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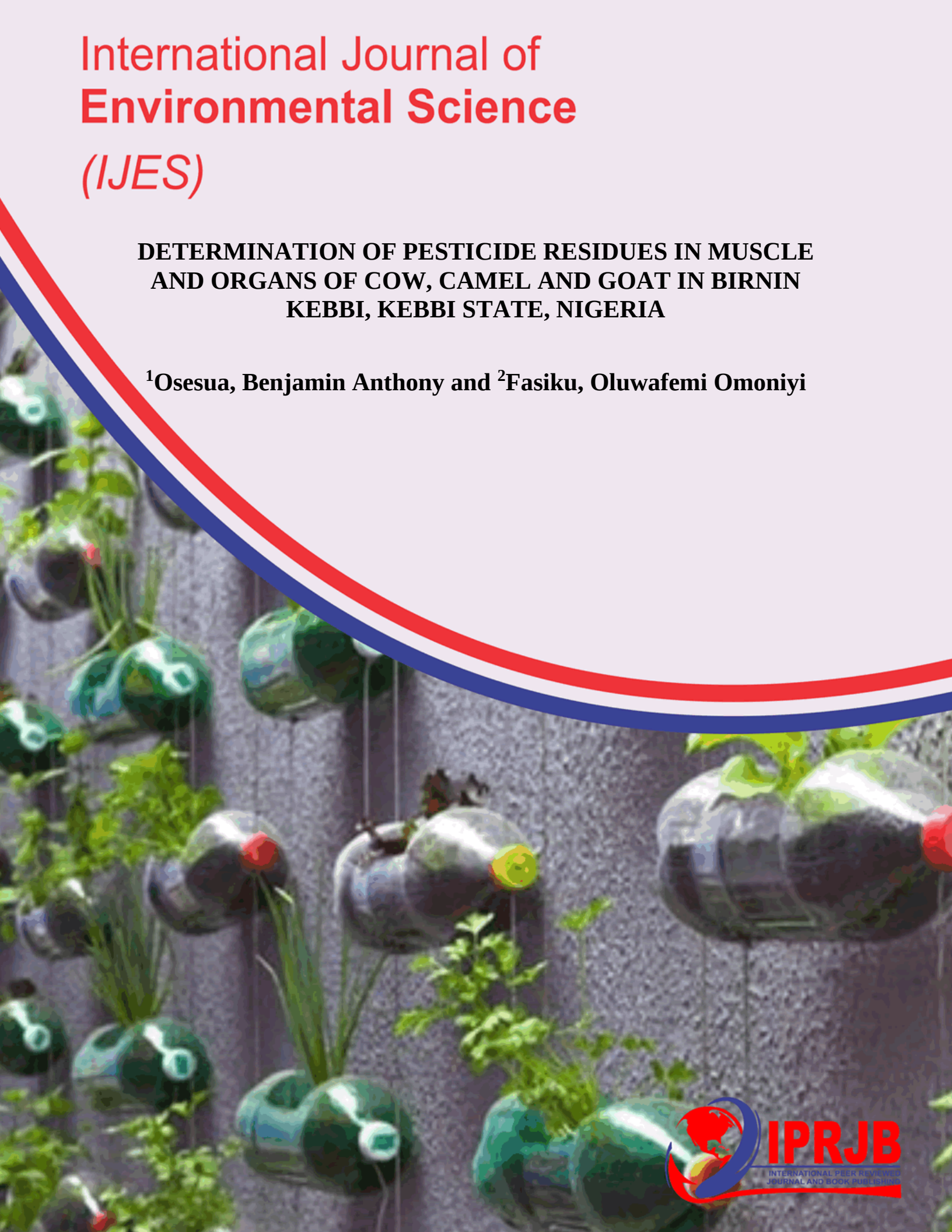

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DETERMINATION OF PESTICIDE RESIDUES IN MUSCLE AND ORGANS OF COW, CAMEL AND GOAT IN BIRNIN KEBBI, KEBBI STATE, NIGERIA.

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Abstract

Pesticide residues in muscle and organs of farm animals (cow, camel and goat) were assessed. Samples of muscle (meat), liver, kidney and heart were collected from central abattoirs in the four emirate councils of Kebbi State in order to determine their level of contamination. Residues were extracted from the samples using standard operating procedures and analysed with GC-MS (Intuvo 9000 model) coupled with ECD. Total pesticide residues in the samples ranged from 0.113 to 2.532µg/Kg (muscle), 0.095 to 2.341µg/Kg (liver), 0.047 to 0.931µg/Kg (kidney and 0.026 to 0.361µg/Kg (heart). Pesticide residues concentrations were in the order; muscle > liver > kidney > heart. Analysis of the results obtained also shown that 12.150% of samples from Yauri emirate were contaminated with pesticide residues, 11.249% were contaminated from Argungu, 7.834% from Zuru and 5.784% from B/Kebbi. Samples of muscle and organs from cow accumulated 47.04% of the detected pesticide residues, Camel accumulated 44.48% of the detected residues and goat muscles and organs accumulated 8.48% of the detected pesticide residues. The concentrations of all the assessed pesticides observed in the muscle and organs were however lower than the recommended maximum residual limits (MRLs).

Keywords: *Residues, Muscle, Organs, Contamination, Organochlorines*

INTRODUCTION

The anxiety amongst farmer to increased food security and eliminate crop pests and diseases has resulted to the accumulation of obsolete and toxic pesticides in the environment (Schwab, Jager, Stoll, Gorgen, Prexterschwab, and Attenburger, 2015). Although the use of pesticides has become overtly accepted as an imperative and integral part of modern agriculture in the control of insects, weed and crop diseases, there is a growing concern on their persistence in the environment and toxicity to non-target organisms. Pesticide residues in food items especially in different parts of meat and organs of livestock are of growing concern due to possible adverse effects on humans (Kelce, Stone, Laws, Gray, Kemppainen, and Wilson, 2015).

Ingestion of contaminated foods especially meat and meat products constitute the most frequent and principal pathway of exposure to pesticides (Nwude, Babayemi, and Abhulimen, 2011). Health effects of pesticides to humans have been documented by several scholars, and the contamination of food especially meat and meat products by pesticide residues have become a topical issue of considerable concern globally (Ross, 2005; Kaulock, 2016; Lebel, Dodin, Ayotte, Marcoux, Ferron, and Dewailly, 2018).

Grazing livestock are exposed to pesticides through direct treatment with pesticides, inhalation of pesticide contaminated air, drinking water contaminated with pesticide residues or through ingestion of contaminated soils and fodder (Kalanzi *et al*., 2011). Nigeria is currently at a crossroad battling with free open grazing, cattle and other livestock are allowed to graze freely and consequently predisposing them to high levels of contaminants in the environment. One of the basic characteristics of pesticides is that they are highly lipophilic which accounts for their massive accumulation in organs and other fatty tissues of animals, thus providing a major route for human exposure when these fatty parts are consumed as food (Saqib *et al*., 2015).

Livestock meat and their products constitute a significant part of most diet in Nigeria, since it is a rich source of protein and nutrients. According to the study reported by FAO. (2004), Nigeria has a high per–capita consumption rate for meat of 8.6 g/person/day. Different parts of domestic and farm animals (muscle and organs) serve as delicacies in most diets of an average Nigerian, since they are readily available and can be consumed in different forms. The growing awareness over pesticide toxicity in the environment has raised serious concerns in the possible accumulation of pesticides in tissues of domestic and livestock animals that serve as food to the majority of the populace (Pardio *et al*., 2012; Nasr *et al*., 2017).

In Nigeria, pesticides are predominantly used by farmers in the Northwestern Region including Kebbi State. In this region pesticide residues have been detected in various types of fish (Osesua *et al*., 2017a), in water and sediment (Osesua *et al.*, 2017b), in blood samples of farmers (Osesua *et al*., 2018) and in fruits and vegetables (Osesua *et al.*, 2019). Livestock and domestic animals reared in this pesticide contaminated environment are therefore prone to accumulating these bio-active chemicals primarily by having contacts with the various environmental samples or feeding directly on pesticide treated foliage. Although residual levels of pesticides in meat and meat products have been documented in several places by different researchers, in Japan (Matumoto e*t al*., 2006), in Indian (Amaraen and Pillale, 2011), in Aragon, Northwestern Spain (Lazaro *et al*., 2018) and Egypt (Sallam *et al*., 2018), there is a dearth of information concerning pesticide residues in meat and organs of livestock in Kebbi state, and to the best of our knowledge no work

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on the determination of pesticide residues in meats and organs of livestock in Kebbi state has been done. It therefore becomes very imperative the need to assess the levels of commonly used pesticides residues in meats and organs of some animals that freely graze in this locality. This will provide a baseline information on the levels of residues in these samples and the possible human health risk associated with consuming meat and meat products from this region.

