

Determination of Residual Pesticides in Selected Grains, Fruits and Vegetables Commercially Sold in Eke Awka Market, Awka, Anambra State, Nigeria

Dike-Iheanyi Evelyn Ifeyinwa, Ifemeje J.C., Okoye Chinenye Justy, Okoroigwe F.C. and Ezeanyanwu V.C.

Department of Biochemistry, Faculty of Natural Science Chukwuemeka Odumegwu Ojukwu University, Uli, Anambra State, Nigeria

ABSTRACT

Six samples from cucumber (*Cucumis sativus*), apple (*Malus domestica*), cabbage (*Brassica oleracea*), Tiger nuts (*Cyperus esculentus*), Beans (*Phaseolus vulgaris*), Green peas (*Pisum sativum*) were purchased from Eke Awka market, Awka in Anambra state, and were evaluated for their residual pesticides. Estimation of Pesticides residues were carried out using American Public Health Association Method. The result of the pesticide residues analysis showed that all the samples contain pesticide residues but the most prevalent one was Isopropylamine. The pesticide residues in beans have highest concentration of Isopropylamine ($0.094 \pm 0.001\%$), followed by cucumber ($0.058 \pm 0.01\%$) and then apple ($0.040 \pm 0.001\%$) while the least was cabbage ($0.023 \pm 0.001\%$). Among all samples studied, beans have the highest level of pesticide residues (0.245 mg/kg) followed by Apple (0.206 mg/kg) and cucumber (0.178 mg/kg) and least was cabbage (0.121 mg/kg). Among all the pesticides studied, lindane was above maximum residue limit (0.01 mg/kg) in apple and tiger nut ($0.048 \pm 0.001\%$) and $0.043 \pm 0.001\%$ respectively. Conclusion: Pesticide residues in most of the samples analyzed were above limits which portends danger. There is urgent need to for action by policy makers and farmers respectively.

Keywords: Pesticide residue, Grains, Fruits, Vegetables

INTRODUCTION

Pesticides are widely used in modern agriculture to protect crops from pests, diseases, and weeds, thereby enhancing agricultural productivity and ensuring food security [1,2,3]. However, the widespread use of pesticides raises concerns about their potential health and environmental impacts, particularly when pesticide residues remain in food products [4,5]. Residual pesticides in food can pose significant health risks to consumers, ranging from acute poisoning to long-term effects such as cancer, endocrine disruption, reproductive disorders, and neurotoxicity [6,7]. Therefore, monitoring and controlling the levels of pesticide residues in food are crucial for safeguarding public health.

Grains, fruits, and vegetables are vital components of the human diet, providing essential nutrients, vitamins, minerals, and fiber [8]. However, these food items are also among the most frequently contaminated with pesticide residues due to their

extensive exposure during cultivation, storage, and transportation. The levels and types of pesticide residues found in these foods can vary significantly depending on the agricultural practices, the type of pesticide used, and the environmental conditions [9,10]. Monitoring the levels of pesticide residues in food products is particularly important in markets where diverse and often locally sourced food items are sold, such as Eke Awka Market.

Given the extensive use of pesticides in agricultural practices across Nigeria and the limited regulation and monitoring of pesticide residues in food, there is an urgent need to assess the safety of the food products sold in this market [12]. Understanding the levels of pesticide residues in these commonly consumed food items is essential for evaluating potential health risks to consumers and ensuring compliance with international safety standards [13].

This research study aimed to determine the levels of residual pesticides in selected grains, fruits, and vegetables commercially sold in Eke Awka Market. By employing analytical techniques, this study will identify and quantify the presence of various pesticide residues, including Isopropylamin, Adrin, Glyphosphate, Tetradifon, DDT, Dicophol, Endosufan, Carbofuran, Lindane which are among the most commonly used pesticide groups in agricultural practices. The findings of this study will provide valuable data on the safety and quality of these food materials highlighting potential health risks associated with pesticide residues and informing both consumers and policymakers.

Sample collection

The samples for this investigation were purchased at the Eke Awka Market in Awka, Anambra State. To avoid spoiling, the fruits and vegetables were collected in an air-ventilated environment. The samples were separated into three groups. Pesticide residues were grouped in one fraction of the samples, trace metals in the second, and micronutrients, phytochemicals, and bioactive substances in the third.

