



Evaluation of Organochlorine Pesticides in Water, Soil and Plants Samples from Irrigated Farms at Hweng, Barkin Ladi LGA, Plateau State, Nigeria

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Abstract

The research work focused on the evaluation of organochlorine pesticides (OCP) in water, soil and plant samples (tomato, carrot and garden egg) from irrigated farms at Hweng, Foron District in Barkin-Ladi LGA, Plateau State, Nigeria. The water samples were collected from the Yakubu Gowon Dam (as control) and the irrigated farms; while soil and plant samples were collected from the farms. Gas Chromatographic Mass Spectrophotometer (GC MS) was used to determine the concentrations of OCP. The OCPs were δ -Lindane, α -Lindane, γ -Lindane, Heptachlor, Aldrin, Heptachlor-Epoxyde, Endosulfan 1, pp-DDE, Endrin, Endosulfan II, pp-DDD, pp-DDT, Methoxychlor. OCPs ranged from 0.08 to 113.00 mg dm⁻³ in water samples from the wastewater exit point, 0.03 to 59.47 mg dm⁻³ in water samples from the irrigated farms, 0.14 to 97.41 mg kg⁻¹ in soil samples and 0.06 to 50.2 mg kg⁻¹ in the plant samples. All the results in water were higher than their respective controls, except heptachlor- epoxyde (0.08 mg dm⁻³) and DDT (0.10 mg dm⁻³). Also, all the results in the plant samples were higher than the WHO recommended values (0.495 mg kg⁻¹). Aldrin content was predominantly the highest in all the samples, water (113.00 mg dm⁻³), soil (96.41 mg kg⁻¹), and plant samples (50.2 mg kg⁻¹). Correlation coefficient was done and the levels of OCP in all the water, soil and plant samples had strong relationship and significant difference. Therefore, the elevated level of organochlorine pesticides in this study indicates that awareness on the implications of the use of agrochemicals beyond the recommended dosage by farmers should be intensified.

Key words: Gas chromatographic mass spectrophotometer, Hweng, Organochlorine pesticides, plant, soil, water.

Introduction

Due to climate change, water scarcity will affect two third of mankind by 2035, while global food production must increase by at least 50% to feed 9 billion people by 2050. To overcome water scarcity, 15 million m³/day of untreated wastewater is used globally for crop irrigation [1]. The

appropriateness of using treated wastewater for irrigating crops or agricultural products remains a problem among experts and policymakers [2].

Man took into irrigation as a profession to harness and sustain the primary usefulness and the highly priced qualities of the plants in our environment. The practice of wastewater 'urban agriculture'

occurs because of local demands of fresh food products, and the use of wastewater resulted in cases of health problems [3].

Obiofor *et al.* 2018 [4] analyzed the impact of textile wastewater on whole plants of *Corchorous* spp, *Telfairia* spp, *Celosia argentea* and *Amaranthus hybrids* cultivated within farmlands in Ibeshe, Ikorodu, Lagos State and observed that when pollutants in the environment were in very minute amount or in low concentrations, they were not toxic to plants and animals so they had short residence time in the environment; they were described as ‘contaminants’.

Lindane is *hexachlorocyclohexane*. It is an organochlorine chemical that has been used both as an agricultural insecticide and a pharmaceutical *p, p'*- DDT is *dichlorodiphenyltrichlorethane* which is an insecticide used in agriculture. It is currently used for health emergencies only. It has a formula $C_{14}H_9Cl_5$.

Endrin is an organochlorine that is solid, almost odorless and dissolved in a liquid carrier. It belongs to the class of monoterpenoids which are compound containing a chain of two isoprene unit. It is used as a pesticide to control insects, rodents and birds, it is persistent, and residues in soil, water and animal tissues. It has a formula $C_{12}H_8Cl_6O$ *p, p'* - DDD is *dichlorodiphenyldichloroethane*, it is used to kill pest, it is slightly irritating to the skin. Its formula is $C_{14}H_{10}Cl_4$ [5].

p, p' – DDE (*dichlorodiphenyldichloroethylene*) is similar to DDT but has no commercial use. It lost

treatment for lice and scabies. It has other isomers as delta and alpha, there general formula is $C_6H_6Cl_6$. Heptachlor is *heptachlorotetrahydromethioindene*, it is used as an insecticide against termites and soil insects. Its formula is $C_{10}H_5Cl_7$. Heptachlor-epoxide is *hepta-2,3-epoxytetrahydromethioindene* used as a pesticide. Its formula is $C_{10}H_5Cl_7O$ [6].

