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Thermal Degradation and Health Risk Assessment of Dieldrin Residues in Grains and Legumes Consumed in Nigeria.

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Abstract- Extensive application of pesticides in agriculture and food crop preservation has resulted in varying values of residual presence of pesticides in stored food products. This study investigated Dieldrin pesticide residue concentrations in legumes, grains and associated wastewater. 18 food crop types were collected from retail markets in Awka, Nigeria between May and August, 2024. They were washed, boiled at different degrees of temperature, extracted, cooled and screened for Dieldrin pesticides present in the boiled food crops and associated waste water after boiling and cooling using Gas Chromatography - Mass Spectrometry. Dieldrin residue was detected in some of the food samples, with concentrations ranging from 0.00 (Not detected) to 0.376 mg/kg. White iron beans had a Dieldrin concentration of 0.258 mg/kg at normal temperature but reduced slightly to 0.213 mg/kg upon heating to 100ºC, highlighting the limited effectiveness of heat in removing Dieldrin residues. A strong positive loading exists among local rice (r = 0.99), yellow corn (r = 1.00), red guinea corn (r = 1.67), pearl millet (r = 1.67), white soft wheat (r = 1.19), and white hard wheat (r = 1.46). The hazard index of Dieldrin in legume followed the decreasing order of Iron white beans (13.10) > brown beans (12.39) > pigeon pea (0.36), implying that Dieldrin in iron white beans and brown beans could cause significant health effects like neurological disorders and cancer. Evidence of the study shows that mere boiling as it is done in Nigeria cannot significantly reduce Dieldrin pesticide concentration in stored food crops. It is therefore recommended that Dieldrin should not be used for food preservation under any circumstance. Natural alternatives like diatomaceous earth and hermetic storage have shown effectiveness with minimal health risk.

Key words: Dieldrin residue; Legumes; Grains; Thermal treatment; Health Risk; Wastewater

I. INTRODUCTION

Human population constantly engages in struggle against competitors and diseases; one way to gain advantage over many of those ecological interactions is through the use of pesticides, which are substances used to protect crop plants, livestock, domestic animals, and people from damage and diseases caused by microorganisms, fungi, insects, rodents, and other pests, and to defend crops from competition with unwanted but abundant weeds. However, pesticide use has become much more common in modern times, and an enormously wider variety of these substances are being used. At least 300 insecticides, 290 herbicides, 165 fungicides, and many other pesticide chemicals are available in more than 3,000 formulations (Handford et al., 2015). Erhunmwunse et al., (2012) described pesticide as a product which consists of formulations of several chemicals; the "active ingredient" which attacks the pest and various "inert ingredients" which enhances its effectiveness. Larger numbers of commercial products are available, because many involve similar formulations manufactured by different manufacturing outfits. The active ingredients in some of them are based on natural biochemicals that are extracted from plants grown for that purpose, while others are inorganic chemicals based on toxic metals or compounds of arsenic (Handford et al., 2015). Most modern pesticides, however, are organic chemicals that have been synthesized by chemists. Unfortunately, the considerable benefits of pesticides are partially offset by damage they cause to ecosystems due and sometimes to human health. Although developing countries account for only about 20% of global pesticide use, they sustain about half of the poisonings (Eddleston et al., 2002). This is because relatively toxic insecticides are used in many developing countries, by a workforce whose widespread illiteracy hinders the understanding of instructions for proper use, and whose safety is further compromised by poor enforcement of regulations and by inadequate use of protective equipment and clothing (Bradberry et al., 2005).

Many inert ingredients in pesticides are, however, biologically active; they are not really passive substances (EPA et al., 2008). Generally, the percentage of other inert ingredients in a pesticide is specified on the product label, but sometimes their identity and concentrations are not given because they are considered to be proprietary information of commercial value (Raimi et al., 2021). Some inert ingredients exhibit risks, hence causes toxicity via normal use of the pesticide (Raimi et al., 2021), few reports show that pesticide usage has increased significantly during the last three decades with consequent changes in farming practices and the increase in intensive agriculture (Francis et al., 2015; Sulaiman et al., 2021). Extensive use of pesticides has resulted in the presence of its residues in various environmental matrices such as food crops, soil, water, domestic and wild animals, proving a possibility of high risk of these chemicals to human health and the environment (Oyeyiola et al., 2017). A particular concern is dieldrin; a synthetic organo-chlorine pesticide derived from the oxidation of aldrin used to control termites mainly on food crops, fruits and vegetables. When sprayed, dieldrin bind tightly to soil and slowly evaporate to the air. Dieldrin is one of the most persistent chlorinated hydrocarbons, and is highly resistant to biodegradation and abiotic degradation. Dieldrin sorbs tightly to soil and sediment, particularly if substantial amounts of organic carbon are present. Dieldrin is toxic to aquatic organisms, birds, and mammals and is capable of producing carcinogenic, teratogenic, and reproductive abnormalities in man (Jorgenson, 2001). Teratogenic effects include cleft palate, webbed foot, and skeletal anomalies. Reproductive effects include decreased fertility, increased fetal death, and effects on gestation. Pesticide residues in agricultural crops are usually monitored with reference to maximum residue limits (MRLs), which represent the highest concentration of pesticide residues that is legally permitted or accepted in food commodities after the use of pesticides (Darko & Akoto, 2008).

The aim of this work is to determine if dieldrin pesticide residue is present in food grains sold in markets in Anambra State, Nigeria and to determine if 'folkloric' use of hot water to wash grains actually reduces pesticides levels, and the percentage reduction effect of boiling and washing with hot water on pesticide residues levels and the dietary intake studies.

II. MATERIALS AND METHODS

2.1: Reagents used

Commercial standard analytical reagents including sodium chloride (NaCl), acetone, methanol, anhydrous magnesium sulphate ($MgSO_4$), gradient grade acetonitrile, primary secondary amine (PSA), sodium sulfate and sulfuric acid were purchased from Chemi-science Nigerian Limited Owerri. They were all within >95% purity.

