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Seasonal Concentrations of Pesticides Residues in Water and Soil Samples from Agricultural Sites in Katsina, Nigeria

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ABSTRACT

This study assessed the seasonal concentrations of pesticide residues in water and soil samples from agricultural sites in Katsina State, Nigeria. Samples were collected during both the rainy and dry season from Daura, Dutsi, Ma'adua, Mani, Mashi, Sandamu, and Zango Local Government Areas (LGAs). Pesticides extraction was performed using QuEChERS methods, and residues were analyzed Gas chromatography-Mass spectrometry (GC-MS). Results revealed the presence of both organochlorine and organophosphate residues in all study locations. Organochlorine compounds such as α-BHC, β-BHC, lindane, aldrin, DDT derivatives and dieldrin were detected in concentrations ranging from 0.045 to 2.36 ppm in water and soil samples, while organophosphates including dichlorvos, carbofuran, butachlor, ethion, and pirimophos-methyl ranged between 0.12 and 3.69 ppm. The highest contamination was recorded in Mani and Mashi LGAs, where butachlor (3.69 ppm) and dieldrin (2.36 ppm) far exceeded the EU maximum residue limits (0.0001 -0.1 ppm) residue levels were generally higher during the rainy season, indicating increased runoff and leaching of pesticides into irrigation water and agricultural soils. The detection of banned organochlorines such as DDT and aldrin highlights persistent contamination and potential ecological and health risks. Continuous monitoring, strict enforcement of pesticide regulations, and farmer education on safe pesticide use are recommended to mitigate future contamination.

Keywords: Pesticide residue, Katsina State, GC-MS, QuEChERS, Organochlorine, Organophosphate, seasonal variation.

INTRODUCTION

Pesticides are indispensable tools in modern agriculture because they help increase crop productivity and prevent yield loses caused by pests, diseases, and weeds [1]. However, excessive and indiscriminate application of pesticides has led to widespread contamination of environmental

compartments such as soil and water, thereby posing risks to human health and ecological systems [2, 3].

In developing countries like Nigeria, the misuse of agrochemicals- especially by smallholder farmers has been associated with contamination of agricultural soils, irrigation waters, and food crops [4]. In northern Nigeria, particularly Katsina State, agriculture is largely seasonal and dependent on irrigation systems during the dry season. Farmers cultivate vegetables and grains extensively, applying various classes of pesticides to ensure high yields [4]. Consequently, pesticide residues tend to accumulate in both soil and water bodies surrounding farmlands, such as Ajiwa and Zobe reservoirs, which serves as irrigation sources [5].

Studies have shown that these residues persist across seasons, influenced by environmental factors like rain fall, temperature, evaporation rate, soil texture [2, 6].

Seasonal variation plays a significant role in determining pesticide residue levels. During the wet season, increased surface runoff and leaching transport pesticides from farmlands into nearby water bodies, leading to elevated concentrations in rivers and reservoirs [3]. In contrast, during the dry season, limited rainfall, high evaporation rates, and frequent irrigation practices concentrate pesticide residue in topsoil and stagnant irrigation channels [7]. Thus, seasonal monitoring provides critical insight into the dynamics of pesticides fate and persistence in agro ecosystems [1, 2]

Reliable analytical determination of pesticide residues in environmental samples requires robust and validated methods. The QuEChERS extraction technique, coupled with gas chromatography- mass spectrometry (GC-MS) or liquid chromatography- mass spectrometry (LC-MS), has become the standard approach for multiresidue determination in complex matrices such as soil and water [8]. This method provides high recovery rates and efficiency across a wide range of pesticide classes, including organochlorine, organophosphates, and carbamates, which are commonly used in Nigerian agriculture [9].

