

# Analysis of Pesticide Residues in Palm Oil obtained from Ankpa, Olamaboro and Dekina Local Government Areas of Kogi State

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## ABSTRACT

In this study, sample extraction and clean-up were achieved by employing the multi-residue techniques consisting of the QuEChERS and acetonitrile sample extraction methods. Analysis was done using gas chromatography with a mass spectrometric detector (GC-MS). The concentration ( $\mu\text{g}/\text{kg}$ ) of pesticides residue in palm oil samples were: chlorpyrifos (0.0125-0.02), DDVP (0.068-0.068), parathion (0.028-0.028), Dimethoate (0.0175-0.2875), BHC (0.043-0.160), endosulfan I (0.017-0.017), dieldrin (0.095-0.098), endrin (0.070-0.041), endosulfan II (0.058-0.058), DDT (0.05-0.140) and methoxychlor (0.008-0.0525). Dieldrin showed the highest concentration value in the palm oil samples, while chlorpyrifos had the least. The results revealed that the pesticide residue concentration analyzed samples was below maximum residue limits, indicating that palm oil in Kogi L.G.A. is less contaminated and less harmful.

**Keywords:** Palm oil, pesticides residues, GC-MS, QuEChERS

## 1. INTRODUCTION

Nowadays, palm trees can be attacked by a diversity of pests and unwanted plants that could reduce the quality and quantity of the palms used to produce palm oil. In this respect, the extensive use of pesticides has played a vital role in controlling or preventing these pests and harmful plants and increasing world palm oil production. On the

other hand, the use of pesticides in agricultural production has acute toxicity to human and animal bodies. Hazardous pesticides exposure concerns the general population, especially the agricultural population and residents living near industrial or contaminated areas [1](Lambropoulou and Albanis, 2013). Therefore, the excessive use of pesticides often leads to residues heavily going beyond the limits. Even when applied following good agricultural practices, they can leave residues, harming food safety [2](Lentza Rizo *et al.*, 2012).

According to [3](Vanguard News Paper, August, 2015), food commodities from Nigeria were banned from Europe till June 2016. The European Food Safety Authority said that the rejected food commodities were found to contain between 0.03 mg/kg to 4.6 mg/kg of dichlorvos pesticide, when the acceptable maximum residue limit is 0.01 mg/kg.

Organochlorine and deadly chemicals in Nigeria are used because they are cheaper than newer and safer pesticides [4] (Erhunmwunse *et al.*, 2012). The research confirmed the use of dichlorvos in a multiple response schedule and discovered that aluminium phosphate tablets ranked 80 % for storage pesticide while dichlorvos was 60 %, DDT 35 % [5](Pii *et al.*, 2019). Others such as endosulfan, gamalin, carbofuran, carbendazim and permethrin were between

5-15%. In addition, the study discovered the use of many restricted and obsolete pesticides within Kogi State and in outrageous quantities which could cause health challenges to consumers of these food products.

Therefore, it is very essential to carry on the inspection and control of pesticide residues. The Codex Alimentarius Committee on Pesticide residues and the Joint FAO/ WHO Meeting on Pesticide Residues have established maximum pesticide residue limits for some of the pesticides in palms destined for oil production [6](International Codex Alimentarius Committee on Pesticide Residues 2021). However, it should also be noted that there are no harmonized maximum residue limits (MRLs) have not yet been established for pesticide residues in palm oil. The National Committee on Agricultural Commodity and Food Standards (2008) issued a notification entitled the Thai Agricultural Standards on Pesticide Residues: Maximum Residue Limits (TAS 9002-2008) for palm oil on 31 July 2006 which was published in the Royal Gazette (Thai Agriculture Standard, TAS 9002-2008). Therefore, this could help to ensure the safe use of pesticides in Nigeria especially in the consumption of food crops.