Aldrin is *hexachloro hexahydrodimethionaphthalen*. It is an insecticide that binds strongly to soil particles and very resistant to leaching into ground water. Its formula is $C_{12}H_8Cl_6$. *Endosulfan* is an off-potent organochlorine insecticide and acaricide, effective against aphids, fruits worms, beetles, leafhoppers, moth larvae and others. Its formula is $C_9H_6Cl_6O_3S$.

hydrogen chloride from DDT and became its impurity. Formula is $C_{14}H_8Cl_4$. *Methoxychlor* is an organochlorine insecticide used in agriculture against flies, mosquitoes, cockroaches and a wide variety of other insects. Its formula is $C_{16}H_{15}Cl_3O_2$ [7].

This source of the polluted water for the irrigation in this study are the rivers flowing into Yakubu Gowon Dam collecting sewage, and water from water treatment plant and agricultural farms.

The aim of this work is to evaluate the organochlorine pesticides concentration in water, soil and selected plant samples from irrigated farms at Hweng, B/Ladi L. G.A, Plateau State, Nigeria.

Materials and Methods

Materials

Sampling Sites

The sampling sites were three (3) different locations, the inlets, the treatment plant waste water discharged point and the irrigated farms at Hweng-

Foron in Barkin Ladi Local Government Area, Plateau State, Nigeria.

Description of the Study Area

On the Jos Plateau, mining activities took place and more steams came up, their minerals are washed into streams and as well as farmlands for agricultural poses. Similarly, the streams passed into the Yakubu Gowon Dam. This dam has its treatment plant at Hweng which treats and supplies drinking water to Jos-South and Barkin Ladi LGAs of Plateau State. The study area was Hweng in B/ Ladi LGA with Yakubu Gowon dam at the eastern side of Bukuru, Jos-South LGA Headquarter and about 12 miles away.

Table 1: Geographical Coordinates of Longitudes and Latitudes of the Sites

<u>Sampling sites</u>	<u>Latitude</u>	<u>Longitude</u>
Rnorth	09 ° 45 57.8 'N	08 ° 58 23.6 'E
Rcentral	09 ° 45 56.4 'N	08 ° 58 22.8 'E
Rsouth	09 ° 45 49.5 'N	08 ° 58 19.6 'E
P ₁	09 ° 45 24.8 'N	08 ° 58 04.7 'E
P ₂	09 ° 45 10.8 'N	08 ° 58 24.7 'E

Key: Rnorth, Rcentral, Rsouth – entrances to the Treatment plant,

P1 – Wastewater exit from the treatment plant,

P2 – Irrigated farm

Sample Collection and Materials

Collection of samples

Water samples were collected from three different sites of the Dam, from the Treatment Plant wastewater discharged point (P₁) and irrigated farm site (P₂).

The soil and plant (tomato, carrot and garden egg) samples were collected in polythene bags; the soil was collected at a depth of 2-3 cm and from several sites of the farm, using a sampler.

Materials

Gas Chromatograph-Mass Spectrometry (GC/MS) (Model: Agilent Technologies 7890A coupled to inertMS 5975C; Germany); Metler analytical balance: 0.0001 – 200g (min: 0.1g) (Model 2004).

