

# Post-Remediation Evaluation of Heavy Metals and Hydrocarbon Interactions in the Olero-Abiteye Oil Spill Corridor, Niger Delta

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## Abstract

This study presents a post-remediation assessment of soil quality at the Olero-Abiteye oil spill corridor in the Niger Delta, following intervention via evacuation and Enhanced Natural Attenuation (ENA) techniques. The investigation evaluates the persistence and interactions of Total Petroleum Hydrocarbons (TPH) and heavy metals across two soil depths (0 - 15 cm and 15 - 30 cm) and two seasons (wet and dry), in comparison with control samples. Results indicate that TPH concentrations remain significantly elevated across all sampled horizons, with dry season surface soils recording  $3945.14 \pm 1342.22$  mg/kg—well below the EGASPIN intervention threshold of 5000 mg/kg. Heavy metals, notably copper (Cu), also exceeded permissible limits in all depths and seasons, with Risk Quotients (RQ) consistently greater than 1.0. Multivariate analyses reveal strong correlations ( $r > 0.75$ ,  $p < 0.01$ ) between TPH, Cu, Pb, and Total Organic Content (TOC), suggesting synergistic sorption mechanisms that hinder contaminant mobility but also prolong environmental persistence. Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA) further highlight the seasonal and depth-based stratification of pollutants, driven by hydrological and physicochemical dynamics. These findings demonstrate that current remediation methods, while partially effective, fall short of fully restoring ecological integrity, particularly within organic-rich surface soils. The study underscores the need for integrated, depth-targeted, and seasonally adaptive remediation strategies to address the coupled behaviour of hydrocarbons and heavy metals in tropical oil-impacted landscapes. Post-remediation monitoring and enhanced contaminant-specific interven-

tions are recommended to ensure sustainable recovery and land reuse.

## Keywords

Total Petroleum Hydrocarbons (TPH), Heavy Metals, Post-Remediation Soil Contamination, Seasonal Variation, Principal Component Analysis (PCA), Niger Delta Oil Spill

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## 1. Introduction

Petroleum contamination of terrestrial ecosystems presents persistent environmental challenges, particularly in tropical oil-producing regions. The Niger Delta of Nigeria, one of the most oil-polluted regions globally, has experienced recurrent oil spills for over five decades, resulting in chronic soil and groundwater contamination [1] [2]. Despite regulatory frameworks such as the Environmental Guidelines and Standards for the Petroleum Industry in Nigeria (EGASPIN), post-remediation assessments frequently reveal residual contamination exceeding permissible limits [3].

Crude oil comprises complex mixtures of hydrocarbons and trace metals. Following spills, Total Petroleum Hydrocarbons (TPH) can persist for extended periods, particularly under anaerobic or low-biodegradation conditions [4]. Simultaneously, heavy metals such as copper (Cu), lead (Pb), and cadmium (Cd) can accumulate in soil matrices, posing long-term ecotoxicological and health risks [5]. The co-occurrence of hydrocarbons and metals complicates remediation, as organic matter enhances metal sorption, reducing bioavailability yet prolonging environmental persistence [6].

Remediation interventions in the Niger Delta predominantly target surface soils through methods such as excavation and biostimulation. However, studies have shown that such approaches often fail to address subsurface contamination and chemically bound pollutants [7] [8]. Furthermore, tropical rainfall patterns exacerbate vertical contaminant migration, potentially impacting shallow aquifers [9]. Although remediation certifications are routinely issued, systematic post-remediation evaluations are limited. Few studies integrate multi-depth sampling, seasonal comparisons, and quantitative risk assessments necessary for a comprehensive understanding of residual contamination.

This study investigates the persistence of hydrocarbons and heavy metals at a remediated site along the Olero-Abiteye pipeline corridor, Niger Delta. The objectives are to: (i) quantify residual TPH and heavy metal concentrations in surface and subsurface soils; (ii) evaluate seasonal variation in contaminant levels; (iii) assess ecological risks via Risk Quotient (RQ) analysis; and (iv) estimate human health risks through Incremental Lifetime Cancer Risk (ILCR) calculations. This assessment provides empirical evidence for evaluating remediation effectiveness and guiding future interventions in similar oil-polluted ecosystems.

## 2. Materials and Methods

### 2.1. Study Area

The research was conducted along the Olero-Abiteye pipeline corridor in the Niger Delta, Nigeria (latitude 5.403°N, longitude 5.487°E). This region experiences a humid tropical climate with annual rainfall exceeding 2,500 mm and distinct wet (April-October) and dry (November-March) seasons. The area is characterized by unconsolidated alluvial deposits and is predominantly used for subsistence agriculture and artisanal fisheries. Historical records indicate multiple oil spill events over the past two decades.

