

Occurrence, Distribution, And Ecological Risk of Pesticide Residues in Surface Water and Sediments of the Orashi River, Niger Delta, Nigeria

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Abstract- Farming practices along riverine areas are one of the significant sources of pesticide pollution of the water-bodies in the developing nations. The study was therefore aimed at assessing the incidence, spatial distribution and ecological hazard of pesticide residues in surface water and sediment of five stations along the Orashi River of Niger Delta in Nigeria. Extraction of the sample was done using the liquid - liquid and ultrasonic methods and analysis by gas chromatography mass spectrometry (GC-MS). Eighteen pesticide residues were found in surface water (organophosphates, synthetic pyrethroids, carbamates, a fungicide, and an insecticide), and total concentration of the pesticide was between 0.463 ± 0.032 and 67.045 ± 0.143 $\mu\text{g/L}$. Dichlorvos recorded the highest individual concentration (21.873 ± 0.067 $\mu\text{g/L}$). Sediments had 16 residues, ranging in total amounts between 1.422 ± 0.284 and 4.053 ± 0.877 mg/kg , and the highest concentration was found in p,p-DDE (2.233 ± 0.035 mg/kg), which revealed historic DDT use. Some of the pesticides such as aldrin, dieldrin, lindane, heptachlor and carbaryl surpassed the international guidelines. Aldrin (RQ = 382.3), dieldrin (RQ = 185.0) and carbaryl (RQ = 4.87) were highly problematic contaminants judging from risk quotient analysis. The concentrations of the pesticides were significantly higher in sediments compared to surface water, which underscores them as long-term pollutant storage. The prevalence of the past and present-day pesticides, is an indicator of nonstop anthropogenic contributions and may have ecological and human health hazards. The ecosystem of the Orashi River and the communities that rely on it are in severe need for protection through regular monitoring and enforcement of the regulations as well as introducing sustainable agricultural methods.

Keywords: Pesticide, Residues, Surface Water, Sediment, Ecological Risk

I. INTRODUCTION

Modern agriculture is highly dependent on pesticides as an improvement to crop production and a means of pest control, but a significant proportion of the pesticides produced end up in the water-body due to surface runoff, leaching, atmospheric deposition, and haphazard disposal of the chemical compounds. Pesticides can accumulate in the water and separate in the water and sediments once they are introduced into freshwater systems and have the effect of causing toxicity in non-target organisms, thus altering the structure and functioning of the ecosystem (Singh & Mandal, 2013; López-Pacheco et al., 2019; Schulz et al., 2021).

Sediments are the key factors in the aquatic contamination processes, as they can both act as long-term sinks of hydrophobic pesticides like organochlorines as well as act as secondary sources by resuspending and diffusing to the overlying waters. Surface water, in its turn, constitutes a direct exposure route in the case of aquatic organisms and human populations that use untreated river water when providing domestic needs (Akoto et al., 2016; Danladi & Akoto, 2021). In turn, the simultaneous evaluation of pesticides in water and sediments is an exhaustive perspective of ecological risk and environmental pollution.

Poor regulatory implementation, especially in the tropical developing countries such as Nigeria, and extensive small scale agricultural activities have aggravated pesticide application in most of them, most of the time without proper training and compliance with safety regulations. Niger Delta is a

region that is at an especial risk because of a high density of rivers, agriculture, as well as its reliance of the local people on surface water resources. In the Nigerian freshwater systems, previous research has noted the presence of organophosphorus, carbamate, and pyrethroid pesticides in water, sediments, and aquatic organisms detectably and often exceeding the international guidelines (Ezemonye et al., 2015; Asiegbu et al., 2022; Awe et al., 2022).

Most pesticides are of endocrine-disrupting, neurotoxic, carcinogenic, and acute toxicity which are commonly associated with acetylcholinesterase inhibition, especially in the case of organophosphorus (Parker et al., 2017; Karami-Mohajeri et al., 2017). Even low doses when chronic might lead to sub-lethal ecological outcomes and long-term human health consequences associated with bioaccumulation and trophic transfer (Rani et al., 2020; Xu et al., 2020). Over three million cases of pesticide poisoning are reported throughout the world every year, which indicates how urgent monitoring of the environment and risk evaluation in the endangered areas are.

Although the agrochemical contamination in the Niger Delta is becoming a major issue of concern, there are limited data regarding the occurrence of the pesticide residues together in the Niger Delta surface water and sediments of the Orashi River. Orashi River sustains many riverine communities that rely on this water-body to supply their domestic water and fishing activities which makes contamination a possible public health problem.