Pretreatment of Samples

Sample pretreatment/digestion

GC-MS method of extraction/Determination of organochloride

Pretreatment of Water Samples

A 10.00 cm³ water sample was placed in a 15.00 cm³ centrifuge tube and 2.50 cm³ dichloromethane was added. Tubes were capped and vortexed for 3 minutes before adding 4.00 g MgSO₄, 1.50 g sodium acetate and 1.00 g NaCl. The tubes were vortexed vigorously for 1 minute and centrifuged for 10 minutes at 7000 RPM then 1.00 cm³ of the supernatants were transferred into 2.00 cm³ vials containing 500 mg MgSO₄ each. The vials were shaken and subsequently centrifuged. The extracts were transferred into a PTFE capped vials (obtained from Agilent GC-MS) for GC-MS analysis (USEPA Method 3535a SPE, 2007) [8].

Pretreatment of Solid Samples

A 5.00 g of solid sample was placed in centrifuge tube and 10.00 cm³ acetonitrile was added and the mixture was shaken vigorously. Further, 1.50 g trisodium citrate dihydrate, 4.00 g anhydrous magnesium sulphate, and 1.00 g sodium chloride

were added and the mixture was shaken for 1 minute using vortex mixer and then centrifuged for 10 minutes at 7000 RPM. Afterward, a 7.50 g anhydrous magnesium sulphate, 0.50 g of C18 and 0.125 g PSA were added.

The tube was vortexed and centrifuged. The aliquot of the supernatant was transferred into a glass test tube and allowed to evaporate to nearly dryness under a stream of nitrogen and the residue was re-dissolved in 1.50 cm³ dichloromethane prior to its injection into the GC-MS system (USEPA Method 3535a SPA, 2007) [8].

Data Analysis

The concentration was analyzed statistically to determine the distribution of OCPs. The results were summarized using descriptive statistics (mean and standard deviation).

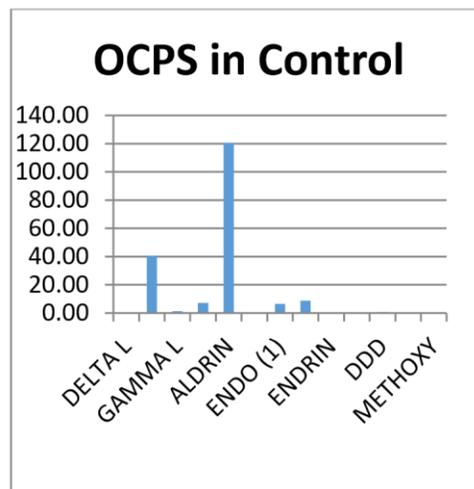
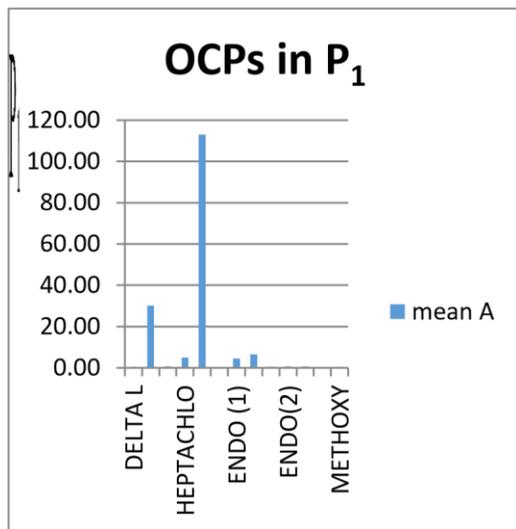
Pearson Correlation Coefficient (SPSS, 2017 version) in the different environmental compartments and assess potential risk to human health and ecosystems.

Results and Discussion

Results

Specifically, organochlorine pesticides (OCPs) of the water samples collected from the dam as the control sample (Figure 2), the water samples from the Treatment Plant waste exit (P₁) (Figure 1).

3.1.1.1 Comparing water samples and control



Figures 1 and 2: Water samples from the Treatment Plant discharged point (P₁) and control

Figure 3 and 4 showed water samples from the irrigated farm (P₂) compared with the control.

