



Research Article

Pesticides Residues in Stored Cowpea (*Vigna unguiculata* L.Walp)  
Grains in Three Markets in Maiduguri, North-Eastern Nigeria

<sup>1</sup>Goniri A. L., <sup>1</sup>Hauwa T., A\*., <sup>1</sup>Umar. A. and <sup>2</sup>Babamai, S.

<sup>1</sup>Department of Zoology, University of Maiduguri Borno State Nigeria

<sup>2</sup>Departments of Veterinary Physiology and Biochemistry, University of Maiduguri, Borno State, Nigeria

\*Corresponding author's Email: [tati66.ng@gmail.com](mailto:tati66.ng@gmail.com), [doi.org/10.55639/607.02010031](https://doi.org/10.55639/607.02010031)

ARTICLE INFO: ABSTRACT

Keywords:

Cowpea,  
Pesticide residues,  
HPLC,  
Organophosphates,  
Pyrethroids

Cowpea (*Vigna unguiculata* L. Walp) is a vital staple crop in Nigeria, serving as a key protein source and economic commodity. However, postharvest losses caused by insect pests, particularly *Callosobruchus maculatus*, have led to widespread pesticide application for storage protection. This study assessed pesticide residues in stored cowpea grains from three major markets in Maiduguri: Muna Garage, Baga Road, and Monday Market. Thirty cowpea samples were randomly collected and analyzed for organochlorine, organophosphate, pyrethroid, and carbamate residues using High-Performance Liquid Chromatography (HPLC). Results indicated the absence of organochlorine and carbamate residues, but pyrethroid (cypermethrin) and organophosphate (DDVP) residues were detected. Cypermethrin residues were found in all study sites, with concentrations ranging from 0.003–0.017 mg/kg, while DDVP residues ranged from 0.003–0.035 mg/kg, with higher concentrations observed at the bottom layers of storage sacks. Some values exceeded the WHO Maximum Residue Limits (MRLs), raising food safety concerns. The findings highlight the need for stricter pesticide regulation, increased awareness among grain traders, and improved cowpea storage management to mitigate health risks associated with pesticide residues.

Corresponding author: Hauwa T., A, Email: [tati66.ng@gmail.com](mailto:tati66.ng@gmail.com)

Department of Zoology, Faculty of Life Sciences, University of Maiduguri, Maiduguri, Nigeria

## INTRODUCTION

Cowpea is also popular crop in Nigeria and other parts of African due to its drought tolerance and high protein content, making it a valuable crop for food security (Popoola *et al.*, 2024). Nigeria is the largest producer and consumer of cowpea accounting for 48% and 46% of production in Africa and World, respectively (IITA, 2024). Borno state is one of the very high production and large markets area of cowpea in Nigeria (Nwagboso *et al.*, 2024). Maiduguri is a major commercial hub in North-Eastern Nigeria where cowpea and other agricultural produce are marketed during harvest and off season. The Muna Garage market, Baga road market and Maiduguri Monday market in Maiduguri serve as the primary trading centers for grains.

One of the primary challenges to cowpea storage in Nigeria is the post-harvest losses caused by storage insect pest, *Callosobruchus maculatus* that commence infestation of cowpea from field to stores where its population build up causes substantial losses. To prevent postharvest loss due to this insect pest, in Northern Nigeria, 99% of wholesale traders treat their cowpea with insecticides to prevent insect infestation in the store and during sales (Akintobi *et al.*, 2018). The application of synthetic insecticides to storage facilities has been studied in different regions of Nigeria. For instance, a study by Fadina *et al.*, (2021) assessed the organochlorine residues in cowpea (*Vigna unguiculata* L. WALP) from selected markets in Ibadan, Oyo State, Nigeria; Ekhuemelo *et al.*, (2021) found significant levels of pesticides residues in cowpea samples obtained from markets in north central Nigeria; Ogah *et al.* (2011) also indicated evidence of organochlorine pesticides residues in maize in Lagos markets; Omokpariola *et al.*, (2023) revealed the concentration levels and risk assessment of organochlorine and organophosphate pesticide residue in selected cereals and legumes sold in Anambra State, south-eastern Nigeria; Asogwa *et al.*, (2023) also analyzed and quantified the levels of

insecticide residues in maize sold in South Eastern Nigeria; Ekhuemelo (2023) evaluated pesticides residues in cowpea grains samples from Plateau State.