In spite increasing awareness, data on the seasonal variations of pesticide residues in water and soil from agricultural sites in katsina remain scarce. Previous localized studies have reported the presence of banned or restricted organochlorine pesticides such as DDT, aldrin, and dieldrin in water and sediments from reservoirs in northern Nigeria [3; 5]. However most of these studies lack comprehensive temporal datasets covering both wet and dry seasons. Therefore, a systematic assessment focusing on seasonal concentration levels of pesticide residues in both soil and water

from selected agricultural sites in Katsina is essential. Such a study will provide valuable information for environmental monitoring, inform policy interventions and guide farmers and regulators on safer pesticide management practices [2; 9],

Therefore, this study aims at assessing the seasonal concentration levels of pesticide residues in soil and water samples from selected agricultural sites in Katsina, Nigeria. The objectives of the study are to identify and quantify the pesticide residues in soil and water samples collected from agricultural sites, compare the seasonal variations (rainy and dry seasons) in pesticide residue concentrations in both soil and water samples, and assess potential environmental and public health implications of detected pesticide residues in the study area.

MATERIAL AND METHODS

Study area

The study was conducted in the North-eastern part of Katsina, located in the Sudan-Sahel ecological zone of north-western Nigeria. Katsina state lies between latitudes 11^{o}07 and 13^{o}23'N and longitudes 6^{o}52' and 9^{o}09E, sharing an international border with the republic of Niger to the north and internal boundaries with Kano, Jigawa, and Zamfara States [10]. The state is divided into 34 local government areas (LGAs), and the North-eastern part encompasses areas such as Daura, Mashi, Mani, Mai'adua, Zango, Sandamu and Dutsi – predominantly agrarian communities where cultivation is intensive and pesticides are widely used.

Sample preparation

Soil samples were collected from selected farms during rainy and dry seasons. Soil sample points were randomly selected at 0–20 cm depths in each sampling site, because soils at this depth are expected to be more contaminated. The samples were collected using a stainless steel trowel, then wrapped carefully in aluminum foil sheets, clearly labeled, sealed in closed air plastic bags and delivered to analytical laboratory where the samples were well kept at the 20 ^{o}C prior to extraction and subsequent analysis.

Water samples were collected from selected farms in the study area.

Extraction

In this study, the QuEChERS extraction techniques were used for the extraction and cleanup of the samples. QuEChERS extraction method is one of the latest extraction and clean up techniques for pesticide residue analysis in sample matrices which is a short form for Quick, Easy, Cheap,

Effective, Rugged and Safe. A representative 10 g of thoroughly homogenized prepared sample was weighted in a 50 mL polypropylene centrifuge tube. Then 10 mL of acetonitrile was added into the centrifuge tube. The centrifuge tube was then closed properly and shaken vigorously for 30 s by the use of a vortex mixer. Then, 4 g of anhydrous $MgSO_4$ and 1 g of NaCl was added into the centrifuge tube, and shaken immediately by the vortex mixer for 1 minute to prevent the formation of magnesium sulfate aggregates. Afterwards, the extract was centrifuged for 5 min at 5000 rpm. An aliquot of 3 mL of the acetonitrile layer was transferred into a 15 mL micro centrifuge tube containing 600 mg anhydrous $MgSO_4$ and 120 mg Primary Secondary Amine (PSA). Then it was thoroughly mixed by vortex for 30 s and centrifuged for 5 minutes at 4000 rpm. After centrifuge, a 1 mL supernatant was filtered by a 0.2 μm polytetrafluroethylene (PTFE) filter, and then taken in a clean GC vial for injection.

For the water sample, 10 ml of the sample was mixed 10 ml of acetonitrile and the tube vortex for 30 s to mix the water and acetonitrile very well. Anhydrous $MgSO_4$ (4 g) and 1g of NaCl were added, the tube closed and shaken very well. Then the tube was put in the centrifuge and spin at 400rpm for 5minutes, and two layers were formed. The top layer which is the acetonitrile contains the pesticide, while the bottom layer contains water and salts. The acetonitrile layer (6 mL) was pipetted and transferred to 10 ml tube. Then 1 ml of the acetonitrile extract was taken and added into a small tube containing 150 mg $MgSO_4$ and 50 mg PSA vortex for 30 seconds and then centrifuged for 5 minutes to get a clear upper part which is the cleaned extract. This was evaporated to dryness and re-dissolved in hexane before transferring into vial for GC-MS analysis.