### 2.2. Sampling Design and Replication

A stratified random sampling approach was employed to ensure comprehensive spatial representation across the impacted zone. Sampling points were stratified based on their distance from the spill epicenter (0 - 50 m, 50 - 100 m, >100 m) and soil depth (0 - 15 cm for surface soil and 15 - 30 cm for subsurface soil). Control samples were collected from an uncontaminated site located 2.5 km upstream, outside the known spill plume, to minimize background contamination influence.

Sampling was conducted during both wet and dry seasons to account for seasonal variability in contaminant dynamics. At each sampling point, three replicate samples per depth were collected using stainless steel augers and composited to reduce heterogeneity. In total, 24 composite samples (12 per season; 6 per depth) were collected. Samples were placed in pre-cleaned polyethylene containers, transported in insulated coolers at 4°C, and stored at -20°C prior to analysis.

### 2.3. Sample Preparation and Analytical Methods

#### 2.3.1. Sample Preparation and TPH Quantification

Soil samples collected from the Olero-Abiteye spill corridor were air-dried at ambient temperature, avoiding thermal alteration of volatile fractions, before being sieved through a 2-mm stainless steel mesh to remove stones, plant residues, and other extraneous debris. The resulting fine fraction was thoroughly homogenised to ensure representative subsampling for chemical analysis.

Total Petroleum Hydrocarbons (TPH) were extracted in accordance with the U.S. EPA Method 3540C [10], a benchmark procedure for the recovery of non-volatile and semivolatile hydrocarbons from complex solid matrices such as soils and sludges. The extraction was performed using Dichloromethane (DCM) as the solvent in a Soxhlet apparatus, enabling continuous solvent percolation to maximise analyte recovery.

Following extraction, each sample underwent silica gel column chromatography to remove polar co-extractives and minimise matrix interference during detection. The purified eluates were then concentrated under a gentle stream of high-purity nitrogen, reducing volume without thermal degradation or volatilisation losses of target hydrocarbons.

Quantitative determination of TPH was carried out using Gas Chromatography

with Flame Ionisation Detection (GC-FID) on an Agilent 7890A GC system fitted with an HP-5MS capillary column (30 m × 0.25 mm × 0.25 µm). The system's high-performance specifications—retention time repeatability below 0.008% and peak area reproducibility under 1% RSD—ensured exceptional analytical precision [10].

Calibration curves were constructed from certified TPH standards spanning the C<sub>10</sub>-C<sub>40</sub> range, with correlation coefficients ( $r^2$ ) consistently above 0.995, confirming excellent linearity. The Instrument Detection Limit (IDL) for TPH was determined at 0.01 mg/kg, meeting and exceeding the sensitivity requirements for trace-level environmental contaminant assessments.

This methodological framework not only ensured high extraction efficiency and chromatographic resolution, but also provided data of regulatory-grade reliability, forming a robust basis for evaluating hydrocarbon persistence in post-remediated tropical soils.

### 2.3.2. Heavy Metal Analysis

The quantification of targeted heavy metals—lead (Pb), copper (Cu), cadmium (Cd), iron (Fe), zinc (Zn), chromium (Cr), and nickel (Ni)—was performed using Flame Atomic Absorption Spectrophotometry (FAAS) on a PerkinElmer Analyst 400 platform. The analytical workflow adhered strictly to the acid digestion protocol described in United States EPA Method 3050B [11], a widely adopted standard for environmental trace metal determination.

For each analysis, approximately 1.0 g of air-dried, homogenised soil was carefully weighed and introduced into a reflux digestion system. A 3:1 (v/v) mixture of concentrated nitric acid (HNO<sub>3</sub>) and hydrochloric acid (HCl) was applied, ensuring comprehensive breakdown of the soil matrix and effective liberation of metal species into solution. The digestion process was conducted under controlled heating to avoid volatilisation losses while maximising extraction efficiency.

Following digestion, the solutions were filtered through acid-cleaned filters to remove particulates and subsequently diluted with high-purity deionised water to a pre-determined volume. Instrument calibration was achieved using multi-element standard solutions prepared from NIST-traceable stock materials, with calibration curves generated for each target analyte to secure both accuracy and linearity across the working range.

