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Full Length Research Paper

Spectral depth analysis for determining the depth to basement of magnetic source rocks over Nkalagu and Igumale areas of the Lower Benue Trough, Nigeria

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Structural interpretation of aeromagnetic data over Nkalagu-Igumale area of the Lower Benue Trough of Nigeria was carried out to determine the depth to magnetic basement and delineate the basement morphology and the structural features associated with the basin and their trends. The aeromagnetic data were subjected to series of computer based image and data enhancement techniques before spectral analysis. Results of the 2-D spectral analysis revealed two depths source models with the depth model (D1) for deep magnetic source bodies which are associated with intra-basement discontinuities and faults ranging from 2.15 to 5.25 km while the depth model (D2) of the shallow magnetic source bodies range from 0.35 to 0.99 km. From an economic viewpoint, the results indicate possible mineralization and existence of a reasonable Cretaceous sedimentary thickness in the area which is deep enough for hydrocarbon accumulation. The average sedimentary thickness obtained in the area is 3.75 km.

Key words: Benue Trough, depth to magnetic basement, aeromagnetic data, spectral analysis, intrusive bodies.

INTRODUCTION

Magnetic method is a measurement of the earth's magnetic field intensity, typically involving the total magnetic field and/or vertical magnetic gradient, horizontal or vertical component or horizontal gradient of the magnetic field (Biswas et al., 2017). Anomalies in the earth's magnetic field are caused by induced or remnant magnetism. Induced magnetic anomalies are the result of secondary magnetization induced in a ferrous body by the earth's magnetic field. The shape, dimensions and amplitude of an induced magnetic anomaly is a function of the orientation, geometry, size, depth and magnetic susceptibility of the body, as well as the intensity and inclination of the earth's magnetic field in the survey area (Biswas, 2016; Biswas and Acharya, 2016). For exploration purposes, both ground and aeromagnetic data have been used to investigate the presence of

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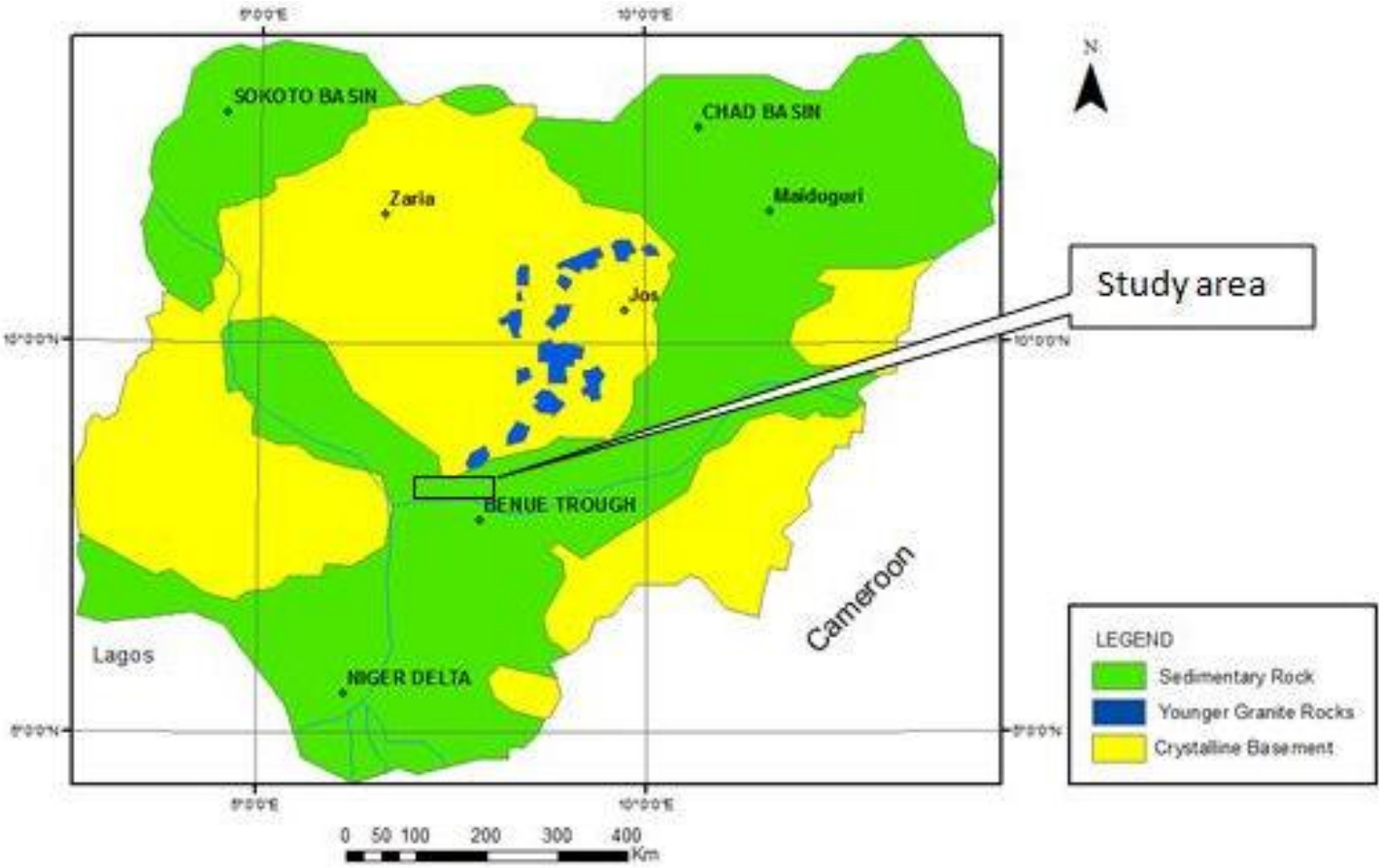

Figure 1. Map of Nigeria showing the study area.
Source: Jatau and Nandom (2013).

mineral deposits in combination with gravity. In the mining industry, both gravity and magnetic methods are still widely used as exploration tools for mapping subsurface geology and to estimate ore reserves for some massive ore bodies (Mandal et al., 2015; Biswas and Sharma, 2016). Nowadays, aeromagnetic surveys are very useful for determining depths to magnetic source rocks of the anomalies over an area, and utilized the principle that the magnetic field measured at the surface can be considered an integral of magnetic signatures from all depths. These anomalies arise as a result of the interactions between the magnetic field and the rocks of the earth crust and also from secondary mineralization along the fault planes in many sedimentary basins. The average depth to sources of ensembles (sedimentary thickness) across the geological area can be obtained from the energy (power) spectrum of the surface field when plotted against frequency in a logarithmic scale. Interpretation of aeromagnetic data can be done quantitatively and qualitatively. Quantitative interpretation involves making numerical estimates of the depth and dimensions of the sources of the anomalies and this often takes the form of modelling of the sources which could in theory, replicate the anomalies recorded in the survey (Biswas, 2015, 2016; Biswas et al., 2017). The computational efficiency and storage capacity of modern digital computers now enable the utilization of a wide range of sophisticated mathematical techniques for data processing and interpretation. Fourier spectral analysis as a mathematical technique has attracted increasing attention in recent years as a powerful tool for the interpretation of potential field data (magnetic anomalies).

The study area lies within the southern portion of the Lower Benue Trough and consists of Sheet 302 (Nkalagu sheet) and Sheet 288 (Igumale sheet) bounded within longitudes 7° 30’ and 8° 00’ East and latitudes 6° 30’ and 7° 00’ North and covering an area of about 6000 km^2. Figure 1 shows the map of Nigeria and location of the study area.

The Lower Benue Trough as a rift system and aulacogen (Olade, 1976) is an area of importance in terms of economic mineral deposits. Consequently, this has inspired a lot of interest on geophysical investigations by many researchers (Ofoegbu and Onuoha, 1991; Obi et al., 2010; Ugwu and Ezema, 2012) to re-examine the economic potentials of this mineral belt using different

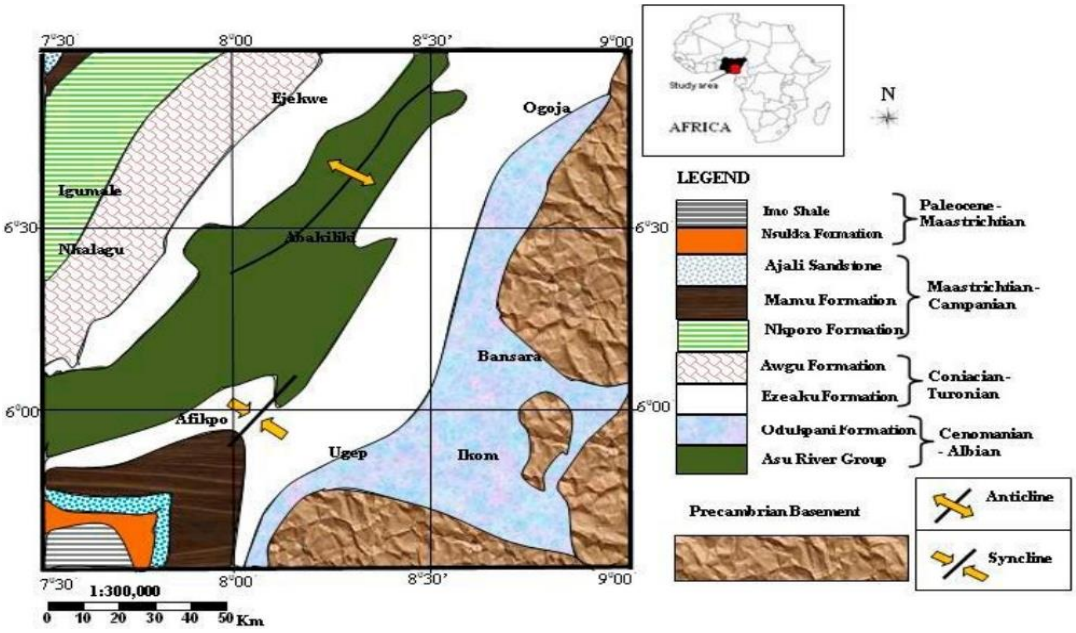

Figure 2. Geological map of some parts of Lower Benue Trough, including the study area. Source: Onuba et al. (2013).

geophysical methods. Previous magnetic anomaly studies over the Lower Benue Trough has shown that the anomalies in the area can best be explained in terms of the combined effects of deep lying basement and intermediate intrusions at both shallow and deep depths (Ofoegbu, 1984, 1985; Ofoegbu and Onuoha, 1991; Ugwu and Ezema, 2012). Ofoegbu (1985) estimated the sedimentary thickness in the Lower and Middle Benue Trough to vary between 0.5 and 7.0 km. Some of the anomalies were interpreted in terms of dykes and volcanic plugs (Ofoegbu, 1984; Ugwu and Ezema, 2012). Previous studies also suggested that Benue Trough has a typical network of fractures and lineaments with dominant trends of NE-SW direction. The intrusive bodies are of variable thickness and different directions of magnetization, which suggest that although they are derived from a common basic mantle material, they were probably emplaced at different polarity epochs (Ofoegbu, 1984).

To contribute to a better understanding of the depth and nature of mineralization, geology and development of the tectonic history in the Lower Benue Trough, we have considered the use of spectral analysis of the aeromagnetic data over Nkalagu and Igumale areas to compute for depths to magnetic sources.

