



## Organochlorine and Organophosphorus Pesticides Residues in Commercial Poultry Feed Samples in Lagos State, Nigeria

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**ABSTRACT:** Pesticides are among Persistent Organic Pollutants (POP) used in agricultural practices to increase production efficiency. They enter the food chain, bioaccumulate and are toxic to human, animals and the environment. The presence of pesticide residues in animal feeds has been rarely monitored and reported in Nigeria. This study evaluated the levels of pesticide residues in different brands and types of poultry feeds that are produced, marketed and used in Lagos, Nigeria. Five types of five commonly used commercial brands (25 samples) of poultry feeds were purchased from animal feed markets in Lagos, Nigeria. Simultaneous detection and quantitation of selected thirteen (13) OCPs and eight (8) OPs residues in the feeds was carried out using Gas Chromatography-Electron Capture Detector (GC-ECD) and Gas Chromatography-Flame Ionization Detector (GC-FID) respectively. Results revealed that all the feed samples had multiple pesticide residues levels which ranged from 0.10 - 1.83 mg/kg for OCPs and 0.25 - 6.04 mg/kg for OPs. These values were above the Maximum Residual Limit (MRL). The detected pesticide residues order of violation of the MRL was Methoxychlor > Dieldrin > Endrin ketone for OCPs, and Trichlorfon > Malathion > Diazinon for OPs. There was no significant ( $p > 0.05$ ) differences in the pesticide residues levels among the brands and types of the samples. Present study indicates extensive and unwholesome use of pesticides in the agro industry in Nigeria suggesting need to enforce Good Agricultural Practices (GAP), Good Storage Practices (GSP), Good Manufacturing Practices (GMP) and constant monitoring of pesticide residues thereby implementing the Good health and Wellbeing (Goal 3) of the 2030 Sustainable Development Goal.

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Organochlorine pesticides (OCPs) and Organophosphorus pesticides (OPs) are persistent in the environment, they also bioaccumulate and biomagnify in the food chain. Continuous use and poor management of pesticides in crop production, transportation, and storage has resulted in pesticide residues in animal feeds and feedstuffs – which is the main route of exposure to farm animals (Ledoux, 2011). Consumption of contaminated feeds by animals lead to the bioaccumulation of pesticide residues in fatty tissues of animal products (poultry, fish, meat, eggs, milk) which is finally transmitted to man via the food chain (Waliszenwski *et al.*, 2003), resulting in human health complications. Foods of animal origin are the major route of pesticide contamination to humans, and are now of major food safety concern worldwide (Kumar *et al.*, 2018). Other sources of animal and human exposure include environmental

exposure via inhalation and dermal exposure [Zezelew and Mekassa, 2018). Modern large scale/commercial intensive poultry production system makes poultry feed an important source of pesticide residue, (Khilare *et al.*, 2016; 2017). To avoid large scale contamination of food of animal origin, there is need to continuously monitor and control pesticide residues in animal feeds before consumption by animals (Panseri *et al.*, 2013). The toxic effects of pesticide residues in birds include heavy mortality, reproductive impairment, skeletal abnormalities, impaired differentiation of the reproductive and nervous system, acute mortality, sub-lethal stress, suppression of egg production, egg shell thinning, impaired incubation and chick rearing behavior, (Jayaraj, 2016). Pesticides also affect the animal microbiome, modifying the microbiome of the animal and this compromise could result in reduced immunity, (Syromyatnikov *et al.*, 2020). The use of

dichlorodiphenyltrichloroethane DDT was banned in developed nations in 1980. And at the 2001 Stockholm Convention on Persistent Organic Pollutants, 179 countries signed an international treaty to stop the use of twelve POPs including DDT, because of their persistence and bioaccumulation in the environment (Mahmood *et al.*, 2016). However, many developing countries still produce and use large quantities of these pesticides in crop protection and environmental sanitation e.g. to control pests and rodents, (Kumar *et al.*, 2019; Adewunmi and Fapohunda, 2018; Carvalho, 2006). Several studies have confirmed the presence of significant concentration of these pesticide residues in crop and animal products, (Panseri *et al.*, 2013; Ogah *et al.*, 2012; Gwary *et al.*, 2011; Akoto *et al.*, 2013). Earlier studies have reported that, foods of animal origin have maximum contamination of pesticides followed by leafy vegetables and garden fruits (Rathore *et al.*, 1996). Poultry products (meat and eggs) are important sources of nutrients and nutraceutical to the human population. Both are widely consumed. The presence of contaminants (pesticides) in such foods is of public health concern. The presence of pesticide residues in animal feed is rarely monitored and reported in Nigeria. This study therefore investigates, compare and report the contamination level of selected OCPs and OPs in selected commercial poultry feed samples that is produced and used in Lagos, Nigeria.

