



## Assessment of Herbicide Residues in Some Internal Organs of Cattle from Jos Markets

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### Abstract

In Nigeria use of agrochemicals especially herbicides is growing. This poses a possible threat to humans through bioaccumulation of these chemical in animals as a result of predominant open grazing activities. The levels of five herbicide residues (alachlor, atrazine, butachlor, metolachlor and propanil) were therefore analysed in three organs of cattle (heart, liver and kidney) obtained from three different markets (Abbatoir, Bukuru and Yanshanu) in Jos metropolis of Plateau state, North Central Nigeria. The study was carried out in two monitoring seasons (October- January) and (May-July). Ledoux extraction method was used for all samples. Extracts were concentrated with rotary evaporator after clean-up with silica gel (60-120 mesh) and analyzed using gas chromatography equipped with flame ionization detector. The result obtained for herbicide residues are in the following ranges: atrazine (0.1- 8.78 ppm), propanil (0.001-7.41 ppm), alachlor (0.0003-0.356 ppm) and butachlor (0.00-0.911 ppm). No metolachlor residue was detected in all the samples. Some of the samples recorded high concentrations of these herbicides when compared with Food and Agricultural Organisation of the United Nation, FAO/WHO [1], TAS [2] and European Food Safety Authority, EFSA [3] established Maximum Residue Limits (MRLs). This implies that consumption of cattle internal organs might be threatening to human health. Therefore relevant health authorities should consider monitoring and controlling the use as well as the presence of these chemicals in meat sold in the country.

**Keywords:** Herbicide; Residue; Liver Organ; Heart Organ; Kidney Organ; Cattle; Bioaccumulation; Persistency

### Introduction

Herbicides pollution is one of the major global concerns because of their environmental persistency. The high usage of herbicides in order to maximize crop yield has aggravated the problem even though there is a shift to natural ways of increasing crop yield [4,5]. Herbicide residues bioaccumulate and bio-magnify along the food chain [6,7], this unique characteristics leads to accumulation in edible tissues and biological samples of ruminants [8-14], which is a threat to human health. There is thus the need to continuously monitor and assess these residues in food commodities and their possible ecological damaging effects.

### Materials and Methods

Herbicide standards: Alachlor, atrazine, butachlor, metolachlor and propanil were prepared in concentrations of 2.5 ppm, 5 ppm and 10 ppm.

### Equipment

Shaker (Gallenkamp 400rpm), Rotary evaporator (RE100, Bibbystiril Ltd, England) and Gas Chromatography (GC\_2010 Shidmazu) equipped with flame ionization detector (FID) at Oupearl Scientific Laboratory, Oshodi, Lagos. The operating conditions were as follows: Initial column temperature was 80°C hold for 1 minute, rate 10°C to 180°C hold for 2 minutes, rate 10°C to 250°C hold for 1 minute. The injector temperature was 250°C.

### Sampling

Samples of cattle heart, kidney and liver were collected from three different markets in Jos metropolis (Abbatoir (A), Bukuru (B) and Yanshanu (Y)) covering 20km for two seasons (October –January, 2013 and May-July, 2014). Samples were wrapped immediately with aluminium foil and placed in a cooler packed with ice and then transported to laboratory for analysis. The map of sample sites is shown in figure 1.

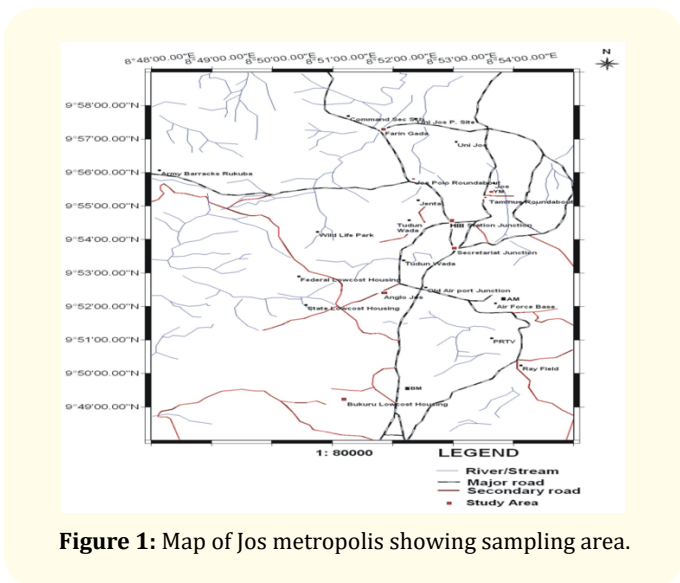


Figure 1: Map of Jos metropolis showing sampling area.