MATERIALS AND METHODS

Study Site

This study was carried out in Kebbi State, one of the states located in North-West Nigeria. Kebbi state has a population of over three (3) million people who are predominately farmers. Administratively, the state is divided into four (4) emirates councils with headquarters in Birnin-kebbi, Argungu, Yauri and Zuru. An average distance of 100Km separates one emirate council from another. Continuous cropping mode of farming engaged in by the farmers in this region accounts for their heavy reliance on the use of agro-chemicals to improve crops yield.

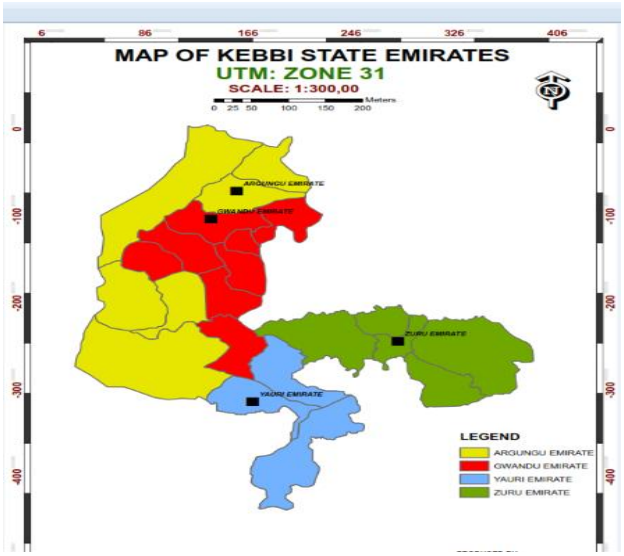

Fig.1: Map of Kebbi State showing the sampling sites

Sample Collection

All experiments using animal specimens were performed according to the guidelines of the Committee of Animal Care and Use, Usmanu Danfodiyo University, Sokoto. The major abattoirs feeding the meat markets in each of the emirate council were sampled according to the method stipulated and developed by FAO/WHO, (2016). Samples of meats (muscles) and organs were collected between the months of July – September. Each sample was taken twice in a month for three (3) months (2x3=6) for each animal part, muscle, heart, liver and kidney (6x4=24) for the three different samples, cow, camel and goat (24x3=72), and for the four different sampling sites of Birnin-Kebbi, Argungu, Yauri and Zuru (72x4=288). A total of 288 samples of muscles, hearts, liver and kidney were taken from cow, camel and goat. Samples were wrapped in polythene zipper

bags, labeled, placed on ice box and taken to the laboratory. Samples were then stored at -20 °C prior to analysis.

Pesticides Standards:

Pure pesticides (DDT, endosulfan, HCH, parathion, chlorpyrifos, endrin, fenitrothion, methylparathion, dichlorvos, diclorvos, heptachlor, aldrin, dieldrin and glysophate) were purchased from Bristol Scientific Company Limited, a subsidiary of Sigma Aldrich in Nigeria. Standard solution of 0.5ppm, 1ppm, 3ppm, and 5ppm were prepared in methanol for analyses using GC-MS coupled with $_{63}$Ni electron capture detector.

Sample Preparation, Extraction, Clean-up and Analysis

Pesticide standard preparation were done according to the method developed by Murphy, (2011) and modified by Pardio *et al*., (2012), extraction, clean-up and analysis were performed according to standard procedures reported by Ahmed *et al*., (2010); Peighambarzadeh *et al*., (2011); Shahzadi *et al*., (2013). Samples were each weighed and homogenized using a meat blender. Fifty (50) g of the homogenized samples was placed into a beaker containing 30 g anhydrous sodium sulfate and thoroughly mixed. This mixture was then extracted using 150 cm^3 of acetone-hexane mixture in a soxhlet extractor. The extracts were filtered and concentrated using a rotary evaporator at 40 °C.

Clean-up of the sample was done by quantitatively transferring the extracts into a chromatographic column which contained one gram (1 g) activated florisil (60–100mesh) and 1 g layer of anhydrous sodium sulfate to absorb moisture. Ten (10) cm^3 of hexane was used to condition the column before transferring one (1 cm^3) each of the sample blanks into the column. The chromatographic column was eluted with 10 cm^3 of hexane at a rate of 1–2 cm^3/min. The eluate was then concentrated to dryness using a rotary evaporator holding the temperature at 40 °C. One (1) cm^3 ethyl acetate was used to dissolve the residue and then transferred into 2 cm^3 injection vials ready for analysis with GC-MS (Intuvo 9000 model) coupled with electron capture detector.

Quality Control

All analytes were subjected to stringent quality control methods. Prior to sample analysis, the instruments were calibrated with calibration standards. The target analytes were identified and quantified by comparing the retention times and peak area of the sample with those of the calibrated external standards (reference standards). The detection of linearity was assessed using linear regression analysis of multi-level calibration curves for each analyte, while the limit of detection (LOD) and limit of quantification (LOQ) were calculated from the curve obtained from the recovery studies. The correlation coefficient (r) of the standard calibration curves ranged from 0.9723 to 0.9997, while the LOD and LOQ ranged from 0.00001 to 0.001 µg/kg and 0.0001 to 0.01 µg/kg respectively. The average recoveries of the target analytes ranged from 80 to 100% with relative standard deviation (RSD) being less than 10%, indicating that the analytical procedures were sufficient (Su, 2008).

Statistical Analysis

Data collected for each emirate council abattoir were summarized separately using descriptive statistics (Microsoft Excel 7.0 programe). Statistical differences between the different target

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pesticides residues in the various meat parts were evaluated using Analysis of variance (ANOVA), SPSS 20 software and $p < 0.05$ was considered significant.

RESULTS AND DISCUSSION

Contamination Profile of Pesticide Residues in the Various Tissues

Pesticides application in modern agriculture is extremely essential if the food need of the ever-growing population is to be met. However, pesticide contamination in edible tissues of livestock are of growing concern because of their accumulative properties and health risks posed to man (Lebel *et al*., 2018). Since most pesticides are lipophilic, the lipid content was used for the estimation of pesticide residual concentration in all the tissues. The mean percentage concentration of lipids in tissue samples were 4.8% (muscle), 4.7% (liver), 2.8% (kidney) and 3.1% (heart). The observed results of lipid concentration are similar to that reported in previous studies (Ahmed *et al*., 2010: Blankson-Arthur *et al*., 2012: Pardio *et al*., 2012). Varying levels of pesticide contamination in edible tissues of livestock and other domestic animals from selected abattoirs in Kebbi State, Nigeria were observed. The concentrations of total pesticide residues (µg/kg) from the abattoirs in the four emirate councils are shown in Tables 1-4.

