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Assessment of Heavy Metals and Pesticide Residues in Soils in Adamawa State, Nigeria

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Abstract

Pesticides and heavy metals are persistent, non-biodegradable contaminants capable of bioaccumulating through ecological food chains such as soil–plant–food and seawater–marine organism–food pathways. Consequently, accurate monitoring of their concentrations is critical for ensuring environmental and public health safety. This study assessed and monitored the concentrations of heavy metals and pesticide residues in soils from various agricultural sites in Adamawa State, Nigeria. Soil samples were collected from Waja, Shashau, Munga, and Garaha Larh in Hong Local Government Area. Heavy metals were analyzed using Atomic Absorption Spectrophotometry (AAS), while pesticide residues—specifically organochlorine and organophosphate compounds—were identified and quantified using Gas Chromatography-Mass Spectrometry (GC-MS). Organochlorine pesticides detected in the soil included Lindane (2.136 mg/kg), Aldrin (1.022 mg/kg), Heptachlor (0.358 mg/kg), and Dieldrin (0.167 mg/kg), all of which exceeded the Codex Maximum Residue Limits (MRLs). Organophosphate pesticides such as Diazinon (6.133 mg/kg), Dichlorvos (0.848 mg/kg), and Chlorpyrifos (0.215 mg/kg) were also detected, with Diazinon and Dichlorvos exceeding MRLs. Heavy metals identified in soil

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included Copper (0.630–16.77 mg/kg), Zinc (12.183–246 mg/kg), Lead (3.648–9.056 mg/kg), Cadmium (0.563–3.842 mg/kg), and Chromium (1.784–5.378 mg/kg). In several locations, Cadmium concentrations exceeded permissible limits, indicating localized contamination. The high levels of Lindane and other contaminants are likely due to excessive use of agrochemicals and potential pollution of irrigation water. The findings underscore the urgent need for regulated agrochemical usage, improved irrigation practices, and ongoing environmental monitoring to mitigate health and ecological risks.

Keywords: Organochlorine; Organophosphate; Soil; Pesticide Residues; Heavy Metals

INTRODUCTION

The soil is a very essential component for all the living organisms. Especially for plants, it's considered as the basic living factor. Soil serves as a nutrient media for the growth of plants (Mitra *et al.,* 2022). The soil is not essential for agriculture production but also towards maintained all life form. The quality of water and air is of immediate concern for most people because we all consume these natural resources on a daily basis. The importance of soil, the generally thin layer of unconsolidated material on bedrock, is more difficult to grasp for an average citizen or politician. Nonetheless, the soil is the "the biogeochemical engine of Earth's life support system". It provides us with food, fodder, fiber, and fuel. In addition to these readily ratable agriculture and forestry goods, soils deliver ecosystem services that cannot be easily traded in markets. These life-supporting functions include, for example, recycling of carbon and essential nutrients of all living materials, filtering, and storage of water, regulation of the atmosphere and biological control of pests. Heavy metal appears to include all metals of the periodic table with atomic numbers greater than 20, generally excluding the alkali metal and the alkali earth. Heavy metals are metallic, naturally occurring compounds that have a very high density greater than $5g/cm^3$; compared to other metals at least five times the density of water.

Heavy metal contamination refers to the excessive deposition of toxic heavy metals in the soil caused by human activities. Heavy metals in the soil include some significant metals of biological toxicity, such as mercury (Hg), cadmium (Cd), lead (Pb), chromium (Cr) and arsenic (As), etc. They also include other heavy metals of certain biological

toxicity, such as zinc (Zn), copper (Cu), nickel (Ni), stannmu (Sn), vanadium (V), and so on. In recent years, with the development of the global economy, both type and content of heavy metals in the soil caused by human activities have gradually increased, resulting in the deterioration of the environment (Shen *et al.,* 2022). Heavy metals are highly hazardous to the environment and organisms. It can be enriched through the food chain. Once the soil suffers from heavy metal contamination, it is difficult to be remediated. In the past, soil contamination was not considered as important as air and water pollution, because soil contamination was often with wide range and was more difficult to be controlled and governed than air and water pollution. However, in recent years the soil contamination in developed countries becomes to be serious.