## 2. MATERIALS AND METHODS

### 2.1. Reagents

The following analytical high grade chemicals were used: Chloroform, potassium iodide (KI), glacial acetic acid (99.0 %), sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ), carbon tetrachloride, potassium iodide (Lobachemie, India), anhydrous  $\text{MgSO}_4$  (Lobachemie, India), gradient grade acetonitrile, analytical graded acetone was obtained from Aldrich, silica gel (Lobachemie, India) and primary secondary amine (PSA).

### 2.2. Equipment

Pesticide quantification was achieved by using Hewlett Packard (HP) 6890 GC/MS,

equipped with dual injector and column, sample vials was used alongside Gilson pipette, 200 mL beaker, spatula, weighing balance, ultrasonic bath (Clean 120-HD), extraction tubes, rotary evaporator (Buchi R215), sintered glass column for liquid chromatography (2 mm diameter), (HP) 5 Column (Length; 30 mL).

### 2.3. Study Area

This study was conducted in Anпка, Dekina and Olamaboro local government areas being the major palm oil production areas in Kogi state, Nigeria. Kogi State lies between longitudes  $5^{\circ}40'$ E and  $7^{\circ}49'$  E and latitudes  $6^{\circ}33'$  N and  $8^{\circ}44'$  N. It is bounded to the South by Anambra and Edo States; and to the North by Niger, Nassarawa and Federal Capital Territory; to the East by Benue and Enugu States. On the Western flank it shares a common border with Ondo, Ekiti and Kwara States (Kogi A.D. P, 1993). It has a population of 3,278,487 inhabitants (National Population Commission, NPC, 2007). It covers a land area of 30,354.74 square kilometers ( $\text{Km}^2$ ). The people in these local government areas are also known for their trading and fishing livelihood. The trading is mostly practised by women [7](Abdullahi, 2004). Figure 1 shows Map of Kogi State with the study areas.

### 2.4. Sample Collection

The sampling is focused on three local Governments areas in Kogi State branch market, which is palm oil's primary distribution or production. Thirteen (13) palm oil samples were collected randomly from three local government areas, Kogi State in different council wards. These samples were purchased from 4 different market sellers and were composited. This process was carried out for all the palm oil samples in the markets in December, 2021. The samples were stored in amber- coloured bottles with tight covers to protect them from contamination. Samples were stored in a refrigerator at  $4^{\circ}\text{C}$  until their analysis.

