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DETERMINATION OF PESTICIDES RESIDUES IN BEANS OBTAINED FROM SOME LOCATIONS IN HONG LOCAL GOVERNMENT AREAS OF ADAMAWA STATE, NIGERIA

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Abstract: **The people of Hong Local Government Area of Adamawa State depend heavily on agricultural practices and they depend mostly on the use of pesticides to boost agricultural production. This research aimed at Assessing the Pesticides residues in beans samples produced in some location in Hong local Government areas, the locations are Hong, Pella, Kwakwa'a Garaha, Gashala Kufom and Uba. The pesticide residues analysis was carried out using GC/MS, data analysis was carried out on SPSS. using one-way analysis of variance (ANOVA) while mean was separated according to Duncan's Multiple Range, (P<0.05) was considered statistically significant. The pesticide residues analyzed are organochlorines and organophosphate pesticides residues. The organochlorine pesticide includes ∞-HCH, β-HCH, y-HCH, chlorothalonil, Hepatechlor, Aldrin, Heptatachlor Epoxide, endosulphan I dieldrin, Endrin, Endosulphan II, P_1 P^1 –DDT and endosulphan sulphate. 13 organophosphate pesticides residues were detected across the areas studied, they are Diclorvos, mevinfos, Diazinon, Diclofenthion, Phosphamidon, Pirimophos-methyl, Chlorpyrifos, Parathion, Fenthion, Isofenfos, Bromophos and Ethion. Organochlorine pesticides residues was found to have highest concentration in the beans sample than in the control sample, it was also seen that in most of the location studied it have relatively high concentration than Organophosphate pesticides residues. Some of the Organophosphate pesticides' residues were detected in few of the location studied with relatively low concentration compare to Organochlorine. High concentration of the pesticides residues indicates the high level of contamination of the beans by the pesticides at the time of the research. These results indicate the need for action to be taken to allow the revision of practices in the administration of pesticides during both cropping and storage of beans in Nigeria to reduce risks of pesticides poisoning.**

Keywords: **Pesticides, Organochlorine, Organophosphate, Residues, Pollution. Bioaccumulation.**

1. INTRODUCTION

Pesticides are chemical compounds or mixture of substances aimed at preventing, combating, and mitigating the effect of pests and vectors on agricultural plants and domestic animals. They are design to kill animals, plants and insects in agricultural and domestic backgrounds such as herbicides, insecticides, fumigants, and fungicides with diverse methods (Suleiman *et al.*, 2020). Pesticides have become widespread pollutants and of global concern because of their persistence

in the environment, bioaccumulation potential in the tissues of animals and humans through the food chain, and their toxic properties for humans and wildlife (FAO/WHO, 2021).

Basically, human exposure to pesticides can occur through several pathways, including dermal absorption and inhalation of air particulates, but the ingestion of contaminated agricultural food accounts for more than 90% of total exposure (Okechukwu, *et al*.,2019, Shalaby *et al*., 2021). The human health effects associated with short term exposure to hazardous pesticides includes headache, dizziness, nausea, vomiting, and convulsion while the long-term exposure is associated with a broad spectrum of possible health effects in human that include neurotoxicity, reproductive toxicity and cancer (Otito *et al*., 2021).

Organochlorine (OCPs) These compounds consist of hydrogen, carbon and chlorine, along with other elements like oxygen and Sulphur. DDT and other organochlorine pesticides have been used extensively ever since the 1940s due to their high performance in preventing diseases and controlling pests (Inobeme *et al*., 2020). OCPs were utilized to manage parasites on cotton and other crops Safiatou, (2007). They have been known to have poor water solubility, lipid-soluble, long - lasting persistence, long-range transport nature, toxicities, and bioaccumulation properties (Cemile,2016). Organophosphates pesticides.

Organophosphates are chemical compounds formed when phosphoric acid and alcohol react and hence include all pesticides containing phosphorus. In early 1940s the discovery of organophosphates becomes as a substituent or alternative to persistent organochlorine derivatives due to their less persistence in the environment (Olawale et al., 2021). These chemicals can cause immediate health problems when consumed, inhaled, or absorbed through the skin (Asiegbu *et al*., 2022). Organophosphates kill insects by interfering with their brain and nervous system activity. They can affect both humans as well as wild animals' nervous systems due to similarities in brain biochemistry (Olalekan *et al*., 2021).