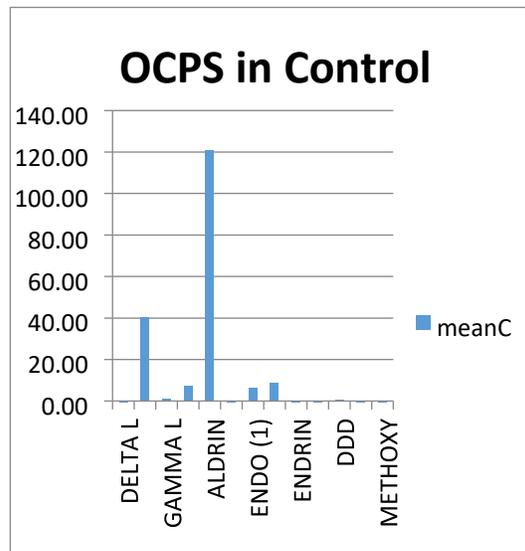
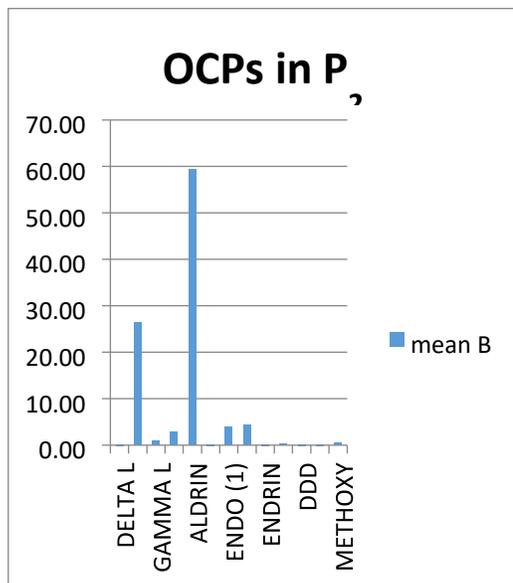


Figure 3 and 4: Water at the farm and control

Table 1 shows the concentration of OCPs in the soil samples (mean ± SD). Comparing Soil samples and Maximum Residual Limit (MRL) all in mg/kg.

Table 1: Concentration of Organochlorine pesticides in soil samples

OCPs (mg/kg)	MRL	OCP in Soil
δ-Lindane	0.01	0.10 ± 0.05
α-Lindane	0.01	36.70 ± 0.9
γ-Lindane	0.01	0.31 ± 0.02
Heptachlor	0.014	0.14 ± 0.05
Aldrin	0.01	97.41 ± 4.78
Heptachlor-Epoxyde.	0.014	0.04 ± 0.00
Endosulfan 1	0.05	2.36 ± 0.35 pp-
DDE	0.05	6.08 ± 2.22 Endrin
0.01	0.66 ± 0.27	Endosulfan II 0.05
0.70 ± 0.35 pp-DDD		0.05 0.58 ±
0.31 pp-DDT	0.05	0.14 ± 0.00
Methoxychlor	0.01	0.53 ± 0.19

Key: (Mean ± SD)

Table 2: Organochlorine Pesticides (OCPs) in Plant sample

OCPs (mg/kg) (Mean ± SD)	WHO	OCP in Plant
δ-Lindane	0.01	0.17 ±
0.04 α-Lindane	0.01	20.3 ±
0.15 γ-Lindane	0.01	0.28 ±
0.13 Heptachlor	0.002	0.06 ±
0.03 Aldrin	0.10	50.2 ± 3.32
Heptachlor-Epoxyde	0.003	0.05 ± 0.06
Endosulfan 1	0.05	0.33 ± 0.06
pp-DDE	0.05	2.75 ± 0.03
Endrin	0.10	1.06 ± 0.06
Endosulfan II	0.05	0.17 ± 0.28
pp-DDD	0.05	0.14 ± 0.60
pp-DDT	0.05	0.14 ± 0.07
Methoxychlor	0.01	0.24 ± 0.16

Pearson Correlation Coefficient of mean concentration of OCPs

Table 3: Correlation of mean concentration of OCPs in P₁ and in P₂

		OCP in P ₁	OCP in P ₂
OCP in P ₁	Pearson Correlation	1	0.986**
	Sig. (2 – tailed).		0.000
	N	13	13
OCP in P ₂	Pearson Correlation	0.986**	1
	Sig. (2-tailed)	0.00	
	N	13	13

** Correlation is significant at the 0.01 level (2-tailed).

Correlation Coefficient of mean concentration of OCPs in Water samples and Control

Table 4: Correlation of mean concentration of OCPs in P₁ and Control.

