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Assessment of Physicochemical parameters and Organochlorine pesticide residues in selected vegetable farmlands soil in Zamfara State, Nigeria

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ABSTRACT

In this study, physicochemical and organochlorine pesticide residues in soil were determined from selected vegetable farmland in Zamfara State, Nigeria. The QuEChERS, and GC-MS were used for sample preparation and organochlorine analysis, respectively. The results of physicochemical analysis obtained showed that the soil from farmland A has sand, clay, slit, pH, organic matter and electrical conductivity content of 28.80±0.164 %, 51.0±0.78% 17.18±10.17%,6.45±0.082, 3.15±0.0098% and 45.00±26.46 (µS/cm), respectively,. The soil from farmland B recorded sand, clay, slit pH organic matter and electrical conductivity content of 50.09±0.001%,25.70±0.755%, 19.01±0.861%, 6.39±0.156%, and 96.33± 75.16µs/cm, respectively while 32.30±0.059%, 56.35±0.399%, 13.6±0.56%, 6.29±0.734, 5.44±0.592% and 200.33±148.30 µs/cm for sand, clay, slit, pH, organic matter, and electrical conductivity respectively were values of parameters from the analysis of soil from farmland C. Seven organochlorine pesticide residues were found in farmland A, namely; alpha lindane, detalindane, endosulfan I, isodrine, DDMU, dieldrine, endosulfan II, with concentration of 0.62 mg/kg, 0.016 mg/kg, 0.069 mg/kg, 0.004 mg/kg, 0.003 mg/kg, and 0.011 mg/kg respectively. Endosulfan I and dieldrin are foundin farmland B with concentration of 0.124 mg/kg and 0.023 mg/kg while six organochlorine pesticide residues were determined in farmland C this include alpha lindane, detalindane, isodrine, heptachlor II, DDMU, and dieldrine with concentration of 0.05 mg/kg, 0.054 mg/kg, 0.028 mg/kg, 0.006 mg/kg, 0.003 mg/kg and 0.006 mg/kg, respectively. The amounts of dieldrin and alpha lindane were above the maximum residual limit accepted in the environment. The presence of the banned organochlorine pesticide residues in soil samples indicates that these chemicals are still in use illegally on vegetable farm land for pest control. Regular monitoring of pesticide residues in the study area is necessary for the prevention, control, and reduction of environmental pollution.

Keywords: Physicochemical, organochlorine, pesticide residues, soil, GC-MS

INTRODUCTION

The use of pesticide to improve the agricultural good yield by vegetable farmers has brought a great relief and availability of various vegetables to the consumers, because the pesticides including organochlorine are use to combat the pest that destroyed vegetables.[1]

However, the incidence of residual deposition of this organochlorine pesticides and long time distance movement of these chemicals to non target area, such as water, air, atmospheric pollution and environment in general has generated a serious concern because of its adverse health effects. [2]

Soil is the repository of chemicals including organochlorine pesticide which have high affinity for soil and may be taken up by crops and eat by grazing animals and hence back to food chain. Soil is considered to be an important agricultural resource which has ability to retained agrochemicals including organochlorine pesticides. [3]

It is known that organochlorine pesticide persist in the soil because they are very slow in degradation process, volatilization, and strong affinity to the soil organic matter.The affinity of organochlorine pesticides to the soil organic matter and other soil physiochemical properties does not prevent plants of taken them up the residues. Consequently this has led to the migration of residues of organochlorine pesticides into growing plant, which in turn gains entrance to food chain. [4]

Organochlorine pesticides can be found in environmental samples such as water, agricultural products and soil. It is widely known that organochlorine pesticides still contaminated in soil because they do not decompose due to their physical properties. The stability of organochlorine pesticides makes it possible to exist in the soil for long period of time which sorption by plants or washed by rainfall from point of used to non target area. This called for global concern, thereby ban by government. [5]

Some of the ban organochlorine pesticides are DDT, lindane, endusulfan, deildrin, heptachlor, HCB and HCH because it resist degradation by chemical means, microbiological and their half life are range from months to years. [6]

