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Original Article

Assessment of Pollution load of Persistent Organic Pollutants in the Lagos Lagoon, Nigeria

Adogame L.[1], Aneni T.I.[*2], Chikwendu C.[3] and Ahaneku C.[4]

[1]Sustainable Research and Action for Environmental Development (SRADev) Nigeria
[2]Sustainable Environment Development Initiative (SEDI) Nigeria
[3]Friends of the Environment (FOTE) Nigeria
[4]Nigerian Environmental Study/Action Team (NEST) Nigeria

***Corresponding Author**
Aneni T.I.
Sustainable Environment Development Initiative (SEDI) Nigeria
E-mail: tomaneni1@yahoo.com

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Abstract

Eighteen Persistent Organic Pollutants (POPs) residue were measured in water, sediment, fish, soil and egg samples. These samples were collected from selected areas (Ilaje, Oko Oba, Iddo and Apapa) based on their proximity to anthropogenic activities that could result in the deposition of these pollutants in the lagoon. The analysis was done using gas chromatograph with pulsed flame photometric detector. The results show that total POPs concentration for all the sampling stations ranged from 2.12 - 5.66µg/L (water), 8.55 - 15.31µg/kg (sediment), 3.83 - 10.96µg/kg (fish), 1500.84 - 2495.73µg/kg (soil) and 3.92 - 9.56µg/kg (egg). The highest concentrations of individual OCPs were 4.53µg/kg (Endosulfan) (Apapa-water), 6.28µg/L (Endrin)(Ilaje-sediment), 5.83µg/kg (Endosulfan)(Ilaje-fish), 1710.06µg/kg (Endosulfan)(Apapa-soil), 4.42µg/kg (Endosulfan)(Ilaje-egg). The concentration of total POPs at the different sampling stations increased in this order; 2.12µg/L (Iddo), 3.88µg/L (Oko Oba), 3.96µg/L (Ilaje) and 5.66µg/L (Apapa) for water samples; 8.55µg/kg (Apapa), 8.94µg/kg (Iddo), 14.6µg/kg (Oko-Oba) and 15.31µg/kg (Ilaje). Sediment sample values were 3.83µg/kg (Iddo), 5.29µg/kg (Apapa) and 10.96µg/kg (Ilaje) and 9.58µg/kg (Oko Oba). Fish sample values were 1500.84µg/kg (Iddo), 1592.31µg/kg (Oko Oba), 1783.72µg/kg (Apapa) and 2495.75µg/kg (Ilaje). Soil sample values were 3.92µg/kg (Apapa), 4.12µg/kg (Iddo), 7.81µg/kg (Oko Oba) and 9.56µg/kg (Iddo). The concentration of total POPs in the various media increased in this order; 3.91µg/L (water), 6.36µg/kg (egg), 7.41µg/kg (fish), 11.85µg/kg (sediment) and 1843.16µg/kg (soil). This study indicates that water, sediment, fish, soil and egg samples collected from the Lagos Lagoon are contaminated with varying amounts of persistent organic pollutants, and need for enforcement of relevant legislation against polluters.

1. Introduction

Persistent Organic Pollutants (POPs) are chemical substances that persist in the environment, bioaccumulate through the food web, and pose a risk of causing adverse effects to human health and the environment [1]. Organochlorine pesticides (OCPs) are highly toxic synthetic organic chemicals (carbon-based) that are used in industry and agriculture, as well as created unintentionally through chlorine combustion processes [2]. They are a group of chemicals which are very resistant to natural breakdown processes and are therefore extremely stable and long lived. These chemicals include the group of DDT (dichloro-phenyltricholoroethane), isomers of BHC (hexachlorobenzene), aldrin, dieldrin, endrin, chlordane, toxaphene, endrin, heptachlor, heptachlor epoxide, Methoxychlor and HCH (hexacyclochlorobenzene).

