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Determination of pesticide residues in *Solanum tuberosum* (Irish potato) from selected local government areas of Plateau state, Nigeria

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Abstract: This study was conducted to determine the level of pesticide residues in irish potato (Solanum tuberosum) from Mangu, Bokkos and BarkinLadi Local Government Areas of Plateau State, Nigeria. Liquid-Liquid extraction procedure was employed and extract cleanup was done using ethyl acetate, cyclohexane and solid phase extraction. The potato and soil samples were collected from two (2) farms each, from the study areas and analyzed for pesticides residues by gas chromatography equipped with electron capture detector. The temperature of injector operating in a split less mode was held at 250 °C and electron capture detector was set at 280 °C. Fourteen (14) different pesticide residues were detected; HCB, p,p'-DDE, Endosulfans, p,p'-DDD, Glyphosphate, Dichlorvos, aldrin, Emamectin, Lindane, t-Nonachlor, 4,4-bipyridinium, Isopropylamine, g-chlordane, heptachlor. The concentrations of pesticide residues in the potato ranged from 0.0001-0.5074 mg/kg and 0.0001-1.1377 mg/kg in soil. HCB, 0.3579 mg/kg was detected to have the highest concentration in the potato samples while Aldrin, 0.0001 mg/kg had the lowest concentration. In the soil samples analyzed, HCB (1.1377 mg/kg) was detected with the highest level while glyphosphate (0.0001 mg/kg) was found to have the lowest level. The continuous consumption of potato with high pesticide levels can accumulate and could result in detrimental chronic effects in the health of consumers.

1. Introduction

The production of Irish potatoes, sweet potatoes and cocoyam in these local governments contribute immensely to the food security of the nation. However, several factors limit their productivity, mainly insect pests and diseases. As several insect pest and diseases attack these crops, they are produced under very high input or application of insecticides. This increasing trend has been observed in the last decade. With the increase in population coupled with modernization, farmers realized the need to apply chemicals to their crops in order to prevent invasion by pests. It is estimated that as much as 45 % of the world's crop are destroyed by pests and diseases (Bhanti and Taneja, 2007). Pesticides are chemical substances that derive their name from the French word "Peste", which means pest or *plague* and the Latin word "caedere", to kill (Akunyili and Ivbijaro, 2006). Pesticides are widely used in most sectors of the agricultural production to prevent or reduce losses by pests and thus, can improve yield as well as quality of the produce, even in terms of cosmetic appeal, which is often important to consumers (Oerke and Dehne, 2004; Cooper and Dobson, 2007; Bazzi *et al.*, 2013).

Plant protection products (more commonly known as pesticides) are widely used in agriculture to increase yield, improve quality, and extend the storage life of food crops (Crenstil and Augustine, 2010). Pesticides have improved the standard of human health by controlling vector-borne diseases. However, long term and indiscriminate use has resulted in serious health effects. As pesticide use has increased over the past few decades, the likelihood of exposure to these chemicals has also increased considerably (Kanwal *et al.*, 2015). The poor efficiency of the spraying process leads to economic losses, environmental contamination, reducing in biological efficacy of the applied pesticide. Public authorities' preoccupation with those problems has increased significantly during the last few years, particularly in relation to both health and environmental risks.

Pesticides can also improve the nutritional value of food and sometimes its safety (Boxall, 2001), (Narayanasamy, 2006). There are also many other kinds of benefits that may be attributed to pesticides, but these benefits are often unnoticed by the general public (Cooper and Dobson 2007), (Damalas, 2009). Thus, from this point of view, pesticides can be referred to as an economic, labor- saving and efficient tool of pest management with great popularity in most sectors of the agricultural production (Damalas and Eleftherohorinos, 2011). According to World Health Organization (WHO, 2004), about 3,000,000 cases of pesticide poisoning and 220,000 deaths are reported in developing countries each year. About 2.2 million people, mainly belonging to developing countries are at increased risk of exposure to pesticides. Some people are more susceptible to the toxic effects of pesticide than others, such as infants, children, agricultural farm workers and pesticide applicators (WHO, 2004).

