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# Analysis of Pesticide Residue and Heavy Metals in Soil of Some Farm Area of Akko, Billiri and Kaltingo Local Government Area of Gombe State, Nigeria

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**ABSTRACT**

This study examined pesticide residues and heavy metals in farms in Akko, Billiri, and Kaltungo local government areas, Gombe State, Nigeria. A 2.5 cm spiral auger gathered soil samples at 0-10 cm, 10-20 cm, and 20-30 cm depths. The three research regions' soil samples were bulked together to produce a 1 kilogramme composite sample and digested using a 5:1:1 trio-acid combination (70% HNO3, 65% HClO4, and 70% H2SO4) at 80oC. The Atomic Absorption Spectrophotometer (AAS, shimadzu japan 6800) measured heavy metals and GC-MS measured pesticide residues. Thirteen organochlorine pesticide residues including Mn, Cd, Pb, Zn, and Cu were found. Higher quantities of organochlorine pesticide residue and heavy metals in this research may be attributable to soil composition, agricultural methods, and environmental variables. The study of organochlorine pesticide residues and heavy metal in Akko, Biliri, and Kaltungo Local Government Areas shows that agricultural pesticides may harm human health. Public understanding of this pesticide's dangers and precautions are needed immediately.

**Keywords:** Soil, Pesticide, Heavy Metals and human health

## INTRODUCTION

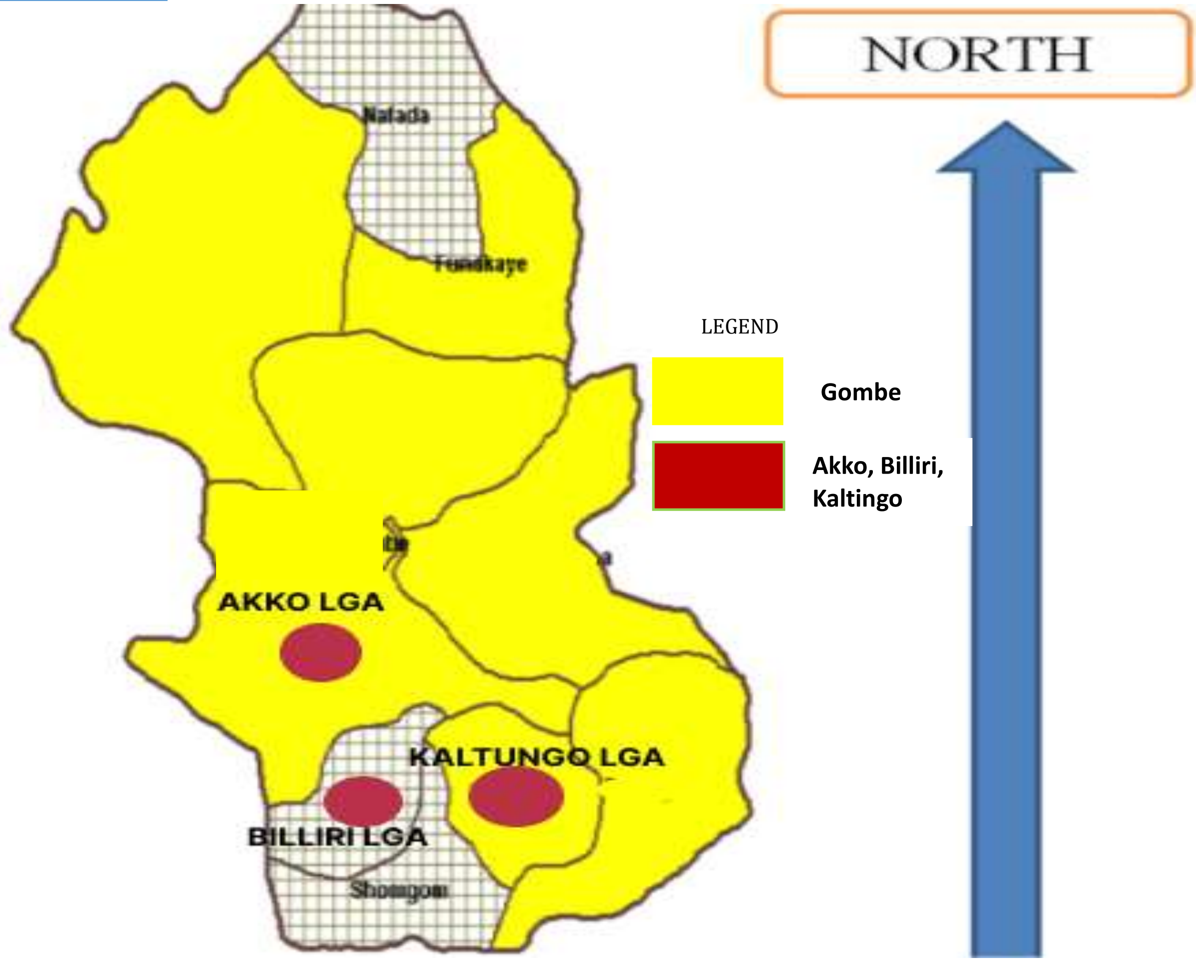
The use of agro-chemicals of various types as well as the performance of some activities such as mining has caused the increase in the level of heavy metal and pesticides residues in farming soil [1]. Therefore, regular check of the level of pesticides residues as well as heavy metals in soil and plant, based on standards of Food Agriculture Organization/ World Health Organization [2], has become an imperative. Pesticide residues constitutes a danger to soil micro fauna and micro flora and their toxic effect manifest on humans when bioaccumulation occurs along the food chain after initial plant uptake, and can cause deleterious effect on soil fertility and crop productivity [3]. Pesticides that are applied on crops are retained to the soil [4]. They enter into cyclic environmental process such as absorption by soil, leaching by $H_2O$ and contaminate both the biosphere and lithosphere. Some arsenic compounds may render the soil permanently infertile. Because the use of pesticides decreases the general biodiversity in the soil. Not using the chemicals results in higher soil quality [5]. Heavy metals are known as non-biodegradable, and persist for long durations in aquatic as well as terrestrial environments [6]. They might be transported from soil to ground waters and vice versa or may be taken up by plants, including agricultural