Organochlorine Pesticide Residues in water, sediments, Fin and Shell-fish samples from Lagos Lagoon Complex, Nigeria.

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Abstract: The organochlorine pesticide (OCPs) residues were measured in Fin fish (*Oreochromis niloticus*), shell fish (*Callinectes pallidus*), sediments and water samples. These were collected from Ologe lagoon, Kuramo lagoon and Lagos Lagoon. The analysis was done using Gas Chromatograph with Electron Capture Detector. The overall range OCPs in ng/l (*with their mean and standard deviation*) in water samples were: Aldrin ND-658.0(242.5±361.5), Chlordane 218.0-702.0(393.7±267.9), Endrin ND-2551.0(959±1388.3), DDT ND-115.0(41.3±63.9), DDD 20.3-268.0(120.5±130.4), pp-DDE 0-176.0(99.6±90.2), α-HCH ND-230.0(117±115.1), γ-HCH ND-783.0(273.6±441.5), β-HCH 28.9-518.0(245.9±249.1), δ-HCH 0-498.0(211.0±257.5), HCB ND-7.9(2.6±4.5), Dieldrin 14.2-175.0(102.4±81.5), Endosulfan ND-3726.0(1549.7±1940.4), and Heptachlor ND-1405.0 (525.7±766.4). While the concentrations in sediment ranged between 0.4 – 43.5μg/kg. The highest levels of OCPs were detected in shellfish and sediment of Ologe lagoon which is bordered by heavy industrial. These concentrations were found to exceed in several folds the recommended limit/guideline (10ng/l) for freshwater aquatic life in Canada and 0.01ppm by Federal Environmental Protection Agency (FEPA) now Federal Ministry of Environment in Nigeria.
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Key words: organochlorine pesticides, fin-fish, shell-fish, Lagos lagoon complex, sediment.

1. Introduction

Organochlorine pesticides (OCPs) are highly toxic synthetic organic chemicals (carbon-based) that are used in industry and agriculture, as well as created un-intentionally through chlorine combustion processes (USEPA, 2002). OCPs are a group of chemicals which are very resistant to natural breakdown processes and are therefore extremely stable and long-lived. They are known to be toxic to man (Ademoroti, 1996). Some of the symptoms of pesticide poisoning according to Bouman *et al.* (1990) and Winter (1992); may be acute (including: irritation, dizziness, tremor), or/and chronic (mainly: reproductive failures, birth defects, endocrine disruption, immune system dysfunction, cancer, tonic and chronic convulsion). Studies also revealed that OCPs have strong potentials to cross placental barriers even in minute concentration and cause serious neonatal damage (Saxena, 1981). DDT in particular can block potassium influx across the membranes of nerve fibres and cause increased negative after-potentials. DDT also induces the mixed function oxidize system thereby altering the metabolism of xenobiotics and steroid hormones (Ademoroti, 1996).

Most water bodies in Nigeria, especially Lagos serve as a sink for the disposal of waste from about 2000 medium and large scale industries located in the metropolis (Anetekhai *et al.*, 2007). This situation has become worrisome as a result of increased urbanization and industrialization, and laxity in enforcing environmental regulations in developing countries (Biney *et al. 1994*). Organochlorine pesticides are among the first set of pesticides used and still in use in Nigeria despite their ban in developed countries due to the associated problems of bioaccumulation and environmental persistence, and potency. The chemical stability, high lipid solubility and toxicity to man and animals have led governments and researchers to be concerned with their presence in the environment. Although most of the pesticides used in Africa are imported, there are a few production facilities in some countries for OCPs, e.g. Nigeria, Senegal, South Africa, Côte d'Ivoire and Egypt. It is estimated that about 25,000 tonnes of OCPs are in use in the region. Pesticides have low solubility in water (e.g. DDT: 1.2*μ*g/l), being lipophilic, can be concentrated to harmful levels in the aquatic environment through bioaccumulation, biomagnification and biogeochemical processes (Edwards, 1977). However, the lack of scientific and ecotoxicological data on chemical pollutants, effective for the control and prevention of aquatic pollution has been recognized in Africa (Biney *et al.*, 1987). Meaningful development of management policies and regulatory framework for the protection of the aquatic environment in Nigeria can only be achieved on the availability of reliable and adequate scientific data generated in the region. However, there have not been holistic data coupled with descriptive and comparative foci. Thus this report will serve as a valuable tool in assessing the biota of the various trophic levels for the bio-accumulation of organochlorine pesticide residues in the Lagos lagoon complex.