Tuber crop cultivation serves as a source of livelihood to some farmers in Plateau state and its environs. Most farmers use synthetic pesticides to control pests on crops in order to increase yield. Due to the indiscriminate use of the pesticides in terms of wrong concentration and frequency of application, residues are left in crops. The main concern is that the levels of pesticide residues in the crops can have adverse effects on human health (Salem et al., 2013). Many of the farmers spray the same wide range of pesticides on all vegetables and ignore pre-harvest intervals (Ntow et al., 2006). Sometimes farmers spray pesticides one day before harvest to sell good looking vegetables. This practice, in particular, exposes consumers to pesticides. Studies conducted indicated that farmers currently use higher than recommended doses of pesticides (Khanel et al., 2023; Sun et al., 2023; Horna et al., 2007). Many of the most widely used pesticides have been classified as Persistent Organic Pollutants (POPs), meaning that pesticides have long life-spans, do not biodegrade well, and have the ability to bio-accumulate in living tissue. When large amounts of pesticides build up in food sources, they contaminate the food chain of nearby communities. (http://www.pan-international.org/panint/files/PAN-Global-Report.pdf) In the modern-day agriculture, the role of pesticides is considered indispensable because of the enormous benefits that come with their application, which include increase in crop productivity and elimination of pests. However, the continued use of pesticides has harmed the ecosystem as a whole. Indiscriminate pesticide use does not distinguish between targeted and untargeted organisms, but it also kills or harms all life forms exposed to it, and it also has the potential to destroy or cause loss of biodiversity.

2. Methodology

2.1 Materials and Reagents

The materials used were Sieve (2mm), mechanical shaker, Whatmann filter paper No. 542, separatory funnel, round bottom flask, rotary evaporator, G.C-ECD. The Reagents used include acetone, hexane, distilled water, anhydrous sodium sulfate, ethyl acetate, cyclohexane, propandiol.

2.2 Sourcing and preparation of samples

Irish potato sample was collected from Chisu in Mangu LGA, Kafiyabo in Barkinladi LGA and Mbar in Bokkos LGA all in Plateau state. The sampling sites were divided into small grids of dimension 10 x 5 m each. Ten of such sites were obtained in each sampling location. The tuber samples and the corresponding soil samples were taken from each grid. Tuber samples were carefully uprooted using a stainless knife. The corresponding soil samples were collected at a depth of 0-15 cm using a stainless-steel hand trowel. Soil and the tuber samples collected were stored in sterilized black polythene bags and labeled according to location for easy identification. The soil and tuber samples were transferred to the laboratory within 48 hours of collection and were stored at temperature of 4 °C (Udosen *et al.*, 2016).

2.2.1 Soil Samples

The soil samples (1.0 kg) were air-dried, ground and homogenized. The samples were then sieved using a 2 mm sieve. The method reported by (Tahir *et al.* 2009) was adopted for the extraction of pesticides residues in soil. Soil sample (50.0 g) was weighed into a conical flask and then 50 cm³ of a mixture of acetone and hexane (1:1) was added. This was shaken for 1 hour with the help of mechanical shaker at a rate of 300 rpm. The mixture was filtered through a glass wool plug with whatmann filter paper No 542 into a separatory funnel. The extract was washed with distilled water (2 x 100 cm³). The lower aqueous layer was discarded and a few grams of anhydrous sodium sulphate was added. The aliquot (20.0 cm³) was transferred to a round bottom flask and evaporated to dryness at 40 °C in a rotary evaporator. The content of the flask was mixed with 6 cm³ ethyl acetate and cyclohexane (1:1) mixture. The sample was dried again and 1 cm³ ethyl acetate was added and then passed through high flow super cell. The samples were further dried under vacuum and 1 cm³ ethyl acetate was added, and then analysed on GC-ECD (Osesua *et al.*, 2017).

