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Full Length Research Paper

Evaluation of the levels of organochlorine pesticide residues in water samples of Lagos Lagoon using solid phase extraction method

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Water samples were collected from 6 different locations of the Lagos lagoon at 0.5 and 2.5 m depths, respectively and from 2 other river bodies in Lagos and analyzed for the presence of 9 OCPs (organochlorine pesticides) residues. Samples were extracted using solid phase extraction method and were analyzed with gas chromatography. The analytes quantified included chlordane (0.006 to 0.950 µg/L), heptachlor (N.D: 0.067 µg/L), methoxychlor (N.D: 0.123 µg/L), hexachlorobenzene (0.015 to 0.774 µg/L), endosulfan (0.015 to 0.996 µg/L), dichlorodiphenyltrichloroethane (0.012 to 0.910 µg/L), Dichlorodiphenyldichloroethylene (0.005 to 0.477 µg/L), dieldrin (0.015 to 0.996 µg/L) and aldrin (0.080 to 0.790 µg/L). The ratio of DDE/∑DDT obtained was 0.812 and this indicates that the degraded metabolite formed a significant proportion of the total DDTs. The overall mean total concentration of aldrin was the highest while heptachlor was the lowest. Concentration of methoxychlor and heptachlor were relatively low and below the detection limit in 50 and 57% of samples analysed respectively. The mean concentrations of organochlorine pesticides were higher than European Community allowable residual limit (0.1 µg/L) for individual OCPs in drinking water in 37.3% of samples analyzed.

Key words: Organochlorine, pesticides, Lagos, gas-chromatography, solid phase extraction.

INTRODUCTION

Pesticides are synthetic organic compounds that despite their benefits also pose considerable hazards to the environment. They are broadly divided into many classes of which the most important are organochlorine pesticides (OCPs). The organochlorine pesticides are broad spectrum insecticides, and are the most widely used in many countries including Nigeria for agricultural purposes and control of mosquitoes (Bouman, 2004; Blaso et al., 2005). Organochlorine pesticides are very stable compounds and it has been cited that the degradation of dichlorodiphenyltrichloroethane (DDT) in soil ranges from 4 to 30 years, while other chlorinated stable for many years after application, due to a high resistance to chemical and biological degradations (Afful et al., 2010). The chemicals are very persistence lipo-soluble compounds and are capable of bioaccumulating in the fatty parts of biological beings such as breast milk, blood and fatty tissues (William et al., 2008) via the food chains. As a result of its position in the food chain (end of the food chain), man is greatly exposed to the effect of the micropollutants by eating foods either coming from contaminated earth or water (Belta et al., 2006; Raposo et al., 2007).

Organochlorine pesticides have become ubiquitous contaminant and have been implicated in a broad range of deleterious health effects in laboratory animals and man. The toxic effect include reproductive failures (Bouman, 2004), immune system malfunction (Kolpin et al., 1998), endocrine disruption (Ize et al., 2007) and

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breast cancers (Garabrant et al., 1992). Previous studies have shown that DDT 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).

The ability of the most prevalent metabolite of DDT, dichlorodiphenyldichloroethylene (DDE) to bind to androgen receptor in animals has been reported (Saxena et al., 1981), while a long exposure to dieldrin and aldrin could results in chronic convulsions (Belta et al., 2006). Epidemiological reports have also suggested an etiological link between exposure to organochlorine pesticides and Parkinsons diseases (Fleming et al., 1994). The aquatic environment often serves as a sink for many potentially harmful organic pollutants emitted from industrial and domestic sources and contamination of water bodies by pesticide residues has been an issue of serious concerns due to the health risks associated with them (Golfinopoulos et al., 2003).

The US-EPA has set allowable residual limit of individual and total concentration in drinking water set at 0.1 and 0.5 µg/L, respectively. Despite the ban on the production and use of OCPs in accordance with Stockholm convention in 2001 (Ennacer et al., 2008) and replacement with less persistent organophosphates and carbamates, some developing countries have resumed the use of OCPs such as DDT, because of its early spectacular success in malaria eradication and fighting of life-threatening typhus. Although, so far there is no local production in Nigeria, over 100 different brands of pesticides are being imported in to the country. It is worth noting that dieldrin, aldrin and other organochlorine pesticides are still in use for the control of pest of cotton, cocoa, fruits, cereals and vegetables (Ize et al., 2007).