These authors reported that several pesticides including, organochlorine, organophosphates, Pyrethroids, carbamide etc were detected in cowpea samples, with some exceeding the maximum residue limits (MRLs) set by regulatory bodies (IUPAC, 2006; Codex Alimentarius, 2022; NAFDAC, 2020).

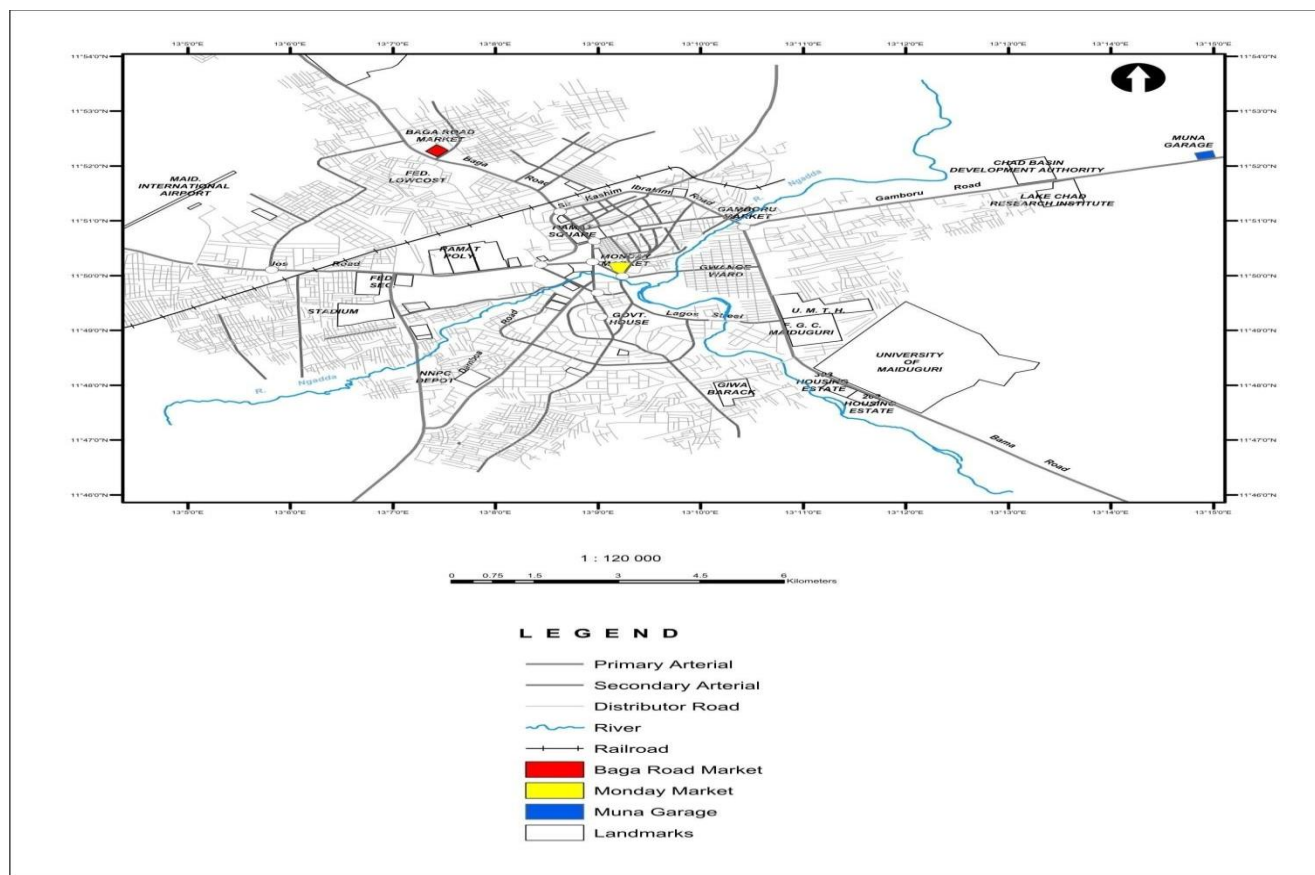
It has been reported that 2.2 million people in Nigerian state are at risk due to exposure from various agricultural inputs such as pesticides and other related chemicals (Almas *et al.*, 2021). These pesticides residues have toxicological implications (Stephen *et al.*, 2011; Adebayo *et al.*, 2023) therefore should be avoided in food intakes for healthy living.

There is little studies on pesticide residues on Cowpea grains in Maiduguri, therefore, the present study was conducted to provide update on the level of pesticides contamination of stored cowpea in Maiduguri to guide policy decision on the marketing and improving food safety and public health in Nigeria.

