
**STUDIES ON LEVELS OF PESTICIDE RESIDUES IN BEEF OBTAINED FROM
ABATTOIR OF JIMETA, ADAMAWA STATE, NIGERIA**

*¹Ndahi J. A., ¹Maitera O.N., ¹Kubmarawa D. and ¹Joseph J. I.

¹Department of Chemistry, Faculty of Physical Sciences, Modibbo Adama University of
Technology, Yola, Nigeria

Corresponding author: jipsy1965@gmail.com

ABSTRACT

The current work assessed levels of Organochlorine and Organophosphorus pesticide residues in ten sampled cattle obtained from abattoir of Jimeta metropolitan, Yola North Local Government Area of Adamawa State, Nigeria. Forty samples made up of beef, intestines, kidneys and livers were prepared using Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) method of sample preparation. Analysis was carried out with Gas chromatography – mass spectrometer (GC-MS). Results of the analysis showed that Organochlorine pesticide (OCP) was not detected in all the samples. Organophosphorus pesticide (OPP) residues were detected below the threshold of 0.001 mg/kg. They include: anthracene, chlorpyrifos, dichlorvos, dichlorpyrifos, diazinon, dimethoate, primifos-methyl, and malathion. Pesticide residues from 1st, 4th, 6th and 10th cattle were below the threshold limit (0.001 mg/kg). In cow 2, concentration of dichlorvos was 0.059 mg/kg in intestine and 1.021 mg/kg in liver respectively and in cow 9 it was 0.039 mg/kg in intestine and 0.081 mg/kg in liver. Mean concentrations of chlorpyrifos pesticides in intestine of cows 3, 5, 7 and 8 were 0.034, 0.027, 0.011 and 0.012 mg/kg respectively. In the liver samples of cows 3, 5, 7 and 8 the concentrations of chlorpyrifos were 0.011, 0.014, 0.09 and 0.007 respectively. The concentration of chlorpyrifos in the intestine was above the maximum residue limit (MRL) values of 0.1 mg/kg. Lower concentration of dichlorvos in the intestine and liver was a sign of its lower effect on the environment and may not cause acute effect when consumed. Presence of chlorpyrifos above MRL is not acceptable health wise. It can be derived from this studies that farmers in Jimeta and its environs use less organochlorine pesticides in their farming activities compared to organophosphate pesticides. Beef consumed in Jimeta contained moderate concentration of pesticides. Therefore, it is recommended to occasionally monitor levels of pesticides in livestock and other food sources.

Keywords: Chlorpyrifos, dichlorvos, organochlorines, organophosphorus, pesticide,

INTRODUCTION

The ever increasing pressure to increase food production to meet up with the current demand due to population growth requires protection of crops from pests [1]. Hence, the emergence of pesticides of various types which over the years have become inevitable integral part of modern farm management [2]. Among other agents, pests diminish crop productivity and yield. This prompted development and application of pesticides in order to combat the pests [3]. The term pesticide includes herbicides, insecticides, insect growth regulator, nematicides, termiticides, molluscicides, piscicides, avicides, rodenticides, predacides, bactericides, insect repellent, antimicrobial, fungicides, disinfectants and sanitizers [4,5]. Pesticide means any chemical substance or mixture of substances intended for preventing, destroying, repelling or mitigating the effect of pest on plants and animals. Pesticides are substances with high toxic effects and persistence in the environment [6] and are primarily designed to kill insects, fungi and weeds. These properties are unique and constitute a threat to man's health and environment [7].

Over the years, agriculture production system moved from subsistence to market oriented large scale farming, thereby resulting to alarming increase in pesticide usage [8]. Pesticide usage has a positive and dramatic effect on agriculture production through protection of crops against insects, pest and diseases. On the other hand, the indiscriminate usage of pesticides in agriculture, domestic and veterinary institutions has brought about increase in consumption or intake of pesticide residues in crops and meat. In Nigeria, this trend has increased over the years, more especially in the North east where farming and rearing of animals are the predominant occupations.