Organochlorine pesticide residues are toxic to both human and animals with serious short and long time effects such as immune system and reproductive damage, mutation and carcinogenic. Despite the ban and restriction of these pesticides for agricultural and household uses, their presents are been identified in fruits and vegetables by various researchers in Nigeria. [7, 8]

For this reasons, this study was conducted to assess the level of organochlorine pesticide residues and physiochemical analysis of vegetable farmland soil samples obtained from Sunami, along Wanke in GusauZamfara State. The area was selected because of their agricultural contribution of vegetables such as tomatoes, different verities of pepper, onion, and vegetables leave. Most of these vegetable are always under threat of pest and diseases, therefore, farmers may result to the use of these potential toxic compound to combat the pest and diseases, and the deposition of the persistence residues can bio accumulate and transcend across the tropic levels.

MATERIALS AND METHODS

Study Area

Samples of soil were collected from three major vegetable farm lands in Sunamialongwanke Gusau, Zamfara State. The three farmlands were in UnguwaMallamBawa, Gidan Halilu and Sarkir Noma as shows in figure 1, along Wake road in central region of Gusau in Zamfara.

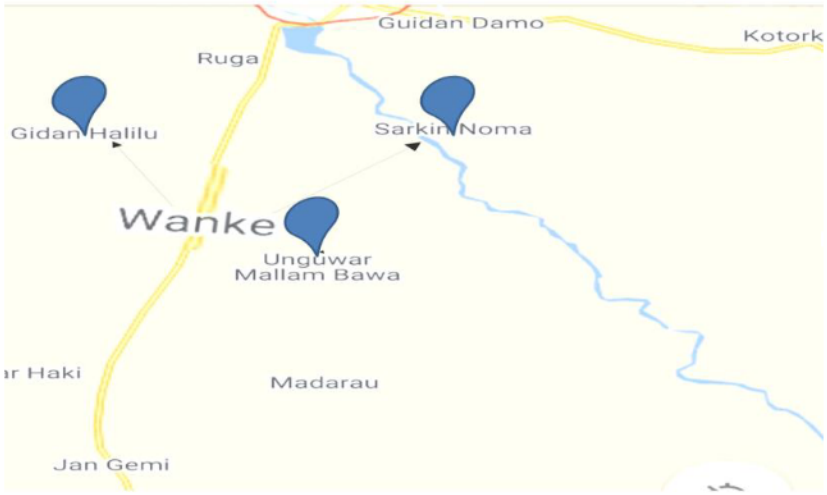

Figure 1. Map of sample collection Area

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Sample Collection treatment

Three vegetable farmlands were selected for samples collection, in each farmland two quadrate of 60 × 10 cm were marked and six soil samples were collected randomly with the aid of soil sample collector (Auger) to a depth of about 20 cm. The samples were composited. Total of 30 samples were collected from each farmland, these were placed in a cleaned well labeled polythene bags and transport to the laboratory for analysis. The samples were dried in the laboratory at ambient temperature. The coarse particles were removed and the samples were sieve and stored in polyethylene bags for further analysis.

Physicochemical Properties

Physicochemical analyses were carried out to determined the soil pH, electric conductivity (EC), soil organic matter (SOM), soil texture (such as sand, slit, and clay contents) following the methods described in the literature. [2-6]

Soil pH

10.0g of each soil samples and 50cm^3 of distilled water were added to each. The mixtures were shaken and stirred with a glass rod for about three hours and allowed to stand overnight. The mixtures were then filtered into another clean beaker, the pH meter was then calibrated (with buffer solutions pH x, pH y and pH z) and the electrode immersed in the solution and the readings were recorded. The electrode was rinsed after each measurement.