2.2.2 Potato Samples

The potato tubers of each sample (25.0 g) were blended separately in blender (2.0 dm³ Blender/Grinder QBL-1861A), with 50 cm³ acetone for 3 minutes. To the mixture, 50 g sodium sulphate, 50.0 cm³ cyclohexane and ethyl acetate (1:1) were added and further blended for another 2 minutes. The whole mixture was allowed to stand for 20 minutes and then 30.0 cm³ of clear supernatant was measured out. Three drops of 10 % propandiol was added and evaporated on rotary evaporator. The remaining liquid was air dried to get rid of the solvent completely. This was reconstituted in ethyl acetate in a volumetric flask (5.0 cm³), and analyzed by Gas Chromatography.

2.3 Sample Analysis

Quantification of different pesticides in soils and tuber extracts was accomplished by Perkin Elmer Autosystem Gas Chromatography equipped with ECD under the parameters; Column: 17 m methyl 10 and phenyl silicone 0.32 mm ID, 0.5 µm film thickness. Injector tempt,: 2200 °C Detector temp.: 350 °C, Detector makeup, Nitrogen: flow 33.3 ml/min, Oven: Temp. 80 °C, initial time 0 min. Rate 20 °C/min. Final temp. 280 °C stay for 0 min, final time 10 min. Injection:1µl splitless, integration: peak height was used for quantitation (Tahir *et al.*, 2009).

2.4 Quality Assurance

Quality assurance involved instrument calibration using certified standards. A mixture of pesticides standards (M8081SC standard, supplied by AccuStandard, New Haven CT, USA) containing 17

Pesticides: dieldrin, Biphenyl, HCB, p,p'-DDE, endosulfans, p,p'-DDD, glyphosphate, dichlorvos, aldrin, emamectin, lindane, t-Nonachlor, 4,4-bypyridinium, isopropylamine, g-chlordane, heptachlor and DDT was used to prepare the standard solutions in acetone and n-hexane. The 17 pesticides were well resolved and eluted within 45 min. The correlation coefficients of the standards were 0.990 (99%) in all runs.

3. Results and Discussion

3.1 Data Presentation

Figure 1 represents the concentrations of pesticide residues in potato and soil from Mangu local government area while Figure 2 shows the concentrations of pesticide residues in potato and soil from Bokkos local government area. Figure 3 represents the concentrations of pesticide residues in potato and soil from Barkin ladi local government area. While Figure 4 shows the concentration of pesticide residues in potato in the three local government areas.

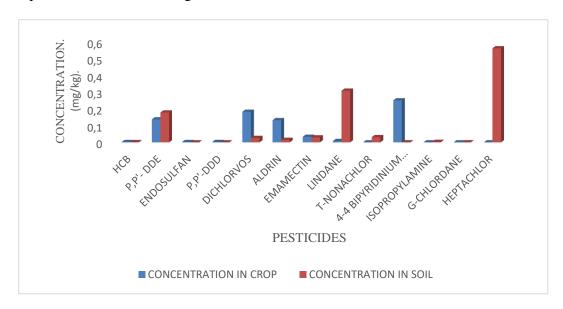


Figure 1: Concentration of Pesticide residues in potato and soil from Mangu local government area

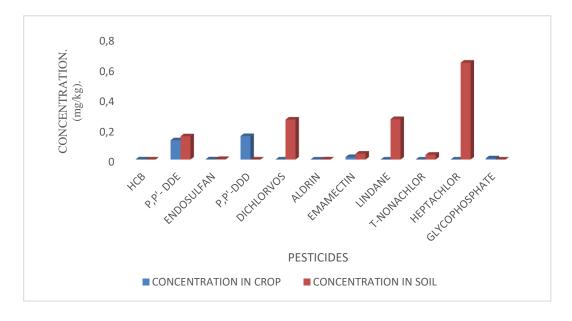


Figure 2: Concentration of Pesticide residues in potato and soil from Bokkos local government area