Crop pesticides can undergo volatilization, photolysis chemical, and microbial degradation while still on the surface, all these activities can decrease original pesticides concentration but can also activate some metabolites in the crops, (Mshelia *et al*., 2022). Volatilization of the pesticides usually ensues instantly when applied in the field. The volatilization process mainly depends on the velocity of vapour density of the pesticides, pesticides with high vapour pressure usually volatilize rapidly into the atmosphere while those with low vapour pressure stay longer on the surface, (Emmanuel *et al*.,2023). Volatilization rate also depends on conditions such as location temperature and wind speed. The pesticides will evaporate further as the faster the wind speed and the higher the temperature intensifies. (Ramadan *et al*.,2020).

Photolysis takes place when molecules absorb sunlight energy resulting in pesticide degradation. The indirect reaction can also be triggered by some other chemicals being broken by the sunlight and their product reacting with pesticide, and use the pesticides as nutrients thereby breaking them into carbon dioxide and other component (Bando *et al*.,2023). Although degradation of pesticides is influence by different environmental field condition, volatilization remains process that affects pesticide. This work aimed at determining the pesticide residues levels in some common beans samples obtained from some part of Hong local government areas of Adamawa state, Nigeria. so as to evaluate their suitability for human consumption.

Study Area

Hong LGA is located between latitudes 10° 00′ 00″ N and 10° 35′ 00″ N and longitudes 12° 35′ 00″ E and 13° 20′ 00″ E. It has a total land area 2,419.11 km^2 (Bashir and Raji, 1999). Hong is one of the 21 Local Government Areas of Adamawa State created in 1987 during the defunct Gongola State. It has its headquarters in Hong town being the largest settlement and is classified by Ilesanmi (1999) as a third order core urban settlement in Adamawa state. Hong Local Government Area consists of seven (7) districts namely; Hong, Dugwaba, Pella, Kulinyi, Hildi, Gaya and Uba. The people leaving in the location are mostly farmers by occupation.

Collection of Samples

Samples of white beans was collected from six locations in Hong local government areas which are Hong, Pella, Kwakwa'a, Garaha, Gashala Kufom and Uba. The samples were code-named and stored in glass bottles with tight covers to protect them from moisture and contamination. They were then stored in the refrigerator at 4^oC until ready for further action.

Preparation of Samples

The samples were cleaned by picking out stones and other extraneous materials. Each sample was thoroughly mixed and 200g portion was taken and milled to 20 mesh particle size to produce a good homogenate. The grinding machine was cleaned up with acetone. The milled samples were then stored in glass bottles with appropriate labels in a refrigerator at 4^{o}C. Duplicate portions (200g) of the samples was stored as whole grains in labeled glass bottles in the refrigerator as backup samples. (Lozowicka,2017)

Extraction and Clean-up of Samples

Extraction was carried out according to Akan *et al*., (2015) The milled sample was properly mixed and 2g was weighed into a 20ml sample vial. Anhydrous sodium sulphate (1g) was added and mixed with the sample to absorb any moisture present. The sodium sulphate was heated at 650^{o}C for one hour and stored in a desiccator. Ethyl acetate (10ml) was added to the vial. The mixture was vortex mixed for 5min and then allowed to stand for 45min. It was then mixed again and centrifuged for 5min. at 2500 rpm. The supernatant was carefully transferred into a flask. The residue was further extracted twice as described above, using 10ml ethyl acetate each time. The supernatants were combined and reduced to about 5ml using a rotary evaporator at 35^{o}C. The solution was transferred to a sample tube and reduced to about 1ml under a gentle stream of nitrogen gas using a nitrogen evaporator at 36^{o}C. This was taken for florisil cleanup. For the clean-up, solid phase extraction cartridges (Florisil™, 500mg/6ml) was used. Each cartridge was conditioned with 5ml of the eluting solvent mixture (hexane/ethyl acetate 50:50) and the sample extract (1ml) was loaded on the Florisil™.

