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Organochlorine Pesticide Residues in Selected Surface Sediments within Ijebu-Ode and Environs, Ogun State, Nigeria

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Abstract

Organochlorine pesticides (OCPs) are a class of synthetic chemicals known for their persistence, toxicity, and bioaccumulation in the environment. These compounds are resistant to degradation by biological, chemical, microbiological, or physical processes, with half-lives ranging from months to decades. They are highly toxic, causing acute and chronic health effects. Despite being banned in many countries, organochlorine pesticides (OCPs) are still detected in the Nigerian environment and food chain, likely due to persistence, illegal trade, and improper disposal of old stockpiles. The detection frequencies of the OCPs in ascending order were 50.0 %, 56.3%, 62.5 %, and 62.5% at Ijagun, Ososa, Imoru, and Ogbogbo, respectively. Based on the detection frequencies, Imoru and Ososa are more polluted. Conversely, the total mean concentrations revealed that Ososa and Ogbogbo had the highest levels of OCP at 74.54 mg/kg and 79.56 mg/kg, respectively. Based on these findings, it can be concluded that the sites studied are polluted with OCPs. As such, removal strategies, such as phytoremediation or bioremediation procedures, should be developed to eliminate these pollutants from the environment.

Keywords: ***bioaccumulation, detection frequencies, GC-MS, surface sediments***

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Introduction

Organochlorine pesticides (OCPs) are a class of synthetic chemicals known for their persistence, toxicity, and bioaccumulation in the environment. These compounds, including dichlorodiphenyltrichloroethane (DDT), aldrin, dieldrin, and lindane, were widely used from the 1950s to the 1980s to enhance agricultural productivity and control vector-borne diseases like malaria and typhus (Baqar et al., 2018; Ferracane et al., 2023; Ma et al., 2020). Meanwhile, OCPs are resistant to degradation by biological, chemical, microbiological, or physical processes, with half-lives ranging from months to decades (Ferracane et al., 2023; Oyinloye et al., 2021). They accumulate in the fatty tissues of living organisms, including humans, leading to higher concentrations up the food chain (Mahugija et al., 2014; Oyinloye et al., 2021). This group of compounds is highly toxic, causing acute and chronic health effects such as gastrointestinal disorders, neurological damage, reproductive issues, endocrine disruption, and cancer (Fosu-Mensah et al., 2016; Mahugija et al., 2014; Opuni et al., 2023; Siddique et al., 2023). Less than 0.1% of applied OCPs reach their target pests, with the majority dispersing into the environment, contaminating soil, water, and non-target species (Baqar et al., 2018; Siddique et al., 2023). They can travel long distances from their application sites, even reaching polar regions and accumulating in the fat tissues of wildlife (Fosu-Mensah et al., 2016; Mahugija et al., 2014). More so, high ecological risks for the OCPs, endosulfan, and endrin, were reported in a study conducted in Pakistan (Baqar et al., 2018). Due to their adverse effects, many OCPs have been banned or restricted under international agreements like the Stockholm Convention on Persistent Organic Pollutants (Ferracane et al., 2023; Ma et al., 2020). Despite being banned in many countries, OCPs are still detected in the Nigerian environment and food chain, likely due to persistence, illegal trade, and improper disposal of old stockpiles (Iwegbue et al., 2024; Oyinloye et al., 2021; Taiwo et al., 2020). Furthermore, their usage could be linked to being cost-effective compared to alternative pesticides,

thereby making users overlook their downsides.