		OCP in P ₁	Control
OCP in P ₁	Pearson Correlation	1	0.998**
	Sig. (2 – tailed).		0.000
	N	13	13
Control	Pearson Correlation	0.998**	1
	Sig. (2-tailed).	0.00	
	N	13	13

** Correlation is significant at the 0.01 level (2-tailed).

Table 5: Correlation of mean concentration of the OCPs in P₂ and Control.

		OCP in P ₂	Control
OCP in P ₂	Pearson Correlation	1	0.995**
	Sig. (2- tailed).		0.000
	N	13	13
Control	Pearson Correlation	0.995**	1
	Sig. (2-tailed).	0.000	
	N	13	13

** Correlation is significant at the 0.01 level (2-tailed)

Comparative analysis

Figure 1 showed the mean concentration of the organochlorine pesticides (OCPs) as 0.55 ± 0.34 (δ -lindane), 30 ± 6.05 (α -lindane), 0.66 ± 0.24 (γ -lindane), 4.94 ± 3.00 (heptachlor), 113 ± 2.73 (aldrin), 0.08 ± 0.00 (heptachlor-epoxide), 4.47 ± 1.78 (endosulfan I), 6.48 ± 2.18 (p, p'-DDE), 0.46 ± 0.11 (endrin), 0.63 ± 0.11 (endosulfan II), 0.66 ± 0.36 (p, p'-DDD), 0.10 ± 0.03 (p, p'-DDT), and 0.51 ± 0.03 mg dm⁻³ (methoxychlor) respectively in the water samples from discharged point of the treatment plant (P₁) and control (water sample from the dam). The results ranged from 0.08 ± 0.00 to 113 ± 2.73 mg/dm³.

The highest result of 113 ± 2.73 mg/dm³ for aldrin corresponded with the result of [3]. All the values of the organochlorine pesticides in Figure 1 were higher than their respective control values except heptachlor-epoxide (0.08 ± 0.00 mg/dm³), aldrin

(113.00 ± 2.73 mg/dm³), DDT (0.10 ± 0.03 mg/dm³), α -lindane (30.13 ± 6.05 mg/dm³) and γ -lindane (0.06 ± 0.24 mg/dm³) in P₁ which were lower than their corresponding control values of 0.10, 120.00, 0.60, 40.0 and 0.14 mg/dm³ respectively. Possible use of Decis (commercial product) for pest control increased the amount of aldrin in the environment. This may be used in this study area, α -Lindane appeared as the next high value.

The results in Figure 3 ranged from 0.07 ± 0.02 to 59.47 ± 0.39 mg/dm³ showing the mean concentration of OCPs as 0.18 ± 0.10 (δ -lindane), 26.53 ± 1.50 (α -lindane), 1.05 ± 0.12 (γ -lindane), 2.00 ± 0.09 (heptachlor), 59.97 ± 0.95 (aldrin), 0.02 ± 0.01 (heptachlorepoide), 4.05 ± 1.02 (endosulfan I), 4.70 ± 0.02 (p, p'-DDE), 0.24 ± 0.07 (endrin), 0.35 ± 0.17 (endosulfan II), 0.25 ± 0.09 (p, p'-DDD), 0.43 ± 0.02 (p, p'-DDT), and 0.55 ± 0.29

mg dm⁻³ (methoxychlor) respectively. In Figure 3, all the P₂ values were higher than their control values (Figure 4) except α -lindane (26.53 \pm 1.50 mg/dm³), heptachlor (2.0 \pm 0.09 mg/dm³), aldrin (59.97 \pm 0.95 mg/dm³), heptachlor-epoxide (0.02 \pm 0.01 mg/dm³), DDE (4.70 \pm 0.02 mg/dm³) and DDT (0.43 \pm 0.02 mg/dm³) in P₂ which were lower than their corresponding control values of 40.00, 2.70, 120.00, 0.10, 4.80 and 0.60 mg/dm³ respectively. The concentration of OCPs in P₂ were lower than P₁ as the stream goes down.