**Extraction**

Solid-liquid extraction was carried out on the samples as described by Ledoux [15]. Exactly 10g of each test sample was homogenized with the aid of porcelain mortar and pestle, then 30 mL of hexane (HEX) was introduced into the sample in a coked conical flask and vitroused at 400 rpm for 30 minutes. The solution was

filtered and concentrated using Rotary Evaporator with water bath maintained at 60 degrees after clean- up, this procedure was repeated with Acetonitrile and Methanol successively. Finally, extracts were stored in amber coloured bottles for GC analysis.

**Clean-Up**

Removal of other co-extractives was achieved by using silica gel (60 – 120 mesh). Slurry of 30 g of silica gel with hexane was made, poured to the column that was conditioned with hexane. Extracts were then introduced into the column and eluates were collected for GC analysis.

**Preparation of reference samples**

Standards of herbicide concentrations of 2.5 ppm, 5.0 ppm and 10 ppm were prepared in acetone for the analysis.

**Results**

Herbicide standards at various concentrations are shown on Table 1.

Peak areas and height obtained as shown on the chromatograms figures 2-4. Concentration of herbicide residues in cattle organs from Abattoir, Bukuru and Yanshanu markets in Jos, are shown on Table 2.

Alachlor		Atrazine		Butachlor	Propanil		
Area (mv.s)	Conc (ppm)	Area (mv.s)	Conc (ppm)	Area (mv.s)	Conc (ppm)	Area (mv.s)	Conc. (ppm)
1170653	2.50	927182	2.56	20009	1.52	119635	2.42
2323459	4.96	1812598	5.00	49990	3.80	242839	4.91
46727877	9.97	36076277	9.97	1431833	10.9	479046	9.69

Table 1: Herbicide standards at various concentrations in ppm.

Samples	Butachlor Rt=13.129	Atrazine Rt=13.906	Propanil Rt=15.784	Alachlor Rt=15.912
A1H(HEX)	BDL	0.7774	BDL	0.0012
A1K(HEX)	BDL	BDL	BDL	0.0038
A1L(HEX)	BDL	BDL	BDL	BDL
A1H(ACN)	BDL	BLD	BDL	BDL
A1K(ACN)	BDL	BDL	BDL	BDL
A1L(ACN)	BDL	BDL	BDL	BDL
A1H(METH)	BDL	BDL	BDL	0,1011
A1K(METH)	0.9109	6.6606	1.6391	0.0858
A1L(METH)	BDL	BDL	0.5117	0.3555
A2H(HEX)	BDL	0.1918	0.0017	0.0046
A2K(HEX)	BDL	8.7829	BDL	0.1047
A2L(HEX)	BDL	BDL	BDL	BDL
A2H(ACN)	BDL	0.4311	BDL	0.0272
A2K(ACN)	BDL	BDL	BDL	BDL
A2L(ACN)	BDL	0.7773	BDL	0.0023
A2H(METH)	BDL	BDL	BDL	0.0263
A2K(METH)	BDL	BDL	BDL	0.0067
A2L(METH)	BDL	BDL	BDL	BD

Table 2: (a) Concentration of herbicide residues in cattle organs obtained in Abattoir market.

KEY: A1-Abattoir market, 1st monitoring seasons of herbicides; A2- Abattoir market, 2nd monitoring seasons of herbicides; H- Heart; K- Kidney; L- Liver; HEX-Hexane; ACN-Acetonitrile; METH-Methanol; ND-Not detected; Rt-Retention time; BDL-Below detection limit.

Samples	Butachlor Rt=13.129	Atrazine Rt=13.906	Propanil Rt=15.784	Alachlor Rt=15.912
B1H (HEX)	BDL	BDL	BDL	0.0527
BIK (HEX)	ND	6.0870	0.1347	0.0401
B1L (HEX)	BDL	BDL	0.0698	0.0329
B1H (ACN)	BDL	BDL	0.306	0.1075
BIK (ACN)	BDL	BDL	BDL	BDL
B1L (ACN)	BDL	BDL	BDL	0.0202
B1H (METH)	BDL	BDL	BDL	0.0074
B1K (METH)	BDL	BDL	0.0189	0.0024
B1L (METH)	BDL	0.9983	0.0825	0.0330
B2H (HEX)	BDL	BDL	BDL	BDL
B2K (HEX)	BDL	0.2510	0.0171	0.0033
B2L (HEX)	BDL	BDL	BDL	0.0024
B2H (ACN)	BDL	BDL	BDL	0.0003
B2K (ACN)	BDL	BDL	BDL	0.0014
B2L (ACN)	BDL	BDL	BDL	BDL
B2H (METH)	BDL	BDL	BDL	0.0026
B2K (METH)	BDL	BDL	BDL	0.0030
B2L (METH)	BDL	BDL	BDL	0.0050