Table 1: Mean Conc. (µg/kg) of Organochlorine (OC) and Organophosphorus (OP) residues in meat and organ from abattoir in B/Kebbi

Pesticides	Cow				Camel				Goat			
(µg/kg)	Ms	Li	Kd	Ht	Ms	Li	Kd	Ht	Ms	Li	Kd	Ht
ORGANOCHLORINE												
Op′ DDT	0.312±0.007	0.101±0.002	0.009±0.002	0.006±0.001	0.239±0.099	0.223±0.081	0.031±0.004	0.043±0.002	0.047±0.009	0.012±0.003	0.009±0.002	0.007±0.002
Pp DDD	0.187±0.011	0.077±0.019	0.022±0.004	0.002±0.001	0.110±0.003	0.065±0.005	0.009±0.002	0.003±0.001	0.005±0.002	0.003±0.001	0.003±0.001	0.002±0.001
Op DDE	0.287±0.077	0.213±0.008	0.013±0.009	0.008±0.003	0.163±0.091	0.041±0.005	0.044±0.008	0.021±0.003	0.044±0.004	0.006±0.003	0.005±0.001	0.005±0.002
γ-HCH	0.331±0.022	0.135±0.030	0.023±0.007	0.009±0.002	0.011±0.004	0.008±0.004	0.008±0.003	0.004±0.001	0.007±0.003	0.007±0.004	0.004±0.001	0.002±0.001
Endosulfan	0.006±0.002	0.003±0.001	ND	ND	0.008±0.003	0.005±0.002	ND	ND	0.003±0.001	ND	ND	ND
Heptachlor	0.008±0.003	0.006±0.002	0.006±0.001	ND	0.008±0.002	0.009±0.003	ND	ND	0.007±0.002	0.005±0.002	ND	ND
Aldrin	0.024±0.008	0.021±0.004	0.009±0.003	0.006±0.002	0.011±0.007	0.013±0.008	0.010±0.004	0.008±0.002	0.013±0.007	0.011±0.006	0.007±0.003	0.005±0.003
Dieldrin	0.017±0.006	0.017±0.003	0.007±0.001	0.007±0.004	0.012±0.005	0.015±0.007	0.010±0.006	0.009±0.001	0.009±0.004	0.007±0.003	0.004±0.002	0.004±0.001
Endrin	0.013±0.008	0.012±0.005	0.004±0.001	0.005±0.002	0.015±0.006	0.017±0.009	0.009±0.005	0.008±0.003	0.010±0.004	0.011±0.007	0.006±0.001	0.007±0.002
ORGANOPHOSPHORUS												
Chlorpyrifos	0.271±0.011	0.211±0.041	0.033±0.006	0.009±0.002	0.277±0.012	0.233±0.099	0.087±0.009	0.007±0.002	0.133±0.014	0.121±0.040	0.066±0.005	0.007±0.002
M/parathion	0.008±0.002	0.007±0.002	0.004±0.001	ND	0.012±0.008	0.011±0.006	0.008±0.004	0.005±0.002	0.004±0.001	0.004±0.001	0.003±0.001	0.003±0.002
Parathion	0.005±0.003	0.007±0.003	0.005±0.002	0.002±0.001	0.009±0.004	0.009±0.003	0.004±0.001	0.004±0.002	0.003±0.001	0.003±0.001	ND	ND
Glyphosate	0.244±0.021	0.241±0.011	0.176±0.009	0.055±0.007	0.231±0.007	0.201±0.012	0.087±0.008	0.005±0.002	0.009±0.003	0.007±0.003	0.004±0.001	0.004±0.003
Fenitrothion	0.005±0.002	0.002±0.001	ND	ND	0.006±0.003	ND	ND	ND	ND	ND	ND	ND
Dichlorvos	0.002±0.001	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Σpesticide	1.720±0.184	1.053±0.132	0.311±0.046	0.109±0.025	1.112±0.254	0.850±0.244	0.307±0.054	0.117±0.021	0.294±0.055	0.197±0.074	0.111±0.018	0.046±0.019

NOTE: ND=Not Detected, Ms=muscle, Li=liver, Kd=kidney, Ht=heart. Mean ± SD of triplicate result.

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Table 2: Mean Conc. (µg/kg) of Organochlorine (OC) and Organophosphorus (OP) residues in meat and organ from abattoir in Argungu

Pesticides	Cow				Camel				Goat			
(µg/kg)	Ms	Li	Kd	Ht	Ms	Li	Kd	Ht	Ms	Li	Kd	Ht
ORGANOCHLORINE												
Op' DDT	0.221±0.011	0.202±0.009	0.110±0.007	0.077±0.004	0.312±0.044	0.299±0.077	0.112±0.043	0.055±0.008	0.041±0.004	0.042±0.006	0.006±0.002	0.003±0.002
Pp DDD	0.189±0.009	0.170±0.005	0.097±0.007	0.055±0.006	0.200±0.033	0.122±0.008	0.099±0.044	0.051±0.004	0.054±0.009	0.052±0.011	0.032±0.004	0.004±0.003
Op DDE	0.330±0.088	0.299±0.054	0.144±0.005	0.081±0.008	0.302±0.111	0.300±0.004	0.203±0.081	0.062±0.010	0.091±0.006	0.087±0.008	0.042±0.006	0.012±0.007
γ-HCH	0.351±0.101	0.304±0.110	0.233±0.033	0.044±0.005	0.302±0.010	0.299±0.006	0.190±0.037	0.077±0.009	0.093±0.004	0.091±0.006	0.055±0.005	0.009±0.005
Endosulfan	0.007±0.002	0.005±0.002	ND	ND	0.009±0.003	0.004±0.001	ND	ND	0.003±0.001	ND	ND	ND
Heptachlor	0.005±0.002	0.005±0.001	0.002±0.001	ND	0.011±0.005	0.009±0.003	ND	ND	ND	ND	ND	ND
Aldrin	0.112±0.039	0.100±0.022	0.077±0.005	0.008±0.005	0.154±0.012	0.120±0.007	0.078±0.006	0.012±0.007	0.044±0.003	0.043±0.005	0.008±0.003	0.005±0.003
Dieldrin	0.101±0.031	0.113±0.007	0.081±0.009	0.010±0.008	0.122±0.023	0.109±0.055	0.091±0.008	0.004±0.002	0.063±0.004	0.041±0.004	0.011±0.006	0.007±0.003
Endrin	0.009±0.003	0.009±0.004	0.004±0.002	0.003±0.001	0.009±0.004	0.008±0.003	ND	ND	0.005±0.002	0.005±0.003	0.003±0.001	ND
Organophosphorus												
Chlorpyrifos	0.322±0.044	0.312±0.033	0.066±0.006	0.041±0.005	0.321±0.011	0.301±0.031	0.061±0.008	0.032±0.006	0.067±0.012	0.052±0.013	0.033±0.006	0.006±0.002
M/parathion	0.005±0.003	0.002±0.001	ND	ND	0.007±0.003	0.003±0.001	ND	ND	ND	ND	ND	ND
Parathion	0.008±0.004	0.008±0.003	0.004±0.002	ND	0.005±0.002	0.005±0.002	0.003±0.001	ND	0.003±0.001	ND	ND	ND
Glyphosate	0.211±0.071	0.199±0.077	0.110±0.008	0.042±0.004	0.261±0.033	0.206±0.035	0.093±0.009	0.006±0.004	0.088±0.007	0.041±0.004	0.006±0.003	0.004±0.002
Fenitrothion	0.006±0.003	ND	ND	ND	0.007±0.003	0.003±0.001	ND	ND	ND	ND	ND	ND
Dichlorvos	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
∑pesticide	1.877±0.411	1.728±0.328	0.928±0.085	0.361±0.046	2.022±0.297	1.788±0.314	0.931±0.235	0.299±0.050	0.552±0.053	0.454±0.060	0.196±0.036	0.050±0.027

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Table 3: Mean Conc. (µg/kg) of Organochlorine (OC) and Organophosphorus (OP) residues in meat and organ from abattoir in Yauri