GC-MS detection of the pesticide residues

The extracts were analyzed for pesticide residues using a Shimadzu Gas chromatography-mass spectrometer (GCMS-QP2010 SE) equipped with an AOC-20i auto –injector. The GC-MS combines the high separation capability of gas chromatography with the selective detection and identification power of mass spectrometry, making it ideal for multi-residue pesticide analysis. Following QuEChERS extraction and clean-up, 1 μm of the purified extract was injected into the GC at an inlet temperature of 250 ^oC. The sample was vaporized and transported through Rxi5sil MS capillary column (30 m x 0.25 mm. i.d 0.25 μm film thickness) using helium as the carrier gas at a constant flow rate of 1.0 ml/min. The oven temperature was optimized for efficient separation of multiple pesticide residues as follows: initial temperature at 70 ^oC (held for 2 min) ramped to

180 ^{o}C at 25 ^{o}C/min, then to 280 ^{o}C at 5 ^{o}C/min, and held for 10 min. This temperature program allowed effective separation of both organochlorine and organophosphate pesticides.

RESULTS AND DISCUSSION

Tables 1 to 14 show rainy season and dry season concentrations of both organochlorine and organophosphate pesticide residues in water samples from selected farms in the study area. Tables 15 to 27 show the rainy season and dry season concentrations of both organochlorine and organophosphate pesticide residues in soil sample from selected farms in the study area.

Table 1: Rainy and dry seasons concentrations of organochlorine pesticides in water used in irrigation in Daura LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL IN PPM (EU)
a-BHC	0.04613	0.04599	0.00010
b-BHC	0.07989	0.07688	0.00010
Lindane	0.06525	0.06506	0.00010
d-BHC	ND	ND	
Heptachlor	ND	ND	
Aldrin	0.05599	0.05198	0.00010
Heptachlorepoxide	ND	ND	
Gama-chlordane	ND	ND	
a-chlordane	ND	ND	
P-P'-DDE	ND	ND	
Dieldrin	ND	ND	
Endrin	ND	ND	
Endosulfan 11	ND	ND	
P-P' DDD	0.07595	0.07554	0.00010
Endrin aldehyde	ND	ND	
P-P' DDT	ND	ND	
Endrin ketone	ND	ND	

Table 2: Rainy and dry seasons concentrations of organophosphate pesticides in water used in irrigation in Daura LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION (PPM)	DRY SEASON CONCENTRATION (PPM)	MRL
Dichlorvos	0.14093	0.12379	0.00010
Carbofuran	ND	ND	
Diazinone	ND	ND	
Pirimiphos methyl	ND	ND	
Bromophos-ethyl	ND	ND	
Butaclor	ND	ND	
Ethion	ND	ND	
Permethrine	0.10992	0.09655	0.00010

Table 3: Rainy and dry seasons concentrations of organchlorine pesticides in water used in irrigation in Dutsi LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION (PPM)	DRY SEASON CONCENTRATION (PPM)	MRL
a-BHC	0.04631	0.03871	0.00010
b-BHC	ND	ND	
Lindane	ND	ND	
d-BHC	ND	ND	
Heptachlor	ND	ND	
Aldrin	ND	ND	
Heptachlorepoxide	ND	ND	
Gama-chlordane	ND	ND	
a-chlordane	ND	ND	
P-P'-DDE	ND	ND	

Dieldrin	ND	ND	
Endrin	ND	ND	
Endosulfan 11	ND	ND	
P-P' DDD	0.07595	0.07332	0.00010
Endrin aldehyde	ND	ND	
P-P' DDT	ND	ND	

Table 4: Rainy and dry seasons concentrations of organophosphate pesticides in water used in irrigation in Dutsi LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.13674	0.13507	0.00010
Carbofuran	0.29021	0.27850	0.00010
Diazinone	ND	ND	
Pirimiphos methyl	0.25053	0.24699	0.00010
Bromophos-ethyl	ND	ND	
Butaclor	ND	ND	
Ethion	ND	ND	
Permethrine	ND	ND	