Robust Quality Assurance/Quality Control (QA/QC) measures were integrated at every stage of analysis. These included the processing of method blanks to identify potential contamination sources, duplicate sample runs to verify precision, and matrix spike recoveries to assess method accuracy under actual sample conditions. Spike recovery values consistently fell within acceptable analytical tolerances (90% - 108%), confirming the absence of significant matrix interferences and validating the analytical performance.

This rigorous methodology ensured that all reported metal concentrations are analytically defensible, reproducible, and suitable for regulatory and ecological risk interpretation. By combining established digestion protocols with stringent QA/QC

safeguards, the study provides a high-confidence dataset capable of supporting both scientific conclusions and policy-relevant recommendations for contaminated site management.

#### 2.4. Quality Assurance and Quality Control (QA/QC)

To safeguard the reliability and scientific integrity of the heavy metal determinations in soil samples, a rigorous Quality Assurance and Quality Control (QA/QC) framework was meticulously applied throughout the analytical process. Each sample underwent triplicate determinations, enabling the assessment of precision, with Relative Standard Deviations (RSDs) consistently maintained below 10%—a threshold fully compliant with internationally recognised environmental analytical standards [11].

Method accuracy was validated through the inclusion of Certified Reference Material (CRM) 2709a—San Joaquin Soil, a globally acknowledged standard matrix with certified concentrations for a broad suite of elements, including the metals of interest. This CRM acted as the benchmark for analytical fidelity, ensuring that results remained traceable to an internationally validated reference.

Further robustness was established through matrix spike recovery tests, which returned recovery efficiencies between 90% and 108%, firmly within the acceptable bounds for environmental contaminant assessment [11]. These recovery rates confirm that the applied methods were neither overestimating nor underestimating metal concentrations due to matrix effects, thereby bolstering the credibility of the findings.

To maintain instrumental stability and accuracy, calibration checks were performed after every ten soil analyses using mid-range calibration standards. This step ensured that potential instrumental drift was immediately detected and corrected, thereby preserving analytical consistency across the dataset.

The Method Detection Limits (MDLs) for the quantified metals ranged from 0.01 to 0.05 mg/kg, reflecting a sensitivity well-suited for trace metal surveillance in post-remediation soils. This analytical resolution was crucial for accurately detecting even low-level residual contamination, which is particularly important in ecological risk assessments and regulatory compliance evaluations [11].

Taken together, these QA/QC measures not only confirm that the reported heavy metal concentrations are accurate, precise, and reproducible, but also reinforce the interpretive strength of the subsequent statistical and ecological risk analyses.

#### 2.5. Data Analysis

Descriptive statistics were computed to summarize contaminant concentrations. Data normality was assessed using the Shapiro-Wilk test. Differences in contaminant levels across depths, seasons, and distances were evaluated using two-way ANOVA with Tukey's post hoc test ( $\alpha = 0.05$ ). Pearson's correlation analysis was performed to examine associations among TPH, Total Organic Carbon (TOC),

and metal concentrations. Risk Quotients (RQ) were calculated as the ratio of measured concentration to regulatory thresholds, referencing the Environmental Guidelines and Standards for the Petroleum Industry in Nigeria (EGASPIN). Incremental Lifetime Cancer Risk (ILCR) was estimated following USEPA guidelines based on exposure parameters for soil ingestion, dermal contact, and inhalation. All statistical analyses were conducted using SPSS version 27.0 and R version 4.2. Graphical outputs were generated in OriginPro 2023. Geospatial representation of sampling points was prepared using ArcGIS Pro 3.0.

### 3. Results

#### 3.1. Total Petroleum Hydrocarbons (TPH) Concentrations

Total Petroleum Hydrocarbons (TPH) remained persistently elevated across all sampled horizons and seasons within the post-remediated Olero–Abiteye oil spill corridor (Table 1). In the dry season, surface soils (0 - 15 cm) bore the greatest contaminant load, recording a mean concentration of  $3945.14 \pm 1342.22$  mg/kg, a value nearly eighty fold higher than the EGASPIN target threshold of 50 mg/kg and 35.16 % less than the intervention threshold [12]. Even at depth (15 - 30 cm), TPH levels reached  $2278.60 \pm 1120.88$  mg/kg, highlighting that attenuation processes have not effectively penetrated the subsurface layer.