2.2: Study site

Samples of food crops were collected from Awka, Anambra State, known mostly for its residential, commercial and scant industrial activities. It covers an area of 1,425 km^2 with a density of 760 people per km^2 as seen in Fig.1. The city is located between latitudes 06° 06′ N and 06° 16′ N and longitudes 07° 01′ E and 07° 10′ E.

2.3: Sample Collection

Eighteen food crop samples (local rice, foreign rice, yellow corn, white corn, red guinea corn, white guinea corn, pearl millet, finger millet, red soft wheat, white soft wheat, white hard wheat, white iron beans, oloka beans, brown beans, white soya beans, red soya beans, red pigeon pea and white pigeon pea) were sampled (purchased) from five selected markets (Eke Awka, Nkwo Amaenyi, Ifite second market, Nodu market and Ezinwafor mini market) between May to August, 2024 in Awka, Anambra State. Each grain sample was bought in three separate portions of 1000 g and put in black-colored polyethylene bags, labeled and transported to the laboratory.

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2.4: Chemical analysis

At the laboratory, the eighteen samples were sorted to remove impurities such as stones, shafts and other debris. They were each divided into three (3) portions of 300 g each. The first portion of the food crop (100 g) was thoroughly washed with normal water a 25 ºC, then decanted and the waste water stored in a well labeled plastic bottle (already washed and rinsed with distilled deionized water) while the washed food sample was ground with electric grinder for extraction. The second portion of each food crop sample was washed and then boiled at 50 ºC, decanted and the waste water after boiling kept (well-labeled), and the food samples was ground. The third portion was boiled at 100 ºC, waste water decanted after boiling and the food sample ground. The same procedure was repeated for all the samples.

2.5: Sample extraction and clean up

Extraction of Dieldrin pesticide residue from the foodstuff samples was carried out using the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method as described by Anastassiades et al. (2003) with slight modifications to suit local matrices. A representative portion of each homogenized sample (10.0 ± 0.1 g) was weighed into a 50 mL centrifuge tube. Subsequently, 10 mL of acetonitrile (GC grade) was added as the extraction solvent. The mixture was vortexed vigorously for 1 minute to ensure complete contact between solvent and matrix. A pre-weighed salt mixture containing 4 g anhydrous magnesium sulfate ($MgSO_4$) and 1 g sodium chloride (NaCl) was then added to each tube to induce phase separation. The tube was immediately capped, shaken vigorously for another 1 minute, and centrifuged at 4,000 rpm for 5 minutes.

Cleanup of the extract was achieved by Dispersive Solid-Phase Extraction (d-SPE); an aliquot (6 mL) of the acetonitrile supernatant was transferred into a 15 mL centrifuge tube containing 150 mg primary–secondary amine (PSA), 150 mg C18 sorbent, and 900 mg anhydrous $MgSO_4$ for dispersive solid-phase cleanup. The tube was vortexed for 30 seconds and centrifuged again at 4,000 rpm for 3 minutes. The resulting supernatant (about 2 mL) was filtered through a 0.22 µm PTFE syringe filter into amber GC vials and stored at 4 °C prior to instrumental analysis. All extractions were performed in triplicate, and blank samples (solvent only) were processed simultaneously to monitor potential contamination.

2.5: Dieldrin analysis by GC-MS

An Agilent 7010C Triple Quadrupole Gas Chromatograph–Mass Spectrometer equipped with an auto-sampler was used for the analysis of pesticide residues in the cleaned extracts. A 1.5 mL aliquot of each extract was transferred into pre-cleaned amber GC vials and automatically injected into the GC–MS/MS system, where analytes were vaporized, separated, and subsequently detected.

The chromatographic separation was achieved on an Agilent AT-1 fused-silica capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness). High-purity helium (99.99%) served as the carrier gas at constant flow rate of 1.0 mL/min. The injector was operated in split-less mode with an injection volume of 1.0 µL, and the injector temperature was maintained at 250 °C. The oven temperature program was set as follows: initial temperature 70 °C (held for 2 min), ramped at 25 °C/min to 150 °C, then at 5 °C/min to 280 °C, and finally held for 10 min. The transfer line temperature was maintained at 280 °C.

The mass spectrometer operated in electron ionization (EI) mode at 70 eV, with the ion source temperature at 230 °C and quadrupole temperatures at 150 °C. Data were acquired in Multiple Reaction Monitoring (MRM) mode for high selectivity and sensitivity. For each pesticide, two ion transitions (precursor → product) were monitored; one for quantification and one for confirmation. Collision gas (nitrogen) was used in Q2 for fragmentation of precursor ions.

Analyte identification was confirmed by comparing both the retention times and ion ratios of sample peaks with those of certified reference standards and with mass spectra from the NIST (National Institute of Standards and Technology) 2023 library. Quantification was performed using matrix-matched calibration curves prepared by spiking blank crop extracts with analytical standards at known concentrations to compensate for matrix effects. Final pesticide concentrations were expressed in mg/kg fresh weight.

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2.6: Quality Control/Assurance

Reliable determination of Dieldrin pesticide residues in foodstuff matrix requires rigorous quality assurance (QA) and quality control (QC) procedures to ensure analytical validity and data integrity. Given the complexity of sample matrices and the trace-level concentrations of pesticides, every stage of the analytical workflow, from sample extraction to instrumental quantification, was carefully monitored to prevent contamination, verify instrument performance, and maintain reproducibility. These were achieved through calibration curve development, LOD/LOQ determination, instrument stability; recovery studies using certified matrix spikes etc.