Geology of the study area

The geology and evolution of the lower Benue Trough is now fairly well documented (Wright, 1968, 1976; Nwachukwu, 1972; Olade, 1975; Ofoegbu, 1985; Obaje, 2009). The lower Benue Trough which is underlain by a thick sedimentary sequence was formed as a result of series of tectonics and repetitive sedimentation in the Cretaceous time. The depositional history of the Benue Trough is characterized by phases of marine regression and transgression (Reyment, 1965; Short and Stauble, 1967; Murat, 1972). The major component units of the lower Benue Trough include the Anambra Basin, the Abakaliki Anticlinorium and the Afikpo Syncline. The oldest sediment of the sequence belongs to the Asu River Group which unconformably overlies the Precambrian basement complex that is made up of granitic and magmatic rocks (Ofoegbu and Onuoha, 1991). The Asu River Group whose type outcrops in Abakaliki has an estimated thickness of about 2000 m (Ofoegbu, 1985) and is Albian to Cenomanian. It comprises of argillaceous sandy shales, laminated sandstone units and minor limestones with an interfingering of magnetic volcanics (Nwachukwu, 1972). The shales are fissile and highly fractured. Deposited on top of these Asu River Group sediments in the area are the Upper Cretaceous Eze-Aku shales. The Turonian Eze-Aku shales consist of nearly 1000 m of calcareous flaky shales and siltstones (Reyment, 1965). The geological map of the study area is shown in Figure 2.

MATERIALS AND METHODS

Two aeromagnetic maps were acquired from the Nigerian Geological Survey Agency (NGSA). They are Sheets 302 (Nkalagu) and 288 (Igumale). The data were acquired along a flight line trend of 135° with a spacing of 500 m and an average flight elevation of

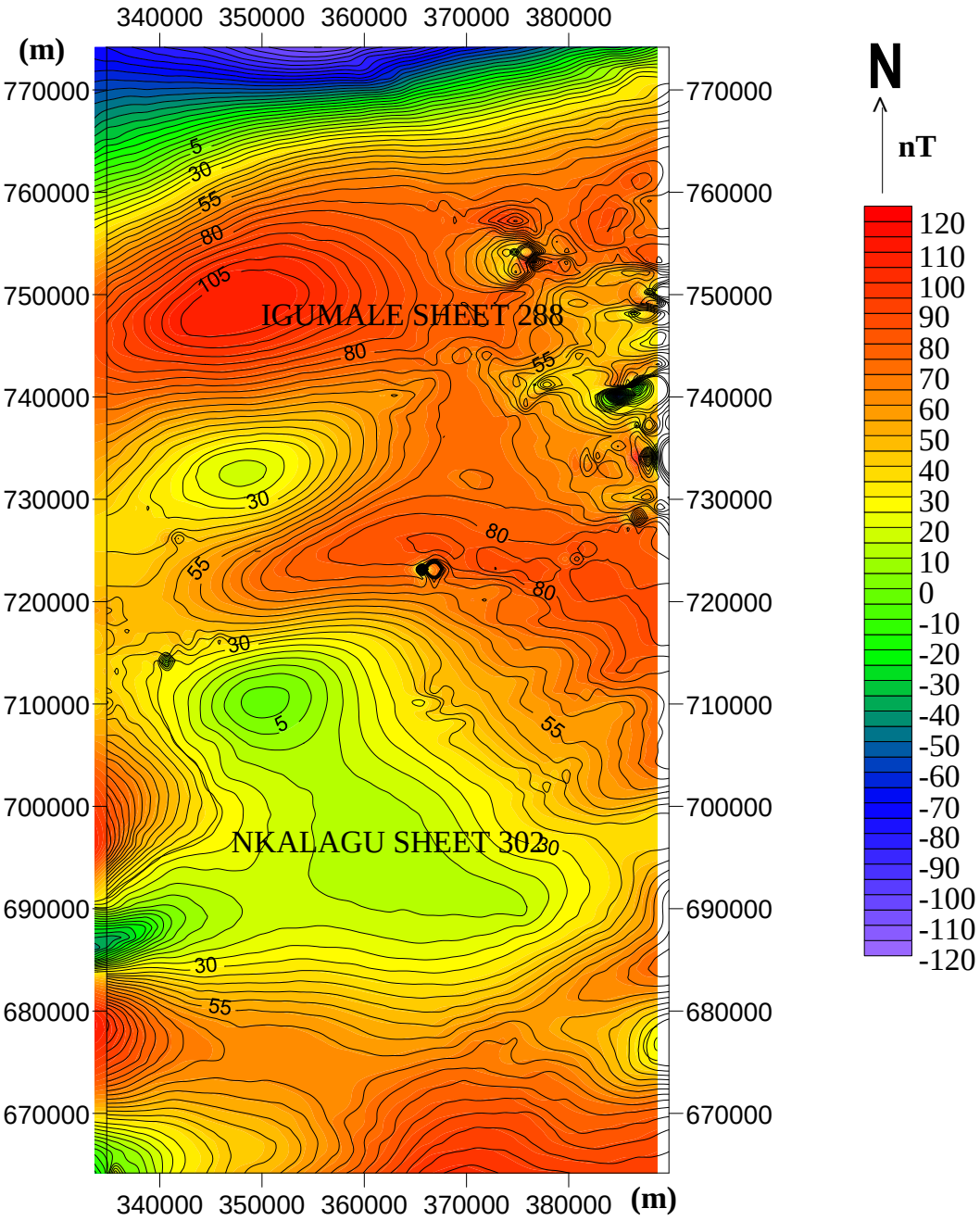

Figure 3. Total magnetic field intensity map of the study area.

75 m, while the tie lines trend and spacing are 225° and 500 m, respectively. The data were made available in digital form on scale of 1:100,000.

The geomagnetic gradient was removed from the data using the International Geomagnetic Reference Field (IGRF). The data sheets of Nkalagu (302) and Igumale (288) were combined using Microsoft Excel 2010 to form the study area covering a total area of about 6000 km^2. The combined sheet was used to produce the total magnetic field intensity (TMI) map (Figure 3) of the study area employing WINGLINK Software and Surfer 10 binary grids. The regional-residual separation was done by polynomial fitting. Here, all the regional fields were calculated as two-dimensional first degree polynomial surfaces. Figure 4 shows the residual anomaly map of the study area after the regional-residual separation. The residual field was also gridded into horizontal and vertical derivatives (Figures 5 and 6). The 3-D surface anomaly map of the study area is also shown in Figure 7.

In order to calculate the depth to basement of the study area using spectral analysis, the study area was divided into six profiles with 18 cells containing 18 × 18 data points. In doing this, it was ensured that essential parts of each anomaly were not cut off in the cells as each cell was made to contain more than one maximum, as suggested by Hahn et al. (1976). To achieve this, the cells were made to overlap each other using Excel 2010 program by applying

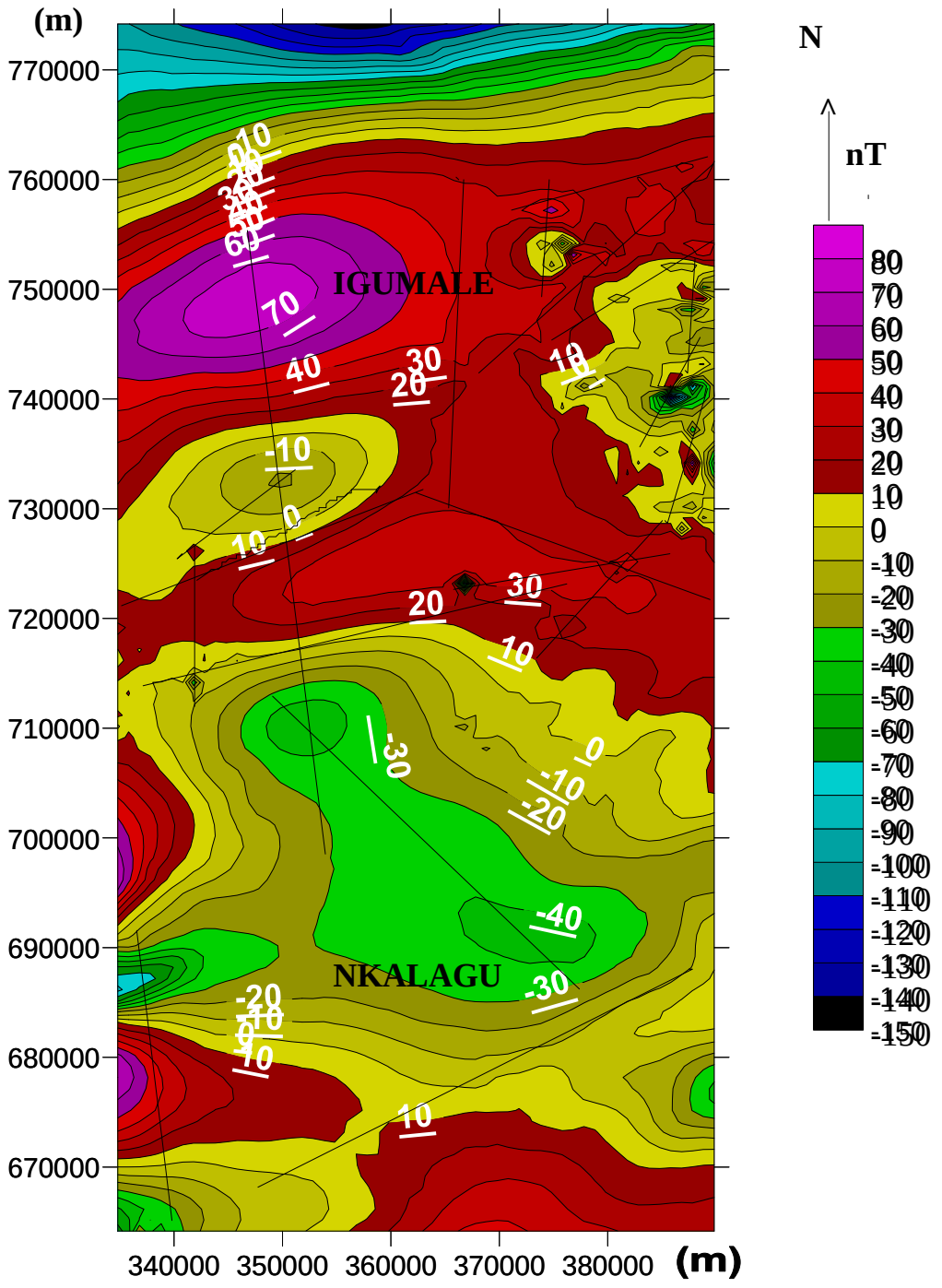

Figure 4. Residual anomaly map of the study area.

inequality formulas in which degree of overlap was insignificant. The Excel software which has the Fourier mathematical algorithm was used to compute the Fast Fourier Transform (FFT) magnitude and FFT frequency. The energy (magnitude) which is in logarithmic scale was plotted against logarithm frequency to produce a decay-curve segment which decreases in slope with increasing frequency. The depths to sources of ensembles were obtained by manually fitting a straight line to each linear interval of the logarithmic energy-decay curve which yields a negative slope. The line of the first linear interval was fit from the deviation of the decay curve from the vertical axes of the logarithmic energy. The second line of the second linear interval was drawn from the deviation of the decay curve from the line of the first linear interval. This was done sequentially such that there is no other possible linear interval between the first and second linear intervals.