2. Materials and Methods

Sampling Area: Isebor *et al.,* (2006) described the Lagos Lagoon as a brackish coastal water body found on the Western part of Nigeria with latitudes 6º26' - 6º30'N and longitudes 3º23' - 4°20'E, and cuts across the southern part of the metropolis, linking the Atlantic Ocean (in the west and south), Lekki and Kuramo Lagoon (in the east). It is about 700sq km in area and 285km in perimeter and it is the largest along the West African coast (figure 1). The lagoon is shallow, with an average depth of about 1.5 m. Shoals of sand are scattered in the lagoon and are usually exposed during low tides. Apart from marine transportation and fishing, complex mixtures of domestic and industrial effluents enter the Lagos Lagoon daily. About 80-85% of the industries in Nigeria are located in Lagos State and they all discharge their effluents into the Lagos lagoon complex. According to Osibanjo, *et al* (1993) River Ogun has been found to be contaminated with most of these Organochlorines which are break down products from industrial and domestic effluents and this river which traverses three states also discharges into the Lagos lagoon.

Sampling: Fin fish (*Oreochromis niloticus*), shell fish (*Callinectes sp.*), sediment and water samples were collected from Kuramo, Ologe and Lagos Lagoon. The fin and shell fish samples were refrigerated and extraction done within few days of collection. All samples were identified and analyzed at the Laboratory of the Nigerian Institute of Oceanography and Marine Research. All chemicals and reagents were of analytical grade and of highest purity possible. Dichloromethane and n-hexane used for the extraction and clean-up was obtained from Fisher Scientific. Silica gel, acetone and anhydrous sodium sulphate used was supplied by BDH laboratories.

Extraction of Finfish and Shell-fish: The extraction was carried out following methods from Osibanjo and Adeyeye (1995) and Osibanjo and Tongo (1985) as described by Saleh and Lee (1978) with some modification. The fish sample were dissected and filleted to obtain the fish flesh. The fillet was wrapped in a labelled aluminum foil paper and weighed. Each fish flesh sample was removed from the foil paper and put into a mortar. Sodium sulfate was added to it and a pestle was used to homogenize the mixture. The homogenous blend was allowed to dry overnight for a period of 18hours. The shell fish was spilt open into two to remove the carapace. This was carefully removed to reveal the fleshy part which was weighed and wrapped up in a labelled aluminum foil paper. Each of the shell fish flesh was removed from the foil paper and put into a mortar. Sodium sulfate was added to it and a pestle was used to homogenize the mixture. The homogenous blend was allowed to dry overnight for a period of 18hours.