## MATERIALS AND METHODS

**Equipment and Reagents:** BUCHI R215 Rotary Evaporator, Clean 120-HD Ultrasonic Bath, Agilent 78790A GC equipped with an Electron Capture Detector GC-ECD or Flame Ionization Detector GC-FID was used for the chromatographic separation, Agilent HP 5 Column (Length 30 m x 0.32 mm x Phase Thickness 0.25  $\mu\text{m}$ ), Weighing Balance, n-Hexane, Silica gel (Lobachemie), Anhydrous Sodium Sulphate from Lobachemie (India), Acetone, Nitric acid.

**Sample Collection:** Twenty five feed samples from twenty five bags of feeds, i.e. one bag (25 kg) each of five types (Starter, grower, layer, chick and finisher mash) of five feed brands (AF, TF, HF, CF, UF) were purchased from feed markets and farms in Lagos, Nigeria

**Sample Preparation:** Each bag of feed was thoroughly mixed, and sample taken from different points for grinding. 5 g of ground sample was transferred into a beaker, which was added 20 ml of Hexane: Acetone (1:1) mix. The beaker and its contents were placed in an ultrasonic bath to sonicate for 20 minutes. The mixture was then allowed to settle in order to decant

the solvent layer. The mixture was later concentrated to 2 ml using a rotary evaporator. A glass column was packed with 4 g of Silica gel (previously activated for 6 hours at 130°C in petri dish loosely covered with foil) and 2 g of anhydrous sodium sulphate. 10 ml hexane was also added to the top of the column to wet and rinse the sodium sulphate and silica gel. The sample was then transferred onto the column. 20 ml hexane was further used to elute the column, and the eluate was concentrated to 2 ml using a rotary evaporator.

**Identification of OCPs using GC-ECD:** To identify the OCP residues in the sample, a gas chromatograph (GC) equipped with an electron capture detector (ECD) was used. The cleaned extract was dried and re-dissolved in 1  $\text{cm}^3$  of Analar grade Isooctane for injection into the gas chromatograph (Pandit *et al.*, 2001). Blank runs were made for background correction and performance of the system. OCPs II EPA method 8081a [Mix AB #3, cat. #321415 (ea)] was used for the analyses. The detection and determination of the residues were performed by injecting 1  $\mu\text{L}$  of the 1.0  $\text{cm}^3$  purified extract into the injection port of the gas chromatograph with a  $^{63}\text{Ni}$  ECD (GC- $\mu\text{ECD}$  Agilent Technology 7890A) equipped with Chem station software. The column consisted of a DB-5 fused silica capillary column (30 m length  $\times$  0.32 mm i.d.  $\times$  0.25  $\mu\text{m}$  film thickness). The column temperature was programmed from 50°C at a rate of 25 °C/min to 100°C, held for 1 minute, and then at a rate of 5 °C/min to 300 °C, held for 5 minutes. The temperatures of the injector and detector were 250 °C and 300 °C, respectively. The injection was carried out on a splitless injector at 250 °C and the purge activation time was 30 seconds. The carrier gas was helium while nitrogen gas was used for the makeup flow. The run time was 17 minutes.

**Identification of OPs using GC-FID:** Samples were analyzed using an Agilent 7890B Gas chromatograph equipped with a flame ionization detector (FID), fitted with a HP-5 capillary column coated with 5% Phenyl Methyl Siloxane 30 m length x 0.32 mm diameter x 0.25  $\mu\text{m}$  film thickness (Agilent Technologies). 1  $\mu\text{L}$  of the samples were injected in splitless mode at an injection temperature of 220 °C, at a pressure of 14.861psi 37 and a total flow of 21.364 mL/min. Purge flow to split vent was set at 15 mL/min at 0.75min. The oven was initially programmed at 100 °C (2 min), then ramped at 10 °C/min to 280 °C (4 min) and then ramped to 300 °C at 10 °C/min. Flame ionization detector (FID) temperature was 300 °C with Hydrogen: Air flow at 30 mL/min: 300 mL/min, Nitrogen was used as the makeup gas at a flow of 18 mL/min. Identification of pesticide residues in both

procedures was accomplished using reference standards and relative retention time techniques, while the concentration of the residues was determined by comparing the peak heights of the samples with the corresponding peak heights of the reference standards of known concentrations. Stock solutions of the OCP and OP standards were purchased from Restek Corporation, USA.