The sample tube was rinsed three times with 1ml eluting solvent, and the rinses added to the florisil column. The sample was then eluted with 5ml of the same solvent mixture into a receiving glass tube. The Florisil™ column was rinsed with another 3ml of the eluting solvent mixture into the same receiving glass tube. The eluate was then evaporated to dryness under a gentle stream of nitrogen gas and the residue reconstituted in 1ml ethylacetate for GC-MS analysis. Preparation of Calibration Curves Stock solutions of the organochlorine pesticides was prepared and then serially diluted to produce different concentrations (0.05, 0.5, 1.0, 2.0, 4.0 and 8.0 µg/ml) of individual pesticides. Stock standard solutions was stored in amber colored bottles at 4^{o}C in a refrigerator and working standard solutions was prepared fresh before use. Standard solutions of the pesticides were run on GC/MS under the set chromatographic conditions and mean peak areas was plotted against concentrations to obtain calibration curves of individual pesticides.

Analysis of Pesticide Residue Content

All compounds were determined and quantified with the aid of a gas chromatograph equipped with a mass selective detector (GC-MS), an autosampler and a split less injector. The DB-5 fused silica capillary column of 30m x 0.25µm i.d. x 0.25µm film thickness was coated with cross-linked 5% phenyl dimethyl polysiloxane. The carrier gas was helium (99.999% purity) at a flow rate of 1.0 ml/min. Oven temperature 0^{o}C was maintained initially at 70^{o}C for 1min, increased at 000015 C/min to 175 C, then at 2C/min to 215 C, at 000010 C/min to 265 C and finally at 20 C/min to 290 C and held for 8min. Injection volume was 1µL, injected in 0 split less mode at injection temperature of 250^{o}C. The mass spectrometer (Trace 2000 Series, Thermo Quest, Italy) was operated in electron impact (EI) ionization mode with a detector voltage of 700 V, 0 ion source temperature of 200^{o}C, GC interface temperature of 320^{o}C and emission current of 150 µV. Acquisition mode was selected ion monitoring (SIM).

Identification and Quantification

Pesticide residues were identified if the retention times matched those of the standards and the relative abundances was within 10% of those of the standards. Identified pesticides was quantified using the external standard method of comparing sample peak areas with those of the pesticide standards under the same conditions. Each sample was analyzed three times and the mean values was obtained. The pesticide residue content of each sample was calculated as: Pesticide Content = Wts As ´ CF Vf Where As = peak area of sample Vf = final volume of clean extract Wts = weight of sample extracted CF = calibration factor. The calibration factor of each pesticide was calculated as: CF = Total Amount of Standard Injected Peak Area of Standard Mean and maximum concentrations of pesticide residues found in the samples.

Data Handling

All analysis was done using SPSS. Results was presented as mean ± SD. Statistically significant Figures was establish using one-way analysis of variance. (ANOVA). Mean was separated according to Duncans Multiple Range Analysis. $P \leq 0.05$ was considered statistically significant

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2. RESULTS

Table 1 and 2: Present the Mean Concentration of Organochlorine Pesticides Residues in (mg/kg) in Beans and Control Samples Obtained from Some Locations in Hong Local Government Areas.

The table below shows about fifteen (15) organochlorine pesticides residues were studied in both the Bean and the control sample. the organochlorine pesticides residues studied are ∝-HCH, β-HCH, γ-HCH, Chlorothalonil, Heptachlor, Aldrin, Heptachlor-epoxide, Endosulphan I, Dieldrin Endosulphan II, P_1P^1-DDD, Endosulphan sulphate and P_1P^1- DDT,

in Hong study areas. All the organochlorine pesticides residues were detected, with Heptachlor having the highest concentration and Aldrin was found to have the least concentration, while in the control sample Aldrin, Dieldrin, Endrin, Endosulphan II and P_1P^1-DDT were not detected. In Pella all the organochlorine pesticides residues were detected with the exception of Aldrin and Heptachlor epoxide which are found to be below the detection limit, while in the control sample only β-HCH, Endosulphan II, P_1P^1- DDD and Endosulphan sulpahte were detected in Pella, the rest were found to be below the deduction limit. In Kwakwa'a study area both the sample and the control contained all the organochlorine pesticides residues except chlorothalonil, Heptachlor epoxide, Aldrin, Endosulphan I, Dieldrin and Endosulphan II .

