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Seasonal Concentration of Pesticides Residues in Rice from Selected Agricultural Sites in Katsina State, Nigeria

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ABSTRACT

Rice is one of the most widely consumed staple foods worldwide and plays a major significant role in food security in Nigeria. However, the increasing use of pesticides in rice cultivation raises a concern about potential contamination of food crops with pesticide residues. This study investigated the seasonal concentration of pesticide residue in rice samples collected from selected agricultural sites in Katsina North, Katsina State, Nigeria. Rice samples were collected during both rainy and dry seasons and analyzed for organochlorine and organophosphate pesticide residues using the QuEChERS extraction method followed by gas chromatography – mass spectrometry (GC-MS). The results showed the presence of several pesticide residues including α-BHC, β-BHC, lindane, aldrine, dieldrin, p,p'-DDD, dichlorvos, butaclor, and carbofuran in varying concentrations were observed during the rainy season compared with dry season. However, all detected pesticide residues were below the maximum residue limits (MRLs) of 0.01 ppm established by international regulatory body the European Union. The findings indicate that although pesticide residues are present in rice cultivated in Katsina North, their concentrations do not exceed recommended safety limits. Continuous monitoring and improved pesticide management practices are nevertheless recommended to minimize potential health risks.

Keywords: GC-MS). QuEChERS, *Oryza sativa*, pesticides, Katsina State, Seasonal variation

Introduction

Rice (*Oryza sativa*) is one of the most widely consumed staple foods globally and serve as a major source of calories for over half of the world's population. In Nigeria rice consumption has increased significantly due to rapid population growth, urbanization, and changing dietary preferences. As a result, rice cultivation has expanded across many agricultural zones, including northern regions of the country where favorable climatic conditions support its production [3]. However, increasing rice production has been accompanied by intensified use of pesticides to control pests that threaten crop yield and quality.

Pesticides, particularly organophosphates and organochlorines, are commonly applied in rice farming to minimize crop losses caused by insects and other agricultural pests. Although these chemicals are effective in pest management, their excessive or improper application may result in the accumulation of pesticide residues in food crops and the environment. Residues of pesticides in agricultural commodities have become a major public health concern because many of these compounds are toxic, persistent, and capable of bioaccumulating in the food chain [1, 2]. Long-term exposure to pesticide residues through contaminated food has been associated with various adverse health effects including neurological disorders, endocrine disruption, and carcinogenic risks.

Seasonal variation is one of the important factors influencing pesticide residue levels in agricultural products. Environmental conditions such as rain fall, temperature, humidity, and sunlight intensity can affect pesticide application rates, degradation, mobility, and persistence in crops and soils. During rainy season, runoff and leaching may redistribute pesticides within agricultural fields, while increased pest pressure often leads to higher pesticide application by farmers. Conversely, higher temperatures and increased photodegradation during the dry season may reduce pesticide persistence in crops [4]. These seasonal differences may therefore result in significant variations in pesticide concentrations detected in rice grains harvested at different times of the year.

In Nigeria, several studies have reported the presence of pesticide residue in food crops such as maize, vegetables, and rice, sometimes exceeding international maximum residues limits (MRLs) established by regulatory bodies such as the European union and Cordex Alimentarius commission [5,7]. Despite these findings, there is still limited information on the seasonal distribution of pesticide residues in rice cultivated in many northern Nigeria agricultural zones, including Katsina State. Katsina North is an important agricultural region where rice cultivation is practiced under both rainy season and dry season irrigation farming systems. The frequent use of pesticides in these systems raises concerns about possible contamination of rice grains consumed by the local population.

Understanding seasonal patterns of pesticide residues in rice is essential for evaluating potential human exposure risks and for developing appropriate strategies to improve pesticide management practices.

Therefore, this study aimed to assess the seasonal concentration of pesticide of pesticide residues in rice collected from selected agricultural sites in katsina north, katsina state Nigeria. The findings of this study will contribute to existing knowledge on pesticide contamination in staple foods and provide scientific data that can support food safety regulation and sustainable agricultural practices in the region.

