Full Length Research Paper

Levels and distribution of chlorinated pesticide residues in water and sediments of Tarkwa Bay, Lagos Lagoon

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Microlayer water, mixed layer water, epipellic and benthic sediments were collected from Tarkwa Bay end of Lagos Lagoon to analyse for chlorinated pesticide residues. Sampling was conducted between December 2008 and September 2009 during the dry and wet seasons to study effects of seasonal variation on the samples. Water samples were subjected to liquid-liquid extraction using dichloromethane while sediment samples were subjected to cold extraction with petroleum ether/acetone (1:1 v/v) mixture and clean-up on silica gel adsorbents. The water and sediment samples were analysed for aldrin, dieldrin; endrin, DDT, heptachlor and their metabolites; HCH and endosulfan isomers; α, γ-chlordane and methoxychlor. The detection and determination of the pesticide residues were performed by gas chromatography. Pesticide residues in the epipellic and benthic sediments were higher than residues in the water. The microlayer water showed reduced levels of residues when compared with the mixed layer water. The residue levels were higher during the dry season than the wet season and levels of residues in the water and sediments were within the permissible limits.
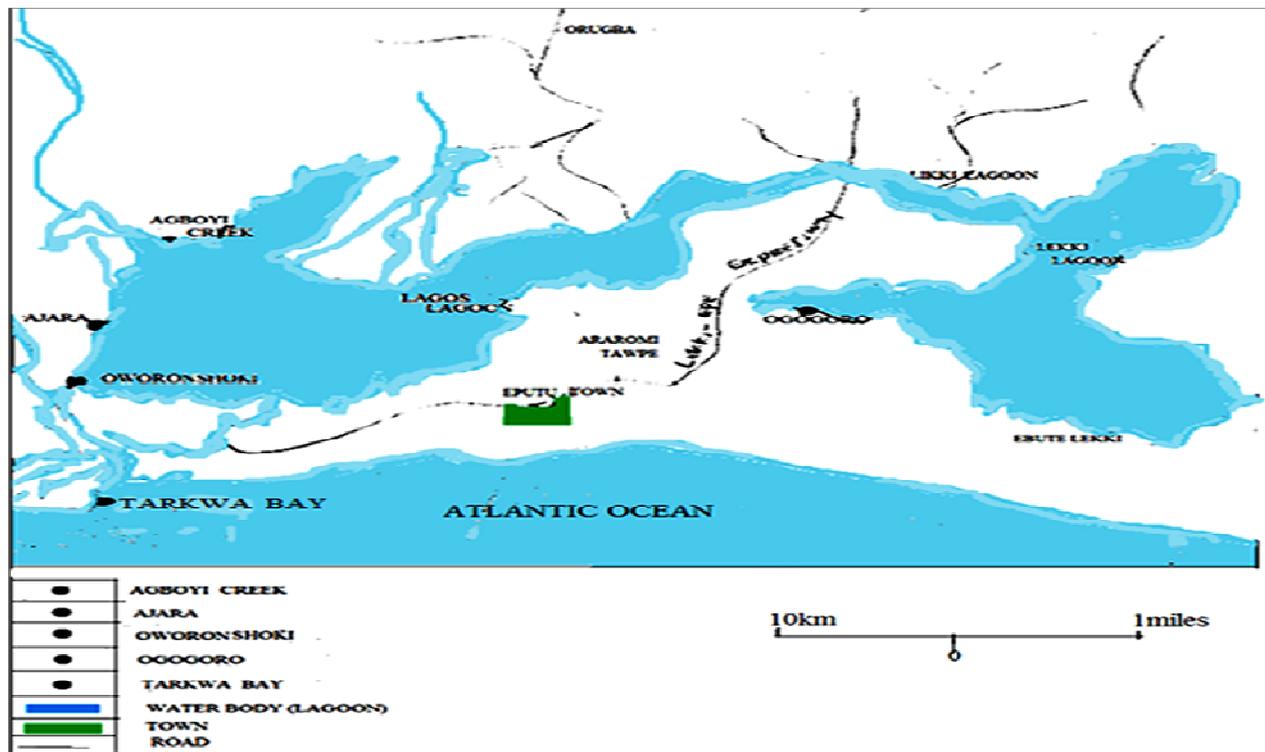
Keywords: Tarkwa Bay, chlorinated pesticides, microlayer water, mixed layer water, epipellic sediment, benthic sediment.

