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Determination of the level of pesticide residues in irrigation soil from Akko, Gombe State, Nigeria

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ABSTRACT

This study determined pesticide residues levels in irrigation soil from Akko, Gombe State, Nigeria. A total of 54 soil samples were collected from three different irrigation farmlands, and the pesticide residues were extracted and analyzed using Agilent GC 7890B, coupled with MSD 5977A. The mean concentration of dichlorvos in the soil samples ranged from 0.00 - 0.089 mg·kg^{-1}, while the concentrations of dimethoate, lindane, aldrin, cypermethrin, and pyrazophos were below the detectable limit in all the studied samples. Paraquat dichloride ranged from 0.00 - 0.084 mg·kg^{-1}, while chlorpyrifos ranged from 0.00 - 0.079 mg·kg^{-1}, and p,p'-DDT ranged from 0.078 - 0.098 mg·kg^{-1}, while heptachlor concentration ranged from 0.00 - 0.086 mg·kg^{-1} in the soil samples. The levels of cyhalothrin ranged from 0.00 - 0.095 mg·kg^{-1}, dieldrin ranged from 0.00 - 0.085 mg·kg^{-1}, while that of endrin ranged from 0.00 - 0.097 mg·kg^{-1}. The concentrations of endosulfan ranged from 0.00 - 0.106 mg·kg^{-1}. This considerable level of these pesticide residues may accumulate in food crops through the uptake of soil and have a negative impact on human health. However, monitoring and continuous stringent regulations should be imposed with regard to the usage of pesticides in soil and foodstuff for public health protection.

KEYWORDS: Determination, irrigation, pesticide residues, soil.

1. INTRODUCTION

Pesticides are substances commonly used in modern agricultural practices to protect crops from various pests and diseases.[1-3] Many farmers use pesticides in order to increase yield and to protect their farms from diseases. However, after pesticide application, residues often remain in the soil and crops even after harvest, eventually entering the food chain.[4,5] Pesticides are toxic to humans both acute and chronic health effects depending on the quantity and types of exposure.[6] Conversely, the use of pesticides has significantly contributed to the increase in global food production over the past several decades.[7]

Globally, approximately 3 million tons of pesticides are used each year, representing a market value of US$40 billion.[8] In the European Union, nearly 500 substances are approved for pesticide use, with annual sales averaging 374,000 tons between 2011 and 2016.[9] Although pesticides enhance agricultural productivity and play a crucial economic role, their excessive use has raised significant environmental and public health concerns.[10] The widespread issue of diffuse agrochemical pollution has emerged as a significant threat to soil health[11], potentially undermining multiple United Nations Sustainable Development Goals related to the soil environment.[12,13]

Soil contamination poses significant threats to soil functions, biodiversity, and food safety, while also facilitating the off-site spread of pollutants through wind and water erosion. This dispersal can disrupt sink ecosystems and create additional exposure pathways for humans and non-target organisms to harmful contaminants.[13,14] Despite the significant impacts of soil contamination, EU regulations do not require monitoring pesticide residues in soil, in contrast to the mandatory water monitoring established by the Water Framework Directive.[7] There is a lack of extensive international studies on pesticide residues in soil, as existing research typically analyzes only individual pesticides or a small group of them.[7,15,16]

Several studies have characterized the occurrence of both currently used and banned pesticides in soils at national or regional levels.[17-22] Effective control of pesticide levels in food can only be achieved through systematic residue monitoring. In recent years, increased surveillance efforts have been implemented, emphasizing proper pesticide application rates and regulatory adherence.[23-25] Despite the public health risks, the usage of these pesticides is increasing, and they are used for both

agricultural and non-agricultural purposes in Nigeria.[5] Hence, this study to investigate the level of pesticide residues in irrigation soil from Akko, Gombe State, Nigeria.