Chronic exposure to OCPs is linked to serious health issues, including liver and kidney damage, central nervous system disorders, and various cancers (Opuni et al., 2023; Siddique et al., 2023). They disrupt ecosystems, affecting both target and non-target species and causing long-term ecological harm (Fosu-Mensah et al., 2016; Siddique et al., 2023). In summary, while OCPs were initially valued for their effectiveness and low cost, their persistent and toxic nature has led to significant environmental and health concerns, prompting global efforts to regulate and mitigate their impact. One of the ways to achieve this is by monitoring their presence in the environment to ascertain their levels within the ecosystem. Hence, this study focused on investigating the presence of sixteen OCPs, including α-BHC, β-BHC, δ-BHC, and γ-BHC (BHC stands for benzene hexachloride, which is a chemical compound also known as hexachlorocyclohexane (HCH)). Other OCPs of interest in this study include heptachlor, aldrin, heptachlor epoxide, endosulfan I, dieldrin, endrin, endosulfan II, 4,4'-DDD, endrin aldehyde, endosulfan sulfate, 4,4'-DDT, and methoxychlor. Regarding the OCP contamination status within Ijebu-Ode, this study is one of the few that have investigated their occurrence pattern in the study area and Nigeria at large. It, therefore, aims to add to the pool of information regarding the presence of OCPs in surface sediments of Ijebu Ode.

Methodology

Description of the study area

The study area, Ijebu-Ode and its environs, is a town in Ogun State and close to the A121 highway. It is within the Southwestern geopolitical zone of Nigeria. Ijebu Ode is located 110 km by road Northeast of Lagos and within 100 km of the Atlantic Ocean in the eastern part of Ogun State. Purposive sampling was employed to collect surface sediments samples from four

sampling sites within this region, and the coordinates of each site were recorded using a GPS coordinate application. These sites included Ijagun (N6°46'54.8976", E3°55'51.42108"), Imoru (N6°48'24.07644", E3°55'29.63388"), Ososa (N6°48'7.87212", E3°51'46.79388") and Ogbogbo (N6°50'54.03048", E3°56'26.97468"), as shown in Figure 1. Composite samples were collected in December 2024, at four different strata from each site, making a total of sixteen samples collected from all the sites. Afterwards, representative samples were collected for each site using the quartering and coning methods. These samples were kept in ice chest coolers (to retain the integrity of the sample) for onward transfer to the laboratory. Sample analyses were carried out at the Ctxion Analytics Limited, Ikeja, Lagos, Nigeria.

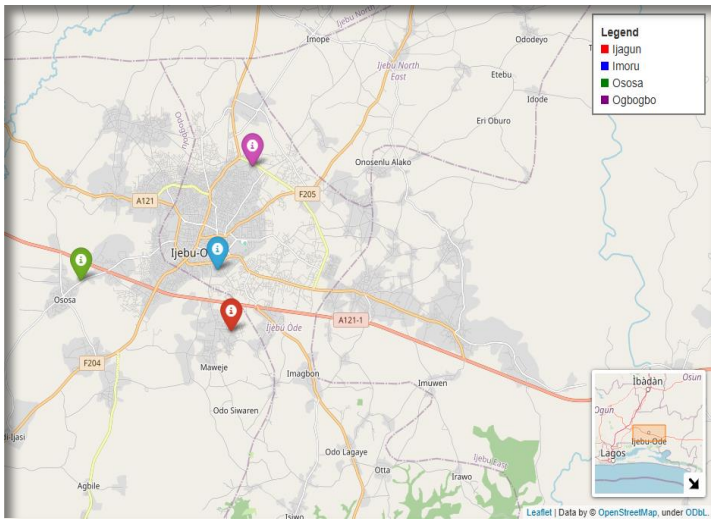

Fig. 1: Sampling site. Source: (Fieldwork, 2024)

Preparation of OCP standards and instrumental conditions

Standards containing sixteen OCPs (α-BHC, β-BHC, δ-BHC, γ-BHC, heptachlor, aldrin, heptachlor epoxide, endosulfan I, dieldrin, endrin, endosulfan II, 4,4'-DDD, endrin aldehyde, endosulfan sulfate, 4,4'-DDT and methoxychlor), (Catalog Number: M-527-BDE) were purchased from AccuStandard, United States of America. Six-point serial dilution calibration

standards (0.25, 0.125, 0.06, 0.03, 0.015, 0.0075 ppm) were prepared from the stock solution of these standards (2000 ppm) and used to calibrate the GC-MS, with all analyte OCPs eluted in less than 15 mins. QUECHERS Kit (Composition: 4g MgSO4 and 1g NaCl) was procured from Agilent, USA. An Agilent 7890B GC with an Agilent 5977B mass spectrometer detection (MSD) system equipped with a split/splitless inlet, and an Agilent 7693 Automatic Liquid Sampler was used for this analysis. The instrumental conditions used in the data acquisition in this study was informed by previous research works on OCPs (Bai et al., 2015; Devi et al., 2015), and are shown in Table 1. All the samples were analyzed in duplicates. The data obtained in this study were interpreted using Microsoft Excel.