crops [7]. The soil contamination by heavy metals can transfer to food and ultimately to consumers. For instance, plants accumulate heavy metals from contaminated soil without physical changes or visible indication, which could cause a potential risk for human and animal [8]. Based on its persistent and cumulative nature, as well as the probability of potential toxicity effects of heavy metals as a result of consumption of leafy vegetables and fruits, there is a need to test and analyze the consumed food item and soil on which they grow to ensure that the levels of these trace elements meet the agreed international requirements [9]. Soil is the most important component of the environment, despite the fact that it is among the most undervalued, misused and abused earth's resources. Soil contamination has become a serious problem in all industrialized areas of the country [6]. Soil is equally regarded as the ultimate sink for the pollutants discharged into the environment, most plants and animals depend on soil as a growth substrate for their sustained growth and development. In many instances the sustenance of life in the soil matrix is adversely affected by the presence of deleterious substances or contaminants. The entry of the organic and inorganic form of contaminants results from disposal of industrial effluents [6].

Pesticides are a complex mixture of substances used to kill, eliminate, or repel insects, weeds, rodents, fungi, or other organisms [2]. They are being extensively used around the world. Millions of people are exposed to pesticides both environmentally and occupationally. Although exposure to pesticides causes hazardous genetic effects, they are still being used in agriculture [10]. Despite their benefit in increasing crop yield and reduction in postharvest losses in agriculture, their extensive use results in the accumulation of pesticide in food residue, in soil, in runoff water, etc [11]. Pesticides show bio-magnification by entering into the food chain due to high levels of exposure and slow biodegradation [10]. Thus, pesticides pose a threat to public health and the country's economy [4]. The source of the organic and inorganic elements of the soil of contaminated area are mainly caused by the release of untreated effluent on the ground. The contamination of soils with heavy metals or micronutrients generates adverse effects not only on plants but also poses risks to human health [10]. Afterwards, the consumption of contaminated edible plants constitutes an important route of heavy metal exposure to animals and human health. Abandoned waste dumpsites have been used extensively as fertile grounds for cultivating vegetables, though research has shown that the vegetables are capable of accumulating high levels of heavy metals from contaminated and polluted soils [7].