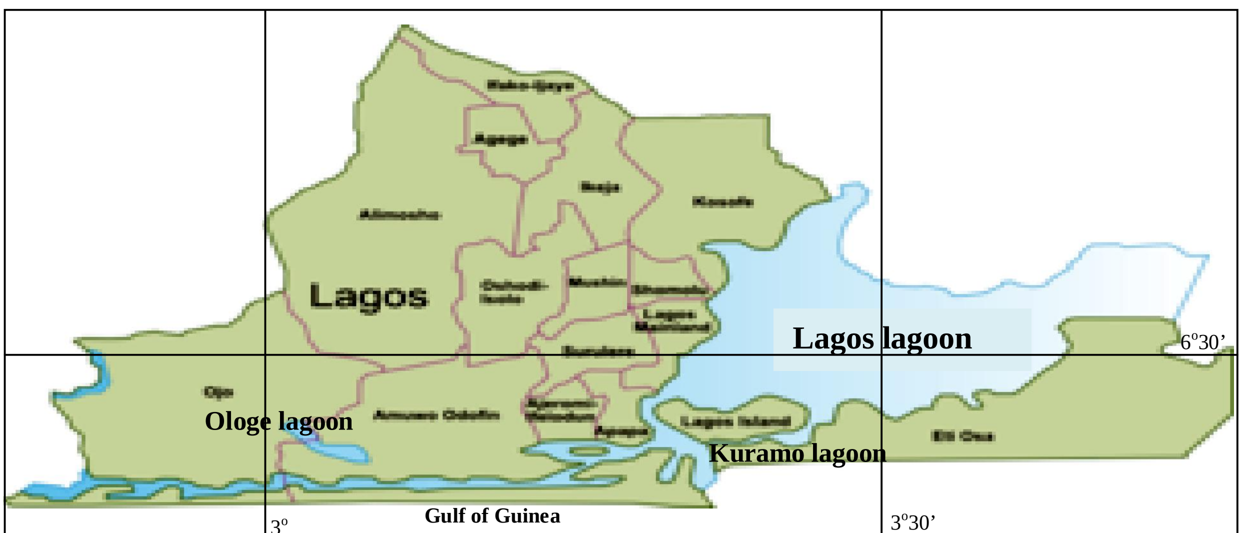

Figure 1: **Map of Lagos-western Nigeria showing the Lagos lagoon complex and the sample stations**

A mixture of acetone and petroleum ether was used instead of the DCM used in water extraction because a more aggressive chemical is required for the extraction of fats. 30ml of the mixture was added to the homogenous blend in a burette and the reagent was allowed to be absorbed by the blend. After 10minutes, another 70ml of the mixture was added. The extract dripped into the beaker drop by drop for over 60minutes. At the end of the extraction process, the extract was transferred into a round bottom flask connected to a pre-weighed receiver through a Liebig condenser, and concentrated to about 10 ml on a water bath maintained at 90ºC. The remaining solvent in the concentrated extract was evaporated using a rotary evaporator. The receiver contained the fat extract (0.1ml, 0.5ml and 0.5ml) for Kuramo, Ologe and Lagos Lagoon sample stations respectively.

Sample Clean Up: A micro column plastic injector was clamped to a retort stand, filled with glass wool; 1gm of deactivated silica gel and 5ml of hexane was added. 2ml of hexane was used to mix each of the extract and injected separately into the micro column and after the extract had completely drained into the silica gel, 40ml of hexane was added. Each fraction was concentrated to 1 ml in a stream of nitrogen before they were analyzed using the gas chromatograph.

Gas chromatography: No clean up was done for the water sample. The level of the pesticide residues were determined using the Gas Chromatograph, model 5890 using Electron Capture Detector. The following conditions were maintained. Gas pressure was 60 psi and injector temperature was 220^{o}C, column temperature was 190^{o}C, detector temperature was 270^{o}C, 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 injection volume was manual and the compounds were sorted by signal. The retention time (mins) were used to identify the compounds present in the samples.

The study on Organochlorine Pesticide Residues (OCPs) in the Lagos lagoon complex presents various stunning results in water, fin-fish, shell-fish and sediment samples. Tables 1, 2, 3 and 4 shows the combinations of the data sets in water, fin-fish, shell-fish and sediment samples from three (3) sampling stations, with their statistical analysis. The samples analyzed contained relatively high concentrations of OCPs residues, which includes: Aldrin, Chlordane, Endrin, DDT (pp'1,1,1-trichloro-2,2-bis-(4-chlorophenyl) ethane), DDD, DDE (pp1,1,-dichloro-2, 2-bis-(4-chlorophenyl) ethylene), α-HCH, γ-HCH, β-HCH, δ-HCH (1,2,3,4,5,6-hexachlorocylohexane), HCB (Hexacholorobenzene), Dieldrin (1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,8,8a octahydro-1,4,5,8 dimethanonaphthalene), Endosulfan, and Heptachlor.