Livestock generally accumulate pesticide residues in two ways: Either applied to animals directly as insecticide such as impregnated ear tag, spray, self-treatment back rubber, dust bags, inhalation or through ingestion of contaminated water, soil and fodder [9]. The lipophilic nature of pesticide residues is responsible for causing them to accumulate in the fatty parts of meat and meat products such as milk and fatty tissues and organs [6]. Such animal contaminated products may end in food chain where these toxic residues may cause unhealthy situation to the public.

Several pesticides have been banned in many countries, more especially group of pesticides termed the 'Dirty dozen.' The dirty dozen includes; camphechlor, chlordane dibrochloropropane

(DBCP), dichlorodiphenyltrichloroethane (DDT), aldrin, ethylenedibromide (EDB), hexachlorocyclohexane/benzene hexachloride (HCH/BHC), paraquat, parathion, pentachlorophenol, and 2,4,5-trichlorophenoxyacetic acid (2,4,5 T). Members of this group of pesticides are mostly derivatives of chlorine [2]. Chlorine is the basis for many of the most toxic and persistent chemicals known. This is because of its ability to combine with hydrocarbon to form toxic, persistent and bio accumulative organochlorines [10]. Despite the legal restriction on these pesticides in most of the developed worlds, they are still imported and used by some developing worlds, where they find use without checks, safeguard and in untrained hands and may likely end up infiltrating soil, water and air thereby ravaging the food chain and play havoc with human as well as the ecological health [2].

Studies have showed presence of pesticide residues in fruits and vegetables [11, 12], various tissues of fish [13], cropland soils [14] and animal tissues [15]. This work therefore, assessed levels of Organochlorine and Organophosphorus pesticide residues in cattle slaughtered in the study area. This served as a way of monitoring levels of toxic pesticides residues in our environment and in animal products.

MATERIALS AND METHODS

The following materials were used in the current work: acetonitrile, magnesium sulphate (MgSO_4) and sodium chloride (NaCl). All pesticide grades were obtained from reagent trade chemical provider. Other materials include distilled water, polythene zipper bag, electric chopper, and centrifuge. Gas chromatography – mass spectrometer (GC-MS)

Sampling and Sample Preparation

Study Area

The study area of the work was Yola slaughterhouse in Jimeta, Yola North local government Area of Adamawa State, Nigeria. The town lies along River Benue with coordinates; $9^{\circ}16'45''\text{N}$ $12^{\circ}6'45''\text{E}$. The slaughterhouse is the biggest abattoir in Adamawa State. It serves not only the state capital but also some of the satellite towns in its vicinity. The state is known for farming and rearing of livestock especially cattle.

Sampling

Sampling was carried out according to method outlined by Herrera et al [16]. Ten cattle were randomly selected among those slaughtered on each sampling day for ten consecutive weeks. The meat was collected/ purchased along with the intestine, kidney and liver. A total of forty (40) samples were collected / purchased within a span of two and half months. The samples were packed in polythene zipper bags and labeled as; B₁= beef of first sampled cow, I₁ = intestine, of first sampled cow, K₁= kidney of first sampled cow and L₁= liver of first sampled cow. Similar labeling procedure was used for the other sampled cattle up to; B₁₀ = beef of tenth sampled cow, I₁₀ = intestine, of tenth sampled cow, K₁₀ = kidney of tenth sampled cow and L₁₀ = liver of tenth sampled cow. Thereafter, the samples were transported to the laboratory for preparation and analysis.

