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Contamination Levels of Organochlorine and Organophosphorous Pesticide Residues in Water and Sediment from River Owena, Nigeria

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Authors' contributions

This work was carried out in collaboration among all authors. Authors AFA, YAA and AS designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript and managed literature searches. Authors YAA and AS managed the analyses of the study and literature searches. All authors read and approved the final manuscript.

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ABSTRACT

River Owena functions as the source of water for the Owena dam which supplies drinking water to the communities in the region, the river also plays an important economic role in irrigation and fish farming in the region. But there has been an indiscriminate used of pesticide because residents of the study area are not well orientated concerning the negative impact of pesticide in the environment. Analysis of organochlorine and organophosphorous pesticide residues was carried out in water and sediment from river Owena, the objective of the research is to determine the contamination levels of organochlorine and organophosphorous pesticide in the water and sediment of river Owena. Analysis was carried out using gas chromatography coupled with electron capture detector in order to identify and quantify the concentration of organochlorine and organophosphorous pesticide in the water and sediment samples from river Owena. Analysis

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revealed the contamination of water and sediment with organophosphorous pesticide residues at different contamination levels in both dry and rainy season, while a low levels of organochlorine pesticide residues only during the dry season. The TOPP for sediment samples ranges from 1.045mg/L during the dry season and 2.279mg/L during the rainy season, while the TOPP for water samples ranges from 7.163mg/L during the dry season to 7.83mg/L during the rainy season. The TOCP values ranges from 1.288mg/L to 0.015mg/L for sediment and water samples respectively during the dry season, while organochlorine pesticides were not detected during the rainy season. The occurrence and seasonal variation of organophosphorous pesticide residues in river Owena can be attributed to the intense agricultural and urban activities in the vicinity of river Owena.

Keywords: Organochlorine; organophosphorous; contamination; sediment; residue.

1. INTRODUCTION

Pesticides are organic artificially synthesized, toxic bioaccumulative agents which are Chemicals or mixtures of chemicals that are used for killing, repelling, mitigating or reducing pest damage [1-5]. Pesticides are one of the many components used in modern agricultural practices as a result, pesticides play an important role in agricultural development and in pest control worldwide but the indiscriminate use of pesticides to fight pests and improve agricultural production constitutes a risk for water quality, aquatic organisms and agricultural food products. Thus, pesticide residues have been detected by monitoring surface waters, ground waters, sediments and agricultural food products [6-9]. Pesticide residues are the deposits of pesticide active ingredient, its metabolites or breakdown products present in some component of the environment after its application, spillage or dumping [10]. Pesticide residue analysis provides a measure of the nature and levels of pesticide contamination within the environment and of its persistence and fate in the environment [11]. Pesticides used in agriculture, public health and domestic pest control can enter into the environment in a number of ways depending upon the method and proficiency of application, as a result of accidents or through the unauthorized dumping of unwanted pesticide products or their containers. When pesticides are applied on fields, gardens, parks, and other places a percentage of the chemicals end up as runoff. Rain washes some of the pesticides into streets gutters, where the pesticide contaminated water goes through storm drains and pipes and eventually flows into nearby creeks, and rivers. Some of the pesticides also end up in groundwater systems by leaching down through the soil. Small amounts of pesticides volatize into the atmosphere, and then later fall back to land as precipitation. As a result of all these pathways, pesticides are widely found in rivers, streams, lakes, and even in drinking water [12]. Pesticides are of environmental concern in streams in both the water column and sediment. Those pesticides that are more hydrophobic tend to be detected more frequently in sediment; thus, measuring pesticides in sediment is important for tracking their fate in the environment and evaluating for potential toxicity [13,14]. The amount of pesticides in water varies greatly, both geographically and seasonally, based on pesticide usage patterns. In agricultural areas, herbicides are the most frequently found type of pesticide in streams and groundwater. In urban areas, there is a greater prevalence of insecticides in streams than in agricultural areas. Pesticide contamination levels vary based on variation in rainfall, also seasonally based on agricultural practices [15-17].

