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EXECRABLE CONDITION OF URBAN RIVERS WITH REFERENCES TO ORGANOCHLORINE PESTICIDE AND POLYCHLOROBIPHENYL IN OGBA RIVER, BENIN CITY NIGERIA

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ABSTRACT: *The levels of organochlorine pesticide (OCPs) and polychlorobiphenyl (PCBs) residues in water and sediments of Ogba River Benin, Nigeria were investigated. The water and sediments samples were collected on monthly basis from April 2017 to March 2018 and analyzed using a gas chromatograph fitted with an election capture detector. The surface water revealed the following pesticides and their ranges in (ng/l) 8.15 – 19.90 (HCH), 24.77 – 44.90 (chlordane), 7.82-20.00 (DDT), 35.87- 98.80 (endrin), 8.90 -33.60 (dieldrin), 36.55 -77.40 (endosulfan) and 150.00 – 547.00(PCBs). Seasonally, the persistent organic pollutants in the surface water were; wet season; (in ng/l), PCBs (415 ± 5.90) endrin (82 ± 2.30), endosulfan (62 ± 2.10) , chlordane (32 ± 1.90) , dieldrin (24 ± 1.12), DDTs (14.80 ± 1.40) , HCHs (15.40 ± 0.60); dry season (in ng/l),), PCBs (398 ± 7.10) endrin (72 ± 1.40), endosulfan (46 ± 1.40) , chlordane (23 ± 1.20) , dieldrin (18 ± 0.32), DDTs (11.20 ± 0.70) , HCHs (12.10 ± 0.20). The OCPs in the bottom sediment had the following concentrations; (µg/kg) 7.62 – 39.99 (HCH), 13.62 – 149.00 (chlordane), 47.36 – 58.55 (DDT), 16.32 -112.20 (endrin), 54.93-99.20 (diedrin), 4.19 – 97.62 (endosulfan) and 440.00 – 701. 00 (PCBs) The sediments seasonal variation were; wet (in µg/kg); PCBs (645 ± 8.40), chlordane (132 ± 3.40), endosulfan (92 ± 0.90), dieldrin (86 ± 2.62), DDT(51± 1.50), HCHs (28 ± 0.70); dry PCBs (580 ± 9.10), chlordane (13 ± 1.20), endosulfan (81 ± 0.50), dieldrin (68 ± 1.02), DDT(45±0.30), HCHs (21 ± 0.20).The OCPs and PCBs were below the values established by the regulatory bodies. Though, the pesticides had low concentration in the River at that point in time it has little or no effect on the biota. Although, if not monitored over the years, it can cause harm to the environment, bioaccumulate and biomagnifies along the food chain.*

KEYWORDS: Organochlorine Pesticides, Polychlorobiphenyl, Water, Sediments, Ogba Rivers.

INTRODUCTION

Organochlorine pesticides and polychlorinated biphenyls (PCBs) are chemicals found widely in the air, aquatic organism, wildlife and humans, but, fortunately due to legislative action, levels of many are now slowly declining (UNEP, 2006). Direct application to farm to eliminate pest, accidental spillages and poor storage methods have been blamed for organochlorine pesticides route to the environment and to the food chain (Ritter *et al*., 1995). Organochlorine and PCBs can enter the body through many routes, such as via polluted air, water or through the skin in countries where they are in used. However, in developed countries where they are no longer used, it is through contaminated food, and the most

contaminated foods are the oily fish and foods of animal origin such as fatty meats and dairy products (EU, 2006).

As soon as in the body these contaminants have a tendency to accumulate in fatty tissues and can stay there for years (Covaci *et al*., 2002a), and can circulate in the fatty area of the blood serum (Thomas *et al*., 2006), and have been reported in the amniotic fluid, human placentas, fetuses, and umbilical cord (Foster *et al*., 20002).