Water bodies are a key recipient of xenobiotic contaminants such as pesticides in the ecosystem (Barcelo, 1991). These contaminants can enter water either through point sources like sewage plants, sewer overflows and poor management practices of farmers or through diffuse sources such as atmospheric deposition, run offs and leaching from agricultural applications (Guruge et al., 2001; Mayon et al., 2006; Shukla et al., 2006). The environmental persistence and toxicity of OCPs have led to its ban in many countries and are in the priority list of European Community.

Pesticides are often found in trace quantities in complex matrices and for efficient study of fate of pesticides in natural waters, very low detection limit must be reached. As a result, both efficient sample preparation and a high performance analytical instrument are required for accurate determination at trace levels in which they occur in complex matrices. The determination of pesticide residues in water is necessary for solving various environmental and biological problems. The pollution of water bodies in Nigeria (Lagos lagoon especially) through agricultural activities and direct deposition is on the rise. Meanwhile, a large proportion of the populace depends on it for potable and recreational water, as well as a source of cheap and affordable protein in form of fish. There are general reports on ground water contami-nation from pesticides in the country (Nwankwoala and Osibanjo, 1992; Ize et al., 2007) but relatively few studies have focused on samples from Lagos lagoon.

The objective of this study was to evaluate the spectrum of organochlorine pesticides residues in water samples of Lagos Lagoon by solid phase extraction method and analysis with GC-ECD. The OCPs investigated include 1,1,1-trichloro-2,2-bis(4-chlorophenyl)ethane (DDT), 1,1-dichloro-2,2-bis (*p*-chlorophenyl)ethylene (DDE), 1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a,octahydro-1,4,5,8-dimethanonaphthalene (Dieldrin), Hexachlorobenzene (HCB), 1,4,5,6,7,8,8-Heptachloro-3a,4,7,7a-tetrahydro-4,7-methano-1H-indene (heptachlor), 1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-dimethanonaphthalene, (aldrin), 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepine-3-oxide (endosulfan), octachloro-4,7-methanohydroindane (chlordane), and 1,1,1-trichloro-2,2-bis(4-methoxyphenyl)ethane (methoxychlor).

MATERIALS AND METHODS

Sampling area

Lagos is a port and populous conurbation city in Nigeria with more than 12 million people and estimated to be the fastest growing city in Africa. Lagos lagoon (Figure 1) is about 50 km long and 3 to 13 km wide, separated from Atlantic Ocean by long sand and spit 2 to 5 km wide with swampy margins on the Lagoon side. Lagos Lagoon empties into the Atlantic via the Lagos harbor, a main channel through the heart of the city, 0.25 to 1 km wide and 10 km long. The principal ocean port of Lagos is located at Apapa in a broad western branch off the main channel of the harbor. Lagos Lagoon is fairly shallow and the city spread along more than 30 km of the Lagoons South western and western shoreline. The area west of the lagoon is not well provided with road, and many communities there traditionally relied on water transport.

Chemicals and reagents

The OCPs standards; aldrin (98.1%), chordane (98.43%), DDE (99.5%), DDT (99.6%), dieldrin (97.9%), β-endosulfan (99.9%), heptachlor (99.7%), methoxychlor (99.5) and hexachlorobenzene (99.6%) were obtained from Supelco (Belle–Fonte, USA). The stock solutions of each analyte (1.0 mg/L) were prepared in acetone and a fresh working solution containing a mixture of each analyte was prepared by stepwise dilution of the stock solution. Solvents used were of pesticide grade standard. HPLC/UV grade acetone, dichloromethane and n-hexane were purchased from Merck Chemical (Bonn, Germany). Ultra high purity water was generated from a Millipore alpha-Q-system supplied by Millipore (Molsheism, France). Granular anhydrous sodium sulfate (AR grade) was heated to 400°C for 4 h prior to use. C-18 SPE catridges(62ESEB) containing ultra pure silica gel were obtained from Gueboc OC, Canada. The standard solution of OCPs, each containing the target