Materials and Methods

Study area

The study was conducted in Katsina north geopolitical zone of Katsina State Nigeria (Figure 1), 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 [8]. The state is divided into 34 local government areas (LGEAs), and the katsina north 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.

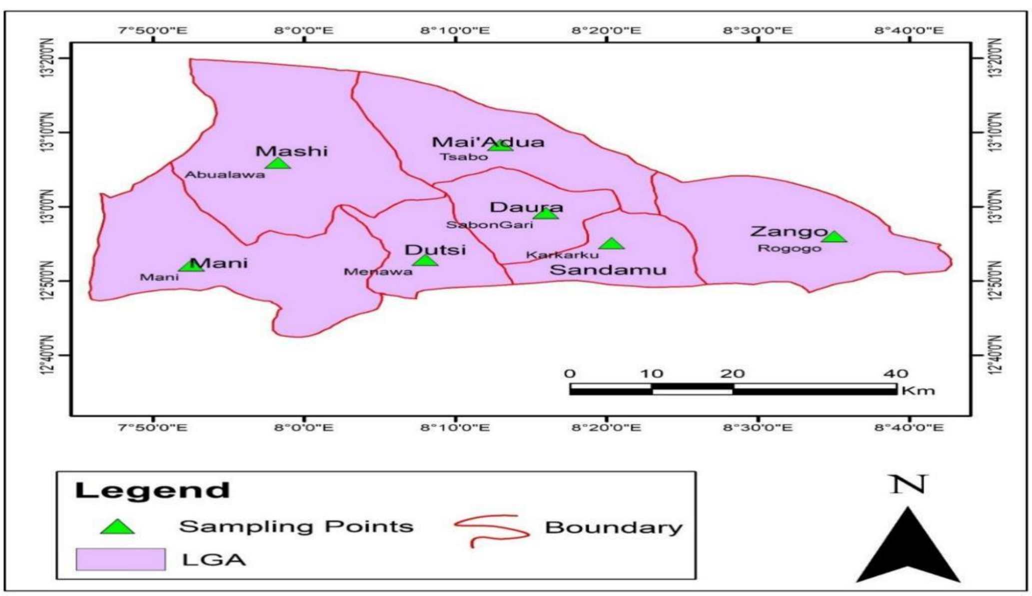

Figure 1: Map of the study area in Katsina State.

Sample preparation

Collected rice grain samples were first cleaned to remove extraneous materials such as stones, dust, and plant debris. The grains were air dried at room temperature and then ground into a fine powder using a laboratory grinder. The homogenized samples were stored in clean polyethylene containers at 4℃ until analysis to prevent degradation of pesticide residues.

Extraction of Pesticide Residues

In this study, the QuEChERS extracted techniques were used for the extraction and cleanup of the samples. QuEChERS extraction method is one of the latest extraction and cleanup techniques for pesticide residue analysis in sample matrices which is a short form for Quick, Easy, Cheap, Effective, Rugged and Safe [2,9]. A representative 10 g of thoroughly homogenized prepared sample was weighed 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[10]. Afterwards, the extract was centrifuged for 5 min at 5000 rpm. An aliquot of 3ml 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 400 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 [2].

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 [3]. Following QuEChERs extraction and clean-up, 1µm of the purified extract was injected into the GC at an inlet temperature of 250℃. The sample was vaporized and transported through Rxi5sil MS capillary column (30m x 0.25mm. i.d 0.25 micrometer film thickness) using helium (99.999% purity) as the carrier gas at a constant flow rate of 1.0ml/min. The oven temperature was optimized for efficient separation of multiple pesticide residues as follows: initial temperature at 70℃ (held for 2 min) ramped to 180℃ at 25℃/min, then to 280℃ at 5℃/min, and held for 10min.Tthis temperature program allowed effective separation of both organochlorine and organophosphate pesticides.

Result and Discussion

Tables 1 to 14 show the rainy and dry seasons mean concentrations of both organochlorine and organophosphate pesticide residues in rice samples from the selected study area.