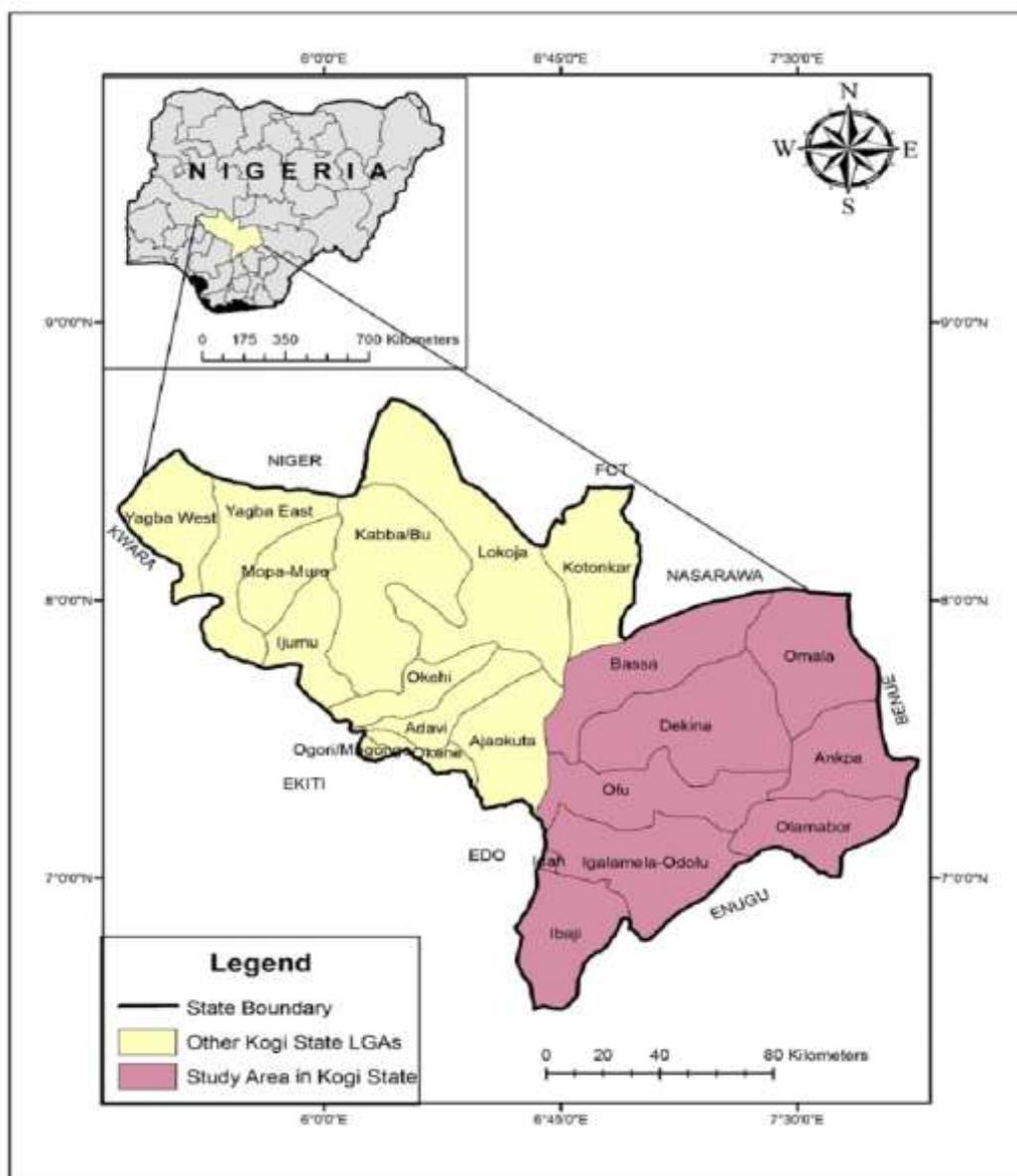


Figure 1: Map of Kogi State showing the study area

### 2.5 Sample Preparation

The collected palm oil samples from the different locations were homogenized, extracted, pre-concentrated (when needed), cleaned-up and stored at room temperature for further analysis.

### 2.6 Sample Clean-up

The extraction method and clean-up used in this study was a modification of the method for multi-residue analysis consisting of QuEChERS sample extraction method as outlined by [2](Lentza-Rizos *et al.*, 2012). Both the cartridges (GCB, PSA) were first conditioned with 5 mL acetonitrile. An

aliquot (2 mL) of the upper layer of the acetonitrile extract was transferred into a GCB (graphitized carbon black) column, after which the entire GCB cartridge was attached to the top of the PSA (primary secondary amine) cartridge with a use of a PTFE adaptor. The extract was initially allowed to flow under gravity, and then a gentle pressure was applied to achieve a flow of approximately one drop per second. The collection of the eluate into a 10 mL graduated vial started at this point. The column was then eluted with an additional 2 mL acetonitrile and the volume

collected was adjusted to 5 mL. Eluate was then mixed and ready for GC- MS analysis.

### 2.7 Preparation of Stock Solution of Pesticides/ Calibration of the Instrument

Stock solutions of individual pesticides were prepared by dissolving the appropriate volumes of their respective concentrates with HPLC-grade methanol in separate 100 mL vials. A solution of pesticide mixture with a concentration 10 ppm was prepared by pipetting the appropriate volume of each stock solution into a 10 mL vial and making it up to the mark with methanol. Then, the volumes of the pesticide mixture were serially diluted to produce working standards with different concentrations of 0.5, 0.25, 0.10 and 0.01 ppm.