Table 5: Rainy and dry seasons concentrations of organchlorine pesticides in water used in irrigation in Maiadua LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
a-BHC	0.04707	0.04599	0.00010
b-BHC	0.09887	0.07952	0.00010
Lindane	0.0665	0.06383	0.00010
d-BHC	0.12639	0.11117	0.00010
Heptachlor	ND	ND	
Aldrin	0.10229	0.21043	0.00010
Heptachlorepoxide	ND	ND	
Gama-chlordane	ND	ND	
a-chlordane	0.07396	0.07184	0.00010
P-P'-DDE	ND	ND	
Dieldrin	ND	ND	
Endrin	ND	ND	
Endosulfan 11	ND	ND	
P-P' DDD	0.07942	0.07584	0.00010
Endrin aldehyde	ND	ND	
P-P' DDT	0.15745	0.15182	0.00010
Endrin ketone	ND	ND	

Table 6: Rainy and dry seasons concentrations of organophosphate pesticides in water used in irrigation in Maiadua LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.13642	0.12817	0.00010
Carbofuran	ND	ND	
Diazinone	ND	ND	
Pirimiphos methyl	ND	ND	
Bromophos-ethyl	ND	ND	
Butaclor	0.24758	0.22831	0.00010
Ethion	ND	ND	
Permethrine	ND	N	

Table 7: Rainy and dry seasons concentrations of organchlorine pesticides in water used in irrigation in Mani LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
a-BHC	ND	ND	
b-BHC	0.07971	0.07991	0.00010
Lindane	ND	ND	
d-BHC	ND	ND	
Heptachlor	ND	ND	
Aldrin	ND	ND	
Heptachlorepoxide	ND	ND	
Gama-chlordane	0.07562	0.07374	0.00010
a-chlordane	ND	ND	
P-P'-DDE	ND	ND	
Dieldrin	0.50657	0.09902	0.00010
Endrin	ND	ND	

Endosulfan 11	ND	ND	
P-P' DDD	0.07641	0.07586	0.00010
Endrin aldehyde	ND	ND	
P-P' DDT	ND	ND	
Endrin ketone	ND	ND	

Table 8: Rainy and dry seasons concentration of organphosphate pesticides in water used in irrigation in Mani LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.14386	0.13445	0.00010
Carbofuran	ND	ND	
Diazinone	0.27248	0.26961	0.00010
Pirimiphos methyl	ND	ND	
Bromophos-ethyl	0.10662	0.01667	0.00010
Butaclor	3.69162	0.49769	0.00010
Ethion	0.48483	0.38621	0.00010
Permethrine	ND	ND	

Table 9: Rainy and dry seasons concentrations of organchlorine pesticides in water used in irrigation in Mashi LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
a-BHC	0.07507	0.04754	0.00010
b-BHC	0.08225	0.07947	0.00010
Lindane	0.08514	0.07654	0.00010
d-BHC	0.13808	0.12477	0.00010
Heptachlor	ND	ND	

Aldrin	ND	ND	
Heptachlorepoxide	ND	ND	
Gama-chlordane	ND	ND	
a-chlordane	ND	ND	
P-P'-DDE	ND	ND	
Dieldrin	0.5580	0.02582	0.00010
Endrin	ND	ND	
Endosulfan 11	0.06713	ND	0.00010
P-P' DDD	0.07904	O.07549	0.00010
Endrin aldehyde	ND	ND	
P-P' DDT	0.16027	0.15205	0.00010
Endrin ketone	ND	ND	

Table 10: Rainy and dry seasons concentrations of organphosphate pesticides in water used in irrigation in Mashi LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.13651	0.13466	0.00010
Carbofuran	ND	ND	
Diazinone	ND	0.27140	
Pirimiphos methyl	ND	ND	
Bromophos-ethyl	ND	ND	
Butaclor	0.24778	0.14387	0.00010
Ethion	ND	ND	
Permethrine	ND	ND	