Their toxic effect includes reproductive failures (Bouman, 2004), immune system malfunction (Kolpin *et al*., 1998), endocrine disruption (Ize – Iyamu *et al.*, 2007) and breast cancers (Garabrant *et al*., 1992). Previous studies have shown that organochlorine has the ability to block potassium influx across membranes of nerve fibres, thereby causing increased negative after-potentials. It also induces mixed function oxidize system thereby, alters the metabolism of xenobiotics and steroid hormones (Colborn and Smolen, 1996).

Presently, unrestricted and illegal use of OCPs for crop protection and household disinfection is still a common practice in Nigeria, and is at the peak in the Niger delta region. However, limited data are available on the occurrence and distribution of OCPs in surface sediments of Rivers in this region (Ezemonye *et al.*, 2008). Ize-Iyamu *et al.* (2007) reported that there is no research on the levels of organochlorine or polychlorine biphenyls in Rivers in Edo state, Nigeria. This study being the first of the kind, will investigate the presence of organochlorines and polychlorobiphenyls in Ogba River, Nigeria

Ogba River is in Benin City, South – south Nigeria, and is located at the Southwest region of the outskirt of Benin City in Edo State. It lies between Latitude 6^0 16'8'' N and 6^0 19'12'' N (Fig.1), and its upper part stretches from Ogbe Ibuya area to Ogba Community. The River is about 42km long and takes its source at Ekewan and flows in a Southeast direction through Ogba village and empties to Osiomo River, which in turn empties into Benin River, then into the Atlantic Ocean.

In the Ogba River watershed are Ogba Water Works, wood processing industry, an open prison, farms, a zoological garden and a Scout Camp. The communities in the area depend on the River for their farming and fishing activities, as well as for domestic activities. Other human activities in the area are swimming, washing of clothes, cars and motor bikes and religious activities (Anyanwu, 2012).

MATERIALS AND METHODS

Sampling Stations

Four sampling stations were created along the main course of the river from upstream to downstream with a distance of more than 500metres between the stations (Figure 1). The entire sampled stretch was fresh water and the selection of the sampling stations was governed primarily by the human activities going on in the area. Three of the stations (2, 3 and 4) witnessed the most human effects; sewage disposal, agricultural activites, bathing, laundry, idol sacrifices, water baptism and washing of cars and motorbikes. Station 1 witnessed no activity throughout the study period