## Methods and Materials

### Study Area

Akko is a local government area of Gombe, Gombe State, Nigeria. Its headquarters is in Kumo town on the A345 highway south of the state capital Gombe. The town of Akko, from which the name of the local government area is originated, is west of Gombe at 10°17′00″N 10°58′00″E. The local government area covers 2,627 km² and had a population of 337,853 at the 2006 census. Billiri (or Biliri) is a Local Government Area of Gombe State, Nigeria. Its headquarters are in the town of Billiri in the northeast of the area on the A345 highway. It is situated at 9°51′53″N 11°13′31″E coordinates. It has an area of 737 km² and a population of 202,144 at the 2006 census. Kaltungo is one among the 11 Local Governments Area of Gombe State, Nigeria. Its headquarters is in the town of Kaltungo in the western part of the Local Government Area on the A345 highway at 9°48′51″N 11°18′32″E. It has a landmark area of 881 km² and a population of 149,805 according to 2006 census.




**Map of Gombe state showing the study area**

**Soil Sampling Protocol**

Before sampling, the surface is cleaned herbs, organic residues and other residues for surface clean soil. Using a spade which has been previously washed with detergent and rinsed with water and acetone. Plant and stones materials has to be off the soil sample. The soil samples will be collected at the depth of 0-10cm, 10-20 cm and 20-30 cm using a spiral auger of 2.5cm. The collected soil sample will be bulked together to form a composite sample of 1 kg according to the three different area of studies namely, Akko, Biliri and Kaltungo LGAs before being placed in a clean labeled plastic bags and transported to the laboratory. The sample will be used for heavy metals and pesticides residue analysis [4] and [12].

**Extraction of soil**

QuEChERS extraction was carried out as described by the European Committee for Standardization. (2018). 5g of the pulverized sample was weighted into a 50 mL polypropylene centrifuge tube, 10 mL of acetonitrile (MeCN) was added followed by the use of a vortex mixer to shake the centrifuge for 30s. NaCl (1 g) and Anhydrous $MgSO_4$ (4g) were added followed by immediate shaking with the vortex mixer for 60 seconds to pre-empt the $MgSO_4$ from becoming aggregates and then, the extract was centrifuged at 2,900 g for 5 minutes. A 3 mL aliquot of the MeCN layer was moved into a 15 mL micro centrifuge tube holding 600 mg of and 120 mg of anhydrous $MgSO_4$ and Primary Secondary Amine, respectively. The vortex was again used to thoroughly 30s before centrifuged at 4000 rpm for 5 minutes. Finally, a 0.2 µm PTFE filter was applied in filtering 1 mL supernatant before taking in a clean vial for injection

## Digestion of Soil

1g of both soil and plants samples were placed into 100 ml beaker separately to which 15 ml of trio-acid mixture (70% $HNO_3$, 65% $HClO_4$ and 70% $H_2SO_4$) were added in ratio 5:1:1. The mixture was digested at 80$^o$C until the solution became clear indicating complete digestion. The resulting solution was then filtered and diluted to 50 ml and later analyzed for metals concentration [13].

## Determination of Pesticide Residues

The SHIMADZU JAPAN GC-MS (Qp 2010), equipped with electron capture detector was used for the chromatography separation and was achieved using a Hp - 5 ms 5% phyenyl methyl siloxane column. The oven was programmed as follows: Initial temperature 60$^0$C for 0.5 min. then 20$^0$C/min. to 300$^0$C for 9 min with a final run times of 21.5 min and a constant column flow rate of 1m/min. The detection of pesticides was 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 was operated in MSN mode and performed tandem MS function 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 was compared with those of the standards for quantization.