Table 11: Rainy and dry seasons concentrations of organchlorine pesticides in water used in irrigation in Sandamu LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
a-BHC	0.04703	0.04431	0.00010
b-BHC	0.08048	0.07449	0.00010
Lindane	0.06583	0.06262	0.00010
d-BHC	ND	ND	
Heptachlor	ND	ND	
Aldrin	ND	ND	
Heptachlorepoxide	ND	ND	
Gama-chlordane	ND	ND	
a-chlordane	0.07375	0.07179	0.00010
P-P'-DDE	ND	ND	
Dieldrin	ND	ND	
Endrin	ND	ND	
Endosulfan 11	ND	ND	
P-P' DDD	ND	ND	
Endrin aldehyde	0.53498	ND	0.00010
P-P' DDT	ND	ND	

Table 12: Rainy and dry seasons concentrations of organphosphate pesticides in water used in irrigation in Sandamu LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.15891	0.12628	0.00010
Carbofuran	ND	ND	
Diazinone	ND	ND	
Pirimiphos methyl	ND	ND	
Bromophos-ethyl	ND	ND	
Butaclor	ND	ND	
Ethion	ND	ND	
Permethrine	ND	ND	

Table 13: Rainy and dry seasons concentrations of organchlorine pesticides in water used in irrigation in Zango LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
a-BHC	0.04668	0.04723	0.00010
b-BHC	ND	ND	
Lindane	0.06585	0.07035	0.00010
d-BHC	0.12543	0.12689	0.00010
Heptachlor	ND	ND	
Aldrin	ND	ND	
Heptachlorepoxide	ND	ND	
Gama-chlordane	ND	ND	

a-chlordane	ND	ND	
P-P'-DDE	ND	ND	
Dieldrin	ND	ND	
Endrin	ND	ND	
Endosulfan 11	0.06414	0.06860	0.00010
P-P' DDD	ND	0.07544	0.00010
Endrin aldehyde	ND	ND	
P-P' DDT	ND	ND	
Endrin ketone	ND	ND	

Table 14: Rainy and dry seasons concentrations of organphosphate pesticides in water used in irrigation in Zango LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.15123	0.13474	0.00010
Carbofuran	0.29160	0.28996	0.00010
Diazinone	ND	ND	
Pirimiphos methyl	ND	ND	
Bromophos-ethyl	ND	ND	
Butaclor	0.24778	ND	0.00010
Ethion	ND	ND	
Permethrine	ND	ND	

Table 15: Rainy and dry seasons concentrations of organochlorine pesticides in soil used in planting food crops in Daura LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL IN PPM
a-BHC	0.04639	0.04599	0.1
b-BHC	0.08055	0.07680	0.1
Lindane	ND	ND	
d-BHC	0.12535	0.12453	0.1
Heptachlor	ND	ND	
Aldrin	0.09805	0.05357	0.1
Heptachlorepoxide	ND	ND	
Gama-chlordane	ND	ND	
a-chlordane	ND	ND	
P-P'-DDE	ND	ND	
Dieldrin	ND	ND	
Endrin	ND	ND	
Endosulfan 11	ND	ND	
P-P' DDD	0.07657	0.07556	0.1
Endrin aldehyde	ND	ND	
P-P' DDT	ND	ND	
Endrin ketone	ND	ND	

Table 16: Rainy and dry seasons concentrations of organophosphate pesticides in Soil used in planting food crops in Daura LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.13594	0.12627	0.1
Carbofuran	ND	ND	
Diazinone	ND	ND	
Pirimiphos methyl	ND	ND	
Bromophos-ethyl	ND	ND	
Butaclor	ND	ND	
Ethion	ND	ND	
Permethrine	0.46774	0.07269	0.1

Table 17: Rainy and dry seasons concentrations of organochlorine pesticides in soil used in planting food crops in Dutsi LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION I N PPM	MRL
a-BHC	0.04751	0.03964	0.1
b-BHC	ND	ND	
Lindane	ND	ND	
d-BHC	ND	ND	
Heptachlor	ND	ND	

Aldrin	ND	ND	
Heptachlorepoxide	ND	ND	
Gama-chlordane	ND	ND	
a-chlordane	ND	ND	
P-P'-DDE	ND	ND	
Dieldrin	ND	ND	
Endrin	ND	ND	
Endosulfan 11	ND	ND	
P-P' DDD	0.07646	0.07339	0.1
Endrin aldehyde	ND	ND	
P-P' DDT	0.15214	0.15134	0.1
Endrin ketone	ND	ND	