Extraction of pesticide residue in beef/ organs

Extraction of the samples were carried out according to method outlined by Maitera et al [6] in which 10 g of each of the samples was weighed chopped and then homogenized. The ground beef was transferred into a 50-mL centrifuge tube. The sample was extracted using 2 mL water and 10 mL acetonitrile (ACN), followed by vigorous shaking for 1 minute. About 4 g MgSO₄ and 1g of NaCl was also added and vigorously shaken for 1 minute. Thereafter the sample was transferred to the centrifuge and ran for 3 minutes at 4000 rpm where 1 mL aliquot of the supernatant (top layer) was taken for dSPE clean up. Other samples were sequentially treated accordingly.

dSPE Cleanup

The cleanup was when 1-mL aliquot of supernatant was transferred to a 2-mL dSPE cleanup tube that contains 150 mg of magnesium sulfate, 50 mg PSA sorbent, and 50 mg C₁₈ sorbent (p/n 186004830). The content was shaken vigorously for 1 minute and a portion of the supernatant was transferred to the LCMS Certified Vial for GC/MS analysis.

Analysis

The analysis was carried out according to method outlined by Maitera et al [6] whereby 1 mL aliquot of the supernatant which was transferred into a certified vial for gas chromatography-mass spectrometry where the Pesticides (organochlorides and organophosphorus) residue level in

samples was determined with GC condition: System – Agilent 7890 AMSD 5975CM; Columns: HP-5MS30(M) 0.250 DIAM (MM) 0.25 film (UM); Temp Limit: 60 to 325 degree Celsius; Gas – Helium, flow. The analysis of all the animal samples was carried out using the software CSW 32 for the GC-MS instrumentation. The peak height, area under curve and the type of pesticide used were obtained.

Statistical Analysis

Statistical Packages for Social Sciences (SPSS) was used to arrive at the mean.

RESULTS AND DISCUSSION

Results of analysis of the samples were presented in Figures 1- 10. The mean concentration values of pesticides detected in the samples with values above the threshold were presented as charts. No organochlorine pesticide was detected. Some organophosphorus pesticides detected at trace levels below threshold of 0.001 mg/kg were anthracene, chlorpyrifos, dichlorvos, dichlorpyrifos, diazinon, dimethoate, primifos-methyl, and malathion in some samples.

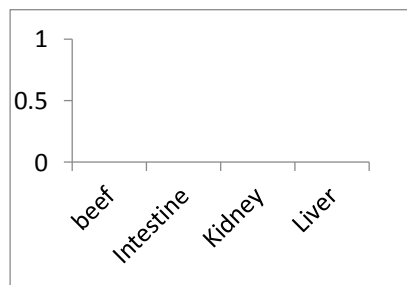


Figure 1: Organophosphate pesticide in sample C1 (mg/kg x 10⁻²)

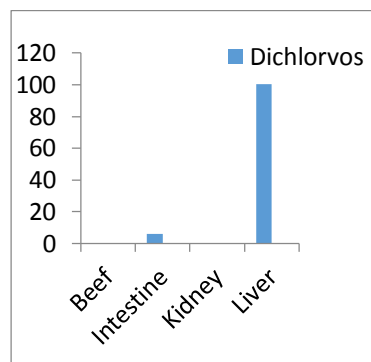


Figure 2: Organophosphate pesticide in sample C2 (mg/kg x 10⁻²)

All the pesticides from cow 1, 4, 6 and 10 were below the detection limit (0.001) mg/kg of the instrument. Only dichlorvos was detected in both liver and intestines samples of cow number 2 (C2) figure 2 and cow number 9 (C9) figure 9. Dichlorvos and chlopyrifos were both detected in cows; 3, 5, 7 and 8 as contained in Figures 3, 5, 7 and 8.

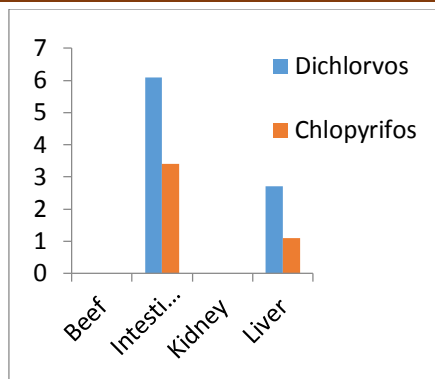


Figure 3: Organophosphate pesticide in sample C3 (mg/kg x 10⁻²)