Organochlorines are chlorinated hydrocarbon, they are among the oldest and most toxic synthetic insecticides, they are neurotoxic and some organochlorine compounds are carcinogenic leading health related problems. They are synthetic and anthropogenic environmental contaminant, the production and use of organochlorine pesticide have been restricted or banned in most industrialize world but considerable amount are still found in the ecosystem due to the persistent and lipophilic nature of organochlorine pesticides [18-20]. Organophosphorous pesticides are synthetic organic compounds which are esters, amide or thiol derivatives of phosphoric, phosphonic, phosphorothioic and phosphonothioic acid. Compared to the mostly banned in U.S. and Europe organochlorine pesticides, organophosphorous pesticides are less persistent in the environment, organophosphates were the insecticides used on a large scale to replace the organochlorines. Because of their unstable chemical structure they disintegrate into harmless radicals within days of application and do not persist in body tissues or the environment.

These features justify their application in the agricultural and veterinary practices of the modern world [21,22]. The River Owena play an important economy role in water supply to the Owena dam, irrigation of farmlands and fish farming in the region, there has been an indiscriminate use of pesticide in the region due to the lack of orientation concerning the negative impact of pesticide to the environment. The aim and objective of this research is to provide information on the contamination levels of organochlorine and organophosphorous pesticide in the water and sediment of river Owena.

2. MATERIALS AND METHODS

2.1 The Study Area

River Owena is located on latitude $6^0$33'55.3" and longitude $5^0$8'24.83" in Nigeria. The estimate terrain elevation above sea level is 10 meters. River Owen is a major source of domestic water supply to the people of Akure and the neighboring towns. The Owen dam, which is a major dam in Ondo State, is constructed as a result of damming the river Owena. Fishing and farming and domestic activities takes place in the vicinity of the river Owena rendering it prone to environmental contamination.

2.2 Sampling and Pre-treatment of Samples

Water samples were collected in a 2L glass bottles, while sediment samples were collected using a soil hugger. Water and sediment samples were during the dry season (February 2015) and rainy season (June 2015) from the study area. The water samples were subjected to liquid-liquid extraction while the sediment samples were air dried for two weeks and pulverized using laboratory mortar and pestle. It was later sieved using a 2 mm mesh size sieve, and then it was subjected to solid-liquid extraction. All chemicals used were of analytical grade.

2.2.1 Liquid-liquid extraction for water samples

The water samples were subjected to liquid-liquid extraction using Method 3510 as described by USEPA [23]. 50mL of dichloromethane was introduced into a separating funnel containing 100mL of the water sample and shaken vigorously for 5 minutes. The sample was allowed to settle for 30 minutes to facilitate effective separation of the organic and aqueous phases. After separation, the organic layer was filtered into a 250mL volumetric flask through anhydrous sodium sulphate (Na_2SO_4) that has been prewashed with dichloromethane. The extraction was repeated twice using 50mL of dichloromethane. The extracts were later combined to make a whole. The extracts were concentrated to 5mL using a rotary evaporator, during concentration the solvent is exchanged with n-hexane. The level of organochlorine and organophosphorous pesticide residues in the water samples were determined using gas chromatography coupled with Electron capture detector (GC-ECD).

2.2.2 Solid-liquid extraction for sediment samples

The sediment samples were subjected to solid-liquid extraction using Method 3550C as described by USEPA [24]. A mixture of 20g of sediment samples and 20g of anhydrous sodium sulphate (Na_2SO_4) was thoroughly mixed with a mixture of 50mL acetone and n-hexane (1:1 v/v). The mixture was sonicated for 30 minutes in a high frequency ultrasonic bath at 60^0C, the organic extract was decanted. The extraction process was repeated twice using 50mL of a mixture of acetone and n-hexane (1:1 v/v). The extracts were later combined to make a whole. The extracts were concentrated using a rotary evaporator.

2.2.3 Clean up procedure for sediment samples

The sediment samples were subjected to a cleanup process in order to avoid interferences. The extracts from the sediment samples were clean up using a column packed with 2g of activated silica gel and 2g of anhydrous sodium sulphate (Na_2SO_4). Prior to the clean up, the column was conditioned with 20mL of n-hexane. The extract was introduced into the column and eluted using a mixture of n-hexane and diethyl ether (1:1 v/v).The elute was concentrated to 5mL using a rotary evaporator, during concentration the solvent is exchanged with n-hexane.

2.2.4 Gas chromatographic conditions

The following instrumentals conditions were maintained. Gas pressure was 60 psi and injector temperature was 220^oC, GC column temperature was 190^oC, detector temperature was 270^oC, the carrier gas was nitrogen (at 30 ml/min), column length 200 cm, id 2 mm, the glass spiral column packed with 1.5% 0V - 17 and 1.95% 0V-210 on chromosorb WHP 80/100 mesh. There were no peaks when solvents and blanks were chromatographed, before the samples were analyzed under the same condition. Known standards, were also chromatographed, the retention time were used to identify the compounds present in the samples.