Pesticides	Cow				Camel				Goat			
(µg/kg)	Ms	Li	Kd	Ht	Ms	Li	Kd	Ht	Ms	Li	Kd	Ht
ORGANOCHLORINE												
Op' DDT	0.410±0.055	0.342±0.011	0.078±0.043	0.044±0.009	0.398±0.091	0.377±0.041	0.091±0.008	0.031±0.004	0.054±0.005	0.055±0.007	0.030±0.004	0.007±0.005
Pp DDD	0.228±0.081	0.214±0.071	0.099±0.007	0.052±0.009	0.226±0.006	0.231±0.009	0.076±0.007	0.009±0.006	0.008±0.005	0.009±0.005	0.005±0.003	0.003±0.002
Op DDE	0.338±0.049	0.320±0.009	0.091±0.004	0.047±0.007	0.318±0.082	0.259±0.005	0.057±0.006	0.012±0.006	0.066±0.008	0.067±0.005	0.011±0.009	0.008±0.004
γ-HCH	0.422±0.021	0.344±0.055	0.060±0.008	0.009±0.003	0.423±0.088	0.399±0.041	0.044±0.005	0.006±0.001	0.055±0.006	0.041±0.003	0.007±0.003	0.004±0.002
Endosulfan	0.078±0.003	0.074±0.004	0.004±0.002	0.003±0.001	0.081±0.009	0.079±0.008	0.055±0.003	0.012±0.004	0.006±0.002	0.003±0.001	ND	ND
Heptachlor	0.055±0.004	0.042±0.005	0.003±0.001	ND	0.061±0.004	0.063±0.007	ND	ND	0.004±0.001	0.003±0.002	ND	ND
Aldrin	0.081±0.007	0.080±0.004	0.007±0.004	0.004±0.002	0.093±0.005	0.076±0.009	0.006±0.003	0.006±0.004	0.009±0.005	0.007±0.005	0.006±0.004	0.004±0.001
Dieldrin	0.078±0.011	0.073±0.006	0.008±0.003	0.005±0.003	0.079±0.007	0.081±0.009	0.008±0.004	0.003±0.001	0.007±0.003	0.005±0.003	ND	ND
Endrin	0.093±0.011	0.067±0.006	0.008±0.004	0.005±0.003	0.099±0.024	0.093±0.006	0.006±0.002	0.003±0.001	0.003±0.001	ND	ND	ND
Organophosphorus												
Chlorpyrifos	0.440±0.008	0.412±0.071	0.081±0.007	0.008±0.003	0.482±0.021	0.441±0.009	0.102±0.055	0.091±0.005	0.055±0.004	0.062±0.006	0.007±0.002	0.006±0.002
M/parathion	0.022±0.004	0.021±0.007	0.007±0.002	0.007±0.003	0.023±0.009	0.013±0.003	0.007±0.002	0.004±0.003	0.031±0.007	0.009±0.004	ND	ND
Parathion	0.037±0.009	0.035±0.004	0.006±0.002	0.005±0.004	0.022±0.005	0.015±0.005	0.009±0.004	0.005±0.002	0.005±0.001	0.005±0.002	0.003±0.001	ND
Glyphosate	0.246±0.022	0.211±0.033	0.110±0.009	0.100±0.008	0.203±0.077	0.214±0.033	0.031±0.009	0.008±0.002	0.122±0.040	0.102±0.007	0.081±0.011	0.020±0.005
Fenitrothion	0.004±0.001	ND	ND	ND	0.006±0.002	ND	ND	ND	ND	ND	ND	ND
Dichlorvos	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
∑pesticide	2.532±0.286	2.235±0.286	0.562±0.096	0.289±0.054	2.514±0.430	2.341±0.185	0.493±0.108	0.190±0.039	0.425±0.088	0.362±0.047	0.150±0.037	0.052±0.021

NOTE: ND=Not Detected, Ms=muscle, Li=liver, Kd=kidney, Ht=heart. Mean ± SD of triplicate result.

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Table 4: Mean Conc. (µg/kg) of Organochlorine (OC) and Organophosphorus (OP) residues in meat and organ from abattoir in Zuru

Pesticides	Cow				Camel				Goat			
(µg/kg)	Ms	Li	Kd	Ht	Ms	Li	Kd	Ht	Ms	Li	Kd	Ht
ORGANOCHLORINE												
Op' DDT	0.280±0.021	0.214±0.077	0.091±0.006	0.006±0.002	0.229±0.012	0.152±0.023	0.008±0.003	0.006±0.002	0.012±0.005	0.011±0.003	0.005±0.002	0.004±0.002
Pp DDD	0.113±0.056	0.110±0.009	0.007±0.003	0.004±0.001	0.112±0.081	0.116±0.012	0.098±0.010	0.007±0.002	0.009±0.003	0.009±0.004	0.005±0.002	0.004±0.001
Op DDE	0.279±0.043	0.224±0.007	0.014±0.004	0.005±0.002	0.150±0.044	0.125±0.004	0.007±0.002	0.005±0.002	0.011±0.008	0.010±0.004	0.008±0.003	0.006±0.002
γ-HCH	0.181±0.054	0.147±0.009	0.009±0.003	0.004±0.001	0.149±0.011	0.120±0.055	0.014±0.007	0.008±0.003	0.008±0.004	0.008±0.002	0.004±0.002	ND
Endosulfan	0.044±0.004	0.021±0.005	0.008±0.003	ND	0.034±0.005	0.027±0.007	0.005±0.001	ND	0.005±0.002	0.005±0.001	ND	ND
Heptachlor	0.031±0.003	0.023±0.007	0.011±0.004	0.004±0.001	0.033±0.005	0.031±0.004	ND	ND	0.004±0.001	0.004±0.002	0.003±0.001	ND
Aldrin	0.130±0.009	0.103±0.004	0.009±0.002	ND	0.133±0.041	0.140±0.004	0.007±0.001	0.005±0.002	0.010±0.005	0.009±0.002	ND	ND
Dieldrin	0.100±0.004	0.009±0.002	0.006±0.001	0.003±0.001	0.120±0.007	0.110±0.005	0.009±0.001	ND	0.005±0.001	0.004±0.001	ND	ND
Endrin	0.131±0.007	0.023±0.002	0.008±0.002	0.007±0.002	0.134±0.003	0.102±0.004	0.008±0.001	0.004±0.001	0.006±0.001	0.004±0.001	0.003±0.001	ND
Organophosphorus												
Chlorpyrifos	0.312±0.009	0.300±0.034	0.056±0.006	0.007±0.002	0.322±0.011	0.320±0.004	0.051±0.005	0.011±0.003	0.007±0.001	0.007±0.002	0.005±0.001	0.004±0.001
M/parathion	0.061±0.007	0.063±0.005	0.006±0.001	ND	0.052±0.004	0.047±0.003	ND	ND	0.007±0.002	0.005±0.001	ND	ND
Parathion	0.067±0.003	0.048±0.004	0.007±0.001	0.005±0.001	0.071±0.004	0.065±0.003	0.008±0.003	ND	0.007±0.001	0.007±0.002	0.004±0.001	ND
Glyphosate	0.277±0.004	0.213±0.008	0.066±0.005	0.031±0.003	0.258±0.021	0.211±0.007	0.033±0.003	0.012±0.004	0.022±0.004	0.012±0.007	0.010±0.006	0.008±0.003
Fenitrothion	0.006±0.001	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Dichlorvos	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
∑pesticide	2.012±0.225	1.498±0.173	0.298±0.041	0.076±0.016	1.797±0.249	1.566±0.135	0.248±0.037	0.058±0.019	0.113±0.038	0.095±0.032	0.047±0.019	0.026±0.009

NOTE: ND=Not Detected, Ms=muscle, Li=liver, Kd=kidney, Ht=heart. Mean ± SD of triplicate result.

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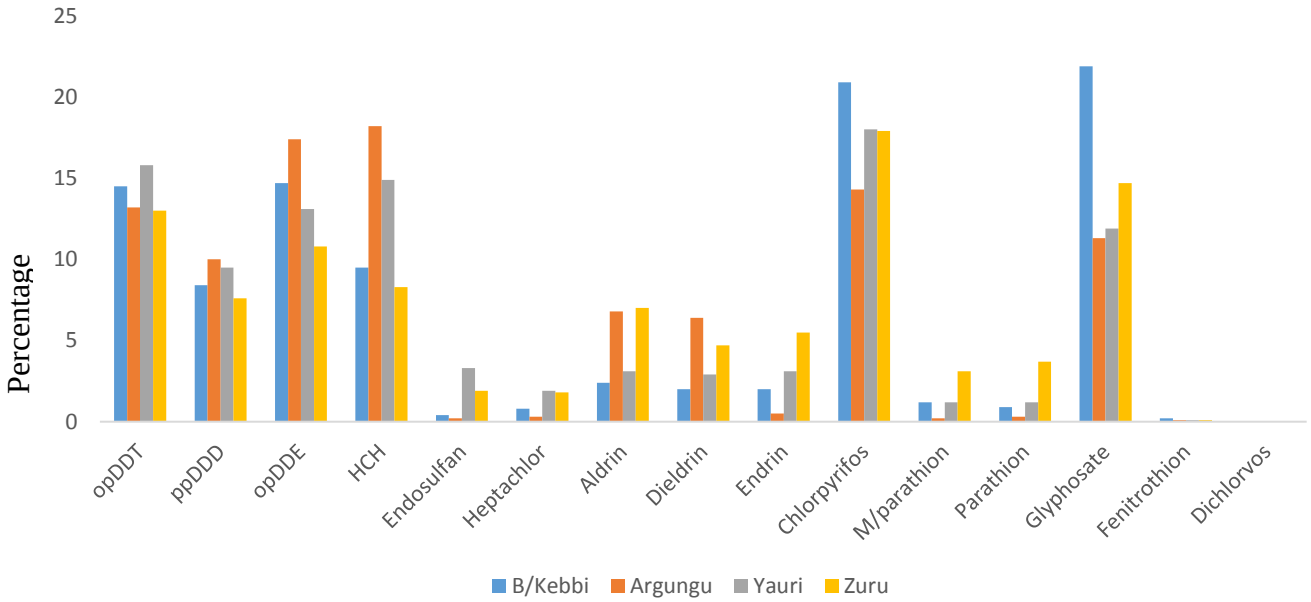