Electrical conductivity (EC)

To 10.0 g of the sample, 50cm^3 of distilled water was added. The solution was then put on shaker for 1 hr and xx rpm. The solution was then decanted into a beaker. The calibrated conductivity U dip the entire meter in the sample? meter was then submerged into the sample and reading were taken after the meter stabilizes.[4]

Percentage Sand, Clay and Silt

The soil was air dried and sieved to remove coarse particles. 50g of dried sieved soil samples were weighed and 100 cm^3 of 25% sodium hexametaphosphate was added and shaken for 16 hours with an electric shaker. The mixture was then placed in a bouyance blender cup and stirred for 2 minutes. The content of the cup was placed in a 2000 cm^3 sedimentation cylinder and filled to the mark with dieionzed water. The suspended solid was measured with a hydrometer after 40 seconds of decantation. The first reading was estimated to be soil content, second as clay content while silt was calculated as the difference between the sum of sand and clay content.

Soil Organic Matter

Loss of ignition method as described by Walkley and Black was used for the determination of soil organic matter. 10g of dried soil sample was weighed into crucible and ignited at 550°C for 2 to 4 hours in muffle furnace until constant mass was achieved[4,5]. Soil organic matter content was then calculated as the Loss of ignition (LOI) using the equation Equation xx

$$LOI(\%) = \frac{DM}{Ms} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots EquationXX$$

Where; DM is loss of mass of the soil after ignition (g), Ms is mass of the soil dried at 105°C and LOI is Loss of ignition.

Organochlorine pesticide residues in soil

The QuEchERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method was used for sample preparation. This method is valued for its simplicity, low cost, and ability to extract pesticides from various matrices. [9, 10]

The QuEChERS method is based on a salting-out extraction with solvent mainly actonitrile and dispersive solid phase extraction. This method is very flexible, modifiable and has a growing popularity. The method has been use to determine wide range of pesticide residues in various sample matrices including soil samples. [9]

Extraction of OCP in Soil

10g of the sieved soil sample was weighed into a cleaned test tube and mixed with 15g anhydrous magnesium sulphate, 20 cm^3 of acetonitrile was added. The mixed was shaken for 20 minutes and allowed to stand for 2 hours. itwas then centrifuge at 3000 rpm for 10 minutes. The cleare supernatant was transferred into 50cm^3 test tube for clean up. [5]

Cleanup of the extract

Primary secondary amine (PSA) is a weak anion exchange sorbent used to extract strong acid and poly-acidic compounds from aqueous samples. After the centrifugation, the sample was clean up using a solid-phase extraction cartridge. The extract was run through the dual pesticide extraction cartridge and eluted with acetonitrile. The process was repeated for all the samples.

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Table 1. The Condition of GC-MS

Parameter	Optimized conditions
Colum temperature	250°c
Detector temperature	300°C
Injection system	Split mode
Injection temperature	300°C
Injection volume	1.0µL
Carrier gas	Helium
Colum length	300 mm
Rate of carrier gas	2.17/min

The analyses were carried out using GC-MS model 7890 Agilent Technologies, equipped with auto sampler, capillary column HP 5ms of length 30 mm and internal diameter of 0.320 mm and 0.25 micrometer thickness. The conditions of analysis are given in Table 1. Split less injection of 1µL was carried out at 300 °C injector temperature with a purge flow of 3ml/minute; the carrier gas used was helium with 99.9% purity and flow rate of 2.17ml/minute while the pressure was 150 kpa. The interface temperature was 300°C.

Sample Analysis for Organochlorine Pesticides Residues

Internal standard technique was employed to analyze the soil extracts. The organochlorine standards used were alphalindane, delta lindane, Endulfan I&II, heptachlor, aldrin, Isodrin, trans-chloro, P-chlorophenyl ethylene (DDMU), DDT, P,P-DDE, dieldrin, Endrin, Endrinketo, melhoxychlor and delta pentachlorocyclohexane with different concentrations ranging from 0.100 ppm to 2.00ppm of the internal standard.

The control and the extracts from the samples were analyzed under the same condition as the standards. Residual level in mg/kg was calculated using the formula below.

$$\text{Residuelevel (mg/kg)} = \frac{peakareaofthesample}{peakareaofthestandard} \times \frac{mg/kgofstandard}{mg/kgofsampleinjected}$$

Analytical quality assurance and control

All the glass ware and tools used during laboratory analysis were properly washed with detergent and running tap water, there after rinsed with distilled water and dried. It was further rinsed with acetone before use.