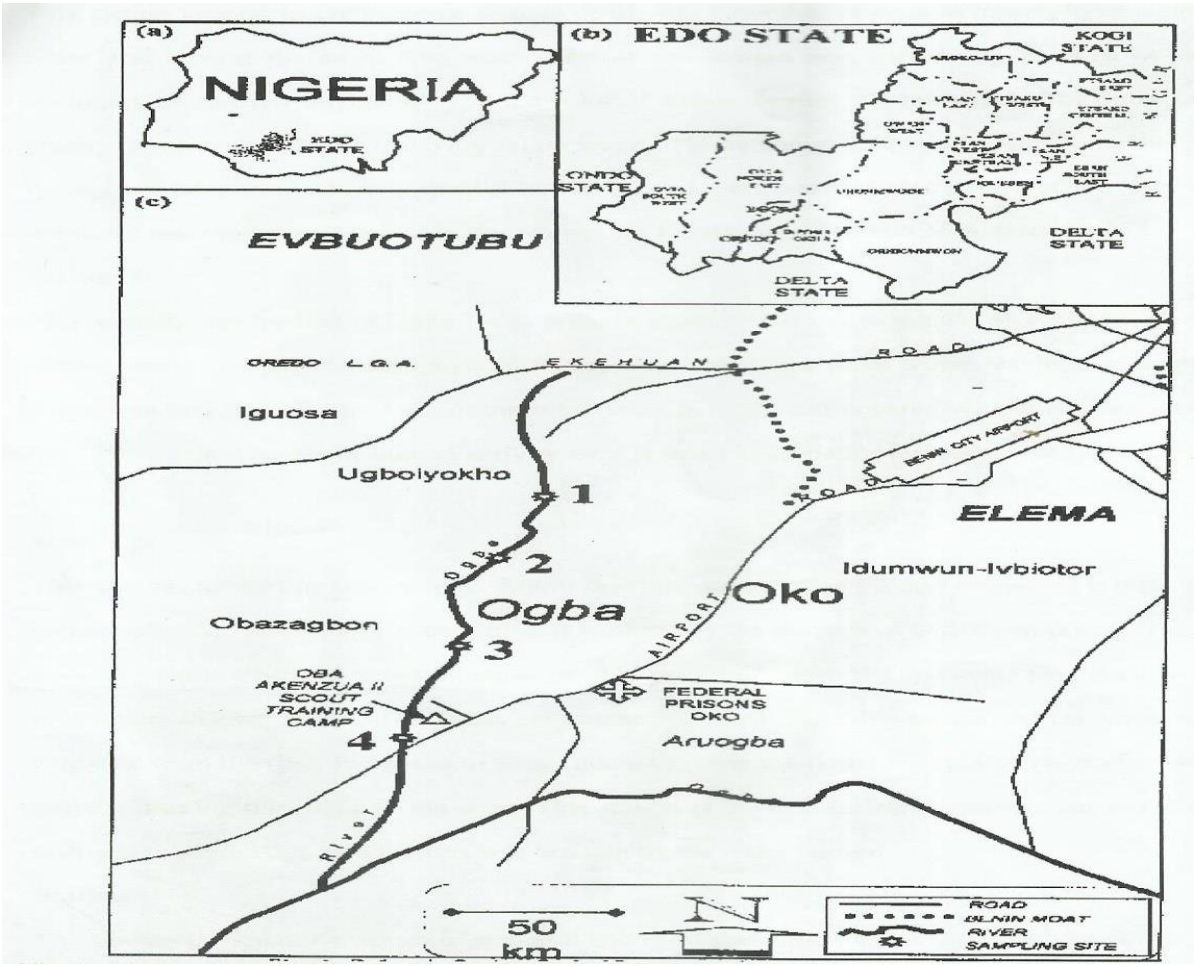

Figure 1: Map of Benin City Showing the Sampling Stations in Ogba River

Source: Ministry of Lands and Survey, Benin City, Edi State.

Station 1: This station located in Ogba Ibuya area, and is open with a number of aquatic macrophytes within the water and around the edge. The upper part of this station was completely blocked by aquatic macrophytes and not navigable. There is lots of debris floating on the water. The substratum is muddy and contains a lot of decaying fallen leaves. No human activity was observed in this station throughout the period of study, probably due to the lonely and secluded nature of the nature of the station. The dominant macrophytes observed in this station are *Vossia cuspidate* and *Nymphaea lotus.*

Station 2: This station, also located in Ogba Ibuya area, is about 0.66km downstream of station 1. *Axonopus compressus* is the major macrophyte in this station. This is a point where Benin City wastewater discharges into the River as a result the substratum is sandy and covered with debris brought in by the waste water. The major human activity here is that Fulani herdsmen occasionally brought cattle for water. By the edge of the River are large expanses of cultivated land.

Station 3: This station, located in Oko area, is 0.6km downstream of station 2 and witnesses a high level of human activities in the form of bathing and washing by the inmates of Oko Open prison. The prison inmates also cultivate a large expanse of this area for vegetable, cassava, plantain and maize production all year round. The farms are extensively fertilized with animal waste while water is extracted from the River by means of water pump to water the farms. The major vegetation is *Vossia cuspidate* lining the edge of the river. This station is made up of sandy substratum and due to its nearness to station 2; a lot of debris was brought by the water current.

Station 4: This station is 2km downstream of station 3 and is located within Ogba Community near the bridge. The human activities here include, washing of clothes, cars and motor bikes as well as idol worshiping and water baptism. The substratum is a mixture of sand and mud. The major vegetation is *Vossia cuspidate* and *Eichornia natans*.