Thus, the research objectives included (i) determine the occurrence and spatial distribution of pesticide residues in surface water and sediments from selected stations along the Orashi River, Niger Delta, Nigeria; (ii) the assessment of the accumulation patterns across the environmental compartments; (iii) the determination of the possible risks of the ecological and human health based on the risk quotient model. The results provide baseline information that will be used in environmental management and regulatory decision-making in the region.

II. MATERIALS AND METHODS

Study Area

Sampling was done along the middle reaches of Orashi River, in the eastern Niger Delta, Nigeria between longitude 06°26'32.5"–06°30'05.0" E and latitude 05°01'20.4"–05°08'24.6" N. It was carried out in five stations on a downstream-upstream gradient: Odieke (Station I), Odiobo (Station II), Mbiama (Station III), Akinima (Station IV), and Oshiobele (Station V). The river passes through mostly agricultural settlements with subsistence farming, fishing and hunting activities which are likely sources of the pesticide input.

Water and sediment sampling

Surface water samples were collected in pre-cleaned amber glass bottles of 1.5 L at approximately 3 cm depth below the water surface. Sample water was used to rinse bottles before collecting them. An Ekman grab sampler was used to take samples of sediments, which were transferred to solvent-rinsed glass containers. All samples were transported in ice to the laboratory and stored at 4 °C before the analysis.

Extraction and clean-up

Water samples were filtered through 0.45 µm fiberglass filters and extracted using liquid-liquid extraction with n-hexane according to Pandit et al. (2006). Sediment samples were air-dried, homogenized, mixed with anhydrous sodium sulfate and subjected to ultrasonic extraction using dichloromethane:n-hexane (2:3, v/v). A rotary evaporator was used to concentrate extracts at 40 °C.

GC–MS Analysis

The amount of residue of pesticides was measured with the help of GC-MS (Varian 3800/4000) equipped with an HP-5MS (30 m × 250 µm × 0.25 µm) capillary column. Electronic impact ionization was operated at 70 eV. The identification was done based on the retention time corresponding and mass spectral comparison with established reference standards and library spectra. The quantification was done through external calibration curves.

A dataset of all the detected pesticide residues across sampling stations of water and sediment samples is provided as supplementary Table S1, while Tables 1 and 2 present compounds consistently detected above quantification limits and/or relevant to risk assessment.

Quality assurance and quality control

Procedural blanks and triplicate analyses were performed to ensure analytical reliability. Calibration curves for all target pesticides showed correlation coefficients (R^2) ≥ 0.995 . Method detection limits ranged from 0.001–0.01 $\mu\text{g/L}$ for water and 0.001–0.01 mg/kg for sediments. Recovery experiments conducted by spiking samples with known pesticide concentrations yielded recoveries between 82–106%. Relative standard deviations for replicate analyses were below 10%. Concentrations below detection limits were reported as $< \text{d.l}$

Human health risk assessment

Potential human health risk associated with pesticide exposure via surface water was evaluated using the Risk Quotient (RQ):

$$RQ = C / GV \quad (1)$$

where C is the measured pesticide concentration ($\mu\text{g/L}$) and GV is the corresponding guideline value (WHO/EU/USEPA). RQ values < 1 indicate low risk, while $RQ \geq 1$ suggests potential health concern.

III. RESULTS

Pesticide residues in surface water

Eighteen pesticide residues belonging to organochlorines, organophosphates, synthetic pyrethroids, carbamates, a fungicide, and an insecticide were detected in surface water samples collected from the Orashi River (Table 1). Total pesticide concentrations ranged from 0.463 ± 0.032 to $67.045 \pm 0.143 \mu\text{g/L}$, with the highest cumulative burden recorded at Odieke station, indicating strong localized anthropogenic input.

Table 1. Concentrations of pesticide residues in surface water from the Orashi River ($\mu\text{g/L}$, mean \pm SD, $n = 3$)