All the chemicals and reagent used were of analytical grade and obtained from BDH and Sigma & Co and used as purchased. The instruments were calibrated and test run before use, in the case of pH meter and electric conductivity meter, their electrode were rinsed before and after each measurement.

RESULTS AND DISCUSSION

Table 2. Physicochemical Characteristics of Farmland soils

	Physicochemical Characteristics					
Farms	Sand (%)	Clay (%)	Silt (%)	pH	O.M (%)	EC(µS/cm)
A	28.80 ±0.164	51.0±0.78	17.18±10.17	6.45±0.082	3.15±0.0098	45.00±26.46
B	50.09 ± 0.001	25.79±0.755	19.01±0.861	6.39±0.156	3.09±0.303	96.33±75.16
C	32.30 ±0.059	56.35±0.399	13.6±0.56	6.29±0.734	5.44±0.592	200.33±148.30

Table 2 shows results of sand, clay, silt, pH, organic matter, and electrical conductivity of the soils from the farmlands A, B, and C. From the Table, the result shows that soil sample B has sand content of 50.09±0.001, clay content of 25.79±0.755 and silt content of 19.01±0.861, while soil sample A has sand, clay and silt content of 28.80±0.164, 51.0±0.78 and 17.18±10.17 respectively. Soil sample C has sand content of 32.30 ±0.059, clay content of 56.35±0.399 and silt content of 13.6±0.56 Soil sample C with highest clay content is expected to decrease the mobility of the pesticide and have a high water retention capacity because their small particle size. Therefore, the high content of clay in farmland A and C imply more retention of pesticide molecules and their metabolites. Soil sample B has

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the highest percentage sand content but low in clay content. It is expected to have low water retention because of its larger particles size.

Generally the proportion of sand, clay and silt affect the movement of water in soil, sandy soil with large pores and thus high permeability allows rapid flow of water, while clay textures of the soil have low permeability and hence more water retention capacity and tendency to absorb more chemical substances onto it. The behavior of pesticide in the soil environment is greatly influenced by the soil texture. Sandy soil tends to facilitate leaching, clay soils help accumulation through colloid formation. Sandy soil has negative correlation to pesticide because it decreases the retention capacity of the organochlorine pesticide in the soil and in turn reduces the absorption of pesticide by the roots of crops. The clay and silt has positive correlation to the pesticide and have high retention capacity toward the pesticide in the soil and thus increase in absorption of pesticide by the root of crop. [11, 12]

Sample C has highest of organic matter content, 5.44±0.592 with pH of 6.29±0.734, while soil samples A and B has 3.15±0.0098 of organic matter and pH of 6.45±0.082 and 3.09±0.303 of organic matter and pH 6.39±0.156 respectively.

The soil pH and organic matter content determine the leaching and absorption capacity of the soil. Soil pH has a profound effect on soil organic matter preservation and decomposition. The degradation of organic matter is greater under acidic conditions than alkaline condition. Soil sample B was more acidic with low organic matter therefore; decomposition of organic matter is expected to be high. Soil pH is also a factor which influences the bio-availability and transportation of pesticide in the soil. [13]

Organic matter of the soil has a positive correlation with organochlorine pesticide because organochlorine pesticides bind strongly with organic matter of the soil. [14]Therefore, soil C with high organic matter is expected to retain more pesticide and it metabolites.

Pesticides are designed to be adsorb onto organic matter, therefore, the more organic matter in the soil the greater chance that pesticides are being held in the soil and available for its intended use.

EC is an important parameter use to estimate the level of dissolved salt in soil. Farmland A has electrical conductivity of 45.00±26.46, farmland B (96.33±75.16) and farmland C (200.33±148.30) farmland C has the highest electrical conductivity, follow by farmland B and C respectively. It is important that the electric conductivity does not get too high either but within the range of 16-175 µS/cm as recommended by FAO/WHO. Therefore, farmland soil A and B are within the range of recommended FAO/WHO but farmland C is higher than recommended FAO/WHO. Electrical conductivity determined the soil healthiness and nutrient such as dissolved mineral ions including sodium and magnesium.