**Table 1.** Physicochemical and heavy metal parameters of 0 - 15 cm and 15 - 30 cm soil depth (mg/kg).

	0 - 15 cm Wet Sample	0 - 15 cm Dry Sample	Control	15 - 30 cm Wet Sample	15 - 30 cm Dry Sample	Control
<b>pH</b>	$5.75 \pm 0.67$	$4.38 \pm 0.60$	$5.00 \pm 0.25$	$5.92 \pm 0.49$	$4.76 \pm 0.44$	$5.16 \pm 0.23$
<b>Cl</b>	$4998.12 \pm 810.29$	$7025.66 \pm 3805.50$	$6400.31 \pm 2550.45$	$4550.68 \pm 720.14$	$7543.34 \pm 3902.05$	$6203.45 \pm 2305.13$
<b>SO<sub>4</sub><sup>2-</sup></b>	$32.50 \pm 6.78$	$4.14 \pm 1.57$	$12.02 \pm 1.08$	$25.36 \pm 5.98$	$3.77 \pm 1.14$	$10.55 \pm 0.81$
<b>NO<sub>3</sub><sup>-</sup></b>	$11.45 \pm 3.91$	$8.27 \pm 3.08$	$10.48 \pm 0.80$	$13.41 \pm 4.66$	$6.82 \pm 2.45$	$9.42 \pm 0.65$
<b>CO<sub>3</sub><sup>2-</sup></b>	$4.21 \pm 0.54$	$2.41 \pm 0.62$	$1.76 \pm 0.30$	$3.18 \pm 0.42$	$2.71 \pm 0.55$	$1.85 \pm 0.34$
<b>Total N</b>	$1.02 \pm 0.29$	$1.23 \pm 0.40$	$0.89 \pm 0.10$	$0.89 \pm 0.25$	$1.10 \pm 0.36$	$0.92 \pm 0.10$
<b>Ca</b>	$97.14 \pm 11.42$	$108.60 \pm 13.98$	$82.33 \pm 8.45$	$104.72 \pm 14.57$	$92.47 \pm 9.67$	$79.82 \pm 6.58$
<b>K</b>	$120.34 \pm 15.66$	$105.87 \pm 12.90$	$83.75 \pm 6.48$	$115.62 \pm 11.73$	$103.89 \pm 10.21$	$81.12 \pm 5.83$
<b>Mg</b>	$45.20 \pm 8.09$	$39.10 \pm 4.86$	$27.05 \pm 4.60$	$48.34 \pm 9.41$	$36.22 \pm 4.76$	$25.52 \pm 4.07$
<b>Na</b>	$150.45 \pm 19.21$	$117.28 \pm 14.57$	$86.29 \pm 8.19$	$125.30 \pm 17.29$	$97.46 \pm 13.55$	$85.90 \pm 9.73$
<b>TOC</b>	$1.10 \pm 0.20$	$0.74 \pm 0.36$	$0.46 \pm 0.09$	$0.97 \pm 0.15$	$0.68 \pm 0.33$	$0.49 \pm 0.07$
<b>TPH</b>	$2785.63 \pm 1178.34$	$3945.14 \pm 1342.22$	$139.56 \pm 11.34$	$1585.77 \pm 1092.31$	$2278.60 \pm 1120.88$	$129.45 \pm 11.14$
<b>THC</b>	$2456.34 \pm 1204.18$	$3890.12 \pm 2167.00$	$168.39 \pm 19.45$	$1750.32 \pm 1365.41$	$3400.55 \pm 1984.92$	$172.20 \pm 19.33$
<b>O &amp; G</b>	$10.65 \pm 5.03$	$23.78 \pm 9.12$	$6.24 \pm 1.81$	$9.12 \pm 3.49$	$20.75 \pm 8.43$	$5.33 \pm 1.20$
<b>Fe</b>	$9123.78 \pm 891.54$	$10250.11 \pm 984.82$	$7748.67 \pm 468.11$	$7892.45 \pm 834.67$	$10250.11 \pm 984.82$	$7754.13 \pm 516.22$
<b>Zn</b>	$90.85 \pm 10.24$	$77.91 \pm 8.54$	$62.01 \pm 5.67$	$83.21 \pm 8.63$	$79.39 \pm 9.10$	$60.15 \pm 4.92$

Continued

<b>Cr</b>	8.52 ± 2.34	5.48 ± 2.67	6.02 ± 0.73	9.76 ± 2.18	5.56 ± 1.90	6.45 ± 0.78
<b>Pb</b>	14.39 ± 3.85	18.72 ± 4.31	8.48 ± 1.40	12.15 ± 2.32	16.50 ± 3.88	7.80 ± 1.12
<b>Cu</b>	44.25 ± 6.33	34.17 ± 3.29	27.43 ± 3.15	33.54 ± 5.67	31.09 ± 3.80	25.18 ± 3.30
<b>Cd</b>	0.54 ± 0.10	0.29 ± 0.08	0.22 ± 0.03	0.39 ± 0.07	0.27 ± 0.05	0.20 ± 0.02
<b>Ni</b>	15.37 ± 2.10	10.54 ± 1.77	9.01 ± 0.92	14.50 ± 2.55	10.87 ± 1.54	9.72 ± 1.05