## Determination of Heavy Metals

The instrumental methods reported by [7], were adapted for the determination of heavy metals in soil and plant samples. Determination of Mn, Cd, Pb, Zn, and Cu was made directly on each final solution using Atomic Absorption Spectrophotometer (AAS, shimadzu japan 6800). Standard solution of each sample Mn, Cd, Pb, Zn, and Cu was prepared according to Sc 2000 manufacturer procedure for Atomic Absorption Spectroscopy was used. A known 1000mg/l concentration of the metal solution was prepared from their salts. Cathode lamps for each element was used as radiation source. Air acetylene gas was used for all the experiments. This method provides both sensitivity and selectivity since other elements in the sample were not generally absorb at the chosen wavelength and thus, were not interfere with the measurement.

## RESULTS AND DISCUSSION

Concentrations of OCPs in soil from two different farms (A and B) from Billiri, Kaltingo and Akko. Compounds identified included Delta Lindane, Alpha Lindanes, Gamma Lindane, Heptachlor, Aldrin, Heptachlor epoxide, Endosulfan I., P,P' – DDE, Endrin, Endosulfan II, P,P' – DDD, P,P' – DDT and Methoxyclor. Figure.1 shows the observed pesticides concentration occurrences in Farm A and B from Billiri, Kaltungo and Akko.

## Presence of Lindane compounds

The concentration of lindane (Delta, alpha and gamma lindanes) was detected in all the samples. The highest concentration of Delta Lindane was found in farm A from Billiri, coinciding with the highest concentration in soil (16.02 ppm); this is therefore the most polluted location in the study area. High concentrations were also found at farm B Billiri (9.03 ppm), and farm A (7.03 ppm) and farm B (9.02 ppm) in Kaltungo. The highest concentration of Alpha Lindane was found in farm A (18.04 ppm) from Billirin while the lowest was found in farm B (3.06 ppm) from Kaltungo. The highest concentration of Gamma Lindane was found in farm A (15.05 ppm) from Billiri while the lowest was found in farm B (2.07 ppm) from Kaltungo. The presence of lindane in the soil samples may suggest the historical use or illegal use of technical HCH mixtures in the study area [14]. Lindane was associated with risk of glioma [15]. The mean lindane value recorded in this study was higher than those reported in soil samples from different locations Numan LGA of Adamawa State [16] and from coco farms in Ghana ]14].

## Heptachlor and Heptachlor epoxide

Heptachlor and Heptachlor epoxide were found in all the soil samples analysed, with concentrations ranging from 4.00 to 13.02 ppm and 5.05 – 24.01 ppm respectively. The highest level of Heptachlor (13.02 ppm) was found at farm A in Billiri and the lowest level (4.00 ppm) was found at farm B in Kaltungo. The highest level of Heptachlor epoxide (24.01 ppm) was found at farm A in Billiri and the lowest level (5.05 ppm) was found at farm A in Kaltungo. The ubiquitous distribution of OCPs in soil suggested an extensive use of these pesticides in the region [17]. The measured concentrations of Heptachlor and Heptachlor epoxide in the studied soils confirmed the previous use or current use of lindane in the study area [14].

**Aldrin and Endrin**

Aldrin and endrin residues were found in soil samples, indicating their ongoing presence in the study area. Soil samples from farm A in Billiri contained the highest aldrin levels (224.93 ppm), followed by farm B in Biliri (40.14 ppm), farm A in Kaltungo (20.14 ppm) and farm B in Kaltungo (14.11 ppm). Endrin concentrations were highest in soil from farm A in Kaltungo (20.03 ppm), followed by farm B in Kaltungo (18.07), farm B in Billiri (18.03 ppm) and farm A in Biliri (9.05 ppm). Aldrin and endrin, known for their persistence and potential health risks, highlight the importance of sustainable pesticide management practices. The elevated levels detected underscore the need for effective strategies to mitigate their presence and protect agricultural ecosystems and human health [18]. Aldrin use was associated with a non-statistically significant elevated risk for uterine cancer based on four exposed cases. Only one case-control study has examined OC exposures and endometrial cancer [15].