The sensitivity and linearity of the detector response to the analyte were assessed by the calibration curve constructed from a series of

six pesticide standards listed as 0.01, 0.05, 0.08, 0.10, 0.50 and 1.00  $\mu\text{L/mL}$ .

The instrumental limit of detection (LOD) and limit of quantitation (LOQ) were also determined from the injection of matrix-matched standard solutions with low concentration levels giving a signal-to-noise ratio of 3 and 10, respectively.

### 2.8 Analysis

The residues are performed using a Hewlett Packard (HP) 6890 GC/MS, equipped with dual injector and column that allowed the detection of contaminants even at trace level (in the lower micrograms per gram range). The GC conditions and detector response will be adjusted so as to match the relative retention time and response. The Hp 6890 conditions used for PAH analysis are presented in Table 1

Table 1: Instrumental Conditions for Pesticide Analysis in Palm Oil using GC-MS

Analysis	Description
Column Flow Rate	RTX – XLB2.0 milliliters/minutes (mL/min)
Carrier gas	Helium
Flow rate	1.0 and 29 mL/min
Injection temperature	280 °C
Make – up gas	Argon/Methane
Oven temperature	130°C hold for 1 minute (min) 5 °C/min to 300 °C, 9 min at 300 °C
Detector temperature	320 °C
Column oven temperature	60 °C for 2 min and at 180 °C/min. Up to 300 °C
GC injection	1.0 microliter ( $\mu\text{L}$ )
Data system	HP Chem Station

## 3. RESULTS AND DISCUSSION

### 3.1. Quality Control Parameters

The quality control parameters of pesticide are presented in Table 2. The RSD ranged from 3.2 to 10.9%, LOQ ranged from 0.0015 to 10.130 ppb and LOD ranged from 0.0648 to 1.8362 ppb indicating the high sensitivity of the gas chromatograph at the operating conditions. The mean and standard deviation were calculated from the detectable values, and values below the detectable limit were considered not-detected (ND). The calibration curves of the analyzed pesticides presented good regression lines in the range of explored concentrations.

Table 2: Quality Control Parameters showing the Mean Concentration and Relative Standard Deviation of Pesticides determined in palm oil

Quality Control Parameters						
Name of Pesticide	Mean (PPb)	S.D (ppb)	LOD (ppb)	LOQ (ppm)	RSD (%)	R.T (mins)
BHC	0.4770	0.4355	1.3063	4.3554	9.1	5.293
Endosulfan I	0.0695	0.2799	0.8399	2.7997	4.02	8.484
Chlorpyrifos	0.0495	0.0015	0.0047	0.0015	3.2	11.878
Dieldrin	0.6700	0.6121	1.8362	6.1206	9.1	11.989
Endrin	0.6700	0.6121	1.8362	6.1206	9.1	12.588
Endosulfan II	0.2133	0.0232	0.0697	0.2324	10.9	15.831
DDVP	0.2767	0.0216	0.0648	0.2160	7.8	15.981

Parathion	1.1000	1.0130	3.0391	10.130	9.2	16.562
DDT	0.0417	0.0432	0.1295	0.4317	10.3	16.734
Dimethoate	0.0695	0.2799	0.8399	2.7997	4.03	18.063
Methoxychlor	0.4333	0.4367	1.3102	4.3672	10.1	19.653