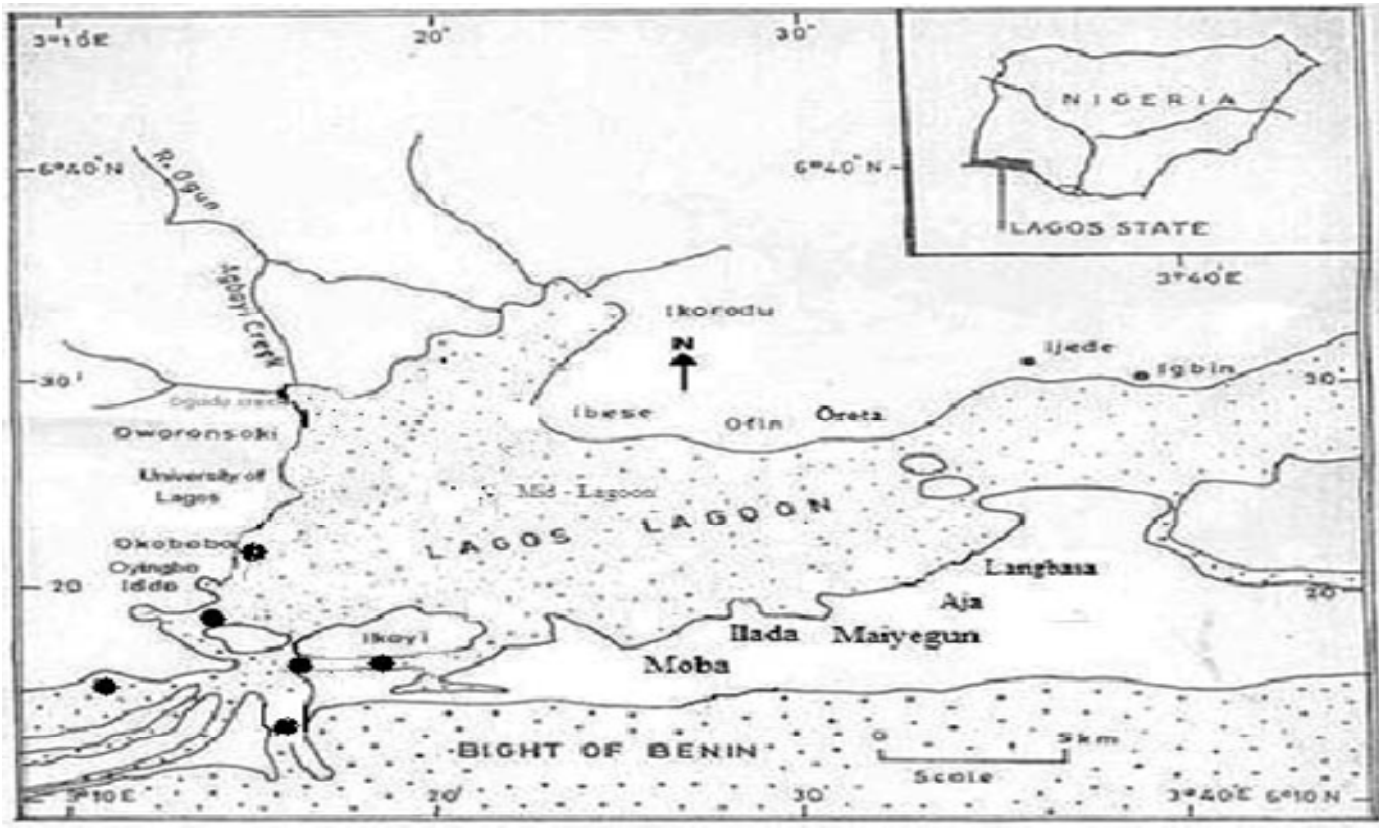

Figure 1. Sketch of the Map of Lagos lagoon; Represents the sampling points.

analyte (Aldrin, chlordane, DDE, DDT, dieldrin, endosulfan, heptachlor, methoxychlor and hexachlorobenzene) were prepared by diluting the standard mixtures to a desired concentration (0.1 to 2.0 µg/L) with n-hexane.