Fig. 2: Percentage distribution of pesticide residues in samples around the various emirate councils

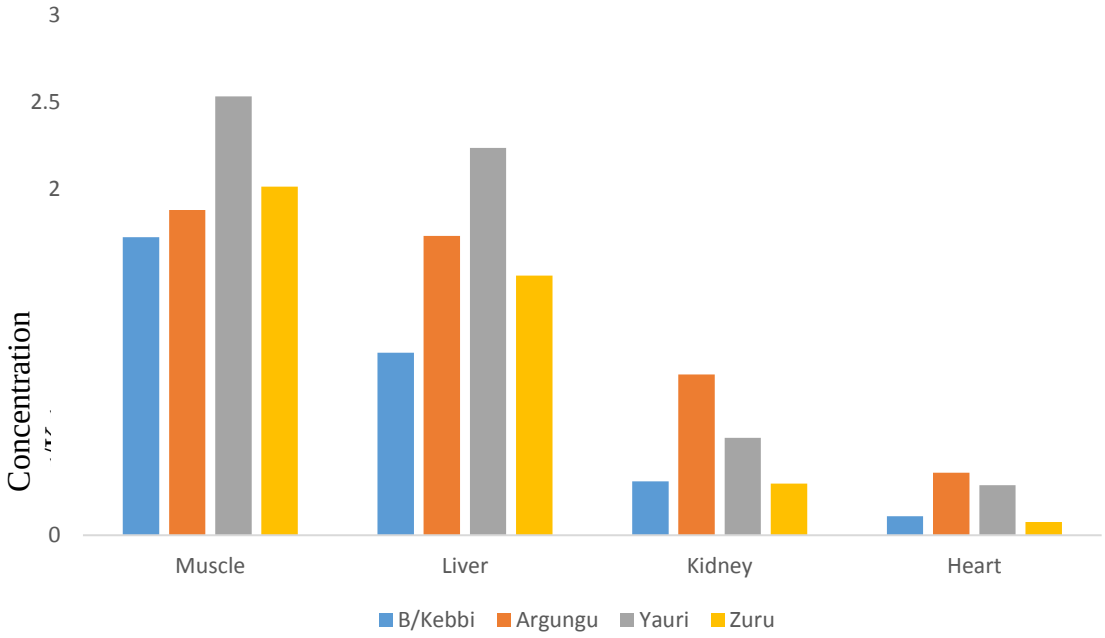

Fig.3: Mean Distribution of Pesticide Residues in Cow Muscle and organs

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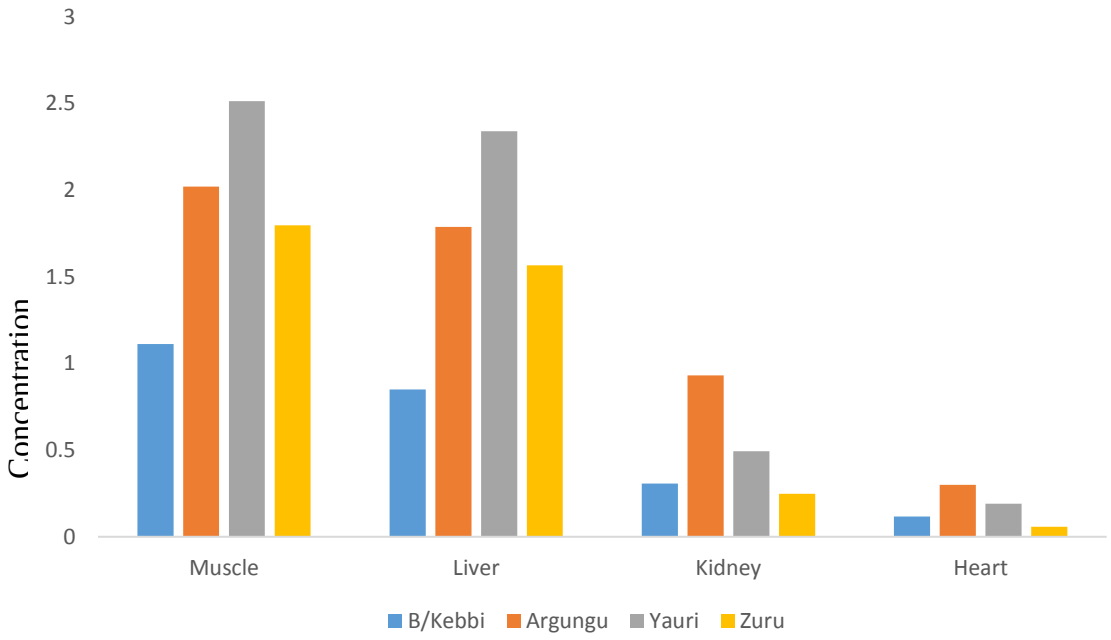