### 3.2. Concentration of Pesticide Residues in Palm Oil

A total of 15 palm oil samples were collected from Ankpa, Dekina and Olamaboro in Kogi State for investigation of 23 pesticides residues. The general descriptive results of pesticide residues as presented for palm oil shows, seven (7) organophosphate pesticides, organochlorine pesticides thirteen (13), pyrethroid and carbamates three (3) commonly used pesticides were determined. The commonly occurring residues were those of chlorpyrifos, DDVP, parathion, dimethoate while dichlorvos, ethion and diazinon were not detected for the organophosphate pesticides. However, the organochlorine pesticides residue are BHC, endosulfan I, O-Terphenyl, dieldrin, endrin, endosulfan II, methoxychlor, DDT while adrin, heptachlor epoxide, mirex and chlordane were not detected. The pyrethroid and carbamates were not detected (cypermethrin, deltamethrin and carbonyl). Summary statistics of concentration of the pesticides residue that were detected in organophosphate and organochlorine are presented in table (3) and (4) respectively.

**Chlorpyrifos:** The mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of chlorpyrifos in palm oil in the different local government areas in table (3) shows that dekina ( $0.02 \pm 0.02$ ) has the highest mean concentration. The LOD and LOQ were  $4.7 \times 10^{-3}$  and  $1.5 \times 10^{-3}$  respectively. The concentrations of chlorpyrifos in the sample palm oil are below its acceptable maximum residual limits (0.5 mg/kg) set by [8] Codex Alimentarius, (2021). The study by [9] Roszko *et al.*, (2012) reported a concentration of chlorpyrifos ( $0.026 \text{ mg kg}^{-1}$ ) in Iran which is higher than the current research work.

High use of chlorpyrifos could lead to the poisoning of non-target species likes humans and also the environment [10](Perry *et al.*, 2020). This may result to health

abnormalities mainly in the nervous system, cardiovascular, respiratory system and even death [11] (Rezg *et al.*, 2010).

**DDVP:** Table (3) shows the concentration ( $\mu\text{g}/\text{kg}$ ) of pesticide residues in palm oil. The concentrations of DDVP in the samples are as follows: Olamaboro ( $0.068 \pm 0.068$ ), Ankpa and Dekina were below detection limit. The LOD and LOQ were  $6.48 \times 10^{-2}$  and  $2.16 \times 10^{-1}$  respectively. The DDVP mean concentration are below the maximum residue limits (0.1 mg/kg) set by [6] (International Food Standards/Codex Alimentarius FAO/WHO, 2021). The mean value ( $0.032 \text{ mg kg}^{-1}$ ) recorded by [9] Roszko *et al.*, (2012) is lower than the value obtained from this study. It is one of the most commonly used organophosphate pesticides in developing countries, and is classified by WHO as a class IB, 'highly hazardous chemicals' [12] (Suchismita, 2013).

**Parathion:** The mean concentration ( $\mu\text{g}/\text{kg}$ ) of parathion in palm oil is  $0.028 \pm 0.028$  from Olamaboro while Ankpa and Dekina were all below detected limit. The mean value recorded in this study is lower than its acceptable maximum residue limits (0.05 mg/kg) set by [6] (International Food Standards/Codex Alimentarius FAO/WHO, 2021). This organophosphate compound which was widely used for agricultural purposes, and may have resulted in human exposure during its application, and residues on or in foods can result in exposure to humans by ingestion [13] (Lenčič and Biljana, 2013). All use of parathion has been banned or restricted in most developing countries to mitigate the risk of human exposure [14](Dyson *et al.*, 2015).

**Dimethoate:** The mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of dimethoate in palm oil in the different local government areas in kogi state in Table (3) ranged between

0.0175-0.2875. This entails that Olamaboro ( $0.2875 \pm 0.13168$ ) has the highest mean concentration followed by Dekina ( $0.0175 \pm 0.0175$ ) and the mean value of Ankpa was below detection limit. However, the recorded mean dimethoate concentration in this research work fell below its acceptable maximum residue limits (1 mg/kg) set by [6](International Food Standards/Codex Alimentarius FAO/WHO, 2021). The mean value obtained in this study area is lower than that reported by [15]Elhom *et al.*, (2012) 1.5 - 3.5 ng g<sup>-1</sup>. The widespread use of Dimethoate may pose a health hazard to animals and humans because of its persistence in soil and crops. The Environment Protection Agency (EPA) classifies dimethoate as a class II toxicity-moderate toxic compound [16] (Sunita and Dhairaj 2014).