2.2.5 Data analysis

Data generated on the concentrations of each pesticide was analyzed for spatial variation using T-test statistical analysis with SPSS package version 20.

3. RESULTS AND DISCUSSION

The average use of pesticides in the region on an annual basis in liters varies ranging from Gamma 20 (939.876), Kokotine (298,885), Copper sulphate (2,694), Caicobre sandoz (551), Perenox (789) and others [25]. Organophosphorous pesticides are the commonly available pesticides in the region due to the banned on organochlorine pesticides as a result of its persistency in the environment. The residents of the study areas are not well educated concerning the use of organophosphorous pesticide as a result of this, in order to control the rapid growth of weeds during the rainy season there is an increase in the indiscriminate use of organophosphorous pesticides in the active ingredient form of 480g/L Glyphosateisopropylamine salt [26]. The results of the findings in this research are presented in tables and figures, also the chromatogram of blank and standards for organophosphorous and organochlorine pesticides analyzed are presented in figures.

Table 1 and Fig. 1 shows the mean value of the concentration of organophosphorous pesticide residues in sediment samples from river Owena, from the 17 organophosphorous pesticide residues analyzed in the sediment samples from the river during the dry and rainy season of 2015. Methyl parathion (1.01±0.007 mg/L) and Pirimiphos-methyl (0.035±0.001 mg/L) were detected during the dry season with a with a corresponding TOPP value of 1.045±0.004 mg/L, while Diazinon (0.382±0.001 mg/L), Phosphamidon (0.176±0.001mg/L), Dichlofenthion (0.158±0.00mg/L), Methyl parathion (0.79±0.003mg/L), Pirimiphos-methyl (0.409±0.004mg/L) and Parathion (0.364±0.003mg/L) were detected during the rainy season with a corresponding TOPP value ranging from N.D-2.279±0.39mg/L. The other organophosphorous pesticides in sediment samples were not detected during the dry and rainy season of 2015.

Table 1. T-test table for the concentration (mg/L) of organophosphorous pesticide residues in sediment samples

Organophosphorous	Dry season	Rainy season	P-value
Dichlorvos	0.00±0.00[a]	0.00±0.00[a]	-
Mevinfos	0.00±0.00[a]	0.00±0.00[a]	-
Carbaryl	0.00±0.00[a]	0.00±0.00[a]	-
Dimethoate	0.00±0.00[a]	0.00±0.00[a]	-
Diazinon	0.00±0.00	0.382±0.001	0.000
Pirimicarb	0.00±0.00[a]	0.00±0.00[a]	-
Phosphamidon	0.00±0.00	0.176±0.001	0.000
Dichlofenthion	0.00±0.00[a]	0.158±0.00[a]	-
Methyl Parathion	1.01±0.007	0.79±0.003	0.001
Pirimiphos-methyl	0.035±0.001	0.409±0.004	0.000
Fenthion	0.00±0.00[a]	0.00±0.00[a]	-
Parathion	0.00±0.00	0.364±0.003	0.000
Isofenphos	0.00±0.00[a]	0.00±0.00[a]	-
Bromophos-ethyl	0.00±0.00[a]	0.00±0.00[a]	-
Ethion	0.00±0.00[a]	0.00±0.00[a]	-
Carbofenothion	0.00±0.00[a]	0.00±0.00[a]	-
Malathion	0.00±0.00[a]	0.00±0.00[a]	-
TOPP	1.045±0.004	2.279±0.39	0.006

Values ± S.D with superscript a are not significantly different. P<0.05 is significant
TOPP= total organophosphorous pesticide, OPP= organophosphorous pesticide, S.D= standard deviation

