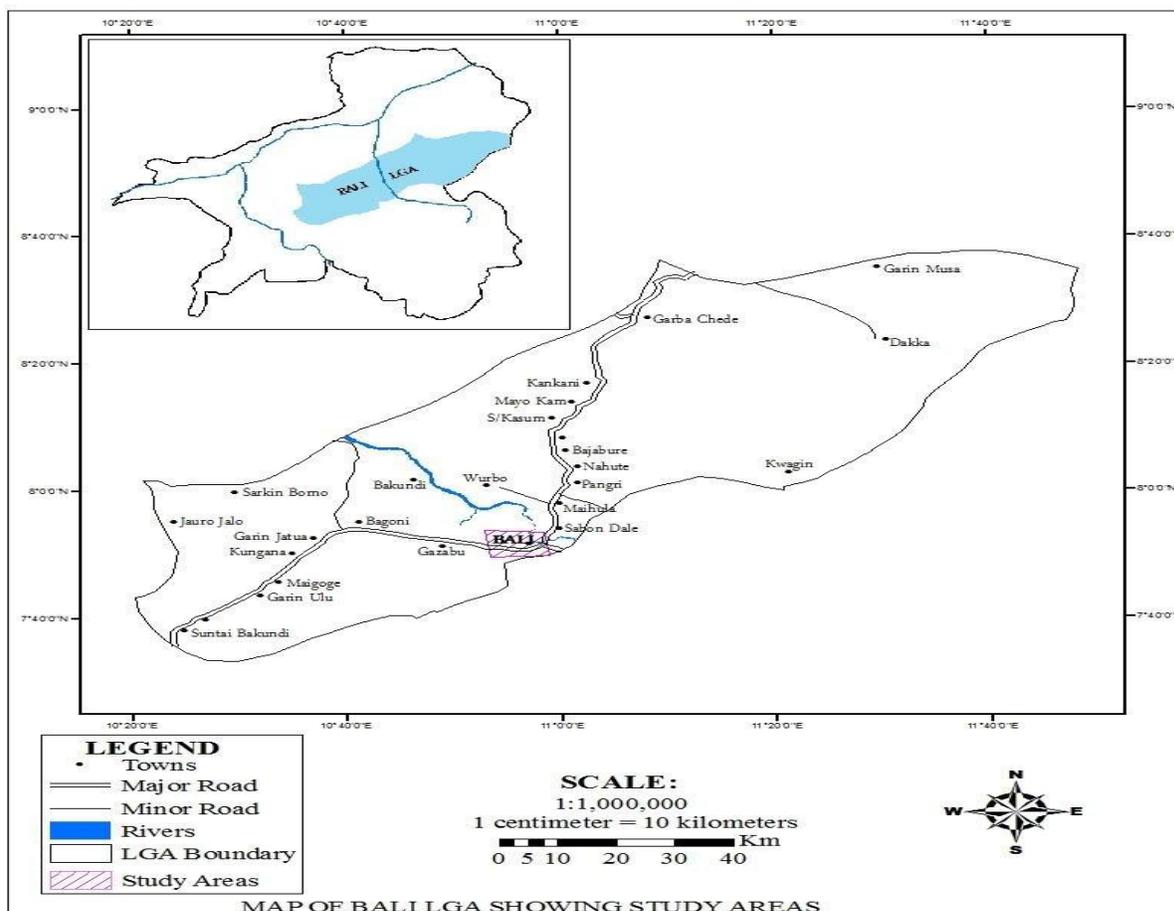


Figure 1: Map of the Study Area

Extraction of water samples for Pesticide Residues

Water samples were first filtered using Whatman No. 1 filter paper to remove suspended particulate matter. A 20 mL aliquot of the filtered water was transferred into a 250 mL glass beaker, after which 20 mL of an organic solvent mixture (acetone: hexane, 1:1 v/v) was added. This solvent system is widely recognized for its efficiency in extracting pesticide residues from aqueous matrices. The mixture was shaken vigorously for 10 minutes using a mechanical shaker at 200 rpm, ensuring thorough contact between the aqueous and organic phases. Following agitation, the mixture was allowed to stand for 5 minutes to facilitate phase separation. The

organic solvent layer was carefully separated using a separation funnel and transferred into clean amber vials to minimize photodegradation. These extracts were subsequently subjected to analysis by GC-MS, a standard technique for sensitive and reliable quantification of pesticide residues in environmental samples [9].