Table 1: Instrumental conditions used in the data acquisition

GC	Agilent 7890B coupled with 5977B Mass selective Detector
Sampler	Agilent 7693 Injector tower with 10µL syringe
Carrier	Helium [flow; 1.2 mL/min]
Injection	1µL Splitless, 250 °C
Column	Rtx-1ms 15 m, 0.25 mm ID, 0.25 µm
Oven	50 °C (hold 0 min) to 270 °C at 15 °C/min (hold 0 min)
MSD	Scan mode, Transfer line temp [280] Quadrupole temp [150 °C] Ion source temp [230 °C]

Source: Instrumental conditions guided and informed by Bai et al. (2015) and Devi et al. (2015)

Sediment Extraction and Clean-up Procedures

Surface sediments were analyzed following a method previously developed by Anastassiades et al., (2003). This method included the sample extraction and clean-up procedures, as shown in Figures 2a and 2b. Briefly, the samples were sieved, and an aliquot (5 g) was transferred into a centrifuge tube where water, acetonitrile, and the content of the QuEChERS kit were added. The tube was vortexed and centrifuged to have a supernatant containing the analytes (OCPs) of interest. Further, this supernatant was cleaned up using

the dispersive solid phase extraction (dSPE) technique. The cleaned supernatant was then transferred into the GC vial for analysis on the GC-MS.

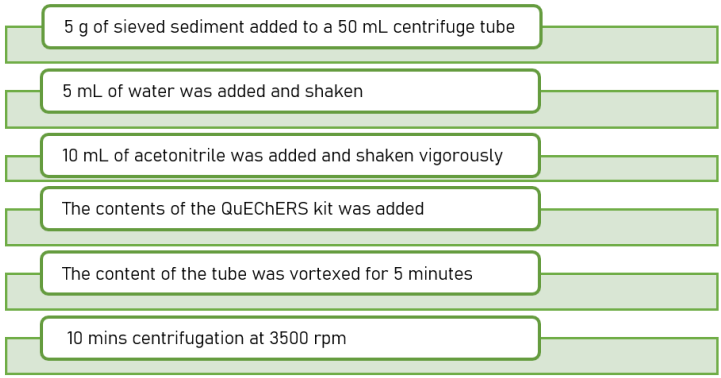

Fig. 2a: Extraction procedure for OCPs in surface sediments. Source: Guided and informed by Anastassiades et al., (2003)

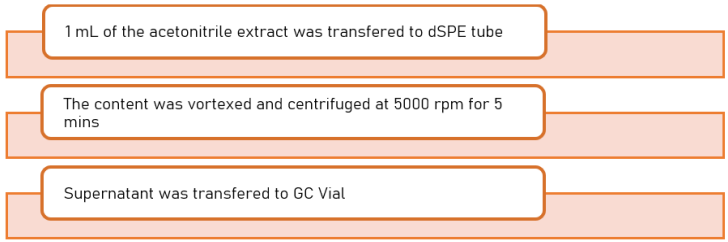

Fig. 2b: Clean-up procedure after surface sediments extraction. Source: Guided and informed by Anastassiades et al., (2003)

Quality Control Measures

Quality control measures taken during the OCP detection in sediments and on a general basis, are crucial for ensuring reliable and accurate results. These measures included blank analysis, and recovery OCP standards to assess contamination, and to ascertain the retention times of the analytes. Additionally, proper cleaning of glassware and equipment were carried out to prevent cross-contamination.

Results and Discussion

Surface sediments were collected from four different sites within Ijebu-Ode, Ogun State, pretreated, and analysed for the detection of sixteen OCPs on a gas chromatograph coupled with a mass spectrometer. A sample chromatogram of the analyte OCPs detected in this study is shown in Figure 3.