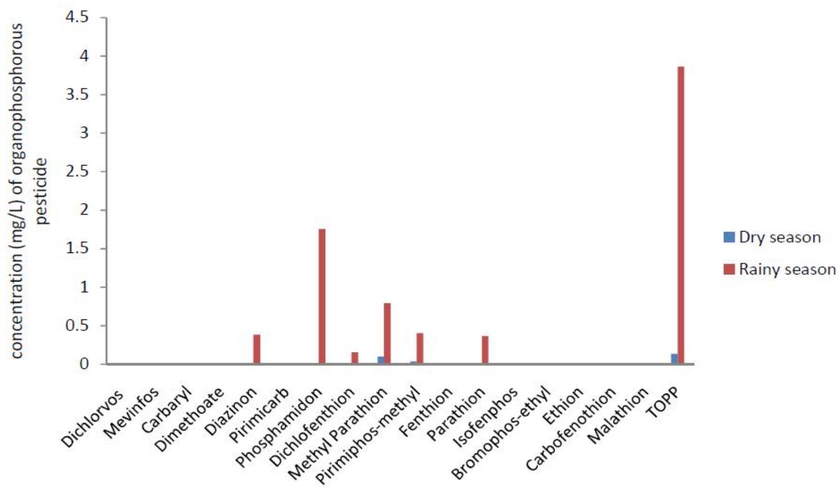

Fig. 1. Showing the seasonal variation of organophosphorous pesticides in sediment samples

Table 2 and Fig. 2 shows the mean value of the concentration of organophosphorous pesticide residues in water samples from river Owena during the dry and rainy season of 2015. From the 17 organophosphorous pesticide residues analyzed from the water samples in river Owena, Carbaryl (0.214±0.004mg/L), Diazinon (0.125±0.004mg/L), Phosphamidon (2.210±0.014mg/L), Dichlofenthion (0.055±0.035mg/L), Methyl Parathion (0.675±0.005mg/L), Pirimiphos-methyl (0.53±0.03mg/L), Parathion (0.301±0.001mg/L), Bromophos ethyl (0.934±0.004mg/L), Ethion (1.447±0.003mg/L), Carbofenothion (0.672±0.001mg/L) were detected during the dry season with a corresponding TOPP value of 7.163±0.001mg/L, while Diazinon (0.443±0.002mg/L), Phosphamidon (2.022±0.016 mg/L), Dichlofenthion (0.354±0.005mg/L), Methyl parathion (1.226±0.02mg/L), Pirimiphos-methyl (0.705±0.006mg/L), Parathion (0.571±0.001mg/L), Bromophos ethyl (0.591±0.001mg/L), Ethion (0.935±0.006 mg/L) and Carbofenothion (0.983±0.003mg/L) were detected during the rainy season with a corresponding TOPP value of 7.83±0.014mg/L. The other organophosphorous pesticides were not detected in the water samples.

Table 2. T-test table for the concentration (mg/L) of organophosphorous pesticide residues in water samples

Organophosphorous	Dry season	Rainy season	P-value
Dichlorvos	0.00±0.00[a]	0.00±0.00[a]	-
Mevinfos	0.00±0.00[a]	0.00±0.00[a]	-
Carbaryl	0.214±0.004	0.00±0.00	0.00
Dimethoate	0.00±0.00[a]	0.00±0.00[a]	-
Diazinon	0.125±0.004	0.443±0.002	0.000
Pirimicarb	0.00±0.00[a]	0.00±0.00[a]	-
Phosphamidon	2.210±0.014	2.022±0.016	0.006
Dichlofenthion	0.055±0.035	0.354±0.005	0.007
Methyl Parathion	0.675±0.005	1.226±0.02	0.001
Pirimiphos-methyl	0.53±0.03	0.705±0.006	0.014
Fenthion	0.00±0.00	0.00±0.00	0.000
Parathion	0.301±0.001	0.571±0.001	0.000
Isofenphos	0.00±0.00	0.00±0.00	0.000
Bromophos-ethyl	0.934±0.004	0.591±0.001	0.005
Ethion	1.447±0.003	0.935±0.006	0.000
Carbofenothion	0.672±0.001	0.983±0.003	0.000
Malathion	0.00±0.00[a]	0.00±0.00[a]	-
TOPP	7.163±0.001	7.83±0.014	0.000

Values ± SD with superscript a are not significantly different. P<0.05 is significant;
TOPP= total organophosphorous pesticide, OPP= organophosphorous pesticide, S.D= standard deviation