These 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 [3]. Pesticides are among the priority pollutants that has been monitored in a wide variety of matrices such as honey [4], waters [5,6], soils [7], stream sediments [8], crops especially leafy vegetables [9] and potato crops [10] and their levels may represent a serious hazard to human health. 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 [11]. Organochlorine pesticides (OCPs) residue reaches the aquatic environment through direct run-off, leaching, equipment washing and careless disposal of empty containers etc.[12].

Ecological effects of OCPs include interference with reproductive success of organisms high on the food chain, especially fish eating birds such as osprey, pelicans, falcons and eagles. OCPs especially DDT have estrogenic and enzyme inducing properties. Ortho and para isomers of DDT compete with estradiol for binding to estrogen receptors in uterine cytosol thus causing changes in steroid metabolism and alter the ability of birds to mobilize calcium to produce strong egg shells[13-17]. They are known to be toxic to man[18]. According to Bouwman *et al*[19] Some of the symptoms of pesticide poisoning 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). This has led to

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the prescription of tolerances such as maximum residue level (MRL) and no observable adverse effect level (NOAEL) for various pesticides in food and water, especially by the Codex Alimentarius Commission [20].

1.1 Objective

The objective of this study was to determine the contamination level of POPs in water, sediment, fish, soil and eggs from selected areas of the Lagos Lagoon close to significant anthropogenic activities which could inadvertently lead to deposition of POPs residue in the water body. An important aspect of this study is to protect public health.

2. Materials and Methods

2.1 The Study Area

Isebor *et al*[21] 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' - 4o20'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). The lagoon is shallow, with an average depth of about 1.5 m. Shoals of sand due to sediment deposit 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[21]. Four sampling stations based on criteria of proximity to point of effluent discharge from Factories and degree of human activities in the area along the lagoon was selected. Map showing sampling locations are shown in figure 1. Sampling coordinates are shown in Table 1.

Fig. 1: Map of the Sampling Stations

Fig. 1: Map showing sampling locations on the Lagos lagoon

Table 1: Sampling stations and their coordinates

Sampling Station	Description	Coordinates
A	Ilaje	N06⁰31.324¹ E3⁰24¹
B	Oko-Oba	N06⁰29.323¹ E3⁰23¹
C	Iddo	N06⁰27.322¹ E3⁰22¹
D	Apapa	N06⁰25.321¹ E3⁰21¹

2.2 Community Approval for Sampling and Analysis

The Ilaje community was chosen as a pilot community due to its relatively large population and size. They had shown great interest in the preliminary stakeholder consultation process. The community was visited on the 19 September 2012. The traditional ruler was represented by his chiefs (Ayegbeni Felix; Gbadebo Ipensi; Sunday Kukute and Felix Titiola). Approval was sought and obtained for water sampling and collection of sample materials for analysis.

2.3 Sampling Stations

Four sampling stations based on criteria of proximity to point of effluent discharge from factories and degree of human activities in the area along the lagoon was selected.

Station A

This station is close to the Ilaje community (Plate 1).

Plate 1: Station A

Station B

This station is at Oko-Oba (Plate 2). Surface water is visibly oily and close to the timber industrial area. Saw millers were visibly present and use of wood preservatives was observed.

Plate 2: Oko-Oba

Station C

This station is at Iddo (Plate 3). Effluents from domestic and industrial activities are major sources of pollution. Industries include FRIGOGLASS, CocaCola bottling etc.

Plate 3: Iddo

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Station D

This station is at Apapa, close to the Folawiyo Tank Farm (Plate 4). Surface water is visibly oily and close to industries (Oil and gas, transportation and haulage, shipping, food).