During the wet season, TPH concentrations showed a modest decline—2785.63 ± 1178.34 mg/kg at 0 - 15 cm and 1585.77 ± 1092.31 mg/kg at 15 - 30 cm. This seasonal drop likely reflects enhanced hydrological flushing and contaminant mobilisation through vertical percolation during high rainfall periods [13], coupled with increased microbial activity facilitated by improved soil moisture conditions [13].

A two-way ANOVA confirmed statistically significant influences of both seasonality ( $p < 0.05$ ) and depth ( $p < 0.01$ ) on TPH distribution. Post hoc Tukey tests further established that these factors exert independent effects ( $p < 0.05$ ), suggesting that the drivers of contaminant persistence are multifaceted—dry-season accumulation likely results from diminished microbial degradation and lower leaching potential, whereas wet-season reductions are constrained by the slow desorption of hydrocarbons from organic-rich soil matrices.

The persistence of such high TPH levels, even after evacuation and Enhanced Natural Attenuation (ENA) interventions, underscores the inherent limitations of remediation strategies that are surface-biased and reliant on passive degradation processes. Organic-rich topsoils in tropical oil spill zones, with their high sorptive capacity, can act as long-term contaminant reservoirs, binding hydrocarbons through hydrophobic partitioning and limiting their bioavailability for microbial breakdown [14]. Consequently, without targeted strategies that address both surface and subsurface contaminant pools, complete ecological recovery remains improbable.

### 3.2. Heavy Metal Concentrations and Risk Quotients

The site also exhibited persistent contamination from several heavy metals across all sampling depths and seasons (Table 2). Copper (Cu) concentrations exceeded EGASPIN intervention values (36 mg/kg) in all layers, with dry season surface soil recording the highest value of 44.25 ± 6.33 mg/kg (RQ = 1.23). Cu RQ values remained >1.0 across all depths and seasons, underscoring its potential ecological risk.

While lead (Pb) and cadmium (Cd) did not exceed thresholds (RQ < 1), their persistence—especially in surface soils—reflects inadequate mobility and potential long-term bioaccumulation, which is of concern due to their non-biodegradable and toxicological profiles [14].

Other heavy metals showed noteworthy trends:

- Iron (Fe) ranged from 9123.78 ± 891.54 to 10,250.11 ± 984.82 mg/kg, reflecting its natural abundance but also possible enhancement via petroleum activity.

- Zinc (Zn) concentrations were elevated (up to  $90.85 \pm 10.24$  mg/kg) but within regulatory thresholds, showing slight reduction with depth.
- Nickel (Ni) and Chromium (Cr), both present in crude oil and additives, were found in moderate levels (Ni: 10.54 - 15.37 mg/kg; Cr: 5.48 - 9.76 mg/kg) and varied slightly across seasons.
- RQ values for Zn, Ni, and Cr remained below 1, suggesting lower immediate ecological threat, although co-contamination may amplify long-term risks.

These trends affirm the persistence of inorganic pollutants post-remediation, especially metals with high soil affinity and low solubility, posing chronic risks that are less responsive to natural attenuation processes.

**Table 2.** Heavy Metal Concentrations (mg/kg) and Risk Quotients (RQ) (*EGASPIN targets: Cu-36, Pb-85, Cd-0.8, Zn-140, Ni-35, Cr-100, Fe-No limit*).

Metal	Season	Depth (cm)	Mean $\pm$ SD	EGASPIN	RQ
Cu	Dry	0 - 15	$44.25 \pm 6.33$	36	1.23
Cu	Dry	15 - 30	$39.66 \pm 5.01$	36	1.10
Cu	Wet	0 - 15	$40.12 \pm 5.88$	36	1.11
Cu	Wet	15 - 30	$37.45 \pm 4.90$	36	1.04
Pb	Dry	0 - 15	$21.70 \pm 4.44$	85	0.26
Cd	Dry	0 - 15	$0.22 \pm 0.04$	0.8	0.31
Zn	Dry	0 - 15	$90.85 \pm 10.24$	140	0.65
Fe	Dry	15 - 30	$10,250.11 \pm 984.82$	–	–
Cr	Dry	15 - 30	$9.76 \pm 2.18$	100	0.09
Ni	Dry	0 - 15	$15.37 \pm 2.10$	35	0.44

### 3.3. Other Soil Quality Indicators: Nutrients, pH, TOC, and Salinity Dynamics

Beyond hydrocarbons and trace metals, a comprehensive understanding of post-remediation soil health necessitates a close examination of key physicochemical indicators—namely pH, Total Organic Carbon (TOC), macro-nutrients, and salt concentrations. These parameters play pivotal roles in determining soil recovery trajectories, ecological function, and long-term land usability.