Identification and quantification: the standard mixture contained OCPs. Quantification was done based on area count match with those of known concentration of the standards.

CALCULATIONS

Concentration of Pops in Water Sample (ppm)

=Area of counts of POPs) (Final volume of water

(Rf factor)(Initial volume of water)

Where: Initial volume of water = 200ml,

Final volume of water = 0.10ml,

Rf factor = 1.59094 X 10¯6

Conc. of Pops in Fish Sample (ppm)

= (Area of counts of POPs)(Final volume of fish extract)

(Rf factor)(Weight of fish flesh)

Where: Weight of fish flesh = 200ml,

Final volume of fish extract = 0.10ml / 0.50ml.

Sediment samples: The extraction and clean up of sediments for semi-volatile organics was carried out following the internal standard methods from Patricia and Jui-Lan (1996).

3. Results

The overall range in ng/l (*with their mean and standard deviation*) in water samples were: Aldrin ND-658.0(364.7±416.1), Chlordane 218.0-702.0(393.7±267.9), Endrin ND-2551.0(959±1388.3), DDT ND-115.0(41.3±63.9), DDD 20.3-268.0(120.5±130.4), pp-DDE 0-176.0(99.6±90.2), α-HCH ND-230.0(117±115.1), γ-HCH ND-783.0(273.6±441.5), β-HCH 28.9-518.0(245.9±249.1), δ-HCH 0-498.0(211.0±257.5), HCB ND-7.9(2.6±4.5), Dieldrin 14.2-175.0(102.4±81.5), Endosulfan ND-3726.0(1549.7±1940.4), and Heptachlor ND-1405.0(525.7±766.4).

The range in concentration of OCPs in sediment samples (µg/kg) from the lagoons were: Aldrin ND – 2.96, Dieldrin 0 – 1.83, Chlordane 0.62-43.54, Endrin ND – 15.6, Endosulfan ND – 32.13, Heptachlor ND – 7.15, HCB ND – 1.3, α-HCH 0 – 2.42, β -HCH 0 - 3.18, γ -HCH 0 – 5.16, δ-HCH 0 - 8.94, DDD ND – 3.83, DDE 0 – 5.27, and DDT 0 – 1.09. The lowest OCP during the period of study is HCB, while chlordane is the highest. The total concentration of OCPs in sediments samples between the three sample stations ranged between 6.22 and 109.28µg/kg, with Ologe lagoon being the most pollution.

DISCUSSION AND CONCLUSION

Study was carried out to assess the presence and levels of pesticides in sediments, water, fin and shell fish; and the ecological impacts of specific groups of Osibanjo, *et al., (1995) m*ost freshwater sediments in Africa are contaminated with organochlorine compounds. Except for high levels of DDT and metabolites (1.73–877 ng/g dry weight).