Table 1: Rainy and dry seasons mean concentrations of organochlorine pesticides in rice samples, collected from Daura LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
a-BHC	0.00405	0.00325	0.01
b-BHC	0.00969	0.00217	0.01
Lindane	0.00558	0.00228	0.01
Aldrin	0.00695	0.00207	0.01
P-P' DDD	0.00654	0.00433	0.01
d-BHC	N.D	N.D	
Heptachlorepoxide	N.D	N.D	
Gama-chlordane	N.D	N.D	

Ppm= parts per million, MRL= maximum residue limit, a-BHC=alpha-benzene hexachloride, b-BHC=beta-benzene hexachloride, d-BHC= delta-benzene hexachloride, P-P' DDD=para, para-dichlorodiphenyldichloroethane.

Table 2: Rainy and dry seasons mean concentrations of organophosphate pesticides in rice samples, collected from Daura LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
Dichlorvos	0.00140	0.00113	0.01
Permethrine	0.00014	0.00021	0.01
Diazinone	N.D	N.D	
Pirimiphos methyl	N.D	N.D	
Bromophos-ethyl	N.D	N.D	
Butaclor	N.D	N.D	
Ethion	N.D	N.D	
Carbofuran	N.D	N.D	

Table 3: Rainy and dry seasons mean concentration of organochlorine pesticides in rice samples, collected from Dutsi LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
a-BHC	0.00463	0.00113	0.01
P-P' DDD	0.00656	0.00211	0.01
b-BHC	N.D	N.D	
d-BHC	N.D	N.D	
Heptachlor	N.D	N.D	
Aldrin	N.D	N.D	
Heptachlorepoxide	N.D	N.D	
Gama-chlordane	N.D	N.D	

Table 4: Rainy and dry seasons concentration of organophosphate pesticides in rice samples, collected from Dutsi LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL
Dichlorvos	0.00154	0.00142	0.01
Carbofuran	0.00016	0.00012	0.01
Pirimiphos methyl	0.00048	0.00027	0.01
Diazinone	N.D	N.D	
Bromophos-ethyl	N.D	N.D	
Butaclor	N.D	N.D	
Ethion	N.D	N.D	
Permethrine	N.D	N.D	

Table 5: Rainy and dry seasons concentration of organchlorine pesticides in rice samples, collected from Maiadua LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
a-BHC	0.00311	0.00221	0.01
b-BHC	0.00598	0.00358	0.01
Lindane	0.00578	0.00108	0.01
d-BHC	0.00245	0.00127	0.01
Aldrin	0.00151	0.00122	0.01
a-chlordane	0.00245	0.00165	0.01
P-P' DDD	0.00268	0.00106	0.01
P-P' DDT	0.00233	0.00127	0.01

Table 6: Rainy and dry seasons concentration of organophosphate pesticides in rice samples, collected from Maiadua LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
Dichlorvos	0.00190	0.00173	0.01
Butaclor	0.00270	0.00133	0.01
Diazinone	N.D	N.D	
Pirimiphos methyl	N.D	N.D	
Bromophos-ethyl	N.D	N.D	
Carbofuran	N.D	N.D	
Ethion	N.D	N.D	
Permethrine	N.D	N.D	

Table 7: Rainy and dry seasons concentration of organchlorine pesticides in rice samples, collected from Mani LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
b-BHC	0.00249	0.00137	0.01
Gama-chlordane	0.00442	0.00212	0.01
Dieldrin	0.00361	0.00221	0.01
P-P' DDD	0.00161	0.00111	0.01
Heptachlor	N.D	N.D	
Aldrin	N.D	N.D	
Heptachlor epoxide	N.D	N.D	
a-BHC	N.D	N.D	

Table 8: Rainy and dry seasons concentration of organophosphate pesticides in rice samples, collected from Mani LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
Dichlorvos	0.00143	0.00118	0.01
Butaclor	0.00183	0.00193	0.01
Diazinone	N.D	N.D	
Pirimiphos methyl	N.D	N.D	
Bromophos-ethyl	N.D	N.D	
Carbofuran	N.D	N.D	
Ethion	N.D	N.D	
Permethrine	N.D	N.D	