Pesticides

Pesticides are used in agriculture to protect crops against insects, fungi, weeds and other pests as well as to protect public health in controlling the vectors of tropical diseases like mosquitoes. They can also be used to prevent, destroy, repel or mitigate any pest and can either kill pests or render them ineffective (Rosas-Sánchez *et al*., 2022). Pesticides are used on fruits, vegetables, wheat, rice, olives, canola pressed into oil and on non-food crops such as cotton, grass and flowers. Pesticides applied to food crops in the field can leave potentially harmful residues (Sreekumar and Prathapan, 2021). According to Ahmadi *et al.,* (2024) after pesticides are applied to the crops, they may interact with the plant surfaces, be exposed to the environmental factors such as wind and sun and may be washed of during rainfall. The pesticide may be absorbed by the plant surface (waxy cuticle and root surfaces) and enter the plant transport system (systemic) or stay on the surface of the plant (contact).

The pesticides that get into the plant tissues may be transformed (metabolised) or sequestered in the tissues to form the pesticide residue. Pesticide residues are the deposits of pesticide active ingredient, its metabolites or breakdown products present in some component of the environment after its application, spillage or dumping (Wondimu and Geletu, 2023). The presence of pesticide residues is a concern for consumers because pesticides are known to have potential harmful effects to other non-targeted organisms than pests and diseases (Aznar-Alemany *et al*., 2021). Infants, children and adults are commonly exposed to pesticides by eating them on and in our food (Rose *et al.,* 2020). Consumption of conventionally grown fruits and vegetables is a major source of non-

occupational pesticide exposure (Wondimu and Geletu, 2023). Vegetables consumption is usually encouraged because according to Aznar-Alemany *et al.*, (2021) they are essential for a healthy and balanced diet, as well as adding variety, interest and flavor to the menu.

METHODS

Study Area

Hong Local Government, lies between longitudes$10^0$1354N and latitude $12^0$5549E. Adamawa is a state in the Northeast Nigeria, with its capital in Yola. Hong is a local government which fall under Adamawa Central Senatorial District with an area of about 2,626 Km^2 and with also the population of about 226,100 (NPC, 2016). The study area will cover four villages in Hong namely (Waja, Shashau, Muga, and Garaha Larh).

Sampling and Sample Collection

A total of twenty samples was collected form four villages, which include four soil samples the soil sample was collected at four cardinal points on each farm land at the depth of 0-20 cm using the soil auger. The soil was dried in the laboratory and extraneous materials was removed and it was sieved through 2 mm mesh sieve and the sample was stored in polythene bag clearly label prior to analysis (den Braver-Sewradj *et al.,* 2021).

Digestion of Sample for Heavy Metal Analysis

1g sample was weighed using a weighing balance and placed in 250 ml beakers separately and 15 ml of aqua regia (HCl and H_2SO_4 in 3:1 ratio) was added. The mixture was digested at 70°C till the solution became transparent. The resulting solution was filtered through filter paper and transfer into a sample bottle and diluted to the mark with distilled water. The sample solution was analyzed for concentrations of Cr, Zn, Cd, Cu and Pb using an atomic absorption spectrophotometer (Sreekumar and Prathapan, 2021).

Extraction of Pesticide

A mixture of 25 g sample and 50g granular sodium sulphate was ground into a powdery consistency using a mortar and pestle. The ground sample was extracted with 150 cm^3 of a mixture of n-Hexane and acetone (1:2). The extract was transferred into a round bottomed flask and concentrated to about 20 cm^3 on a water bath maintained at 50°C - 55°C. The remaining solvent in the concentrated extract was evaporated using a rotary evaporator to about 5 cm^3. The concentrated extract was quantitatively transferred to a centrifuge tube,

concentrated on a nitrogen evaporator to 0.5 cm^3 and diluted to 2 cm^3 in hexane prior to GC-MS analysis. (Wondimu and Geletu, 2023).