(i) Calibration Curve Development: Six-point calibration curves were generated for each analyte within the range of 0.005–1.000 mg/kg (equivalent to 5–1000 µg/kg). Each calibration level was analyzed in triplicate, and calibration curves were constructed by plotting the mean peak area (y-axis) against the corresponding standard concentration (x-axis).The coefficient of determination (R^2) values obtained for all analytes ranged from 0.992 to 0.999, indicating excellent linearity throughout the working range. Calibration curves were evaluated for slope consistency, intercept significance, and residual deviation; none exceeded 15% of the mean response, confirming compliance with SANTE/12682/2019 performance criteria.

(ii) Limit of Detection/Quantification (LOD/LOQ): The limit of detection (LOD) and limit of quantification (LOQ) were determined based on signal-to-noise (S/N) ratios of 3:1 and 10:1, respectively. The LODs obtained ranged between 0.001 and 0.005 mg/kg, while the LOQs were between 0.003 and 0.015 mg/kg, depending on the compound's ionization efficiency and matrix effect.

(iii) Recoveries and Matrix Effects: Spike-and-recovery experiments were carried out to evaluate the accuracy, precision, and possible matrix effects associated with the analytical determination of all target pesticides. Blank crop matrices confirmed to be free from pesticide contamination were fortified with mixed pesticide standards at three concentration levels representing low, medium, and high spikes (0.01, 0.05, and 0.10 mg/kg). Each fortified sample was extracted and analyzed in triplicate using the validated QuEChERS–GC–MS/MS method under identical conditions as the test samples. The percentage recovery for each analyte was calculated as: $\text{Recovery (\%)} = (C_s / C_a) \times 100$ Where C_s is the measured concentration after spiking and C_a is the known added concentration. The method precision was expressed as relative standard deviation (RSD, %) calculated from the triplicate results.
Mean recoveries for all analyzed pesticides ranged between 81.3 ± 4.2% and 109.8 ± 5.4%, with RSDs below 10%, fulfilling the acceptance range of 70–120% recovery and ≤20% RSD stipulated by SANTE/12682/2019 guidelines.

These measures collectively ensured that the gas chromatography mass spectrometry (GC–MS) method generated results that were accurate, precise and reproducible

2.7: Statistical analysis

This study utilized Microsoft Excel 2016 for its descriptive statistics, and PAST (V.4.03) for the One-Way ANOVA, Pearson correlation, Principal Component and Bray-Curtis Hierarchical analyses. The One-Way ANOVA was conducted to determine whether there is statistically significant difference in Dieldrin concentration across the food types at different temperature treatments. The Bray-Curtis analysis assessed the similarity in Dieldrin levels across the food types. Pearson correlation was performed to evaluate the linear relationship between Dieldrin concentrations in grains and legumes and their associated wastewater. Prior to Principal Component and hierarchical cluster analyses, dataset was standardized. The Shapiro-Wilk's test for Normality showed $p > 0.05$, indicating that all variables follow a normal distribution.

2.8 Health Risk Assessment

Considering health exposure, it is necessary to compare pesticide exposure estimates to established toxicological criteria. Such are achieved through estimation of daily intakes (EDI) and Health Risk index (HI).

2.8.1 Estimation of Daily Intakes

Estimated daily intake was determined using United States Environmental Protection Agency guidelines (US EPA), involving multiplying the concentration of Dieldrin residual level obtained with the food consumption rate (kg/person/day) and dividing by the average adult body weight as stated by equation (1) (Akoto et al., 2015; Oyeyiola et al., 2017; Wang et al., 2011).

$$\text{EDI} = \frac{C_p\ x\ F_R}{Bw} \qquad 1$$

Where EDI is the Estimated Daily Intake, C_p is the concentration of pesticide residue level (mg/kg of food), F_R is the Food consumption rate (kg of food/person/day), and B_w is the Body weight (kg). The average adult body weight of 65 kg was used for the study, while a food consumption rate of 0.33kg for cereals and grains were used for the derivation of the daily intake (WHO, 2017).

2.8.2 Health Risk Index

The health risk for chronic exposure was calculated as ratio between estimated daily intakes (EDI) and World Health Organization's acceptable daily intake (0.0001mg/kg) (Oyeyiola et al., 2017).

$$\text{HI} = \frac{EDI}{ADI} \qquad 2$$

Where ADI is the acceptable daily intake. When HI > 1, it is considered a risk, hence not safe for human health (Donkor et al., 2016).

III. RESULTS AND DISCUSSION

3.1. Pesticide residue concentrations in food crops

The concentrations of Dieldrin pesticides in different food crops studied are shown in Table 1. Dieldrin residue was detected in some of the food samples, with concentrations ranging from 0.00 (not detected) to 0.376 mg/kg. Dieldrin residue concentrations in local rice was 0.030 mg/kg at normal temperature, and it remained constant when boiled at 50ºC, and slightly decreases to 0.027 mg/kg at 100ºC. These concentrations exceeded the maximum residue limit (MRL) of 0.01 mg/kg, indicating a significant health risk from regular consumption (Oyeyiola et al., 2017). Dieldrin residue concentrations in foreign rice were lower (0.013 mg/kg at normal temperature) when compared to local rice, and was similarly unaffected by boiling, showing significant variability in Dieldrin residue concentrations between locally produced rice (Anam) and imported rice (Thailand). Higher concentrations of Dieldrin in local rice might be attributed to differences in farming practices, pesticide application methods, farm processing and regulatory oversight between countries. In contrast, imported rice tends to have lower Dieldrin residue levels, possibly due to stricter enforcement of international Dieldrin residue standards in the countries of origin (Kubiak-Hardiman et al., 2023). The detection of Dieldrin in yellow corn at 0.071 mg/kg shows an alarming level of Dieldrin residue. Despite boiling, only minor reductions were observed, highlighting the attachment and strong binding factor of Dieldrin to food crops. Low levels of D-ieldrin were detected in both red and white guinea corn (0.005–0.007 mg/kg) and pearl millet (0.005 mg/kg). The consistent bonding of Dieldrin to food crops at different temperature range, emphasizes a potential cumulative risk with regular consumption. Notably, white soft wheat showed the highest Dieldrin levels of 0.376 mg/kg, which exceeds the MRL by a significant margin, underscoring concerns about wheat safety in the local diet.