*Statistical Analysis:* The data were subjected to descriptive analysis using Statistical Package for Social Sciences (SPSS) software 20 version was used to check the presence of significant difference at 95% confidence and the significance difference were determined at the  $p < 0.05$  level.

## RESULTS AND DISCUSSION

The accuracy of the instrument in terms of percentage recovery of each pesticide was evaluated for the feed samples. The result is presented as shown in Table 1. Result for the OCPs ranged from 65% - 118% while OPs ranged from 67% - 113%. This recovery is acceptable for pesticides in spiked samples, (EU, 2002). Figs 1 and 2 show the chromatogram of the standard mix of OCPs and OPs respectively. The system precision was evaluated by studying the reproducibility of the instrument response relative to the retention time and area of the analyte. Specificity was evaluated by visual observation of spiked and unspiked samples chromatographic signals at the

retention times of the specific pesticides. The following selected OCPs were identified in the feed samples: endosulfan sulfate, endosulfan, endosulfan II, endrin aldehyde, endrine ketone, dieldrine, p,p'-DDT, p,p'-DDD, p,p'-DDE, methoxychlor, heptachlor, endrin, and heptachlorepoxyde. Also, the selected OPs identified in the samples are coumaphos, diazinon, dichlorvos, dimethoate, ethyl parathion, malathion, parathion, and trichlorfon. One hundred percent of the feed samples (brands and types) contained multiple OCP and OP pesticide residues. Figs 3 and 4 show the chromatogram for a poultry feed sample showing OCP and OP residues respectively. Table 2 shows the mean concentration (mg/kg) of the different OCPs and OPs identified in the different feed types. All the OCPs and OPs identified were present in the different types of feed under study. Comparatively, the levels of the OCPs and OPs identified were higher than the maximum residual level MRL, but were not significantly different ( $p > 0.05$ ) from one another, except dichlorvos ( $p < 0.05$ ), in the different feed types. Our report supports the work of Zelelew and Mekassa, 2018. The authors reported values higher than the MRL for broiler and layer feeds. Contrarily, they reported that the pesticide residues in broiler feeds were significantly higher than in layer feeds, probably due to higher percentage of protein source in broiler feeds.

**Table 1:** Percentage Recovery of Organochlorine and Organophosphorus Pesticides from spiked feed samples

Organochlorine	% Recovery	Organophosphorus	% Recovery
p,p'-DDT	89%	Coumaphos	78%
p,p'-DDE	65%	Diazinon	67%
p,p'-DDD	68%	Dichlorvos	101%
Dieldrin	77%	Dimethoate	79%
Aldrin	71%	Ethyl Parathion	99%
Endosulfan	69%	Malathion	90%
Endosulfan II	93%	Parathion	91%
Endosulfan Sulfate	88%	Trichlorfon	113%
Endrin	67%		
Endrin Aldehyde	70%		
Endrin Ketone	102%		
Methoxychlor	118%		
Heptachlor	65%		