Samples Collection: The four sampling stations were visited monthly from April 2017 to March 2018. During this period a total number of 12 sampling visits were made to all the stations between 10.00hrs and 1.00hrs. On each sampling day, sediments and water samples were collected concurrently at all stations. The water samples were collected in clean labelled 250ml amber reagent bottles while the bottom sediments samples were collected with a clean hand trowel into a plastic basin before it wrapped with Aluminum foil and labelled. The samples were transported to the laboratory immediately for analysis.

Water Sample Preparation: The method employed was EPA method 3510c with slight modification was used for water sample extraction. 25 mls of dichloromethane was added to 250 mls of water sample (unfiltered) in its original sample bottle. The bottle was closed tightly with an aluminium lined cap. 20 µg/l of internal standard, decafluorobiphenyl was added. The bottle was shaken manually for 15 minutes so that the vortex formed at the surface reaches almost to the bottom of the bottle. The contents of the bottle were transferred to 500 ml separatory funnel and the aqueous layer was returned to the sample bottle. The 500 ml separatory funnel was rinsed twice with dichloromethane; 10 ml at first and then 15ml, transferring the solvent to the sample bottle after each rinsing.

The shaking, separation and rinsing procedure was repeated twice. After the third separation the organic layer was transferred to the 250 separatory funnel and the sample discarded. The 500 ml separatory funnel was rinsed again and the contents added to the 250 ml separatory funnel. The 250 mls separatory funnel was shaken for 2 minutes and allowed to stand for 10 minutes (Dean, 2003). 1 g of anhydrous sodium sulfate was placed in a 125 ml sintered glass funnel, and set up to drain into a 250 ml round bottom flask. The organic layer in the 250 ml separatory funnel was drained into the filtration column. 15 ml of dichloromethane was added to the aqueous layer remaining in the separatory funnel and shaken for 2 minutes and then allowed to stand for 10 minutes. The organic layer was drained again through the sodium sulfate filter column and the remaining aqueous layer phase discarded. The 50 ml separatory funnel was rinsed twice with 10 ml of dichloromethane and passed through the sodium sulfate column. The sodium sulfate column was washed with 10 mls of dichloromethane. The combined sample extracts were evaporated under vacuum using a rotary evaporator at 30 °c-35 °c to 5 or 6 ml. the concentrate was transferred with 4x 1 ml rinsing to a 15 ml graduated glass tube with conical bottom. The evaporation was finished to 3 ml under gentle stream of nitrogen at 50 to 60°c at atmospheric pressure. The extract is solvent exchanged to isooctane.

Sediment Sample Preparation for Analysis: The procedures EPA 3570, and Steindwandter and Shutler (1978) with slight modifications were used. Approximately 10.0 grams of anhydrous sodium sulfate was added to a pre-cleaned mortar and 5grams of fresh wet sediments was added to the mortar and homogenized to a complete mixture with a pestle. The mixture was carefully transferred to a pre-cleaned PTFE extraction tube which has a PTFE screw cap. 5 and 10 pre-cleaned glass beads were added. 2.5ml of a mixture of acetone and petroleum spirit (1:1) was added to the 100 ml TFE extraction tube; the extraction tube was tightly capped and allowed to stand for minimum of 20 minutes. This allows complete permeation of solvent to the matrix. 20 µg/l of the internal standard decaflourobiphenyl in iso-octane directly was added to the sediment and sodium sulphate mixture. The tube was shaken vigorously until the slurry is free-flowing. Any chunks were broken manually with the glass rod, working quickly but gently. The cap was replaced immediately after the breaking of the chunks. More sodium sulfate was added and manually mixed as necessary to produce free-flowing, finely divided slurry. The samples were extracted by rotating end-over-end for at least 30 minutes. Care was taken to release pressure by opening and closing the flasks at intervals.

The solids were allowed to settle for one to two minutes. The solvent layer was filtered through a small glass funnel containing a layer of anhydrous sodium sulfate over a plug of glass wool into a receiving conical flask. The sodium sulfate was thoroughly pre-wetted with acetone before sample filtration. The sodium sulfate layer was rinsed with 2 to 3 ml of acetone as soon as the surface is exposed.