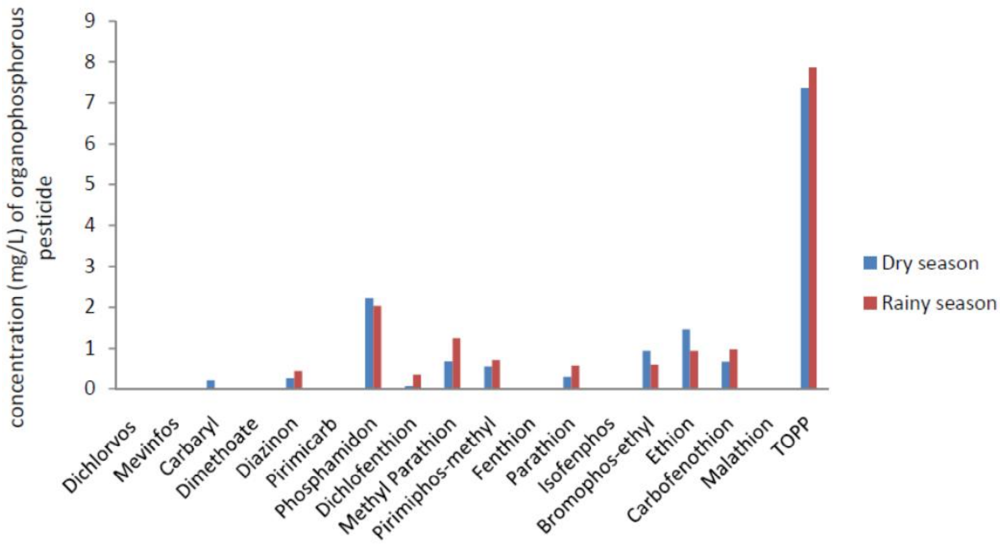

Fig. 2. Showing the seasonal variation of organophosphorous pesticides in water samples

Tables 3 and 4 shows the mean values of the concentration of organochlorine pesticide residues analyzed in the water and sediment samples respectively from river Owena during the dry and rainy season of 2015. From the 12 organochlorine pesticide residue analyzed in the water sample during the dry season of 2015 only Endosulfan II was detected with a concentration of 0.015±0.002 mg/L, while organochlorine pesticides residues were not detected during the rainy season of 2015. From the 12 organochlorine pesticide residues analyzed in the sediment samples from river Owena Lindane (0.163±0.002mg/L), Chlorothalonil (0.362±0.001mg/L), Dieldrin (0.159±0.002mg/L), Endosulfan II (0.32±0.001mg/L) and P-P DDT (0.284±0.002mg/L) were detected in the sediment samples from river Owena during the dry season of 2015 with a corresponding TOCP value of 1.288±0.001 mg/L. Organochlorine pesticide residues were not detected in the sediment samples from river Owena during the rainy season of 2015.

Fig. 3 shows a chromatogram of the blank solution with no peak for organochlorine or organophosphorous pesticide but Figs. 4 and 5 show a chromatogram with standards for the analyzed organophosphorous and organochlorine pesticides residues.

Table 3. T-test table for the concentration (mg/L) of organochlorine pesticide residues in water samples

Organochlorines	Dry Season	Rainy Season	P-value
Alpha BHC	0.00±0.00[a]	0.00±0.00[a]	-
Beta BHC	0.00±0.00[a]	0.00±0.00[a]	-
Lindane	0.00±0.00[a]	0.00±0.00[a]	-
Chlorothalonil	0.00±0.00[a]	0.00±0.00[a]	-
Delta Lindane	0.00±0.00[a]	0.00±0.00[a]	-
Heptachlor	0.00±0.00[a]	0.00±0.00[a]	-
Aldrin	0.00±0.00[a]	0.00±0.00[a]	-
Heptachlor epoxide	0.00±0.00[a]	0.00±0.00[a]	-
Dieldrin	0.00±0.00[a]	0.00±0.00[a]	-
Endosulfan II	0.015±0.002	0.00±0.00	0.008
P,P'-DDD	0.00±0.00[a]	0.00±0.00[a]	-
P-P' DDT	0.00±0.00[a]	0.00±0.00[a]	-
TOCP	0.015±0.002	0.00±0.00	0.004

Values ± S.D with superscript a are not significantly different. P<0.05 is significant
TOCP= total organochlorine pesticide, OPP= organophosphorous pesticide, S.D= standard deviation

Table 4. T-test table for the concentration (mg/L) of organochlorine pesticide residues in organochlorines sediment samples