**Table 2: (b)** Concentration of herbicide residues in cattle organs obtained from Bukuru market.

KEY: B1-Bukuru market,1st monitoring season;  
 B2-Bukuru market,2nd monitoring season  
 Rt-Retention time; H-Hear; K-Kidney; L-Liver; HEX-Hexane;  
 ACN-Acetonitrile; METH-Methanol; BDL-Below detection limit

Sample	Butachlour Rt=13.129	Atrazine Rt=13.906	Propanil Rt=15.784	Alachlor Rt=15.912
Y1H (HEX)	BDL	BDL	BDL	0.0180
Y1K (HEX)	BDL	2.8126	7.4060	BDL
Y1L (HEX)	BDL	BDL	BDL	BDL
Y1H (ACN)	BDL	BDL	BDL	BDL
Y1K (ACN)	BDL	BDL	DBL	0.0088
Y1L (ACN)	BDL	BDL	BDL	0.0071
Y1H (METH)	BDL	BDL	BDL	0.0454
Y1K (METH)	BDL	BDL	BDL	BDL
Y1L (METH)	BDL	BDL	BDL	BDL
Y2H (HEX)	BDL	BDL	BDL	BDL
Y2K (HEX)	BDL	BDL	BDL	0.0032
Y2L (HEX)	BDL	BDL	BDL	BDL
Y2H (ACN)	BDL	BDL	BDL	BDL
Y2K (ACN)	BDL	0.190	BDL	0.0061
Y2L (ACN)	BDL	BDL	BDL	0.0239
Y2H (METH)	BDL	BDL	BDL	0.0084
Y2K (METH)	BDL	BDL	BDL	0.0081
Y2L (METH)	BDL	BDL	BDL	BDL

**Table 2: (c)** Concentration of herbicide residues in cattle organs obtained from Yanshanu market.

KEY: Y1-Yanshanu market, 1<sup>st</sup>, monitoring season;  
 Y2-Yanshanu market, 2<sup>nd</sup> monitoring season; H-Heart;  
 K-Kidney; L-Liver; HEX-Hexane; ACN-Acetonitrile;  
 BDL-Below detection limit