The top of the sodium sulfate layer was not allowed to go dry. The sediment sample was extracted twice by adding approximately 15 ml of acetone/petroleum spirit mixture to the sample, capping the extraction tube tightly, and shaking vigorously by hand for 2 minutes. All the extracts are combined and poured into the round bottom flask of the rotary evaporator.

The round bottom flask of the rotary evaporator is placed in a constant temperature hot water bath so that the concentrator flask is partially, but not completely, immersed. The temperature of the bath was adjusted and the position of the apparatus so that, the solvent heat evenly. The sample volume was reduced to approximately 1.0 ml.

Cleanup for Water and Sediment: A 600 mm x 19 mm id cleanup was prepared by blocking the hole with glass wool and adding 3g of activated silica gel (60 to 100 mesh) calcined at 650 °c for 4 h, and then stored at 130 °c until use. Before use the silica gel was deactivated with 1 ml distilled water. The column was topped with 1 cm of preheated Na_2SO_4 previously heated at 650 °c for 18 h, and stored in a clean bottle in a desiccator. The column was eluted with 4 ml hexane and discarded. The concentrated extract in iso-octane was transferred to the column and eluted with 50 ml of 20 + 80 DCM/ hexane (vv ratio). The eluent was collected in a 100 ml round bottom flask. This fraction (referred as eluent1) contains PCBs and about 14 OCs (Dean, 2003).

Sample Analysis

An HP-5 fused silica capillary column (30 m length, 0.25 mm i.d.,0.25 lm film thickness) coated with 5% phenyl 95% methylpolysiloxane was used for the analyses. The oven temperature program started at 100C (holding time 1 min), increased to 120^{o}C at 20^{o}Cmin^{-}1, to 240 ^{o}C at 4 ^{o}Cmin^{-}1 (holding time 12 min) and finally to 300^{o}C at 4^{o}Cmin^{-}1 (holding time

15 min). Injector and detector temperatures were 280 and 310^{o}C, respectively. Helium and nitrogen were used as carrier (0.33 ml min^{-}1) and make-up (60 ml min^{-}1) gases, respectively. Compound identification was confirmed by GC coupled to mass spectrometry in the chemical ionization mode and negative ion recording (Fisons 8000 Series, Mass Selective Detector 800 Series).

Solutions of tetrachloronaphthalene and octachloronaphthalene were added to the vials before injection. Water and sediment extracts were injected into a Hewlett Packard 5890 Series II GC-ECD. Calibration curves were performed for each compound to be quantified. The range of linearity of the detector was evaluated from the curves generated by plotting detector signal vs amount injected. All measurements were performed in the ranges of linearity found for each compound.

Statistical Analysis of Results

In addition to the basic significant measurements of central tendency and dispersion, interstation comparisons were carried out to test for significant differences in the organochlorine's values. If significant 'H' values ($p < 0.05$) were obtained, non-parametric multiple comparisons were performed to determine the location of significant difference.

RESULTS

Water Samples: The distribution of pesticides residues in the surface water of Ogba River revealed the present of the following pesticides and their ranges in (ng/l) between 8.15 – 19.90 (HCH), 24.77 – 44.90 (chlordane), 7.82-20.00 (DDT), 35.87- 98.80 (endrin), 8.90 - 33.60 (dieldrin), 36.55 -77.40 (endosulfan) (Table 1) and 150.00 – 547. 00 (PCBs) (Figure 2)

Seasonally, the OCPs and PCBs residues in the surface water are shown in Figure 3. The distribution revealed that the wet season had the following concentrations; (in ng/l), PCBs (415 ± 5.90) endrin (82 ± 2.30), endosulfan (62 ± 2.10) , chlordane (32 ± 1.90) , dieldrin (24 ± 1.12), DDTs (14.80 ± 1.40) , HCHs (15.40 ± 0.60), and the dry season; (in ng/l), PCBs (398 ± 7.10) endrin (72 ± 1.40), endosulfan (46 ± 1.40) , chlordane (23 ± 1.20) , dieldrin (18 ± 0.32), DDTs (11.20 ± 0.70) , and HCHs (12.10 ± 0.20).