Table 9: Rainy and dry seasons concentration of organchlorine pesticides in rice samples, collected from Mashi LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
a-BHC	0.00216	0.00116	0.01
b-BHC	0.00193	0.00113	0.01
Lindane	0.00237	0.00135	0.01
d-BHC	0.00171	0.00101	0.01
Dieldrin	0.00123	0.00093	0.01
P-P' DDD	0.00166	0.00116	0.01
P-P' DDT	0.00157	0.00131	0.01
Gama-chlordane	N.D	N.D	

Table 10: Rainy and dry seasons concentration of organophosphate pesticides in rice samples, collected from Mashi LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
Dichlorvos	0.00171	0.00109	0.01
Butaclor	0.00091	0.00119	0.01
Diazinone	N.D	N.D	
Pirimiphos methyl	N.D	N.D	
Bromophos-ethyl	N.D	N.D	
Carbofuran	N.D	N.D	
Ethion	N.D	N.D	
Permethrine	N.D	N.D	

Table 11: Rainy and dry seasons concentration of organchlorine pesticides in rice samples, collected from Sandamu LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL
a-BHC	0.00174	0.00134	0.01
b-BHC	0.00143	0.00105	0.01
Lindane	0.00108	0.00072	0.01
a-chlordane	0.00116	0.00108	0.01
Heptachlor	N.D	N.D	
Aldrin	N.D	N.D	
Heptachlorepoxide	N.D	N.D	
Gama-chlordane	N.D	N.D	

Table 12: Rainy and dry seasons concentration of organophosphate pesticides in rice samples, collected from Sandamu LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
Dichlorvos	0.00141	0.00127	0.01
Butaclor	0.00139	0.00107	0.01
Diazinone	N.D	N.D	
Pirimiphos methyl	N.D	N.D	
Bromophos-ethyl	N.D	N.D	
Carbofuran	N.D	N.D	
Ethion	N.D	N.D	
Permethrine	N.D	N.D	

Table 13: Rainy and dry seasons concentration of organchlorine pesticides in rice, collected from Zango LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
a-BHC	0.00167	0.00149	0.01
b-BHC	0.00118	0.00102	0.01
Lindane	0.00102	0.00099	0.01
d-BHC	0.00182	0.00124	0.01
a-chlordane	0.00116	N.D	0.01
Endosulfan 11	0.00036	0.00015	0.01
P-P' DDD	0.00126	0.00104	0.01
Gama-chlordane	N.D	N.D	

Table 14: Rainy and dry seasons concentration of organophosphate pesticides in rice, collected from Zango LGA, Katsina State, Nigeria.

Pesticides	Rainy Season Mean Concentration (ppm)	Dry Season Mean Concentration (ppm)	MRL [11]
Dichlorvos	0.00183	0.00143	0.01
Carbofuran	0.00028	0.00014	0.01
Butaclor	0.00225	0.00165	0.01
Pirimiphos methyl	N.D	N.D	
Bromophos-ethyl	N.D	N.D	
Diazinone	N.D	N.D	
Ethion	N.D	N.D	
Permethrine	N.D	N.D	

The result obtained from this study revealed the presence of several organochlorine and αorganophosphate pesticide residues in rice samples collected from selected agricultural sites in Katsina North, Katsina State. However, the detected concentrations across all locations and seasons were generally lower than the maximum residue limits (MRLs) of 0.01 ppm established for these pesticides. This suggests that although pesticide residues are present in rice samples, their levels remain within the acceptable safety limits for human consumption. Similar findings have been reported in previous studies on pesticide residues in staple food crops in Nigeria, where detectable residues were observed but often remain below international safety thresholds of 0.01ppm [5, 7]

A consistent seasonal trend was observed in most of the study locations, where higher pesticide residue concentrations were generally recorded during the rainy season compared to the dry season. For instance, in Daura Local Government Area, organochlorine pesticides such as α-BHC, β-BHC, lindane, aldrin, and p,p'-DDD exhibited higher concentrations during the rainy season than in the dry season . Similarly, organophosphate pesticide such as dichlorvos showed slightly higher concentrations during the rainy season. This pattern suggest that increased pesticide application during the rainy season may contribute to a higher residue levels in rice grains. Farmers often apply pesticides more frequently during this period due to increased pest infection and crop diseases associated with higher humidity and favorable pest breeding conditions [4].