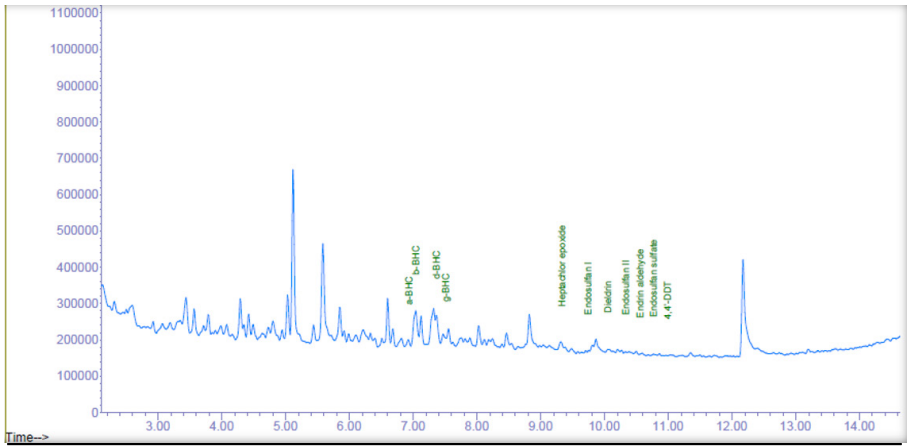

Fig. 3: Sample chromatogram of detected OCPs in the surface sediment samples. Source: (Author's sample chromatogram, 2024)

The levels of OCPs measured in the surface sediment samples are presented in Table 2. The limit of detection (mg/L) for the OCPs were 0.007, 0.009, 0.002, 0.004, 0.019, 0.022, 0.004, 0.023, 0.003, 0.034, 0.019, 0.022, 0.034, 0.019, 0.042 and 0.014 mg/L for α-BHC, β-BHC, δ-BHC, γ-BHC, heptachlor, aldrin, heptachlor epoxide, endosulfan I, dieldrin, endrin, endosulfan II, 4,4'-DDD, endrin aldehyde, endosulfan sulfate, 4,4'-DDT and methoxychlor, respectively. The sediment samples analysed showed the presence of the targeted OCPs in varying concentrations from non-detected (ND) in some sites, as shown in Table 2 to 28.13±0.05 mg/kg of β-BHC at Ogbogbo. However, variance analysis showed no statistically significant site differences ($p > 0.05$) in the mean concentrations of the detected OCPs in the sediment samples.

Table 2: Mean concentration (mg/kg) of detected OCPs in the studied surface sediment samples

	Ijagun		Imoru		Ososa		Ogbogbo	
OCP ANALYTES	Mean	S.D	Mean	S.D	Mean	S.D	Mean	S.D
α-BHC	ND	_	ND	_	ND	_	1.60	0.02
β-BHC	3.54	0.20	3.19	0.15	24.87	0.05	28.13	0.05
δ-BHC	2.41	0.11	6.98	4.68	0.12	0.04	8.56	0.06
γ-BHC	5.08	0.12	3.61	1.35	3.86	0.04	2.39	0.03
Heptachlor	ND	_	ND	_	ND	_	ND	_
Aldrin	ND	_	ND	_	ND	_	ND	_
Heptachlor epoxide	3.26	0.08	3.08	0.26	2.74	0.04	4.08	0.04
Endosulfan I	ND	_	3.70	3.70	3.68	0.04	1.23	0.07
Dieldrin	5.78	0.04	2.91	2.91	10.5	0.08	4.11	0.05
Endrin	ND	_	ND	_	ND	_	ND	_
Endosulfan II	8.45	0.03	5.99	2.43	8.81	0.03	8.06	0.04
4,4'-DDD	ND	_	ND	_	ND	_	ND	_
Endrin aldehyde	11.34	0.08	11.26	0.16	10.86	0.04	11.39	0.03
Endosulfan sulfate	7.35	0.15	7.43	0.23	9.1	0.06	10.31	0.05
4,4'-DDT	ND	_	0.01	0.01	ND	_	1.30	0.02
Methoxychlor	ND	_	ND	_	ND	_	ND	_