Smoothing was done to the signals to filter and determine a trend (linear curvature) on the decay curve by removing high frequency noise. The effect of this filter was to average the value at a given grid

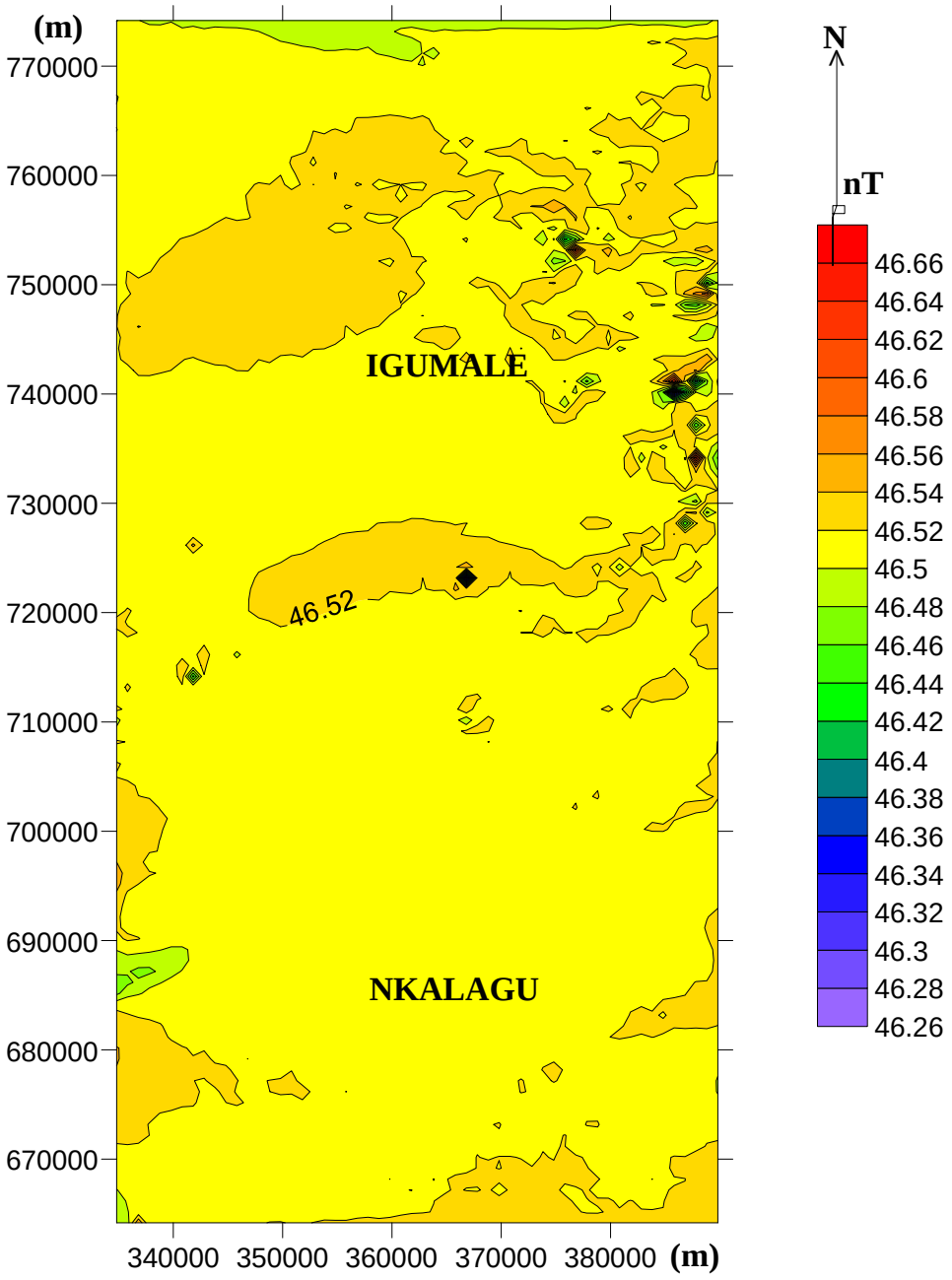

Figure 5. Vertical derivative anomaly map of the study area.

node together with the values at its eight nearest neighbours to give a new value at the centre point. The smoothing operator was moved repeatedly row-by-row over the whole grid to produce new gridded values. The problems of aliasing effect and Gibb's phenomenon which are normally encountered in the course of performing spectral analysis were taken care of by the use of small sampling intervals to reduce frequencies greater than Nyquist frequency.

RESULTS

The spectral plots of logarithmic energy against frequency for the six representative cells of the profiles are shown in Figures 8 to 13. Two-depth models have been estimated for all the cells and these values are summarized in Table 1. The slope is given by the equation:

$$S = \frac{\Delta LogE}{\Delta F} = \frac{LogE_2 - LogE_1}{F_2 - F_1}$$

While the depth to slope relationship is given by D= -S/4π when frequency is in cycle/km (Bonde et al., 2014).

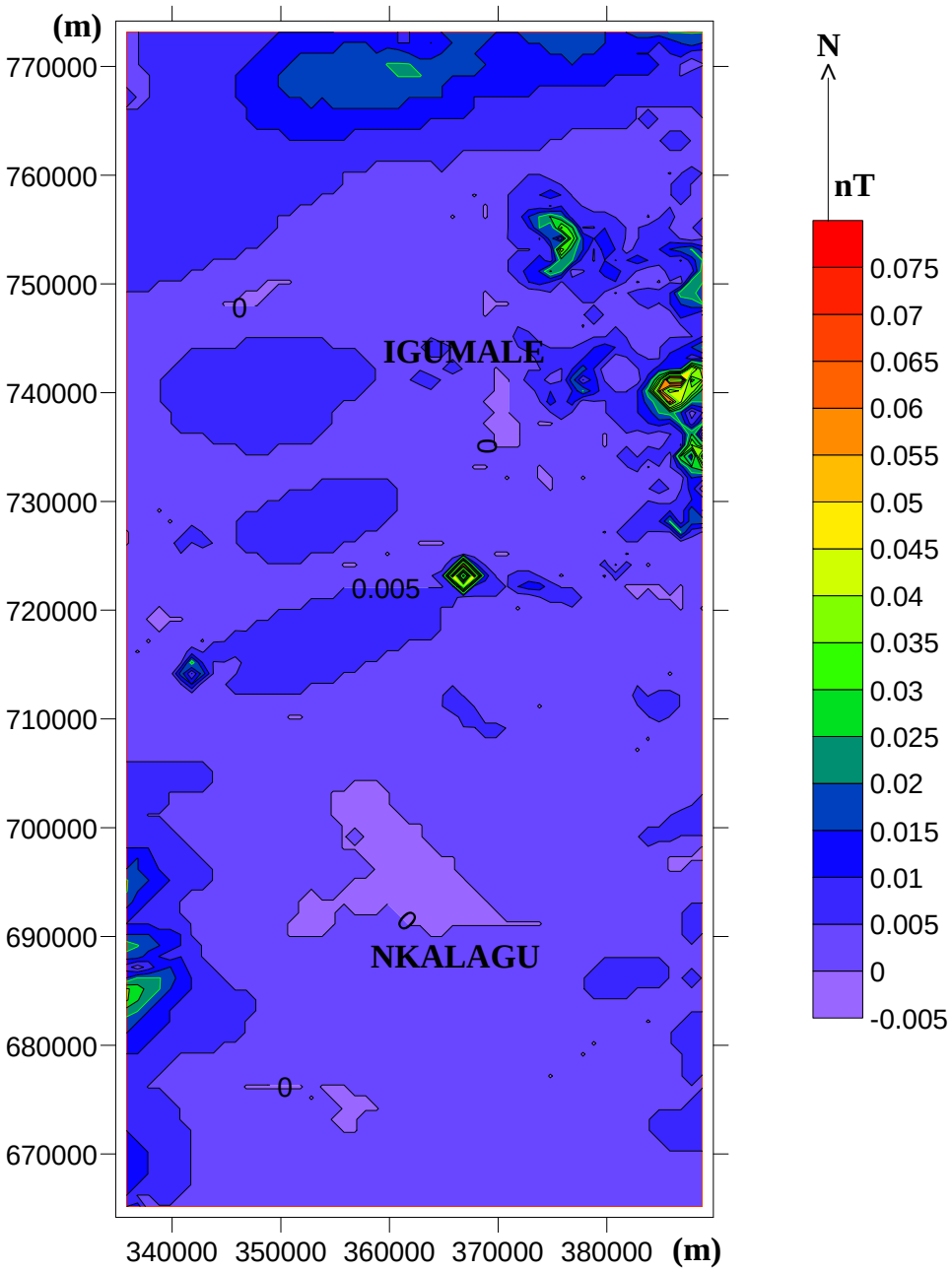

Figure 6. Horizontal derivative anomaly map of the study area.

DISCUSSION

A visual inspection (subjective) of the total magnetic intensity (TMI) and residual maps show that the contour lines of the central and southern parts are widely spaced, suggesting that the depths to magnetic basement in these areas are relatively high. At the northern part of the maps, the contour lines are more closely together, suggesting that the depth to basement is shallow at this part. Also, there are spikes at the northeastern part which shares a geologic boundary with Ejekwe (very similar to Abakaliki, geologically) indicating the presence of magnetic mineral rocks or intrusive bodies within the sedimentary cover. So it is possible that the existence of intrusive bodies within the Abakaliki anticlinorium of the Benue Trough (Ofoegbu and Onuoha, 1991; Ugwu and Ezema, 2012; Abdulahi et al., 2014) extends to Igumale. The distribution of high magnetic intensities from just above the central part toward the southern part of the study area suggests the existence of deep penetrating fractures within the area while the low magnetic intensity indicated by closures and the linear sub parallel

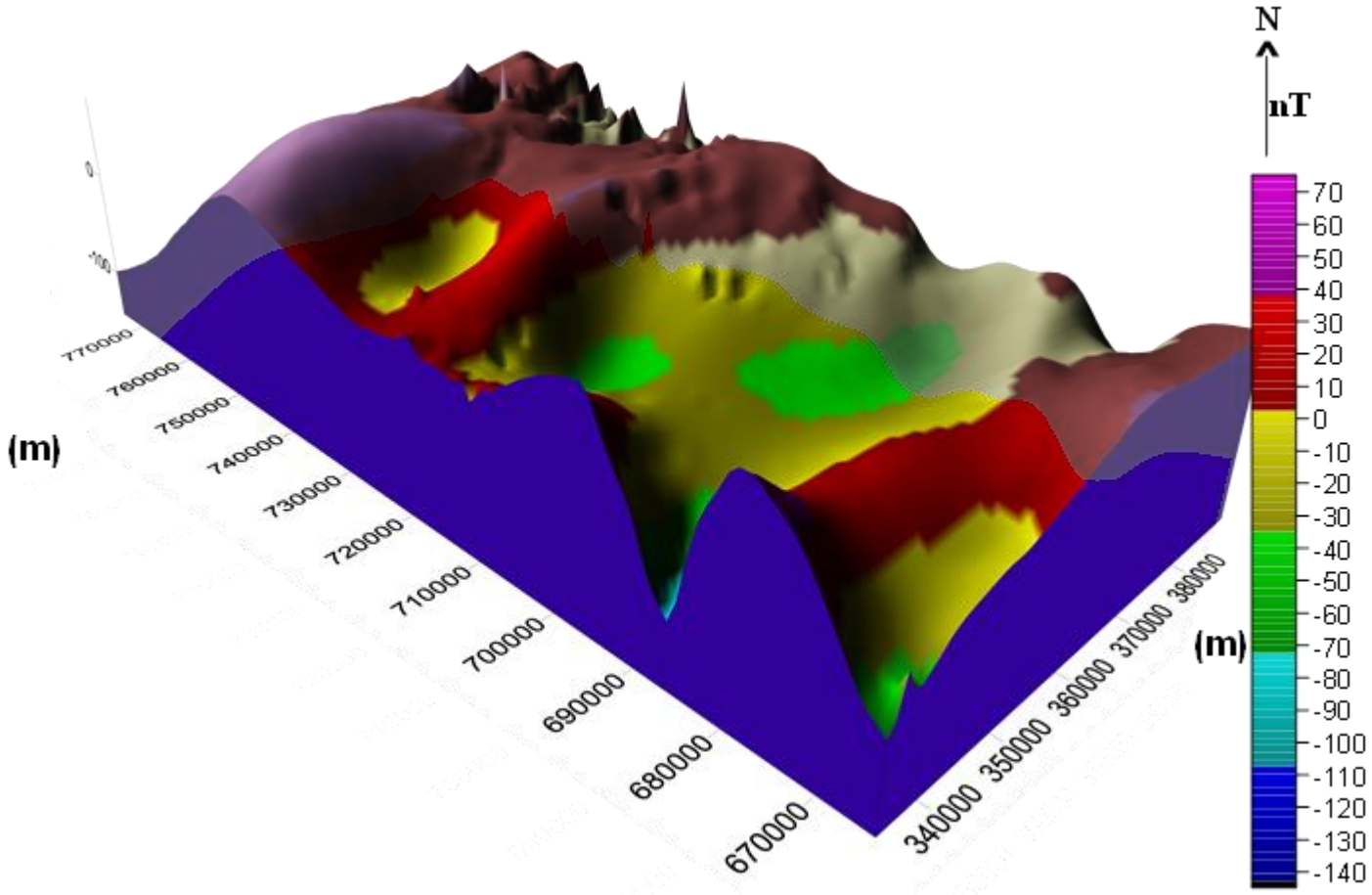

Figure 7. 3-D surface anomaly map of the study area showing the basement topograghy.