**BCH:** Mean residue concentration ( $\mu\text{g/kg}$ ) of BHC ranged from 0.025-0.160. The highest concentration was recorded in Olamaboro ( $0.160 \pm 0.096$ ), Dekina ( $0.148 \pm 0.085$ ) and the lowest was recorded in Ankpa ( $0.043 \pm 0.025$ ) in the palm oil sample. The value obtained from this work is less than the maximum residue limits [6](set by International Food Standards/Codex Alimentarius FAO/WHO, 2021). The mean value obtained in this research work is higher than the value recorded ( $0.0054 \text{ mg/kg}^{-1}$ ) by [17] Gonzalez *et al.*, (2007) in vegetable oils. According to the World Health Organization (WHO), BHC or lindane is moderately acutely noxious. This is why its usage in agriculture has been regulated or banned [18] (Vijgen *et al.*, 2013). Its higher amount can affect the nervous system and its production process harms the environment.

**Endosulfan I and II:** The mean residue concentration ( $\mu\text{g/kg}$ ) of endosulfan I in palm oil in Table (4) shows that Dekina recorded  $0.0175 \pm 0.0175$  while Olamaboro and Ankpa were all below the detection limit. While in endosulfan II Olamaboro and Dekina had  $0.058 \pm 0.058$  respectively while Ankpa was below the detection limit. The recorded

mean concentration of endosulfan I and II in this research work is less than the maximum residue limits [6](set by International Food Standards/Codex Alimentarius FAO/WHO, 2021), which poses no risk to humans and the environment.

The extensive use of endosulfan is a global concern over its toxicity. Because of its persistence in the environment, its usage has been banned in most developed countries. However, it is still being used in the developing countries because of its availability through illegal importation [19] (Vaikosen *et al.*, 2019)

**Dieldrin:** Dieldrin is an organochloride insecticide. The concentration ( $\mu\text{g/kg}$ ) of this pesticide residue in palm oil was found to be  $0.098 \pm 0.033$  in Olamaboro,  $0.095 \pm 0.059$  in Dekina and Ankpa was below the detection limit. The mean concentration value recorded is lower than its maximum residual limit (0.2 mg/kg) [6] set by International Food Standards/Codex Alimentarius FAO/WHO, 2021.

Dieldrin has proved to be highly effective insecticide and was very widely used during the 1950s to early 1970s. However, it is a highly persistent organic pollutant; it does not break down easily. Furthermore, it tends to biomagnify as it is passed along the food chain [20] (Pang *et al.*, 2020).

**Endrin:** The mean residue concentration ( $\mu\text{g/kg}$ ) of endrin in table (4) shows that Olamaboro recorded  $0.07 \pm 0.041$  while Dekina and Ankpa were below the detection limit. The mean concentration value recorded in this study is higher than its maximum residual limit (0.01 mg/kg) set by [6] International Food Standards/Codex Alimentarius FAO/WHO, 2021.

The most significant route of exposure is most likely ingestion of imported foods and also from groundwater contaminated with endrin. However, this has made the usage of endrin to be restricted in most countries worldwide and which helps to reduce the potential for human exposure [21] (Freire *et al.*, 2014). The high concentration of endrin

**DDT:** The mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of DDT (Dichlorodiphenyltrichloroethane) ranged from 0.035 to 0.14. Ankpa recorded  $0.05\pm 0.058$ ,  $0.14\pm 0.052$  in Dekina and was below detection limit in olamaboro. The mean concentration value recorded is lower than its maximum residual limit (0.5 mg/kg) set by [6] International Food Standards/Codex Alimentarius FAO/WHO, 2021. The value ( $0.02 \text{ mg}/\text{kg}^{-1}$ ) reported by [9] Roszko *et al.*, (2012) is lower than the value recorded in this study. The human exposure to DDT and its breakdown products is primarily through dietary ingestion, particularly of meat, fish, poultry, and root and leafy vegetables [22] (Yang *et al.*, 2017), which is very persistent in the environment and causes toxic effects on humans and wildlife.