## MATERIALS AND METHODS

### Study Area and Periods

This study was conducted in three markets in Maiduguri the capital of Borno state, which lies between Latitude 11.83° N and 13.15° E Longitude. The state occupies the greater part of the lake Chad Basin and is in the North Eastern part of Nigeria. It is Located in the Sahel Savannah Vegetation region of north east Nigeria. The climate of Maiduguri and Jere is with a mean annual rainfall and temperature of Maiduguri and Jere Local Government Areas about 650 mm and 32°C, respectively. The months of March and April are the hottest periods of the year with the temperatures ranging between 30°C and 40°C. It is usually cold and dry during the Harmattan months of November to January being the coldest months (BSMI, 2017).



**Figure 1:** Map of Maiduguri and Jere Local Government Areas.

### Sample Collection and Preparation

Thirty (30) samples of two kilograms of stored cowpea grains were randomly collected in triplicate at different levels (top, middle and bottom) of the sacks at the Maiduguri Monday market, Baga Road fish/grain market and Muna Garage grain market (Figure 1) using grain sampler. Untreated cowpea grains were used as control. The collected cowpea grain samples were transported to the Department of Biological Sciences, University of Maiduguri, for identification of varieties, cleaning and storage of the grains before taking to the National Agency for Food and Drug Administration and Control (NAFDAC) laboratory, Maiduguri, for further investigations.

### Extraction/Processing Procedure

A gram portion of each sample was crushed using a pestle and mortar. The powder was weighed into a pre-extracted Whatman extraction thimble. The sample extraction was performed in a Soxhlet extractor for 4 hours using dichloromethane (DCM) as the solvent.

The extract was concentrated by distilling off the solvent (DCM) to about 3 ml using a distillation kit operated at 39-41°C. The concentrated extract was cooled down to room temperature, and then further reduced extract was preserved for chromatographic cleanup prior to chromatographic analysis (AOAC, 2012).

### Extraction of insecticide

The insecticide fractions from the grains were extracted with acetonitrile and methanol (4:1). Powdered seed samples were mixed with 25 ml of acetonitrile and methanol. The mixtures were shaken vigorously and then allowed to stand for one hour. The supernatants were filtered using the Whatman number 4 filter paper. The extracts were transferred into a sterile bottle for chromatography Analysis (Gesald, 2009).

### Cleanup Experiment

For the cleanup experiment, a column of about 15 cm x 1 cm was packed with about 5 grams of activated silica gel prepared in a slurry form in n-3 hexane. About 0.5 cm of anhydrous sodium sulfate was placed at the top of the Column to

absorb any water in the sample or the solvent. The Column was pre-eluted with 15 ml of n-hexane without exposing the sodium sulfate layer to air to prevent drying up and breaking of the adsorbent. The reduced extract was turned into the Column and allowed to migrate below the sodium sulfate layer. Elution was done with 2 x 10 ml portions of the extracting solvent (DCM). The eluate was then collected, dried with anhydrous sodium sulfate and evaporated to dryness under a stream of analytical-grade nitrogen (99.99%).

### **High-Performance Liquid Chromatography (HPLC)**

HPLC was used to separate the components of biological samples or similar synthetic chemicals from each other during the production process of biological products (Gerber *et al.*, 2004). Chromatography can be described as a mass transfer process involving adsorption. Higher-performance liquid chromatography relies on pumps to pass a pressurized liquid and sample mixture through a column filled with a sorbent, leading to the separation of the sample components. The extracted insecticides were analyzed by injecting 1 ml of each extract into bulk scientific Column BLC 10/11 high-performance liquid chromatography with a flame ionization detector. The separate compounds were detected and quantified from eluting chromatographic columns. It should not exceed 1 ml of extract; each grain was pipetted into a small test tube. 3 ml of methanol-acetonitrile was added and swirled to mix. After 1 ml of 0.05 M sodium phosphate buffer was added and swirled to mix. The sample loop of the HPLC was loaded with a 100 µl syringe, and the sample was injected into a column; the peaks that came off the system were recorded and compared to a standard to identify and measure the compounds present (Gesald, 2009). The insecticide residue was identified using reference standards and relative retention time techniques. In contrast, the concentration of the residues was determined by comparing the peak

areas of the samples with the corresponding peak areas of the reference standards of known concentrations (Williams, 2011).

### **Quality control measures**

Quality control measures such as determination of percentage recovery and detection limit were adopted to ensure that the analytical procedures used comply strictly with appropriate standards and specifications and that the results obtained are reliable, valid and of high quality.