Extraction procedure

Water samples were collected from 6 different locations during a high tidal in Lagos lagoon at 0.5 and 2.5 m depth and from 2 other river bodies within Lagos metropolis which are quite distant from the Lagoon with low instances of intense Agricultural practices and industrialization (thereafter referred to as OKB, AP, OB, VI, EE, CMS, Ag and Aj, between March and April, 2010) in a pre-cleaned glass jars. Global positioning system (Global tech Ltd, Hong Kong) was used to identify the location where samples were collected were collected, while the physicochemical parameters of water samples including total suspended solids, pH, conductivity, turbidity, salinity, alkalinity, temperature and dissolved oxygen (Table 1) were measured with Horiba U-10 (Horiba Ltd, Japan) multiparameter water quality metres. On arrival in the laboratory, samples were filtered through 0.45 µm glass filter (Whatman GF/F) to remove particulate matter, acidified with dilute hydrochloric acid to pH 2.5 and stored in the dark at temperature between 0 to 4°C prior to extraction. SPE using pre-packed reversed-phase octadecyl (C-18 bonded silica) contained in cartridges was used for sample extraction. Methanol (5 ml) was added to the water samples to aid extraction. Prior to the extraction, the C-18 bonded phase, which contains 500 mg of bonded phase was conditioned with 5 ml methanol and equilibrated with 2 ml ultra high purity water. 10 ml of the sample was passed at a rate of 0.3 ml/min through the conditioned cartridge. The analyte was allowed to percolate through the cartridge under vacuum for 15 min after which time they were eluted with 5 ml n-hexane. The eluate was evaporated to complete dryness under a gentle stream of nitrogen gas and analyte was reconstituted in 0.5 ml n-hexane for GC analysis. For recovery studies, 10 ml aliquots of ultra high purity Milli-Q water samples (pH adjusted to 6.5) and spiked with standard solutions (0.05, 0.5 and 1.5 µg/L) of the analytes were similarly extracted in triplicates as described (Spyros et al., 2003).

Gas chromatography analytical conditions

Analyses were performed on an Agilent GC-6820 (Santa Clara, C. A, U.S.A) equipped with ^{63}Ni electron capture detector (ECD). Zebron ZB-multiresidue-1 column (30 × 0.25 mm i. d × 0.25 µm film thickness) manufactured by phenomenex (Torrence, USA) was employed in the separation of the analytes. Helium was used as the carrier gas while nitrogen was used as the make-up gas. The analytical conditions are as shown in Table 2. A 1 µl volume was injected in a splitless mode. The residues of organochlorine pesticides were determined by comparing the peak areas of the samples and the calibration curves of the standards.

Quality assurance

For every set of 10 samples, a procedural blank and spiked sample consisting of all reagents was run to check for interference and cross-contamination. The range of linearity of the detector was evaluated from the curves generated by plotting the detector signal

Table 1. Phsicochemical parameters of water samples. Each sampling location was marked with coordinate. [X;] Water samples collected from other water bodies in Lagos. OKB; Oko baba, AP (Apapa), OB (Obalende), VI (Victoria Island), EE (Ebute- Ero),CMS (CMS), Ag (Agbelekale), Aj (Ajasa). Value in bracket represents the depth at which the water is collected in metres.