Lekki Lagoon, Nigeria, a semi-enclosed water body with several rivers draining into it, provides a hot spot with very high mean values of some compounds, e.g. Dieldrin (4,560 ng/g) and *ρ,ρ'*-DDE (263 ng/g dry weight). Concentrations above 100 ng/I were found for *γ*-HCH, DDE and total DDT in Egyptian lakes, Ibadan streams, Nigeria (DDT only) and PCBs in Hartbeespoort Dam, South Africa respectively, indicating pollution by these chemicals. The results are similar to level of DDT reported in Colombia OCPs in the Lagos lagoon complex. The catchments areas are largely sites of urbanization and industrialization. They are characterized by the presence of hotels, recreational centers, fishing activities, inland water transportation, agriculture, palm plantations and intense industrial activities. In the investigation carried-out in: Lagos, Kuramo and Ologe lagoons, fourteen [14] organochlorine pesticides were detected. Relatively, as indicated in the results; more information on a wide range of organochlorine pesticides has been reported, in particular on Aldrin, Dieldrin, DDT and metabolites and PCBs (IPEP, 2006). As opined by (GEMS/WATER) and the *γ*-HCH values reported for Some major rivers in the United Kingdom (Croll, 1991), Japan (Suzuki *et al*., 1974) and India (Ramesh *et al*., 1990). Contrary to the findings in this study, Merriman and Metcalf, (1988) opined that the majority of the African inland waters have *γ*-HCH concentrations below 10ng/l which is within the recommended guideline for the protection of freshwater aquatic life in Canada, however, this is now obsolete since exceedingly high Dieldrin levels of over 1000 ng/l were found in some Nigerian surface waters, especially Lagos lagoons (Osibanjo, *et al.* 1995).

Table 1: Concentration of Organochlorine Pesticide Residues (OCPs) in water from three (3) samples Stations in the Lagos lagoon complex, Nigeria.

PESTICIDES	Fin-fish (ng/g)			
	Kuramo lagoon	Lagos lagoon	Ologe lagoon	Mean and SD
Aldrin	ND	30240	534	15387±21005
Dieldrin	13	ND	517	265±356.3
Chlordane	26	381	5480	1962±3051.7
Endrin	ND	11430	7630	9530±2687
Endosulfan	ND	37780	2850	20315±24699
Heptachlor	ND	13260	1040	7150±86408
HCB	13	ND	ND	12.8±0
α-HCH	0	3510	868	1459±1828.2
γ-HCH	ND	11490	1410	6450±7127.6
β-HCH	0	55740	639	18793± 31999
δ-HCH	16	ND	990	502.8±689
ρ,ρ' DDD	38	421	408	288.9±217.7
ρ,ρ'DDE	0	120	1030	383.3±563.2
DDT	2.6	ND	60.3	31.47±40.8

Table 2: Concentration of OCPs in Fn-fish in the Lagos lagoon complex, Lagos-Nigeria.

PESTICIDES	WATER (ng/l)			
	Kuramo lagoon	Lagos lagoon	Ologe lagoon	Mean and SD
Aldrin	ND	658	69.5	363.7± 416.1
Dieldrin	14.2	118	175	146.5± 81.5
Chlordane	261	218	702	460± 267.9
Endrin	ND	2551	326	1438.5± 1573.3
Endosulfan	ND	923	3726	2324.5± 1982
Heptachlor	ND	1405	172	788.5± 871.9
HCB	7.86	ND	ND	7.8 ± 0
α-HCH	0	230	121	175.5± 115.1
γ-HCH	ND	783	37.8	410.4± 526.9
β-HCH	28.9	518	191	354.5± 249.1
δ-HCH	0	498	135	316.5± 527.6
ρ,ρ' DDD	20.3	268	73.3	170.65± 130.4
ρ,ρ'DDE	0	123	176	149.5± 90.3
DDT	8.96	ND	115	115 ± 74.9

Abbreviations: ng/l = nanogram per liter, ng/g = nanogram per gram, ND = Not Detected, SD = Standard Deviation.

Table 3: Concentration of OCPs in shell-fish in the Lagos lagoon complex, Nigeria.