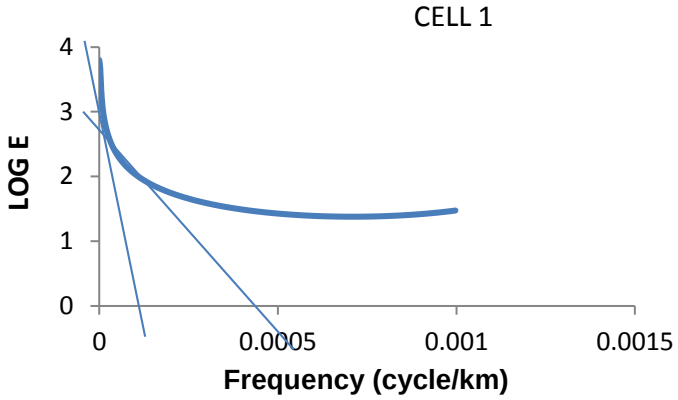

Figure 8. Cell 1 of Profile One.

orientations of the contours at the uppermost northern part of the map suggest a shallow geologic structure. Structural trends within the study area as shown by delineation in the residual map are mainly NE-SW and NW-SE directions which agree with the fault orientation within the Benue Trough, with the NE-SW trends being dominant.

The 3-D surface map of the study area (Figure 7) shows high basement, relief indicating folded and undulating topography, which can be interpreted as part

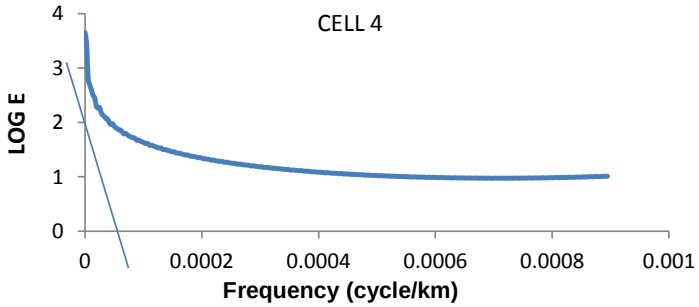

Figure 9. Cell 5 of Profile Two.

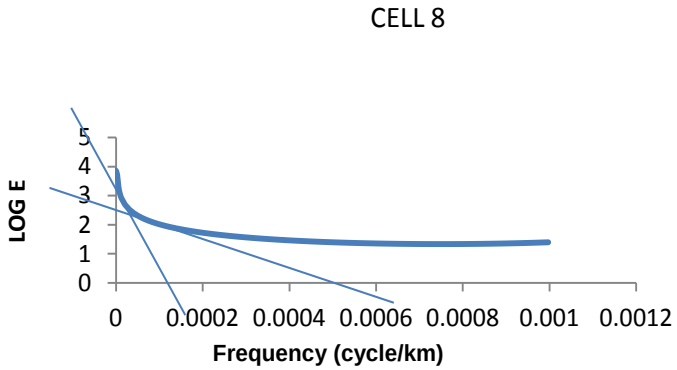

Figure 10. Cell 8 of Profile Three.

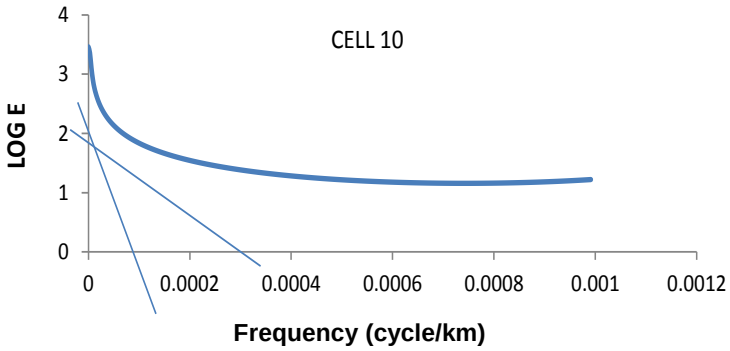

Figure 11. Cell 10 of Profile Four.

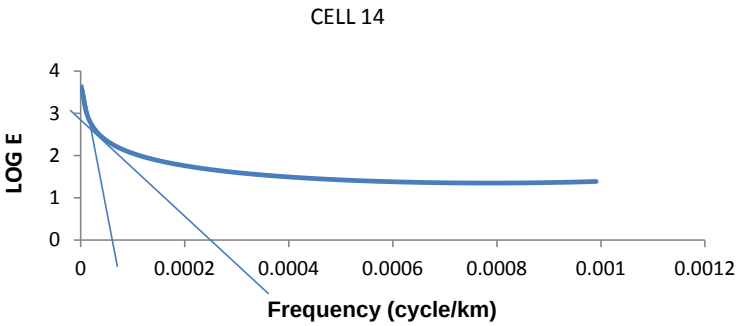

Figure 12. Cell 14 of Profile Five.

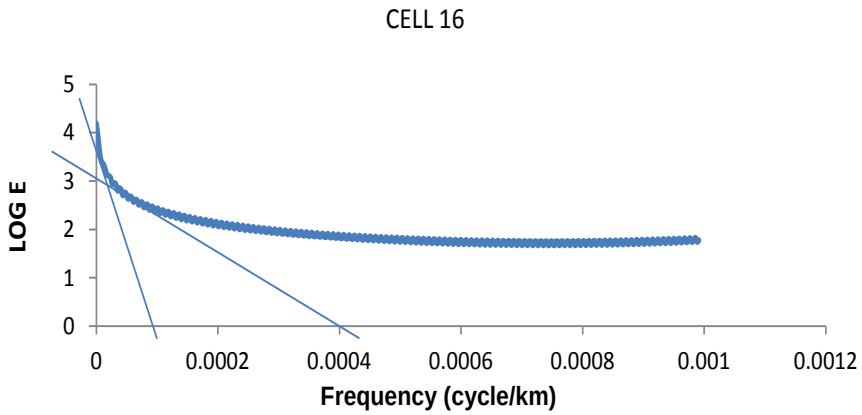

Figure 13. Cell 16 of Profile Six.

Table 1. Summary of the estimates of spectral depths to the magnetic basement in the study area.

Cell 16	Cell 17	Cell 18	Profile 6
D1 = 2.67 km	D1 = 4.97 km	D1 = 3.98 km	Average D1 = 3.87 km
D2 = 0.46 km	D2 = 0.94 km	D2 = 0.50 km	Average D2 = 0.63 km
Cell 13	**Cell 14**	**Cell 15**	**Profile 5**
D1 = 2.39 km	D1 = 4.77 km	D1 = 3.98 km	Average D1 = 3.71 km
D2 = 0.48 km	D2 = 0.35 km	D2 = 0.45 km	Average D2 = 0.43 km
Cell 10	**Cell 11**	**Cell 12**	**Profile 4**
D1 = 4.30 km	D1 = 2.99 km	D1 = 2.98 km	Average D1 = 3.42 km
D2 = 0.40 km	D2 = 0.46 km	D2 = 0. 82 km	Average D2 = 0.56 km
Cell 7	**Cell 8**	**Cell 9**	**Profile 3**
D1 = 2.15 km	D1 4.64 km	D1 = 3.28 km	Average D1 = 3.36 km
D2 = 0=0.42 km	D2 = 0.66 km	D2 = 0.59 km	Average D2 = 0.56 km
Cell 4	**Cell 5**	**Cell 6**	**Profile 2**
D1 = 3.71 km	D1 = 3.58 km	D1 = 5.25 km	Average D1 = 4.18 km
D2 = 0.53 km	D2 = 0.64 km	D2 = 0.82 km	Average D2 = 0.66 km
Cell 1	**Cell 2**	**Cell 3**	**Profile 1**
D1 = 4.38 km	D1 = 3.56 km	D1 = 4.00 km	Average D1 = 3.98 km
D2 = 0.80 km	D2 = 0.66 km	D2 = 0.99 km	Average D2 = 0.82m

of the Abakaliki folded belt. The results of the vertical derivative anomaly maps indicate the lithologic boundaries between different formations underlying the area. The horizontal derivative map shows the possibility of faults or local fractured zones passing through the north central and central part of the study area.

Results of the spectral analysis of the aeromagnetic data over the study area indicated a two-depth source model. The shallow depth model is between 0.35 and 0.99 km which probably indicates the presence of intrusive bodies. The deep source lies at a depth that varies between 2.15 and 5.25 km. These deep sources represented by the first segment of the spectrum in all the cells of the six profiles reflect the Precambrian basement of the study area. Profile two shows the thickest sedimentary cover in the Nkalagu area (cells 4, 5 and 6 of Table 1). This result closely agrees with the results from other aeromagnetic works which had indicated

depth (sedimentary thickness) of 1.5 to 4 km in Nkalagu (Nur et al., 1994; Obi et al., 2010). The range of the average profile depths implies a nearly evenly distributed sedimentary thickness within the study area and ranges between 3.5 and 4.2 km.

Conclusion

Quantitative interpretation of the aeromagnetic data over Nkalagu and Igumale areas of the Lower Benue Trough has been successfully carried out using spectral analysis to determine depth to the magnetic basement. Structural interpretation of the residual anomaly map was also used to delineate the basement morphology, relief, and the structural features associated with the basin and their trends. The results of the study have shown that the area is characterized with an average sedimentary thickness of 3.75 km for the deep source model. From economic point of view, the results indicate possible mineralization within Igumale area due to presence of a few intrusive bodies within the area. The results also show very high possibility for hydrocarbon occurrence due to the existence of folded basement, faults/fractures capable of trapping hydrocarbons. The occurrence of reasonable Cretaceous sedimentary thickness in the area supports the hydrocarbon potential of the area.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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Analysis of organochlorine and organophosphorus pesticide residues in blood samples of sheep and rabbits from villages of Jimeta-Yola, Adamawa State, Nigeria

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Blood samples of sheep and rabbits from five different villages of Jimeta-Yola, Adamawa State, Nigeria were collected for determination of organochlorine and organophosphorus pesticide residues. Preparation of blood samples was carried out using standard procedure of QuEChERS method of extraction. The concentrations of all the pesticides in blood samples of sheep and rabbits were determined using Gas Chromatography–Mass Spectrometry (GC-MS) Shimadzu (GCMS-QP2010), equipped with electron capture detector. Organochlorine and organophosphorus pesticide residues were significantly higher in the blood samples of sheep than in the blood samples of rabbits. According to the concentration and detection frequency, o,p'DDE, p,p'DDD, aldrin and dieldrin were the most dominant compounds among the organochlorine pesticide residues. The results of this study showed that highest concentration of o,p-DDE in blood samples of sheep was detected at Namtari village with a mean value of 0.034±0.001 mg/l. For o,p'-DDT, the highest concentration (0.014±0.001 mg/l) was observed at Wurojabbe, p,p'-DDD had the highest concentration at Doubeli with a value of 0.028±0.001 mg/l. Also, the highest concentration of aldrin in the blood samples of sheep was detected at Namtari with a value of 0.028±0.001 mg/l. For dieldrin, the highest concentration was recorded also at Namtari with a mean value of 0.036±0.001 mg/l. Despite the bans and restrictions on the use of some of these pesticides in Nigeria, the observed concentrations of the studied pesticides from these villages could explain either their persistence in the environment or continued use in the study area. Hence, routine monitoring of pesticide residues in these villages is necessary for prevention, control and reduction of environmental pollutions, so as to minimize health risks.