Fig.4: Mean Distribution of Pesticide Residues in Camel Muscle and organs

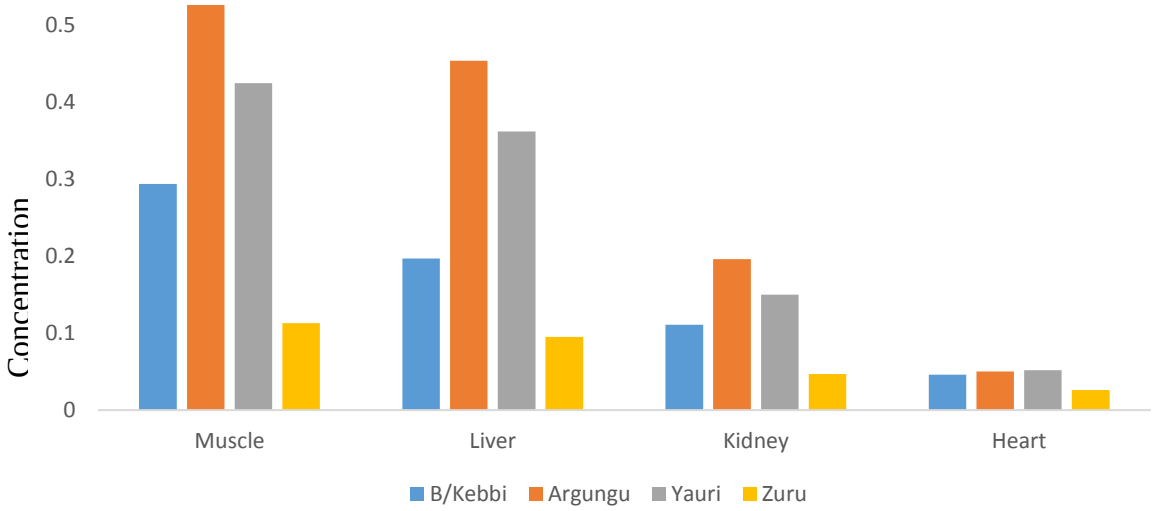

Fig.5: Mean Distribution of Pesticide Residues in Goat Muscle and organs

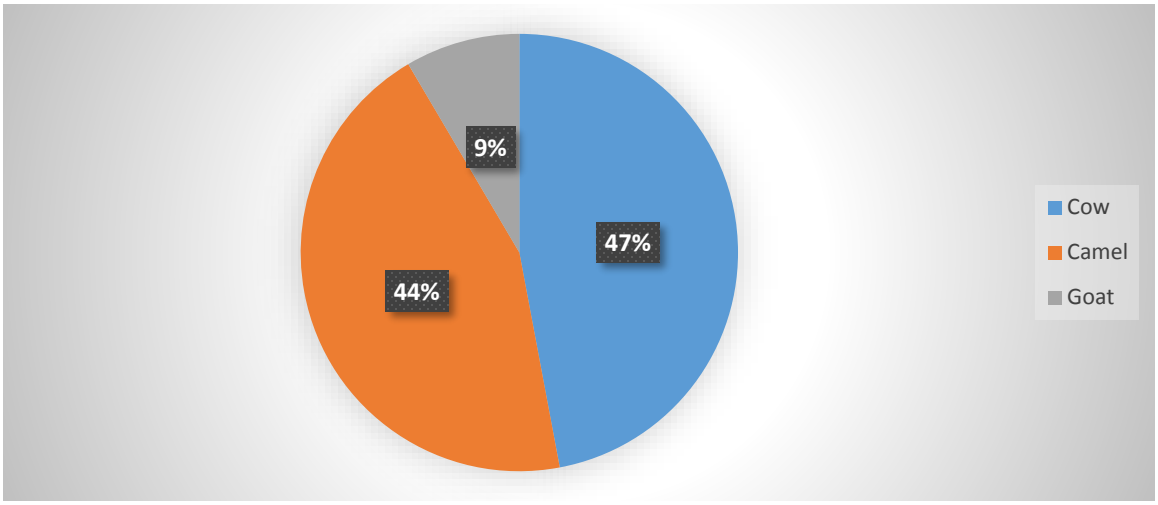

Fig. 6: Accumulative potentials of pesticide residues by the various animals.

DISCUSSION

In Table 1, the highest mean concentration (1.720±0.184µg/Kg) of pesticide residues was found in the muscle of cow, and the lowest mean concentration (0.046±0.019µg/Kg) was detected in the heart of goat. Analysis of samples from Argungu abattoir revealed the highest mean concentration (2.022±0.297µg/Kg) of residues in the muscle of camel (Table 2). Tables 3 and 4 with the highest mean concentration (2.532±0.286µg/Kg and 2.012±0.225µg/Kg) of residues respectively were detected in the muscle of cow. The results reflect different levels of pesticide residues in the muscle, liver, kidney and heart samples of cow, camel and goat. Residual pesticide concentrations in the tissues were in the order: muscle > Liver > Kidney > heart. The difference in the levels of pesticides in the tissues of these animals may be attributed to the levels of contaminants in pastures where these animals graze/drink, the type of husbandry practices, the quantity of contaminated folder consumed, the physical and chemical properties of the pesticide and also the location or proximity of the slaughter houses or abattoirs to pesticide contamination (Hiba, 2015). The muscle was observed to have the highest residual pesticide concentration compared to the other tissues, concentrations were however not statistically significant ($p > 0.05$, $F = 1.54$). Higher levels in the muscle may be due to the high lipid content due to the overlying adipose tissues which serve as primary site for pesticide accumulation (Khalid *et al*., 2013; Aulakh *et al*., 2016).

DDT

DDT was detected in all tissue samples analysed. The highest mean concentration (0.410±0.055µg/Kg) was detected in the muscle of cow from Yauri region (Table 3) with percentage distribution of 15.8% Fig.2. The distribution pattern was in the order; muscle > Liver > Kidney > heart. This result is similar to the study reported by Mahmound *et al* (2013), in the muscle and organ samples of buffalo from Egypt with mean concentration of 62.83 ng/g lipid weight. Much higher concentrations of DDT have been reported in milk samples (Kaphalia *et al*., 2010), muscle (Ahmed *et al*., 2010; Saschenbrecker, 2016), liver, kidney and muscle tissues (Pardio *et al*., 2012). The mean concentrations of residues observed in this study

were however higher than what was obtained in liver and kidney tissues of buffalo from Egypt (Mahmound *et al*., 2013). Although the use of DDT has been banned in Nigeria, residual concentrations in tissues of livestock observed in this study could be attributed to its persistence in the environment or due to illegal and indiscriminate recent use. The reported concentrations of individual DDT in this study were however lower than the recommended MRLs as stipulated by the Japan Food Chemical Research Foundation (JFCRF, 2016). The existence of DDT along with its metabolites DDD and DDE in this study could be an indication of both past and recent applications.

DDD

DDD, one of the isomers of DDT was detected in all the samples analysed (Tables 1-4), with the highest mean concentration (0.231±0.009µg/Kg) found in the liver of camel from the Yauri emirate council (Table 3). Concentrations of DDD residues in the liver were higher than other organs, being 1.1 times higher than residues concentrations in the muscle, 7.6 times higher than concentrations in the kidney and 18.5 times higher than concentrations detected in the heart. The differences in tissue concentrations were however not statistically significant ($P > 0.05$, $F = 3.65$). Higher concentrations of residues detected in the liver may be attributed to the fact that the liver is the primary site of detoxification of xenobiotics (Hotton *et al*., 2011; Khalid *et al*., 2013). This result is similar to the residual concentrations reported by Manirakiza *et al*., (2002) and Glynn *et al*., (2007) who reported DDD concentrations in adipose tissues (0.7 µg/kg lipid) and fat (0.6 µg/kg fat) of cattle from Sweden and Sene-Gambian region respectively. However, higher concentrations (53 µg/kg fat basis) was reported in Ghana by Darko and Acquaah (2007), in their study of organochlorine pesticide residues in meat. The high prevalence of DDD in the muscles and organs observed in this study may be attributed to the high volatility of this product which increases uptake via deposition or sorption from the atmosphere onto pasture surface which in turn is consumed by farm animals or by direct treatment with pesticides (Pardio et al., 2012).

DDE

DDE is the most stable DDT isomer, it has slow elimination rate and thus has the tendency to accumulate in animal tissues over time as compared to other isomers (Pardio *et al*., 2012; Dogheim *et al*., 2016). DDE was detected in all the samples analysed. The highest mean concentration (0.338±0.049µg/Kg) was detected in the muscle of cow from the Yauri council (Table 3). Concentrations observed in this study were however the highest amongst the assessed DDT isomers. However, the differences in tissue concentrations among the tissues were not significant ($P > 0.05$, $F = 0.51$). Higher concentrations in the muscle may be attributed to the fact that DDE is known to be highly lipophilic (Pardio *et al*., 2012), hence increased accumulation in the muscle tissue which was more fatty due to the overlaying adipose tissues. DDE concentrations observed in this study is comparable with the findings of Manirakiza et al., (2002), who reported DDE concentrations (0.2 µg/kg fat) in cattle fat samples from butchers and abattoirs in Sene-Gambian region. The mean residues concentration in the liver tissues obtained in this study were however lower than levels reported by (Covaci *et al*., 2014) in liver tissues of pork from Romania. Residual Levels were similar to what was obtained for DDD isomer in which the muscle and the liver had the highest concentration. DDT and its metabolites (DDD and DDE) are known for their lipophilicity, hence higher concentration in the fatty organs could be attributed to the fact that these

organs have higher concentrations of fat compared to the other organs (kidney and heart). The differences in the organ concentrations were however not significant ($P > 0.05$, $F = 1.71$). DDD+DDE/DDT ratios below 1 are often used as indicators of fresh input of DDT into the environment (Kozul *et al*., 2008; Pardio *et al*., 2012). In this study the mean DDD+DDE/DDT ratios in all the tissues assessed were above 1, indicating that there was no fresh input of the parent DDT into the environment. There was also no statistical significance ($p > 0.05$, $F = 0.31$) in the DDD+DDE/DDT ratios among the organs. The residue concentrations of DDE observed in the muscle, liver, kidney and heart tissues were below the MRL recommended by the Japan Food Chemical Research Foundation (JFCRF,2016) and the European Union (EU, 2015).