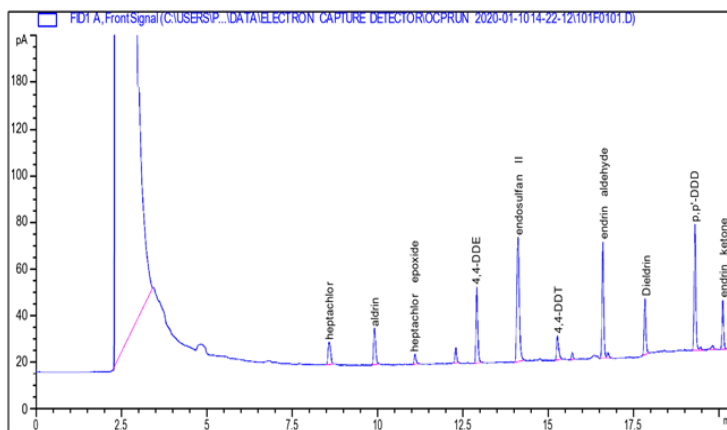


Fig 1: Chromatogram of Organochlorine in Standard Mix

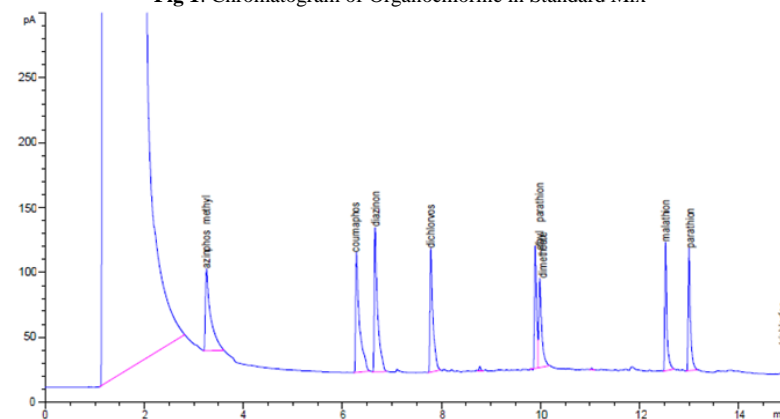


Fig 2: Chromatogram of Organophosphorus in Standard Mix

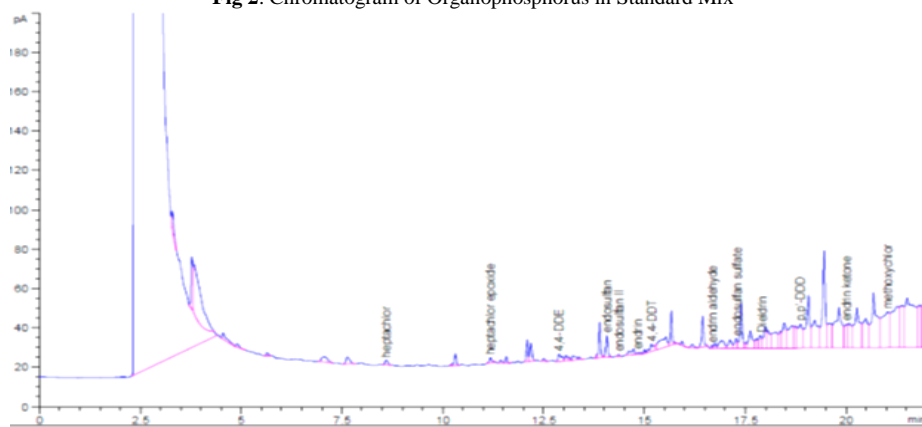


Fig 3: Chromatogram of organochlorine residue in a feed sample

Table 3 shows the mean concentration (mg/kg) of the different OCPs and OPs in the different feed brands. Comparatively, the levels of the OCPs and OPs identified were higher than the MRL, but were not significantly different ( $p > 0.05$ ) from one another except parathion ( $p < 0.05$ ). The presence of pesticide residues in the different feed brands is consistent with the work of Zelelew and Mekassa, 2018. The authors reported the presence of multi-pesticide residues in poultry feeds obtained from different feed mills in

Ethiopia. The non-significant difference ( $p > 0.05$ ) in pesticide residues between the different feed brands agrees with previous works of Zelelew and Mekassa, 2018, Khilare *et al* 2016., Aulakh *et al.*, 2006. This indicates a general indiscriminate and unwholesome use of pesticides by the crop farmers and non-implementation of Good Agricultural Practice and Good Manufacturing Practice in pesticide use by crop farmers and in poultry feed production respectively in the area/region under study.

