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# Residue analysis of organochlorine pesticides in water and sediments from Agboyi Creek, Lagos

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**Microlayer water, mixed layer water, epipellic and benthic sediments were collected from Agboyi Creek, Lagos to analyse organochlorine 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 sediments were subjected to cold extraction with petroleum ether/acetone (1:1 v/v) mixture and clean-up on silica gel adsorbents. The samples were analyzed for aldrin, dieldrin, endrin, DDT, heptachlor, HCH, endosulfan, chlordane and methoxychlor. The detection and determination of the pesticide residues were performed by injecting 1 µL of purified extract into the injection port of a gas chromatograph with a $^{63}$Ni electron capture detector (GC-µECD Agilent 7890A) equipped with ChemStation software. Pesticide residues in the epipellic and benthic sediments were higher than the residues in the water. The mixed layer water showed enhanced levels of residues when compared with the microlayer water. The residue levels were higher during the dry season than the wet season. The levels of residues in the water and sediments were below the maximum permissive residue limits.**

**Key words:** Agboyi Creek, organochlorine pesticides, microlayer water, mixed layer water, epipellic sediment, benthic sediment.

## INTRODUCTION

Organochlorine pesticides (OCPs), namely aldrin, dieldrin, endrin, chlordane, dichlorodiphenyltrichloroethane (DDT), heptachlor, mirex, toxaphene, hexachlorobenzene (HCB) and industrial chemicals and byproducts, including PCBs, dioxins and furans, constitute the twelve chemical substances called the "dirty dozen" and defined under the Stockholm Convention. The manufacture and use of chlorinated pesticides have been banned or restricted in developed countries. Although these bans and restrictions were enacted during the 1970s and 1980s, some developing countries are still using OCPs for agricultural and public health purposes because of their low cost and versatility in controlling various pests (Xue et al., 2006). Again, they are being used in most developing countries, including Nigeria, due to a lack of appropriate regulatory control and management of the production, trade and use of these chemicals (Darko and Acquaah, 2007).

Pesticides are chemicals used to kill or control pests. They are classified according to their chemical class or intended use. OCP residues enter aquatic environments 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). OCPs could distribute to the components of the ecosystem, such as water and sediment, and accumulate in the biota. The sediment reservoir is important because it serves as

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a sink from which water and biota are continuously polluted. Thus, the quality of sediment is essential in assessing the pollution status of the ecosystem (Doong et al., 2002).

The toxicity of pesticides could be acute and chronic. There is growing evidence on cancer, neurological damage, endocrine disruption and birth defects arising from exposure (Williams et al., 2013). OCP levels are usually monitored 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. The indiscriminate use of pesticides in Nigeria has resulted in the occurrence of the residues in biota and other abiotic compartments (Okoya et al., 2013; Izelyamu et al., 2007; Adeyemi et al., 2008; Williams et al., 2013). It is necessary to ascertain the distribution, behaviour and fate of these compounds in various environmental compartments. Agboyi Creek empties into Lagos Lagoon which is the ultimate sink for the disposal of sewage, domestic, industrial and agricultural wastes in Lagos. This study was therefore, undertaken to determine the pesticide residues in microlayer water, mixed layer water, epipellic and benthic sediments from Agboyi Creek, Lagos, Nigeria.

## MATERIALS AND METHODS

### Study area

The study area for this investigation is Agboyi Creek, which lies between latitude 6.56492 and longitude 3.41086 on the Western part of Nigeria.

### Sampling strategy

Sampling was conducted at Agboyi Creek (AGR) between December 2008 and September 2009. Field investigations were carried out four times during the dry season months of December and February and the wet season months of May and September to study effects of seasonal variation on the samples. Sampling locations were identified with a hand-held Garmin-GPSMAP 76S-type global positioning system.

Prior to sampling, sample bottles and glass wares were washed with detergent, rinsed with distilled water and pure acetone (99.9%) and then heated in an oven overnight at 100°C.

The microlayer water was collected with clean glass from a depth of 1 cm while the mixed layer water was sampled with a 5 L Goflon water sampler with the aid of a boat. Water samples were collected (31 N 0545254, UTM 0726974) in three labelled amber glass bottles to form composite samples. After collection, samples were stored in ice-packed coolers. The water samples were kept in the refrigerator in the laboratory at 4°C to inactivate microbes and thus preserve the integrity of the samples (Radojevic and Bashkin, 1999).