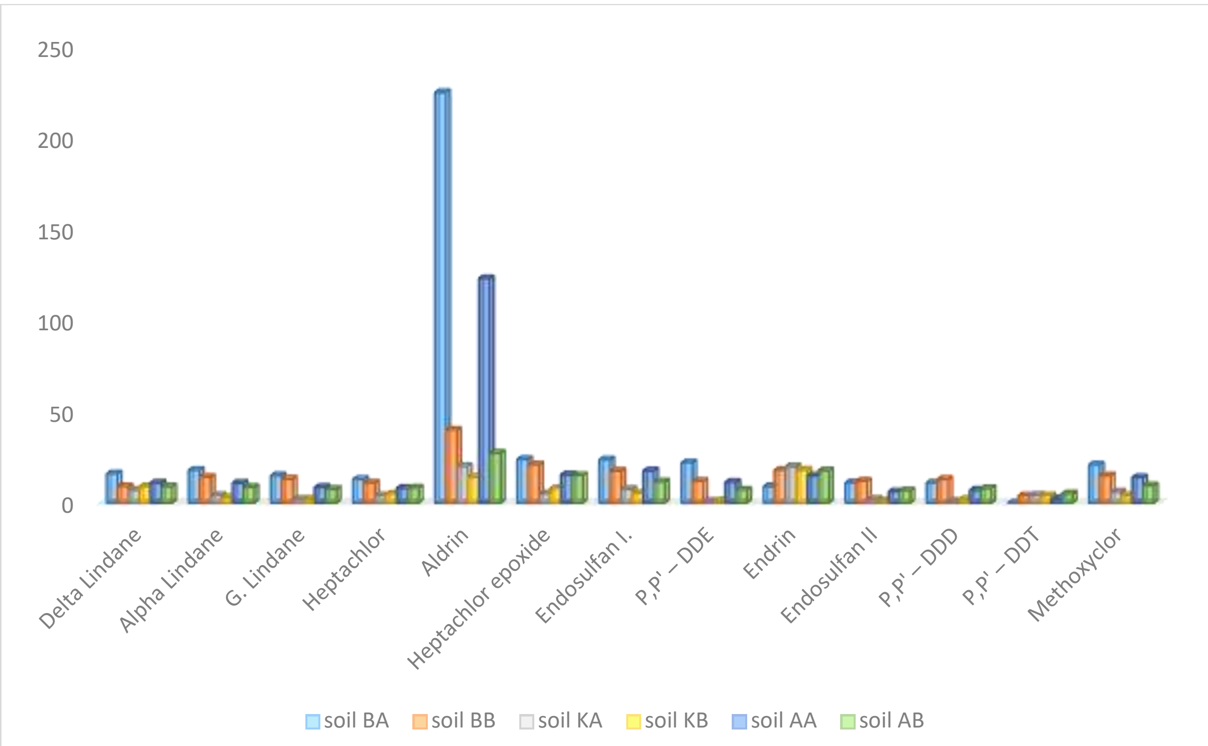



**Figure: 1: Mean Concentration of organochlorine pesticide residues in soil from farm A and B of Billiri, Kaltungo and Akko LGA of Gombe State**

**DDT**

p,p'-DDE and p,p'-DDD were detected in all the soil samples. p,p'-DDT were detected in all soil samples but farm A from Billiri. The contribution of individual metabolites showed differences. The concentration of total p,p'-DDE reached maximum value at farm A (22.14 ppm) while the lowest value was found at (1.02 ppm) from farm A Kaltungo, farm A (17.01 ppm) and farm B (14.21 ppm). Similarly, the concentration of p,p'-DDD reached maximum value at farm B from Billiri (13.01 ppm) while the lowest was recorded at farm A from Kaltungo (1.01 ppm). The minimum value of p,p'-DDT was recorded at farm A from Billiri (0.00 ppm), whereas the other three farms followed almost an equal trend of DDT distribution of 4.02 ppm (farm B from Billiri), 4.22 ppm (farm A from Kaltungo), and 4.00 ppm (farm B from Kaltungo). Among the metabolites of DDT, *p,p′*-DDE was found to be much more significant than other metabolites *p,p′*-DDE and *p,p*-DDT. The occurrence of DDT isomer is predominant in the following order: *p,p′*-DDE > *p,p′*- DDD > *p,p′*-DDT. This implies that DDT is likely to be dechlorinated to DDE in the anaerobic condition in deep layer soil. These concentrations raise concerns about potential bioaccumulation and human exposure through dietary consumption. The persistence of DDT residues is attributed to historical usage and the compound's environmental stability [17]. The findings emphasize the need for remediation strategies to reduce soil contamination and minimize further accumulation in the food chain. The concentrations of DDT reported in this study were higher than the mean values of reported in soils from Numan LGA in Adamawa State, Nigeria [16].