Sample code	Coordinate	Total suspended solid (Mg/L)	pH	Conductivity (S/M)	Turbidity (Mg/L)	Salinity (Mg/L)	Alkalinity	Temperature (℃)	Dissolved oxygen (Mg/L)
OKB (0.5)	$06^{0}28^{1}56^{11N}$	10	7	31.5	12	20.1	20	29	1.5
OKB(2.5)	$03^{0}23^{1}34^{11E}$	45	8	31.8	77	20.1	22	29	1.8
AP(0.5)	$06^{0}26^{1}11^{11N}$	7.5	7	21.8	45	17.5	18	30	1.7
AP(2.5)	$03^{0}21^{1}39^{11E}$	21	8	28.8	59	18.0	24	29	1.6
OB (0.5)	$06^{0}26^{1}02^{11N}$	5.0	8	28.6	4	17.8	20	30	1.2
OB(2.5)	$03^{0}21^{1}52^{11E}$	8.0	8	32.4	3	2.4	20	29	1.6
VI(0.5)	$06^{0}21^{1}57^{11N}$	1.0	8	28.4	10	17.5	22	31	1.6
VI(2.5)	$08^{0}22^{1}05^{11E}$	8.0	8	37.1	6	23.8	22	30	2.3
EE(0.5)	$06^{0}27^{1}16^{11N}$	6.0	8	34.7	3	20.1	20	31	6.8
EE(2.5)	$03^{0}23^{1}03^{11E}$	8.0	8	34.2	1	21.8	16	30	3.0
CMS(0.5)	$06^{0}28^{1}56^{11N}$	8.0	8	29.6	13	18.4	20	29	3.2
CMS(2.5)	$03^{0}23^{1}34^{11E}$	2.9	8	32.9	1	21.8	18	28	1.7
Ag(0.5)[x]	$05^{0}22^{1}24^{11N}$	13	9	27.6	13	16.7	16	32	1.5
Aj (0.5)[x]	$04^{0}23^{1}34^{11N}$	21	8	25.4	47	15.8	28	31	1.6

Table 2. Gas chromatograph conditions.

Conditions	Values
Career gas flow	2 mL/min
Make up gas flow	20 mL/min
Oven temperature	50℃ (1 min). - 120℃ (2 min) at 30℃/min - 250℃ (5 min) at 15℃/min - 280 ℃ (5 min) at 30℃/min
Injector temperature	280℃
Detector temperature	300℃

versus the amount injected.

RESULTS AND DISCUSSION

Analytical quality assurance

The GC chromatogram of 2.0 µg/L OCPs standards mixture is as shown in Figure 2. The method detection limits of OCPs in samples were determined as the concentration of analyte in a sample that gives rise to a peak with a signal-to-noise ratio (S/N) of 3. The detection limit was lowest for methoxychlor (0.004 µg/L) and highest for dieldrin (0.012 µg/L), while the detection limit of other analytes lies within the range. The average recoveries (n = 3) for OCPs through the analytical procedures (laboratory treatment) was 82.5 to 100.2% as determined by spiking OCPs mixtures in ultrapure milli-Q water samples. The relative standard deviations (RSD) were below 6.0%. They all met the requirement of US-EPA (Recovery: 70 to 130%, RSD is < 30%) showing that the analytical protocols used in this study can effectively determine OCP residue in water. The quantitative data were however not corrected for surrogate recoveries.

Concentrations of OCPs

The sample location and the relative values of physico chemical parameters of water samples are as shown in Table 1, while the mean concentration of OCPs residues in water samples are as shown in Table 3. Turbidity is a measure of cloudiness, while total suspended solids is a measure of suspended particles and salinity is a measure of dissolved salt content of the water samples. Total suspended solids are relatively higher at OKB and CMS while turbididy were relatively higher at OKB, AP and AJ. The temperature of the water bodies lies between 28 to 32℃, while the pH of the samples was either neutral or slightly alkaline. It appears that there is no correlation