Pesticide	Odheke	Odiobo	Mbiama	Akinima	Oshiobebe	Guideline value
Methomyl	2.153 \pm 0.021	0.043 \pm 0.012	0.037 \pm 0.006	0.037 \pm 0.006	0.050 \pm 0.010	3.5
Penconazole	3.357 \pm 0.045	0.087 \pm 0.023	0.103 \pm 0.006	0.103 \pm 0.006	0.113 \pm 0.015	200
Dichlorvos	21.873 \pm 0.067	20.987 \pm 0.037	9.423 \pm 0.025	9.423 \pm 0.025	9.623 \pm 0.021	20
Lindane	2.127 \pm 0.023	2.203 \pm 0.006	1.293 \pm 0.021	1.293 \pm 0.021	1.300 \pm 0.000	2
Diazinone	1.107 \pm 0.012	1.200 \pm 0.30	0.087 \pm 0.015	0.087 \pm 0.015	0.107 \pm 0.015	10
Carbofuran	1.043 \pm 0.040	1.207 \pm 0.012	1.257 \pm 0.023	1.257 \pm 0.023	1.303 \pm 0.031	7
Aldrin	10.170 \pm 0.061	9.217 \pm 0.012	12.337 \pm 0.035	12.337 \pm 0.035	12.303 \pm 0.025	0.03
Pirimiphos methyl	3.260 \pm 0.026	0.173 \pm 0.006	2.127 \pm 0.023	1.127 \pm 0.023	2.113 \pm 0.023	1
Carbaryl	4.773 \pm 0.035	12.320 \pm 0.026	11.343 \pm 0.021	11.343 \pm 0.021	11.307 \pm 0.012	2.1
Dieldrin	1.203 \pm 0.006	2.130 \pm 0.112	8.143 \pm 0.038	8.143 \pm 0.038	8.120 \pm 0.017	0.03
Heptachlor	0.127 \pm 0.012	1.230 \pm 0.017	1.233 \pm 0.042	1.233 \pm 0.042	1.223 \pm 0.025	1
Bifenthrin	0.183 \pm 0.025	0.130 \pm 0.010	1.297 \pm 0.015	1.297 \pm 0.015	1.297 \pm 0.015	0.01

Guideline values from WHO (2009). USEPA (2012); EFSA (2016).

Among individual compounds, dichlorvos exhibited the highest concentration, reaching $21.873 \pm 0.067 \mu\text{g/L}$, accounting for approximately 34% of total residues. Elevated levels of aldrin (up to $12.337 \pm$

$0.035 \mu\text{g/L}$), dieldrin ($8.143 \pm 0.038 \mu\text{g/L}$), carbaryl ($11.343 \pm 0.021 \mu\text{g/L}$), and chlorpyrifos ($6.510 \pm 0.030 \mu\text{g/L}$) were also observed across multiple stations (Table 1). These concentrations exceeded WHO and USEPA guideline values for freshwater.

Spatial variation showed consistently higher pesticide loads at Odieke and Odiobo, followed by Mbiama, Akinima, and Oshiobebe, reflecting proximity to intensive agricultural activities. Organochlorine pesticides, despite regulatory restrictions, were detected at all stations, highlighting continued environmental persistence or possible illegal application.

Pesticide residues in sediments

Sixteen pesticide residues were detected in sediment samples (Table 2), with total concentrations ranging from 1.422 ± 0.284 to 4.053 ± 0.877 mg/kg.

Sediments contained substantially higher pesticide loads than surface water, confirming their role as long-term contaminant sinks.

The metabolite p,p'-DDE recorded the highest individual concentration (2.233 ± 0.035 mg/kg), accounting for over 50% of total sediment residues at some stations, indicating historical DDT usage. Other dominant sediment contaminants included α -lindane, dieldrin, aldrin, and carbofuran (Table 2). Most detected residues exceeded FAO/WHO sediment guideline values.

Table 2 Concentrations of pesticide residues in sediments from the Orashi River (mg/kg dry weight, mean \pm SD, n = 3)

Pesticide	Odheke	Odiobo	Mbiama	Akinima	Oshiobebe	MRL FAO/WHO
δ -Lindane	0.060 \pm 0.010	0.100 \pm 0.026	0.070 \pm 0.017	0.110 \pm 0.017	0.153 \pm 0.015	0.01
α -Lindane	0.097 \pm 0.006	0.083 \pm 0.012	0.283 \pm 0.050	0.333 \pm 0.025	0.363 \pm 0.006	0.01
p,p'-DDT	0.150 \pm 0.026	0.173 \pm 0.031	0.130 \pm 0.010	0.193 \pm 0.015	0.300 \pm 0.030	0.01
p,p'-DDE	2.233 \pm 0.035	2.167 \pm 0.023	0.053 \pm 0.023	0.107 \pm 0.021	0.120 \pm 0.020	0.01
Dieldrin	0.093 \pm 0.006	0.130 \pm 0.040	0.103 \pm 0.023	0.120 \pm 0.020	0.167 \pm 0.025	0.02
Aldrin	0.063 \pm 0.025	0.063 \pm 0.015	0.050 \pm 0.026	0.067 \pm 0.012	0.067 \pm 0.012	0.02
Carbofuran	0.120 \pm 0.017	0.053 \pm 0.012	0.103 \pm 0.023	0.100 \pm 0.010	0.117 \pm 0.015	0.2

Maximum residues limit (MRL); (CODEX, 2006; WHO, 2009)

The distribution pattern revealed higher sediment contamination at Odieke and Odiobo, mirroring surface water trends and suggesting common pollution sources.