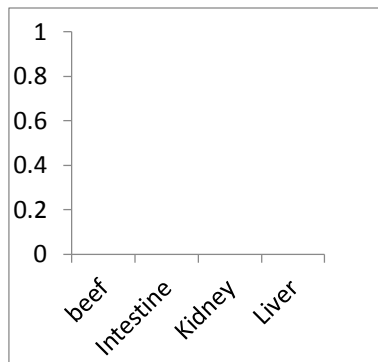


Figure 4: Organophosphate pesticide in sample C4 (mg/kg x 10⁻²)

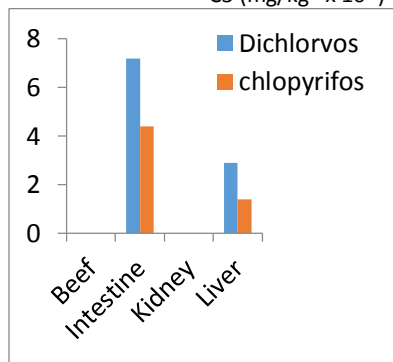


Figure 5: Organophosphate pesticide in sample C5 (mg/kg x 10⁻²)

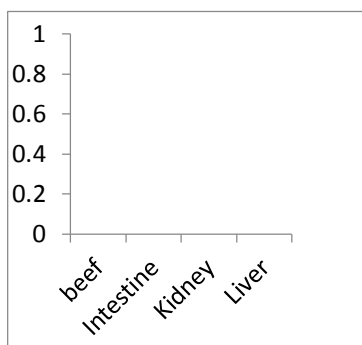


Figure 6: Organophosphate pesticide in sample C6 (mg/kg x 10⁻²)

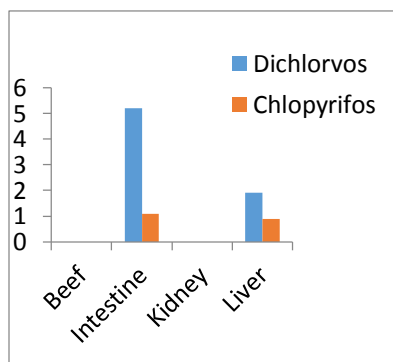


Figure 7: Organophosphate pesticide in sample C7 (mg/kg x 10⁻²)

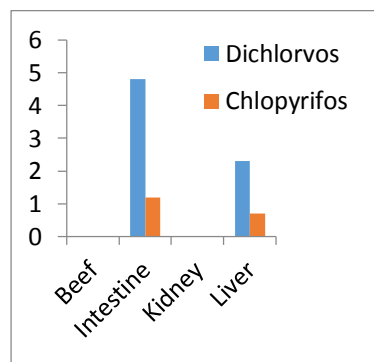


Figure 8: Organophosphate pesticide in sample C8 (mg/kg x 10⁻²)

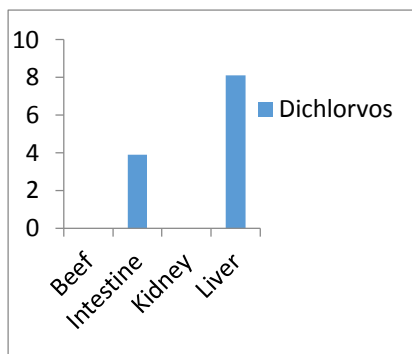


Figure 9: Organophosphate pesticide in sample C9 (mg/kg x 10⁻²)

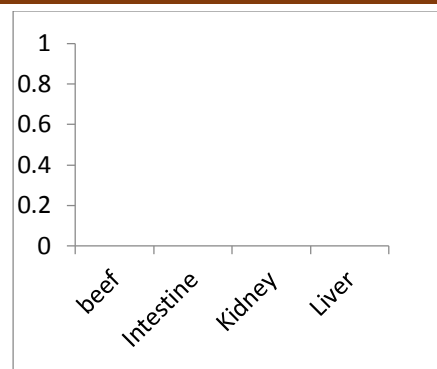


Figure 10: Organophosphate pesticide in sample C10 (mg/kg x 10⁻²)

Similarly, two pesticides were detected in intestines and livers each in figures 7, 8 and 9, which showed results for the various samples analyzed for cow number 7 (C7), cow number 8 (C8) and cow number 9 (C9) respectively..