Iron white (Beans) variety exhibited a Dieldrin residue concentration of 0.258 mg/kg, which, like many other crops, exceeds the MRL. Upon regulated heating to 100ºC, it slightly decreased to 0.213 mg/kg; this further highlights the limited effectiveness of heat in removing harmful residues. The detection of Dieldrin in brown beans (0.244 mg/kg) and the non-detection in other varieties suggest variability in Dieldrin application or soil contamination across different legumes. In Pigeon Peas, residues were minimal (0.007 mg/kg), indicating potentially lower exposure risks compared to other legumes. However, the presence of Dieldrin still raises concern about overall dietary safety. Of the eighteen food crops analyzed at different thermal range, Dieldrin residue was not detected in six crops (about 33.3%); white corn, finger millet, oloka beans, white soya beans, red soya beans and white pigeon pea. 38.89% of the food crops had Dieldrin concentrations exceeding the MRL of 0.01 mg/kg, highlighting a potential health risk. In Qatar, 90 % of residues of organochlorines pesticides (OCPs) in vegetables and fruits were above the MRL (Al-Shamary et al., 2016). Further, Dieldrin concentrations in beans (range: 0.21 – 0.25 mg/kg), therefore, within the maximum concentration (0.23 mg/kg) reported in Nigeria (Nwajideobi & Onye, 2025), however, the pesticide was not detected in cocoa beans and pods from Akure-North, Nigeria (Idowu et al., 2022).

3.2 Dieldrin concentrations in wastewater after thermal treatments

From Table 2, the concentrations of Dieldrin in waste water of food crops reduced from 0.007 mg/L (normal temperature) to 0.004 mg/L (100^{o}C) in the Anam local rice, and 0.010 mg/L (normal temperature) to 0.001 mg/L (100^{o}C) in the foreign rice. However, notable increase was observed in the Dieldrin concentrations of Guinea corn (0.001 to 0.011 mg/L) and Millet (pearl) (0.003 to 0.004 mg/L) as the heating progresses to 100^{o}C. The same are true of Wheat (red soft) (0.001 to 0.002 mg/L), Wheat (White soft) (0.005 to 0.007 mg/L), Wheat (White hard) (0.004 to 0.015 mg/L), Beans (Iron white) (0.003 to 0.014 mg/L), and Beans (brown) (0.011 to 0.017 mg/L). Boiling had a limited impact on reducing Dieldrin levels in the food crops. The reduced Dieldrin concentrations in the waste water suggest that minor leaching occurred during boiling, inferring that Dieldrin has strong binding affinity to the food crops, hence the amount in waste water may not constitute environment menace, but on prolonged accumulation will constitute a public health risks. In some food crops, the residue levels remained relatively unchanged. The persistence of Dieldrin across all temperature ranges further confirms its relative heat stability. Dieldrin's stability means it can accumulate in soil and aquatic ecosystems, presenting long-term risks to both environmental and human health (Islam et al., 2022). This aligns with other research indicating that boiling may not effectively reduce pesticide residues in certain cases (Oyeyiola et al., 2017). This observation is crucial as it challenges the folkloric assumption that simple boiling is sufficient to mitigate Dieldrin contamination. Previous studies have shown mixed results regarding the efficacy of boiling; while some pesticides are volatile and can evaporate during cooking, others, particularly those that are persistent and bind tightly to organic matter or soil, remain resistant (Francis et al., 2015).This suggests that boiling may not be a sufficient method for decontaminating food from Dieldrin contamination.

3.3: Analysis of Variance (ANOVA) of Dieldrin in the food crops and waste water.

A one-way ANOVA was conducted to examine the effect of heat on Dieldrin levels of the food crop. Levene's test confirmed that the assumption of homogeneity of variances was met ($F (2, 33) = 0.018$, $p = 0.90$). The ANOVA revealed non-significant difference in Dieldrin residue level between the food crops, $F (2, 33)$, $p = 0.98$ (Table S1). Using Dunn's post hoc comparison, no significant difference was found between the Dieldrin in the food crops boiled at 50^{o}C and normal temperature ($p = 0.51$), between the 100^{o}C and normal temperature (0^{o}C) ($p = 0.39$), or between 100^{o}C and 50^{o}C ($p = 0.83$) (Table S1). The same analysis was conducted for waste water samples; the ANOVA result followed the same trend with food crops earlier discussed and indicated a statistically significant effect of heat on Dieldrin level, $F (2, 33)$, $p = 0.79$ (Table S2).