Plate 4: Folawiyo Tank Farm

2.4 Sampling

Water samples were collected at each sampling station at a depth of 2m below the water surface with a pre-cleaned 1L glass bottles. Surface sediment samples were collected with a stainless steel eckman bottom sampler [22]. The top 20cm of the bottom sediments were carefully removed wrapped in a foil paper before being stored in glass vials. Surface soil samples were collected from communities around the sampling stations using a soil auger at a depth of 15cm. The soil samples were wrapped in foil paper before storage in glass vials. Fish samples were collected from the sampling stations, wrapped with foil paper and put in polyethylene bags. Egg samples from free range roosters were collected from communities near the sampling stations. They were wrapped with polyethylene bags and stored in plastic vials. All samples (water, sediments, fish, soil and eggs) were transported to the laboratory in ice coolers. In the laboratory, they were at -20^{0}C until extraction. Samples at each sampling station were collected as composites of 3 sampling areas per station.

2.5 Sample Extraction

The extraction was carried out following the modified standard methods of American Society for Testing Material[23] and United State Environmental Protection Agency[24]. The fish samples 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. A mixture of acetone and petroleum ether was used 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 sub-sampled material was pulverized using the laboratory milling machine of make Janke and Kunkel (IKA Labortechnik) in the laboratory. The pulverized samples were weighed and kept in the fridge/freezer at a temperature that was less than 4^{0}C until analysis. 10.0 g of the weighed sample was extracted after the addition of the surrogate standard solution to the sample and later transferred to the extracting bottle that was cocked with TFE-flourocarbon. 50 ml of the phosphate buffer was added, followed by the pH measurement with the addition of sulphuric acid for pH adjustment. 1.0g of the sodium chloride salt was added to the sample, sealed and shook to dissolve the salt. 20.0ml of the redistilled analytical grade methylene chloride was measured and poured into the sample. The sample was extracted for 30minutes. The extract was filtered into the Erlenmeyer flask. The extraction was repeated two more times with fresh solvent and the filtrate was combined. The combined extract was dried by pouring through a drying column containing a 10-cm column of anhydrous sodium sulphate (previously rinsed with methylene chloride), and the filtrate was concentrated in the concentrator flask with a stream of nitrogen. The wall of the concentrator flask was rinsed with extracting solvent so as to bring the final volume of the extract to 5.0ml. The effluent sample was extracted by measuring 500ml of the sample and the same procedure of extraction as outlined above was followed in the borosilicate separatory funnel. The extraction and clean up of sediments for semi-volatile organics was carried out following the internal standard methods from [24].

2.6 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. The clean up of the concentrated extract was followed by packing the column with the florisil. The concentrated extract was eluted with the Hexane and later concentrated to the required final volume of 5ml.

2.7 Sample analysis

The OCPs residues were analyzed by Hewlett Packard Gas Chromatograph 6890 with a Pulse Flame Photometric Detector (PFPD) and HP ChemStation Software. The following conditions were maintained; injection temperature - Split Injection, Split Ratio - 20:1, Carrier Gas – Hydrogen, Flow Rate - 1.0ml/min Inlet Temperature - 250^{0}C, Column Type - HP 5MS, Column Dimensions -30m x 0.25mm x 0.25µm, Oven temperature program - Initial @ 80^{0}C for 1 minute, First Ramp @ 10^{0}C/min to 200^{0}C, Second Ramp @ 12^{0}C/min to 300^{0}C constant at 1mins, Detector Temperature - 300^{0}C, Hydrogen Pressure - 22psi, Compressed Air - 28psi. 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) was used to identify the compounds present in the samples.

2.8 Analytical quality control

The quality control included the analysis of pre-extracted and spiked lagoon water, sediments, soil, and egg samples (10 µg/L) (N=8) and analysis of samples from each sampling site shows good recoveries as shown in Table 2. All spiked samples consisted of all reagents exposed to all glassware and equipments, were run to check for interference compounds. None of the target compounds were detected.