γ-Hexachlorocyclohexane (γ-HCH)

The mean concentrations of γ-HCH in muscle, liver, kidney and heart of all the animals are presented in Tables 1-4. and further illustrated in Fig.2. The highest mean concentrations of γ-HCH was found in the muscle (0.423 ± 0.088 µg/kg) of camel from the Yauri council. Concentrations of γ-HCH were not statistically significant ($p > 0.05$, $F = 0.05$) amongst the organs. The result of this study is similar with the findings of Mahmound *et al*., (2013), who reported higher concentrations of γ-HCH in tissues of buffalo from slaughter houses in Egypt. The observed residual levels of γ-HCH in the organs from this study were lower than concentrations in reported studies on various parts of food animals (Kaphalia *et al.*, 2005; Nath *et al*., 2008; Singh and Chaw, 2008; Sharma *et al*., 2009; Kannan *et al*., 2012; Polder *et al*., 2012). In Nigeria few studies reported higher residual concentration of γ-HCH in parts of food animals (Osibanjo and Adeyeye, 1997; Maitera *et al*., 2018). Comparing with recent studies, the mean residue levels observed in this study were higher than concentrations reported by Pardio *et al*., (2012); Mahmound *et al*., (2013), who worked on organochlorine pesticide residues in bovine and buffalo tissues respectively. Higher levels observed in this study indicate an upsurge in the use of technical HCH and Lindane on pastures in the region. Only the (γ) isomer (lindane) of HCH was detected in all the samples analysed from this region probably because it is the most persistent in the tissues of animals. The concentration of γ-HCH in the various muscle and organs were in the sequence muscle > liver > kidney > heart (Table 1-4). The presence of γ-HCH in all the analysed samples with the exception of heart tissues of goat from the Zuru region (Table 4) is apparently due to the continuous use of lindane in these emirate councils. Osibanjo and Adeyeye, (1997), reported higher levels of γ-HCH in muscle (14.0 µg/kg lipid), liver (30.0 µg/kg lipid), kidney (25.0 µg/kg lipid) and issues from cattle in South Western Nigeria. High concentrations of γ-HCH in various parts of food animals has also been reported by various researchers (Manirakiza *et al*., 2002; Darko and Acquaah, 2007; Sallam *et al*., 2008; Covaci *et al*., 2014. The concentrations of γ-HCH observed in the muscle, liver, kidney and heart tissues were below the MRL recommended by the Japan Food Chemical Research Foundation (JFCRF, 2016) and the European Union, (EU, 2015).

Endosulfan

The mean concentration of endosulfan in the muscles, liver, kidney and heart tissues are presented in Tables 1-4. Endosulfan was detected in 58% of the samples with the highest residual concentration (0.009 ± 0.003µg/Kg) found in the muscle of camel from Argungu emirate council. Concentrations were no statistically significant ($p > 0.05$, $F = 0.213$) among the tissues. Higher concentrations of endosulfan was reported in bovine fat (Panseri *et al*., 2013) and cattle meat

(Muhammad *et al*., 2010). Varying residues concentration of endosulfan have been reported in cow milk, human tissues and cord blood samples (Cooper et al., 2001), adipose tissue (Amaraen and Pillale, 2011) and human breast milk samples (Campoy *et al*., 2001; Saleh *et al*., 2016).

The observed residue concentrations of endosulfan in this study were lower than the concentrations reported in Grasscutter tissues (Blankson-Arthur *et al.*, 2012), higher concentrations of residues were also reported in game meat (Saschenbrecker, 2016). Although endosulfan has been banned, its prevalence in this study could be attributed to its persistence in the environment or continuous illegal use of the pesticide. Concentrations observed in this study were however lower than the established MRLs (IARC, 2008; JFCRF, 2016).

Heptachlor

Residual concentrations of heptachlor in the muscle, liver, kidney and heart tissues of cow, camel and goat are presented in Tables 1-4. The highest mean concentration (0.063±0.007µg/Kg) was observed in the liver of camel from Yauri emirate council (Table 3). However, residue concentrations in the tissues were not significantly different ($p > 0.05$, $F = 0.32$). Heptachlor binds strongly to soil particles and migrates very slowing (FAO/WHO, 1992; Blankson-Arthur *et al*., 2011) and could therefore be taken in by farm animals during feeding. Food intake is considered to be the major source of exposure to heptachlor (FAO/WHO, 1992; 1995). The relatively high residues concentration observed in this study could be attributed to its persistence in the environment. Similar studies reported by Blankson-Arthur *et al*., (2011) on the concentration of heptachlor residues in Grasscutter tissues revealed higher concentrations in muscle (0.695 µg/kg wet) and kidney (0.403 µg/kg wet), while Schwab *et al*., (2015) reported higher concentrations (1.391 µg/kg wet) in game meat from Ghana. Heptachlor residual concentrations observed in this study for the different organs are lower than the MRLs as stipulated by FAO/WHO, (2016).

Aldrin, dieldrin and endrin

Aldrin, dieldrin and endrin are group of organochlorine pesticides known to be closely related chemically and are extremely persistent in the environment (Newman, 2010). Although, these group of organochlorines have been ban (Abdel-Wahab et al., 2004), residual concentrations have been reported in tissues of food animals by several researchers (Matsumolo *et al*., 2006; Lazaro *et al*., 2016; Sallam *et al*., 2018). Concentrations of aldrin, dieldrin and endrin in the tissues were found to follow a particular order: Muscle > liver > kidney > heart (Tables 1-4). Tissues concentrations of aldrin were generally found to be higher than dieldrin concentrations. Higher concentrations of aldrin in relation to dieldrin could be attributed to recent and indiscriminate application of the parent aldrin as aldrin is known to rapidly metabolizes to deildrin in a wide range of organisms (Blankson-Arthur *et al*., 2011; Shahzadi *et al*., 2013). However, variation in the residue concentrations among the organs were not significantly different ($p > 0.05$).

In this study concentrations of aldrin and endrin were found to be lower than the mean values of 0.058 and 0.127 mg/kg reported respectively for aldrin and endrin by Abdel-Wahab *et al*., (2004), who assessed organochlorine residues in cow milk from Sohag and Qena governorates. Higher residual concentrations of dieldrin and endrin were also reported by Ahmed *et al*., (2010), in bovine muscles in Egypt while a similar value (0.174 µg/kg) was reported for aldrin. Similarly, higher values were reported for aldrin and endrin in chicken muscles while a lower value (0.259 µg/kg)

was reported for dieldrin (Blankson-Arthur *et al*., 2012). Moreover, higher concentrations were reported for aldrin in grasscutter tissues and Game meat from Ghana (Bankson-Arthur *et al*., 2011). The residual levels of these organochlorines observed in this study were lower than the MRLs established by the Japan Food Chemical Research Foundation (JFCRF, 2016) and the European Union (EU, 2015) for pesticide residues in food animals. The prevalence of these banned organochlorines residues in the assessed tissue of food animals call for concern as the meat from these animals constitutes a major part of most diet in Nigeria. These pesticides are known to be highly toxic and can bio-magnify along the food chains.

Chlorpyrifos

Chlorpyrifos one of the organophosphorus residues analysed in this study, was detected in all the samples. The highest mean concentration (0.482±0.021µg/Kg) was found in the muscle of camel from Yauri emirate council (Table 3). Concentrations were however not significant ($p > 0.05$. $F = 0.23$) among the organs. The increased prevalence of chlorpyrifos residues in this study could be attributed to the compounds wide range of uses against ecto – and endoparasites, active in the vapour phase or in the soil, and its uses against crop and public health pests (Garrido *et al*., 2007; Basheer and Lee, 2014; Argauer *et al*., 2017). Chlorpyrifos is also known to form a major component of many pesticide formulations (Battu *et al*., 2004; Pradeep *et al*., 2008). The results obtained from this study is in agreement with that reported by Ahmed *et al*., (2019), in their study of chlorpyrifos residues in the muscles and livers of camel, sheep and cattle carcasses. Bindu *et al*., (2018), reported higher levels of malation and chlorpyrifos residue in pork and chicken meat samples; randomly collected from markets in Pakistan. Dallegrave *et al*., (2018), detected chlorpyrifos residues in meat, fish and egg samples, and all the detected residues were within the acceptable limit, which is in agreement with the current findings. Another study reported in Faisalabad to compare the residue of chlorpyrifos and other pesticides residues in meat samples revealed higher residual levels above the MRL (Sultatos, 1994; Muhammad *et al*., 2010). Residual concentration of chlorpyrifos in this study were found to be within the MRL as stipulated by FAO/WHO, (2016).