RESULTS AND DISCUSSION

The results for analysis of heavy metals in soil

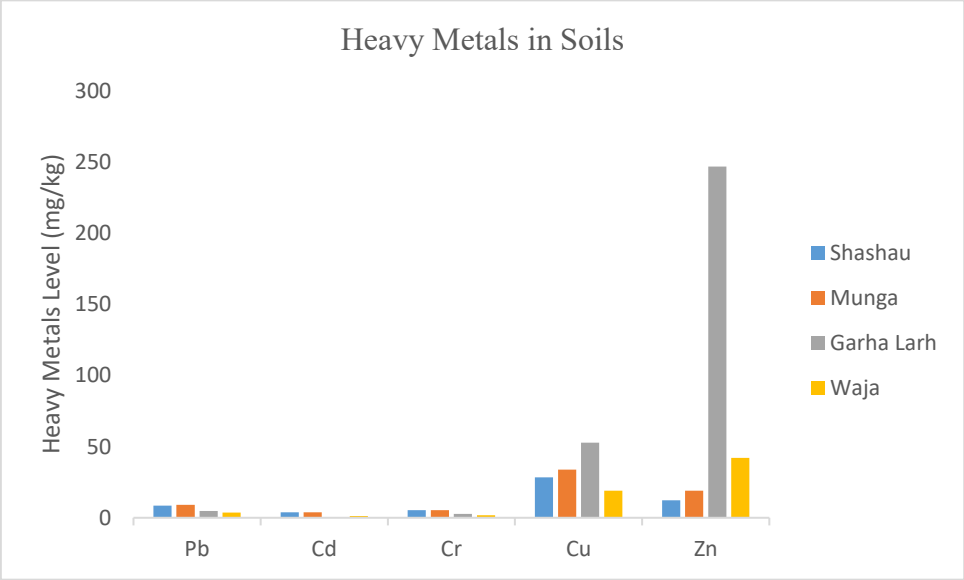


Fig 1. Heavy metals concentration of cereal grains and soil from Waja

Discussion on heavy metals

Heavy metals in soil at Shashau

Fig. 1 Has shown that copper (Cu) has the highest concentration over all the metals analyze soil, the concentration of copper (Cu) was found to be 28.448 mg/kg. The concentration of heavy metals in soil varies Cu>Zn>Pb>Cr>Cd. Pb 3.831 mg/kg was found to be above maximum residue limit while other element detected in the soil samples were found to be within the permissible limit.

Heavy metals in soil at Munga

Fig. 1 Has shown that copper (Cu) was found to be in higher concentration over all the metals analyze in soil sample in Munga. The concentration of copper was found to be 33.877 mg/kg. The concentration of the metals varies Cu>Zn>Pb>Cr>Cd respectively.

The concentration of all these heavy metals in soil falls within the safe limit with exception of lead 3.842 mg/kg which was found to slightly above the safe limit.

Heavy metals in soil at Garaha Larh

Fig. 1 Revealed that Zinc (Zn) was found to be in higher concentration over all the metals analyze in soil samples at Garaha Larh. The concentration of Zinc was found to be 246.531 mg/kg. the concentration of the heavy metals varies Zn>Cu>Pb>Cr>Cd respectively. All the heavy metals found were within the permissible limit.

Heavy metals in soil at Waja

Fig. 1 Has shown that zinc (Zn) was found to be in high concentration over all the heavy metals analyze in soil sample at Waja. The concentration of zinc was found to be 42.121 mg/kg. All the heavy metals were found in the soil samples and the concentration varies Zn>Cu>Pb>Cr>Cd. The concentration of these heavy metals was found to be within the safe limit.