Table 2: Calibration curve correlated coefficient of the selected intermediate standards

S/N	Compound	Correlation Coefficient
1.	Mecoprop	0.99989
2.	Pentachlorophenol	0.99908
3.	2, 4, DB	0.99952
4.	DDT	0.99969
5.	Endrin	0.99984
6.	Dieldrin	0.99616
Average		**0.99903**

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3. Results

The results of the detection of POPs in water, sediment, fish, soil and eggs are shown in Table 3. The values shown indicate the range, mean and standard deviation of the concentration of POPs in the various samples collected. The total POPs concentration for all the sampling stations ranged from 2.12 - 5.66µg/L (water), 8.55 - 15.31µg/kg (sediment), 3.83 - 10.96µg/kg (fish), 1500.84 - 2495.73µg/kg (soil) and 3.92 - 9.56µg/kg (egg). The highest concentrations of individual POPs were 4.53µg/kg (Endosulfan)(Apapa-water), 6.28µg/L (Endrin)(Ilaje-sediment), 5.83µg/kg (Endosulfan)(Ilaje-fish), 1710.06µg/kg (Endosulfan)(Apapa-soil), 4.42µg/kg (Endosulfan)(Ilaje-egg). The mean concentration of sum POPs for Ilaje, Oko-oba, Iddo and Apapa sample stations were 0.23±0.69µg/L, 0.23±0.69µg/L, 0.13±0.28µg/L and 0.33±1.08µg/L (water), 0.85±1.58µg/kg, 0.81±1.41µg/kg, 0.50±0.96µg/kg and 0.48±0.96µg/kg (sediment), 0.65±1.47µg/kg, 0.56±1.10µg/kg, 0.22±0.42µg/kg and 0.31±0.51µg/kg (fish), 138.65±548.66µg/kg, 88.46±352.82µg/kg, 83.38±334.64 and 99.10±402.15µg/kg (soil) and 0.53±1.08µg/kg, 0.43±0.83µg/kg, 0.23±0.33 µg/kg, and 0.22±0.30µg/kg (eggs) respectively. The concentration of total POPs at the different sampling stations increased in this order; 2.12µg/L (Iddo), 3.88µg/L (Oko Oba), 3.96µg/L (Ilaje) and 5.66µg/L (Apapa) for water samples; 8.55µg/kg (Apapa), 8.94µg/kg (Iddo), 14.6µg/kg (Oko-Oba) and 15.31µg/kg (Ilaje) for sediment samples; 3.83µg/kg (Iddo), 5.29µg/kg (Apapa) and 10.96µg/kg (Ilaje) and 9.58µg/kg (Oko Oba) for Fish samples; 1500.84µg/kg (Iddo), 1592.31µg/kg (Oko Oba), 1783.72µg/kg (Apapa) and 2495.75µg/kg (Ilaje) for Soil samples and 3.92µg/kg (Apapa), 4.12µg/kg (Iddo), 7.81µg/kg (Oko Oba) and 9.56µg/kg (Iddo). The concentration of total POPs in the various media increased in this order; 3.91µg/L (water), 6.36µg/kg (egg), 7.41µg/kg (fish), 11.85µg/kg (sediment) and 1843.16µg/kg (soil). Table 4 shows POPs in water samples and World Health Organization (WHO) guideline limits for water quality. The total concentration of POPs in the sampling stations is shown in Figs. 2-6.

Table 3: Total OCPs concentrations in water, sediment, fish, egg and soil from selected areas of the Lagos lagoon