3.4 Health Risk Assessment

Persistent Diedrin residues can pose chronic health effect; hence, assessment of its health risk is required. The health risks of Dieldrin residue ingestion in the food crops at different heating temperatures (normal room temperature (0^{o}C), 50^{o}C and 100^{o}C) are shown in Tables 3 - 5. Evaluating the health risk of ingestion of Dieldrin residue in the food crop at the normal temperature (Table 3), the grains had hazard index (HI) values ranging from 0.25 to 19.09, with wheat (white soft) showing the highest value (19.09), this is followed by the white hard wheat (10.92). The high hazard index value of Dieldrin in the wheat grains suggested a severe health risk for the consumers, and contrast the low overall risk of pesticide residue in milk types of Tehran (Amininejad & Movassaghghazani, 2025). Other grains with hazard index values > 1 were local rice (Anam) and corn (yellow). However, guinea corn (red & white), millet (pearls), wheat (red soft) and foreign rice (Thailand) had hazard index values < 1, indicating insignificant non-cancer risk. Based on these findings, regular consumption of the Anam local rice (HI: 1.52) will lead to Dieldrin exposure above the acceptable daily intake. A lower risk is expected by consumption of foreign rice (Thailand) with a HI of 0.66; it is also a safer option compared to local rice, but on prolonged consumption, risk may be involved due to accumulation. Yellow corn had a notably high HI of 3.60, which signifies a substantial health risk and continuous consumption could lead to disastrous health effects (Donkor et al., 2016). The hazard index (HI) of Dieldrin in the legume (Table 3) followed the decreasing order of Iron white beans (13.10) > brown beans (12.39) > pigeon pea (0.36), implying that Dieldrin in Iron white beans and brown beans could cause significant health effects like neurological disorders, reproductive effects, endocrine disruption, thyroid issues and birth defects (Ansari et al., 2024). A health risk index > 1 for Dieldrin in brown beans has been reported in Nigeria (Abugu et al., 2023). At 50ºC (Table 4), consuming local rice (HI: 1.52), corn yellow (HI: 3.45), white soft wheat (HI: 18.63) and white hard wheat (HI: 10.71) could cause significant health effects. Further, the Dieldrin in the foreign rice, guinea corn (red & white), millet (pearls) and wheat (red soft) had similar normal temperature hazard index values earlier discussed. Regular consumption of these grains could lead to Dieldrin exposure above the acceptable daily intake. Also, legumes (Table 4), followed the same trend with normal temperature hazard index values. At 100ºC, the Dieldrin residue in Anam local rice (HI: 1.37), corn (yellow) (HI: 3.35), white soft beans (HI: 18.02), white hard beans (HI: 10.71), Iron white beans (HI: 10.81), and brown beans (HI: 10.71) could be described as "risky" since HI > 1. Dieldrin is associated with a range of toxic effects, including carcinogenic, teratogenic, and reproductive impacts (Handford et al., 2015). The presence of Dieldrin in several crops, particularly wheat and beans, underscores potential health risks for the exposed consumers. Chronic exposure could lead to cumulative health effects, particularly for vulnerable populations such as children and pregnant women.

3.5: Principal Component Analysis (PCA)

The PCA of Dieldrin in the food crops and waste water were conducted to explore the dataset underlying structure and reduce its dimensionality while preserving as much variance as possible. In Table 6a and Fig. 2, the analysis of the food crop showed that PC 1 had an eigen value of 2.66, indicating that it accounted for 89 % of the total variance in the standardized data. In contrast, the PC 2 and PC 3 yielded eigen values of 0.34 and 0.00, explaining 11 % and < 1 % of the variance respectively. Based on the Kaiser criterion (Kaiser, 1960), eigenvalues > 1 were utilized for this study, hence, only PC1 qualifies as a significant contributor to data structure, suggesting a strong unidimensional pattern within the dataset. PC 1 had a strong positive loading of local rice (r = 0.99), yellow corn (r = 1.00), red guinea corn (r = 1.67), pearl millet (r = 1.67), white soft wheat (r = 1.19), and white hard wheat (r = 1.46). A negative strong loading was also observed among foreign rice (r = -1.40), white guinea corn (r = - 2.32), red soft wheat (r = - 2.32), and red pigeon pea (r = - 2.32).

Considering the waste water (Table 6b & Fig.3), PC 1 had eigen value > 1, accounting for 68 % of the total variance, with a strong positive association of local rice (r = 1.78) and foreign rice (r = 2.66).

3.6: Hierarchical cluster analysis of Dieldrin contamination

To investigate the similarity in Dieldrin contamination levels across the food types (grains and legumes) and their wastewater, a Bray-Curtis hierarchical cluster analysis was performed as presented in Fig. 5. Four clusters were grouped for the food types based on the clustering algorithm (Fig.5a). The first cluster comprised grains and legume (red pigeon pea, red soft wheat, and white guinea corn), all of which had a correlation of 99 %. The second also comprised grains and legume (local rice and iron white beans) showing a maximum similarity of 98 %. In contrast, the third and fourth clusters only comprised of red guinea corn and pearl millet (99 %), and white soft wheat and yellow corn (99 %) respectively, all of which are grain food. In the waste water (Fig. 5b), three clusters comprising of legumes and grains were formed. The first cluster comprised of white guinea corn and red pigeon pea (90 %), the second: white soft wheat and brown beans (90 %), and the third: yellow corn and iron white beans (82 %). These clustering patterns reflect differences in surface area of the food types or pesticide application practices during storage, hence the need for targeted monitoring of specific food groups in pesticide residue surveillance programs.

3.7: Pearson correlation analysis of Dieldrin concentrations in food matrices and thermal wastewater fractions.

A Pearson correlation analysis was performed to study the inter-relationships between Dieldrin concentrations in legumes, grains, and their associated wastewater fractions collected at normal temperature (0^{o}C), 50^{o}C and 100^{o}C. This was with a view to understand whether thermal treatment influences the distribution of Dieldrin between solid food matrices and their aqueous residues. Looking at Fig. 6 and Table S3, there was a significant strong positive correlation ($p < 0.05$) between Dieldrin concentrations in foreign rice and white guinea corn ($r = 1.0$), foreign rice and red soya beans ($r = 1.0$) and white guinea corn and white hard wheat ($r = 1.0$) amongst others. These results suggest a similar contamination trends across these food types, possibly due to similar storage or pesticide application methods.

Dieldrin concentrations in wastewater at different treatment levels showed a significant strong negative correlation with Anam local rice and iron white beans ($r = -1.0$), foreign rice and red soft wheat ($r = -0.9$) and local rice and soft white wheat ($r = -0.9$). However, significant strong positive correlation was observed among pearl millet and red guinea corn ($r = 1.0$), yellow corn and white soft wheat ($r = 1.0$), white guinea corn and red guinea corn ($r = 1.0$), suggesting that thermal processing, especially at high temperature facilitated the leaching of Dieldrin from food crops into aqueous state, potentially harming the ecosystem through wastewater discharge.