INTRODUCTION

Chlorinated pesticides are persistent organic pollutants which have caused worldwide concern as toxic environmental contaminants (Covacia et al., 2005). They are lipophilic, hydrophobic and are ubiquitous contaminants that have been detected far from their sources of origin because of long-range transport stemming from atmospheric exchange, water currents, animal migration and other pathways (Zhang et al., 2007). Chlorinated pesticides namely aldrin, dieldrin, endrin, chlordane, dichlorodiphenyltrichloroethane (DDT), heptachlor, mirex, toxaphene and hexachlorobenzene (HCB) are among the twelve chemical substances called "dirty dozen" and defined under the Stockholm Convention. The manufacture and use of some pesticides have been banned or restricted in developed countries. However, some developing countries are still using chlorinated pesticides for agricultural and public health purposes (Xue et al., 2006). They are being used

in developing countries including Nigeria due to lack of appropriate regulatory control and management on the production, trade and use of these chemicals (Darko and Acquaah, 2007).

Chlorinated pesticide residues enter aquatic environment through effluent release, discharges of domestic sewage and industrial wastewater, atmospheric deposition, runoff from agricultural fields, leaching, equipment washing, disposal of empty containers and direct dumping of wastes into the water systems (Yang et al., 2005). Pesticide residues could distribute among the components of an ecosystem, such as water and sediment. Due to their persistence, pesticides in water can be transferred into the food chain and accumulate in aquatic organisms like plankton. Pesticides can be bio-concentrated through biogeochemical processes and can be scavenged from the water through sorption onto suspended material before they get deposited to the bottom substrate. The sediment component of aquatic ecosystems deposits pesticides. Sediment is one of the

Figure 1. Map of Lagos Lagoon showing Tarkwa Bay sampling point Source: Author's field survey (http://maps.google.ca/maps)

principal reservoirs of environmental pesticides, representing a source from which residues can be released to the atmosphere, groundwater and living organisms (Xue et al., 2006). Persistence of these organic pollutants in sediment is possible due to their low solubilities and tendency to associate with suspended particulate matter. Due to their low water solubility, chlorinated pesticides have a strong affinity for particulate matter. They are hydrophobic compounds that tend to adsorb to suspended particulate matter and benthic sediments in aquatic ecosystems. Sediments serve as ultimate sinks for them. Indirect exposure to contaminated sediments takes place when fish feeds on benthic invertebrates that are ingesting particulate matter. Direct exposure through the sediment takes place by release of contaminated particulate matter into the water column by both natural and anthropogenic processes. The ill effects from pesticides may result from short or long-term exposure, low or high-level exposure through dermal absorption, inhalation and oral ingestion. Some of the symptoms of pesticides poisoning include irritation, dizziness, tremor, tonic and chronic convulsion (Winter, 1992).

Persistent organic pollutant (POP) contamination is usually monitored by measuring levels either in inorganic ecosystem compartments such as water, air and sediment or in biota. Monitoring in inorganic compartments has the advantage of producing an immediate, geographically localized measure of contamination. Lagos Lagoon is a depository of last resort for a large number of surface runoffs, drainage channels and important rivers flowing from the interior of Southwestern Nigeria to the Atlantic Ocean. The surrounding landmass to the lagoon is also among the most densely populated areas in the country. The introduction of deleterious materials into it is thus expected. The quality of Lagos Lagoon must be constantly monitored. There is a need to monitor concentrations of chlorinated pesticides in pelagic columns in order to assess compliance with the POPs Convention. Given the potential for contamination by pesticides, this study was undertaken to determine the chlorinated pesticide residues in microlayer water, mixed layer water, epipellic and benthic sediments from Tarkwa Bay, Lagos Lagoon, Nigeria.