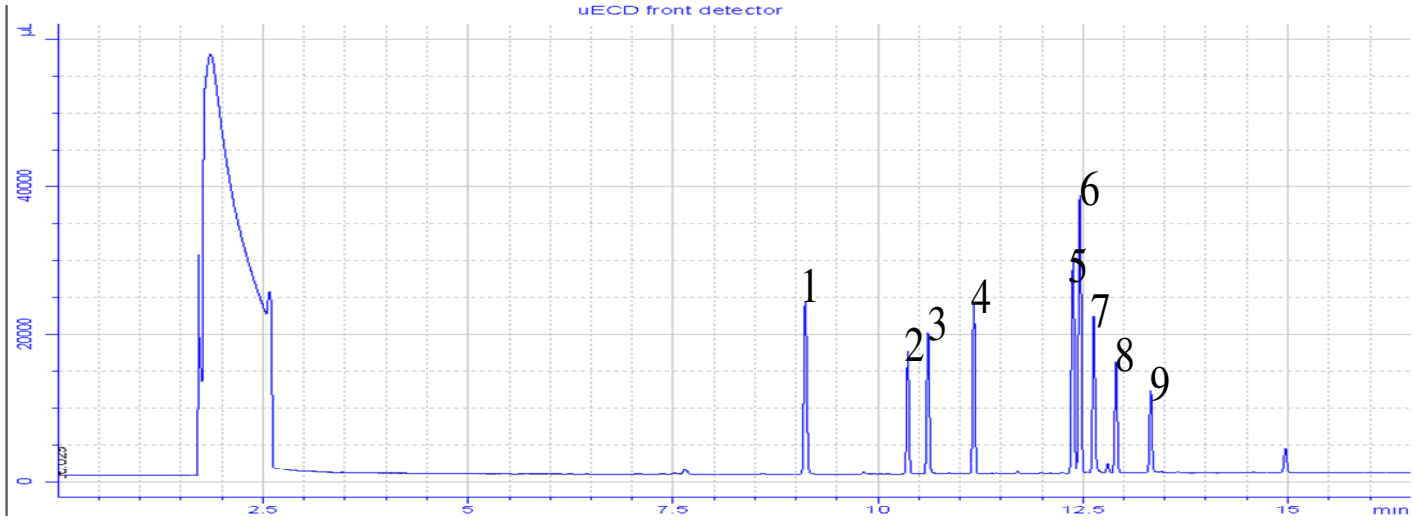

Figure 2. Gas-Chromatogram of OCPs standard mixture (2.0 µg/L): 1, DDT, 2, Dieldrin, 3, DDE, 4, Chlordane, 5, heptachlor, 6, HCB, 7, Endosulfan, 8, Methoxychlor, 9, Aldrin.

Table 3. Mean concentrations of OCPs residues (µg/L) at various locations sampled.

Sample code	DDT	DDE	Dieldrin	Aldrin	Chlordane	Heptachlor	Methoxychlor	HCB	Endosulfan
OKB(0.5)	0.049	0.030	0.037	0.417	0.006	0.032	ND	0.015	0.084
OKB(2.5)	0.056	0.005	0.038	0.287	0.007	ND	ND	0.015	0.067
AP(0.5)	0.910	0.063	0.954	0.128	0.818	0.060	0.032	0.608	0.936
AP (2.5)	0.245	0.073	0.015	0.270	0.904	0.040	0.005	0.034	0.616
OB(0.5)	0.368	0.324	0.150	0.359	0.342	0.020	ND	0.488	0.660
OB(2.5)	0.032	0.087	0.996	0.295	0.950	0.064	0.038	0.774	0.996
VI(0.5)	0.032	0.078	0.017	0.402	0.078	ND	0.005	0.048	0.068
VI(2.5)	0.095	0.061	0.071	0.637	0.312	0.052	0.052	0.077	0.298
EE(0.5)	0.026	0.123	0.235	0.125	0.010	ND	ND	0.034	0.050
EE(2.5)	0.012	0.477	0.137	0.736	0.008	ND	ND	0.156	0.236
CMS(0.5)	0.083	0.270	0.780	0.790	0.034	ND	ND	0.320	0.200
CMS(2.5)	0.071	0.064	0.296	0.700	0.009	ND	ND	0.034	0.050
Ag(0.5)^{x}	0.051	0.030	0.076	0.080	0.012	0.067	0.016	0.087	0.015
Aj(0.5)^{x}	0.052	0.032	0.140	0.100	0.009	0.061	0.123	0.532	0.321

N.D; Non detectable; *: Water samples collected from other water bodies in Lagos. OKB: Oko baba, AP: Apapa; OB: Obalende; VI: Victoria Island; EE: Ebute- Ero; CMS: CMS; Ag: Agbelekale; Aj: Ajasa; Value in bracket represent the depth at which the water is collected in metres.

between the organochlorine pesticides residues distribution and the measured physicochemical parameters of the samples.