GC-MS Analysis

Extracts were analyzed using an Agilent 7890B Gas Chromatograph coupled with a 5977B Mass Selective Detector (Agilent Technologies, USA) equipped with a DB-5MS capillary column (30 m × 0.25 mm i.d., 0.25 μm film thickness). Helium (99.999%) was used as the carrier gas at a constant flow rate of 1.0 mL/min. Samples (1 μL) were injected in splitless mode at 250 °C. The oven program was initial at 70 °C (2 min), ramped at 10 °C/min to 180 °C, then 3 °C/min to 280 °C (10 min hold). The MS was operated in electron impact (EI) mode at 70 eV, with source temperature 230 °C, quadrupole 150 °C, and transfer line 280 °C. Data were acquired in selected ion monitoring (SIM) mode for quantification, with full scan (m/z 50–500) used for confirmation [10].

Calibration, Detection, and Quantification

Certified pesticide residue standards (Sigma-Aldrich, USA) were used to prepare calibration curves within the concentration range of 0.01–2.0 mg/L. Calibration was performed using a weighted linear regression (1/x), and correlation coefficients (R^2) were consistently ≥ 0.995 , ensuring accuracy and reliability of the analytical method. An internal standard (PCB-209) was added to all samples to correct for variability in extraction efficiency and instrument injection.

The limit of detection (LOD) was defined as a signal-to-noise ratio (S/N) of 3, while the limit of quantification (LOQ) was defined as an S/N of 10. Identification of pesticide residues was based on retention time matching within ± 0.2 minutes of the calibration standard, ion ratio consistency within $\pm 20\%$ of the reference value, and spectral similarity of at least 80% when compared with the NIST mass spectral library. Quantification was carried out using external calibration with internal standard correction, and results were expressed in mg/kg [11].

RESULTS AND DISCUSSION

Concentration of Pesticide Residues in Water from Sampling Sites around Mahanga Lake

The water samples are presented with their various retention times which are shown from Table 1 to Table 4. The Delta-lindane, Alpha-lindane, Gamma-lindane, Heptachlor, Aldrin, Heptachlor Epoxide, Endosulfan I, P, P – DDD, P, P- DDT and Methoxychlor were the pesticide residue analysed.

Concentration of pesticide residues in water sample around Mahanga lake from Northern sampling point

Table 1: Pesticide Residues from Northern Water Sample around Mahaga Lake.

Pesticide Residues	Types of Residues	Molecular Formula	Molecular Mass (g/Mol)	RT (Min)	Concentration (mg/kg)
Delta-Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	5.313	0.05
Alpha-Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	5.862	0.01
Gamma-Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	6.634	0.02
Heptachlor	Insecticide	C ₁₀ H ₅ Cl ₇	373	6.76	0.02
Aldrin	Insecticide	C ₁₂ H ₈ C ₁₆	365	7.298	0.29
Heptachlor Epoxide	Insecticide	C ₁₀ H ₅ Cl ₇ O	389	7.996	0.02
Endosulfan I	Insecticide	C ₉ H ₆ Cl ₆ O ₃ S	407	8.626	0.66
P,P,-DDE	Insecticide	C ₁₄ H ₈ C ₁₄	318	9.043	0.06
Endrin	Insecticide	C ₁₂ H ₁₆ N ₃ O ₃ PS	313	9.49	0.24
Endosulfan II	Insecticide	C ₉ H ₆ Cl ₆ O ₂ S	407	9.77	0.09
P,P-DDD	Insecticide	C ₁₄ H ₉ C ₁₅	354	9.93	0.13
P,-DDT	Insecticide	C ₁₄ H ₉ C ₁₅	354	10.503	0.02
Methoxychlor	Insecticide	C ₁₆ H ₁₅ Cl ₃ O ₂	346	11.441	0.02

RT = Retention Time

Pesticide residue of water from Northern sampling site had the following concentration, Delta-lindane (0.053 mg/kg), Alpha-Lindane (0.02 mg/kg), Gamma-lindane (0.02 mg/kg), Heptachlor (0.014 mg/kg) Endosulfan I (0.66 mg/kg), P, P- DDE (0.06 mg/kg), Endrin (0.24 mg/kg), Endosulfan II (0.09 mg/kg), P, P-DDD (0.12 mg/kg), P, P-DDT (0.02 mg/kg), and Methoxychlor (0.02 mg/kg). At the northern sampling point, endosulfan I recorded the highest concentration (0.66 mg/kg), while alpha-lindane was the lowest (0.01 mg/kg). The dominance of endosulfan I is consistent with its known persistence and higher proportion in technical formulations compared to endosulfan II [12]. The detection of aldrin and endrin at significant levels is concerning, as these compounds are highly toxic and have been banned globally due to their persistence and bioaccumulation [13]. Similar results were obtained in River Tella, Taraba State where Organochlorine pesticides (OCPs) detected in water and fish, with endosulfan residues present but at lower concentrations than Mahanga Lake [14].

