

# **ORIGINAL ARTICLES**

# Occurrence of Organochlorine Pesticides (OCPs) in Surface Sediments of the Niger Delta, Nigeria

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# ABSTRACT

The occurrence, sources and toxicity of organochlorine pesticides (OCPs) in surface sediments of selected rivers, canals and streams in the Niger Delta were investigated. The concentrations of 21 target OCPs ranged from 20 to 313 ng/g with a mean value of 102 ng/g. Hexachlorocyclohexanes (HCHs) were the dominant components accounting for approximately 50% of the total OCPs detected. The high concentration of HCHs could be attributed to the use of HCHs pesticides (lindane) in the region. The compositional profiles of dichlorodiphenyltrichloroethanes (DDTs) in most samples suggested that DDTs were not derived from fresh inputs, but rather related to anaerobic biodegraded products. The sediments can be considered to be highly polluted with HCHs when compared to similar samples from other parts of the world. An assessment using the widely cited sediment quality guidelines for OCPs indicated that there were potential ecological and human health risks for HCHs in the area.

*Key words:* Organochlorine Pesticides; Surface sediments; Niger Delta; Hexachlorocyclohexanes, Dichlorodiphenyltrichloroethanes.

## Introduction

Organochlorine pesticides (OCPs) have received much attention due to their persistent nature, bioaccumulation, long -range environmental transport and toxicity to wildlife and humans (Fillmann *et al.*, 2002; Shen *et al.*, 2005; Wan *et al.*, 2005. These compounds have been widely used globally as insecticides for agriculture and public health purposes. The use of OCPs was banned or restricted in the developed countries in the 1970s and 1980s. These compounds still remain major pollutants in many developing countries because of the weak enforcement program on the usage and the presence of residues from previous use (Iwata *et al.*, 1994; Tanabe *et al.*, 1994; Shen *et al.*, 2005). The OCPs could still be a problem in marine sediments which were regarded as an important sink of organic contaminants (Yang *et al.*, 2005). OCPs can enter aquatic environment through effluent release atmospheric deposition, runoff and other means (Itawa *et al.*, 1993; Yang *et al.*, 2005). They are subsequently removed from the water and absorbed onto particulate matter and finally settles in the bottom sediments.

The Niger Delta is one of the major oil producing regions of the world and covers an area of about 75,000 km<sup>2</sup> which represents 7.5% of Nigeria's total land area. Oil exploration activities in the region have led to many claims and counter claims by the inhabitants on the pollution of rivers in the area. OCPs are believed to have been used for farming practices and domestic purposes before the discovery of oil in the region. Presently, unrestricted and illegal use of OCPs for crop protection and household disinfection is still a common practice in the Niger delta. However, limited data are available on the occurrence and distribution of OCPs in surface sediments of rivers in the area (Ezemonye *et al.*, 2008, 2009). The present study was conducted to determine the concentration, distribution and possible source(s) of OCPs in surface sediments of some rivers, canals and streams in the Niger Delta. The potential biological risk of these organic pollutants was also assessed.

### Materials and method

#### Sample Preparation and Instrumental Analysis:

Sample collection and preparation were carried out according to methods already described in Sojinu *et al.*, 2010. Surface sediment samples (0-20cm) were collected from rivers, canals and streams in some parts of the Niger Delta (Fig. 1 and Table 1).



Fig. 1: The map of the study area showing sampling locations, the inset maps show the location of the study area (top left) in Nigeria.