Table 3. GC-MS Result of Organochlorine Pesticides Residues in Soil

OCPS	A (mg/kg)	B (mg/kg)	C(mg/kg)	MLR
Deta pent	ND	ND	ND	NR
Alpha lindane	0.626	ND	0.050	0.04
Delta lindane	0.016	ND	ND	0.04
Endosulfan I	0.069	0.124	0.054	NR
Heptachlor I	ND	ND	ND	NR
Aldrin	ND	ND	ND	NR
Isodrin	0.148	ND	0.028	NR
Heptachlor II	ND	ND	0.006	0.03
DDMU	0.004	ND	0.003	NR
pp-DDE	ND	ND	ND	NR
Dieldrin	0.0031	0.023	0.006	0.02
Endrin	ND	ND	ND	NR
Endosulfan II	0.011	ND	0.014	0.02
Endrinketo	ND	ND	ND	NR
Methoxychor	ND	ND	ND	NR

Key: ND is not detectable, OCP is organochlorine pesticide, A, B, C is soil samples, MRL is maximum residual limit, and NR is no record.

The results of OCPs residues in the soil samples are shown in the Table 3. From the table, seven OCPs residues were detected in sample A, including alpha lindane, beta lindane, isodrin, DDMU, Dieldrin, Endosulfan I and II, and heptachlor. Nonetheless, only Endosulfan I and Dieldrin were found in sample B, while alpha lindane, Endosulfan, isodrin, DDMU, Dieldrin, Endosulfan II and Heptachlor were seen in the soil of farmland C.

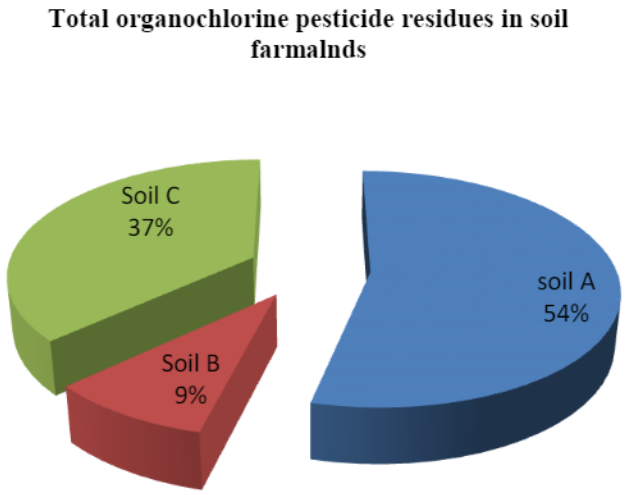

Figure 2. Percentage total organochlorine pesticide residues

The concentration of alpha lindane in farmland A and C were 0.626 mg/kg and 0.05 mg/kg respectively. Alpha lindane was higher in concentration compare to other OCPs residues in soils from all the farmlands (Fig 2). In addition, the value of ∝-lindane was higher than the MRL of 0.04 mg/kg acceptable in environment.[11]

The presence of alpha lindane in soil samples A and C indicated that farmers still use the insecticide in Nigeria as it was also detected in a finding of Ademola, & Gideon[1] in the soil of Cocoa plantation in Ondo State, Nigeria. Alpha lindane was also reported in the findings of in soils from a farmland in Ghana and was lower than MRL.

It is important to note that, alpha lindane is an isomer of hexachlorocyclohexane (HCH), which has been banned for agricultural insecticide because of its neurotoxic effect in human. [15]

Delta lindane is also an isomer of HCH and was only detected in farmland A at concentration of 0.016 mg/kg which is below acceptable MRL for agricultural soil.[16]

The presence of lindane in the farmlands soil indicated the use of hexachlorocyclohexane for control of pest despite the prohibition. The continuous use of the prohibited peaticide by farmers could probably due to its low lost and availability or it considered very active to eradicate pest.