Key words: Animals blood, pesticides, extraction, clean-up, gas chromatography-mass spectrometry (GC-MS).

INTRODUCTION

Various scientists from different parts of the world have attempted to assess the level of pollution in different environmental media and at times related the results with plants and animals living in the environment. The need to preserve our environment is an important component of sustainable development of a society, with a view to maintaining ecological balance, and improving the quality of life and conditions that may affect human and animal

health (Milam et al., 2015).

There is a need for the growing demand for food productivity to meet the needs of the rising global population. This has led to advance agricultural technology and practices in which pesticides play an important role. Pesticides are generally used to increase the productivity of agricultural product (John et al., 2001). Pesticides have been widely used throughout the world since the middle of the 20th century. Pesticide plays a vital role in agricultural and animal production, though they contain substances with high toxic effects and persistence in the environment (Beyer and Biziuk, 2008). The toxicity of these pesticides goes beyond their biological activity and unfortunately includes toxicological actions toward animals (Imran et al., 2002). Pesticides generally have bioaccumulation ability; their persistence in the environment make them not only remain where they are applied but instead are found between the soil profiles and are absorbed in the soil several kilometers from where they were initially applied (Agarwal, 2009).

Organochlorine (OC) compounds are lipophilic and metabolized very little in sheep and rabbits. Therefore, an exposure of animal to this organochlorine (OC) compounds results in bioaccumulation and persistence in animal tissues (Falandysz et al., 2004). Pesticides containing organochlorine (OC) compounds undergo bioamplification through the food chain (Angulo et al., 1999; Borgå et al., 2001). Fagnani et al. (2011) defined organophosphorus (OP) pesticides as esters, amides or thiol derivatives of phosphoric acid. OP pesticides are easily hydrolyzed and therefore do not persist in the environment. However, their toxicity and the possibility of their accumulation especially fat-soluble OPs in animal blood, tissues, milk, and eggs pose risks for human health (Fagnani et al., 2011).

Pesticide residues in rabbits and sheep, generally accumulate by several ways. These are either applied to livestock through insecticide-impregnated ear tag, spray, self-treatment back rubber, dust bags injectable or spray of agricultural crops and fodder. The advantages of the use of pesticide on agricultural production range from protection of crops against insects to control of pests and diseases (Eskenazi et al., 2009). EU European Commission (2007) reported that animal blood is the most accessible body fluid for ascertaining the pesticide residue levels. The evaluation of serum levels of pesticides can be used as a biomarker of exposure for evaluating the health effects at certain levels (EU European Commission, 2007).

To ensure consumers safety and commercialization, permissive residue levels (PRLs) have been determined for pesticide residues in products of plant and animal origin. Legislation in the European Union has established MRLs of 40 and 0.8 ng/g for the target OC pesticides dichloro-diphenyl-trichloroethane and endrin, respectively, in milk. The PRL for chlorpyrifos is ≤10 mg/g (Ullah et al., 2010). Most authorities of developing countries, however, maintain that they cannot afford to ban certain chemicals for reasons of cost, efficacy or both. As a result, most of these chemicals have been or continue to be used in large quantities in many countries, including sub-Saharan Africa (Ahmed, 1989). Several studies on soil and water pollution have been investigated in Egypt, where wide use of OC and OP pesticides in the 1960s and 1970s prompted the government to prohibit their use in 1980s (El-Sebae and Soliman, 1982; Soliman et al., 1997). However, OC and OP pesticides are still being used for agricultural practices in sub-Saharan Africa (Amr et al., 1995; Dogheim et al., 1990; California Department of Pesticide Regulation (CDPR), 2010b). Therefore, this study was conducted to assess the current status of OC and OP pesticide residue contamination in blood of sheep and rabbits in Yola, Adamawa State, Nigeria, particularly since the ban on the use of these pesticides in agriculture.

MATERIALS AND METHODS

Blood samples for pesticides analysis were collected severally from randomly selected sheep and rabbits from five different villages of Jimeta-Yola- Namtari, Ngurore, Jambutu, Doubeli and Wurojabbe. First, composite of the samples was made; threreafter 5 ml of blood samples was collected and transferred into residue free heparinized glass vials containing 200 USP units of heparin in 0.2 ml solution with the help of sterilized syringe. Blood samples were transported in dry ice to the laboratory and stored at -20°C until analyzed.

Extraction of pesticide residues in blood

Extraction was based on the method of California Department of Pesticide Regulation (CDPR) (2010b). With some modifications, 5 ml of blood sample was diluted with 25 ml distilled water; thereafter 2.0 ml of saturated brine solution was added and transferred to a separatory funnel and extracted with hexane by shaking the separatory funnel vigorously for 2 to 3 min, releasing the pressure intermittently. The layers were allowed to separate; afterwards, the extracts were also passed through anhydrous $MgSO_4$ and concentrated to about 1 to 2 ml using rotary vacuum evaporator.

Dispersive solid-phase extraction (dSPE) cleanup

From the extract obtained in blood, 1.0 ml was transferred to a 2-ml dSPE Cleanup tube that contains 150 mg of $MgSO_4$, 50 mg PSA sorbent and 50 mg C-18 sorbent and was shaken vigorously for 1 min. Then, the portions of the supernatant were transferred to the

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Table 1. Mean concentrations (mg/l) of some organochlorine pesticide residues in blood samples of sheep from some selected villages of Jimeta-Yola, Adamawa State, Nigeria.

Villages	o,p '-DDE	o,p '-DDT	p,p'-DDD	aldrin	dieldrin	Chlordane
Ngorore	0.023±0.001	0.009±0.001	0.013±0.001	0.016±0.001	0.021±0.001	<0.001
Namtari	0.034±0.001	0.013±0.001	0.024±0.001	0.028±0.001	0.036±0.001	<0.001
Wurojabbe	0.022±0.001	0.014±0.001	0.006±0.001	0.005±0.001	0.019±0.001	<0.001
Doubeli	0.016±0.001	0.008±0.001	0.028±0.001	0.002±0.001	0.007±0.001	<0.001
Jambutu	0.018±0.001	0.002±0.001	0.009±0.001	0.006±0.001	0.012±0.001	<0.001

Instrument detection limit (IDL): 0.001.

Table 2. Mean concentrations (mg/l) of some organochlorine pesticide residues in blood samples of rabbits from some selected villages of Jimeta-Yola, Adamawa State, Nigeria.

Villages	o,p '-DDE	o,p '-DDT	p,p'-DDD	aldrin	dieldrin	Chlordane
Ngorore	0.012±0.001	0.003±0.001	0.006±0.001	0.002±0.001	0.004±0.001	<0.001
Namtari	0.008±0.001	0.001±0.001	0.011±0.001	0.004±0.001	0.008±0.001	<0.001
Wurojabbe	0.002±0.001	<0.001	0.001±0.001	0.002±0.001	0.004±0.001	<0.001
Doubeli	<0.001	0.001±0.001	0.003±0.001	0.001±0.001	0.006±0.001	<0.001
Jambutu	0.003±0.001	0.002±0.001	0.001±0.001	0.002±0.001	0.008±0.001	<0.001

Instrument detection limit (IDL): 0.001.

liquid chromatography–mass spectrometry (LC-MS) certified vial for gas chromatography–mass spectrometry (GC-MS) analysis.

Determination of pesticide residues

The Shimadzu Japan GC/MS (GCMS – QP2010), equipped with electron capture detector was used for the chromatographic separation and was achieved using a HP-5MS 5% Phenyl Methyl Siloxane Column. The oven was programmed as follows: initial temperature 60°C for 0.5 min, then 20°C/min to 300°C for 9 min, with a final run time of 21.5 min and a constant column flow rate of 1 mL/min. The detection of pesticides were performed using the GC-ion trap MS with optional MSn mode. The scanning mode offered enhances selectivity over either full scanned or selected ion monitoring (SIM). In SIM, at the elution time of each pesticide, the ratio of the intensity of matrix ions increase exponentially versus that of the pesticide ions as the concentration of the pesticide approach the detection limit, decreasing the accuracy at lower levels. The GC-ion trap MS was operated in MSn mode and tandem MS function was performed by injecting ions into the ion trap and destabilizing matrix ions, isolating only the pesticide ions. The retention time, peak area and peak height of the samples were compared with those of the standards for quantization.

RESULTS

The mean concentrations of some organochlorine pesticide residues in blood samples of sheep from different villages of Jimeta-Yola, Adamawa State are presented in Table 1. The concentrations of o,p'-DDE range from 0.016±0.001 to 0.034±0.001 mg/l; o,p'-DDT from 0.002±0.001 to 0.014±0.001 mg/l; p,p'-DDD from 0.009±0.001 to 0.028±0.001 mg/l. For aldrin, the concentrations range from 0.002±0.001 to 0.028±0.001 mg/l, while the concentrations of dieldrin range from 0.007±0.001 to 0.036 mg/l, whereas the concentration of chlordane is less than the detection limit. For rabbits, the concentrations of organochlorine pesticide residues in blood samples of rabbits from some selected villages of Jimeta-Yola, Adamawa State, are as presented in Table 2. The concentrations of o,p'-DDE range from 0.002±0.001 to 0.012±0.001 mg/l; o,p'-DDT from 0.001±0.001 to 0.003±0.001 mg/l; and p,p'DDD from 0.001±0.001 to 0.011±0.001 mg/l. Then, for aldrin, the concentrations ranged from 0.001±0.001 to 0.004±0.001 mg/l, whereas the concentrations of dieldrin ranged from 0.004±0.001 to 0.008±0.001 mg/l.

The mean concentrations of some organophosphorus pesticide residues in blood samples of sheep from selected villages of Jimeta-Yola, Adamawa State are presented in Figure 1. The concentrations of dichlorvos ranges from 0.008±0.001 to 0.032±0.001 mg/l; Malathion from 0.005±0.001 to 0.028±0.001 mg/l; Chlorpyrifos from 0.003±0.001 to 0.019±0.001 mg/l; Parathion from 0.004±0.001 to 0.017±0.001 mg/l; and Phosphamidon from 0.013±0.001 to 0.023±0.001 mg/l; whereas the concentration of Ethion was not detected in the entire blood samples analyzed. For blood samples in rabbits as presented in Figure 2, the mean concentrations of Dichlorvos ranges from 0.001±0.001 to 0.012±0.001 mg/l; Malathion from 0.001±0.001 to 0.009±0.001 mg/l;

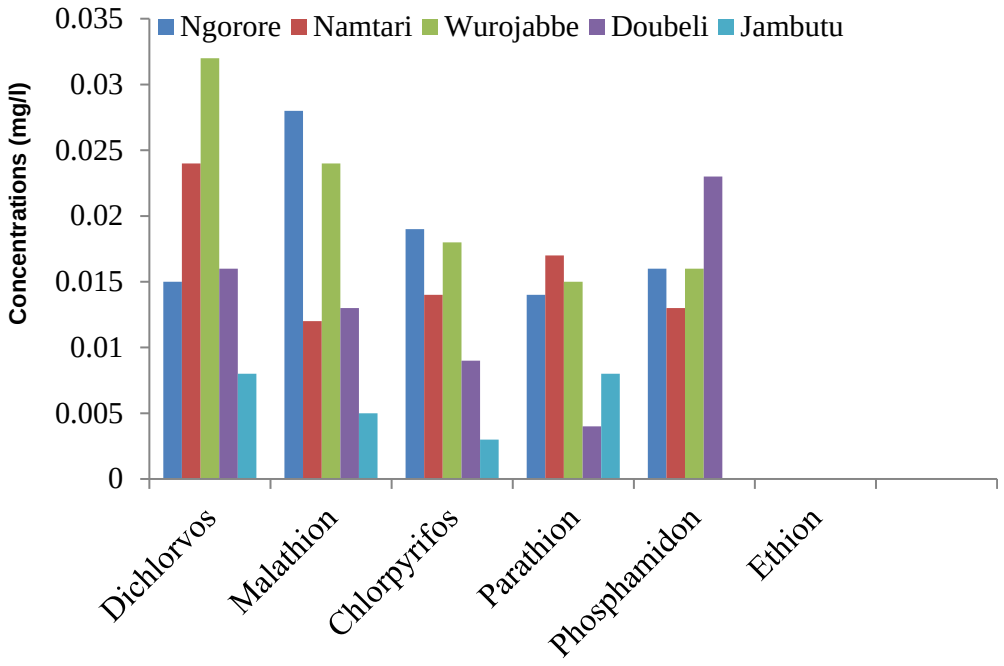

Figure 1. Mean concentrations of some organophosphorus pesticide residues in blood samples of sheep from some selected villages of Jimeta-Yola, Adamawa State, Nigeria.