IV. CONCLUSION

Dieldrin concentrations was detected in some of the food samples, with the highest level observed in white soft wheat across different treatment conditions (range: 0.355 to 0.376 mg/kg), this was followed by iron white beans (range: 0.213 to 0.258 mg/kg), and then white hard wheat (range: 0.211 to 0.215 mg/kg). These concentrations exceeded the maximum residue limit (MRL) of 0.01 mg/kg, indicating a significant health risk from regular consumption. Dieldrin residue concentrations in foreign rice were lower (0.013 mg/kg at normal temperature) when compared to local rice, and was similarly unaffected by boiling, showing significant variability in pesticide residue concentrations between locally produced rice (Anam) and imported rice (Thailand). Of the eighteen food crops analyzed, Dieldrin residue was not detected in six crops (about 33.3%); white corn, finger millet, oloka beans, white soya beans, red soya beans and white pigeon pea while 38.89% of the food crops had Dieldrin concentrations exceeding the MRL of 0.01 mg/kg. The ANOVA revealed non-significant difference in Dieldrin residue level between the food crops, $F (2, 33)$, $p = 0.98$. Boiling had a limited impact on reducing Dieldrin levels in the food crops. The slight reduction in dieldrin concentrations in the waste water suggest that minor leaching occurred during boiling, insignificant enough to still damage body organs. Regular consumption of the Anam local rice (Hazard Index: 1.52) will lead to dieldrin exposure above the acceptable daily intake. PC 1 had a strong positive loading of local rice ($r = 0.99$), yellow corn ($r = 1.00$), red guinea corn ($r = 1.67$), pearl millet ($r = 1.67$), white soft wheat ($r = 1.19$), and white hard wheat ($r = 1.46$). Dieldrin concentrations in wastewater at different treatment levels showed a significant strong negative correlation with Anam local rice and iron white beans ($r = -1.0$), foreign rice and red soft wheat ($r = -0.9$) and local rice and soft white wheat ($r = -0.9$). We recommend further research to explore alternative methods for reducing pesticide residues in food crops, and regular monitoring of Dieldrin levels in food crops to identify potential contamination issues.

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Table 1: Concentration of dieldrin in food crops (mg/kg)

FOOD SPECIE	TYPE	Pesticide	Normal Temp. Mean ± SEM	At 50ºC Mean ± SEM	At 100ºC Mean ± SEM	MRL
Grains	Local rice (Anam)	Dieldrin	0.030± 0.06	0.030± 0.21	0.027± 0.01	0.01
	Foreign rice (Thailand)	Dieldrin	0.013± 0.02	0.013± 0.00	0.010± 0.01	0.01
	Corn (yellow)	Dieldrin	0.071± 0.02	0.068± 0.12	0.066± 0.24	0.01
	Corn (white)	Dieldrin	ND	ND	ND	0.01
	Guinea corn (red)	Dieldrin	0.005± 0.05	0.005± 0.00	0.005± 0.11	0.01
	Guinea Corn (white)	Dieldrin	0.007± 0.03	0.005± 0.21	0.005± 0.05	0.01
	Millet (pearl)	Dieldrin	0.005± 0.10	0.005± 0.00	0.005± 0.02	0.01

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	Millet (finger)	Dieldrin	ND	ND	ND	0.01
	Wheat (red soft)	Dieldrin	0.007± 0.15	0.005± 0.00	0.005± 0.11	0.01
	Wheat (White soft)	Dieldrin	0.376± 0.13	0.367± 0.00	0.355± 0.17	0.01
	Wheat (White hard)	Dieldrin	0.215± 0.01	0.211± 0.00	0.211± 0.12	0.01
Legumes	Beans (Iron white)	Dieldrin	0.258± 0.09	0.250± 0.02	0.213± 0.00	0.01
	Beans(oloka)	Dieldrin	ND	ND	ND	0.01
	Beans (Brown)	Dieldrin	0.244± 0.00	0.214± 0.12	0.211± 0.00	0.01
	Soya beans (white)	Dieldrin	ND	ND	ND	0.01
	Soya beans (red)	Dieldrin	ND	ND	ND	0.01
	Pigeon pea(red)	Dieldrin	0.007± 0.06	0.005± 0.15	0.005± 0.25	0.01
	Pigeon pea (white)	Dieldrin	ND	ND	ND	0.01

SEM= Standard error of mean; ND= not detected

Table 2: Concentration of dieldrin in waste water of food crops (mg/L)

FOOD SPECIE	TYPE	Pesticide	Normal Temp. Mean ± SEM	At 50ºC Mean ± SEM	At 100ºC Mean ± SEM
Grains	Local rice (Anam)	Dieldrin	0.007± 0.13	0.005± 0.05	0.004± 0.02
	Foreign rice (Thailand)	Dieldrin	0.010± 0.10	0.002± 0.21	0.001± 0.05
	Corn (yellow)	Dieldrin	ND	0.001± 0.00	0.001± 0.02
	Corn (white)	Dieldrin	ND	ND	ND
	Guinea corn (red)	Dieldrin	ND	ND	0.004± 0.15
	Guinea Corn (white)	Dieldrin	0.001± 0.01	0.001± 0.04	0.011± 0.05
	Millet (pearl)	Dieldrin	0.003± 0.00	0.003± 0.23	0.004± 0.00
	Millet (finger)	Dieldrin	ND	ND	ND
	Wheat (red soft)	Dieldrin	0.001± 0.00	0.002± 0.05	0.002± 0.09
	Wheat (White soft)	Dieldrin	0.005± 0.02	0.007± 0.10	0.007± 0.06
	Wheat (White hard)	Dieldrin	0.004± 0.06	0.005± 0.00	0.015± 0.05
Legumes	Beans (Iron white)	Dieldrin	0.003± 0.01	0.010± 0.08	0.014± 0.04
	Beans(oloka)	Dieldrin	ND	ND	ND
	Beans (Brown)	Dieldrin	0.011± 0.02	0.014± 0.09	0.017± 0.19
	Soya beans (white)	Dieldrin	ND	ND	ND
	Soya beans (red)	Dieldrin	ND	ND	ND
	Pigeon pea (red)	Dieldrin	ND	0.001± 0.26	0.01± 0.02
	Pigeon pea (white)	Dieldrin	ND	ND	ND