2. MATERIALS AND METHODS

2.1 Study area

The Akko Local Government Area is one of the eleven local governments in Gombe State. Akko is located between the latitudes of 10.97320° E and the longitudes of 10.28899° N of the equator. The area falls within the northern Guinea savanna zone of the Akko, Gombe State. Akko covers a total area of 2,627 km² (1,014 sq mi), with approximate population of 337,853.[26] The area receives an average annual rainfall of approximately 600 mm, which is sufficient for a single farming season. The annual rainfall pattern is erratic at the beginning of the rainy season, starting in April and intensifying as the season advances, raising the average from 600 to 1000 mm. The temperature as high as 41°C and the as low as 16°C. The generally high temperatures and low humidity favor high rates of evapotranspiration, leading to net water deficiencies. Akko L.G.A. is in the interior Savanna woodland, which can sustain large-scale livestock farming, as well as the cultivation of agricultural products like onions, spinach, tomatoes, rice, groundnuts, yams, cassava, and maize. The study area was selected due to of dry farming activities.

2.2 Soil sample collection

Soil samples were collected from three farms (labelled Farm A, Farm B, and Farm C) using a nested sub-sampling approach. Each farm contains six sub-farms; from each sub-farm three soil cores were taken at a depth of 0–20 cm, yielding 18 samples per farm (3 samples × 6 sub-farms = 18). All 18 samples from each farm were composited into a single representative sample for that farm (i.e., one composite for Farm A, one for Farm B, and one for Farm C). Samples were labelled sequentially as A, B, and C corresponding to the three farms. The samples were all label and transported to laboratory for analysis.

2.3 Reagent

Reagents/Solvents: n-hexane and dichloromethane (HPLC grade). Copper powder and concentrated sulfuric acid were obtained from Loba Chemie India.

2.4 Sample Extraction

Five grams of the sample was weighed into a 50 mL centrifuge tube, and 30 mL aliquots of the extraction solvent consisting of a mixture of n-hexane/dichloromethane (1/1, v/v) were added and shaken for 15 min (using a universal shaker, IKA Werke). After 15 minutes, the sample mixture was ultrasonically extracted at 25°C for 20 min. The mixture was centrifuged at 8,000 rpm for 8 minutes, and the resulting supernatant was transferred into a separate container. The extraction process was repeated twice, after which all extracts were combined and concentrated using a rotary evaporator under mild vacuum until dryness. The residue was dissolved in 40 mL of n-hexane and analyzed using GC system: Agilent 7890A GC (Agilent Technologies)

2.5 Sample Clean up

Five grams of copper was added to the dissolved sample mixture, shaken for 5 min, and centrifuged at 8000 rpm for 5 min. The supernatant was collected, 3 mL of concentrated H_2SO_4 was added, and the mixture was vortex for 1 min.The mixture was again centrifuged at 2,000 rpm for 5 minutes, and the resulting supernatant was collected and treated with 1 mL of concentrated sulfuric acid. Vortex-mixed again for 1 min, and then centrifuged at 2000 r/min for 5 min. The supernatant solvent was dried by a rotary evaporator, and the residue was reconstituted with 1 mL n-hexane and then vortex-mixed.

2.6 Instrumental Analysis

A 1 µL aliquot of the reconstituted sample was injected into a gas chromatograph (Agilent 7890B GC coupled with a 5977A MSD) for analysis, using helium as the carrier gas. The column temperature program was set at 150 °C for 1 min, ramp at 25°C /min to 230 °C, hold for 2 min; and ramp at 20°C /min to 240 °C, hold for 1 min; ramp at 20 °C /min to 260 °C, and hold for 5 min; ramp at 3°C /min to 280 °C, and hold for 5 min; ramp at 2°C /min to 285 °C, and hold for 10 min. The injection port temperature and transfer line were set at 240 °C and 250 °C, respectively. The injection was made in the splitless mode with purge on after 0.75 min. [27]