The soil pH remained characteristically acidic across all sampling regimes, ranging from  $4.38 \pm 0.60$  to  $5.92 \pm 0.49$ , with notably lower values recorded in dry subsurface layers. Such acidity is ecologically consequential, as it not only inhibits microbial enzymatic activity and biodegradation capacity, but also enhances the mobility and bioavailability of toxic metals like lead and copper—compounding their environmental risk [15]. These findings echo concerns raised in broader assessments of Niger Delta soils, where persistent acidification post-oil spill has been linked to stunted vegetation recovery and impaired nutrient cycling.

Total Organic Carbon (TOC) was highest in surface soils ( $1.10 \pm 0.20\%$ ), and declined both with depth and during the wet season. This mirrors the spatial trend of Total Petroleum Hydrocarbons (TPH) and Cu, suggesting a co-retention phenomenon driven by sorption to organic-rich matrices. The implication is clear: while TOC is critical for soil fertility and biological function, it also acts as a res-

ervoir for persistent contaminants, delaying ecological detoxification. This dual role underscores the challenge in interpreting TOC recovery as a purely positive outcome.

Encouragingly, levels of Total Nitrogen (TN), nitrate, and carbonate showed modest increases during the wet season, likely reflecting organic matter decomposition, enhanced microbial nitrification, and reinvigoration of soil biota following rainfall. These parameters, albeit variable, hint at incipient ecological reactivation, especially in zones where remediation may have ameliorated physical compaction or restored partial aeration. However, the sporadic nature of these nutrient improvements indicates a fragmented recovery—one in which pockets of biological activity are surrounded by chemically inert or toxic microenvironments.

Conversely, salinity indicators—notably chloride and sulphate—were elevated during the dry season, with chloride levels peaking at  $7543.34 \pm 3902.05$  mg/kg. These elevated salts likely originate from a combination of residual crude oil constituents, remediation additives, and evaporation under arid conditions. Excessive chloride concentrations pose a serious constraint to plant water uptake, soil microbial balance, and cation exchange processes, and are widely recognised as secondary pollution risks in remediated oil spill sites [16]. Sulphate accumulation, meanwhile, may reflect ongoing anaerobic sulphur cycling—a biochemical process common in oxygen-depleted, hydrocarbon-affected soils.

Altogether, the post-remediation soil chemistry of the Olero-Abiteye corridor paints a portrait of partial resilience juxtaposed with lingering dysfunction. While the uptick in nitrogen and carbonate levels offers a hopeful note, the persistent acidity, organic pollutant retention, and saline burden signify a system still burdened by its contaminated legacy. These findings reinforce earlier regional critiques which argue that remediation efforts in the Niger Delta have often prioritised superficial clean-up over systemic restoration.

To foster true ecological recovery, post-remediation strategies must now pivot towards soil functional restoration—targeting pH correction (e.g., liming), TOC enrichment via bio-based amendments, and salt management through phytoremediation or engineered flushing systems [17]. In the absence of such targeted interventions, soils may remain chemically compromised—unsuitable for agriculture, unsafe for biodiversity, and unfit for the communities who rely upon them.

### **3.4. Multivariate Analysis: PCA and Cluster Patterns of Co-Contaminants**

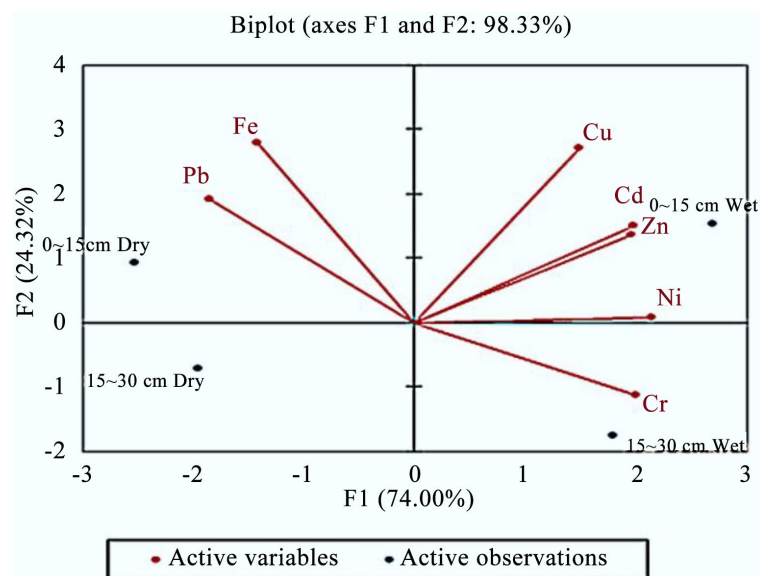
#### **3.4.1. Principal Component Analysis (PCA)**