Estimation of Pesticides Residue in Fruits and Vegetables

This was analyzed according to APHA [14]. This is done in three phases 1, 2 and 3. Phase 1 is the extraction method utilises dichloromethane and hexane After weighing and homogenising the sample, it was combined with 60g of anhydrous sodium sulphate in an agate mortar to absorb moisture. The homogenate was put in a 500ml beaker and extracted with 300ml of n – hexane for 24 hours. The crude extract was evaporated to dryness in a rotating vacuum evaporator at 400°C.

In Phase 2 which is Clean-Up of extract (purification using SPE cartridge), 1 mL filtered residue was dissolved in 50 mL chloroform, transferred to a 100 mL volumetric flask, and diluted to the desired concentration. The majority of the chloroform was evaporated at room temperature, and 1 ml of the reagent (20 vol% benzene, 55 vol% methanol) was added. Sealed and cooked for 10 minutes in a 400°F water bath. After heating, the organic materials were extracted with silicon hexane and water, resulting in a 1:1:1 combination of reagent, hexane, and water (i.e., add 1ml each of hexane and water to the reaction mixture). For 2 minutes, the mixture was violently shaken by hand. If a stable emulsion forms, centrifugation is used to split it up. For injection, about half of the upper hexane phase was transferred to a tiny test tube.

The results of this study could have significant implications for public health, particularly in developing countries where pesticide regulation and enforcement may be less stringent. The data generated could help to raise awareness about the presence of pesticide residues in food, encourage the adoption of safer agricultural practices, and guide the development of effective regulatory policies to protect consumers. Furthermore, this research could serve as a foundation for future studies aimed at monitoring food safety and promoting sustainable agricultural practices that minimize the reliance on chemical pesticides.

METHODOLOGY

Gas Chromatographic Conditions for Pesticide Determination

The final extracts were analysed using a Gas Chromatograph-Buck M910 scientific gas chromatography system with a combipalauto sampler and electron capture detector (ECD), which allowed for the detection of contaminants even at trace levels (in the lower g/g and g/kg range) from the matrix that other detectors did not respond to. The GC conditions employed for the analysis were a capillary column coated with VF-5 (30 m + 10 m EZ guard column x 0.25 mm internal diameter, 0.25 m film thickness) and a capillary column coated with VF-5 (30 m + 10 m EZ guard column x 0.25 mm internal diameter, 0.25 m film thickness). Temperatures for the injector and detector were set at 210°C and 280°C, respectively. The oven temperature was set as follows: 120°C for 4 minutes, then 10°C/min to 180°C for 2 minutes, then 5°C/min to 300°C. Helium was employed as the carrier gas, with a flow rate of 1.0 mL/min, and 29 MI min⁻¹ as the detector make-up gas. The GC injection volume was 1.0 litres. A sample's overall run duration was 31.4 minutes.

Phase 3: Quantification of pesticides residues

Using the external standard technique and peak area, the pesticide residue levels were quantitatively assessed. The detector's linear range was used to conduct the measurements. The concentration was calculated by extrapolating the peak regions whose retention periods matched the standards on their respective calibration curves.

Statistical Analysis

The results were reported as the mean of three replicates plus standard deviation (SD). The Statistical Package for Social Sciences was used to conduct the statistical analysis (SPSS). For comparison, one-way analyses of variance were used, and the findings were submitted to a post hoc test using the least square deviation (LSD). The significance level for all of the findings was set at 0.05.

RESULTS

Pesticides Residues Level

The Pesticide residues level of selected grains, Fruits and Vegetables from Eke Awka market are shown in table 1. The result showed that all the samples analyzed contain pesticide residue. Among all the pesticides studied, Isopropylamine was most prevalent and Endosulfan was less prevalent in grains, Fruits and vegetables studied.

The pesticide residues in beans have highest concentration of Isopropylamine ($0.094 \pm 0.001\%$), followed by cucumber ($0.058 \pm 0.01\%$) and then

apple ($0.040 \pm 0.001\%$) while the least was cabbage ($0.023 \pm 0.001\%$). Among all samples studied, beans has the highest level of pesticide residues (0.245mg/kg) followed by Apple (0.206mg/kg) and cucumber (0.178mg/kg) and least was cabbage (0.121mg/kg). Among all the pesticides studied, lindane was above maximum residue limit (0.01mg/kg) in apple and tiger nut ($0.048 \pm 0.001\%$ and $0.043 \pm 0.001\%$) respectively. The difference in the mean value were significant at $p < 0.05$.