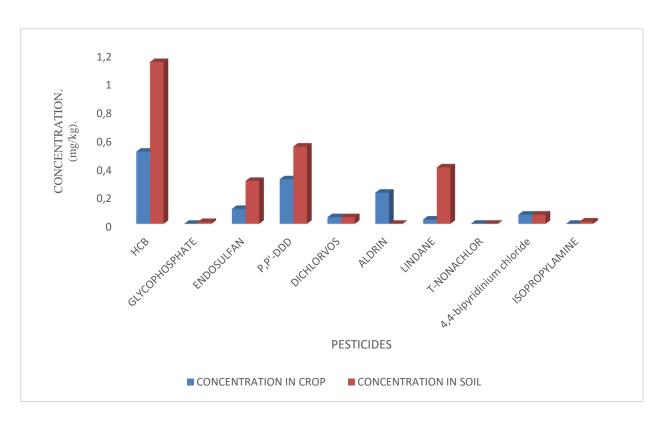


Figure 3: Concentration of Pesticide residues in potato and soil from Barkin ladi local government area

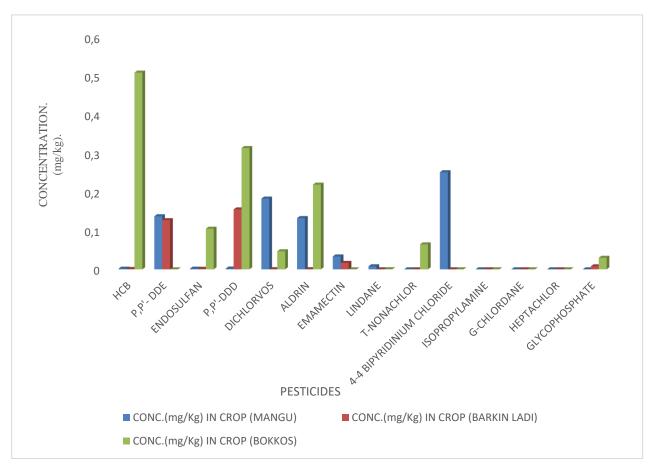


Figure 4: Concentration of pesticide residues in potato in the three local government areas

Figure t-Test Comparison of mean levels of pesticides in potatoes and soils across the three LGAs

Variables	Mangu	Bokkos	BarkinLadi	F	P-value	
Mean level in potatoes	0.0534±0.09	0.0220±0.09	0.0917±0.15	1.548	0.226	
Mean level in potato soil	0.0831±0.16	0.0997±0.18	0.1799±0.33	0.674	0.515	Statistically
significant at P≤0.05						

Table 2: t-Test Comparison of mean levels of pesticides in potatoes and soils between two LGAs

S/N	Dependent	(I) LGA	(J) LGA	Std. Error	P-value	95% Confidence Interval	
	Variable					Lower Bound	Upper Bound
1	Quantity of	Mangu LGA	Bokkos LGA	0.0892	0.981	-0.2340	0.201
	pesticide in potato soil	Mangu LGA	Barkin-ladi LGA	0.0892	0.528	-0.3142	0.120
		Bokkos LGA	Barkin-ladi LGA	0.0892	0.644	-0.2975	0.137
2	Quantity of	Mangu LGA	Bokkos LGA	0.0397	0.710	-0.0653	0.128
	pesticide in	Mangu LGA	Barkin-ladi LGA	0.0397	0.603	-0.1350	0.058
	potato	Bokkos LGA	Barkin-ladi LGA	0.0397	0.198	-0.1664	0.027