Table 18: Rainy and dry seasons concentrations of organophosphate pesticides in Soil used in planting food crops in Dutsi LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.13483	0.13466	0.1
Carbofuran	0.28996	0.28876	0.1
Diazinone	ND	ND	
Pirimiphos methyl	0.24640	0.24640	0.1
Bromophos-ethyl	0.07637	0.07187	0.1

Butaclor	ND	ND
Ethion	ND	ND
Permethrine	ND	ND

Table 19: Rainy and dry seasons concentrations of organochlorine pesticides in soil used in planting food crops in Maiadua LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
a-BHC	0.04617	0.04597	0.1
b-BHC	0.08110	0.07950	0.1
Lindane	0.06511	0.06410	0.1
d-BHC	0.12450	0.10761	0.1
Heptachlor	ND	ND	
Aldrin	ND	ND	
Heptachlorepoxide	ND	ND	
Gama-chlordane	0.07499	0.07005	0.1
a-chlordane	ND	ND	
P-P'-DDE	ND	ND	
Dieldrin	ND	ND	
Endrin	ND	ND	
Endosulfan 11	ND	ND	
P-P' DDD	0.07707	0.07547	0.10
Endrin aldehyde	ND	ND	

P-P' DDT	0.15253	0.14602	0.1
Endrin ketone	ND	ND	

Table 20: Rainy and dry seasons concentrations of organophosphate pesticides in Soil used in planting food crops in Maiadua LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.13518	0.13272	0.1
Carbofuran	ND	ND	
Diazinone	ND	ND	
Pirimiphos methyl	0.24716	0.25661	0.1
Bromophos-ethyl	ND	ND	
Butaclor	0.22704	0.22434	0.1
Ethion	ND	ND	
Permethrine	ND	ND	

Table 21: Rainy and dry seasons concentrations of organochlorine pesticides in soil used in planting food crops in Mani LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
a-BHC	N.D	ND	
b-BHC	0.08022	0.07949	0.1
Lindane	ND	ND	
d-BHC	ND	ND	
Heptachlor	ND	ND	
Aldrin	ND	ND	
Heptachlorepoxide	ND	ND	
Gama-chlordane	0.07286	0.07042	0.1
a-chlordane	ND	ND	
P-P'-DDE	ND	ND	
Dieldrin	0.09698	0.06442	0.1
Endrin	ND	ND	
Endosulfan 11	ND	ND	
P-P' DDD	0.07589	0.07443	0.1
Endrin aldehyde	ND	ND	
P-P' DDT	ND	ND	
Endrin ketone	ND	ND	

Table 22: Rainy and dry seasons concentrations of organophosphate pesticides in Soil used in planting food crops in Mani LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.13518	0.12710	0.1
Carbofuran	N.D	ND	
Diazinone	0.27104	0.26961	0.1
Pirimiphos methyl	N.D	ND	
Bromophos-ethyl	0.08257	0.03278	0.1
Butaclor	1.87154	0.20827	0.1
Ethion	0.46459	0.44268	0.1
Permethrine	ND	ND	

Table 23: Rainy and dry seasons concentrations of organochlorine pesticides in soil used in planting food crops in Mashi LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
a-BHC	0.05290	0.04684	0.1
b-BHC	1.89665	0.47358	0.1
Lindane	0.06821	0.09431	0.1
d-BHC	0.17825	0.12463	0.1

Heptachlor	ND	ND	
Aldrin	ND	ND	
Heptachlorepoxide	ND	ND	
Gama-chlordane	ND	ND	
a-chlordane	ND	ND	
P-P'-DDE	ND	ND	
Dieldrin	2.36299	0.16748	0.1
Endrin	ND	ND	
Endosulfan 11	ND	ND	
P-P' DDD	0.63217	0.35476	0.1
Endrin aldehyde	ND	ND	
P-P' DDT	0.54966	0.27863	0.1
Endrin ketone	ND	ND	