Concentration of pesticide residues in water sample around Mahanga lake from Southern sampling point

Table 2: Pesticide Residues from Southern Water Sample Around Mahaga Lake

Pesticide Residues	Types of Residues	Molecular Formula	Molecular Mass (g/Mol)	RT (Min)	Concentration (mg/kg)
Delta-Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	5.336	0.04
Alpha-Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	5.862	0.03
Gamma-Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	6.52	0.18
Heptachlor Aldrin	Insecticide	C ₁₀ H ₅ Cl ₇	373	6.738	0.03
Heptachlor Epoxide	Insecticide	C ₁₂ H ₈ Cl ₆	365	7.287	0.09
	Insecticide	C ₁₀ H ₅ Cl ₇ O	389	8.008	0.04

Endosulfan I	Insecticide	C ₉ H ₆ Cl ₆ O ₃ S	407	8.683	0.62
P,P,-DDE	Insecticide	C ₁₄ H ₈ C ₁₄	318	9.049	0.02
Endrin	Insecticide	C ₁₂ H ₁₆ N ₃ O ₃ PS	313	9.455	0.01
Endosulfan II	Insecticide	C ₉ H ₆ Cl ₆ O ₂ S	407	9.782	0.06
P,P-DDD	Insecticide	C ₁₄ H ₉ C ₁₅	354	9.942	0.03
P,-DDT	Insecticide	C ₁₄ H ₉ C ₁₅	354	10.503	0.02
Methoxychlor	Insecticide	C ₁₆ H ₁₅ Cl ₃ O ₂	346	11.492	0.01

RT = Retention Time

The pesticide residue concentration in the southern site of the lake had the following concentration levels in the order Delta-Lindane (0.04 mg/kg) Alpha-Lindane (0.03 mg/kg), Aldrin (0.09 mg/kg), Endosulfan I (0.62 mg/kg), P, P-DDE (0.02 mg/kg), Endrin (0.01 mg/kg), Endosulfan II (0.06 mg/kg), P, P-DDD (0.03 mg/kg), P, P-DDT (0.02 mg/kg) and methoxychlor (0.01 mg/kg). The higher concentration of Endosulfan I (0.62 mg/kg) detected in the water sample compare is Endosulfan II (0.06 mg/kg) may be attributed due to the manufacture technical endosulfan, which normally contains about 67% endosulfan I by weight of the total endosulfan content, while endosulfan II constitute only 32% [15]. It is therefore, not unexpected that more endosulfan would be found in the environment when the pesticide is applied. More also, endosulfan I is thermally stable while endosulfan II is unstable and it converts endosulfan I in the environment [11].

Concentration of pesticide residues in water sample around Mahanga lake from Eastern sampling point

Table 3: Pesticide Residues from Eastern Water Sample Around Mahanga Lake

Pesticide Residues	Types of Residues	Molecular Formula	Molecular Mass (g/Mol)	RT (Min)	Concentration (mg/kg)
Delta-Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	5.376	0.06
Alpha-Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	5.851	0.02

Gamma-Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	6.577	0.09
Heptachlor	Insecticide	C ₁₀ H ₅ Cl ₇	373	6.795	0.01
Aldrin	Insecticide	C ₁₂ H ₈ Cl ₆	365	7.241	0.30
Heptachlor Epoxide	Insecticide	C ₁₀ H ₅ Cl ₇ O	389	7.996	0.03
Endosulfan I	Insecticide	C ₉ H ₆ Cl ₆ O ₃ S	407	8.586	0.44
P,P,-DDE	Insecticide	C ₁₄ H ₈ Cl ₄	318	9.112	0.04
Endrin	Insecticide	C ₁₂ H ₁₆ N ₃ O ₃ PS	313	9.456	0.10
Endosulfan II	Insecticide	C ₉ H ₆ Cl ₆ O ₂ S	407	9.782	0.08
P,P-DDD	Insecticide	C ₁₄ H ₉ Cl ₃	354	9.942	0.27
P,-DDT	Insecticide	C ₁₄ H ₉ Cl ₅	354	10.474	0.01
Methoxychlor	Insecticide	C ₁₆ H ₁₅ Cl ₃ O ₂	346	11.521	0.01

RT = Retention Time

In the eastern site water sampling, the concentration of individual sample levels showed that Delta-lindane had concentration of (0.06 mg/kg), Alpha-lindane (0.02 mg/kg), Gamma-lindane (0.09 mg/kg), Heptachlor (0.01 mg/kg), Aldrin (0.30 mg/kg), Heptachlor epoxide (0.03 mg/kg), Endosulfan I (0.44 mg/kg), P, P- DDE (0.04 mg/kg), Endrin (0.10 mg/kg), Endosulfan II (0.08 mg/kg), P,P-DDD (0.27 mg/kg), P,P-DDT (0.01 mg/kg) and Methoxychlor (0.01 mg/kg). At the eastern sampling point, aldrin (0.30 mg/kg) and endosulfan I (0.44 mg/kg) was again dominant, while heptachlor was lowest (0.01 mg/kg). The detection of DDT metabolites (DDD and DDE) alongside lower parent DDT concentrations reflects typical degradation pathways of legacy pesticides, consistent with findings in African freshwater ecosystems where DDE and DDD are more abundant than DDT [16].