### **Statistical Analysis**

The mean values of pesticide residues were computed and compared with standard WHO maximum pesticide residue limit values.

## **RESULTS**

Test for organochlorine (OC), organophosphate (OP), pyrethroids (PY) and carbamate (CA) insecticides in the stored cowpeas was performed, and the following results were obtained. The results showed negativity to OC and CA but positivity to PY and OP as follows:

### **Pyrethroids Residues**

The residues of pyrethroids and cypermethrin were detected in all the study sites (Table 1). The samples collected at Muna Garage Market show that cypermethrin residues were below the WHO limits in samples collected at the storage bags' top (0.011mg/kg) and middle (0.007 mg/kg). However, samples collected at the bottom of the bags have slightly higher limits (0.017mg/kg). The samples collected at Baga Road Market show that residues of cypermethrin were below the WHO limits in samples collected at the top (0.010mg/kg), middle (0.003 mg/kg) and bottom (0.011 mg/kg) of the storage bags. The samples collected at Monday Market show that residues of cypermethrin were below the WHO limits in samples collected at the top (0.016mg/kg), middle (0.012 mg/kg) and bottom (0.015 mg/kg) of the storage bags. Surprisingly, the control untreated samples collected at Muna Garage and Monday market showed negligible limits for cypermethrin concentrations.

**Table 1:** Residues of cypermethrine (PY) in stored Cowpea *Vigna unguiculata* from Maiduguri and Jere Local Government Areas, Borno State

Sampling Sites	Residue concentration (mg/kg) ±S.E				WHO Limit (mg/kg)
	Top	Middle	Bottom	Control	
Muna Garage Market	0.011±0.00363	0.007±0.00363	0.017±0.00363	0.005±0.0002	0.01
Baga Road Market	0.010±0.00363	0.003±0.00363	0.011±0.00363	0.000±0.005	0.01
Monday Market	0.016±0.00363	0.012±0.00363	0.015±0.00363	0.006±0.005	0.01

Each data is a mean value of 10 samples

### Organophosphate Residues

The result indicates the presence of organophosphate residues, DDVP (Dichlorvos 2, 2 Dichlorviny dimethyl Phosphate) in the stored Cowpea samples collected in the three sites (Table 2). The samples collected at Muna Garage Market show that DDVP residues were below the WHO limits in samples collected at the top (0.011 mg/kg), middle (0.003 mg/kg) and bottom (0.019 mg/kg) of the storage bags. The samples collected at Baga Road Market show

that DDVP residues were below the WHO limits in samples collected at the top (0.019 mg/kg), middle (0.014 mg/kg) and bottom (0.030 mg/kg) of the storage bags. The samples collected at Monday Market show that DDVP residues were below the WHO limits in samples collected at the top (0.029 mg/kg), middle (0.007 mg/kg) and bottom (0.035 mg/kg) of the storage bags. The control untreated samples collected at all the sites did not show DDVP pesticide residues.

**Table 2:** Residues of DDVP found in stored Cowpea *Vigna unguiculata* from Maiduguri and Jere Local government areas, Borno State

Sampling Sites	Residue Concentration mg/kg +S.E				WHO Limit (mg/kg)
	Top	Middle	Bottom	Control	
Muna Garage Market	0.011±0.0006	0.003±0.0006	0.019±0.0006	0.000±0.0000	0.02
Baga Road Market	0.019±0.0006	0.014±0.0006	0.030±0.0006	0.000±0.000	0.02
Monday Market	0.029±0.0006	0.007±0.0006	0.035±0.0006	0.000±0.000	0.02

Each data is a mean value of 10 samples

### DISCUSSION

The present study found no residues of organochlorine and carbamate pesticides. This is interesting and indicates that traders and farmers have primarily adhered to the National Agency for Food and Drug Administration and Control (NAFDAC) policy, which banned these chemicals due to their toxicity and their severe health risks. However, detecting pyrethroid (cypermethrin) and organophosphate (DDVP) insecticide residues in the stored cowpea grains remains a cause for concern. When grain merchants were asked to present the samples of the insecticides used in storage, they provided empty cans of 10% EC Cypermethrin (Cypertex©) and DDVP (Dichlorvos 2, 2 Dichlorviny dimethyl Phosphate), 100% EC, Dichlor©. This finding suggests that these

insecticides are still widely used despite concerns over their potential health risks.