Generally all the pesticides residues detected in this work were in the intestine and the liver samples. No traces of organochlorine pesticide was detected in this work. Samples from cow 1, 4, 6 and 10 as shown on Figures 1, 4, 6 and 10 contained organophosphates pesticide residues below detection limit of (0.001 mg/kg). This implies that the values obtained are below the Maximum Residue Limit established by United States Food and Drugs Administration (USFDA). The absence of these pesticides could be attributed to the following; the environment where pesticides were not used and lack of leaching of pesticides through soil to underground water where it may contaminate drinking water for the animals. Only dichlorvos was detected in the intestines and the livers of samples from cow 2 and cow 9 as contained in figures 2 and 9. In cow 2 dichlorvos was detected at mean values of 0.059 mg/kg in intestine and 1.021 mg/kg in livers respectively and in cow 9 at mean values of 0.039 in intestine and 0.081 in liver. Concentrations of dichlorvos in the intestine are below maximum residue limit value in the intestines compared to that of liver which is slightly higher than the MRL value of 1.00 mg/kg established by USFDA. The results implied that the animals ingested the pesticides from contaminated fodder, drinking water or from veterinary drugs and fumigants. Dichlorvos is an insecticide used on crops, stored products and animals or as pest control in homes. Acute and chronic exposure of humans to dichlorvos result in the inhibition of an enzyme,

acetylcholinesterase, with neurotic effects including perspiration, vomiting, diarrhea, drowsiness, fatigue, headache, and at high concentrations, convulsions and coma.

Both dichlorvos and chlorpyrifos pesticides were detected in intestines and livers only of cows 3, 5, 7 and 8 (Figures; 3, 5, 7 and 8) respectively. The mean concentrations of chlorpyrifos pesticides in intestine of cows 3, 5, 7 and 8 were 0.034, 0.027, 0.011 and 0.012 mg/kg respectively. In the liver samples of cows 3, 5, 7 and 8 the concentrations of chlorpyrifos were 0.011, 0.014, 0.09 and 0.007 respectively. The concentration of chlorpyrifos in the intestine are above the MRL values of 0.1 mg/kg, which is about 340% increase in intestine whereas, the liver concentration was found to be slightly above the MRL value of 0.01 mg/kg, which is about 110% increase in the liver. However, dichlorvos concentration in both the intestine and liver are below the MRL values of 1.00 mg/kg with less than 6.1% and 2.7% respectively. This indicates no level of contamination by dichlorvos in the intestine and liver of cow 3. The chlorpyrifos detected in this study were several times more than the stipulated MRL. This is similar to that obtained in milk where it had values ranging from 5 to 18 mg/kg [17]. The concentration of dichlorvos remains same seasonally in muscle, kidney, liver, and lung samples of cattle [17]. Detectability of dichlorvos pesticides could be attributed to its wide use as an insecticide and ectoparasite on livestock [17].

CONCLUSION

All the pesticide residues detected in the current work were organophosphate pesticides. No trace of organochlorine pesticide was detected. Chlorpyrifos and dichlorvos were the only pronounced OPP detected while the rest were beyond the detection limit (< 0.001) of the instrument used. Chlorpyrifos was detected with values many times more than the MRL value established by USFDA. Dichlorvos concentration in both the intestine and liver were below the MRL value of 1.00 mg/kg with less than 6.1% and 2.7% respectively. This indicated that the level of contamination by dichlorvos in the intestine and liver may not cause acute effect when consumed. Presence of chlorpyrifos above MRL is not acceptable health wise. The health and environmental effects associated with OCP include reproductive failures, birth defect, endocrine disruption properties. Human data also indicated possible disruption in semen quality, menstruation, gestation length and duration of lactation. It is recommended therefore that occasional monitoring of levels of pesticides in livestock and other food sources are carried out.