**Entozoan I and II**

Endosulfan I residues were identified in all soil samples, with varying concentrations. Soil samples from farm A in Billiri exhibited the highest endosulfan concentration (23.78 ppm), followed by farm B in Billiri (17.56 ppm), farm A in Kaltungo (7.56 ppm) and farm B in Kaltungo (5.3 ppm). Similarly, endosulfan II concentrations were observed in all the soil samples. Soil sample from farm B in Billiri showed the highest accumulation (12.03 ppm), followed by farm A in Billiri (11.12 ppm), farm A in Kaltungo (2.03 ppm) and farm B in Kaltungo (1.17 ppm). These levels surpassed the recommended soil limit for endosulfan use [16]. The presence of endosulfan residues raises concerns about its potential impact on soil quality, plant growth, and potential exposure through the food chain. The findings underscore the need for stringent regulatory measures and the adoption of alternative pest management practices [19].

**Methoxychlor**

Methoxychlor residues were identified in all the soil samples, indicating its widespread occurrence in the study area. Soil samples from farm A in Billiri exhibited the highest methoxychlor concentration (21.02 ppm), followed by farm B in Biliri (15.00 ppm), farm A in Kaltungo (6.00 ppm) and farm B in Kaltungo (4.03 ppm). The presence of methoxychlor residues raises concerns about potential ecological and health impacts. Methoxychlor, although considered a less persistent alternative to some other organochlorine pesticides, can still pose risks due to its toxic properties and potential for bioaccumulation [17]. Figure 2. Shows the concentrations of heavy metal (Mn, Cd, Pb, Zn, and Cu) in the soil samples from the different farm locations. The soil samples from farm A in Billiri LGA (SOIL BA) determined the highest concentration in Mn (0.171 ppm), followed by Zn (0.085 ppm), Cd (0.083 ppm), Cu (0.082 ppm) and Pb (0.06 ppm). The soil samples from farm B in Billiri LGA (SOIL BB) recorded the highest concentration in Cu (0.164 ppm) followed by Zn (0.149 ppm), Mn (0.121 ppm), Cd (0.085) and Pb (0.07 ppm). The soil samples from farm A in Kaltungo LGA (SOIL KA) detected the highest concentration in Zn (0.141 ppm) followed by Mn (0.081 ppm), Cu (0.051 ppm), Cd (0.050 ppm) and Pb (0.05 ppm). The soil samples from farm B in Kaltungo LGA (SOIL KB) determined the highest concentrations in Cu (0.122 ppm) followed by Zn (0.101 ppm), Mn (0.091), Cd (0.071 ppm) and Pb (0.06 ppm). The soil samples from farm A in Akko LGA (SOIL AA) detected the highest concentration in Mn (0.108 ppm), followed by Cu (0.073 ppm), Cd (0.062ppm), Pb (0.06ppm) and Zn (0.058ppm). The soil samples from farm B in Akko LGA (SOIL AB) determined the highest concentration in Cu (0.142 ppm) followed by Zn (0.123 ppm), Mn (0.108 ppm), Cd (0.091ppm) and Pb (0.05 ppm).