Heavy metals in soil

The Mean concentration of copper (Cu), zinc (Zn), and lead (Pb) were found to be within the permissible limit (fig. 1) While (Cd) at Shashau and Munga were found to be above the maximum residue limit and the concentration at Garaha Larh and Waja were within the maximum residue limit. The excessive concentrations of heavy metals in soil reduce microbial activities and thus lower down the process of recycling of important nutrients, control of pests and maintenance of soil structure (Ikechukwu *et al.,* 2021). The result show that the availability of heavy metals is in the order Zn>Cu>Pb>Cd>Cr. Zinc and copper were found to be in high concentration which indicate the presence of essential nutrient in the soil. The highest concentration of zinc 30.299 mg/kg in soil was at Garaha Larh while the lower concentration was at Shashau 1.382 mg/kg, the high concentration of copper was recorded at Garaha Larh 52.684 mg/kg and low concentration was at Waja 19.108 mg/kg, high concentration of cadmium was found at Munga 3.842 mg/kg and the low concentration was at Garaha Larh 0.563 mg/kg, chromium was high at Munga 5.378 mg/kg and low concentration at Waja 1.784 mg/kg and high concentration of Lead was recorded at Shashau 8.462 mg/kg and low concentration was at Waja 3.648 mg/kg respectively.

Result for analysis of organochlorine pesticide in soil samples.

Table 1 Concentration (mg/kg) of organochlorine pesticide residues in soil

S/N	PESTICIDES	SOIL Waja	RT (Mins)	SOIL Shashau	RT (Mins)	SOIL Garaha Larh	RT (Mins)	SOIL Munga	RT (Mins)	MRL (mg/kg) EU/WHO
1	Lindane	0.312	11.047	0.712	11.043	0.841	11.060	2.136	11.024	0.04
2	p,p'-DDT	ND	16.936	ND	16.936	ND	16.936	ND	16.936	0.10
3	Aldrin	ND	18.145	ND	18.145	ND	18.145	1.022	18.062	0.05
4	Heptachlor	ND	18.637	ND	18.637	ND	18.637	0.358	18.645	0.03
5	Dieldrin	0.015	20.219	0.167	20.336	ND	20.754	ND	20.754	0.05
6	Endrin	ND	20.754	0.001	20.766	ND	20.754	ND	20.754	0.05
7	Endosulfan	ND	22.287	ND	22.287	ND	22.287	ND	22.287	0.20

Result for analysis of organophosphate in soil samples.

Table 2 Concentration (mg/kg) of organophosphate pesticide residues in soil

S/N	PESTICIDES	SOIL (SW)	RT (Mins)	SOIL (SS)	RT (Mins)	SOIL (SG)	RT (Mins)	SOIL (SM)	RT (Mins)	MRL (mg/kg) EU
1.	Dichlorvos	ND	9.367	1.243	9.292	0.848	9.307	11.995	9.233	0.20
2	Chlorpyrifos	ND	14.242	0.015	14.362	ND	14.242	0.215	14.440	0.30
3	Diazinon	0.050	12.941	0.314	12.874	ND	12.909	6.133	12.946	0.40

Discussion on Pesticides Residues

The overwhelming benefits of investigating the concentration of organochlorine pesticide in soil is presented in Table 1. Five of the organochlorine pesticides were detected which include Lindane, Endrin, Heptachlor, Dieldrin and Aldrin. Lindane was detected in all the soil samples from the study area which ranges from 0.312 mg/kg to 2.136 mg/kg. The high and low concentration of lindane was from Munga and Waja. The concentration of lindane in all the soil samples exceeded the maximum residue limit set by EU (2018). Adrine 1.022 mg/kg and heptachlor 0.358 mg/kg were detected in soil from Munga. The concentration of aldrin and heptachlor both exceeded the maximum residue limit, the high