Table 1 shows that all the results of the concentration of the organochlorines pesticides studies in the soil samples had high values than their EC Maximum Residual Limit (MRL) and so the health risk index should be examined whether it could be harmful or not. The range was from 0.10 \pm 0.05 to 97.41 \pm 4.78 mg kg⁻¹ for δ - lindane and aldrin respectively and the mean concentration of the OCPs was 11.21 \pm 6.20 mg kg⁻¹. The soil sample contained highest concentration of aldrin similar to that of Kumah *et al.* 2023 [8].

The results in Table 2 of the OCPs in plant samples were between 0.06 \pm 0.03 and 50.2 \pm 3.32 mg kg⁻¹, they are all higher than the WHO permissible limit. The mean concentration was 5.84 \pm 0.37 mg kg⁻¹. Aldrin dominated with the value of 50.2 \pm 3.32 mg/kg followed by α -Lindane with 20.30 \pm 0.15 mg/kg. OCPs with aldrin should not be used in this study area or when it is used it should be monitored. Plant samples should be properly washed and well cook before consumption.

Aldrin shows a consistent decrease in the result from water sample (P₁) > soil sample > plant sample. Aldrin might have been applied in this farm in some cases as a seed dresser as in the study done by [9].

Correlations

The Pearson Correlations was used in the study. In Table 3 was the correlation of concentration of OCPs in water samples at the discharged point (P₁) and water samples at the irrigated farm (P₂). The correlation coefficient computed ascertained the relationship and the results turnout to be a strong positive linear relationship with r = 0.986, p-value = 0.00 and sample size (n) = 13. This signifies that the organochlorine pesticide in both samples had strong positive relationship and were statistically significant since the p-value was less than 1% (0.01).

In Table 4 was the correlation of concentration of OCPs in water samples at the discharged point of the treatment plant (P₁) and the water samples from the dam (control). This gave result as r = 0.998, p-value = 0.00 and sample size n = 13. Thus, this signified a strong positive relationship and statistically significant since the p-value was > 0.01.

Table 5 showed the correlation OCP concentrations in the water samples at the irrigated farm (P₂) and water samples from the dam (Control). Which gave result as r = 0.995, p-value = 0.00 and sample size n = 13. Thus, this signified a strong positive relationship and statistically significant since the p-

value was > 0.10 . *Correlation Coefficient in mean concentration of Water and Soil samples.*

Table 6: Correlation of mean concentration of the OCPs in P_2 and soil sample

		OCP in P_2	OCP in Soil
OCP in P_2	Pearson Correlation	1	0.997**
	Sig. (2 – tailed).		0.000
	N	13	13
<hr/>			
OCP in Soil	Pearson Correlation	0.997**	1
	Sig. (2-tailed).	0.000	
	N	13	13

** Correlation is significant at the 0.01 level (2-tailed).

Correlation Coefficient in mean concentration of Soil and Plant samples

Table 7: Correlation of the mean concentration of OCPs in the soil and plant samples.

		OCP in Soil	OCP in Plant
OCP in Soil	Pearson Correlation	1	0.999**
	Sig. (2- tailed).		0.000
	N	13	13
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OCP in Plant	Pearson Correlation	0.999**	1
	Sig. (2-tailed).	0.000	
	N	13	13