The HCH has eight isomer, but the common isomers used as insecticide are α-,β-,γ- and δ-HCH. Their common name is lindane and it is partially soluble in water, but binds with organic matter of the soil and absorbed by the plant. Because of it persistence in the soil, it was banned worldwide for agricultural use. [17]

Endosulfan I was detected in farmlands A, B and C but predominant in farmland B. Endosulfan II was found in soils A and C with the concentration been higher in farmland C than A. these amounts were both below the MRL set by FAO in agricultural soil.

The low concentration of endosulfan I and II in all the farmlands may be as result of long time use of the pesticide for pest control. Ademola & Gideon reported high concentration of endosulfan I and II in the soil of cocoa plant Ondo, Nigeria which was above MRL[1].

The concentration of endosulfan I in soil A was higher than endosulfan II in the same soil samples. This may be attributed to the composition of endosulfan which normally contained about 67% endosulfan I by mass of total endosulfan content. Endosulfan I are considered more thermally stable

compared to endosulfan II, therefore, it is expected to be more persistent in the environment. [1]

Isodrin was detected in soils A and C at 0.148 mg/kg and 0.028 mg/kg respectively. The presence of isodrin in farmlands is a prove of its continued application despite prohibition for agricultural use.

Isodrin is chlorinated cyclodiene insecticide and isomer of aldrin and it is very stable in soil because of it low degradability. [17]

Heptachlor was found only in farmland C at a level of 0.006 mg/kg which is less than the MRL acceptable for agricultural soil. Heptachlor organochlorine pesticides are banned for agricultural use due to their health hazard as they have been associated with liver diseases in animal and suspected to be human carcinogens. [18]

The pesticide DDMU was present in farmlands A and C at concentrations of 0.004 mg/kg and 0.003 mg/kg respectively. The presence of DDMU in soils A and C maybe as result of long time use of DDT because DDMU is a metabolite of DDT which has be banned for agricultural use, The DDT was also reported in the findings of Fosu-Mensah et al,[11] with concentration of 0.03 mg/kg. It degrades to DDD, DDE and DDMU and all the products pose health hazards to both humans and animals and become the purpose the pesticide was banned worldwide for agricultural us .

Dieldrin was also detected in farmlands A, B and C, having concentration of 0.0031 mg/kg, 0.023 mg/kg and 0.006 mg/kg respectively. Dieldrin in Farmland C was higher than the concentration of dieldrin in farmland soil A while dieldrin in farmland C has the lowest concentration with value of 0.0031 mg/kg. The higher concentration of dieldrin in farmland C could be as result of recent used of the pesticide for pest control. Dieldrin was also reported in the findings of in the sediment of Cocoa producing area of Ondo State with a mean value of 0.1507 mg/kg which was higher than MRL.[18-20]

Dieldrin is an extremely persistent pollutant which does not easily degrade in the environment but tend to biomagnified when it enters the food chain. When ingested, it causes headache, dizziness, and vomiting. It was also found that this chemical can remain in the soil for decade and accumulate in agricultural produce and is unsuitable to humans. This is the reason dieldrin was banned worldwide.

CONCLUSION

The results from this study showed that residues of organochlorine pesticides were present in the soil from vegetable farmlands. Seven banned organochlorine pesticide residues namely; dieldrine alpha lindane, beta lindane, endosulfan I and II, heptachlor, isodrin, and DDMU, detected were below the maximum residue limit of the US Environmental Protection Agency, except alpha lindane, endosulfan I, and isodrin with concentration of 0.624 mg/kg, 0.069 mg/kg and 0.148 mg/kg, respectively, which were above the MRL. The presence of organochlorine pesticides residues may be attributed to illegal use of the pesticides by farmers in the study area or their historical use. Routine monitoring of pesticide residues in the study area is necessary for prevention, control, and the reduction of environmental pollution in order to minimize health risk to humans. In addition, the dam around the study area should be monitoring for possible pesticide contamination and in addition, there is the need to sensitize the farmers on safe pesticide usage, this will reduce the level of pesticide residue in the soils.

Conflict of interest

The authors hereby declare that there are no conflicts of interest.

Author's contributions

The authors have equally contributed to the work.

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