Statistically significant at P≤0.05

3.2 Data Analysis

3.2.1 Pesticide Residues in Potato

Results for pesticide residue in the potato samples analyzed at Mangu, Bokkos, and Barkin-Ladi are presented in Figure 1, 2 and 3 respectively. Pesticide residues were detected in all the three samples. The predominant pesticides were HCB (0.0009-0.5074 mg/kg), endosulfan (0.0013-1.1047 mg/kg), p,p'-DDD (0.0019-0.3128 mg/kg), dichlorvos (0.0003-0.1824mg/kg), and Aldrin (0.0001-0.2185 mg/kg). p,p'-DDE (0.0332 mg/kg). Emamectin (0.0165 mg/kg) was only found in samples at locations in Mangu and Bokkos while lindane (0.0079-0.0297 mg/kg) and 4,4-bipyridinium chloride (0.0644-0.2504 mg/kg) were present in potato samples collected from Mangu and Barkin-Ladi. Glyphosphate (0.0077 mg/kg) was only found in sample location at Bokkos while t-Nonachlor (0.0003mg/kg) was only found in sample collected from Barkin-Ladi, and at a very low concentration when compared to the EU MRL.

The Concentrations of endosulfan from the three locations (0.0013-0.1047 mg/kg) were lower than the levels (0.057 mg/kg-2.818 mg/kg) in potato from Bangalore Rural District by (Ramesh and Yogananda, 2013). Endosulfan contamination in potato samples was high because OCPS might be used on pests which are active during April to June when the vegetables are growing. Sometimes high temperatures in summer makes the OCPs volatile from their reservoirs such as soil or vegetation and that the edible parts of the crop may have tapped some of the evaporating pesticides (Odhiambo *et al.*, 2009).

The presence of metabolites of DDT (p,p'-DDD,p,p'-DDE) in samples analyzed suggested that the banned chemical is still in use by the farmers in crops cultivation. Furthermore, results from the residue analyzed indicated that dichlorvos (0.0003-0.1824mg/kg), aldrin (0.0001-0.2185 mg/kg) and 4,4-bipyridinium (0.0644-0.2504 mg/kg) in samples collected from Mangu and Barkin-Ladi had values above the MRL, but were observed to be within the permissible limits in Bokkos. Emamectin was high

in Mangu (0.0332 mg/kg), low in Bokkos (0.0165 mg/kg) and not detected in sample from Barkin-Ladi. Lindane (0.0297 mg/kg) was above the (EU, 2007) MRL in sample from Barkin-Ladi, low in sample from Mangu and not detected in sample collected from Bokkos. t-Nonachlor had a negligible concentration of (0.0003 mg/kg) in sample collected from Bokkos. This agreed with the previous findings by (Srivastava *et al.*, 2011) on the evaluation of residues of 48 pesticides in vegetables including 10 synthetic pyrethriods, 13 organochlorines, 8 herbicides and 17 organophosphates. Out of total 48 pesticides, 23 pesticides were detected in all 60 analysed vegetables with the range of (0.005-12.35 mg/kg). The variation in the residue levels in the individual samples were expected because most farmers use different pesticide concentrations at different stages of cultivation.

3.2.2 Pesticide Residues in Soil

The soil is the repository of chemicals such as organochlorine pesticides. Many organochlorine pesticides have a high affinity for soil, which might be taken up by crops and by grazing animals and hence reach the human food chain. They are also washed in run-off from land into watercourse and emitted into atmosphere through volatilization, which results in water and atmospheric contamination (Bidleman and Leone, 2004).

The levels of pesticides detected in soil samples are shown in Figures 1-3. The pesticides include HCB (0.0012-1.1377 mg/kg), p,p'-DDE (0.0081-0.1780 mg/kg), Endosulfans (0.0023-0.3015 mg/kg), p,p'-DDD (0.0010-0.5428 mg/kg), Glyphosphate (0.0001-0.1393 mg/kg), Dichlorvos (0.0150-0.2632 mg/kg), Aldrin (0.0006-0.1501 mg/kg), Emamectin (0.0386-0.0397 mg/kg), Lindane (0.2204-0.3965 mg/kg), t-Nonachlor (0.0003-0.0315 mg/kg), 4,4-bypyridinium (0.0301-0.0645 mg/kg), isopropylamine (0.0010-0.0162 mg/kg), g-chlordane (0.0003 mg/kg), heptachlor (0.5608-1.0829 mg/kg). Chlordane was detected at a concentration of 0.0003 mg/kg in the potato soil sample at Mangu only, was lower compared to the EU MRL. Chlordane persists for more than twenty years in some soils (Beryanan *et al.*, 1981) thus chlordane level in the soil may be the result of past usage.