MATERIALS AND METHODS

Study area

The study area for this investigation is Tarkwa Bay in Lagos Lagoon, which lies between latitude 6° 26' – 6° 37' N and longitude 3° 23' – 4° 20' E on the Western part of Nigeria. Figure1 shows the Map of the lagoon sho-wing Tarkwa Bay sampling point and surrounding areas.

Sampling strategy

Sampling was conducted between December 2008 and September 2009 during the dry and wet seasons to study effects of seasonal variation on the samples. Field investigations were carried out four (4) times at Tarkwa Bay (TBY), where the lagoon discharges into the Atlantic Ocean during the dry season months of December 2008 and February 2009 and the wet season months of May and September 2009. Sampling locations were identified with a hand-held Garmin-GPSMAP 76S-type global positioning system.

Pretreatment of sampling and storage vessels

Prior to sampling, sample bottles and glasswares were washed with detergent, rinsed with distilled water and pure acetone (99.9%) and then dried in an oven overnight at 100^{0}C. Glass containers were used in collecting water samples for chlorinated pesticide residue determination.

Collection of water samples

The microlayer water was collected with clean glass and polythene bottles from a depth of 1 cm while the mixed layer water was sampled with a 5L Goflon water sampler with the aid of a boat. The containers were rinsed three times with the site water prior to collection. Water samples were collected in three labeled amber glass bottles to form composite samples at Tarkwa Bay with coordinate 31N 0543889, UTM 0707582. Filled sample bottles were sealed free of air bubbles with glass stoppers. After collection, samples were properly covered and stored in ice-packed coolers. The water samples were refrigerated in the laboratory at 4°C to inactivate microbes and thus preserve the integrity of the samples (Radojevic and Bashkin, 1999).

Collection of sediment samples

Epipellic sediments were obtained at Tarkwa Bay (31 N 0543893, UTM 0707630) by using a short core sampler to scoop the top 1 to 5 cm of the intertidal sediments. Benthic sediments were obtained at Tarkwa Bay (31 N 0543889, UTM 0707582) with the aid of a Shipek grab sampler. Benthic sediments were collected by lowering the grab sampler into the water bed and raising it aboard after a few minutes. Three grap sediment samples were collected at the site and mixed together to form composite samples and subsequently wrapped in labeled aluminium foil. After collection, the sediment samples were stored in ice-packed coolers to preserve the integrity of the samples. Sediment samples were

refrigerated in the laboratory at 4°C to inactivate microbes. Pebbles, shells and vegetable matter were manually removed.

Treatment of samples

Water and sediment samples were extracted and cleaned up before identification and determination of chlorinated pesticide residues were carried out.

Extraction of water samples

Water samples were subjected to liquid-liquid extraction. $200cm^{3}$ aliquot of water sample was introduced into a $300cm^{3}$ capacity separating funnel and $30cm^{3}$ of HPLC grade of dichloromethane was added. The mixture was well shaken and the stopper removed continuously to release the gas built up in the bottle. The solvent extract was carefully drained into a glass container. The extraction process was repeated with $20cm^{3}$ dichloromethane and the solvent extract was again drained out and combined with the first portion (US EPA Method 3510C, 2007).

Extraction of sediment samples

5g of wet sediment was homogenized with 5g of anhydrous granulated Na_2SO_4 using a mortar and a pestle. Cold solvent extraction (Steinwandter, 1992) was carried out using $50cm^{3}$ HPLC grade petroleum ether/acetone (1:1 v/v) mixtures in a $250cm^{3}$ reagent bottle containing the homogenized sediment sample. The mixture was well shaken and the stopper removed continuously to release the gas built up in the reagent bottle. The mixture was allowed to stand for 30 minutes and then filtered with a filter paper into a glass container (US EPA Method 3570C, 2002).

Pre-concentration of extracts

The solvent extracts were concentrated to $1cm^{3}$ using a rotary evaporator and kept for clean-up procedure.