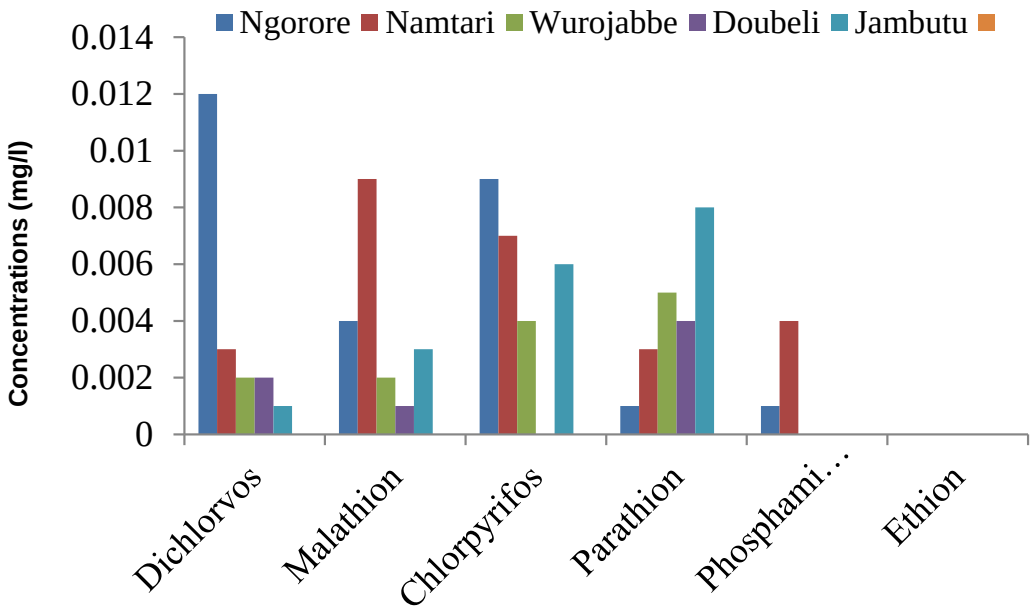

Figure 2. Mean concentrations of some organophosphorus pesticide residues in blood samples of rabbits from some selected villages of Jimeta-Yola, Adamawa State, Nigeria.

Chlorpyrifos from 0.004±0.001 to 0.009±0.001 mg/l; Parathion from 0.001±0.001 to 0.008±0.001 mg/l; and Phosphamidon from 0.001±0.001 to 0.004±0.001 mg/l. Similarly, Ethion was not detected in all the blood samples analyzed.

Comparison of the total mean concentrations of some organochlorine pesticide residues in the blood samples

Figure 3 shows comparison of the concentrations of

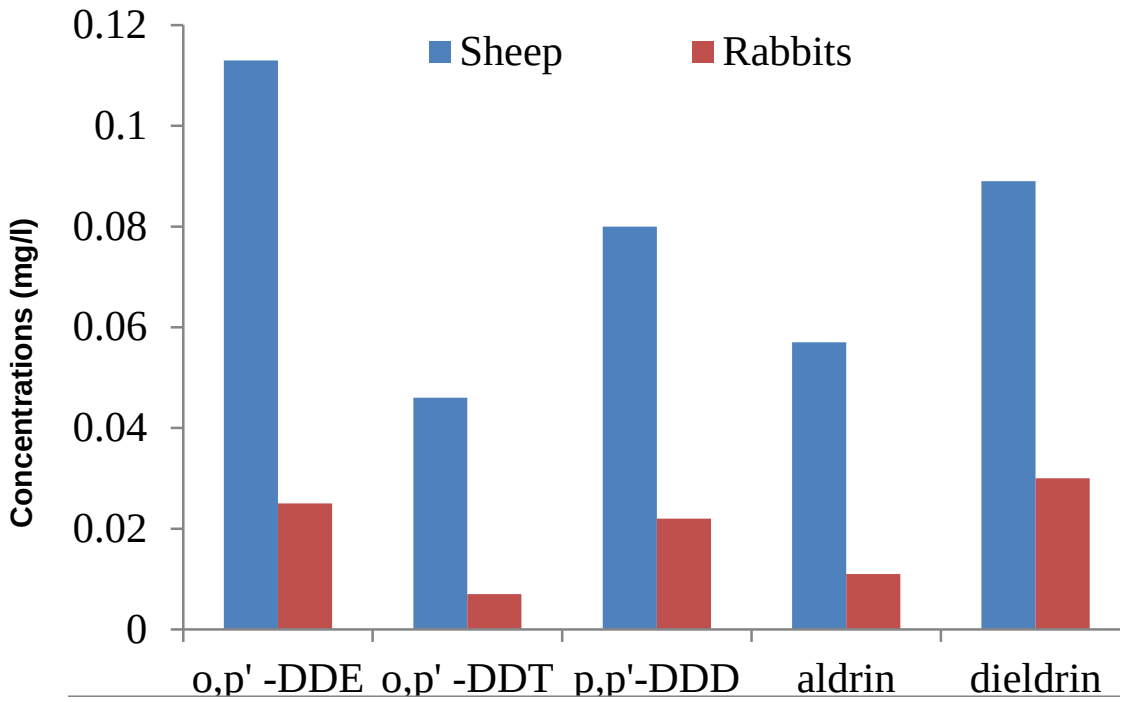

Figure 3. Comparison of the total mean concentrations (mg/l) of some organochlorine pesticide residues between blood samples of sheep and rabbits from selected villages of Jimeta-yola, Adamawa State, Nigeria.

some organochlorine pesticide residues between blood samples of sheep and rabbits. The mean concentrations of o,p'-DDE in the blood samples of sheep was 0.113 mg/l, while in the blood samples of rabbits a total concentrations of 0.025 mg/l was recorded. The mean concentrations of o,p'-DDT in blood samples of sheep was 0.046 mg/l, while 0.007 mg/l was observed in rabbits. Similarly, the mean concentration of p,p'-DDD in sheep was 0.08±0.001 mg/l while 0.022 mg/l was recorded as total concentration in blood samples of rabbits. Also, the mean concentration of aldrin in blood samples of sheep was 0.057 mg/l while total concentration in blood samples of rabbits was 0.011 mg/l. For dieldrin, the mean concentration of 0.089 mg/l in blood samples of sheep was recorded, while 0.03 mg/l in blood samples of rabbits was recorded.

Figure 4 shows comparison of the concentration of organophosphorus pesticide residues between blood samples of sheep and rabbits. The concentration of dichlorvos in blood samples of sheep was 0.095 mg/l, while in rabbits, 0.02 mg/l was recorded. Malathion concentration of 0.082 mg/l was recorded in blood samples of sheep and 0.019 mg/l in rabbits. The mean concentration of chlorpyrifos in blood samples of sheep is 0.063 mg/l, whereas in rabbits, a concentration of 0.026 mg/l was recorded. Also, the total mean concentration of parathion in blood samples of sheep was 0.058 mg/l, while that of blood samples of rabbits was 0.021 mg/l. In addition, the total mean concentration of phosphamidon in blood samples of sheep was 0.068 mg/l, and that of blood samples of rabbits was 0.005 mg/l.

DISCUSSION

Organochlorine pesticide residues (o,p'-DDE, O,p'-DDT, p,p'-DDD, aldrin, diedrin and chlordane) in the blood samples

In blood samples of sheep, the highest concentration of o, p-DDE in blood samples of sheep was detected at Namtari village with a value of 0.034 ± 0.001 mg/l, while the lowest concentration was detected at Doubeli with a value of 0.016 ± 0.01 mg/l. For o,p'-DDT, the highest concentration (0.014±0.001 mg/l) was observed at Wurojabbe, whereas Jambutu village showed the lowest concentration (0.002±0.001 mg/l). Also, p,p'-DDD had the highest concentration at Doubeli with a value of 0.028±0.001 mg/l and the lowest concentration was detected at Wurojabbe with a value of 0.006±0.001 mg/l. Also, the highest concentration of aldrin in the blood samples of sheep was detected at Namtari with a value of 0.028±0.001 mg/l and the lowest concentration was recorded at Jambutu with a value of 0.002±0.001 mg/l. For dieldrin, the highest concentration was also recorded at Namtari with a mean value of 0.036±0.001 mg/l, while the lowest concentration was recorded at Doubeli village with a mean value of 0.007±0.001 mg/l. Whereas,

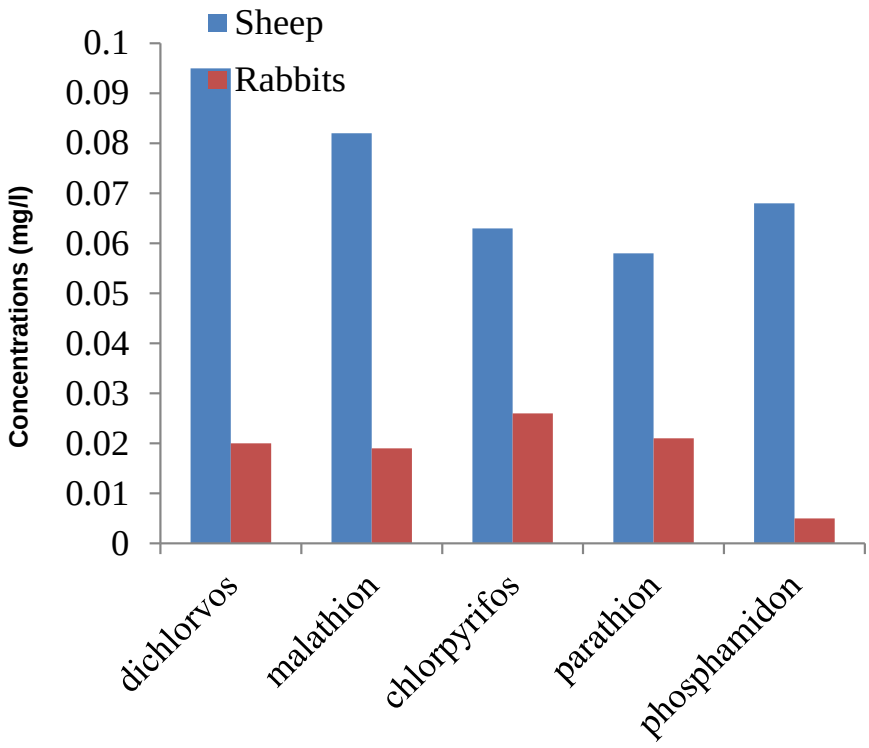

Figure 4. Comparison of the total mean concentration (mg/l) of some organophosphorus pesticide residues between blood samples of sheep and rabbits from selected villages of Jimeta-Yola, Adamawa State, Nigeria.

Chlordane was not detected in any of the blood samples analyzed, because either the animals have not been exposed to it or the chemicals are not available in these areas.