Table 1: Spatial Distribution of Organochlorine Pesticides Residues in the Surface Water of Ogba River, Niger Delta Nigeria

Pesticides (ng/l)	Station 1	Station 2	Station 3	Station 4	P-Value	WHO Limit
HCH	8.41±1.5	18.87±0.72	17.39±1.41	12.86 ±1.05	P < 0.05	2000
∑Chlordane	26.53±3.37	49.51±2.40	34.41±2.79	29.14±2.37	P > 0.05	200
∑DDT	2.12±1.0	25.78±1.25	18.3±1.0	12.51±0.69	P< 0.05	1000
∑Endrin	17.58±5.73	99.04±3.17	88.62±7.19	49.49±4.02	P<0.05	600
∑Dieldrin	3.58±1.02	28.30±1.0	10.36±0.84	9.02±0.93	P> 0.05	
∑Endosufan	16.80±4.61	67.84±4.37	51.89±4.21	39.78±3.23	P> 0.05	*

Sediments: The OCPs and PCBs in the bottom sediment in ogbia River had the following concentrations; (µg/kg) 7.62 – 39.99 (HCH), 13.62 – 149.00 (chlordane), 47.36 – 58.55 (DDT), 16.32 -112.20 (endrin), 54.93-99.20 (diedrin), 4.19 – 97.62 (endosulfan) (Table 2) and 440.00 – 701. 00 (PCBs) (Figure 2)

The bottom sediments seasonal variation were; wet season,(in µg/kg); PCBs (645 ± 8.40), endrin (93 ± 0.30), chlordane (132 ± 3.40), endosulfan (92 ± 0.90), dieldrin (86 ± 2.62), DDT(51± 1.50), HCHs (28 ± 0.70); dry season,(in µg/kg); PCBs (580 ± 9.10), endrin (81 ± 1.20), chlordane (113 ± 1.20), endosulfan (76 ± 0.50), dieldrin (68 ± 1.02), DDT(45±0.30), HCHs (21 ± 0.20) (Figure 3)

Table 2: Spatial Distribution of Organochlorine Pesticides Residues in the bottom Sediments of Ogba River, Niger Delta Nigeria

Pesticides (µg/kg)	Station 1	Station 2	Station 3	Station 4	P-Value	WHO Limit
HCH	9.59±2.40	36.71±1.36	18.19±0.29	15.92±1.30	P<0.05	2000
∑Chlordane	53.50±11.50	125.57±1.27	113.00±1.20	53.83±1.21	P<0.05	2000
∑DDT	12.30±1.00	54.54±4.18	51.58±4.12	47.58±4.12	P<0.05	1000
∑Endrin	48.66±1.52	104.47±4.422	96.88±4.21	87.77±1.45	P<0.05	600
∑Dieldrin	32.77±5.09	87.76±7.44	82.39±7.09	77.78±4.85	P<0.05	
∑Endosufan	4.80±2.83	94.80±2.83	83.14±2.69	76.67±0.38	P<0.05	