Organochlorines	Dry season	Rainy season	P-value
Alpha-BHC	0.00±0.00[a]	0.00±0.00[a]	-
Beta BHC	0.00±0.00[a]	0.00±0.00[a]	-
Lindane	0.163±0.002	0.00±0.00	0.000
Chlorothalonil	0.362±0.001	0.00±0.00	0.000
Heptachlor	0.00±0.00[a]	0.00±0.00[a]	-
Aldrin	0.00±0.00[a]	0.00±0.00[a]	-
Heptachloro epoxide	0.00±0.00[a]	0.00±0.00[a]	-
Dieldrin	0.159±0.002	0.00±0.00	0.000
Endosulfan II	0.32±0.001	0.00±0.00	0.000
P-P'DDD	0.00±0.00[a]	0.00±0.00[a]	-
P-P DDT	0.284±0.002	0.00±0.00	0.000
TOCP	1.288±0.001	0.00±0.00	0.000

Values ± S.D with superscript a are not significantly different. $P<0.05$ is significant
TOCP= total organochlorine pesticide, OPP= organophosphorous pesticide, S.D= standard deviation

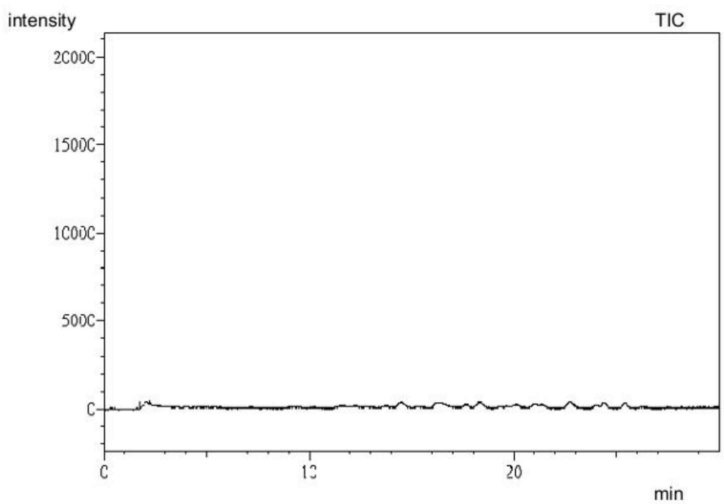

Fig. 3. A chromatogram of the blank solution

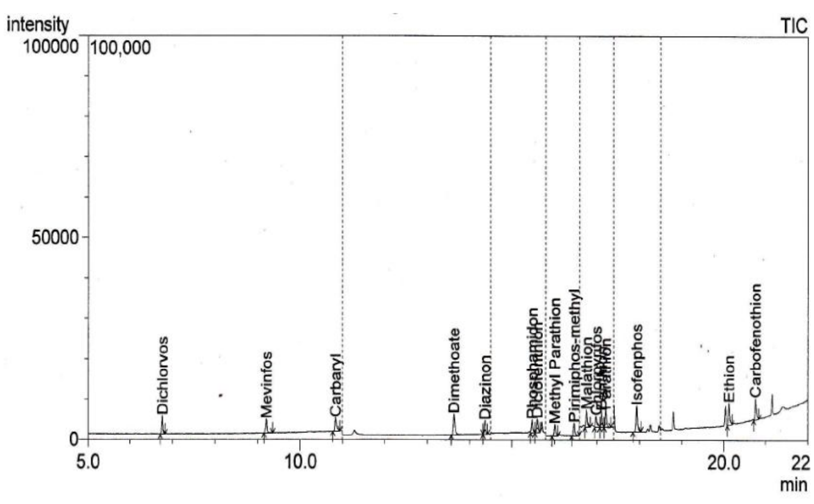

Fig. 4. A chromatogram of standard for the analyzed organophosphorous pesticide residues