REFERENCES

1. Sharma, N., Lakshmanan, V. & Singh, C.M. (1984). Pesticide Residues in Livestock Products with Special Reference to Meat. *Indian Food Industry*. (3):52-56
2. Sharma, B.K. (2011). Industrial Chemistry. Krishna Prakashen Media (P) Ltd. Meerut, India. P. 1300
3. John, P.J., Bakore, N., & Bhanthnagar, P. (2001). Assessment of organochlorine pesticides Residue levels in dairy milk and buffalo milk from Jaipur city, Rajasthan, India, *Environment International*, 26:231:236
4. NAFDAC Decree and Regulation (2004) Federal Republic of Nigeria: National Agency for Food and Drug Administration and Control Pesticide Registration and Regulation. No. 15: 303-307
5. Jacobs, M.B. (1999). The Chemical Analysis of Foods and Food Products. 3rd ed. CBS Publishers and Distributors, New Delhi, India. P. 891.
6. Maitera, N. O, Hitler, L., Bata, S. Y., Adeleye, T. A., Akakuru, U. O. & Magu, O. T. (2018). Comparative Analysis of the Level of Pesticide Residues in Beef, Chevon and Internal Organs of Cows and Goats Slaughtered in Yola Abattoir of Adamawa State, Nigeria. *Current World Environment*. 13(3):416-423
7. Cox, C., & Surgan, M. (2006). Unidentified inert ingredient in pesticides: Implication for human developmental health. *Perspectives 111*:377-382.
8. Sosan, M.B., Amos, E.A., Isaac, A.O. & Muheez A.D. (2008). Insecticide residues In the blood serum and domestic water source cacao farmers in Southwestern Nigeria. *Journal of Chemosphere* 72:781 – 784.
9. Tongo, I. & Ezemonye, L. (2015). Humans health risks associated with residual pesticides levels in edible tissues of slaughtered cattle in Benin City, Southern Nigeria. *Toxicology reports 2*:1117 - 1135
10. Bouwman, H. (2004). South Africa and Stochholm convention on persistent organic pollutants. *South African Journal of Science*. 100:323-328
11. Tahir, S., Anwar, T., Ahmad, I., Aziz, S., Mohammad, A., & Ahad, K. (2001). Determination of pesticide residues in fruits and vegetable in Islamabad market. *Journal of Environmental Biology*. 22(1): 71 – 4

12. Perveen, Z., Khubro, M.T. & Rafig, N. (2005). Monitoring of pesticide residue in vegetable (2000 – 2003) in Karachi, Pakistan.
13. Ssebugere, P., Kiremire, B.T., Kishimba, M., Wandiga, S.O. Nyanzi, S.A.& Wasswa, J, (2009). DDT and metabolites in fish from Lake Edward, Uganda. *Chemosphere* 76, 212-215
14. Jabbar, A.S., Masud, Z., Perveen, Z. and Mubarik, A. (1993). Pesticides residue in Cropland Soils and shallow groundwater in Punjab, Pakistan Bull. *Environmental Contamination & Toxicology*. 51(2): 112 – 8.
15. Sara, A.M.,Sahar, M.A., Abubakar, A. S., Abdelbaqi, A.O., & Abdalla, A.M. (2019). Monitoring of Some Organophosphate and OrganochlorinePesticide Residues in Beef form Khartoum State Slaughterhouses. *Journal of Biomedical Science & Research*. 6(5), 405-409.
16. Herrera, A., Arino, A. A., Conchello, M.P., Lazaro, R., Bayarri, S. & Perez, C. (1994). Organochlorine Pesticide Residues in Spanish Meat Products and Meat of different Species. *Journal of food protection* 57(5),441-444
17. Gazzotti, T. Sticca P., Zironi, E., Ogoboni B., Serraino, A. & Paglica, G. (2008). Determination of 15 organophosphorus pesticides in Italian Raw Milk. *Bulletin of Environmental Contamination and toxicology*, 82, 251-524