The HCB, p,p'-DDE, endosulfans, p,p'-DDD, dichlorvos, emamectin, lindane, t-nonachlor, 4,4-bypyridinium, Isopropylamine and heptachlor were found to be above the EU MRLs for agricultural soil. This indicates that the agrochemicals are still in use by some farmers. Heptachlor was detected in the soil samples though it was not found in any of the crop samples. It tends to stay in the soil for long periods of time (Baah, 2014). Heptachlor has been shown to cause cancer in laboratory animals when exposed at high levels over their lifetimes. This may increase the risk of cancer in humans who are exposed for a long time (Udeh, 2004).

Most of the pesticides detected, were found to be above the EU MRL, which posed a threat to human health as stated earlier, but they are said to be within the permissible limits when compared to maximum residue levels by Tanzania Bureau of Standards (TBS).

3.2.3 Statistical Analysis

Figures 1 to 4 represent the variations between the crops and the soils from the three locations. The levels of pesticide residues in the soils were higher than the residues in the crops which might be as a result of accumulation from spraying the farms with pesticides.

Figure 4 represents the comparism of the pesticide residues in potato samples from the three locations. Bokkos Local Government Area recorded the highest level of pesticide residues. This could be as a result of the increase in the rate at which farmers spray potato with pesticide. The increase may be as a result of outbreak of potato blast disease which has persisted in the locality for the past seven years; where from six weeks of planting, the potato farm is sprayed twice a week to prevent infection with

the disease. There could be other factors responsible for the high levels of these pesticides in the potato which include the fertilizers and the herbicides used on the farm. Another reason could be as a result of uptake through the roots.

Analysis of ANOVA (Table 1) shows that there was no significant difference ($P \le 0.05$) in the concentrations of pesticides in the tubers and soils from the three local government areas, as all the p-values were greater than the confidence limit (0.05). Similarly, from the t-Test of the levels of pesticides in the potato and soil (Table 2) between any two study areas were not significantly different ($P \le 0.05$). indicating even distribution of the pesticides among the sampling sites.

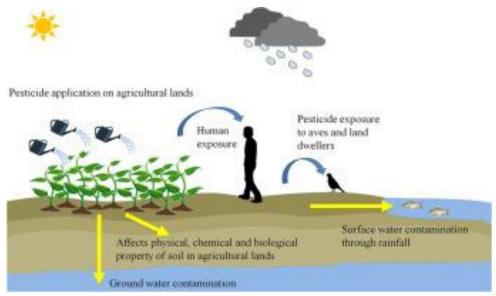


Figure 5: photo abstract on the effect of pesticides on the Environment (Rajak et al., 2023)

Conclusion

The laboratory analysis revealed the presence of banned organochlorine and organophosphate pesticides in tubers and soil samples analyzed which include HCB, p,p'-DDE, endosulfans, p,p'-DDD, glyphosphate, dichlorvos, aldrin, emamectin, lindane, t-Nonachlor, 4,4-bypyridinium, isopropylamine, g-chlordane, and heptachlor. HCB and heptachlor recorded the highest concentrations of (1.1377 mg/kg) and (1.0829 mg/kg) respectively in the soil samples, while HCB had the highest concentration of (0.5074 mg/kg) in the crop samples which was above the EU MRLs. This poses a potential health risk to consumers of potato produced from study areas. Because of their lipid solubility and resistance to metabolism, they can bio-accumulate in human tissues of consumers. So, chronic exposure could pose health problems. The results of the study also revealed the unregulated use of pesticides. The study showed that the organochlorine and organophosphate pesticides are still being used despite the fact that they have been banned from use by FAO/WHO.

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