Human health risk assessment

Risk quotient (RQ) analysis identified aldrin, dieldrin, and carbaryl as major contaminants of concern (Table 3). Aldrin exhibited the highest RQ (382.3), followed by dieldrin (185.0) and carbaryl (4.87), all exceeding the safety threshold of unity and indicating significant chronic exposure risk.

Table 3 Risk quotient (RQ) of selected pesticide residues in surface water of the Orashi River

Pesticide	Mean conc. (μ g/L)	Guideline value (μ g/L)	RQ	Risk level
Aldrin	11.47	0.03 (WHO)	382.3	High
Dieldrin	5.55	0.03 (WHO)	185.0	High

Lindane	1.84	2.0 (WHO)	0.92	Low
Dichlorvos	14.67	20 (USEPA)	0.73	Low
Carbaryl	10.22	2.1 (USEPA)	4.87	High
Penconazole	0.97	200 (EFSA)	0.01	Low

Guideline sources: WHO (2009); USEPA (2012); EFSA (2016)

Even though lindane (RQ = 0.92) and dichlorvos (RQ = 0.73) were less than unity, they were close to the regulatory thresholds, and thus there is a possibility that they will represent the risk in the future, in terms of constant environmental loading. The risk was insignificant in penconazole (RQ = 0.01).

These reports show that the untreated surface water in the Orashi River cannot be used in homes and can be extremely unsafe to the people who rely on rivers. Ecological risk index (ERI)

To further assess ecological concerns, a qualitative Ecological Risk Index (ERI) framework was used, depending on the quantity of residue in relation to guideline limits and the toxicity of the compounds. The ecological risk of organochlorine pesticides particularly aldrin and dieldrin was extremely high because of their high level and persistence. The synthetic pyrethroids and carbamates are of medium ecological hazards due to their high acute toxicity to aquatic invertebrates and fish though they are found in lower concentrations. Organophosphorus compounds added significantly to the total amount of contaminants, which means that there is a pressure on aquatic biota.

The presence of both legacy and current-use pesticides implies the existence of cumulative and mixture toxicity, which can enhance sub-lethal consequences like reproduction impairment, enzymatic derailment, and trophic disruption. Residues related to sediment also amplify ecological vulnerability by way of exposure pathways of benthic. Altogether, these results define the Orashi River as an ecologically pressurized system, which demands immediate intervention.

Risk assessment on human health.

Analysis of risk quotient (RQ) indicated that aldrin, dieldrin and carbaryl had very high potential health risks to the people exposed to Orashi River surface water. Aldrin registered the greatest RQ of 382.3, then dieldrin (185.0) and carbaryl (4.87), which is much higher than the toxic limit of unity, and disclosed unacceptable risk level of chronic exposure. These high RQs are largely due to the very low guideline values of persistent organochlorine compounds with their relatively high measured values.

The lindane and dichlorvos had a lower RQ value less than one (0.92 and 0.73, respectively); however, the concentrations are relatively near to the regulatory threshold, and thus there might be a future risk associated with these compounds as more environmental load occurs. The risk of penconazole was insignificant (RQ = 0.01) due to its comparatively large guideline value.

Of special concern is the fact that aldrin and dieldrin are the most prevalent in the risk profile because both are prohibited or highly limited in most countries due to their persistence, bioaccumulative capacity, and carcinogenicity. Being found in concentrations greater than WHO guideline values by more than two orders of magnitude is an indication of continuing illegal use or recycling of history residues in the catchment.

Altogether, the RQ findings suggest that the direct utilization of Orashi River water as the domestic source may be a threat to human health, especially of the children and pregnant women. These results highlight the necessity of the immediate enforcement of regulations, the provision of alternative water, and the interventions on the population health in riverine communities.

IV. DISCUSSION

The current research shows that there is extensive pollution of the Orashi River by various classes of pesticides, which indicates long-term human activities in the region through agricultural activities. Eighteen pesticide residues were detected in surface water and sixteen in sediments with the concentrations ranging from 0.463 ± 0.032 to 67.045 ± 0.143 $\mu\text{g/L}$ and 1.422 to 4.053 mg/kg respectively. They are higher than those measured in a variety of freshwater systems in Nigeria, such as the Ogbesse River (maximum 28.6 $\mu\text{g/L}$; Ezemonye et al., 2015) and Vea Irrigation Reservoir, Ghana (maximum 19.4 $\mu\text{g/L}$; Danladi and Akoto, 2021), indicating that the pressure of contamination in the Orashi River is relatively high.