Clean-up of extracts

Column chromatography was employed to clean-up the extracts (US EPA method 3630B, 1996). The glass separating column (20 cm) was packed with activated silica gel (90% < 45 µm) and washed down with n-hexane to remove any dirt. The extracts were demoisturized over 1 g of anhydrous granulated Na_2SO_4 and separated into two eluted fractions using mixtures of

Table 1. Recoveries, method detection limits (MDLs) and the relative standard deviation (RSD), limits of detection (LOD) and limits of quantification (LOQ) of chlorinated pesticides in water samples

OCPs	% Recoveries Water	MDLs (ng/mL)	RSD (%) n^a = 5	Mean OCP (ng/mL ± S.D)	LOD (ng/mL)	LOQ (ng/mL)
Alpha-BHC	95.10	0.07	5	8.77 ± 0.61	1.82	6.06
Beta-BHC	96.14	0.18	7	9.17 ± 0.66	1.97	6.58
Lindane	98.65	0.08	6	8.73 ± 0.60	1.81	6.04
Delta-BHC	97.19	0.10	8	8.95 ± 0.52	1.55	5.18
Heptachlor	96.87	0.20	8	8.60 ± 0.64	1.93	6.44
Aldrin	96.05	0.20	6	8.68 ± 0.61	1.84	6.15
Heptachlor-epoxide (B)	94.15	0.25	7	8.67 ± 0.62	1.86	6.21
Cis-Chlordane	90.48	0.30	8	8.70 ± 0.64	1.93	6.45
Trans-Chlordane	93.67	0.28	9	8.78 ± 0.62	1.85	6.17
Endosulfan	96.25	0.14	7	8.80 ± 0.58	1.75	5.84
Dieldrin	93.35	0.22	5	8.70 ± 0.61	1.83	6.10
p,p´-DDE	95.58	0.09	8	8.79 ± 0.58	1.73	5.77
Endrin	92.95	0.15	5	9.48 ± 0.80	2.39	7.96
Endosulfan 11	94.14	0.10	6	9.14 ± 0.56	1.69	5.65
p,p´-DDD	96.45	0.08	7	9.00 ± 0.48	1.43	4.77
Endrin aldehyde	91.85	0.10	8	9.11 ± 0.63	1.90	6.34
Endosulfan sulphate	96.10	0.16	8	9.99 ± 0.67	2.01	6.69
p,p´-DDT	95.74	0.20	10	8.43 ± 0.78	2.34	7.81
Methoxychlor	92.08	0.26	9	10.34 ± 1.22	3.66	12.21
Endrin ketone	90.88	0.12	7	20.20 ± 3.43	10.28	34.29

[a] measurements repeated five times

dichloromethane, hexane and acetonitrile as eluting solvents. For the first fraction, 30cm^3 of a dichloromethane/hexane (20/80) mixture was used, while 30cm^3 of a dichloromethane/hexane/acetonitrile (50/49.5/0.5) mixture was used for the second fraction in order to ensure that the polar acetonitrile eluted any remaining residue. The fractions were combined, concentrated to 1cm^3 using a rotary evaporator and subsequently analyzed.

Identification and determination of chlorinated pesticide residues by gas chromatography

A gas chromatograph equipped with an electron capture detector (GC-ECD) was used for the analysis of the chlorinated pesticide residues. The cleaned-up extracts were dried and re-dissolved in 1.0cm^3 of analar grade isooctane for injection into the gas chromatograph (Pandit et al., 2002). Blank runs were made for background correction and performance of the system. Organochlorine Pesticides II EPA Method 8081A {Mix AB # 3, cat. #32415 (ea.)} was employed for the analyses. The detection and determination of the residues were performed by injecting 1 μL of the 1.0cm^3 purified extract into the injection port of a gas chromatograph with a ^{63}Ni electron capture detector (GC-μECD Agilent Technology 7890A) equipped with the ChemStation software. The injection was carried out on a splitless injector at 250^{0}C and the purge activation time was 30 s. The run time was 17mins. The chromatographic conditions are given in Table 1. Identification of pesticide residues was accomplished using reference standards and relative retention time techniques, while the concentration of the residues was determined by comparing the peak heights of the samples with the corresponding peak heights of the reference standards of known concentrations. The stock solution of the OCP standards was purchased from Restek Corporation, USA. It contained 1000 ppm in n-hexane and was serially diluted to obtain the desired concentrations of 10, 20 and 40 ng/mL. All the extracts from water and sediment samples were analyzed for aldrin, dieldrin; endrin, DDT, heptachlor and their metabolites; HCH and endosulfan isomers; α, γ-chlordane and methoxychlor. The concentrations of the pesticide residues were calculated directly by the gas chromatograph after inputing the volume and weight of the samples. Blank corrections were effected.