Table 1: Concentrations (ng/g, dry wt.), contents of TOC and molecular ratios of OCPs in surface sediments of the Niger Delta.											
OCPs	Calabar	Bakassi	GKMT	GKHT	GKLT	Imo	Oginni	Ughelli	Olomoro	Uzere	Mean
	(3) <sup>d</sup>	$(3)^{d}$	(3) <sup>d</sup>	(3) <sup>d</sup>	(3) <sup>d</sup>	(3) <sup>d</sup>					
α-HCH	6.28	4.73	4.40	0.70	1.53	3.91	31.27	7.76	58.87	11.82	13.13
β-НСН	4.02	5.91	11.73	3.17	10.26	5.27	24.07	39.51	71.59	13.49	18.90
ү-НСН	1.63	9.73	2.50	2.18	3.03	1.60	14.14	8.85	30.52	3.38	7.76
δ-НСН	3.26	13.10	4.90	6.53	1.75	2.33	25.83	13.38	31.81	4.67	10.76
Heptachlor	6.11	0.32	0.21	0.53	1.76	5.41	1.41	0.38	2.10	0.11	1.83
Aldrin	0.13	0.49	0.05	nd	0.02	0.34	15.11	1.81	18.52	0.65	3.71
Heptachlor epoxide II	1.61	2.41	0.79	0.63	1.43	2.64	38.09	7.17	16.56	1.72	7.31
DDMU	0.09	0.69	nd	0.27	0.11	0.37	0.82	0.05	1.05	0.19	0.36
Endosulfan	nd	nd	1.13	0.11	0.15	1.28	37.18	6.40	18.20	3.91	6.84
o,p'-DDE	0.01	0.04	0.03	nd	nd	nd	1.07	0.14	0.53	0.06	0.19
Endosulfan II	0.73	11.76	0.18	0.01	0.19	3.25	12.61	1.23	14.09	0.07	4.41
p,p-DDE	0.14	0.75	0.23	0.04	0.12	0.65	4.37	0.28	1.64	0.17	0.84
o,p-DDD	0.01	1.15	0.17	0.17	0.13	0.93	2.59	0.36	2.96	0.13	0.86
Dieldrin	0.05	nd	0.11	nd	0.05	0.11	7.29	0.73	7.22	0.13	1.57
Endosulfan I	0.35	0.6	0.96	0.88	1.21	1.24	0.88	0.12	11.31	0.68	1.80
p,p'-DDD	0.10	1.74	0.13	0.01	0.06	4.19	1.93	0.13	4.05	0.13	1.25
o,p'-DDT	0.03	1.19	0.07	0.10	0.03	0.11	21.15	2.23	2.79	0.28	2.80
p,p'-DDT	nd	0.10	0.36	0.06	0.00	0.35	5.49	0.57	10.64	0.25	1.78
Endrin Sulfate	nd	0.02	nd	nd	nd	0.03	0.06	0.02	0.65	0.01	0.08
Endrin Aldehyde	nd	1.75	108.84	2.85	0.06	8.88	1.67	1.54	5.68	8.94	14.02
Methoxychlor	0.06	0.01	1.36	0.01	nd	3.78	2.13	0.38	1.83	0.48	1.00
TOC	2.35	1.29	6.38	4.65	5.09	3.93	1.89	1.03	2.13	1.26	3.00
$\sum_{21}$ OCP(ng/g dry wt):	57.9	140	20.0	23.4	48.1	24.9	93.0	313	51.3	102	
26.2											
∑HCHs	15.2	33.5	23.6	12.6	16.6	13.1	95.3	69.5	193	33.4	50.6
∑DDTs	0.29	4.97	0.99	0.38	0.34	5.88	36.60	3.71	22.61	1.06	7.68
α-/ γ-ΗCΗ	3.85	0.49	1.76	0.32	0.50	2.44	2.20	0.22	1.93	3.50	1.79
DDE+DDD/DDTs	0.90	0.74	0.57	0.58	0.91	0.98	0.27	0.25	0.41	0.48	0.61
DDDE/DDD	1.36	0.27	0.87	0.22	0.63	0.13	1.20	0.80	0.31	0.88	0.67

<sup>a</sup>River; <sup>b</sup>Canal; <sup>c</sup>Stream, <sup>d</sup>No of sampling sites

nd., not detected

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Homogenized and freeze-dried samples (20-50g) spiked with known amount of surrogate standards of PCB 67 and PCB 191 were extracted with dichloromethane in a Soxhlet extractor for 48 h. The extracts were treated with activated copper in order to remove elemental sulphur. The extracts were concentrated using a rotary evaporator to approximately 1 mL and solvent-exchanged to hexane (Wang *et al.*, 2007). The concentrated outracts were fractionated on class columns pecked with silica alumina (2:1) into two fractions.

concentrated extracts were fractionated on glass columns packed with silica-alumina (2:1) into two fractions containing aliphatic hydrocarbons and PAHs/OCPs, respectively, by successive elution with 20 mL of hexane and 70 mL of hexane/dichloromethane (7/3 in v/v). PCB 82 was added to the PAH/OCPs fractions as an internal standard prior to gas chromatography/mass spectrometry analyses.