ND = not detected;
α = alpha; β = beta; δ = delta; γ = gamma; DDD = Dichlorodiphenyldichloroethane; DDT = Dichlorodiphenyltrichloroethane. Source: (Author's computation, 2025)

Heptachlor, aldrin, 4,4'-DDD, and methoxychlor were not detected in any of the sampling sites. The highest mean concentrations of OCPs reported for

each site were 11.34±0.08 mg/kg (endrin aldehyde); 11.26±0.16 mg/kg (endrin aldehyde); 24.87±0.05 mg/kg (β-BHC); 28.13±0.05 mg/kg (β-BHC) at Ijagun, Imoru, Ososa and Ogbogbo, respectively. The detection frequencies of the OCPs in ascending order were 50.0 %, 56.3%, 62.5 %, and 62.5% at Ijagun, Ososa, Imoru, and Ogbogbo, respectively. The spatial variation of the concentration of the OCPs shows that Imoru and Ososa are more polluted. Conversely, the total mean concentrations revealed that Ososa and Ogbogbo had the highest levels of OCP at 74.54 mg/kg and 79.56 mg/kg, respectively. Possible sources of the relatively high levels of OCPs found in these sites might be from farming activities that require the use of OCPs, which are prevalent in these areas. According to Figure 4, β-BHC had the highest concentration in the Ogbogbo surface sediment, followed by Ososa surface sediment.

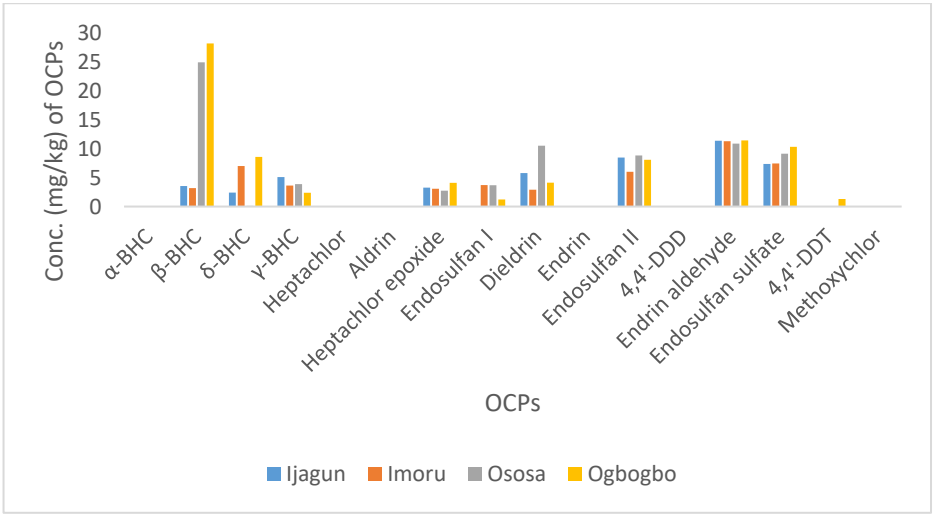

Fig. 4: Graphical representation of the mean levels (mg/kg) of detected OCPs in the studied sites. Source: (Author's computation and interpretation, 2025)

The concentrations of a few of the OCPs obtained in the study are below those reported by Benson et al., (2023) in sediments from the Lagos Lagoon, Gulf of

Guinea. Specifically, Benson and team members reported the levels of α-BHC as 0.03 mg/kg in their studied sites, while this study reported its presence in only one of the four sites under study (1.60±0.02 mg/kg) at Ogbogbo. However, the singular data obtained on the Ogbogbo sediment (1.60±0.02 mg/kg) exceeds those reported in the Lagos Lagoon (Benson et al., 2023). Furthermore, aldrin concentrations reported in the Lagos Lagoon were far above those reported in this study, as this OCP (Aldrin) was not detected in any of the sites studied. In the riverine ecosystem in the south-southern part of Nigeria, OCPs were detected in the sediment samples collected within this area with concentrations up to 0.000988 mg/kg (Ogbeide et al., 2019). This value is far below most of those obtained in this study, which might be due to the dilution effects of the river on the presence of the OCPs in the Ikpoba River. Moreover, concentrations of OCPs were reportedly higher in sediment samples than in water samples (Ogbeide et al., 2019). This could be the reason sediments obtained from rivers have relatively lower concentrations of OCPs compared to the samples obtained from dry lands. Most recently, sediments from the Escravos River basin in Nigeria were analysed for the presence of OCPs alongside other pollutants (Iwegbue et al., 2025). Similarly, Iwegbue and coauthors reported OCP concentrations below those obtained in this study except for α-BHC, which was not detected in this study in some of the sites (Iwegbue et al., 2025).