Epipellic sediments were obtained by scooping the top 1 to 5 cm of the intertidal sediments (31 N 0545069, UTM 0727598). Benthic sediments were obtained (31 N 0545254, UTM 0726974) with the aid of a Shipek grab sampler. Three grab sediment samples were collected and mixed together to form a composite sample and then wrapped in a labelled aluminium foil. After collection, the sediment sample was stored in an ice-packed cooler and kept in the refrigerator in the laboratory at 4°C. Pebbles, shells and vegetable matter were manually removed.

### Treatment of samples

Water samples were subjected to liquid-liquid extraction. 200 $cm^3$ of water sample was extracted with 30 $cm^3$ of HPLC grade dichloromethane in a separating funnel. The extraction process was repeated with 20 $cm^3$ dichloromethane and the extracts were combined (US EPA, 2007).

5 g of wet sediment was homogenized with 5 g of anhydrous granulated $Na_2SO_4$. Cold solvent extraction (Steinwandter, 1992) was carried out using 50 $cm^3$ HPLC grade petroleum ether/acetone (1:1 v/v) mixture. The mixture was shaken and allowed to stand for 30 min and then filtered (US EPA, 2002). The solvent extracts were concentrated to 1 $cm^3$

Column chromatography was used to clean-up the extracts (US EPA, 1996). The glass separating column was packed with activated silica gel (90%, <45 µm) and washed down with n-hexane. The extracts were demoisturized over 1 g of anhydrous granulated $Na_2SO_4$ and separated into two fractions using mixtures of dichloromethane, hexane and acetonitrile as eluting solvents. For the first fraction, 30 $cm^3$ of a dichloromethane/hexane (20/80) mixture was used, while 30 $cm^3$ of a dichloromethane/hexane/acetonitrile (50/49.5/0.5) mixture was used for the second fraction. The fractions were combined and concentrated to 1 $cm^3$.

### Identification and determination of OCP residues by gas chromatography

A gas chromatograph with a $^{63}Ni$ electron capture detector (GC-µECD Agilent Technology 7890A) was used for the identification and determination of the OCP residues. The cleaned-up extracts were dried and re-dissolved in 1.0 $cm^3$ analar grade isooctane before injecting 1 µL of the purified extract into the injection port of the gas chromatograph (Pandit et al., 2002). Organochlorine Pesticides II EPA Method 8081A was employed for the analyses. The stock solution of the OCP standards was purchased from Restek Corporation, USA and was serially diluted to obtain 10, 20 and 40 ng/mL.

Strict cleaning procedures, recovery of spiked standards and monitoring of detector response were some of the quality assurance measures that were adopted. The correlation coefficients of calibration curves were all higher than 0.998.

Recovery study was determined by spiking the previously analysed samples with the pesticide standard.

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

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 standard.

The limits of detection and quantification of the organochlorine pesticide residues were determined by multiplying the standard deviation obtained from six replicates at lowest expected concentration by 3 and 10, respectively (Attallah et al., 2012).

## RESULTS

Concentrations of OCP residues were calculated individually and as the sum of their isomeric forms. Description

**Table 1.** Recoveries in water and sediment, limits of detection (LOD) and limits of quantification (LOQ) of OCPs in water samples.