A significant factor contributing to the high pesticide residue levels is the lack of awareness and education among local peasant farmers and grain merchants. Many traders are illiterate and unable to read or comprehend the safety precautions and recommended application guidelines on pesticide packaging. Similarly, studies conducted in Dawanus Market, Kano State, found that most individuals involved in the grain trade lacked formal education, making them unaware of the consequences of pesticide misuse (Yusuf *et al.*, 2017). The misuse of pesticides, including overdosing and repeated application, is often motivated by economic concerns—traders aim to protect stored cowpea grains from insect infestation for extended

periods, inadvertently leading to excessive pesticide accumulation.

The detection of cypermethrin and DDVP above WHO safety limits in the lower portion of the storage bags raises significant food safety concerns. While the concentrations in the top and middle layers of the sacks were within acceptable limits, those at the bottom exceeded the WHO Maximum Residue Limits (MRLs), potentially posing a health risk to consumers. This uneven distribution of pesticide residues suggests that heavier chemicals may accumulate at lower levels over time due to gravitational settling and absorption by the grains. Other studies have reported similar findings examining pesticide distribution in stored grains (Kemabonta & Akinhanmi, 2013).

The consumption of pesticide-contaminated cowpea grains may have acute and chronic health consequences. Acute exposure to cypermethrin and DDVP can lead to neurological symptoms such as dizziness, headaches, nausea, and muscle weakness (FAO/WHO, 1986). Dichlorvos (DDVP), an organophosphate, is particularly concerning because it inhibits acetylcholinesterase, leading to cholinergic toxicity, which manifests as excessive salivation, respiratory distress, and, in severe cases, paralysis (Goel & Aggarwal, 2007). Chronic exposure to low doses of organophosphates has been associated with endocrine disruption, developmental disorders, and an increased risk of neurodegenerative diseases such as Parkinson's disease (Kim *et al.*, 2017).

Prolonged ingestion of cypermethrin-contaminated food has also been linked to reproductive toxicity, immune suppression, and liver damage (Gasald, 2009). Studies have demonstrated that long-term exposure to pyrethroids can lead to oxidative stress, increasing the risk of metabolic disorders and cancer (Sharma *et al.*, 2020). In developing countries, where pesticide monitoring is less stringent, foodborne pesticide exposure has been implicated in rising chronic kidney disease and liver dysfunction (Adebayo *et al.*, 2023).

The public health implications of these findings are alarming, as past incidents have demonstrated the dangers of pesticide contamination in food products. For instance, 20

fast-food outlets in Nigeria were shut down after reports linked fatalities to pesticide residues in food (Chikwe, 2010). Furthermore, the European Union (EU) banned Nigerian cowpea exports in 2015 due to excessive pesticide residues, emphasizing the economic consequences of pesticide misuse. High DDVP residues in the cowpea grains examined in this study suggest that the grains may not meet international safety standards, further jeopardizing Nigeria's agricultural export potential.

Given these concerns, enhanced pesticide regulation and enforcement are urgently needed to ensure compliance with safety standards. NAFDAC and other regulatory agencies should strengthen monitoring efforts at markets and warehouses to ensure pesticide applications are within the recommended limits. Public health campaigns targeting traders and farmers should also be launched to educate them about the dangers of pesticide misuse and alternative pest control methods, such as hermetic storage bags and biological pest control solutions (Mutungi *et al.*, 2019).

Beyond regulation, food processing industries must implement strict quality control measures to ensure pesticide residues are within safe limits before cowpea grains reach consumers. Washing, soaking, and heat treatments have been reported to reduce pesticide residues in food, but these measures are not foolproof (Albaseer *et al.*, 2019). Consumers must be aware of potential contamination risks and encouraged to adopt safer food preparation practices.

## CONCLUSION

The findings of this study highlight the urgent need for improved pesticide management in cowpea storage. The detection of DDVP and cypermethrin residues exceeding WHO limits in some samples indicates unsafe pesticide application practices, which could have severe health implications for consumers. As Nigeria seeks to improve food safety standards and regain access to international markets, a coordinated effort involving regulatory agencies, traders, and consumers is essential to mitigate the risks associated with pesticide residues in food.