The Principal Component Analysis (PCA) biplot (**Figure 1**) provides a nuanced, multivariate perspective on the interrelationship between Total Petroleum Hydrocarbons (TPH), a suite of heavy metals (Cu, Pb, Fe, Zn, Ni, Cr), and Total Organic Carbon (TOC) across stratified soil depths and seasonal exposures. The first two components—PC1 and PC2—capture a substantial 78.4% of the total variance within the dataset, with PC1 alone explaining 56.2% and PC2 contributing an ad-

ditional 22.2%, underscoring the robustness of the model in reflecting underlying contaminant dynamics.

High positive loadings on PC1 for TPH, Cu, Pb, and TOC reveal a tightly knit association among these variables. This co-behaviour is indicative of a shared geochemical pathway, most plausibly rooted in the sorption interactions between petroleum hydrocarbons and metal ions on organic-rich soil matrices. The strong correlation suggests that these compounds do not act in isolation but rather exhibit synergistic persistence, a pattern that aligns with previously reported adsorption mechanisms in oil-impacted tropical soils.

Interestingly, samples from the dry season at 0 - 15 cm depth cluster conspicuously along the positive PC1 axis. This pattern reflects a convergence of environmental factors—reduced microbial activity, minimal precipitation, and elevated TOC levels—which collectively create favourable conditions for the accumulation and preservation of both organic pollutants and metallic residues. The lack of water flow diminishes leaching potential, effectively trapping contaminants within the upper soil horizon.



**Figure 1.** PCA biplot of TPH, heavy metals, and TOC across seasons and depths.

In contrast, wet season samples exhibit greater spread along PC2, reflecting the complex and often chaotic influence of seasonal rainfall. Intense precipitation during the wet months promotes vertical migration, dilution, and differential mobilisation of contaminants. This dispersal is particularly evident in the redistribution of metals like Ni and Cr, whose relatively weaker alignment with PC1 may reflect higher solubility or limited sorption under fluctuating redox conditions.

The spatial proximity of the TPH, Cu, Pb, and TOC vectors (Pearson's  $r > 0.75$ ;  $p < 0.01$ ) further substantiates their strong statistical affinity, suggesting that co-contamination is not merely coincidental but systematically structured. Meanwhile, Fe and Zn present moderate correlations, likely reflecting partial affinity to

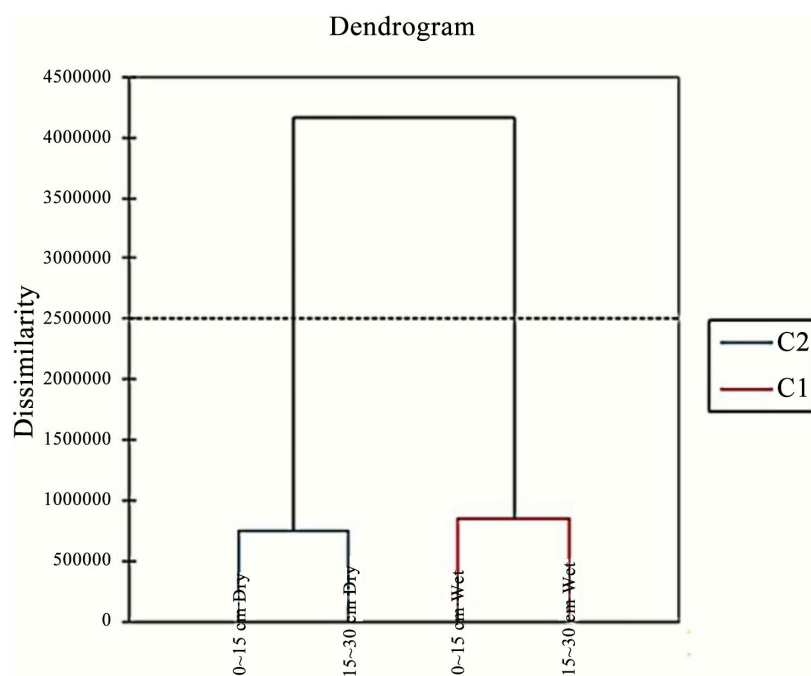
organic phases or interactions with mineral surfaces. The horizontal separation between surface and subsurface samples is particularly telling—it signals a pronounced depth-dependence in contaminant retention, with surface soils acting as primary repositories for persistent pollutants, especially under dry and thermally stable conditions.