In Garaha study area all the organochlorine pesticides residues were detecting with the exception endosulphan I which is below the detection limit, it was found that β-HCH have the highest concentration while endrin was found to have the least concentration. In the control sample ∞-HCH, β-HCH, γ-HCH, Endosulphan II, P_1P^1-DDD, Endosulphan sulphate and P_1P^1-DDT were detected in the control sample. In Gashala Fufon study area, all the organochlorine pesticides residues were detected with the exception of Dieldrin and endosulphan II which was found to be below the detection limit at the time the research was carried out, while in the control sample, all the pesticides residues are present with the exception of β-HCH, Heptachlor, Endosulphan I, Endosulphan II and P_1P^1-DDT. In Uba study are only Endosulphan Sulphate was not detected in the sample studies, meanwhile in the control sample only γ-HCH, Chlorothalonil, ∞-HCH, Heptachlor, Heptachlor epoxite, Endrin and Endosulphan II were detected.

Table 1: Present the Mean Concentration (mg/kg) of Organochlorine Pesticide Residues in Bean Sample from Some Locations in Hong Local Government Adamawa State.

Pesticides	Hong	Pella	Kwakwa'a	Garaha	Gashala Kufom	Uba
∞-HCH	0.279± 0.092	0.237± 0.11	0.237±0.028	0.194±0.045	0.303±0.132	0.145±0.13
β-HCH	0.132±0.087	0.234±0.011	0.258±0.051	0.336±0.072	0.17±0.034	0.378±0.117
γ-HCH	0.124±0.042	0.134±0.19	0.332±0.066	0.176±0.009	0.214±0.036	0.126±0.121
Chlorothalonil	0.264±0.039	0.147±0.041	0.136±0.026	0.275±0.080	0.153±0.049	0.182±0.041
Heptachlor	0.326±0.126	0.226±0.084	0.258±0.072	0.246±0.073	0.104±0.071	0.216±0.073
Aldrin	0.046±0.009	BDL	0.053±0.042	0.190±0.03	0.412±0.131	0.216±0.075
Heptachlor epoxide	0.296±0.051	BDL	0.062±0.035	0.296±0.113	0.332±0.159	0.054±0.072
Endosulphan I	0.232±0.079	0.179±0.051	0.110± 0.03	BDL	0.062±0.037	0.052±0.042
Dieldrin	0.066±0.007	0.122±0.101	0.018±0.007	0.027±0.12	BDL	0.061±0.043
Endrin	0.078±0.034	0.211±0.134	BDL	0.164±0.059	0.231±0.071	0.258±0.159
Endosulphan II	0.178±0.113	0.316±0.084	0.142±0.061	0.371±0.121	BDL	0.397±136
P. P'-DDD	0.281±0.124	0.411±0.137	0.301±0.087	0.297±0.082	0.259±0.110	0.204±0.091
Endosulphan sulphate	0.269±0.081	0.401±0.084	0.249±0.061	0.170±0.054	0.381±0.072	BDL
P, P' DDT	0.182±0.011	0.142±0.051	0.206±0.042	0.263±0.117	0.173±0.082	0.119±0.016

Values are Presented in mean ± standard deviation of triplicate determination. BDL, below detection limits of 0.0015-0.004mg kg^{-2}

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Table 2 Present the Mean Concentration of Organochlorine Pesticides Residues in (mg/kg) in the Control Samples Obtained from Some Locations in Hong Local Government Areas, Adamawa State.

Pesticide	Hong	Pella	Kwakwa'a	Garaha	Gashala Kufom	Uba
∞-HCH	0.113±0.047	BDL	0.081±0.016	0.092±0.014	0.172±0.073	BDL
β-HCH	0.092±0.031	0.164±0.092	0.116±0.073	0.217±0.114	BDL	0.291±0.141
Y-HCH	0.097±0.021	BDL	0.217±0.091	0.079±0.021	0.171±0.049	BDL
Chlorothalonil	0.146±0.098	0.096±0.017	0.072±0.013	0.016±0.009	0.092±0.042	0.194±0.064
Heptachlor	0.312±0.112	0.097±0.034	0.218±0.119	BDL	0.082±0.047	0.192±0.092
Aldrin	BDL	BDL	BDL	BDL	0.216±0.073	0.091±0.018
Heptachlor epoxide	0.146±0.073	BDL	BDL	0.163±0.012	0.173±0.041	0.119±0.072
Endosulphan I	0.149±0.124	0.067±0.017	BDL	BDL	BDL	BDL
Dieldrin	BDL	0.084±0.013	BDL	0.096±0.034	0.172±0.046	BDL
Endrin	BDL	0.074±0.19	0.194±0.083	BDL	0.104±0.039	0.143±0.062
Endosulphan II	0.092±0.043	0.219±0.113	0.098±0.027	0.216±0.124	BDL	0.152±0.119
P. P'-DDD	0.214±0.113	0.287±0.012	0.215±0.102	0.183±0.022	0.143±0.063	BDL
Endosulphan Sulphate	0.213±0.101	0.329±0.115	0.194±0.067	0.211±0.064	0.134±0.027	BDL
P, P' DDT	0.079±0.034	BDL	0.114±0.064	0.214±0.038	0.094±0.024	BDL