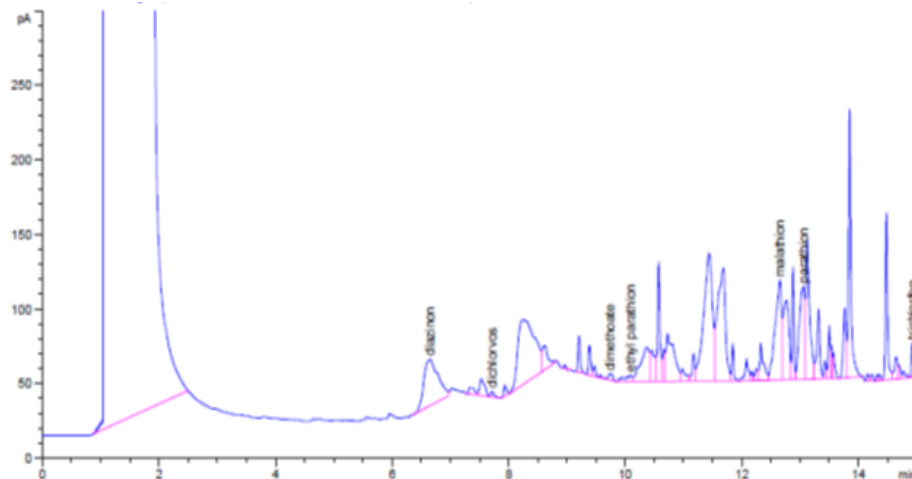


Fig 4: Chromatogram of Organophosphorus in a Feed sample

Table 4 shows the mean concentration of the selected OCPs and OPs in the feed samples (types and brands) compared to MRL. The mean concentrations in our samples were higher than the MRL. The mean concentrations for OCPs: p,p'-DDT, p,p'-DDD, p,p'-DDE, dieldrin, endrin, endrin aldehyde, endrin ketone, endosulfan, endosulfan II, endosulfan sulphate, methoxychlor, heptachlor are 0.29 ±0.04, 0.39±0.06, 0.14±0.03, 1.47±0.38, 0.10±0.02, 0.84±0.22, 0.95±0.37, 0.24±0.04, 0.49±0.19, 0.23±0.02, 1.83±0.44, 0.63±0.06 mg/kg respectively. The respective MRLs are 0.05, 0.05, 0.05, 0.01, 0.01, 0.02, 0.02, 0.1, 0.1, 0.1, 0.5, and 0.01 mg/kg. The mean concentration obtained in this study for p,p'-DDT,

p,p'-DDD and p,p'-DDE are lower than the value of 3.70 - 6.68 mg/kg for total DDT reported by Mahugija *et al.*, (2018), but higher than 4.12±1.79 µg/kg reported in another study Panseri *et al.*, 2013). However our values are consistent with previous report of 0.25 mg/kg (DDT) and 0.91 mg/kg (total DDT) for poultry feed (Kumar *et al.*, 2019). The presence of significant levels of DDT and its metabolites DDE and DDD in our result indicate the consistent use of the banned DDT in poultry feedstuff/feed production chain in Nigeria. The metabolites are products of DDT biodegradation (reductive dechlorination)

Table 2: Mean Concentration of Pesticides in Different Poultry Feed Types (mg/kg ±SD)

Pesticides	Broiler Finisher	Broiler Starter	Chick Mash	Grower Mash	Layer Mash	P value
Organochlorine						
p,p'-DDT	0.20±0.08	0.51±0.28	ND	0.22±0.09	0.16±0.05	0.09
p,p'-DDE	0.30±0.19	0.55±0.51	ND	0.35±0.23	0.36±0.38	0.81
p,p'-DDD	0.13±0.17	0.17±0.19	0.04±0.03	0.17±0.16	0.16±0.17	0.84
Dieldrin	0.80±0.73	1.01±1.43	1.27±1.27	2.30±2.30	1.87±2.38	0.75
Aldrin	ND	ND	ND	ND	ND	
Endosulfan	0.21±0.13	0.27±0.25	0.13±0.00	0.31±0.21	0.25±0.20	0.78
Endosulfan II	1.16±1.98	0.20±0.14	ND	0.38±0.27	0.30±0.25	0.49
Endosulfan sulfate	0.26±0.11	0.27±0.14	0.18±0.12	0.19±0.09	0.23±0.04	0.66
Endrin	0.07±0.02	0.15±0.15	0.06±0.00	0.11±0.07	0.13±0.08	0.62
Endrin aldehyde	0.84±0.82	1.02±1.86	0.07±0.03	1.08±1.11	0.88±0.98	0.81
Endrin ketone	0.64±0.47	2.21±3.83	0.16±0.12	0.60±0.46	0.82±1.01	0.56
Methoxychlor	0.31±0.19	2.04±1.75	1.43±1.94	2.49±2.99	2.71±2.94	0.48
Heptachlor	0.93±0.38	0.49±0.15	0.31±0.00	0.72±0.36	0.61±0.29	0.39
Organophosphorus						
Coumaphos	0.39±0.00	ND	ND	0.11±0.00	ND	-
Diazinon	2.83±3.75	2.52±1.10	2.05±0.73	0.83±0.75	1.99±1.46	0.64
Dichlorvos	4.74±4.08	0.89±0.87	0.11±0.02	0.35±0.27	0.55±0.63	0.02
Dimethoate	1.81±2.12	0.32±0.14	0.36±0.11	0.18±0.11	2.09±2.18	0.32
Ethyl parathion	0.68±0.53	0.06±0.04	2.32±3.62	0.09±0.02	0.46±0.57	0.37
Malathion	4.84±8.25	1.95±2.44	2.73±1.91	0.39±0.41	1.40±1.73	0.54
Parathion	2.39±1.82	1.45±1.34	2.81±0.88	1.27±0.90	1.92±3.25	0.80
Trichlorfon	6.88±4.81	4.45±3.45	1.17±1.27	8.64±7.84	7.32±3.01	0.26