Parathion

Parathion was detected in 79.2% of the analysed samples. The highest mean concentration (0.071±0.004µg/Kg) was detected in the muscle of camel from the Zuru emirate council (Table 4). The high prevalence of parathion observed in this study could be attributed to the massive use of this chemical in the areas for the control of ticks in farm animals. Residual concentrations are however not significantly different among the organs ($p > 0.05$, $F = 1.23$). Sara *et al*., (2019), reported similar levels of residual concentration in their study of OPs and OCs in beef meat. Khalid *et al*, (2013) however reported higher levels of parathion in the liver of male mice. Residues concentration observed in this study are lower than the MRLs stipulated by FAO/WHO, (2016).

Methylparathion

Methylparathion, one of the organophosphates pesticide residues analysed in this study was detected in 66.7% of the samples with the highest mean concentration (0.063±0.005µg/Kg) found in the liver of cow from the Zuru emirate council, (Table 4). The observed higher concentrations of Methylparathion residues in the liver may be attributed to the fact that the liver is the primary

site of detoxification of xenobiotics (Hotton *et al*., 2011; Khalid *et al*., 2013). Concentrations among the organs are not significantly different ($p > 0.05$, $F = 0.02$). Methylparathion is one of the most commonly used insecticide globally (Tchounwoul *et al*., 2000; Compernolle *et al*., 2005), it is commonly used in Nigeria for the control of wide varieties of ecto-parasites in most farm animals (Ezemonye and Tongo, 2009). Despite its intensive use, methylparathion has been implicated in a number of health effects (Ezemonye and Tongo, 2009; Dogheim *et al*., 2016). High concentrations of methylparathion in serum and urine samples of cattle were observed by Peighambarzadeh et al., (2011), with concentrations of 0.739 and 1.389 µg/l respectively. In the present study concentrations were lower than the recommended MRLs as stipulated by FAO/WHO, 2016).

Glyphosate

Glyphosate is one of the major herbicides used in agriculture that readily and permanently binds to soil particles and remains in the upper few centimetres of soil (Cox, 1991), which inevitably is consumed by cattle from soil. There is a dearth of information on the levels of glyphosate pesticides residues in parts of food animals. This could be attributed to the fact that glyphosate has been classified by US EPA as non-carcinogenic to humans (US EPA, 1993). Irrespective of this, recent findings have revealed significant poisoning effects caused by both intentional and accidental exposure to glyphosate in humans and laboratory animals (Witorsch, 2002). The existence of glyphosate residues in animal feeds from pre-harvest glyphosate treatment of cereals or fodder may result in residual concentrations in meat, milk and eggs (JFCRF, 2016). Glyphosate was detected in all the samples analysed in this study with varying concentrations. The highest mean concentration (0.277±0.004µg/Kg) was detected in the muscle of cow from Zuru emirate council (Table 4). The observed mean values were however not significantly different among the tissues ($p > 0.05$, $F = 1.14$). Higher levels in the muscle may be attributed to the physical contact during dipping or a consequence of the overlying adipose tissues due to fat solubility (Mahmound *et al*., 2013). Residual concentrations of glyphosate observed in the muscle and organs in this study were however below the MRL (IARC, 2008; JFCRF, 2016).

Fenitrothion and Dichlorvos

Fenitrothion and Dichlorvos are two organophosphate pesticides that are rarely detected in biological tissues despite their extensive use in modern agriculture for the control of insect pests, due to their high rate of degradation in biological tissues. Serious public concerns regarding environmental and food safety of these chemicals have been raised (Morais *et al*., 2011). In the present study fenitrothion was detected in 18.7% of the analysed samples with the highest mean concentration (0.007±0.003µg/Kg) found in the muscle of camel from the Argungu emirate council (Table 2). However, dichlorvos was detected only in one sample of cow muscle from the B/Kebbi area (Table 1). The low prevalence of these pesticides in the samples could be as a consequence of low usage of these substances by farmers in this region or due to high rate of biodegradation of these chemicals in tissues of farm animals (Osesua *et al*., 2018). The results obtained in this study were similar to what was reported by Sara *et al*., (2019) in their study of organophosphorus and organochlorine pesticide residues in beef meat from Khartoum State. Shahzadi et al., (2013) however reported higher values of fenitrothion concentrations in milk samples of Buffalo (0.60 mg/kg), Cow (0.84 mg/kg), Goat (0.74 mg/kg), Sheep (0.74 mg/kg), and

Camel (0.55 mg/kg). Concentrations recorded in this study were lower than the recommended MRLs as stipulated by FAO/WHO, (2016)

Comparing residues contamination of the samples from the various abattoir revealed that samples from Yauri emirate council have the highest pesticide contamination and B/Kebbi samples the least (Fig.2). Generally, samples contaminations are in the order: Yauri (12.2%) > Argungu (11.2%) > Zuru (7.8%) > B/Kebbi (5.8%). Concentrations in tissues were however not significantly different ($p > 0.05$) among the abattoirs. The high prevalence of residual contamination observed in Yauri samples could be attributed to the high volume of agricultural activities (both wet and dry season) being carried out in this region which necessitated the indiscriminate use of agro-chemicals. Although, the individual residual concentrations observed in this study are lower than the MRLs, the results suggest that farm animal's meat and tissues from the respective abattoirs sold in markets come from the same source. This raises concern over the consequences that might result from accumulation of these pesticide residues in food animal tissues consumed as meat in this region, as their accumulative properties could pose health risks to man and animals (Lebel *et al*., 2018).

Among the organochlorine residues investigated, γ-HCH (18.2%) in Argungu was observed to have the highest prevalence in all the analysed samples (Fig. 2). The high prevalence of γ-HCH in the tissues assessed compared with the other pesticide residues may be attributed to its high volatility which increases uptake via deposition from the atmosphere onto pasture surface which in turn are consumed by farm animals (Pardio *et al*., 2012; Isioma and Lawrence, 2015). Glyphosate (21.9%) and chlorpyrifos (20.9%) are the dominant organophosphate residues detected. Their prevalence could be largely due to the high volume of these pesticides consumed in this region in combatting weeds and insect pests respectively. The ability of the various animals to store pesticide residues in their muscles and organs were depicted in Fig. 6. The cow was found to accumulate the highest level (47.04%) of the analysed residues, the camel accumulated 44% of the detected residues while goat was found to have accumulated only 8.48%. This result could be a reflection of the feeding pattern of the animals, the versatility of the animal as well as the ability of the animal to detoxify the residues and the consequential effective elimination method (Arpana *et al*., 2015; Milam *et al*., 2017). This result is however at variance with the study by Sait *et al*., (2011), who reported higher residues in smaller animals in their study of organochlorine pesticide residues in tissue and blood of goat.

Conclusion

The results from this study has shown that pesticide residues can accumulate in the muscles and organs of farm animals. The concentration levels of all the assessed pesticide residues observed in the muscle, liver, kidney and heart tissues were however lower than the recommended maximum residual limit (MRL) set by FAO/WHO and the Japan Food Chemical Research Foundation for pesticide residues in food animal tissues and thus within safe limits. However, the results revealed that some residues of organochlorine pesticides are present at concentrations close to the WHO Maximum Residue Levels (MRLs) in the meat samples. These pesticides might have originated from the feed of the farm animals or from pesticides used as 'dip' to control ectoparasites on the animals or from maternal transfer through the breast milk. Education, training and information activities on pesticides and their residues should be established and strengthened. Open and free

grazing of farm animals should also be discouraged as this predisposes the animals to high loads of pesticide contamination. The results from this study provides preliminary baseline data on the residues levels detected on the edible parts of farm animals (muscle, liver, kidney and heart) contaminated with pesticide residues in Kebbi State. Regular monitoring of pesticide residues in meat and meat products is therefore necessary to mitigate the impact of these pesticide on the health of general populace consuming these products.

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