Table 1: Pesticides Residue levels of selected grains, fruits and vegetables sold in Eke Awka market, Awka, Anambra state

Pesticides	Cucumber	Apple	Cabbage	Green peas	Beans	Tiger nuts
Isopropylamin	0.058 ± 0.01	0.040 ± 0.001	0.023 ± 0.001	0.023 ± 0.001	0.094 ± 0.001	0.022 ± 0.001
Adrin	0.008 ± 0.001	0.008 ± 0.001	BDL	BDL	0.010 ± 0.000	0.007 ± 0.001
Pipi- DDD	0.007 ± 0.000	BDL	0.001 ± 0.000	0.001 ± 0.000	0.012 ± 0.001	BDL
Glyphosphate	0.023 ± 0.000	BDL	BDL	BDL	0.025 ± 0.01	BDL
Tetradifon	ND	0.003 ± 0.00	0.002 ± 0.001	0.002 ± 0.001	0.001 ± 0.010	0.003 ± 0.001
DDT	0.016 ± 0.001	BDL	BDL	0.020 ± 0.000	0.03 ± 0.001	BDL
Dicophol	0.053 ± 0.001	0.025 ± 0.001	BDL	BDL	0.055 ± 0.001	0.020 ± 0.00
T-nonaclor	0.013 ± 0.001	BDL	0.001 ± 0.000	BDL	0.018 ± 0.001	BDL
Dichlorobiphenly	BDL	0.028 ± 0.001	0.021 ± 0.001	0.001 ± 0.000	BDL	0.020 ± 0.000
Endosufan	BDL	BDL	0.053 ± 0.001	0.021 ± 0.001	BDL	BDL
Carbofuran	BDL	0.039 ± 0.001	0.053 ± 0.001	0.053 ± 0.001	BDL	0.039 ± 0.00
Lindane	BDL	0.048 ± 0.001	BDL	BDL	BDL	0.043 ± 0.001
HCB	BDL	0.015 ± 0.000	BDL	BDL	BDL	0.014 ± 0.00
g-chlordane	BDL	BDL	BDL	BDL	BDL	BDL
∑ Pesticide residues	0.178	0.206	0.08	0.121	0.245	0.168

Values are mean \pm standard of triplicate determinations

Values within the same bearing the same superscript letters are significantly difference at $P < 0.05$.

DISCUSSION

This study evaluated pesticide residues in six different samples: cucumber (*Cucumis sativus*), apple (*Malus domestica*), cabbage (*Brassica oleracea*), tiger nuts (*Cyperus esculentus*), beans (*Phaseolus vulgaris*), and green peas (*Pisum sativum*). The

findings reveal that all samples contained pesticide residues, with varying concentrations.

The study identified multiple pesticide residues across all samples, with Isopropylamine being the most prevalent pesticide detected. The concentration

of Isopropylamine varied among different samples, with beans having the highest concentration ($0.094 \pm 0.001\%$), followed by cucumber ($0.058 \pm 0.01\%$), apple ($0.040 \pm 0.001\%$), and the least in cabbage ($0.023 \pm 0.001\%$).

The presence of these residues indicates that beans, apples, and cucumbers pose a higher risk for consumers compared to the other tested products.

Lindane another pesticide of concern, was found in concentrations above the maximum residue limit (MRL) of 0.01 mg/kg in both apple ($0.048 \pm 0.001\%$) and tiger nut ($0.043 \pm 0.001\%$). This indicates a significant exceedance of safe levels, particularly in these two samples.

Pesticides like Isopropylamine and lindane are associated with various health risks, including neurological, reproductive, and carcinogenic effects [16,17]. The presence of these residues in concentrations above acceptable limits poses a significant risk to human health. For instance, lindane, which was found above MRLs in apple and tiger nuts, is classified by the World Health Organization (WHO) as a Class II moderately hazardous pesticide. Long-term exposure to lindane can lead to serious health concerns, such as immune and endocrine disruption [18,19].