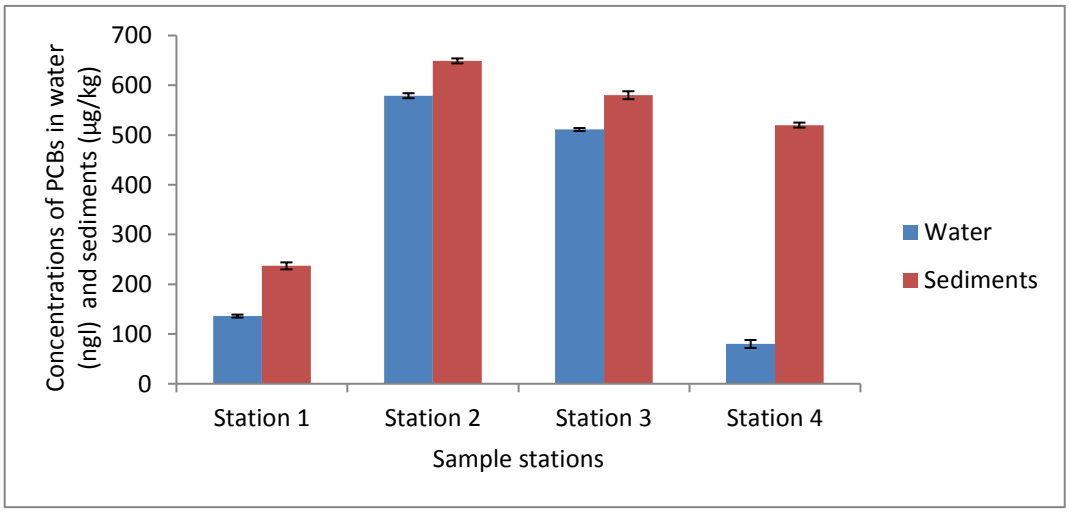

Figure 2: Spatial Distribution of PCBs Residues in the Surface Water and Bottom Sediments in Ogba River, Niger Delta Nigeria.

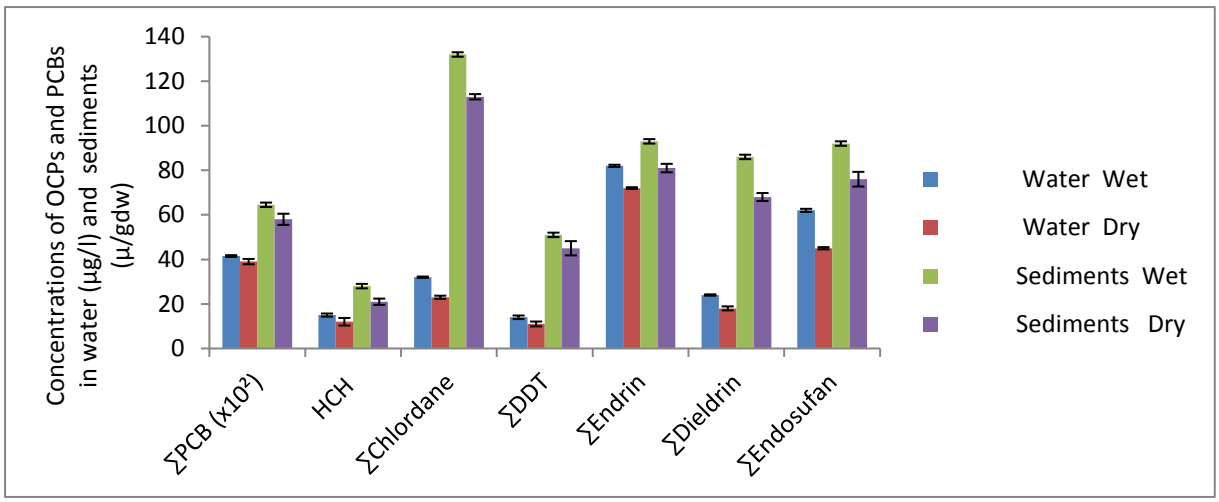

Figure 3: Seasonal Distribution of OCPs and PCBs Residues in the Surface Water and Bottom sediments in Ogba River, Niger Delta Nigeria.

DISCUSSION

The distribution and concentrations of persistent organochlorine compounds in water and sediments from Ogba River Niger Delta Nigeria revealed the present of HCH, chlordane, DDT, endrin, dieldrin, endosulfan and PCBs. This showed their persistence in the environment, even when they have not been used recently.