**Methoxychlor:** The mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of methoxychlor ranged from 0.035 to 0.14. Dekina recorded the highest ( $0.0525\pm 0.0335$ ) followed by olamaboro ( $0.08\pm 0.08$ ) and ankpa recorded the lowest ( $0.008\pm 0.008$ ). The mean concentration value recorded in olamaboro and dekina are above its maximum residual limit except for ankpa ( $0.01 \text{ mg}/\text{kg}$ ) set by [6] International Food Standards/Codex Alimentarius FAO/WHO, 2021.

The detection of methoxychlor may be either as a result of the historical use of DDT of which technically methoxychlor contains about 88 % of the p,p'- isomer together with more than 50 structurally related contaminants, which might have been added to the actual amount of methoxychlor present [23](Agbeve *et al.*, 2014).

**Table 3: Mean Residue Concentration ( $\mu\text{g}/\text{kg}$ ) of Palm oil in the Study Areas (Organophosphate)**

Pesticides/ Locations	N	Min	Max	Sum	Mean $\pm$ Std. Error
Chlorpyrifos					
Olamaboro	4	0	0	0	0
Ankpa	4	0	0.05	0.05	$0.0125\pm 0.0125$
Dekina	4	0	0.08	0.08	$0.02\pm 0.02$
DDVP					
Olamaboro	4	0	0.27	0.27	$0.068\pm 0.068$
Ankpa	4	0	0	0	0
Dekina	4	0	0	0	0
Parathion					
Olamaboro	4	0	0.11	0.11	$0.028\pm 0.028$
Ankpa	4	0	0	0	0
Dekina	4	0	0	0	0
Dimethoate					
Olamaboro	4	0	0.59	1.15	$0.2875\pm 0.1319$
Ankpa	4	0	0	0	0
Dekina	4	0	0.07	0.07	$0.0175\pm 0.0175$

**Table 4: Mean Residue Concentration ( $\mu\text{g}/\text{kg}$ ) of Palm oil in the Study Areas (Organochlorine)**

Pesticides/ Locations	N	Min	Max	Sum	Mean $\pm$ Std. Error
BHC					
Olamaboro	4	0	0.38	0.64	$0.160\pm 0.096$
Ankpa	4	0	0.09	0.17	$0.043\pm 0.0025$
Dekina	4	0	0.31	0.59	$0.148\pm 0.085$
Endosulfan I					
Olamaboro	4	0	0	0	0
Ankpa	4	0	0	0	0
Dekina	4	0	0.07	0.07	$0.017\pm 0.017$
Dieldrin					
Olamaboro	4	0	0.13	0.39	$0.098\pm 0.033$
Ankpa	4	0	0	0	0
Dekina	4	0	0.24	0.38	$0.095\pm 0.059$
Endrin					
Olamaboro	4	0	0.15	0.28	$0.070\pm 0.041$
Ankpa	4	0	0	0	0
Dekina	4	0	0	0	0
Endosulfan II					
Olamaboro	4	0	0.23	0.23	$0.058\pm 0.058$
Ankpa	4	0	0	0	0
Dekina	4	0	0.23	0.23	$0.058\pm 0.058$

DDT					
Olamaboro	4	0	0	0	0
Ankpa	4	0	0.15	0.20	0.050±0.058
Dekina	4	0	0.26	0.56	0.140±0.052
Methoxychlor					
Olamaboro	4	0	0.32	0.32	0.080±0.080
Ankpa	4	0	0.03	0.03	0.008±0.008
Dekina	4	0	0.14	0.21	0.0525±0.03351