concentration of these residues measured in the soil was either a reflection of the usage which resulted in bioaccumulation or probably arising from the application of the insecticides which was intense. According to Mitra *et al.,* (2022), the primary exposure of human beings to Heptachlor is through contaminated foods as Heptachlor is used primarily by farmers to kill termites, ants and soil insects in seed grains and on crops, as well as by exterminators and home owners to kill termites. The use of this organochlorine has been banned or severely restricted in several countries but these are still being produced and exported to the developing countries such as Nigeria finding the chemical to be affordable and versatile in pest control (Wondimu and Geletu, 2023). Dieldrin was also detected in soil from Waja 0.015 mg/kg, endrin 0.001 mg/kg was also detected in Shashau which were all below the maximum residue limit. (DDT) which is moderately hazardous, with high persistence and a half-life of 12-15 years (Aznar-Alemany *et al.*, 2021). The use of DDT is now banned in many countries but it is illegally use in most developing countries. This applies also to Endosulfan, an insecticide which is highly hazardous and has moderate persistence with half-life of fifty days (Oyinloye *et al.,* 2021). p'-DDT and Endosulfon were not detected in all the soil samples.

The concentration of organophosphate pesticide in soil is presented in Table 2. All the organophosphate under study was detected in soil, the concentration of dichlorvos was found to be 0.848 mg/kg at Garaha Larh, 1.243 mg/kg at Shashau, 11.995 mg/kg at Munga and was not detected at Waja, the various concentration of dichlorvos exceed the maximum residue limit. Chlorpyrifos was also found to be 0.015 mg/kg at Shausha, 0.215 mg/kg at Munga and was not detected in Waja and Garaha. The concentration of chlorpyrifos was found to be below the maximum residue limit. Diazinon was detected at Waja, Shashau and Munga and their concentration were found to be 0.050 mg/kg, 0.314 mg/kg, 6.133 mg/kg and diazinon was not detected at Garaha Larh, the concentration at Waja and Shashau were found to be below the maximum residue limit while at Munga it was found to exceed the maximum residue limit.

This study has unveiled the concentration of pesticide in soil collected at four villages in Hong Local Government, some were above the MRLS of WHO and EU, increase use of pesticide in the country's agriculture has invoked under spread concern among specialist about their potential ill effects in human health. The following challenges may result; asthma, birth defects, neurological effect, cancer, hormone disruption and death.

Summary

The study was conducted to determine pesticide and heavy metals in soil samples from four villages which include (Waja, Munga, Shashau and Garaha Larh) in Hong Local Government of Area of Adamawa State. The results showed that organochlorine and organophosphate pesticide residue was detected in most of the soil samples under study. The residues have originated from agricultural activities and use of pesticide during cultivation in the study area because of their persistent, Aldrin record high concentration in soil 1.022 mg/kg at Munga, Dieldrin was also recorded high in Guinea corn 0.279 kg/mg at Shashau, Heptachlor was high in soil 0.358 mg/kg at Munga which was above the EU MRL Other pesticide detected were in conformity with MRL. p,p'-DDT was not detected in all samples analyzed. This implied that some soil sample from the research areas were contaminated with pesticide above the MRL.

The heavy metals analysis results for soil revealed the presence of metals (Cu, Cd, Cr, Pb and Zn) in most of the samples analyses in this study. Some of the metals were within the safe limit while others were found to be above WHO maximum limit.

CONCLUSION

The results of the study revealed that organochlorine and organophosphate pesticides were present in some of the soil samples. The pesticide residue detected from Hong were Lindane, Aldrin, Heptachlor, Dieldrin, Endrin, Dichlorvos, Chlorpyrifos and Diazinon. Lindane pesticide was banned but still in used by and farmers. The results indicated most of the pesticides residues were higher or slightly higher than EU/WHO MRLS while others were lower than the EU/WHO MRLS.

The study also showed that some of the samples were not contaminated with heavy metals as analyzed while others were contaminated. The presence of some of the heavy metals in soil represent a potential hazard to the consumers. If these metals are not eliminated at the rate which they bioaccumulate, they pose serious health risk to their consumers. So, it is essential for agricultural activities and other human activities that increase heavy metal contamination of food stuffs be controlled. Therefore, the opinion of (Oyinloye *et al.,* 2021), that monitoring and systematic gathering of information on heavy metal levels in the environment are essential components of any pollution control system is well supported.

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