For blood samples of rabbits, the highest concentration of o,p-DDE in blood samples was observed at Namtari location with a concentration of 0.008±0.001 mg/l, while the lowest concentration was observed at Wurojabbe with a mean value of 0.002±0.001 mg/l, and also Doubeli location which showed less than detection limit. For o,p'-DDT, the highest concentration was recorded at Ngorore with a mean value of 0.003±0.001 mg/l, whereas the lowest concentration was observed at Namtari and Doubeli location with a mean value of 0.001±0.001 mg/g; meanwhile, less than the detection limit was observed at Wurojabbe. Also, for p,p'-DDD, the highest concentration was detected at Namtari with a value of 0.011±0.001 mg/l and the lowest concentration was observed at Wurojabbe and Jambutu with a mean value of 0.001±0.001 mg/l. Also, aldrin recorded the highest concentration at Namtari location with a value of 0.004±0.001 mg/l and the lowest concentration was detected at Doubeli with a mean value of 0.001±0.001 mg/l. For dieldrin, the highest concentration was detected at Namtari and Jambutu with a mean value of 0.008±0.001 mg/l, while the lowest concentration was detected at Ngorore and Wurojabbe with a total mean value of 0.004±0.001 mg/l. Whereas, Chlordane was not detected in any of the blood samples analyzed.

The total concentrations of dichlorodiphenyltrichloroethane (DDT) and its metabolites in the blood samples of sheep were in the range of 0.046±0.001 to 0.113±0.001 mg/l, while in blood samples of rabbits, it ranges from 0.007±0.001 to 0.025±0.001 mg/l. Based on the concentrations and detection frequency, o,p'DDE, p,p'DDD, aldrin and dieldrin were the most dominant compound among the organochlorine pesticide residues (OCPs). Similar results of OCPs residues in animal's meat have been reported in recent investigations. The concentrations of the degradation products, which are dichlorodiphenyldichloroethylene (DDE) (0.138 mg/l) and dichlorodiphenyldichloroethane (DDD) (0.102 mg/l) in all the blood samples from the villages both in sheep and rabbits, were more than that of the parent compound (0.053 mg/l), DDT, which indicates past usage of the DDT pesticide. DDT normally degrades under aerobic condition to DDE and under anaerobic condition to DDD, thus a higher DDE + DDD/ DDT ratio is an indication of past usage. Although, the use of DDT has been banned in Nigeria since 2008, it is still being used. DDT and its DDE and DDD metabolites persist in the environment and are known to bio accumulate in animal's fat and plant waxes (Akan et al., 2015). DDT, DDD and DDE have all been classified by the National

Agency for Food and Drug Administration and Control (NAFDAC) as probable human carcinogens. Also, long-term exposure to DDT, DDE or DDD induced liver cancer in animals (Agency for Toxic Substances and Disease Registry (ATSDR), 2002). The concentration of o,p'DDT (0.046 mg/l) and aldrin (0.057 mg/l), dieldrin (0.089 mg/l) and its metabolites were higher in the blood samples of sheep when compared with blood samples of rabbits as mentioned above. The high concentration of OC residue in the blood samples of sheep could be attributed to the high level of grazing of these animals in the vicinity. There is also an increase in agricultural activities which has enhanced the use of pesticides in crop and animal production. This is because sheep and other ruminants grazed freely on contaminated environment and drink water from ponds, stream, rivers and other possible contaminated water sources. Sheep in the process are exposed to high levels of organochlorine pesticides in the environment, in agreement with the report of Nwude et al. (2010).

Organophosphorus pesticide residues (Dichlorvos, Malathion, Chlorpyrifos, Parathion, Phosphamidon and Ethion) in the blood samples

The highest concentration of dichlorvos in blood samples was detected at Wurojabbe with a mean level of 0.032±0.001 mg/l, while the lowest concentration was observed at Jambutu location with a mean value of 0.008±0.001 mg/l. For malathion, the highest concentration was recorded at Ngorore location with a mean value of 0.028±0.001 mg/l and the lowest concentration was observed at Jambutu location with a mean value of 0.005±0.001 mg/l. Also, chlorpyrifos had the highest concentration at Ngorore with a mean level of 0.019±0.001 mg/l, whereas the least concentration of chlorpyrifos was observed at Jambutu with a mean level of 0.003±0.001 mg/l. For parathion, the highest concentration was detected at point Namtari with a mean value of 0.017±0.001 mg/l, while the lowest concentration was recorded at Doubeli location with a mean level of 0.004±0.001 mg/l. The highest concentration of phosphamidon was recorded at Doubeli with a total mean value of 0.023±0.001 mg/l, while the least value was detected at Namtari village with a mean value of 0.013±0.001 mg/l, whereas less than the detection limit was observed at Jambutu location. Ethion was not observed in any of the blood samples analyzed.

The highest concentration of dichlorvos in the blood samples of rabbits was observed at Ngorore with a mean level of 0.012±0.001 mg/l, while Jambutu location showed the lowest concentration of 0.001±0.001 mg/l. Also, Malathion recorded the highest concentration at Namtari village with a mean value of 0.009±0.001 mg/l, while the lowest concentration was observed at Doubeli location with a total mean of 0.01±0.001 mg/l. Also, the highest concentration of chlorpyrifos was detected at Ngorore 0.009±0.001 mg/l, while the lowest concentration was recorded at Wurojabbe with a value of 0.004±0.001 mg/l and less than detection limit was observed at Doubeli. For parathion, the highest concentration of 0.008±0.001 mg/l was observed at Jambutu, with the lowest concentration of 0.001±0.001 mg/l at Ngorore. Phosphamidon recorded the highest concentration at Namtari location (0.004±0.001 mg/l), while the lowest concentration was observed at Ngorore with a value of 0.001±0.001 mg/l and less than the detection limit was observed at Wurojabbe, Doubeli and Jambutu. Also, Ethion was not detected in any of the blood samples analyzed.

Organophosphorus pesticides are widely used as agricultural insecticides and also have many uses in households for pest control. The mean concentrations of organophosphorus pesticide residues in the blood samples of sheep ranged from 0.003±0.001 to 0.032±0.001 mg/l, while that of blood samples of rabbits ranged from 0.001±0.001 to 0.012±0.001 mg/l. According to the concentrations and detection frequency, Dichlorvos (0.032±0.001 mg/l), Malathion (0.028.±0.001 mg/l) and Phosphamidon (0.023±0.001 mg/l) were the compounds with the highest organophosphorus pesticide residues in the blood samples of sheep. The high concentration of OP residue in the blood samples of sheep could be attributed to the high level of grazing of these animals in these areas. There is also an increase in agricultural activities which has enhanced the use of pesticides in crop and animal production. The maximum residue limit (MRL) is the maximum amount of the pesticide residue which is found in food substances that will not cause any health hazard (Yasmin and Souza, 2010).

Conclusion

The present study analyzed a wide variety of pesticides in animal blood samples from some selected villages of Jimeta-Yola. Generally, the residues in blood samples of both the sheep and rabbits were at very low concentrations because as soon as it enters into the body, most of the chemicals may get metabolized and the metabolites may accumulate to induce toxic effects. Despite the bans and restrictions on the use of some of these pesticides in Nigeria, the observed concentrations of the studied pesticides from the villages could explain either their persistence in the environment or continued usage in the study area. Hence, routine monitoring of pesticide and its usage in these villages is necessary for prevention, control and reduction of environmental pollution, so as to minimize health risks.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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Assessment of natural radionuclide levels in some Nigerian made poultry feedstuff

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This study investigated the radionuclide contents of naturally occurring radionuclides. Ten samples of some Nigerian made poultry feedstuff and supplements were collected and analysed using NaI(Tl) gamma(γ)-ray spectroscopy for an accumulated time of about 29000 s. The results revealed that activity concentration of primordial radionuclides ^{40}K, ^{226}Ra, and ^{232}Th for the samples ranged from 43.6 to 196.8 Bq/kg, 5.0 to 34.7 Bq/kg and 0.9 to 51.6 Bq/kg, respectively. The presence of anthropogenic radionuclides was not detected. The Nigerian poultry fodders were found to have relatively higher activities of ^{226}Ra and ^{232}Th compared to the activities reported for poultry feeds in other countries. However, the radionuclide concentrations are not alarming.

Key words: Natural radionuclides, activity concentration, poultry, feedstuff, gamma ray spectroscopy.

INTRODUCTION

Among Nigerians dwelling in rural areas, poultry meat and eggs are to some extent still considered luxury foods but in the urban areas, poultry is consumed more often due to the relatively higher income; availability of poultry meat either as fresh or frozen products, and the introduction of a chain of fast food outlets, whose recipes and menus are rich in chicken meat and eggs, is on the increase.

There has been a concern with radiation levels in food, because ingestion is one of the most common way radionuclides enter living organisms. These radionuclides find their way into the food chain from soil or air to plants, plant to animals/humans, or from other lower animals to humans.

Animal feeds are developed from an organic base (plants or animals) and are intended to provide the most complete nutrition possible (Filho et al., 2016). In an attempt to improve nutritional value of feedstuff, feed substances derived from the biota that contain some elevated levels of radionuclide activity may sometimes be added to poultry feeds. This may increase the radionuclide concentration in such feeds.

Many Nigerian made poultry feeds comprised soybean, maize, sodium chloride and a vitamin complex called "Premix Vitamínico Mineral" ("Vitamin Mineral Premix"), bone meal and fish meal. Thus, in these feed samples,

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Table 1. Samples identity and collection locations.

S/N	Sample No.	Location	Brand type	Feed formula
1	Samp 1	Benue	Feed supplement	Lysine
2	Samp 2	Benue	Feed supplement	Layer premix
3	Samp 3	Benue	Feed supplement	Methyonine
4	Samp 4	Benue	Feed supplement	Limestone
5	Samp 5	Benue	Feed supplement	Broilers Premix
6	Samp 6	Kaduna	Compounded feed (Rebson feed)	Layer mash
7	Samp 7	Kaduna	Compounded feed(Rebson feed)	Finisher
8	Samp 8	Kaduna	Compounded feed (PLS)	Chick mash
9	Samp 9	Kaduna	Compounded feed (PLS)	Grower mash
10	Sale 10	Kaduna	Compounded feed (PLS)	Layer mash

one must account for the natural radioactivity present in maize, soybeans and all the other components like limestone, phosphate rock which is another raw material used for the production of the feed supplement (Casacuberta et al., 2010), which supplies calcium for domestic animals such as poultry.

The food chain extends to the uptake of these radionuclides by animals from the feedstuff they eat which is deposited in their tissue and finally consumed by human. Thus, as humans ingest both eggs and poultry meat, it is expedient to monitor the radiation levels in poultry feedstuff since a part of the amount of radionuclides in the feed which animals ingest could possibly be transferred to humans via the radionuclide pathway in the food chain (Mc Donald et al., 1999; Breuninger et al., 2002; Hernandez et al., 2004).

Therefore, every source of radionuclide that has an end effect on humans whether directly or indirectly needs to be checked to ensure that substances meant for public consumption have radioactivity levels As Low As Reasonably Achievable (ALARA).

Around the world, researches have reported natural radioactivity levels in poultry fodders. However, there has been little or no reports on the concentration of natural radionuclides in poultry feeds made in Nigeria. Thus, this present work assessed the amount of natural radionuclides in samples of Nigerian made poultry feedstuff and compared the measured values with feedstuff produced in other parts of the world.

MATERIALS AND METHODS

Ten samples of Nigerian made chicken feedstuff and supplements used for poultry feeds were collected, prepared and analysed. Samples were collected on the basis of production and consumption in Nigeria. Sample collection was done by interaction with farmers, milling (manufacturing) companies and a few samples directly purchased from the market. Table 1 shows the sample collections.

Sample preparation

Collected samples of Nigerian made poultry feeds were pulverized to 200-300 mesh size, with a blender and ceramic mortar, and then homogenized to fine powder with a 2 mm-mesh sieve and packed to fill cylindrical plastic containers with inner dimension of 7.2 cm diameter and 6 cm height. This satisfied the selected optimal sample container height (Ibeanu, 1999). Each container accommodated approximately 300 g of sample which were carefully sealed (using vaseline, candle wax and masking tape) to prevent radon escape and then stored for a minimum of 28 days. This is to allow Radium and Thorium attain secular equilibrium with their (daughters) progenies.