Conclusion

The present study aimed to investigate the levels of OCPs in selected sediments within Ijebu-Ode, Ogun State, Nigeria. The surface sediments were collected from four different sites within Ijebu-Ode, Ogun State, Nigeria, and analysed for the detection of sixteen OCPs using a gas chromatograph coupled to a mass spectrometer (GC-MS). Of the OCPs detected, β-BHC was found with the highest concentration in the Ogbogbo surface sediment, followed by the same OCP (β-BHC) in the Ososa surface sediment. The

findings from this study highlight the ongoing issue of OCP contamination in sediments within the Ijebu-Ode area of Ogun State, Nigeria. Future studies should investigate the associated risks of these detected compounds to both the environment and human health. Additionally, environmental monitoring studies on OCPs and other pollutants should be continuous, especially in the studied sites, as there are few studies on the subject matter in the selected environments.

The detection of OCPs in the studied environments necessitates immediate and coordinated remediation efforts. A thorough environmental assessment should be conducted to determine the extent and severity of the contamination. Several remediation methods, such as bioremediation and phytoremediation techniques, should be employed where feasible, utilizing microbes or plants to degrade or absorb the pesticides. In areas with high contamination levels, excavation and safe disposal of affected soils may be required. Continuous monitoring of soil, water, and biota should be implemented to track progress and prevent further spread. Additionally, public awareness campaigns and stricter enforcement of pesticide regulations are essential to mitigate future risks and promote sustainable land use practices.

REFERENCES

Anastassiades, M., Lehotay, S. J., Štajnbaher, D., and Schenck, F. J. (2003). Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce. *Journal of AOAC INTERNATIONAL, 86*(2), 412-431. doi:10.1093/jaoac/86.2.412

Bai, J., Lu, Q., Zhao, Q., Wang, J., Gao, Z., and Zhang, G. (2015). Organochlorine pesticides (OCPs) in wetland soils under different land uses along a 100-year chronosequence of reclamation in a Chinese estuary. *Scientific Reports, 5*(1), 17624. doi:10.1038/srep17624

Baqar, M., Sadef, Y., Ahmad, S. R., Mahmood, A., Li, J., and Zhang, G. (2018). Organochlorine pesticides across the tributaries of River Ravi, Pakistan: Human health risk assessment through dermal exposure, ecological risks, source fingerprints and spatio-temporal distribution. *Science of The Total Environment, 618*, 291-305. doi:https://doi.org/10.1016/j.scitotenv.2017.10.234

Benson, N. U., Unyimadu, J. P., and Tenebe, I. T. (2023). Distribution of organochlorine pesticides (OCPs) in surface-mixed layer water and intertidal sediments of Lagos lagoon, Gulf of Guinea. *Regional Studies in Marine Science, 67*, 103187. doi:https://doi.org/10.1016/j.rsma.2023.103187

Devi, N. L., Yadav, I. C., Raha, P., Shihua, Q., and Dan, Y. (2015). Spatial distribution, source apportionment and ecological risk assessment of residual organochlorine pesticides (OCPs) in the Himalayas. *Environ Sci Pollut Res Int, 22*(24), 20154-20166. doi:10.1007/s11356-015-5237-5

Ferracane, A., Manousi, N., Kabir, A., Furton, K. G., Tranchida, P. Q., Zachariadis, G. A., Płotka-Wasylka, J., Mondello, L., Samanidou, V. F., and Rosenberg, E. (2023). Monolithic capsule phase microextraction prior to gas chromatography-mass spectrometry for the determination of organochlorine pesticides in environmental water samples. *Microchemical Journal, 186*, 108355. doi:https://doi.org/10.1016/j.microc.2022.108355