2.7 Preparation of Calibration Standards

Working standard solutions were prepared by diluting the stock solutions to 10 µg/mL in ethyl acetate. Appropriate aliquots were taken and further diluted with ethyl acetate to give a series of calibration standard solutions with concentrations of 10, 20, 50, 75, and 100 ng/mL. Extracted calibration standards were prepared by spiking blank water samples (1 L) with the working standard solution prior to extraction to give concentrations of 10 ng/L, 20 ng/L, 10, 20, 50, 75, and 100 ng/L. After extraction and reconstitution (1 L to 1 mL), concentrations of these equate to of 0, 20, 50, 75, and 100 ng/L.[28]

2.8 Method of Data Analysis

The residues were analyzed using a Shimadzu gas chromatograph (GC-2010), equipped with 63Ni, an electron capture detector that allows the detection of contaminants even at trace concentrations. The GC conditions and detector response were adjusted to match the relative retention time and response. The capillary column was coated with ZB5 (30 m x 0.25 mm, 0.25 µm film thickness). Sample preparation, extraction, cleanup, and analysis were performed according to the procedure.[29]

3. RESULTS AND DISCUSSION

3.1 Pesticide residues Percentage in the soil samples

The percentage of pesticide residues is presented in Figure 1. The percentage level of dichlorvos in the soil samples was in the range of 27.50% and 34.85% in farms B and C, respectively, while the percentage level of paraquat dichloride in the soil was 12.90% in farm A and below the detectable limit in farms B and C. The levels of chlorpyrifos in the soils were in the order of 24.38% and 32.78% in farms B and C, whereas they were below detectable in farm A. The p,p'-DDT distribution in the studied soils was 15.05%, 24.07%, and 32.36% in farms A, B, and C, respectively. The heptachlor in the soil wass in the order of 13.21% in farm A, below detectable in farms B and C, while that of cyhalothrin had the following percentages in the same order: 14.59%, and dieldrin sulphate distribution in the studied soils was 13.05% and 24.07% in farms A and B, respectively. The percentage level of endrin in the soils was 14.90% in farm A and below the detectable limit in farms B and C, whereas that of endosulfan was 16.28% in similar orders.