Quality assurance

Before analysis was performed, standards were run to check for the column performance, peak height, resolution and the detection limit. The correlation coefficients of calibration curves of OCPs were all higher than 0.998. The quality assurance measures included strict cleaning procedures, procedural blank, recovery of spiked standards and monitoring of detector response.

Recovery study

Recovery studies were undertaken to evaluate the effi-

Table 2. Recoveries, method detection limits (MDLs) and the relative standard deviation (RSD) of chlorinated pesticides in sediment samples

OCPs	% Recoveries Sediment	MDLs (ng/g)	RSD (%) $n^a = 5$
Alpha-BHC	96.43	0.18	8
Beta-BHC	94.24	0.28	7
Lindane	97.04	0.35	8
Delta-BHC	98.15	0.10	8
Heptachlor	94.80	0.20	9
Aldrin	98.12	0.22	10
Heptachlor-epoxide (B)	96.30	0.36	12
Cis-Chlordane	92.38	0.25	9
Trans-Chlordane	92.18	0.32	10
Endosulfan	94.43	0.30	5
Dieldrin	94.05	0.18	11
p,p´-DDE	94.65	0.20	7
Endrin	90.14	0.45	9
Endosulfan 11	93.89	0.35	8
p,p´-DDD	94.32	0.18	7
Endrin aldehyde	93.80	0.40	6
Endosulfan sulphate	95.25	0.38	8
p,p´-DDT	96.10	0.24	8
Methoxychlor	90.36	0.30	5
Endrin ketone	92.26	0.25	6

ciency of the extraction procedure used. The recovery of chlorinated pesticides was done in replicate and was determined by spiking the previously analyzed samples with the pesticide standard at concentrations similar to those expected in the samples. The recovery values expressed in percentages were calculated from the chromatograms.

$$\% \text{ Recovery} = \frac{CS_2 - CS_1}{CS} \times 100$$

where, CS_1 = concentration of pesticide residues in the sample

CS_2 = concentration of pesticide residues in the spiked sample

CS = concentration of added pesticide

Determination of method detection limits (MDLs)

A solution of the analyte that is 3 times the estimated detection was prepared. The method detection limit was determined according to the formula:

MDL = Student's t value x the standard deviation of replicate measurements.

Determination of limit of detection (LOD)

The limits of detection of the organochlorine pesticide residues were determined by replicate chromatographic runs (6 times) of the least concentration of the OCP standards (10 ng/mL) and then multiplying the standard deviation obtained by 3.

Determination of limit of quantification (LOQ)

The limits of quantification of the organochlorine pesticide residues were determined by replicate chromatographic runs (6 times) of the least concentration of the OCP standards (10 ng/mL) and then multiplying the standard deviation by 10.

Data analysis

Concentrations of OCP residues were calculated individually and as the sum of their isomeric forms. Description of data was performed using a Statgraphics Centurion XV statistical software, with the level of significance maintained at 95% for each test.

RESULTS AND DISCUSSION

Table 1 shows the percentage recoveries, method detection limits (MDLs), the relative standard deviation (RSD), limits of detection (LOD) and limits of quantification (LOQ) of chlorinated pesticides in water samples while the percentage recoveries, method detection limits (MDLs) and the relative standard deviation (RSD) of chlorinated pesticides in sediment samples are shown in Table 2. The mean concentrations

Table 3. Mean concentrations (ng/mL) of chlorinated pesticide residues in microlayer and mixed layer water at Tarkwa Bay during the dry and wet seasons