SEM= Standard error of the mean; ND= Not detected

Table 3: Health Risk of Dieldrin residue in food crops at normal temperature

FOOD SPECIE	TYPE	Normal Temp Mean ± SEM	EDI X 10^{-3} mg/kg/day	HI	Risk/No risk
Grains	Local rice (Anam)	0.030± 0.06	0.152	1.52	Risk
	Foreign rice (Thailand)	0.013± 0.02	0.066	0.66	No risk
	Corn (yellow)	0.071± 0.02	0.360	3.60	Risk
	Corn (white)	ND	-	-	No risk
	Guinea corn (red)	0.005± 0.05	0.025	0.25	No risk
	Guinea Corn (white)	0.007± 0.03	0.036	0.36	No risk
	Millet (pearl)	0.005± 0.10	0.025	0.25	No risk
	Millet (finger)	ND	-	-	No risk
	Wheat (red soft)	0.007± 0.15	0.036	0.36	No risk
	Wheat (White soft)	0.376± 0.13	1.909	19.09	Risk
	Wheat (White hard)	0.215± 0.01	1.091	10.92	Risk
Legumes	Beans (Iron white)	0.258± 0.09	1.310	13.10	Risk
	Beans(oloka)	ND	-	-	No risk
	Beans (Brown)	0.244± 0.00	1.239	12.39	Risk
	Soya beans (white)	ND	-	-	No risk
	Soya beans (red)	ND	-	-	No risk
	Pigeon pea(red)	0.007± 0.06	0.036	0.36	No risk
	Pigeon pea (white)	ND	-	-	No risk

ND: Not detected

Table 4: Health Risk of Dieldrin residue in food crops at At 50ºC

FOOD SPECIE	TYPE	At 50ºC Mean ± SEM	EDI X 10^{-3} mg/kg/day	HI	Risk/No risk
Grains	Local rice (Anam)	0.030± 0.21	0.152	1.52	Risk
	Foreign rice (Thailand)	0.013± 0.00	0.066	0.66	No risk
	Corn (yellow)	0.068± 0.12	0.345	3.45	Risk
	Corn (white)	ND	-	-	No risk
	Guinea corn (red)	0.005± 0.00	0.025	0.25	No risk
	Guinea Corn (white)	0.005± 0.21	0.025	0.25	No risk
	Millet (pearl)	0.005± 0.00	0.025	0.25	No risk
	Millet (finger)	ND	-	-	No risk
	Wheat (red soft)	0.005± 0.00	0.025	0.25	No risk
	Wheat (White soft)	0.367± 0.00	1.863	18.63	Risk
	Wheat (White hard)	0.211± 0.00	1.071	10.71	Risk
Legumes	Beans (Iron white)	0.250± 0.02	1.269	12.69	Risk
	Beans(oloka)	ND	-	-	No risk
	Beans (Brown)	0.214± 0.12	1.086	10.86	Risk

	Soya beans (white)	ND	-	-	No risk
	Soya beans (red)	ND	-	-	No risk
	Pigeon pea(red)	0.005± 0.15	0.025	0.25	No risk
	Pigeon pea (white)	ND	-	-	No risk

ND: Not detected

Table 5: Health Risk of Dieldrin residue in food crops at At 100ºC

FOOD SPECIE	TYPE	At 100ºC Mean ± SEM	EDI X 10^{-3} mg/kg/day	HI	Risk/No risk
Grains	Local rice (Anam)	0.027± 0.01	0.137	1.37	Risk
	Foreign rice (Thailand)	0.010± 0.01	0.051	0.51	No risk
	Corn (yellow)	0.066± 0.24	0.335	3.35	Risk
	Corn (white)	ND	-	-	No risk
	Guinea corn (red)	0.005± 0.11	0.025	0.25	No risk
	Guinea Corn (white)	0.005± 0.05	0.025	0.25	No risk
	Millet (pearl)	0.005± 0.02	0.025	0.25	No risk
	Millet (finger)	ND	-	-	No risk
	Wheat (red soft)	0.005± 0.11	0.025	0.25	No risk
	Wheat (White soft)	0.355± 0.17	1.802	18.02	Risk
	Wheat (White hard)	0.211± 0.12	1.071	10.71	Risk
Legumes	Beans (Ironwhite)	0.213± 0.00	1.081	10.81	Risk
	Beans(oloka)	ND	-	-	No risk
	Beans (Brown)	0.211± 0.00	1.071	10.71	Risk
	Soya beans (white)	ND	-	-	No risk
	Soya beans (red)	ND	-	-	No risk
	Pigeon pea(red)	0.005± 0.25	0.025	0.25	No risk
	Pigeon pea (white)	ND	-	-	-

ND: Not detected

Table 6: Principal Component Analysis (PCA) of dieldrin in food crops and waste water and their biplots.