This study is similar to those by others reported in Nigeria as well as other parts of the developing world. However, they are much higher spatially and seasonally compared to the standards established for the aquatic life. For instance, the values of Endosulfan, DDT, PCB were much higher than the regulatory standard of 1.9 ng/g, 10 µg/L (PCBs and DDT) respectively (CCME, 2002)

The concentration of these pollutants in the surface water followed the order: PCBs > endrin > endosulfan > chlordane > dieldrin > DDTs > HCHs, while in the sediment, the order of distribution is PCBs > chlordane > endrin > endosulfan > dieldrin > DDTs > HCHs. These concentrations of these organochlorine compounds observed in this study could be as a result of their stability, which attributed to their persistence in the environment (Konan *et al*., 2008). The high concentration of the PCBs in the water could be accredited to its resistant to degradation (Gevao *et al*., 2012), and pollutants generated by agro-industrial units located on the shores of this ecosystems. Hexachlorocyclohexane (HCH) as the least pesticide detected in the water column, which may be as a result of it not frequently use for agricultural purposes and the concentration observed could be as a result of the waste discharge from the municipality and other anthropogenic activities.

The concentrations of the organochlorine compounds were higher in the sediment than the surface water. This is due to the fact that the sediment is one of the important sinks and basin for persistent pollutants discharged into the environment. A similar finding was reported by Li *et al*. (2001) in the Sediment Profile of the Pearl River Estuary. The concentrations

reported in the water,though is lower than the sediment concentration, may be attributed to the fact that the sediments may release back to overlying waters the pollutants bound on it, as a result of remobilization due to various diagenetic courses (Li *et al*., 2000). As a result, sediments are now regarded as a vital source of many micro pollutants that seriously affect the balance of the natural ecosystems (Sodergren, 1997).

Spatial distribution indicated that station 2 is most contaminated site with the pattern; station 2 > station 3 > station 4 > station 1. The high level of organic contaminants recorded in station 2 and 3 can be attributed to the pollution load added from the wastewater discharges into the River, increased sewage flow from the metropolis, extensive agricultural and human activities. The low concentration observed in station 4 may be as a result of decreases downstream flow of the water, less human activities and degradation of contaminants by microbial action. Low downstream concentrations of organochlorine have been reported by Puneeta *et al.* (2011) in the surface sediments of River Yamuna in Delhi, India which similar to our findings in the Ogba River's water and sediment.

Seasonal variations revealed that OCPs and PCBs concentrations for both matrixes were higher in the wet season than dry seasons. These variations could be as a result of human activities such as agriculture where farmers use pesticides for farming during wet season and the draining of water through run off from the urban area into the River.

The use of pesticides in illegal fishing, and poor farming methods in most developing countries has led to the introduction of many chemicals such as heavy metals, pesticides and fertilizers in the coastal ecosystem. This notion supports our findings as the persistent organic contaminants were observed in the area that serves as control with no evidence of input of pollutants. Besides human factors, atmospheric transport / disposition could also be accredited with the concentrations observed in the reference station. Comparable to our findings was the report of Joan *et al*. (2004), where they observed persistent organochlorine compounds in soils and sediments of European high-altitude mountain lakes that are free from local anthropogenic sources

CONCLUSION

Many OP and PCBs are extremely hazardous to fish and other aquatic life. Fish kills involving these chemicals have been documented and their potential toxicity and high persistence, led to the enactment of various guidelines for their regulated use or exclusion from the market.

Farmers and landowners who use pesticides can protect aquatic habitats by first considering whether pesticide treatment is really necessary. If pesticides must be used, use the least toxic product that will do the job and apply it according to the label and follow best Management Practices. More attention has been given to sustainable agriculture as shown in evidence in the movement toward more diverse mechanize farming in developed countries. These reduce soil erosion, improved water quality, improve nutrient cycling, and ameliorate pesticide inputs into the environment, this need to be emulated in the developing world.

Discharging of wastewater into the Rivers should be discouraged. Waste water should be treated and made harmless before emptying into the aquatic environment. Similarly, high

toxic soap and detergents should not be used for washing clothes, cars or motor bikes close to aquatic environment.

The OCPs and PCBs concentrations were below the values established by the regulatory bodies. Though, the pesticides had low concentration in the River at that point in time it has little or no effect on the biota. Although, if not monitored over the years, it can cause harm to the environment, bioaccumulate and biomagnifies along the food chain

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