Sediment profiles were mainly dominated by organochlorine pesticides most especially p,p'-DDE that showed a value of 2.233 ± 0.035 mg/kg indicating the historical use of DDT and its long-term presence. The recalcitrant nature of the DDT metabolites in tropical water-bodies has been confirmed in similar DDE dominance in the sediments of Tono Reservoir, Ghana (Akoto et al., 2016). The high levels of sediment-water partitioning used in the study are in line with earlier observations that the hydrophobic pesticides are mostly deposited

in the benthic matrices making the sediments secondary sources of contamination (Caldas et al., 1999; Akan et al., 2014).

In surface water, organophosphorus pesticides took the highest percentage of the residues detected with dichlorvos taking the highest concentration of $21.873 \pm 0.067 \mu\text{g/L}$. Such concentrations are higher in agricultural catchments across Bangladesh ($\leq 8.3 \mu\text{g/L}$) as represented by Hossain et al., (2015) and southeastern Nigeria ($\leq 12.5 \mu\text{g/L}$) (Asiegbu et al., 2022), which represents intensive local use and high mobility due to its high solubility. The prevalence of organophosphates is in line with their popularity today after many organochlorines were prohibited (Essumang & Chokky, 2009; Awe et al., 2022).

It is worth noting that the concentration of aldrin and dieldrin in surface water were more than 300 times and 180 times respectively, greater than the values of the guideline provided by WHO. Similar excrescences had been observed in the river systems of Nigeria, but of much lower magnitude (Ezemonye et al., 2015; Asiegbu et al., 2022). These extreme levels are highly indicative of either further illicit use or reintroduction of debased soils and sediments due to runoffs.

The analysis of risk quotient revealed that the priority contaminants are aldrin, dieldrin, and carbaryl with the RQ of 382.3, 185.0, and 4.87, respectively. The values are significantly greater than unity and manifest intolerable chronic exposure risk to the populations using untreated river water. High-risk analogs of aldrin and dieldrin have been detected in freshwater systems in Ghana and Nigeria (Akoto et al., 2016; Danladi & Akoto, 2021), highlighting their continued topicality in society.

The pesticides of sediment were always found to be higher in the concentration when compared to water, which further supports their ability to serve as permanent reservoirs of pollutants. The availability of both historic organochlorines and modern organophosphates demonstrates a pattern of contamination transition usually observed in developing agricultural areas, where the remnants of

the past coexist with the new wave of pesticides (Schulz et al., 2021).

Locally, the sources of pesticides are increased in Odieke and Odiobo stations which are associated with increased farming activities and human settlement, indicating localized concentrations. The fact that the presence of several classes of pesticides accumulates is an additional reason to worry about mixture toxicity, which can potentially increase ecological impact over and above what single-compound measurements would suggest.

Generally, the results show that Orashi River has been subjected to constant loading of pesticides, which threaten ecological stress and high hazards to human health. In the absence of urgent measures, chronic disease burden in the reliant communities may be increased by the long-term exposure of the communities by use of domestic water and aquatic food webs.

V. CONCLUSION

This study gives empirical evidence of pesticide contamination in the surface water and sediments of the Orashi River, Niger delta, Nigeria. A large number of both historical organochlorines and contemporary organophosphates were detected indicating long-term agrochemical contributions to the river system. The loads of pesticides in sediments were much greater than those in surface water and supported the role of sediments as long-term reservoirs of contaminants and secondary sources of contamination.

In risk quotient analysis, aldrin, dieldrin, and carbaryl were the priority contaminants, and the value of the contaminants exceeded that of safety by far, which implies that the risk of chronic exposure of river-dependent populations to contaminants is unacceptable. The presence of dichlorvos in the surface water also indicates the continued use of the chemical in agriculture and the high levels of p,p' DDE in the sediments indicate that DDT was used in the past.

All these results show that Orashi River is an ecologically affected system that has potential impacts on aquatic biodiversity and human health. The key measures needed to curb further contamination include immediate regulatory action of the prohibited pesticides, regular environmental inspection, supply of safe sources of alternative water, and encouragement of the integrated pest management to take place. The data developed in this research can be used as a baseline on the environmental management and evidence-based policy interventions to protect freshwater resources in Niger Delta region.

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