Sample analysis

Gamma ray spectrometry technique was employed in the spectral collection of the prepared samples using the higher energy region of the γ-line. A 7.62 × 7.62 cm NaI(Tl) detector by Canberra Inc, housed in a 6 cm thick lead shield and lined with cadmium and copper sheets was used. A computer based Multichannel Analyzer (MCA) MAESTRO programme from ORTEC was used for data acquisition and analysis of gamma spectra. The energy calibration of the spectrometer was performed using certified reference material for radiometric measurement from the International Atomic Energy Agency (IAEA), Vienna. Gamma Standard sources includes: Caesium-137 and Cobalt-60; also, calibration energy of 661.60 keV for Cs-137 or 1173.2, 1332.5 keV for Co-60 was used. The 1764 keV γ line of ^{214}B for U was used in the assessment of the activity concentration of ^{226}Ra, while 2614.5 keV γ-line of ^{208}Tl was used for ^{232}Th. The single 1460 keV γ-line of ^{40}K was used in its content evaluation.

The samples were mounted on the detector surface and each counted for 29,000 s in reproducible sample detector geometry. The configuration and geometry was maintained throughout the analysis.

RESULTS AND DISCUSSION

Activity concentration estimation

All the obtained raw data were converted to conventional units using calibration factors to determine the activity concentration of ^{40}K, ^{226}Ra and ^{232}Th. The radioactivity concentration in the investigated samples was obtained as follows (Abel-Ghany et al., 2009).

$$A = (CPS)_{net}/I \times Eff \times M \quad (1)$$

where A is the activity concentration in Bq/kg, (cps) net is

Table 2. Activity concentrations of poultry feedstuff (Bq/kg).

Sample No.	Brand of feed	^{40}K	^{226}Ra	^{232}Th
1	Feed supplement	103.5±7.0	7.6±4.8	11.7±1.5
2	Feed supplement	134.6±3.6	14.5±0.3	12.1±1.5
3	Feed supplement	43.6±4.7	14.9±2.6	10.9±1.9
4	Feed supplement	168.3±5.2	19.6±0.7	0.9±0.3
5	Feed supplement	131.5±5.8	12.2±1.4	11.6±0.3
6	Compounded feed (Rebson feed)	179.8±4.7	19.2±2.2	23.6±1.4
7	Compounded feed (Rebson feed)	48.7±3.9	9.4±1.5	22.3±0.8
8	Compounded feed (PLS)	160.7±5.6	34.7±2.2	49.6±1.9
9	Compounded feed (PLS)	178.7±2.9	7.6±3.3	37.1±7.9
10	Compounded feed (PLS)	196.8±7.2	5.0±0.3	51.6±1.4

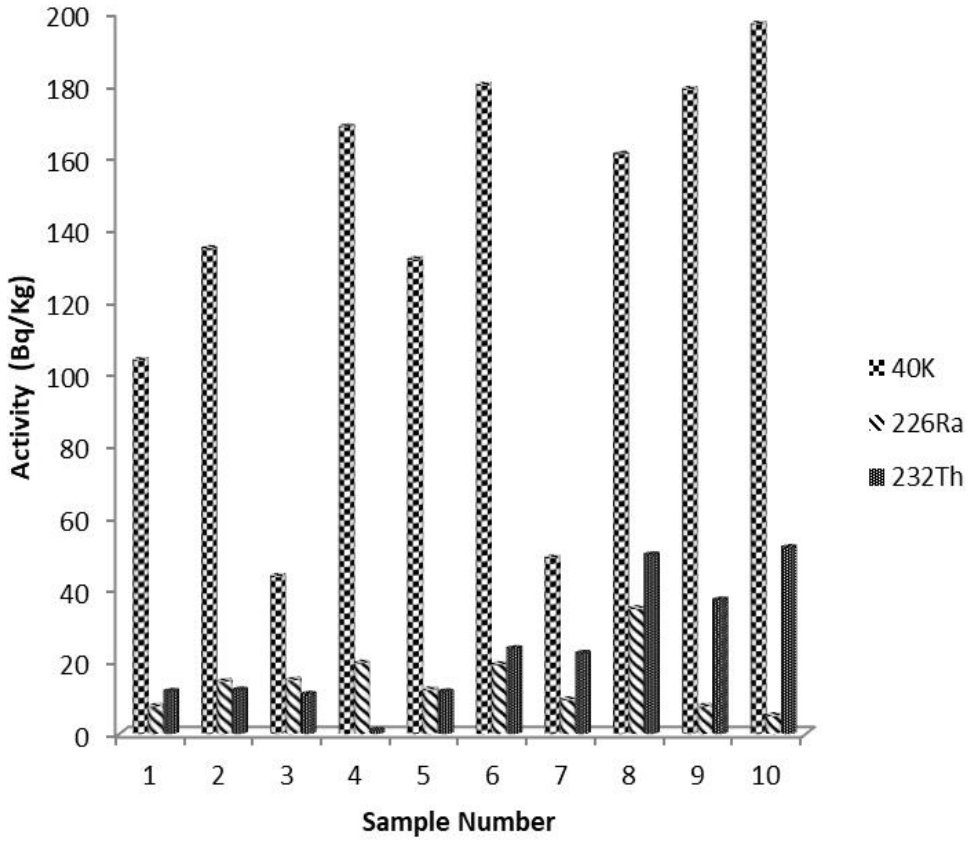

Figure 1. Activity concentration of K-40, Ra-226 and Th-232.

the count per second and equals cps sample-(cps) background. I is the intensity of the γ-line in a radionuclide, E_{ff} is the measured efficiency for each γ-line observed and m is the mass of the sample in kilograms. The quantity $I \times E_{ff} \times m$ is called the conversion factor. The conversion factor for the conversion to Bq/kg for each radionuclide is given thus; K-40 = 0.000643, Ra-226 = 0.000863 and Th-232 = 0.000877.

The activity concentrations of ^{40}K, ^{226}Ra and ^{232}Th were estimated and shown in Table 2 and Figure 1. The results showed that the activity concentrations in feed supplement range from 43.6 to 168.3 Bq/kg for ^{40}K, 7.6 to 19.6 Bq/kg for ^{226}Ra and 0.9 to 12.1 Bq/kg for ^{232}Th, while that of the compounded feed range from 48.7 to 196.8 Bq/kg for ^{40}K, 5.0 to 34.7 Bq/kg for ^{226}Ra, and 22.3 to 51.6 Bq/kg for ^{232}Th. Methionine and layers premix of the

Table 3. Comparison of activity concentration (Bq/kg) of poultry feeds in different locations.

Country	References	^{226}Ra	^{232}Th	^{40}K
Egypt	Harb et al. (2010)	0.35-1.17	0.27-1.07	60.51-91.21
Korea	Choi et al. (2008)	0.026	0.0127	58.5
Brazil	Filho et al. (2016)	0.23-1.51	0.29-1.63	236-402
Nigeria*	Present work	5.0-34.7	22.4-51. 6	48.7-196.8

*****Present research work.

feed supplements had higher values of ^{226}Ra and ^{40}K compared to the other supplements, while chick mash and the two samples of compounded layers mash had higher values of ^{226}Ra and ^{232}Th.

Generally, the compounded feeds had higher values than the feed supplements which is expected, since some of these supplements are used for compounding feeds. The activities' concentrations of ^{226}Ra and ^{232}Th obtained for Nigerian compounded feedstuff shown in Table 3 are relatively higher compared to those of Egyptian, Korean and Brazilian feedstuffs (Harb et al., 2010; Choi et al., 2008; Filho et al., 2016). The presence of anthropogenic radionuclides was not detected which shows that there was no contamination due to artificial radionuclides.

Conclusion

In this study, the activity concentrations of natural radionuclides, ^{40}K, ^{226}Ra and ^{232}Th in different samples of Nigerian poultry feed supplements and compounded feeds were estimated. The results were also compared with other poultry fodders in other parts of the world. Although, the Nigerian poultry fodders had relatively higher activities of ^{226}Ra and ^{232}Th, the radionuclide concentrations are not alarming. By implication, a transfer of such levels to chicken and finally to human in the radionuclide pathway will pose no danger when poultry products of meat and eggs fed from these feeds are consumed eventually by the public.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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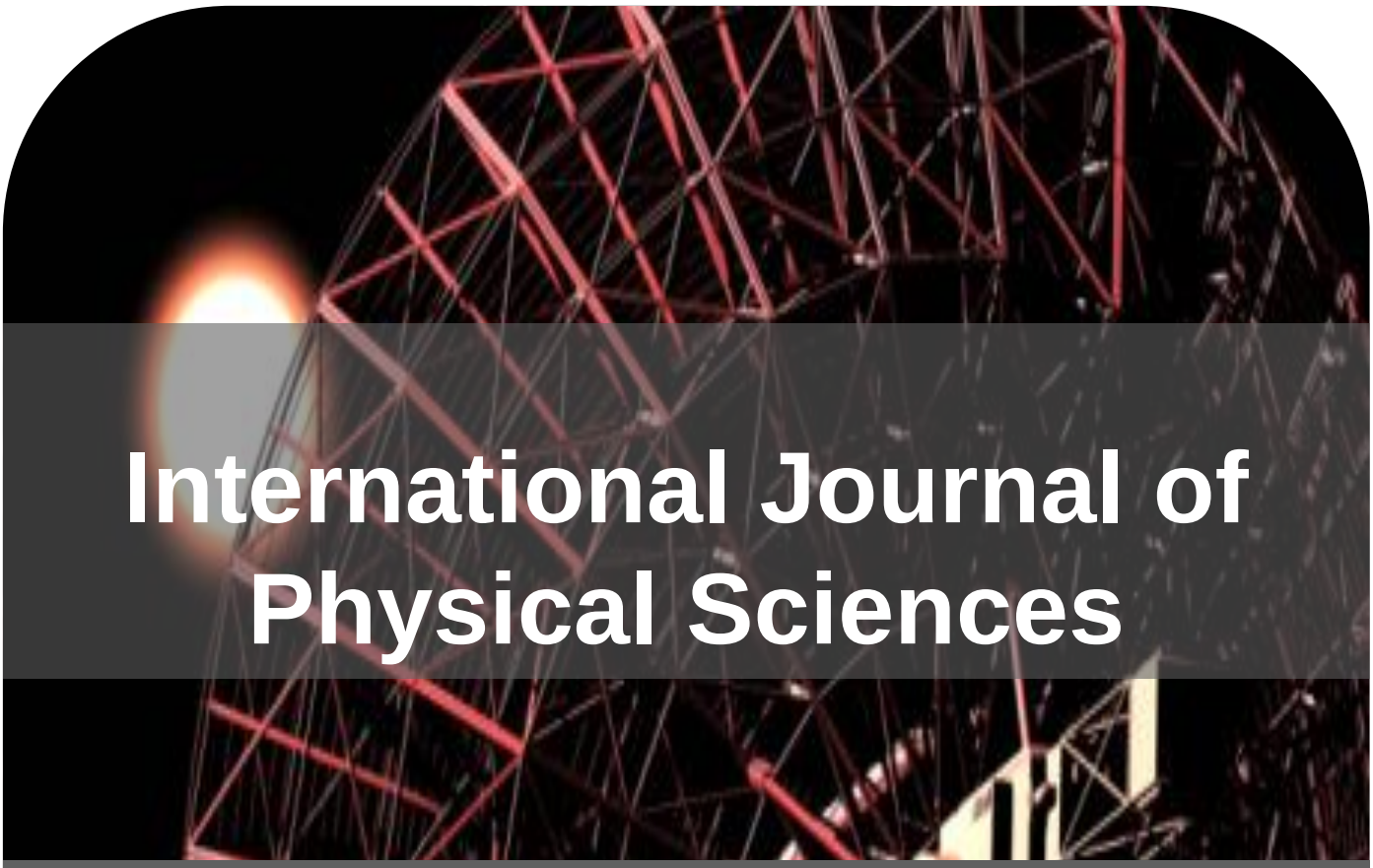

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