OCPs	Dry season		Wet season	
	TBY-SW	TBY-BW	TBY-SW	TBY-BW
Alpha-BHC	5.0±2.1	0.7±0.2	2.0±0.2	0.8±0.1
Beta-BHC	0.4±0.3	3.0±1.3	0.2±0.1	0.5±0.2
Lindane	38.0±4.3	30.0±8.3	0.9±0.2	0.9±0.3
Delta-BHC	3.0±2.3	1.0±0.1	0.9±0.3	0.8±0.1
ΣBHC	46.4±9.0	34.7±9.9	4.0±0.8	3.0±0.7
Heptachlor	23.0±3.2	0.9±0.1	0.9±0.3	21.0±0.4
Heptachlor-epoxide (B)	2.0±1.2	0.6±0.3	0.6±0.3	1.0±0.5
Aldrin	5.0±1.1	6.0±3.3	2.0±1.1	5.0±3.3
Dieldrin	0.8±0.2	0.7±0.2	0.8±0.3	0.7±0.3
Endrin	0.7±0.2	1.0±0.1	1.0±0.2	0.8±0.3
Endrin aldehyde	1.0±0.2	0.4±0.2	0.6±0.3	0.8±0.4
Endrin ketone	0	0	0	0
Cis-Chlordane	0.7±0.4	0.6±0.2	0.6±0.4	0.6±0.5
Trans-Chlordane	0.7±0.6	0.8±0.1	0.5±0.2	1.0±0.4
Endosulfan 1	0.8±0.1	0.8±0.1	0.7±0.3	0.8±0.4
Endosulfan 11	0.5±0.8	0.7±0.6	1.0±0.4	0.7±0.4
Endosulfan sulphate	4.0±3.2	2.0±1.2	20.0±0.7	3.0±1.5
Methoxychlor	0	0	0	0
p,p´-DDE	0.8±0.4	0.8±0.1	0.8±0.3	0.7±0.5
p,p´-DDD	2.0±1.2	2.0±0.3	3.0±0.2	1.0±0.3
p,p´-DDT	1.0±0.4	0.5±0.2	1.0±0.3	0.7±0.3
ΣDDT	3.0±2.0	3.3±0.6	5.0±0.8	3.0±1.1
ΣOCPs	90.0±22.2	52.5±16.9	38.0±6.1	41.0±10.2

TBY-SW = microlayer water at Tarkwa Bay; TBY-BW = mixed layer water at Tarkwa Bay. The mean value was calculated from triplicate determinations.

Table 4. Mean concentrations (ng/g) of chlorinated pesticide residues in epipellic and benthic sediments at Tarkwa Bay during the dry and wet seasons

OCPs	Dry season		Wet season	
	TBY-SS	TBY-BS	TBY-SS	TBY-BS
Alpha-BHC	17.2±2.5	2.1±1.1	29.9±3.2	2.1±3.1
Beta-BHC	102.0±4.3	12.1±3.2	16.6±4.3	6.0±2.2
Lindane	45.7±8.2	4.9±2.2	26.9±2.4	0.5±0.8
Delta-BHC	11.7±1.1	0.8±1.4	19.4±2.2	0.6±0.2
ΣBHC	176.6±16.1	19.9±7.9	92.8±12.1	9.2±6.3
Heptachlor	119.3±5.4	91.8±7.2	556.5±5.5	13.2±5.2
Heptachlor-epoxide (B)	15.4±8.3	2.2±2.1	49.0±6.3	0.7±1.2
Aldrin	31.6±1.2	12.6±2.5	80.4±3.8	2.2±1.3
Dieldrin	27.4±2.3	2.8±1.2	48.8±2.4	0.6±0.2
Endrin	ND	10.7±3.3	49.6±8.5	0.8±0.2
Endrin aldehyde	143.5±2.4	6.1±2.1	375.4±5.5	0.6±0.1
Endrin ketone	238.6±8.3	14.5±2.2	420.6±3.6	0
Cis-Chlordane	60.1±5.2	2.8±4.2	76.6±6.5	0.6±1.2
Trans-Chlordane	45.5±3.3	7.7±5.3	80.5±2.4	1.2±2.1
Endosulfan 1	62.9±4.5	2.0±5.2	32.6±9.4	0.6±1.4
Endosulfan 11	260.9±5.8	2.2±4.2	102.9±6.5	0.8±3.6
Endosulfan sulphate	252.6±9.7	10.7±7.4	178.6±9.6	2.7±7.3
Methoxychlor	84.7±3.4	7.9±8.2	141.6±5.4	0
p,p´-DDE	20.6±6.2	ND	32.2±3.6	0.8±3.4
p,p´-DDD	ND	4.6±3.1	93.1±2.3	2.9±4.1
p,p´-DDT	113.2±8.2	7.4±7.3	101.8±5.7	0.8±5.2
ΣDDT	133.8±14.4	12.0±10.4	227.1±11.6	4.5±12.7
ΣOCPs	1646±90.3	195±74.1	2503±111.1	36±42.8