p@0.05

**Table 3:** Mean Concentration of Pesticides in Different Poultry Brands (mg/kg  $\pm$ SD)

Pesticides	AF	BF	CF	DF	EF	P value
Organochlorine						
<b>p,p'-DDT</b>	0.26 $\pm$ 0.10	ND	0.22 $\pm$ 0.09	0.42 $\pm$ 0.44	0.28 $\pm$ 0.15	0.73
<b>p,p'-DDE</b>	0.21 $\pm$ 0.20	0.46 $\pm$ 0.39	0.14 $\pm$ 0.07	0.36 $\pm$ 0.06	0.64 $\pm$ 0.41	0.37
<b>p,p'-DDD</b>	0.12 $\pm$ 0.17	0.06 $\pm$ 0.01	0.08 $\pm$ 0.05	0.25 $\pm$ 0.21	0.19 $\pm$ 0.17	0.28
<b>Dieldrin</b>	1.03 $\pm$ 0.80	0.81 $\pm$ 0.60	3.28 $\pm$ 3.38	0.57 $\pm$ 0.58	1.52 $\pm$ 1.42	0.17
<b>Aldrin</b>	ND	ND	ND	ND	ND	-
<b>Endosulfan</b>	0.18 $\pm$ 0.11	0.09 $\pm$ 0.04	0.28 $\pm$ 0.22	0.36 $\pm$ 0.26	0.28 $\pm$ 0.11	0.23
<b>Endosulfan II</b>	1.50 $\pm$ 2.28	0.09 $\pm$ 0.04	0.32 $\pm$ 0.28	0.42 $\pm$ 0.24	0.24 $\pm$ 0.13	0.40
<b>Endosulfan sulfate</b>	0.19 $\pm$ 0.10	0.15 $\pm$ 0.05	0.23 $\pm$ 0.07	0.29 $\pm$ 0.15	0.28 $\pm$ 0.06	0.23
<b>Endrin</b>	0.09 $\pm$ 0.02	0.05 $\pm$ 0.00	0.14 $\pm$ 0.09	0.07 $\pm$ 0.02	0.16 $\pm$ 0.14	0.44
<b>Endrin aldehyde</b>	0.62 $\pm$ 0.95	1.37 $\pm$ 1.00	0.17 $\pm$ 0.11	0.42 $\pm$ 0.71	1.95 $\pm$ 1.77	0.09
<b>Endrin ketone</b>	0.51 $\pm$ 0.48	0.33 $\pm$ 0.04	0.20 $\pm$ 0.12	1.01 $\pm$ 0.96	2.97 $\pm$ 4.04	0.17
<b>Methoxychlor</b>	0.71 $\pm$ 0.52	0.53 $\pm$ 0.51	1.92 $\pm$ 1.59	3.89 $\pm$ 3.54	1.83 $\pm$ 2.00	0.13
<b>Heptachlor</b>	0.65 $\pm$ 0.48	0.36 $\pm$ 0.00	0.42 $\pm$ 0.09	0.79 $\pm$ 0.15	0.71 $\pm$ 0.34	0.55
Organophosphorus						
<b>Coumaphos</b>	ND	ND	0.25 $\pm$ 0.20	ND	ND	-
<b>Diazinon</b>	2.51 $\pm$ 1.16	3.07 $\pm$ 1.92	1.66 $\pm$ 1.58	0.16 $\pm$ 0.11	ND	0.10
<b>Dichlorvos</b>	1.16 $\pm$ 1.51	0.74 $\pm$ 0.85	2.39 $\pm$ 5.02	1.36 $\pm$ 1.92	2.11 $\pm$ 2.04	0.91
<b>Dimethoate</b>	0.20 $\pm$ 0.06	0.21 $\pm$ 0.01	1.82 $\pm$ 2.06	1.82 $\pm$ 1.98	0.23 $\pm$ 0.00	0.37
<b>Ethyl parathion</b>	0.26 $\pm$ 0.30	0.30 $\pm$ 0.02	0.43 $\pm$ 0.62	2.87 $\pm$ 3.16	0.10 $\pm$ 0.04	0.09
<b>Malathion</b>	1.51 $\pm$ 1.92	0.59 $\pm$ 0.99	0.85 $\pm$ 1.25	5.94 $\pm$ 7.84	1.80 $\pm$ 1.73	0.24
<b>Parathion</b>	2.21 $\pm$ 0.96	0.70 $\pm$ 0.22	1.39 $\pm$ 1.82	3.98 $\pm$ 2.28	0.43 $\pm$ 0.18	0.02
<b>Trichlorfon</b>	4.10 $\pm$ 1.71	5.76 $\pm$ 5.84	3.05 $\pm$ 3.21	7.71 $\pm$ 6.48	11.31 $\pm$ 0.95	0.12