The results indicate that 88% of samples analysed recorded positive for the presence of OCPs. Substances such as chlordane, endosulfan, hexachlorobenzene, dieldrin and aldrin were the pesticides detected in larger concentration (0.001 to 0.996 µg/L), while heptachlor, methoxychlor, DDT and DDE were detected in lower concentrations (< 0.368 µg/L). On the whole, the overall mean total concentration of aldrin was the highest while overall mean total concentration of heptachlor was the lowest. The concentration of heptachlor and methoxychlor were below the detection limit in 57 and 50% of samples analysed respectively. 37.3% of samples analyzed recorded mean concentrations higher than the European Community allowable residual limits in drinking water set at 0.1 µg/L.

In this study, the concentration of OCPs detected in water samples collected from other sources (freshwater) in Lagos were generally lower compared to samples from Lagos lagoon. This can be attributed to the fact that these other water bodies are located near residential areas where agricultural and industrial activities are very low. The mean concentration of OCPs detected in water samples were much lower than was detected in our

Table 4. Comparison of the mean concentration of OCPs (µg/L) residues in water samples from Lagos Lagoon with other water bodies in some other cities elsewhere.

OCPs	Lagos lagoon (Present study)	Ibadan (Nwakwoala et al., 1991)	Culturama Brazil (Raposo Junior et al., 2007)	Hyderabad city, India. (Shukla et al., 2005)	Okavango Delta, Botswana (Mmualefe et al., 2009)
HCB	0.015-0.217	N.D-0.092	N.D-0.0325	-	61.4
Heptachlor	ND- 0.022	0.004-0.202	N.D-0.0046	-	-
Aldrin	0.08-0.375	N.D-0.04	N.D-0.00116	-	-
Endosulfan	0.015-0.355	N.D-0.43	N.D-0.003	0.21-0.87	-
Dieldrin	0.017-0.311	0.018-0.657	N.D-0.0016	-	-
DDT	0.012-0.169	N.D-1.3	N.D-0.0023	0.15-0.19	-
Chlordane	0.006-0.300	-	N.D-0.0037	-	3.2
DDE	0.005-0.169	-	N.D-0.0149	-	5.3

previous studies (Adeyemi et al., 2008) on fish samples from the same lagoon. This outcome is expected because of the high lipophilic and hydrophobic nature of the compound, and the possibility of being retained on the organic phase of sediment and organisms (Sarka et al., 1997).

The concentrations of OCPs in water samples from Lagos lagoon were also compared to water bodies in other parts of the world as shown in Table 4. The mean concentrations of OCPs in water samples in this study were lower compared to levels detected in water samples from Hyderabad City in India (Shukla et al., 2006) and Okavango Delta in Botswana (Mmualefe et al., 2009) but were higher than those detected in previous studies on water bodies from Ibadan, Nigeria (Nwankwoala et al., 1992) and Culturama in Brazil (Raposo et al., 2007). Although the use of the OCPs has either been banned or restricted, same cannot be said about developing nations like Nigeria. The OCPs are still being used in developing countries such as Indian, Botswana and Nigeria for disease control and increased food production for the growing population.

Relatively low levels of DDT detected could be as a result of metabolism of DDT to DDE. The relative concentration of the parent DDT compound and its biological metabolite DDD and DDE are often used as indices for assessing the possible sources and time of application (Hites and Day, 1992). This is because DDT can be biodegraded to DDE and DDD under aerobic and anaerobic condition respectively. In this study, the ratio DDE/∑DDT was greater than 0.5, which is an indication of less tendency of recent exposure to new sources of DDT and accumulation was probably through indirect sources such as long range transport or historical application (Hong et al., 1999; Zhang et al., 1999). Furthermore, the result reveals a wide-range distribution of the OCPs in the sampled location and the possibility of contamination arising from point sources by way of direct discharge and recent applications of most of the OCPs through agricultural run offs. Since the improvement in crop yield by pesticides application is always concomitant with the occurrence of pesticides residues in soil and water samples, there is need for regulatory control on application and point sources of pesticides in order to forestall serious health hazards on the environment.

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