| OCPs | Water recoveries (%) | Sediment recoveries (%) | Mean OCP (ng/mL ± S.D) | LOD (ng/mL) | LOQ (ng/mL) |
|---|---|---|---|---|---|
| Alpha-BHC | 95.10 | 96.43 | 8.77 ± 0.61 | 1.82 | 6.06 |
| Beta-BHC | 96.14 | 94.24 | 9.17 ± 0.66 | 1.97 | 6.58 |
| Lindane | 98.65 | 97.04 | 8.73 ± 0.60 | 1.81 | 6.04 |
| Delta-BHC | 97.19 | 98.15 | 8.95 ± 0.52 | 1.55 | 5.18 |
| Hepta Heptachlor | 96.87 | 94.80 | 8.60 ± 0.64 | 1.93 | 6.44 |
| Aldrin Aldrin | 96.05 | 98.12 | 8.68 ± 0.61 | 1.84 | 6.15 |
| Heptachlor-epoxide (B) | 94.15 | 96.30 | 8.67± 0.62 | 1.86 | 6.20 |
| Cis-Chlordane | 90.48 | 92.38 | 8.70 ± 0.64 | 1.93 | 6.45 |
| Trans-Chlordane | 93.67 | 92.18 | 8.78 ± 0.62 | 1.85 | 6.17 |
| Endosulfan | 96.25 | 94.43 | 8.80 ± 0.58 | 1.75 | 5.84 |
| Dieldrin | 93.35 | 94.05 | 8.70 ± 0.61 | 1.83 | 6.10 |
| p,p´-DDE | 95.58 | 94.65 | 8.79 ± 0.58 | 1.73 | 5.77 |
| Endrin | 92.95 | 90.14 | 9.48 ± 0.80 | 2.39 | 7.96 |
| Endosulfan 11 | 94.14 | 93.89 | 9.14 ± 0.56 | 1.69 | 5.65 |
| p,p´-DDD | 96.45 | 94.32 | 9.00 ± 0.48 | 1.43 | 4.77 |
| Endrin aldehyde | 91.85 | 93.80 | 9.11 ± 0.63 | 1.90 | 6.34 |
| Endosulfan sulphate | 96.10 | 95.25 | 9.99 ± 0.67 | 2.01 | 6.69 |
| p,p´-DDT | 95.74 | 96.10 | 8.43 ± 0.78 | 2.34 | 7.81 |
| Methoxychlor | 92.08 | 90.36 | 10.34 ± 1.22 | 3.66 | 12.21 |
| Endrin ketone | 90.88 | 92.26 | 20.20 ± 3.43 | 10.28 | 34.29 |

of data was performed using a Statgraphics Centurion XV statistical software.

The results of the analyses are presented in Tables 1 to 3, while the representative chromatograms are shown in Figures 1 to 4. The mean and standard deviation were calculated from the detectable values, and values below the detectable limit were considered not detected (ND). The mean was calculated from triplicate determinations.

## DISCUSSION

Table 1 shows the percentage recoveries in water and sediment, limits of detection (LOD) and limits of quantification (LOQ) of organochlorine pesticides in water samples. The mean concentrations of organochlorine pesticide residues in microlayer and mixed layer water from Agboyi Creek are shown in Table 2, while the mean concentrations of organochlorine pesticide residues in epipellic and benthic sediments from Agboyi Creek are presented in Table 3. The percentage recoveries of the samples validate the methodology that was employed. The water samples contained lower amounts of residues as compared to the sediment samples analyzed. 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.

Dieldrin, α-BHC, endrin aldehyde, endrin ketone, cis-chlordane, trans-chlordane, endosulfan sulphate, methoxychlor, p,p´-DDE and p,p´-DDD were not detected in the mixed layer water of Agboyi Creek. Endrine ketone was not detected in all the samples investigated. The microlayer water at Agboyi Creek had a wider distribution of OCPs, though the total OCP residues were more in the mixed layer water (25.59 ng/mL) than in the microlayer water (19.87 ng/mL). The concentrations of residues did not follow any particular pattern during the dry and wet seasons. Investigations conducted on the OCP levels in Lagos Lagoon at the time this study was carried out showed reduced concentration levels in Agboyi Creek. The concentrations of OCP residues obtained in this study were higher when compared with the residues obtained in the studies of Ovia, Ogba and Ikoro Rivers in Edo State, Nigeria (Ize-Iyamu et al., 2007). The mean pesticide residues obtained were higher than those obtained from studies carried out in some rivers in Nigeria (Tongo, 1985). In similar investigations carried out on Gomti River, India (Malik et al., 2008) and Beijing Guanting reservoir, China (Xue et al., 2006), the total OCP 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 OCPs 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 (Ize-Iyamu et al., 2007; Tongo, 1985). Methoxychlor and p,p´-DDE were not detected in the epipellic sediment of Agboyi during the dry season, while endrin ketone was undetected in the benthic sediment. The OCP levels were within the permissible limits (FAO,

**Table 2.** Mean concentrations (ng/mL) of organochlorine pesticide residues in microlayer and mixed layer water of Agboyi Creek during the dry and wet seasons.