** Correlation is significant at the 0.01 level (2-tailed).

Table 8: Summary of all the Correlations

OCPs in		P ₁	P ₂	Control	Soil	Plant
P ₁	PCC	1	0.986**	0.998**	0.993**	0.989**
	Sig. (2-tailed)		0.000	0.000	0.000	0.000
	N	13	13	13	13	13
P ₂	PCC	0.986**	1	0.995**	0.997**	0.997**
	Sig. (2-tailed)	0.000	13	0.000	0.000	0.000
	N	13	13	13	13	13
Control	PCC	0.998**	0.995**	1	0.997**	0.995**
	Sig. (2-tailed)	0.000	0.000	13	0.000	0.000
	N	13	13	13	13	13
Soil	PCC	0.993**	0.997**	0.997**	1	0.999**
	Sig. (2-tailed)	0.000	0.000	0.000		0.000
	N	13	13	13	13	13
Plant	PCC	0.989**	0.997**	0.995**	0.999**	1
	Sig. (2-tailed)	0.000	0.000	0.000	0.000	
	N	13	13	13	13	13

**Correlation is significant at the 0.01 level (2 tailed).

Table 6 showed OCPs in water sample at the irrigated farm (P₂) was correlated with the soil sample. Pearson correlation coefficient computed between the concentration of OCPs in water samples from the irrigated farm (P₂) and the soil samples ascertained the relationship and the result was a strong positive linear relationship with $r = 0.997$, a p -value = 0.000 and a sample size $n = 13$. This signified that the organochlorine pesticides in both samples had a positively strong relationship and was statistically significant since the p -value was less than 1% (0.01).

While OCPs in soil and plant samples were correlated in Table 7. A Pearson correlation coefficient was computed between the concentration of OCPs in soil samples and in plant samples result turnout to be a strong positive linear relationship with $r = 0.999$, a p -value = 0.000 and a

sample size $n = 13$. This signified that the organochlorine pesticides in both samples had a very strong relationship and were statistically significant since the p -value was less than 1% (0.01).

In Table 8 showed the summary of all the correlation of OCPs and the descriptive statistics of P₁, P₂, soil and plant samples where all had a strong positive relationship and statistically significant since their p – values were less than 1% (0.01).

The statistical analysis of the total Pearson's correlation coefficient was presented in the appendix, it showed the correlation coefficient between various concentrations of OCPs factor indicated that methoxy varied with the variation of α - lindane. The data revealed that α -lindane exhibited significant strong positive (correlation

matrix pooled) with organochlorine pesticides such as aldrin, DDD, DDE, DDT and Methoxy.

Conclusion

The result from the study showed the concentration of the organochlorine pesticides (OCPs) in water, soil and plant samples taken around the Yakubu Gowon Water Treatment Plant at Hweng, Foron in Barkin Ladi LGA, Plateau State. All the concentration of OCPs obtained from the water sample was above the control value except in the case of heptachlor-epoxide, aldrin, α -, γ -lindane and p, p' - DDT in P₁ also α -lindane, heptachlor, heptachlor-epoxide, aldrin, DDE and DDT in P₂, all the concentration of OCPs in soil sample was higher than their respective Maximum Retention Limit (MRL) and all the OCPs concentration in plant sample was also above the WHO permissible limit except p, p' - DDD and p, p' - DDT. In the 13 OCPs studied, aldrin had the highest content in all the samples.

Pearson correlation coefficient was used to quantitatively evaluate the samples and all the results gave a very strong positive relationship and statistically significant.

Therefore,

The evaluation of OCPs in water, soil and plant samples taken, showed all the water samples were contaminated with OCPs, possibly indicating that the chemicals used in the treatment of water in the Treatment Plant could be the source of the OCPs or farmers might also be using OCPs as pesticides to eradicate insect, termites or pests from their farms

leading to high concentration of OCPs in the soil and plant samples.

Recommendation

The exposure to OCPs should be reduced so as to minimize the health hazard that affects the environment and human. There is a definite need to find similar, safer, effective and cheaper alternatives to OCPs when it comes to pesticides. There are still people who are not aware of the consequences of the use of OCPs therefore health education should be given to them. Proper washing of the plants and cooking it well can help eliminate most of these OCPs like aldrin, DDE and DDD, although, the probability of it to cause cancer is low. Possibly, management of the Treatment Plant can be advised on the use of minimal amount of chemicals in treating water, this also is important.

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