## REFERENCES

- Akintobi, O. S., Evinemi, C. A, and Achagwa, S. M. (2018). Analysis of the Problems Associated with Cowpea Storage Among Farmers in Kuje Area Council, Federal Capital Territory Abuja, Nigeria. *FUDMA Journal of Agriculture and Agricultural Technology* **4** (1): 91-98.
- Asogwa, J. C., Nwankwo, E. N., Ezekwesili-Ofili, J. O., Asogwa, K. K., and Okwor, C. R. (2023). Analysis and quantification of the levels of insecticide residues in maize sold in South Eastern Nigeria. *Journal of Applied Entomology*, **3**(4):16-22
- AOAC (2012). Official Method of analysis Association of analytical chemist 19<sup>th</sup> edition Washington DC. U.S.A 121-130.
- Borno State Ministry of Information (BSMI) (2017). National Bureau of statistic Demographic Statistic Bulletin third series 3.
- Codex pesticides residues in food online database: pesticide index [Internet]. Rome: Food and Agriculture Organization/World Health Organization; c2020 [cited 2020 Jul 27]. Available from: <http://www.fao.org/fao-who-codexalimentarius/codex-texts/dbs/pestres/pesticides/en/>
- Ekhuemelo, c. (2023). Assessment of aflatoxin and pesticide residue in cowpea (*Vigna unguiculata* L. walp.) from north central nigeria. *Scientia Africana*, **22** (1):41-54. <https://dx.doi.org/10.4314/sa.v22i1.5>
- Fadina, O. O., Daodu, B. J., Fayinminnu, O. O., and Nwanguma, C. S. (2021). Determination of Organochlorine Residues in Cowpea (*Vigna unguiculata* L. WALP) From Selected Markets in Ibadan, Oyo State, Nigeria. *Journal of Agricultural Studies*, **9**(4): 72-86.
- Gesald, J.D.(2009). Topography Analysis of Higher Performance Liquid Chromatography. The Extracted Insecticide on average cowpea had levels of insecticide residue while maize had lower. *Journal of natural sciences and engineering technology* **11**(1):62-71.
- Gerber, F.Krummen,M. Potgeter, H.Roth, A.Siffrin, C. and Spoendlin, C. (2004). Practical Aspects of Fast Reversed Phase Higher Performance Liquid Chromatography using pharmaceutical development and production working under current good manufacturing practice. *Journal of chromatography A* **1036** (2): 127-133.
- International Institute of Tropical Agriculture (IITA) (2024). [www.iita.org/cropsnews/cowpea/#1620977076242-b594b658-46f3](http://www.iita.org/cropsnews/cowpea/#1620977076242-b594b658-46f3)
- Kalpan, Hajam, Y.A. and Kumar, R. (2022). Management of stored grain pest with special reference to *Callosobruchus maculatus*, a major pest of cowpea: A review. *Helicon*:**8**(1) <https://doi.org/10/j.helicon.2021.e08703>
- Nwagboso, C., Andam,K.S., Amare, M., Bamiwuye, T. and Fasoranti, A. (2024). The economic Importance of cowpea in nigeria; trends and implications for achieving Agric-Food System Transformation, International Food Policy Research Institute. *IFPRI Discussion Paper 02241*
- Ogah,C.O.Coker,H. A. and Adepoju Bello,A.A.(2011).Pesticide Residue levels in Maize Samples from market in Lagos state,Nigeria. *Journal of hospital Medicines* **21**(2):169-174.
- Omokpariola, P. L., Okoye, P. A., Okechukwu, V. U., and Omokpariola, D. O. (2023). Concentration levels and risk assessment of organochlorine and organophosphate pesticide residue in selected cereals and legumes sold in Anambra State, south-eastern Nigeria. *Physical Sciences Reviews. Vol,9, no. 3, pp. 1353-1373.* <https://doi:10.1515/psr-2022-0319>
- Patricia blanch (2012). Different Pesticide mixture standards published talanta Pesticideresidues in food and the Environment and the WHO Core Assessment Group Food and Agricultural Organization Plant Production and Protection Paper156–172.

Popoola, B.O., Ongom, P.O., Mohammed, S.B., Togola, A., Ishaya, D.J., Bala, G., Fatokun, C. and Boukar, O., 2024. Assessing the Impact of Genotype-by-Environment Interactions on Agronomic Traits in Elite Cowpea Lines across Agro-Ecologies in Nigeria. *Agronomy*, 14(2):263.

Stephen, W.C. Chung; Benedict and L.S. Chen. (2011). Determination of Organochlorine pesticide residues in

fatty foods: A critical review on the analytical methods and their testing capabilities". *Journal of Chromatography A* .1218 (33): 5555–5567.

Williams, A. B. (2011). Organochlorine Pesticide residues in Shellfishes and Finfishes from Lagos lagoon. *African journal on Environment science and technology* 5 (6)437-442.