Fosu-Mensah, B. Y., Okoffo, E. D., Darko, G., and Gordon, C. (2016). Assessment of organochlorine pesticide residues in soils and drinking water sources from cocoa farms in Ghana. *SpringerPlus, 5*(1), 869. doi:10.1186/s40064-016-2352-9

Iwegbue, C. M. A., Odali, E. W., Reuben-Okoye, M., Ossai, C. J., Ogwu, I. F., Olisah, C., and Martincigh, B. S. (2025). Organochlorine pesticides and polybrominated diphenyl ethers in sediments around oil production facilities in the Escravos River basin, Nigeria: Implications for ecological and human health risk. *Marine Pollution Bulletin, 213*, 117526. doi:https://doi.org/10.1016/j.marpolbul.2025.117526

Iwegbue, C. M. A., Ossai, C. J., Ogwu, I. F., Olisah, C., Ujam, O. T., Nwajei, G. E., and Martincigh, B. S. (2024). Organochlorine pesticide contamination of soils and dust from an urban environment in the Niger Delta of Nigeria. *Science of The Total Environment, 938*, 172959. doi:https://doi.org/10.1016/j.scitotenv.2024.172959

Ma, Y., Yun, X., Ruan, Z., Lu, C., Shi, Y., Qin, Q., Men, Z., Zou, D., Du, X., Xing, B., and Xie, Y. (2020). Review of hexachlorocyclohexane (HCH) and dichlorodiphenyltrichloroethane (DDT) contamination in Chinese soils. *Science of The Total Environment, 749*, 141212. doi:https://doi.org/10.1016/j.scitotenv.2020.141212

Mahugija, J. a. M., Henkelmann, B., and Schramm, K.-W. (2014). Levels, compositions and distributions of organochlorine pesticide residues in soil 5–14 years after clean-up of former storage sites in Tanzania. *Chemosphere, 117*, 330-337. doi:https://doi.org/10.1016/j.chemosphere.2014.07.052

Ogbeide, O., Uhunamure, G., Okundaye, F., and Ejeomo, C. (2019). First report on probabilistic risk assessment of pesticide residues in a riverine ecosystem in South-South Nigeria. *Chemosphere, 231*, 546-561. doi:https://doi.org/10.1016/j.chemosphere.2019.05.105

Opuni, K. F. M., Kretchy, J.-P., Agyabeng, K., Boadu, J. A., Adanu, T., Ankamah, S., Appiah, A., Amoah, G. B., Baidoo, M., and Kretchy, I. A. (2023). Contamination of herbal medicinal products in low-and-middle-income countries: A systematic review. *Heliyon, 9*(9), e19370. doi:https://doi.org/10.1016/j.heliyon.2023.e19370

Oyinloye, J. A., Oyekunle, J. a. O., Ogunfowokan, A. O., Msagati, T., Adekunle, A. S., and Nety, S. S. (2021). Human health risk assessments of organochlorine pesticides in some food crops from Esa-Oke farm settlement, Osun State, Nigeria. *Heliyon, 7*(7), e07470. doi:https://doi.org/10.1016/j.heliyon.2021.e07470

Siddique, S., Chaudhry, M. N., Ahmad, S. R., Nazir, R., Zhao, Z., Javed, R., Alghamdi, H. A., and Mahmood, A. (2023). Ecological and human health hazards; integrated risk assessment of organochlorine pesticides (OCPs) from the Chenab River, Pakistan. *Science of The Total Environment, 882*, 163504. doi:https://doi.org/10.1016/j.scitotenv.2023.163504

Taiwo, A. M., Philip, T. O., Akingbolahan, A. A., Tosin, B. E., Olajumoke, O. M., Helen, A. B., Lekan, O. S., Ajoke, B. A., Fatimah, M. O., Anthony, A. A., & Ahmad, S. M. (2020). Evaluating the potential health risk of organochlorine pesticides in selected protein foods from Abeokuta southwestern Nigeria. *Environmental Pollutants and Bioavailability, 32*(1), 131-145. doi:10.1080/26395940.2020.1816498