OCPs were analyzed on a Shimadzu Model 2010 GC-MS (Shimadzu, Japan) with an HP-5MS fused silica column (30 m × 0.25 mm i.d., 0.25  $\mu$ m film thickness). Ultra pure helium gas was used as carrier gas. The mass spectrometer was operated in the electron impact mode at 70 eV. The fractions were injected with an auto sampler in the splitless/split mode with a split time of 1 min after injection and the injector temperature was 100°C. Column temperature was programmed from 50°C (held for 1 min) to 150°C at a rate of 10°C/min, then to 210°C at a rate of 2°C/min and finally to 280°C (held for 7mins) at a rate of 35°C/min. Compound identification was based on comparison of the retention times and fragmented ion profiles of the reference standards with the target analytes, and quantification was performed with a conventional internal calibration method (Wang *et al.*, 2007).

#### Determination of Total Organic Carbon:

Total organic carbon (TOC) contents were determined on freeze-dried, ground samples, following acid treatment with 10% HCl for 24 h, to remove carbonate. The carbonate-free samples were rinsed thrice with distilled water to remove acid residues and dried at 60°C for 48 h. The organic carbon content was determined using a Perkin Elmer CHN 2400 elemental analyzer (Hedges and Stern, 1984).

#### Quality Assurance/Quality Control:

Quality assurance and quality control procedures have been described in detail elsewhere (Sojinu *et al.*, 2010). PCB 67, PCB 191 and PCB 82 standards were obtained from AccuStandard (New Haven, CT, USA). The average recoveries of the surrogate standards ranged from 55% to 85% in all the blanks and from 50% to 80% in the samples. All the spiked blanks (standards spiked into solvents) have surrogate standard recoveries in the range of 65 to 85% and the matrix spiked samples had surrogate standard recoveries in the range of 55 to 75%. Less than 15% targets were found in the blanks; therefore, all data were corrected by mean concentrations of blanks. It should be noted that the concentrations were not corrected by the surrogate recovery data.

#### **Results and discussion**

#### Occurrence and Distributions of OCPs in Surface Sediments:

The concentrations of OCPs and TOC contents in sediments of the Niger Delta are listed in Tables 1. Total concentrations of 21 target OCPs ranged from 20 to 313 ng/g with a mean value of 102 ng/g. Among the different OCPs, the levels of hexachlorocyclohexane isomers (HCHs) were the highest in all the locations (Table 1), accounting for approximately 50% of the total OCPs detected. The high HCHs concentrations in the sediments could be attributed to the runoffs from surrounding agricultural areas where HCHs pesticides have been used. The  $\alpha$ -,  $\beta$ -,  $\gamma$ -, and  $\delta$ - HCH isomers in the samples accounted for about 26.0%, 37.4%, 15.4% and 21.3% of the total HCHs, respectively. This percentage distribution is different from what is expected in the technical HCH pesticides. Studies have shown that the percentages of  $\alpha$ -,  $\beta$ -,  $\gamma$ -, and  $\delta$ - HCH in technical HCH pesticides are 55-80%, 5-14%, 8-15% and 2-16%, respectively (Walker *et al.*, 1999; Lee *et al.*, 2001). Many studies have reported the predominance of  $\beta$  - HCH in sediments from river or estuary environment after long -term migration and transformation (Wu *et al.*, 1997; Lee *et al.*, 2001; Doong *et al.*, 2002). It has also been reported that  $\alpha$ - and  $\gamma$ -HCH can also be transformed to  $\beta$  -HCH in the aged environment samples (Walker *et al.*, 1999).