CONCLUSION

The study indicates that pesticide residues in most samples were above the permissible limits, posing potential health risks to consumers. Beans, apples, and cucumbers were particularly concerning due to their high residue levels. The detection of lindane above MRLs in apples and tiger nuts is alarming and calls for urgent action from policy-makers to enforce stricter pesticide regulations, improve monitoring,

The study indicates that several samples had pesticide residues exceeding the MRLs established by international health organizations such as the WHO and Food and Agriculture Organization (FAO). The exceedance suggests that current agricultural practices and regulatory oversight in the region may be inadequate. The findings highlight the need for stricter enforcement of pesticide regulations, monitoring of pesticide application, and education for farmers on the safe use of pesticides to minimize health risks to consumers. The high concentration of pesticides, especially in beans, apples, and cucumbers, suggests a need for improved agricultural practices, such as integrated pest management (IPM) that focuses on minimizing pesticide use and adopting alternative methods for pest control. Farmers should be trained on the importance of adhering to recommended pesticide dosages, application methods, and pre-harvest intervals to reduce the risk of high pesticide residues. There is a need to raise awareness among consumers regarding the potential health risks of consuming products with high pesticide residues. Encouraging practices such as thoroughly washing fruits and vegetables, buying organic or certified pesticide-free produce, and advocating for more transparent labeling can help mitigate these risks.

and promote safe agricultural practices. Farmers should be educated on the risks associated with improper pesticide use, and consumers should be made aware of the potential hazards of pesticide residues in their food. The results call for immediate intervention to ensure food safety and protect public health in the region.

REFERENCES

1. Prem Rajak, Sumedha Roy, Abhratanu Ganguly, Moutushi Mandi, Anik Dutta, Kanchana Das, Sayantani Nanda, Siddhartha Ghanty, Gopal Biswas, Agricultural pesticides – friends or foes to biosphere?, *Journal of Hazardous Materials Advances*, 2023; 10, 100264. <https://doi.org/10.1016/j.hazadv.2023.100264>.
2. Sharma, A., Kumar, V., Shahzad, B. *et al.* Worldwide pesticide usage and its impacts on ecosystem. *SN Appl. Sci.* 2019; 1, 1446. <https://doi.org/10.1007/s42452-019-1485-1>
3. Pathak VM, Verma VK, Rawat BS, Kaur B, Babu N, Sharma A, Dewali S, Yadav M, Kumari R, Singh S, Mohapatra A, Pandey V, Rana N, Cunill JM. Current status of pesticide effects on environment, human health and it's eco-friendly management as bioremediation: A comprehensive review. *Front Microbiol.* 2022; 13:962619. doi: 10.3389/fmicb.2022.962619. PMID: 36060785; PMCID: PMC9428564.
4. Pathak VM, Verma VK, Rawat BS, Kaur B, Babu N, Sharma A, Dewali S, Yadav M, Kumari R, Singh S, Mohapatra A, Pandey V, Rana N, Cunill JM. Current status of pesticide effects on environment, human health and it's eco-friendly management as bioremediation: A comprehensive review. *Front Microbiol.* 2022; 13:962619. doi: 10.3389/fmicb.2022.962619. PMID: 36060785; PMCID: PMC9428564.
5. Md Faruque Ahmad, Fakhruddin Ali Ahmad, Abdulrahman A. Alsayegh, Md. Zeyauallah, Abdullah M. AlShahrani, Khursheed Muzammil, Abdullah Ali Saati, Shadma Wahab, Ehab Y. Elbendary, Nahla Kambal, Mohamed H. Abdelrahman, Sohail