p@0.05

**Table 4:** Comparison of the Mean Concentration of Pesticides (mg/kg  $\pm$  SE) in samples to MRL

Pesticides	Mean (mg/kg)	MRL for animal feed(mg/kg)
Organochlorine		
<b>p,p'-DDT</b>	0.29 $\pm$ 0.04	0.05*
<b>p,p'-DDE</b>	0.39 $\pm$ 0.06	0.05*
<b>p,p'-DDD</b>	0.14 $\pm$ 0.03	0.05*
<b>Dieldrin</b>	1.47 $\pm$ 0.38	0.01*
<b>Aldrin</b>	-	0.01*
<b>Endosulfan</b>	0.24 $\pm$ 0.04	0.1*
<b>Endosulfan II</b>	0.49 $\pm$ 0.19	0.1*
<b>Endosulfan sulfate</b>	0.23 $\pm$ 0.02	0.1*
<b>Endrin</b>	0.10 $\pm$ 0.02	0.01*
<b>Endrin aldehyde</b>	0.84 $\pm$ 0.22	0.02*
<b>Endrin ketone</b>	0.95 $\pm$ 0.37	-
<b>Methoxychlor</b>	1.83 $\pm$ 0.44	0.5*
<b>Heptachlor</b>	0.63 $\pm$ 0.06	0.01*
Organophosphorus		
<b>Coumaphos</b>	0.25 $\pm$ 0.01	0.5**
<b>Diazinon</b>	1.95 $\pm$ 0.10	0.01*
<b>Dichlorvos</b>	1.44 $\pm$ 0.11	0.01*
<b>Dimethoate</b>	0.91 $\pm$ 0.06	0.05**
<b>Ethyl parathion</b>	0.76 $\pm$ 0.04	0.05 - 2.00#
<b>Malathion</b>	2.20 $\pm$ 0.06	0.05*
<b>Parathion</b>	1.87 $\pm$ 0.11	0.01*
<b>Trichlorfon</b>	6.04 $\pm$ 0.35	0.05**

\*\*FAO/WHO 2006; \*EC 2005; # Codex Alimentarius 1996

The concentrations of endrin and endrin aldehyde reported are higher than the values of 4.15 $\pm$ 0.63 and 12.99 $\pm$ 1.57  $\mu$ g/kg reported by Panseri *et al.*, (2013). Dieldrin, endrin, endrin aldehyde, endrine ketone are metabolites of aldrin degradation. The absence of aldrin in our result is contrary to the concentration of 4.53 $\pm$ 1.12  $\mu$ g/kg reported by Panseri *et al.*, (2013). Thus the presence of aldrin metabolites only in our report indicates that aldrin was long used. Aldrin is of much higher toxicity than other OCPs (Panseri *et al.*,

2013). However, presence of endrin shows epoxidation and conversion of aldrin to dieldrin and endrin. Dieldrin is more persistent (relative to aldrin) because of its low biotransformation and evaporation. The levels of endosulfan and endosulfan sulphate reported are consistent with the range of 0.028 - 0.42 mg/kg reported by Kumar *et al.*, 2019 and 0.42 mg/kg (Aulakh *et al.*, 2006) respectively. The presence of the endosulfan group shows the consistent use of endosulfan despite its ban since 2007. The most