PESTICIDES	Shell fish			
	Kuramo lagoon	Lagos lagoon	Ologe lagoon	Mean and SD
Aldrin	ND	177	580	378.5±2972
Dieldrin	1820	211	144170	48734±107353
Chlordane	27.5	1070	7100	2733±12459
Endrin	ND	580	719	649.5±98.3
Endosulfan	ND	858	7300	4079±6383.4
Heptachlor	ND	1420	5490	3455±10006
HCB	27.5	ND	ND	27.5±0
α-HCH	1530	900	3830	2087±2231.3
γ-HCH	ND	675	2300	1488±1366.7
β-HCH	2090	984	1720	1598±1126.2
δ-HCH	0	1500	19530	7010±10909
ρ,ρ' DDD	48.6	0	0	16.2±28.1
ρ,ρ'DDE	0	1800	2360	1387±1233.1
DDT	21.8	440	3720	1394±2025.3

Table 4: Concentration of OCPs in sediment in the Lagos lagoon complex, Lagos-Nigeria.

PESTICIDES	Fin-fish (ng/g)			
	Kuramo lagoon	Lagos lagoon	Ologe lagoon	Mean and SD
Aldrin	ND	400	43400	21900±30405.6
Dieldrin	0	530	1830	786.7±941.6
Chlordane	0	650	43400	14683±24871.5
Endrin	ND	15600	8000	11800±5374.0
Endosulfan	ND	2990	32130	17560±20605.1
Heptachlor	ND	7150	3330	5240±2701.2
HCB	1300	ND	0	650±919.2
α-HCH	0	2420	2320	1580±1369.2
γ-HCH	0	3180	1950	1710±1603.5
β-HCH	0	5160	1350	2170±2675.9
δ-HCH	0	8940	4000	4313±4478.2
ρ,ρ' DDD	3830	ND	1640	2735±1548.6
ρ,ρ'DDE	0	460	5270	1910±2918.9
DDT	1090	0	960	683.3±595.3

Similarly, levels reported for Dieldrin under the GEMS/WATER in Colombia and Malaysia respectively, exceed several fold the 4 ng/I guideline set for Canadian freshwater aquatic life.

Analyzed samples from Kuramo revealed the presence of various OCPs: DDT and its metabolites. ρ,ρ′ DDD was the major degradation compound detected in the water sample, although, ρ,ρ′ DDE is slightly higher in concentration compared to ρ,ρ′ DDD in the sediment sample. This is contrary to other literature where the level of ρ,ρ′ DDE was reported higher than in ρ,ρ′ DDD. Kuramo water exhibits an unusual combination of physical and chemical characteristics, according to Adeboyejo *et al.* (2009); this may be as a result of high eutrophic condition, domestic use of insecticide and temporal fluctuations of the water chemistry, The low level of agricultural and industrial activities around the Kuramo may be responsible for the low concentration of OCPs in the water body, the major source of pollution in this lagoon is domestic (hotels and un-coordinated settlements on the shore-line).

Water samples from Ologe and Lagos lagoon showed a higher concentration of pesticides relative to that obtained in Kuramo waters (Olarinmoye, 2007). Endrin, Endosulfan, Aldrin, Heptachlor, Chlordane, and metabolites of DDT were all detected and found to exceed several folds the recommended limit/guideline (10ng/l) for water. This reveals a high level of pollution which is in accordance with reports from Tongo, (1985), Ogunlowo, (1991), Nwankwoala and Osibanjo, (1992) who reported a general contamination of African inland surface waters by a broad spectrum of Organochlorine pesticides (OCPs). The mean pesticides residue levels obtained for all the water samples were higher than those obtained by past researchers from studies carried out in some rivers in Nigeria. This view was supported by Ize-iyamu *et al.*, (2007) who discovered that pesticides were bio-accumulated at the bottom of the lagoon.

These chemicals tend to bind with soil sediment particles and with tissues of the benthic organisms (shell fishes). Consumption of such contaminated shellfish over a long period may be hazardous to man's health causing carcinogenic, respiratory, reproductive problems and brain damage especially in children.

The Lagos Lagoon complex is considered in this study, to be significantly and highly contaminated with OCPs and therefore may pose significant health threat. There is an urgent need for monitoring of these non-easily metabolized and persistent pesticides in order to prevent long-term environmental health hazards and promote preservation and conservation of the aquatic resources.

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