| OCPs | Dry season | | Wet season | |
|---|---|---|---|---|
| | AGR-SW | AGR-BW | AGR-SW | AGR-BW |
| Alpha-BHC | 0.6±0.1 | ND | 3.0±1.3 | 2.0±2.4 |
| Beta-BHC | 0 | 2.0±0.2 | 2.0±1.5 | 1.0±0.8 |
| Lindane | 0.9±0.3 | 0.5±0.3 | 3.0±1.6 | 13.0±3.3 |
| Delta-BHC | 1.0±0.3 | 0.7±0.1 | 0.9±0.4 | 0.9±0.2 |
| ΣBHC | 2.5±0.7 | 3.2±0.6 | 9.0±4.8 | 17.0±6.7 |
| Heptachlor | 5.0±2.2 | 2.0±1.1 | 0.8±0.3 | 0.9±0.3 |
| Heptachlor-epoxide (B) | 0.5±0.2 | 0.5±0.2 | 0.6±0.1 | 0.6±0.2 |
| Aldrin | 2.0±0.3 | 1.0±0.1 | 3.0±1.4 | 3.0±1.4 |
| Dieldrin | 0.6±0.1 | ND | 0.6±0.3 | 0.6±0.3 |
| Endrin | 0.9±0.2 | 0.9±0.1 | 0.7±0.3 | 0.8±0.6 |
| Endrin aldehyde | 0.9±0.2 | ND | 0.6±0.3 | 0.7±0.4 |
| Endrin ketone | 0 | ND | 0 | 0 |
| Cis-chlordane | 1.0±0.1 | ND | 0.7±0.5 | 0.5±0.5 |
| Trans-chlordane | 0.5±0.1 | ND | 0.5±0.2 | 0.8±0.3 |
| Endosulfan 1 | 0.7±0.2 | 0.9±0.2 | 0.6±0.3 | 0.6±0.1 |
| Endosulfan 11 | 0.7±0.1 | 2.0±1.2 | 0.5±0.4 | 0.6±0.3 |
| Endosulfan sulphate | 1.0±0.2 | ND | 2.0±0.6 | 1.0±0.5 |
| Methoxychlor | 0 | ND | 0 | 0 |
| p,p´-DDE | 0.7±0.2 | ND | 0.7±0.4 | 0.9±0.3 |
| p,p´-DDD | 2.0±0.2 | ND | 1.0±0.6 | 1.0±0.4 |
| p,p´-DDT | 0.7±0.3 | 14.0±9.5 | 0.7±0.5 | 0.6±0.2 |
| ΣDDT | 3.4±0.8 | 14.0±9.5 | 3.0±1.5 | 3.0±0.9 |
| ΣOCPs | 21.7±5.4 | 24.6±2.9 | 22.0±11.0 | 30.0±12.5 |

AGR-SW = microlayer water at Agboyi; AGR-BW = mixed layer water at Agboyi;

**Table 3.** Mean concentrations (ng/g) of organochlorine pesticide residues in epipellic and benthic sediments of Agboyi Creek during the dry and wet seasons.

| OCPs | Dry season | | Wet season | |
|---|---|---|---|---|
| | AGR-SS | AGR-BS | AGR-SS | AGR-BS |
| Alpha-BHC | 2.3±1.5 | 11.9±7.2 | 11.1±3.5 | 28.7±9.4 |
| Beta-BHC | 178.9±5.1 | 60.7±4.5 | 74.3±2.8 | 49.7±2.3 |
| Lindane | 8.2±1.6 | 24.7±2.3 | 25.4±8.3 | 83.3±4.4 |
| Delta-BHC | 9.1±3.2 | 111.2±4.1 | 96.3±5.2 | 20.9±2.3 |
| ΣBHC | 198.5±11.4 | 208.5±18.1 | 207.1±19.8 | 182.6±18.4 |
| Heptachlor | 5.6±2.4 | 44.9±5.6 | 49.5±4.2 | 125.6±8.5 |
| Heptachlor-epoxide(B) | 11.6±2.8 | 403.6±9.3 | 440.3±8.5 | 19.0±1.4 |
| Aldrin | 27.3±6.3 | 14.8±4.2 | 24.2±2.2 | 43.6±2.1 |
| Dieldrin | 17.5±7.6 | 37.2±7.5 | 33.0±8.5 | 38.9±7.5 |
| Endrin | 73.8±2.1 | 139.5±2.3 | 103.0±9.9 | 137.8±2.4 |
| Endrin aldehyde | 135.2±6.5 | 536.2±7.4 | 334.1±4.3 | 101.8±8.5 |
| Endrin ketone | 442.4±8.2 | ND | 426.8±2.8 | 691.7±5.8 |
| Cis-Chlordane | 18.4±3.4 | 28.4±2.5 | 61.1±3.3 | 53.7±3.2 |
| Trans-Chlordane | 16.1±8.1 | 90.8±6.2 | 34.7±7.2 | 40.8±2.3 |
| Endosulfan 1 | 30.2±2.0 | 37.7±3.6 | 39.4±2.1 | 69.5±3.2 |
| Endosulfan 11 | 30.1±4.5 | 55.3±6.3 | 76.9±8.5 | 139.7±8.7 |
| Endosulfan sulphate | 71.5±9.1 | 53.6±4.2 | 78.2±2.4 | 114.9±5.4 |