commonly detected OCPs in animal feed are DDT > heptachlor > methoxychlor > aldrin > aldrin metabolites (Panseri *et al.*, 2013). All these pesticides are present in our samples in the order Methoxychlor > Dieldrin > Endrin ketone > Endrine aldehyde > Total DDT > Heptachlor for OCPs. OPs are biodegradable and less persistent relative to OCPs, but its presence in feedstuffs has been severally reported (Khilare *et al.*, 2017; Singh *et al.*, 1998; Chinniah *et al.*, 1998). OPs persist in food of animal origin (Kumar *et al.*, 2019). Previous study reported wide and intensive use of OPs in Sudan because of its significant levels (above MRL) in meat from slaughter houses in Sudan (Sara *et al.*, 2019). These reports are in agreement with our work. We identified the presence of OP residues at levels above the MRL in our samples. From table 4, the following selected OP residues were identified; coumaphos, diazinon, dichlorvos, dimethoate, ethyl parathion, malathion, parathion, trichlorfon with a mean concentration of  $0.25 \pm 0.10$ ,  $1.95 \pm 0.10$ ,  $1.44 \pm 0.11$ ,  $0.91 \pm 0.06$ ,  $0.76 \pm 0.04$ ,  $2.20 \pm 0.06$ ,  $1.87 \pm 0.11$ ,  $6.04 \pm 0.35$  mg/kg respectively. These values are above the MRL values for the pesticides. There is a paucity of information in the literature on OP pesticide use, its residual level and MRL in animal feed. Some of the MRL values in literature are for feedstuffs/feed ingredients. The mean malathion concentration in our report  $2.20 \pm 0.06$  mg/kg is higher than the mean value of  $6.84 \mu\text{g/kg}$  reported by (Khilare *et al.*, 2017). Malathion  $80.45$ , dimethoate  $0.01$ , diazinon  $4.72 \mu\text{g/kg}$  have been reported in cattle feed (Fagnani *et al.*, 2011). However, higher mean concentration of OP residues of  $4.9 - 22.11$  mg/kg has also been reported (Mahugija *et al.*, 2018). Dichlorvos is a more active form of trichlorfon. Dichlorvos is a cholinesterase inhibitor. Trichlorfon biotransform (rearranges) *In vivo* to dichlorvos. High levels ( $0.03 - 4.60$  mg/kg) of dichlorvos have been reported in Nigerian food crops, (Adewunmi and Fapohunda, 2018). The order of presence of OPs in our samples is Trichlorfon > Malathion > Diazinon. Several studies have reported high contamination levels of pesticides (especially OCPs) residues in Nigerian food crops and feedstuffs mostly cereals, oil seed, bran, agro-industrial by products (Ogah *et al.*, 2011, 2012; Erhunmwunse *et al.*, 2012; Olutona and Aderemi, 2019). We did not see any report yet on pesticide residues in poultry/animal feed in Nigeria. Pesticide residues in food and feeds are potential toxicant to animal and man, and it is against the "One Health" concept. The presence of banned pesticide residues at levels above the MRL in our samples show persistent, indiscriminate and unwholesome use of pesticides in Nigeria despite the government policy on its production and use (Public Health Nigeria, 2019). The high level of pesticide contamination in our report may

also be due to the composition of the feed. Poultry feeds contain variety of contaminated ingredients/feedstuffs (Aulakh *et al.*, 2006). The presence of banned pesticide residues in poultry feed is the main source of exposure to poultry. This has deleterious effect on the health and production efficiency of the birds (Jayaraj *et al.*, 2016; Syromyatnikov *et al.*, 2020). The consumption of pesticide contaminated feed by poultry birds has a cross over effect of pesticide residues in poultry products such as meat and eggs. The presence of pesticide residue in poultry meat and eggs is of public health concerns, as this could be a major source of exposure to humans due to increasing consumption of poultry meat and egg - for its nutritional and nutraceutical properties. The damaging effects of small but continuous pesticide exposure/contamination via food of animal origin to human health have been severally discussed and documented (Khilare *et al.*, 2016, 2017; Panseri *et al.*, 2013, Thompson *et al.*, 2017; Abdollahi *et al.*, 2004; Kumar *et al.*, 2013).

**Conclusion:** Our report on pesticide residues in poultry/animal feeds appears to be the first in Nigeria. The reported high levels of pesticide residues (above MRL) in the poultry feed samples is a risk to animal and human health. This necessitates continuous monitoring of poultry/animal feeds and urgent need to educate farmers, produce merchants and feed producers on responsible use of pesticides, risks associated with pesticide use, observance of Good Agricultural Practices (GAP), Good Storage Practices (GSP) and Good Manufacturing Practices (GMP) respectively.

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