Table 24: Rainy and dry seasons concentrations of organophosphate pesticides in soil samples used in planting food crops in Mashi LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.13466	0.13469	0.1
Carbofuran	N.D	ND	
Diazinone	ND	ND	
Pirimiphos methyl	N.D	ND	

Bromophos-ethyl	ND	ND	
Butaclor	0.22838	0.01110	0.1
Ethion	ND	ND	
Permethrine	ND	ND	

Table 25: Rainy and dry seasons concentrations of organochlorine pesticides in soil samples used in planting food crops in Sandamu LGA, Katsina State, Nigeria

PESTICIDES	RAINY SEASON CONCENTRATION (PPM)	DRY SEASON CONCENTRATION (PPM)	MRL
a-BHC	0.04746	0.04594	0.1
b-BHC	0.08107	0.07476	0.1
Lindane	0.06740	0.06339	0.1
d-BHC	0.12578	0.12477	0.1
Heptachlor	N.D	ND	
Aldrin	1.72095	0.39706	0.1
Heptachlorepoxide	N.D	ND	
Gama-chlordane	N.D	ND	
a-chlordane	0.07815	0.07216	0.1
P-P'-DDE	N.D	ND	
Dieldrin	N.D	ND	
Endrin	N.D	ND	
Endosulfan 11	0.06302	0.06246	0.1

P-P' DDD	0.07711	0.07570	0.1
Endrin aldehyde	N.D	ND	
P-P' DDT	N.D	ND	
Endrin ketone	N.D	ND	

Table 26: Rainy and dry seasons concentrations of organophosphate pesticides in Soil used in planting food crops in Sandamu LGA, Katsina State, Nigeria

PESTICIDES	RAINING SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
Dichlorvos	0.13990	0.13666	0.1
Carbofuran	N.D	ND	
Diazinone	N.D	0.27189	0.1
Pirimiphos methyl	N.D	ND	
Bromophos-ethyl	N.D	ND	
Butaclor	0.28457	0.22897	0.1
Ethion	N.D	ND	
Permethrine	N.D	ND	

Table 27: Rainy and dry seasons concentrations of organochlorine pesticides in soil samples used in planting food crops in Zango LGA, Katsina State, Nigeria

PESTICIDES	RAINING SEASON CONCENTRATION IN PPM	DRY SEASON CONCENTRATION IN PPM	MRL
a-BHC	0.04754	0.04631	0.1
b-BHC	0.09734	0.08845	0.1
Lindane	0.08131	0.06834	0.1
d-BHC	0.12689	0.12479	0.1
Heptachlor	N.D	ND	
Aldrin	N.D	ND	
Heptachlorepoxide	N.D	ND	
Gama-chlordane	N.D	ND	
a-chlordane	N.D	ND	
P-P'-DDE	N.D	ND	
Dieldrin	N.D	ND	
Endrin	N.D	ND	
Endosulfan 11	0.07006	0.06699	0.1
P-P' DDD	0.07987	0.07636	0.1
Endrin aldehyde	N.D	ND	
P-P' DDT	N.D	ND	
Endrin ketone	N.D	ND	

Occurrence of pesticide residues in irrigation water

Analysis of irrigation water samples revealed the presence of both organochlorine and organophosphate pesticide residues across all the study locations as shown in Tables 1 to 14.

Organochlorine compounds such as a-BHC, b-BHC, lindane, aldrin, and P-P' DDD were the most frequently detected, with concentrations ranging between 0.045 – 0.16 ppm in most samples. The highest organochlorine concentration (0.56 ppm dieldrin) was observed in Mashi LGA during the rainy season, far exceeding the EU MRL of 0.0001 ppm, indicating significant

contamination. Similar elevated values of aldrin and BHCs were also observed in Mai'adua and Daura LGAs.

Organochlorine levels tended to be higher in the rainy season than in the dry season, likely due to increased surface runoff and leaching of pesticide residues from cultivated lands into irrigation water [7]. However, in some sites notably Mani and Zango LGAs, dry season residues were comparable, suggesting possible concentration effects due to evaporation and low water dilution [6].