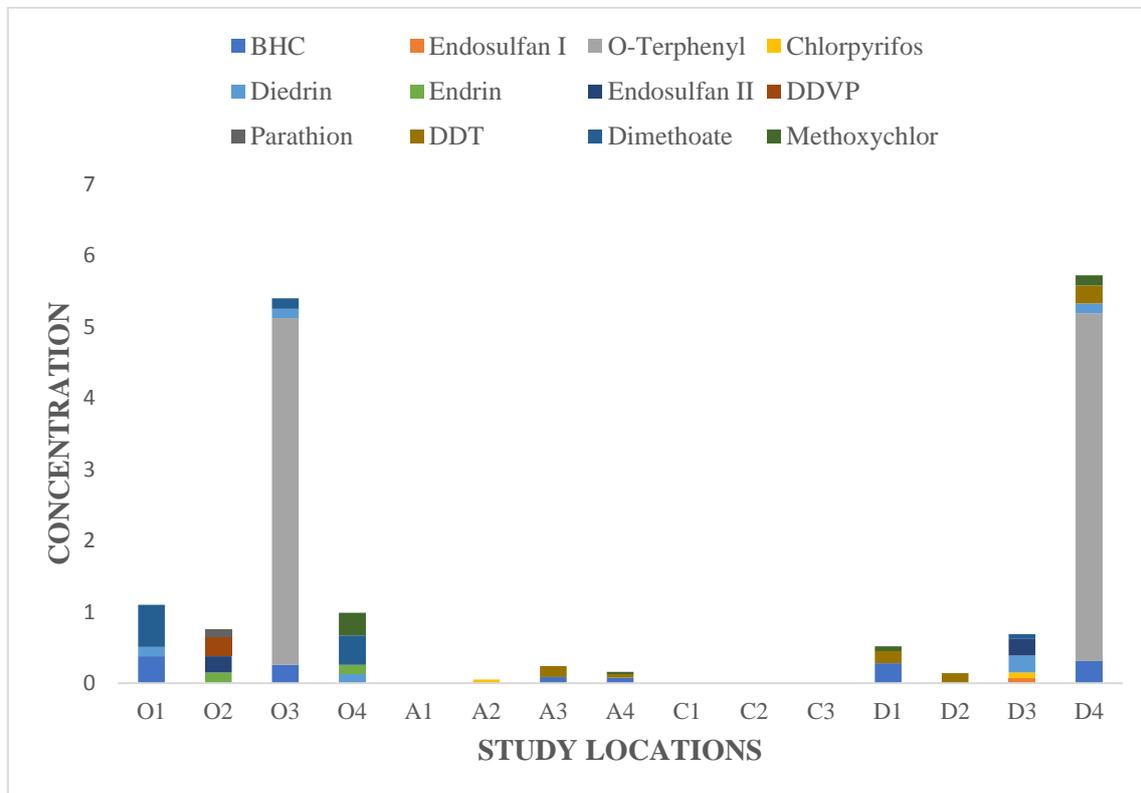


Figure 1: Mean concentration pesticide residues in oil palm from the study area

#### 4. CONCLUSION

The result generally revealed that all samples of palm oil analysed showed some degree of contamination. Pesticide residue levels were generally below their acceptable maximum levels and acceptable daily intake, indicating that palm oil in Kogi L.G.A. of Kogi State is less contaminated and relatively less detrimental.

However, the toxicological importance of pesticide residue data depends not only on the residue content of food but also on the quantity of contaminated food consumed and the length of time over which it is consumed. Thus, incessant consumption of less contaminated food over a long period of time may lead to a perilous high concentration of chemical residues in the body.

Since the improvement in crop yield by pesticide application is always concomitant

with the occurrence of residues in food stuffs, there is need for habitual control of the application of pesticides as this may go a long way towards preventing various environmental and public health hazards.

#### Declaration by Authors

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