DDT and its metabolites were also detected in appreciable quantities in the samples. The concentrations of DDTs (p,p'-DDT, o,p'-DDT, p,p',-DDE, o,p'-DDE, p,p'-DDD and o,p'-DDD) ranged from 0.3 to 37ng/g. The DDTs is dominated by o,p'-DDT. Other OCPs detected in the samples include heptachlor, aldrin,

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heptachlor epoxide II, DDMU, endosulfane, endosulfan II, dieldrin, endosulfan I, endrine sulphate, endrin aldehyde and methoxychlor.

There was no correlation between the OCPs and TOC as shown in Fig. 2 ( $r^2 = -0.1969$  and p = 0.5856 based on a 2-tailed test), suggesting that TOC has no influence on the distribution of OCPs in the sediments. This result indicated that the residue OCPs concentrations in the sediments were a reflection of input source rather than post-depositional sorption by the organic matter.



Fig. 2: Plots of OCPs concentrations versus TOC contents in surface sediments of Niger Delta, Nigeria.

#### Sources of OCPs:

The composition differences of HCHs isomers and DDT (compared with its metabolites) have been used to identify different sources of OCPs contaminations in sediments (Willett *et al.*, 1998; Lee *et al.*, 2001; Tao *et al.*, 2007; Yang *et al.*, 2008). HCH pesticide is available in two formations: technical HCH ( $\alpha$ -HCH > 55%) and lindane ( $\gamma$  - HCH > 99%). High ratio of  $\alpha / \gamma$  - HCH ranging from 3-7 indicates the input of technical HCH, while low ratio close to < 1 suggest dominant use of lindane (Willet *et al.*, 1998; Sarkar *et al.*, 2008; Yang *et al.*, 2008). A higher ratio (> 7) could be attributed to long-range atmospheric or re-cycling of technical HCH because  $\alpha$  -HCH has a longer lifetime than  $\gamma$  isomer by about 25% (Willet *et al.*, 1998). In this work,  $\alpha$ -/ $\gamma$ -HCH ratios were lower than 3, with the exception of sediments from two locations (Calabar and Uzere) where values close to 3 were recorded (Table 1). These values suggest that the HCHs were derived mainly from lindane.

 Table 2:
 Comparison of HCHs and DDTs concentrations in surface sediments of Niger Delta and other rivers and coastal area in the world (ng/g, dry wt.).

Locations	HCHs	DDTs	Reference
Niger Delta	12.6-193 (50.6)	0.29-36.6 (4.58)	This study
Xiamen Harbor, China	0.14-1.12 (0.45)	4.45-311 (42.8)	Hong et al. (1995)
Minjiang River Estuary, China	2.99-16.21 (8.62)	1.57-13.06 (6.7)	Zhang et al. (2003)
Quanzhou Bay, China	0.55-3.74 (1.63)	7.37-49.3 (22.27)	Gong et al. (2007)
Yangtze Estuary, China	0.5-17.5 (6.0)	0.9-33.1 (8.2)	Liu et al. (2008)
Bohai Sea, China	0.16-3.17 (0.83)	0.24-5.67 (1.36)	Hu et al. (2009)
Mandovi River Estuary, India	3.8	73	Iwata et al. (1994)
Bay of Bengal, India	0.04-4.79	0.04-4.79	Babu Rajendran et al. (2005)
Kyeonggi Bay, Korea	0.15-1.2 (0.44)	0.046-4.2 (0.70)	Lee et al. (2001)
Wu-shi Estuary, Taiwan	0.99-14.5 (3.78)	nd-11.4 (2.51)	Doong et al. (2002)
Coastal area of Singapore	3.4-46.1	2.2-11.9	Wurl and Obbard (2005)
Coastal area of Vietnam	nd-1.00	0.31-274	Hong et al. (2008)

The relative concentration of the parent DDT compared to its biological metabolites; DDD and DDE have been used to identify the possible sources of DDT. DDT may be transformed by microorganisms to DDE and DDD under aerobic and anaerobic conditions, respectively (Bossi *et al.*, 1992; Falandysz *et al.*, 2001; Liu *et al.*, 2008). (DDD + DDE)/ DDTs ratios have been used to determine the degree of DDT decomposition and any fresh input (Hong *et al.*, 1999; Lee *et al.*, 2001; Doong *et al.*, 2002). A ratio of (DDD + DDE)/ DDTs of more than 0.5 suggests aged (microbiologically degraded) DDT while a ratio < 0.5 implies

fresh input (Hong *et al.*, 1999). The (DDD + DDE)/ DDTs ratios in most of the studied samples were higher than 0.5, implying aged DDT source. DDE/DDD ratios in the samples were lower than 1, indicating anaerobic conditions in the sedimentary environment (Liu *et al.*, 2008).