**Table 3.** Contd

| | | | | |
|---|---|---|---|---|
| Methoxychlor | ND | 146.7±8.2 | 20.1±5.3 | 74.2±4.2 |
| p,p´-DDE | ND | 43.1±5.6 | 41.2±2.5 | 66.3±8.2 |
| p,p´-DDD | 31.7±5.6 | 52.3±8.2 | 52.6±7.2 | 10.4±4.3 |
| p,p´-DDT | 26.9±3.8 | 44.5±2.4 | 104.9±3.3 | 102.3±8.4 |
| ΣDDT | 58.6±9.4 | 139.9±16.2 | 198.7±13 | 279.0±20.9 |
| ΣOCPs | 1138.9±83.8 | 1939.1±101.6 | 2121±89.3 | 2102±102.5 |

AGR-SS = epipellic sediment at Agboyi Creek; AGR-BS = benthic sediment at Agboyi Creek.

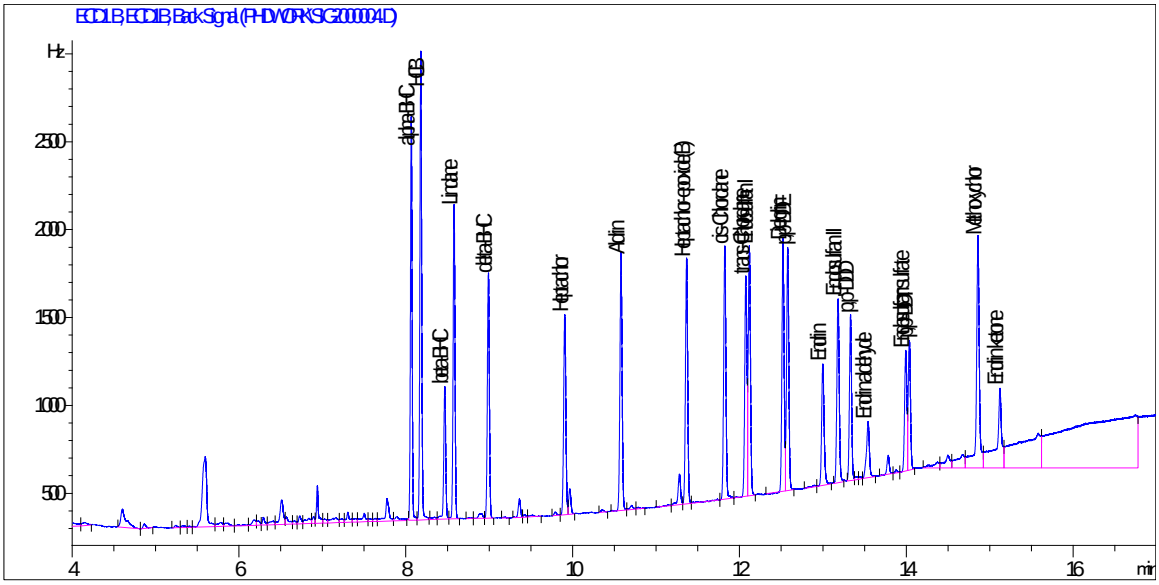

**Figure 1.** Chromatogram of OCP standard (10 ng/mL) run.

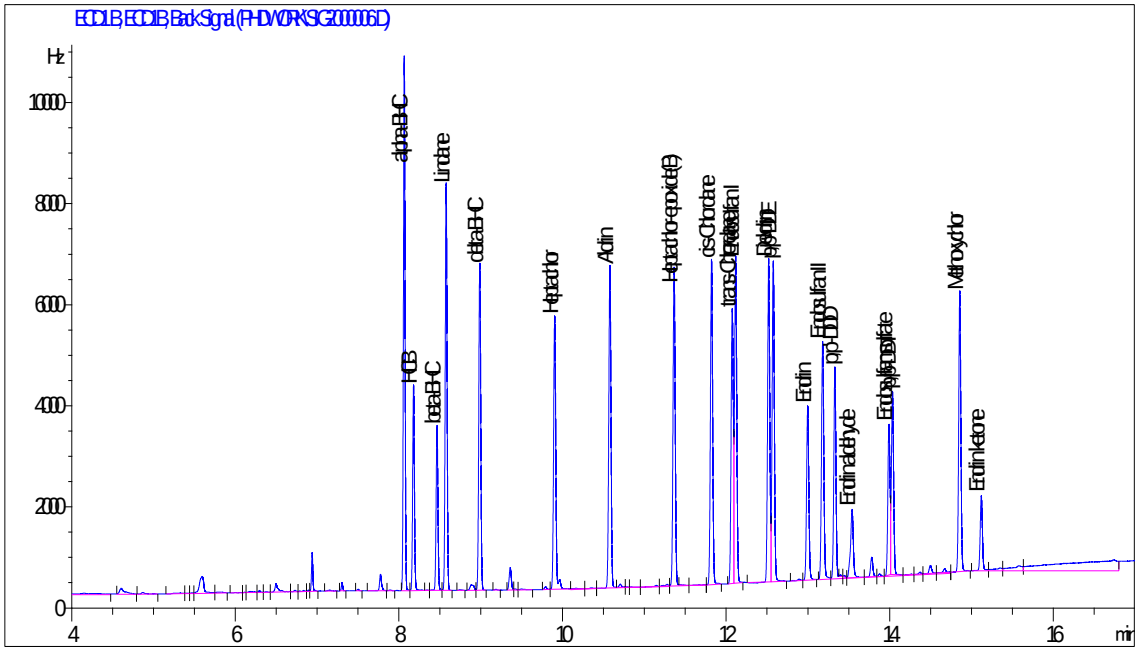