Concentration of pesticide residues in water sample around Mahanga lake from Western sampling point

Table 4: Pesticide Residues from Western Water Sample Around Mahaga Lake

Pesticide Residues	Types of Residues	Molecular Formula	Molecular Mass	RT (Min)	Concentration (mg/kg)
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			(g/Mol)		
Delta- Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	5.416	0.05
Alpha- Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	5.868	0.02
Gamma- Lindane	Insecticide	C ₆ H ₆ Cl ₆	291	6.652	0.16
Heptachlor	Insecticide	C ₁₀ H ₅ Cl ₇	373	6.766	0.01
Aldrin	Insecticide	C ₁₂ H ₈ Cl ₆	365	7.424	0.19
Heptachlor Epoxide	Insecticide	C ₁₀ H ₅ Cl ₇ O	389	7.996	0.02
Endosulfan I	Insecticide	C ₉ H ₆ Cl ₆ O ₃ S	407	8.637	0.51
P,P,-DDE	Insecticide	C ₁₄ H ₈ Cl ₄	318	9.055	0.02
Endrin	Insecticide	C ₁₂ H ₁₆ N ₃ O ₃ PS	313	9.438	0.16
Endosulfan II	Insecticide	C ₉ H ₆ Cl ₆ O ₂ S	407	9.77	0.51
P,P-DDD	Insecticide	C ₁₄ H ₉ Cl ₅	354	9.982	0.05
P,-DDT	Insecticide	C ₁₄ H ₉ Cl ₅	354	10.377	0.02
Methoxychlor	Insecticide	C ₁₆ H ₁₅ Cl ₃ O ₂	346	11.469	0.01

RT = Retention Time

The pesticide residue concentrations from western site showed that Delta-lindane had concentration of (0.05 mg/kg), Alpha-lindane (0.02 mg/kg), Gamma-lindane (0.16 mg/kg), Heptachlor (0.01 mg/kg), Aldrin (0.19 mg/kg), Heptachlor Epoxide (0.02 mg/kg), Endosulfan I (0.51 mg/kg), P,P-DDE (0.02 mg/kg), Endrin (0.16 mg/kg), Endosulfan II (0.05 mg/kg), P,P-DDD (0.02 mg/kg), P,P-DDD (0.02 mg/kg), P,P-DDD (0.02 mg/kg) and methoxychlor (0.01 mg/kg), respectively. At the western sampling point, Gamma-lindane (0.16 mg/kg), aldrin (0.19 mg/kg), and endosulfan I (0.51 mg/kg) was prominent. The repeated detection of aldrin and endrin highlights ongoing contamination, possibly from leaching of old pesticide stocks or residues in soils [17]. Methoxychlor was also detected, which is notable because it was once used as a substitute for DDT but is now recognized as environmentally persistent [12]. The higher concentration of Endosulfan I (0.51 mg/kg) detected in the water sample when compared

Endosulfan II (0.05 mg/kg), may be attributed due to the manufacture technical endosulfan normally contains about 67% endosulfan I by weight of the total endosulfan content, while endosulfan II constitute only 32% [15].

CONCLUSION

The results of water analysis from farmlands around Mahanga Lake demonstrates extensive contamination by multiple organochlorine pesticide residues. Endosulfan I was consistently dominant across all sampling points, reflecting its persistence and continued use, while aldrin, endrin, and lindane isomers were also detected at concerning levels. The presence of DDT metabolites further indicates legacy contamination from historical pesticide applications. Compared to WHO and USEPA permissible limits, the concentrations observed around Mahanga Lake are alarmingly high, posing risks to aquatic ecosystems and human health through drinking water, irrigation, and fish consumption. These findings are consistent with reports from other water bodies, though Mahanga Lake exhibits much higher contamination levels, underscoring localized pollution pressures. To mitigate these risks, there is a pressing need for stricter enforcement of pesticide regulations, improved agricultural practices, and continuous monitoring of water quality. Addressing pesticide pollution in Mahanga Lake will not only protect ecosystem integrity but also safeguard the health of communities that depend on the lake for water, food, and livelihood.

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