#### **Pollution Status:**

The extent of OCPs pollution in the present study was assessed by comparing the concentrations of HCHs and DDTs in the sediments with those reported for similar samples from other parts of the world (Table 2). HCH and DDT have been the most used OCPs in agriculture all over the world (Jones and Voogt, 1999). The major OCP determined in the Niger Delta surface sediments are the HCHs isomers which constitute about 50% of the total OCPs. The HCHs concentrations ranged from 13 to 193 ng/g with a mean value of 51 ng/g (Table 1). The samples can be considered to be highly polluted with HCHs compared to those reported in coastal sediments around the world (Table 2). DDTs concentration recorded in this study ranged from 0.29 to 36.6 ng/g with a mean value of 4.58 ng/g. These values were generally lower than those reported in similar samples around the world.

#### Assessment of Potential Ecological Risk:

The possible toxic effects of OCPs in the study area were assessed based on two widely used sediment quality guidelines as shown in Table 3. Total DDT (o,p'- and p,p'-DDT) concentrations exceed the ERL and TEL values in Bakkasi, Oginni, Ughelli and Olomoro. The DDTs values also exceed the ERL and TEL values at these locations with exception of Ughelli. Concentration levels of DDT (o,p'- and p,p'-DDT) exceed the ERM and PEL values only in 2 locations (Oginni and Olomoro). DDTs values also exceed the ERL and TEL values in sediments from Imo River. The levels of  $\gamma$ -HCH in all the locations exceed the TEL and PEL values. This assessment suggested that there exists a potential ecological risk for  $\gamma$ -HCH in the surface sediments of the studied area.

Location	o,p - and p,p -DDT	p,p -DDE	p,p -DDD	DDTs	γ-HCH
Calabar	0.03	0.1	0.10	0.29	1.63
Bakasi	1.29	0.75	1.74	4.97	9.73
GKMT	0.43	0.23	0.13	0099	2.50
GKHT	0.16	0.04	0.01	0.38	2.18
GKLT	0.03	0.12	0.06	0.34	3.03
Imo	0.46	0.65	4.19	5.88	1.60
Oginni	26.6	4.37	1.93	36.6	14.2
Ughelli	2.80	0.28	0.13	3.71	8.85
Olomore	13.4	1.64	4.05	22.6	30.5
Uzere	0.53	0.17	0.13	1.02	3.38
ERL <sup>a</sup>	1	2.2	2	1.58	-
ERM <sup>a</sup>	7	27	20	46.1	-
TEL <sup>c</sup>	1.19	2.07	1.22	3.89	0.32
PEL <sup>d</sup>	4.77	374	7.81	51.7	0.99

 Table 3:
 Assessment of potential ecological risk of selected OCPs in surface sediments of Niger Delta using two sediment quality guidelines (SQG).

<sup>a</sup> Effect range-low value (Long *et al.*, 1998; Long and MacDonald, 1995)

<sup>b</sup> Effect range-median value (Long et al., 1998; Long and MacDonald, 1995)

<sup>c</sup> Threshold effect level (CCME, 2002)

<sup>d</sup> Probable effect level (CCME, 2002)

#### Conclusions:

This study examined the occurrences, distribution, possible source(s) and potential toxicity of OCPs in surface sediments of some rivers, canals and streams in the Niger Delta, Nigeria. HCHs isomers were the major components of the total OCPs detected, and their levels were much higher compared to values recorded in other riverine /estuarine and coastal regions in the world. Source diagnostic ratios indicated that the HCHs were derived mainly from previous or recent use of lindane while the DDTs are mainly anaerobic byproducts. An assessment based on the sediment quality guideline indicated that there exists a potential ecological risk for HCH in the region.

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