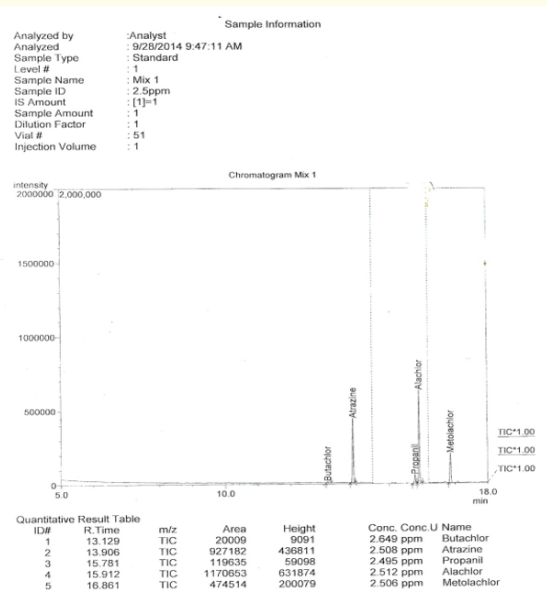


Figure 2

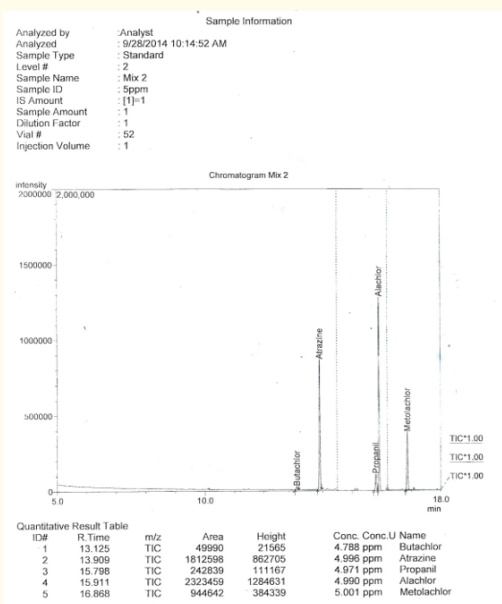


Figure 3

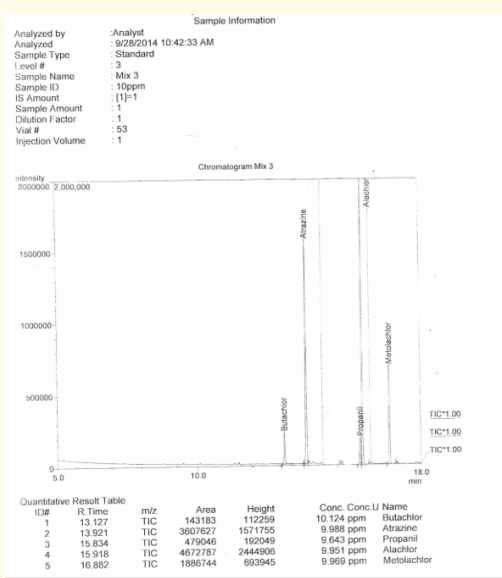


Figure 4

Discussion

Four herbicide residues detected were Alachlor, atrazine, butachlor and propanil, from this study as shown on Table 2. Alachlor concentrations ranged from 0.0003-0.356 ppm, atrazine from 0.1-8.78 ppm while butachlor from 0-0.911 ppm and propanil from 0.001-7.41 ppm. Most samples from the three markets revealed low concentrations of alachlor with the exception of sample A1L (METH) which was 0.356 ppm. Its frequent detections in samples is attributed to its high mobility in the environment which could be used as grazing area for cattle [16]. This result agreed with the work of Choudhury, *et al.* [17] which reported 0.001 to 0.0016g alachlor residues in fish tissues.

The concentration of atrazine residues in cattle organs were high exceeding the 0.05 ppm maximum residue limits by WHO/FAO [1]. Atrazine residues were detected in all the markets. The high concentrations could be attributed to the frequently used atrazine herbicide in Jos metropolis on maize and sorghum farms because they are the most stable foods where the cattle could consume their stalks. Another possible source for these high concentrations could be from contaminated water and soil. Atrazine degrades slowly (13-261 days). Peighambar Zadeh, *et al.* [11] reported the mean concentration of atrazine in serum and urine samples to be 0.739-0.567 ppm and 1.389- 0.633 ppm respectively which supported this study. Dana, *et al.* [18] also detected high amounts of atrazine residues in urine of farmers with their spouses and children after atrazine application on fields.

The concentration of 0.911 ppm butachlor detected in Abbatoir market with none from Bukuru and Yanshanu markets was high. This finding is in agreement with the work of Shad and Andrabi [19] which reported horses poisoned with butachlor from contaminated water. Similarly the works of Ebrahim, *et al.* [20], and ying chu Lo, *et al.* [21] detected butachlor in animals after consumption of contaminated water. Also butachlor ranged from 0.0012 to 0.0014 µg/g was detected in fish tissues from different markets in India [17].

Propanil residues were detected in the range of 0.001-7.41 ppm from all markets. According to European Food and Safety Authority [3], Propanil is not expected to be in livestock products because it is easily metabolized to 3,4-dichloroaniline by the liver. Therefore its detection in livestock commodity calls for its monitoring in animal tissues (Figure a).

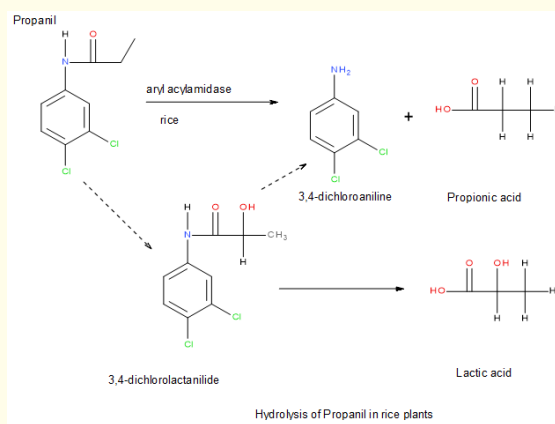


Figure a

Seasonal variation in herbicide residues concentration was noted. More herbicide residues were detected in the 1st monitoring season (October-January) compared with the 2nd monitoring season (May-June). This could be as a result of the incessant crisis in the Plateau State, making farmers maximize the land closer to their domain where irrigation is employed.

## Conclusion

This work shows the need to carry out monitoring studies on pesticides in meat in order to improve food safety since these pesticide residues posed a potential risk to human health. Therefore relevant health authorities should develop a monitoring scheme that will involve periodic assessment of the prevalence of the bio-accumulation of these chemicals.

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