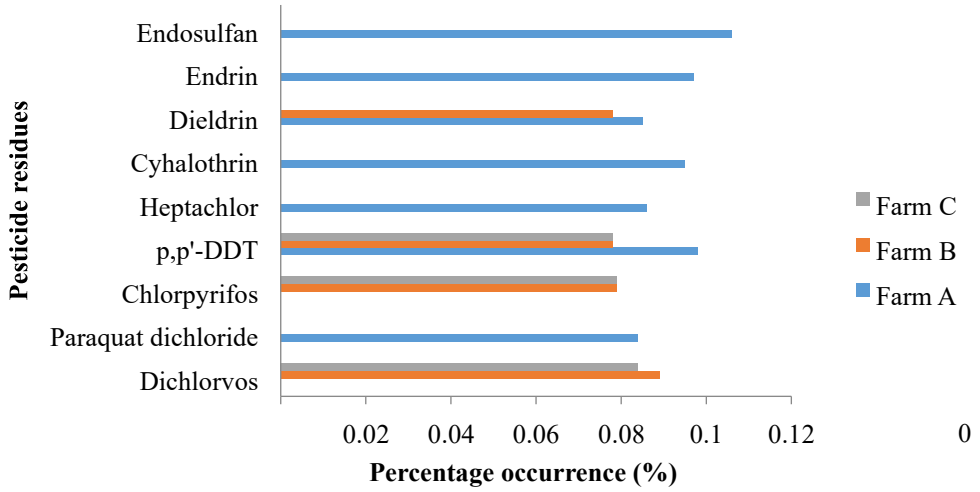

Figure 1: Percentage (%) of pesticide residues in the soil samples

3.2 Levels of pesticide residues in soil samples

The pesticide levels in the soil samples are presented in Table 1. The mean concentration of dichlorvos in the soil samples ranged from 0.00 to 0.089 mg·kg^{-1}, and the concentrations of dimethoate, lindane, aldrin, cypermethrin, and pyrazophos in the samples were below the detectable limit. Paraquat dichloride concentration was 0.084 mg kg^{-1} in farm A and was below the detectable limit in farms B and C. Chlorpyrifos concentration in the samples ranged from 0.00 to 0.079 mg·kg^{-1}, and concentrations of p,p'-DDT in the samples ranged from 0.078 to 0.098 mg·kg^{-1}, while heptachlor concentration ranged from 0.00 to 0.086 mg·kg^{-1} in the samples. The levels of cyhalothrin in the samples ranged from 0.00 to 0.095 mg·kg^{-1}, the dieldrin ranged from 0.00 to 0.085 mg·kg^{-1} in the sampled soils, while that of endrin ranged from 0.00 to 0.097 mg·kg^{-1}. The endosulfan concentrations in the samples ranged from 0.00 to 0.106 mg·kg^{-1}. Han et al. also reported that the content of chlorpyrifos, 7.2 - 77.2 µg/kg, was below the value of 0.079 mg kg^{-1} obtained in this study[31]. The level of p,p'-DDT obtained in this study was above (4.01 µg/kg) that reported in soils from the farms of Kumasi, Ghana.[30] and 34.0 reported from the soils of China.[31]

The level of cyhalothrin was higher than that reported by Han et al.[31] but lower than that the cypermethrin reported by Han et al.[31]. Vincent et al. reported the detection of aldrin in soil 1.20 µg/kg, which is higher than the values obtained in this study.[30] In a Thailand project of food webs in rice paddies, a high level of aldrin was also detected in rice soils (28.6 µg/kg), [32] while dieldrin values were reported as 1.5-3.9 µg/kg by Han et al., which were below the values of 0.078-0.085 mg·kg^{-1} in this study.[31] Moreover, this study showed that the levels of pesticide residues in the studied soils were higher than those in early reported studies conducted in other parts of the world.[22-34] This could be due to unawareness and misuse of the use of OCP pesticides in Nigeria.[7]

Table 1: Levels of pesticide residues in soil (mg kg^{-1}) samples

S/No		Farm A	Farm B	Farm C
1	Dichlorvos	BDL	0.089±0.105	0.084±0.098
2	Dimethoate	BDL	BDL	BDL
3	Paraquat dichloride	0.084±0.105	BDL	BDL
4	Lindane	BDL	BDL	BDL
5	Chlorpyrifos	BDL	0.079±0.078	0.079±0.088
6	p,p'-DDT	0.098±0.089	0.078±0.098	0.078±0.100
7	Aldrin	BDL	BDL	BDL
8	Heptachlor	0.086±0.099	BDL	BDL
9	Cyhalothrin	0.095±0110	BDL	BDL
10	Cypermethrin	BDL	BDL	BDL
11	Pyrazophos	BDL	BDL	BDL
12	Dieldrin	0.085±0.110	0.078±0.101	BDL
13	Endrin	0.097±0.974	BDL	BDL
14	Endosulfan	0.106±0.0970	BDL	BDL

BDL= below detectable limit

4. CONCLUSION

The present study was conducted to determine pesticide residues levels in the irrigation soil of Kunnuwal, Akko Local Government Area Gombe State, Nigeria. Generally, the pesticide residue concentrations in the various soils have been recorded at a reasonable concentration. Hence, the need for regular monitoring of pesticide residues should be encouraged, since an increase in misuse of

pesticides for agricultural produce is still ongoing in Nigeria. Based on the findings of this study, the following recommendations for further action and studies are suggested: the present work critically focused on the pesticide residue assessment of irrigation soil in the study area; the results, therefore, suggest the need for further studies on agricultural produce to help in understanding the chemical form and behavior of the pesticide residues; there is a need to develop an environmental monitoring and management program for pesticide residues in Nigeria.

CONFLICT OF INTERESTS

The authors declare no conflict of interests.

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