Compound	Range	Water (µg/L) Mean± SD	Range	Sediment (µg/kg) Mean ± SD	Range	Fish (µg/kg) Mean ± SD	Range	Soil (µg/kg) Mean ± SD	Range	Egg (µg/kg) Mean ± SD
Mecoprop			0.058-0.061	0.059±0.001			0.044-0.058	0.055±0.007	ND-0.002	0.002±0.001
Atrazine	0.058-0.059	0.058±0.000	0.214-0.218	0.216±0.002	0.058-0.059	0.058±0.000	0.200-0.214	0.210±0.007	0.070-0.073	0.071±0.001
2, 4-D	0.056-0.057	0.057±0.001	0.211-0.215	0.213±0.002	0.056-0.057	0.057±0.001	0.200-0.211	0.208±0.005	0.068-0.071	0.069±0.001
Carbofuran	0.01-0.011	0.011±0.001	0.097-0.101	0.099±0.002	0.010-0.011	0.010±0.001	0.083-0.097	0.094±0.007	0.022-0.025	0.023±0.002
DBCP	0.073-0.074	0.074±0.001	0.228-0.234	0.232±0.003	0.073-0.074	0.074±0.001	0.214-0.228	0.225±0.007	0.085-0.088	0.086±0.001
Pentachlorophenol	0.077	0.077±0.000	0.257-0.260	0.259±0.001	0.077	0.077±0.000	0.243-0.257	0.254±0.007	0.088-0.091	0.089±0.002
Fenoprop	0.027	0.027±0.000	0.180-0.181	0.181±0.001	0.027-0.028	0.028±0.001	0.178-0.180	0.180±0.001	0.153-0.154	0.153±0.001
Alachlor	0.016-0.097	0.055±0.039	0.037-0.059	0.042±0.011	0.186-3.632	2.033±1.439	1.046-2.412	1.728±0.613	0.009-0.084	0.032±0.035
2,4-DB	0.004-0.005	0.005±0.001	0.218-0.220	0.219±0.001	.0004-0.040	0.014±0.018	0.220	0.220±0.000	0.177-0.148	0.155±0.014
Metolachlor	0.069	0.069±0.000	0.083-0.084	0.084±0.001	0.068-0.069	0.069±0.001	0.083	0.083±0.000	0.069-0.083	0.073±0.007
Lindane	0.085	0.085±0.000	0.116-0.425	0.266±0.170	0.129-0.804	0.313±0.000	1.998-36.664	19.732±20.43	0.608-0.820	0.730±0.091
Chlorpyrifos	0.079	0.079±0.000	0.108	0.108±0.000	0.078-0.079	0.079±0.252	0.011-1.081	0.327±0.505	0.079-0.108	0.086±0.015
DDT	0.073	0.073±0.000	0.625-2.646	1.683±1.074	0.673	0.673±0.096	9.766-12.291	11.384±1.148	0.258-0.450	0.364±0.080
Aldrin	0.055-0.093	0.072±0.016	0.633-1.401	0.942±0.341	0.370-0.932	0.666±0.038	1.153-16.333	7.643±7.589	0.021-0.715	0.347±0.354
Endrin	0.116-0.169	0.120±0.046	4.124-6.278	5.061±1.072	0.039-0.250	0.148±0.096	3.444-95.398	35.854±43.29	1.105-1.919	1.368±0.378
Chlordane	0.103-0.135	0.122±0.015	0.061-0.064	0.208±0.290	0.103-0.192	0.148±0.038	1.319-3.080	2.020±0.769	0.175-0.398	0.313±0.097
Endosulfan	1.2-4.534	2.889±1.361	0.156-0.334	0.243±0.090	1.088-5.832	2.945±2.121	1423.69-2335.22	1742.635 ± 413.141	0.581-4.423	2.331±1.910
Dieldrin	0.013-0.060	0.034±0.020	1.020-2.573	1.737±0.772	0.023-0.026	0.024±0.002	14.741-27.758	20.307±5.453	0.064-0.070	0.066±0.005
Sum POPs	2.120-5.662	3.905±1.500	8.545-15.307	11.849±3.832	3.828-10.959	7.414±4.296	1500.84-2495.73	1843.157 ± 492.985	3.921-9.562	6.355±2.994
Internal Standard	0.060	0.060±0.000	0.060	0.060±0.000	0.060-0.061	0.060±0.001	0.060	0.060±0.000	0.060	0.060±0.000

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Table 4: Show POPs in water samples and World Health Organization (WHO) guideline limits for water quality