Values are Presented in mean ± standard deviation of triplicate determination. BDL, below detection limits of 0.0015-0.004mg kg^{-2}

Table 3 and 4: Present the Mean Concentration (mg/kg) of Organophosphate Pesticides Residues in Beans and the Control Samples from Some Location in Hong Local Government Area, Adamawa State.

The total of (14) organophosphate pesticides residues were studied in both the bean and the control samples, they are Diclorvos, mevinfos, Diazinon, Diamethoate, Diclofenthion, phosphomidon, pirimophos-methol, chlorpyrefes, parathion, fenthion, isofenfos, Bromophos and Ethion.

In Hong study area the following organophosphate were detected, diclorvos, mevinfos, Diamethoate, Phosphomidon, parathion, Isofenfos, levinfos, Diamethoate, Phosphomidon, parathion, Isofenfos and Ethion, while in the control sample from the same study area only diclorvos and Ethion were detected. In pella study area only seven (7) organophosphate pesticides residues were detected, they are Diclorvos, mevinfos, Diazinon, phophomidon, chlorpyrifes, parathion and isofenfos, while in the control sample only three (3) organophosphate pesticides residues were detected, which include diclorvos, Diazinon and chlopyrifes. In Kwakwa'a study area six (6) organophosphate pesticide residues were detected, they include diclorvos, Diamethoate, diclofenthion, pirimophose-methyl, fenthion and Bromophos while the rest were below the detection limit at the time of the research. In the control samples, four (4) organophosphate pesticides residues were detected, they are diclorvos, diclofenthion, pirimophos-methyl and Bromophos.

In Garaha study area, six (6) organophosphate were detected they are diclorvos, Diazinon, phosphomidon, pirimophos-methyl, fenthion and Isofenfos, while in the control sample only Bromophos was detected. In Gashala Kufom, diclorvos, mevinfos, diclofenthion, parathion, Isofenfos and Bromophos were detected while in the control sample diclorvos, pirimophos-methyl, chlopyrifes Isofenfos and Ethion were detected. In Uba study area diclorvos, diamethoate, diclofenthion, parathion and Isofenfos were detected, in the control samples from the same study area, diclorvos, diamethoate, pirimophos-methyl, chlorpyrite and Isofenfos were detected, the rest are below the detection limit at the time of the research.

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Table 3: Present the Mean Concentration (mg/kg) of Oganophosphate Pesticides Residues in Bean Samples from Some Location in Hong Local Government Area, Adamawa State.

Pesticide	Hong	Pella	Kwakwa'a	Garaha	Gashala kufom	Uba
Diclorvos	0.332±0.079	0.119±0.023	0.243±0.101	0.051±0.029	0.194±0.021	0.163±0.049
Mevinfos	0.068±0.039	0.041±0.019	BDL	BDL	0.072±0.048	BDL
Diazinon	BDL	0.326±0.114	BDL	0.03±0.01	BDL	BDL
Dimethoate	0.073±0.027	BDL	0.019±0.012	BDL	BDL	0.137±0.012
Diclofenthion	BDL	BDL	0.113±0.069	BDL	0.062±0.014	0.013±0.002
Phosphamidon	0.007±0.002	0.039±0.011	BDL	0.014±0.006	BDL	BDL
Pirimophos-methyl	BDL	BDL	0.137±0.041	0.043±0.017	BDL	BDL
Chlorpyrites	BDL	0.329±0.047	BDL	BDL	0.214±0.027	0.118±0.037
Parathion	0.053±0.042	BDL	BDL	BDL	BDL	BDL
Fenthion	BDL	BDL	0.012±0.009	0.022±0.003	BDL	BDL
Isofenfos	0.037±0.013	0.028±0.009	BDL	BDL	0.039±0.017	0.124±0.032
Bromophos	BDL	BDL	0.052±0.019	0.032±0.032	BDL	BDL
Ethion	0.037±0.019	BDL	BDL	BDL	0.022±0.011	BDL