- Hussain, Pesticides impacts on human health and the environment with their mechanisms of action and possible countermeasures, *Heliyon*, 2024; 10(7): e29128. <https://doi.org/10.1016/j.heliyon.2024.e29128>.
6. Ahmad MF, Ahmad FA, Alsayegh AA, Zeyauallah M, AlShahrani AM, Muzammil K, Saati AA, Wahab S, Elbendary EY, Kambal N, Abdelrahman MH, Hussain S. Pesticides impacts on human health and the environment with their mechanisms of action and possible countermeasures. *Heliyon*. 2024; 10(7):e29128. doi: 10.1016/j.heliyon.2024.e29128. PMID: 38623208; PMCID: PMC11016626.
 7. Pathak VM, Verma VK, Rawat BS, Kaur B, Babu N, Sharma A, Dewali S, Yadav M, Kumari R, Singh S, Mohapatra A, Pandey V, Rana N, Cunill JM. Current status of pesticide effects on environment, human health and it's eco-friendly management as bioremediation: A comprehensive review. *Front Microbiol*. 2022; 13:962619. doi: 10.3389/fmicb.2022.962619. PMID: 36060785; PMCID: PMC9428564.
 8. Liu RH. Health-promoting components of fruits and vegetables in the diet. *Adv Nutr*. 2013;4(3):384S-92S. doi: 10.3945/an.112.003517. PMID: 23674808; PMCID: PMC3650511.
 9. Ssemugabo C, Bradman A, Ssempebwa JC, Sillé F, Guwatudde D. Pesticide Residues in Fresh Fruit and Vegetables from Farm to Fork in the Kampala Metropolitan Area, Uganda. *Environ Health Insights*. 2022; 16:11786302221111866. doi: 10.1177/11786302221111866. PMID: 35846167; PMCID: PMC9277444.
 10. Bajwa U, Sandhu KS. Effect of handling and processing on pesticide residues in food- a review. *J Food Sci Technol*. 2014; 51(2):201-20. doi: 10.1007/s13197-011-0499-5. Epub 2011 Aug 26. PMID: 24493878; PMCID: PMC3907644.
 11. Tudi M, Daniel Ruan H, Wang L, Lyu J, Sadler R, Connell D, Chu C, Phung DT. Agriculture Development, Pesticide Application and Its Impact on the Environment. *Int J Environ Res Public Health*. 2021; 18(3):1112. doi: 10.3390/ijerph18031112. PMID: 33513796; PMCID: PMC7908628.
 12. Ssemugabo C, Bradman A, Ssempebwa JC, Sillé F, Guwatudde D. An assessment of health risks posed by consumption of pesticide residues in fruits and vegetables among residents in the Kampala Metropolitan Area in Uganda. *Int J Food Contam*. 2022;9(1):4. doi: 10.1186/s40550-022-00090-9. Epub 2022 Apr 28. PMID: 35535174; PMCID: PMC9050770.
 13. Adeniyi Adeyeye, Oladele Osibanjo, Residues of organochlorine pesticides in fruits, vegetables and tubers from Nigerian markets, *Science of The Total Environment*, 1999; 231(2-3): 227-233. [https://doi.org/10.1016/S0048-9697\(99\)00067-4](https://doi.org/10.1016/S0048-9697(99)00067-4).
 14. American Public Health Association. Vapour Atomic Absorption Spectrometric Method, Standard Methods for the Examination of Water and Wastewater, 20th Edition, APHA, AWWA, WEF. 1995
 15. Banke I.Y. Concentration of pesticides residues in beans and maize grains in bodija market, Ibadan, Nigeria., www.researchgate.net. 2019
 16. Kaur K, Kaur R. Occupational Pesticide Exposure, Impaired DNA Repair, and Diseases. *Indian J Occup Environ Med*. 2018; 22(2):74-81. doi: 10.4103/ijocem.IJOEM_45_18. PMID: 30319227; PMCID: PMC6176703.
 17. Ahmad MF, Ahmad FA, Alsayegh AA, Zeyauallah M, AlShahrani AM, Muzammil K, Saati AA, Wahab S, Elbendary EY, Kambal N, Abdelrahman MH, Hussain S. Pesticides impacts on human health and the environment with their mechanisms of action and possible countermeasures. *Heliyon*. 2024; 10(7):e29128. doi: 10.1016/j.heliyon.2024.e29128. PMID: 38623208; PMCID: PMC11016626.
 18. Yunjiang Yu, Haibo Chen, Xin Hua, Zhengdong Wang, Liangzhong Li, Zongrui Li, Mingdeng Xiang, Ping Ding, Long-term toxicity of lindane through oxidative stress and cell apoptosis in *Caenorhabditis elegans*, *Environmental Pollution*, 2021; 272,116036. <https://doi.org/10.1016/j.envpol.2020>.
 19. Qi SY, Xu XL, Ma WZ, Deng SL, Lian ZX, Yu K. Effects of Organochlorine Pesticide Residues in Maternal Body on Infants. *Front Endocrinol (Lausanne)*. 2022; 13:890307. doi: 10.3389/fendo.2022.890307. PMID: 35757428; PMCID: PMC9218079.

CITE AS: Dike-Iheanyi Evelyn Ifeyinwa, Ifemeje J.C., Okoye Chinenye Justy, Okoroigwe F.C. and Ezeanyanwu V.C. (2024). Determination of Residual Pesticides in Selected Grains, Fruits and Vegetables Commercially Sold in Eke Awka Market, Awka, Anambra State, Nigeria. IDOSR JOURNAL OF BIOCHEMISTRY, BIOTECHNOLOGY AND ALLIED FIELDS 9(3):1-6. <https://doi.org/10.59298/IDOSR/JBBAF/24/93.16000>