Compound	WHO guideline limits (µg/L)	POPs in water sample (µg/L)	Remarks
Mecoprop	0.01	ND	Below limits
Atrazine	0.01	0.058±0.000	Above limits
2, 4-D	0.5	0.057±0.001	Above limits
Carbofuran	0.1	0.011±0.001	Above limits
DBCP	0.02	0.074±0.001	Above limits
Pentachlorophenol	0.01	0.077±0.000	Above limits
Fenoprop	0.2	0.027±0.000	Above limits
Alachlor	0.1	0.055±0.039	Below limits
2,4-DB	1.0	0.005±0.001	Below limits
Metolachlor	.01	0.069±0.000	Above limits
Lindane	0.01	0.085±0.000`	Above limits
Chlorpyrifos	1.0	0.079±0.000	Below limits
DDT	0.011	0.073±0.000	Above limits
Aldrin	0.03	0.072±0.016	Above limits
Endrin	0.6	0.120±0.046	Below limits
Chlordane	0.014	0.122±0.015	Above limits
Endosulfan	0.6	2.889±1.361	Above limits
Dieldrin	0.03	0.034±0.020	Above limits

ND – Not detected

Fig. 2: Total Concentrations of POPs in water samples at different sampling stations

Fig. 3: Total Concentrations of POPs in sediment samples at different sampling stations

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Fig. 4: Total Concentrations of POPs in fish samples at different sampling stations

Fig. 5: Total Concentrations of POPs in soil samples at different sampling stations

Fig. 6: Total Concentrations of POPs in egg samples at different sampling stations

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4. Discussion

This study is intended to support the development and implementation of risk management strategies that will ensure the safety of the Lagos Lagoon through a scientific basis for the control of POPs constituents present in the Lagoon.

Station D (Apapa) had the highest concentration of endosulfan in water samples (Fig. 2). This could be as a result of a higher concentration of industrial activities at this location.

Sediment samples had relatively high concentrations of DDT, Andrin, Endrin and Dieldrin in all the sampling stations (Fig. 3). This indicates the indiscriminate use of these harmful POPs which are broad spectrum insecticides and also used for wood protection.

The high concentrations of endosulfan, alachlor, aldrin, DDT and lindane in fish samples (Fig. 4) indicates that large quantities of these substances are used, and which are subsequently absorbed by fish and other aquatic organisms in the water body. These ultimately on the long - term could result in damage to vital organs in humans.

A high concentration of endosulfan was observed in soil samples in all the stations (fig. 5). This indicates high pesticide which is absorbed in soil and which is deleterious to human health.

Eggs had relatively high concentrations of endosulfan, chlordane, endrin, aldrin, DDT and lindane (Fig. 6). This concentration indicates the presence of these substances in levels that could be injurious to human health. Mecoprop which is mainly used as a herbicide was not recorded for water and fish samples. In comparison with WHO guidelines for water quality, it was observed that most of the POPs were above the recommended maximum limits. This reveals pollution which is in accordance with Nwankwoala and Osibanjo[25], who reported a general contamination of African inland surface waters by a broad spectrum of Organochlorine pesticides (OCPs). This view is also supported by Ize-iyamu *et al*[26] who discovered that pesticides were bio-accumulated at the bottom of the Lagoon.

5. Conclusion and Recommendations

This study has shown that water, sediment, fish, soil and egg samples collected from the Lagos Lagoon are contaminated with varying amounts of persistent organic pollutants. Thus investigations of sources and point of entry of these residues as well as awareness programmes of the harmful effects of these pesticides especially in fish and eggs which serve as food are recommended. There is need for regular surveillance and policing of the lagoon and enforcement of relevant legislation against polluters of the lagoon by the Lagos State Environmental Protection Agency.

There is need for the neighboring communities and inhabitants to become more vigilant and aware of the need to protect the Lagoon. Further studies of pesticide residues in blood serum of the inhabitants of communities surrounding these selected areas are recommended.

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