Among organophosphates, dichlorvos and carbofuran were the most frequently detected, with dichlorvos concentrations ranging from 0.12 – 0.16 ppm across the sites. Butachlor and ethion were also recorded in Mani and Mai'adua LGAs, with butachlor reaching 3.69 ppm in Mani LGA during the rainy season, which represents severe contamination relative to permissible limits [1, 2]. This indicates heavy pesticide application during the cropping season and improper disposal of agrochemical waste.

Pesticide residues in agricultural soils

Soil samples also showed varying levels of pesticide residues across the locations as shown in Tables 15 to 27.

Organochlorine residues such as a-BHC, b-BHC, DDT derivatives, aldrin, and dieldrin were prevalent in both seasons. The highest concentrations were observed in Mashi and Sandamu LGAs, where dieldrin (2.36 ppm) and aldrin (1.72ppm) levels during the rainy season were substantially higher than EU MRL (0.1 ppm). These elevated values suggest long-term accumulation due to repeated pesticide application and persistence of organochlorine compounds in soil matrices [6].

In most LGAs, residue concentrations decreased slightly during the dry season, possibly due to photodegradation and microbial activity enhanced by higher temperatures [4]. However, certain compounds such as DDT and its metabolites (P-P' DDD, P-P'-DDE) persisted across seasons, reaffirming their environmental stability and bioacculative potentials [5].

For organophosphates residues, dichlorvos, carbofuran, and butachlor were recurrent in several soil samples. Mani and Sandamu LGAs recorded the highest values for butachlor (1.87 ppm and 0.28 ppm, respectively), while ethion concentration (0.46 ppm) in Mani LGA indicated substantial post-application persistence despite its relatively short environmental half-life.

Seasonal variations in organophosphate residues were moderate, reflecting the rapid degradation nature of these compounds relative to organochlorines.

Seasonal and spatial variations

Overall, the study showed clear spatial variability, with Mashi, Mani and Mai'adua LGAs exhibiting higher contamination levels than Dutsi and Zango. The higher residue levels during the rainy season can be attributed recent pesticide application, runoff, and leaching, while lower concentrations during the dry season likely results from volatilization and dilution effects. These finding align with previous reports by Oluwale & Abdulkarim [7], and Abdullahi [5], who observed similar seasonal residue dynamics in northern Nigeria ecosystems.

CONCLUSION

This study assessed the seasonal concentration levels of pesticide residues in water and soil from selected agricultural sites in Katsina State, Nigeria. The aim and objectives of the research were achieved through the identification, quantification, and comparison of organochlorine and organophosphate pesticide residues across rainy and dry seasons using the QuEChERS extraction method coupled with GC-MS analysis. The results demonstrated that both water and soil samples contained pesticide residues, with concentrations ranging from 0.045 to 3.69 ppm, many of which exceeded the EU and WHO maximum residue limits (MRLs). Higher residue levels during the rainy season were attributed to increased surface runoff and leaching, while the persistence of banned organochlorine compounds such as DDT, aldrin, and dieldrin indicated long-term environmental accumulation. The findings highlights that pesticide contamination poses a serious environmental and human health concern in the studied agricultural areas. Continuous exposure through irrigation water and soil may lead to bioaccumulation in food crops and eventual dietary exposure among local populations.

Limitations of the study

Despite its scope, the study was limited by studying only selected organochlorine and organophosphate pesticides, while other classes such as carbamates and pyrethroids were not included due to cost constraints.

Recommendation for future research

Future studies should expand sampling coverage and include more frequent temporal sampling to better understand residue dynamics across planting and fallow periods; incorporate additional

pesticide classes (e.g, carbamates, neonicotinoids, and pyrethroids) to provide a broader contamination profile; evaluate pesticide residues in food crops and sediments to establish possible transfer pathways to human; conduct human health risk assessments and fate modelling to predict long-term ecological and exposure risks and encourage collaboration between regulatory agencies, extension services and local farmers to promote integrated pest management (IPM) and reduce dependence on persistent chemical pesticides

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