A) PCA of dieldrin in food crops (mg/kg)

	PC 1	PC 2	PC 3
LR	0.99	-0.83	-0.01
FR	-1.40	0.22	0.04
YC	1.00	0.07	0.02
RGC	1.67	0.48	-0.02
WGC	-2.32	0.13	-0.02
PM	1.67	0.48	-0.02
RSW	-2.32	0.13	-0.02
WSW	1.19	0.05	0.01
WHW	1.46	0.47	-0.01
IWB	0.28	-1.50	0.00
BB	0.08	0.19	0.06
RPP	-2.32	0.13	-0.02
Eigenvalue	2.66	0.34	0.00
% variance	88.58	11.40	0.02

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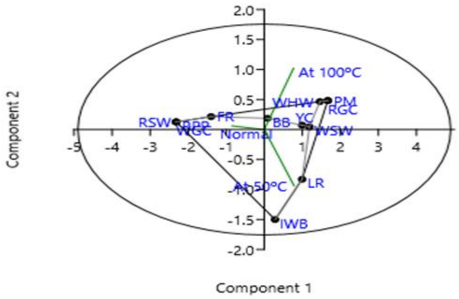


Fig.2: Biplot of dieldrin in food crops (mg/kg)

B) PCA of dieldrin in waste water (mg/L)

	PC 1	PC 2	PC 3
LR	1.78	-0.15	0.15
FR	2.66	-1.82	-0.35
YC	-0.27	1.44	-0.63
RGC	-1.97	-0.99	-0.13
WGC	-1.63	-0.76	0.08
PM	0.61	0.19	0.37
RSW	0.49	0.93	-0.02
WSW	0.78	0.65	0.13
WHW	-0.83	-0.16	0.32
IWB	-0.33	0.83	-0.05
BB	0.53	0.46	0.25
RPP	-1.82	-0.63	-0.10
Eigenvalue	2.04	0.87	0.08
% variance	68.13	29.15	2.73

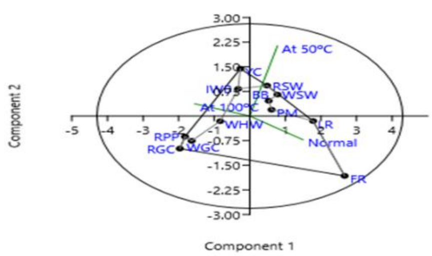


Fig.3: Biplot of dieldrin in waste water (mg/L)

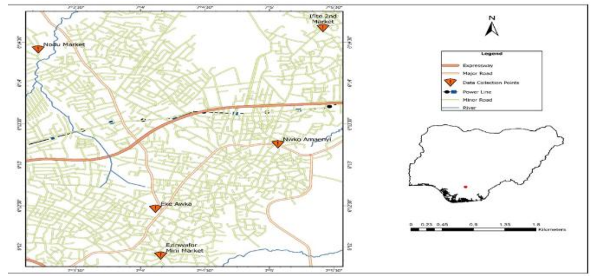
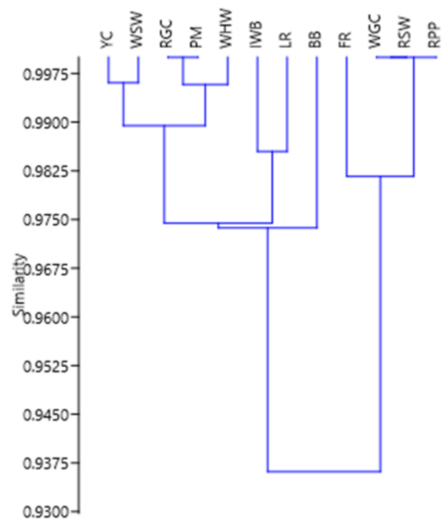
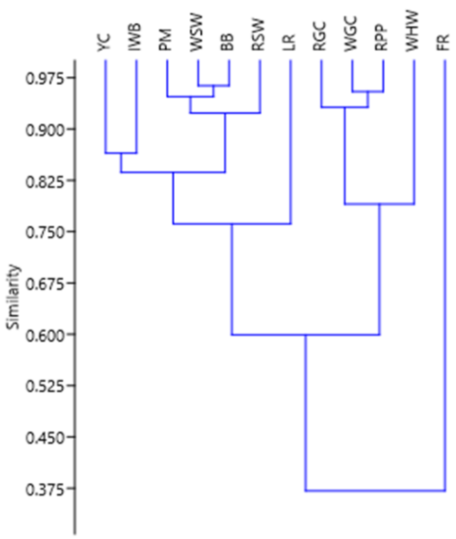


Fig. 1: Map of study area showing sample collection area in Awka Metropolis

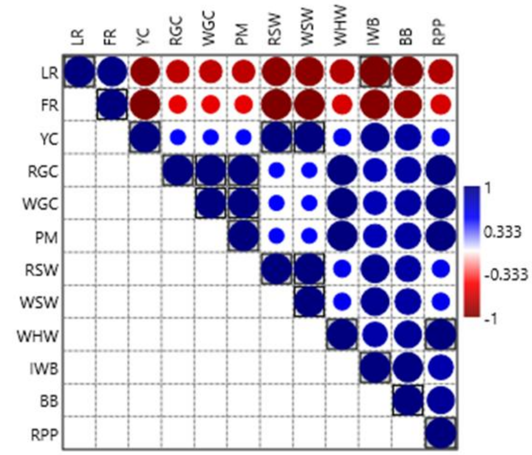


a) Food crops (mg/kg)



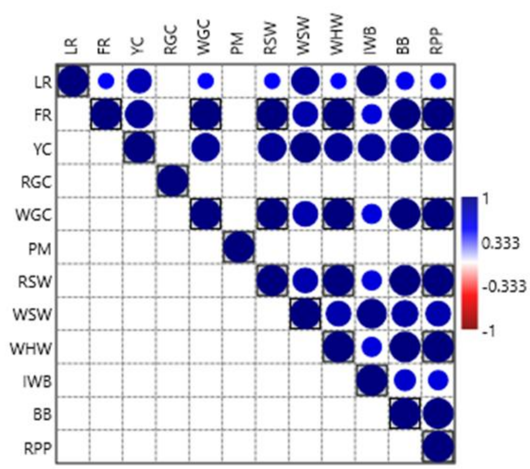
b) Waste water (mg/kg)

Fig.2 : Bray-Curtis hierarchical cluster analysis of dieldrin in the food crops and waste water



Waste water (mg/L)

Fig.3: Pearson Coefficient Correlation of dieldrin in the food crops and waste water. The blue and red colored circles depict the strength of correlation of dieldrin in the matrices. Correlation is significant at p < 0.05 (boxed).



Food crops (mg/kg)