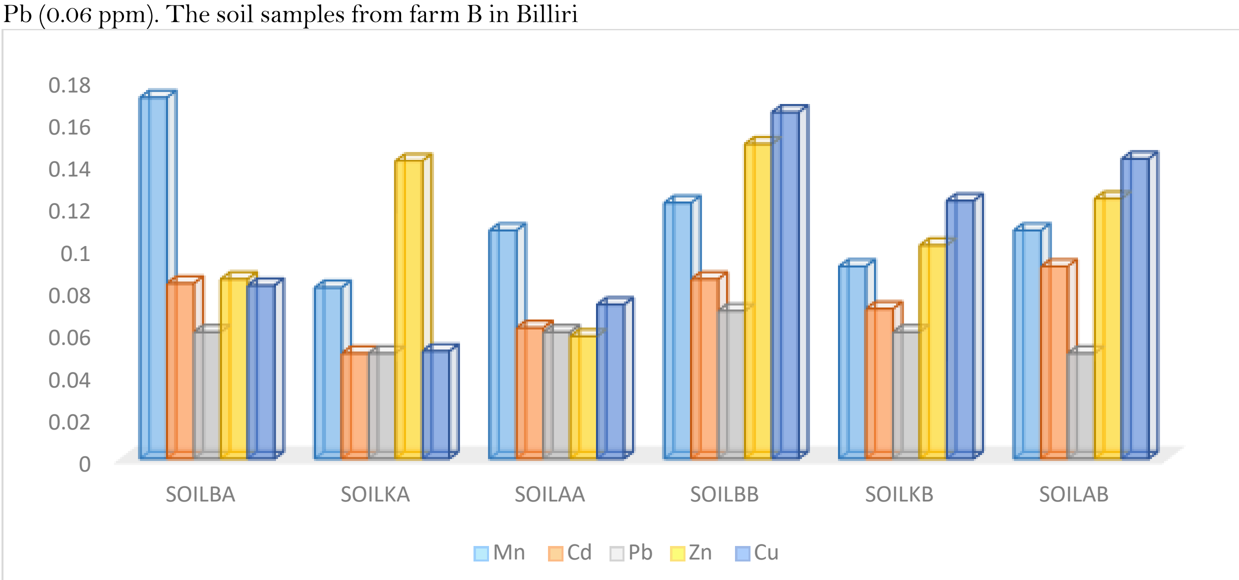


**Figure: 2 Mean Concentrations of Heavy Metals in Soil Samples from Farm A and B in Billiri, Kaltungo and Akko LGAs**

The highest concentration of heavy metals in soil samples attributed variations in metal concentrations to the duration and intensity of the anthropogenic activities associated with a particular land use type, geological characteristics of the different sites, soil type (textural characteristics), pH, sorption capacity, organic matter content, cation exchange capacity and the type of metal, with trace elements which originate from parent rock demonstrate lower mobility than same elements imported to the soil from anthropogenic sources [20]. Studies have shown that high concentrations of heavy metals in soil have strong toxic effects on plants which in turn result in weak growth, yield depression, disorders in plant metabolism and reduced nutrient uptake [5] and [21]. The figure clearly depicts the variations in the heavy metal concentration level between the soil samples from the different farm locations. The higher concentrations of Mn, Cd, Pb, Zn, and Cu in the soil samples from farm A and B in Billiri LGA compared to the Kaltungo and Akko LGA samples are evident from the figure. This shows that the soil samples from the different farm locations exhibit varying levels of heavy metal concentrations. This could be influenced by factors such as the inherent soil composition, agricultural practices, and environmental factors in the respective areas. Which is in line with the work of [20].

## CONCLUSION

Based on this study soil samples from farm A and B of Billiri, Kaltungo and Akko LGA reveals high levels of some heavy metals. Elements such as Mn, Cd, Pb, Zn, and Cu levels indicate the bioaccumulative properties of these elements. The result of the Levels of pesticide residue in soil samples from farm A and B of Billiri, Kaltungo and Akko LGA shows that pesticide residue in soil samples from different subject accumulate differently based on exposure. The lowest organochlorin pesticide residue of soil sample showed that the soil was not much exposed to the pesticide residue, or low agricultural practices [17]. However, the high level of organochlorin in soil may be attributed to the misused of pesticide during application. [22] and [23]. There is a widespread repeated exposure of the population to the pesticides and heavy metals of soil and environmental origin. Such population is forced to undergo continuous stress imposed by combined exposure of the heavy metals and different classes of the pesticides used in agricultural Practice. There is instantaneous need for public awareness about the hazards of this pesticide and heavy metal and take necessary precautionary measures.

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