In Dutsi Local Government Area, only a few organochlorine pesticids such as α-BHC and p,p'-DDD were detected, while several other pesticides were not detected in both seasons. A similar pattern was observed for organophosphate pesticides where dichlorvos, carbofuran, and pirimiphos-methyl were detected at very low concentrations. The relatively limited detection of pesticide residues in this area may indicate lower pesticide usage or faster degradation of these compounds in the local environmental conditions. Environmental factors such as sunlight exposure, microbial activity, and soil composition can significantly influence the degradation and persistence of pesticides in agricultural systems [2].

Mai'adua Local Government Area recorded relatively higher diversity of organochlorine pesticide residue s compared to other study locations. Pesticides such as α-BHC, β-BHC, lindane, δ-BHC, aldrin, α-chlordane, p,p'-DDD, and p,p'-DDT were detected in both seasons. Nevertheless, their concentations remained well below the regulatory limits. The presence of DDT deerivatives such as p,p'-DDD suggests possible historical use of organochlorine pesticides in the area, as thses compounds are known for their persistence in soil and agricultural environment for many years and may continue to be detected long after their application has ceased due to their high chemical stability and lipophilic nature [1].

In Mani Local Government Area, organochlorine pesticides including β-BHC, γ-chlordane, dieldrine, and p,p'-DDD were detected in rice samples, with slightly higher concentrations during the rainy season. Similarly, in Mashi Local Government Area, mmutip;e organochlorine residues such as α-BHC, β-BHC, lindane, δ-BHC, dieldrin, p,p'-DDD, and p,p'-DDT were identified. The occurrence of these residues across multiple locations suggests widespread but low –level environmental contamination that may be associated with past agricultural pesticide applications.

Sanndamu and Zango showed detectable levels of organochlorine residues including α-BHC, β-BHC, lindane, and α-chlordanee. In Zango, additional residues such as δ-BHC, endosulfan 11 and p,p'-DDD were identified, although their concentrations remain their concentrations remained extremely low. The presence of endosulfann residues, despite restrictions on its use in many countries, may indicate their historical persistene in the environment or continued informal usage in some agricultural settings.

Conclusions

The use of QuEChERS extraction method combined with GC-MS detection provided a sensitive and reliable approach for identifying trace levels of pesticide residues in rice samples.

The study shows that for organophosphate pesticides, dichlorvos was the most frequently detected compound across nearly all sampling locations. Butachlor was also detected in several locations Mai'adua, Mani, Mashi, Sandamu, and Zango. The widespread detection of dichlorvos may be attributed to its frequent use as an insecticide in agricultural practices due to its effectiveness against a broad range of pests. However, unlike organochlorine pesticides, organophosphates generally degrade more rapidly in the environment, which may explain the relatively low concentrations detected in the rice samples [2]. Another observation from this study is that a number of pesticides were not detected in the analyzed samples. Compounds such as dizinon, pirimophos-methyl, bromophos-ethyl, ethion, and permethrin were absent in many of the samples. This could be attributed to several factors including limited application by farmers, rapid environmental degradation, or concentrations falling below the detection limits of the analytical instrument.

The results indicated that pesticide residues are present in rice cultivated in Katsina North but at concentrations below internationally accepted safety limits. Although the detected levels do not pose immediate health risks based on regulatory standards, continuous monitoring remains necessary due to the cumulative nature of pesticide exposure through food consumption. Long-term dietary exposure to even low levels of pesticide residues may contribute to chronic health effects including neurological disorders, endocrine disruption, and carcinogenic risks [1, 7].

The findings of this study therefore highlight the importance of promoting good agricultural practices, proper pesticide application, and farmer education to minimize pesticide contamination in food crops. In addition, regulatory agencies should strengthen surveillance programs to ensure compliance with pesticide safety guidelines and to protect public health.

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