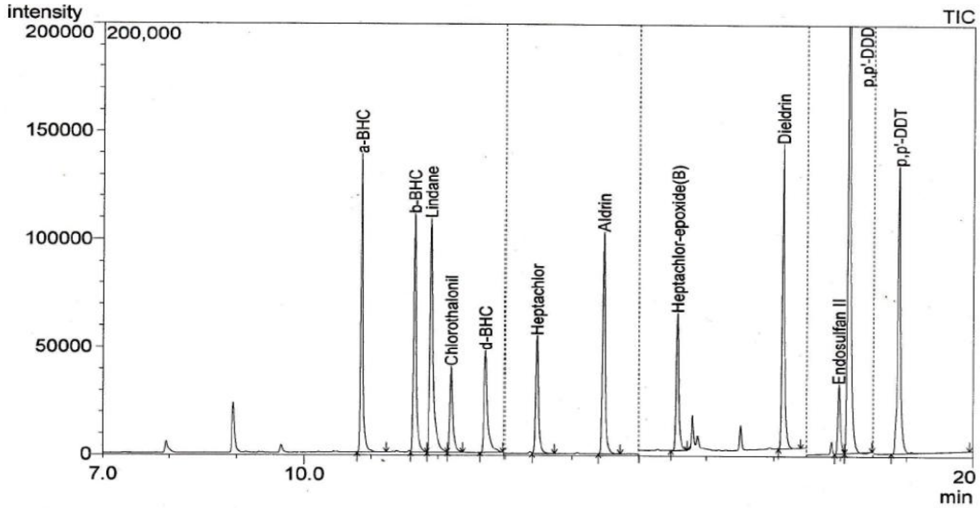

Fig. 5. A chromatogram of standard for the analyzed organochlorine pesticide residues

Table 5. Organophosphorous and organochlorine and retention times

Organophosphorous	Retention time	Organochlorine	Retention time
Dichloros	6.765	a-BHC	10.851
Mevinfos	9.207	b-BHC	11.645
Carbaryl	10.836	Lindane	11.890
Dimethoate	13.630	Chorothalonil	12.194
Diazinon	14.362	d-BHC	12.709
Phosphamidon	15.479	Heptachlor	13.473
Diclofenthion	15.600	Aldrin	14.469
Methyl Parathion	16.010	Heptachlor-epoxide (B)	15.569
Primiphos-methyl	16.465	Dieldrin	17.135
Malathion	16.755	Endosulfan (II)	17.977
Chlorpyrifos	16.980	p,p'-DDD	18.117
Fenthion	17.111	p,p'-DDT	18.878
Parathion	17.205		
Isofenphos	17.938		
Bromophos-ethyl	18.467		
Ethion	20.124		
Carbofenothion	20.755		

The TOPP value (7.83±0.014mg/L) of the water samples during the rainy season was higher than the TOPP value (7.163±0.001mg/L) of the water samples during the dry season, this variation occurs as a result of the increase in the use of organophosphorous pesticide to control or inhibit the fast growth of weeds in farmlands and residential homes during rainy season, also some of the fishermen made use of organophosphorous pesticide to kill fishes [27]. The sediment samples have a higher value of TOPP (2.279±0.39mg/L) during the rainy season and a lower value of TOPP (1.045±0.04mg/L) during the dry season, the higher value of TOPP during the rainy season compared to the lower value of TOPP during the dry season was as a result of the deposition of organophosphorous pesticide residue deposit on the river Owena sediment from water runoff that has been contaminated with organophosphorous pesticide residues. Water runoff from agricultural farmlands after heavy rainfall plays a great role in the higher value TOPP during the rainy season when samples are collected after rainfall compared to a lower value of TOPP during dry season where there is no rainfall [28]. The TOCP values of 1.288±0.001mg/L and 0.015 ± 0.002mg/L in sediment and water samples respectively was only obtained during the dry season as organochlorine was not detected in the water and sediment samples during the rainy samples. The absence of organochlorine pesticide residues in water and sediment samples during the rainy season may be as a result of dilution effect due to the increase in the volume of water body caused by heavy rainfall and also the adsorption of pesticide residue to soil particles during the dry season [29]. It was observed that the concentration of organophosphorous pesticide residues were higher than the concentration of organochlorine pesticide residues in the water and sediment samples from river Owena, this is due to the banned on organochlorine pesticides as a result of the persistence and environmental effect of organochlorine pesticides. Producers and users of organochlorine pesticides are force to switch to organophosphorous pesticides, thereby rendering organophosphorous pesticides the available pesticide product in Nigeria [30-33]. This study has revealed that organochlorine pesticides has been successful replacement with organophosphorous pesticide because of the banned on organochlorine pesticides, but the indiscriminate use of organophosphorous pesticide can lead to contamination of the river Owena thereby impacting negatively on the ecosystem.

4. CONCLUSION

It is important to monitor the contamination levels of pesticides in fresh water environment in order to prevent health problems that are related to pesticide contamination. This study shows the river Owena is contaminated with some levels of organophosphorous pesticide, which is the common pesticide used in the region. While the concentration of organochlorine pesticide is very low, this is attributed to the banned in the use of organochlorine pesticide. The concentration of organochlorine pesticide detected may have resulted from the bioaccumulation of organochlorine pesticide that has been used a long time ago in the region.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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