**Figure 2.** Chromatogram of OCP standard (40 ng/mL) run.

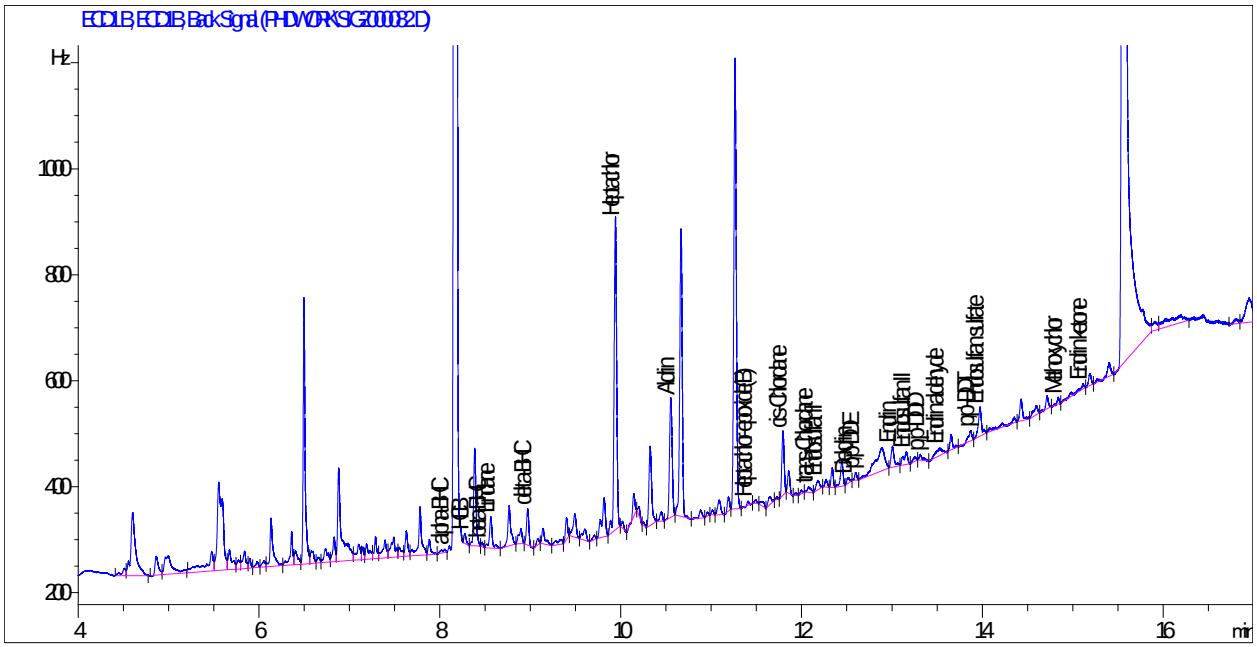


**Figure 3.** Chromatogram of OCP residues present in microlayer water in Agboyi Creek during the dry season.

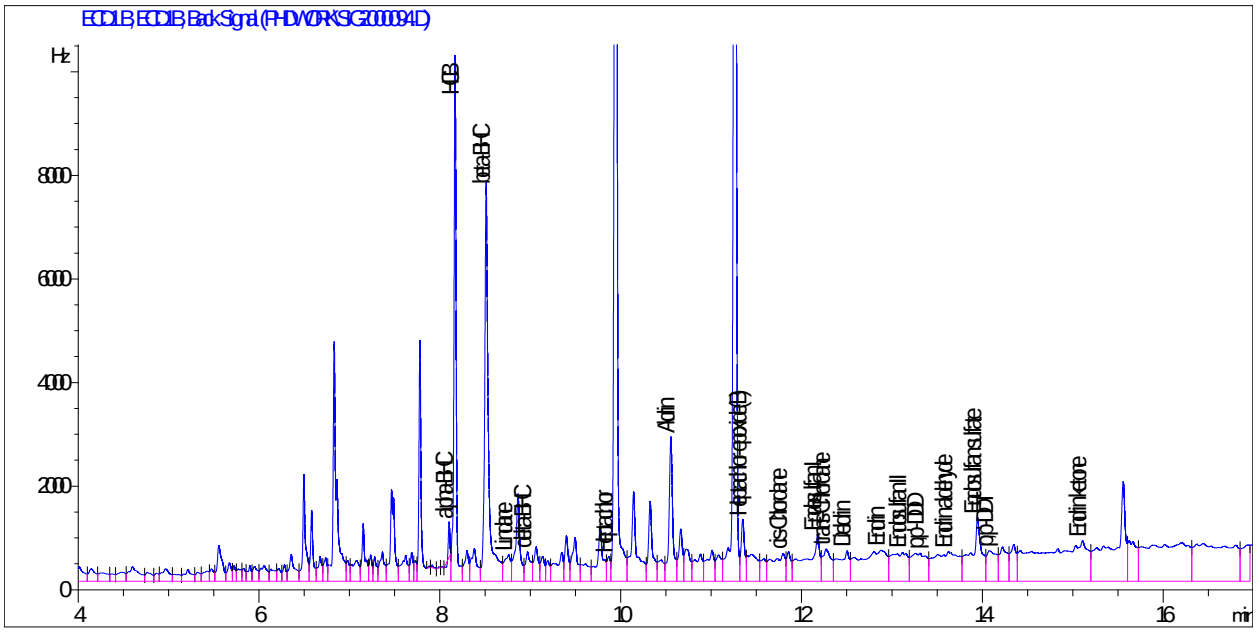


**Figure 4.** Chromatogram of OCP residues present in epipellic sediment in Agboyi Creek during the dry season.

2005;US EPA, 2006).

## Conclusion

A total of 23 OCPs were detected and determined in the two matrices at various sample locations for both dry and wet seasons. Concentrations of OCP residues were calculated individually and as the sum of their isomeric forms. Pesticide residues in the epipellic and benthic sediments were higher than the residues in the micro-layer and mixed layer water. This confirms the hydrophobicity of OCPs. The residue levels were higher during the dry season than the wet season. The levels of residues in the water and sediments were below the maximum permissive residue limits. The present studies could serve as a reference for future work on OCPs in microlayer and

mixed layer water, epipellic and benthic sediments of Agboyi Creek. Future work on the OCP and PCB levels in some biota in Agboyi Creek is encouraged.

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