TBY-SS = epipellic sediment at Tarkwa Bay; TBY-BS = benthic sediment at Tarkwa Bay. The mean value was calculated from triplicate determinations.

of chlorinated pesticide residues in microlayer and mixed layer water from Tarkwa Bay are shown in Table 3 while the mean concentrations of chlorinated pesticide residues in epipellic and benthic sediments from Tarkwa Bay are presented in Table 4. The mean and standard deviation were calculated from the detectable values, and values below the detectable limit were considered not detected (ND). Compared to the sediment samples analyzed, the water samples contained the lowest amounts of residues. During the dry season, the most frequently occurring residues were β-BHC, lindane, δ-BHC, heptachlor, heptachlor epoxide (B), aldrin, endrin, endosulfan1 and p,p′-DDT. Endrine ketone was not detected in all the samples investigated. Total pesticide concentration recorded in the microlayer water was 89.82 ng/mL while 51.49 ng/mL was the concentration in the mixed layer water. The majority of the pesticide residues were found at higher concentrations in the microlayer water than in the mixed layer water. The concentrations of residues did not follow any particular pattern during the dry and wet seasons.

The concentrations of chlorinated pesticide residues obtained in this study were higher when compared to the residues obtained by Ize-Iyamu et al. (2007) in their studies of Ovia, Ogba and Ikoro Rivers in Edo State, Nigeria. The mean pesticide residues obtained were higher than those obtained by Tongo (1985) from studies carried out in some rivers in Nigeria. In similar investigations carried out on Gomti River, India (Malik et al., 2008) and Beijing Guanting reservoir, China (Xue et al., 2006), the total pesticide concentration ranged from 2.16 to 567.49 ng/L and from 16.70 to 791.00 ng/L respectively. It was observed that the levels of pesticide residues in samples collected from the same site in different seasons varied. These differences in concentration could be attributed to tidal changes. Water turbulence might lead to a mixing tendency as earlier reported by Tongo (1985) and Ize-Iyamu et al. (2007). The sediment samples exhibited significantly higher residue levels than the water samples. p,p′-DDE was not detected at the benthic sediment. Endrin and p,p′-DDD were not detected at epipellic sediment. Chlorinated pesticide levels did not show any particular pattern between the epipellic and benthic sediment and during the dry and wet seasons. The pesticide levels were within the permissible limits (FAO/WHO, 2005; USEPA, 2006).

The mean recoveries of the residues in water ranged from 90.48 to 98.65% while the mean recoveries of the residues in sediment ranged from 90.14 to 98.15%. These recovery values indicate good accuracy of the methodology used in this study, thus validating it. The method detection limits ranged from 0.07 to 0.30 ng/mL in water and 0.10 to 0.45 ng/g in sediment. The relative standard deviation in water was in the range of 5 to 10%. The relative standard deviation in sediment ranged from 5 to 12%. The limits of detection ranged from 1.43 ng/g to 10.29 ng/g, indicating the high sensitivity of the gas chromatograph at the operating conditions. The calibration curves of the analyzed pesticides present a good regression line (r > 0.999) in the range of explored concentrations.

CONCLUSION

The chlorinated pesticide residues were detected in all the samples even though the frequency of detection of a few of the residues was less than 100%. Pesticide residues in water were largely lower than residues in the sediments. A higher concentration of the residues was generally observed during the dry season.

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