Values are Presented in mean ± standard deviation of triplicate determination. BDL, below detection limits of 0.0015-0.004mg kg^{-2}

Table 4: Present the Mean Concentration (mg/kg) of Organophosphate Pesticides Residues in Control Sample of Bean from Some Location in Hong Local Government Areas, Adamawa State.

Pesticide	Hong	Pella	Kwakwa'a	Garaha	Gashala kufom	Uba
Diclorvos	0.117±0.029	0.072±0.041	0.162±0.053	BDL	0.163±0.014	0.132±0.057
Mevinfos	BDL	BDL	BDL	BDL	BDL	BDL
Diazinon	BDL	0.273±0.132	BDL	BDL	BDL	BDL
Dimethoate	BDL	BDL	BDL	BDL	BDL	0.104±0.062
Diclofenthion	BDL	BDL	0.093±0.017	BDL	BDL	BDL
Phosphamidon	BDL	BDL	BDL	BDL	BDL	BDL
Pirimophos-methyl	BDL	BDL	0.109±0.013	BDL	BDL	BDL
Chlorpyrites	BDL	0.273±0.104	BDL	BDL	0.112±0.034	0.062±0.013
Parathion	BDL	BDL	BDL	BDL	BDL	BDL
Fenthion	BDL	BDL	BDL	BDL	BDL	BDL
Isofenfos	BDL	BDL	BDL	BDL	0.011±0.012	0.103±0.034
Bromophose	BDL	BDL	0.019±0.012	0.023±0.013	BDL	BDL
Ethion	0.012±0.006	BDL	BDL	BDL	0.012±0.007	BDL

Values are Presented in mean ± standard deviation of triplicate determination. BDL, below detection limits of 0.0015-0.004mg kg^{-2}

3. DISCUSSION

Organochlorine pesticides are persistent organic pollutant which have cause worldwide concern as one of the most toxic environmental pollutant (Mshelia *et al*.,2022). They are lipophilic, hydrophobic and are ubiquitous contaminants which have been detected far from their sources of origin because of their persistency in nature, long-range transport steaming from atmospheric exchange (Zhang *et al*., 2007). These pesticides are Aldrin, dieldrin, Endrin, chlordane, dichlorodiphenyl, trichloroethene (DDT), heptachlor, mirextoxaphene, Hexachlorobenzene (HCB) and others, it was

found that organochlorine pesticides residues were detected in bean samples in almost all the area studies this could be due to the fact that they are persistence in nature and the have long-range of transportation. The outcome of the research shows that most of the bean samples were contaminated with organochlorine pesticide at the times the research was conducted. This could be due to the fact that there was a contact between beans with organochlorine pesticides either during cultivation or during storage. it was observed that the concentration of organochlorine pesticide residues was high in bean sample than in the control sample.

Organophosphate are highly potent compounds used majorly as insecticides in the control and storage of food crops. They are harmful and more often involves in acute poisoning than other classy of pesticides (Otitoju and Lewis, 2021). In the presence studies, wide variation was observed from one location to another. The pesticides studied were found to be present in few locations at the time this research was carried out. The mean concentration of the pesticide's residue was found to be high in some location. Going by the results obtained, it indicates that the bean samples have high concentration of pesticides residues at the time the research was carried out,

4. CONCLUSION

The research studied (15) organochlorine and (14) organophosphate pesticides residues in bean samples obtained from some part of Hong Local Government Areas which includes Hong, Pella, Kwakwa'a, Garaha, Gashala Kufom and Uba study areas. Most of the location studied contained one or more pesticide residues with most occurring at high concentration therefore there is need to monitor the use of pesticides in crops production and storage to minimize the risk of contracting some terminal diseases and to make the environment friendly and habitable.

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