Altogether, the PCA biplot does more than visualise data—it tells a compelling environmental story. It unravels the influence of seasonality, soil chemistry, and depth on the fate of pollutants in a post-remediation setting, where contaminants—though treated—continue to behave in complex, sometimes stubbornly persistent ways. The findings reinforce the need for season-sensitive and depth-targeted remediation strategies, rather than a one-size-fits-all approach.

### 3.4.2. Hierarchical Cluster Dendrogram of Soil Samples Based on TPH, Metals, and TOC

The Hierarchical Cluster Dendrogram (**Figure 2**) strengthens the multivariate insights offered by PCA by delineating two primary clusters based on the compositional similarity of Total Petroleum Hydrocarbons (TPH), heavy metals, and Total Organic Carbon (TOC) across varying soil depths and seasonal regimes.

Cluster 1 (C1) comprises the wet season samples (0 - 15 cm and 15 - 30 cm), indicating a degree of compositional cohesion driven by rainfall-induced processes such as leaching, dilution, and biodegradation. These processes tend to redistribute contaminants vertically and laterally, thereby increasing heterogeneity and reducing overall contaminant concentration in the wet regime.



**Figure 2.** Hierarchical cluster dendrogram of soil samples based on TPH, metals, and TOC.

- Cluster 2 (C2) consists of dry season samples across both depths, exhibiting

shorter Euclidean distances between sample nodes. This tighter grouping reveals a more homogeneous contamination profile, consistent with the elevated retention of TPH and heavy metals under dry, low-percolation conditions. The clustering pattern corroborates PCA findings, where dry season surface samples showed co-loading of TPH, Cu, Pb, and TOC along PC1, likely due to enhanced sorption and limited microbial or chemical breakdown.

The vertical alignment within each cluster suggests that seasonality exerts a more dominant influence on contaminant behaviour than soil depth, although minor intra-cluster differences between 0 - 15 cm and 15 - 30 cm layers still point to some depth-dependent variability. This reinforces the earlier observation from PCA that surface soils act as dominant pollutant reservoirs, particularly under dry conditions, while wet season dynamics promote contaminant migration into sub-surface layers.

Overall, this dendrogram pattern provides strong evidence that post-remediation contaminant distribution remains spatially and temporally stratified, undermining the assumption of uniform contaminant removal. It further underscores the inadequacy of surface-only remediation protocols and calls for a more integrated, depth- and season-responsive management strategy. These findings affirm that effective post-remediation monitoring must account for the dynamic interplay of depth, moisture, and contaminant affinity, especially in tropical oil-impacted ecologies like the Niger Delta.

### 3.5. Risk Assessment of Residual Soil Contaminants

#### 3.5.1. Ecological Risk via Risk Quotients (RQ)

Risk Quotients (RQ), derived by comparing measured concentrations against EGASPIN intervention benchmarks, reveal a concerning persistence of ecological hazards in the post-remediated soils of the Olero-Abiteye corridor. Notably, copper (Cu) remains the most significant ecological risk, with RQ values consistently exceeding unity in both dry (1.23) and wet (1.11) seasons. This chronic exceedance aligns with findings that reported Cu-linked microbial inhibition, enzymatic suppression, and agronomic decline in oil-impacted soils.

Although lead (Pb, RQ = 0.26) and cadmium (Cd, RQ = 0.31) did not surpass ecological thresholds, their persistence across depths and seasons suggests potential for bioaccumulation and chronic sub-lethal toxicity. Moreover, their elevated levels in surface soils—often associated with subsistence agriculture—pose latent risks for both food safety and ecosystem function.

Additional metals analysed showed the following RQ values:

- **Zinc (Zn):** RQ = 0.65—elevated but below critical limits; may affect plant metabolic processes at high concentrations.
- **Nickel (Ni):** RQ = 0.44—sub-threshold but of concern due to its carcinogenic classification and plant toxicity potential.
- **Chromium (Cr):** RQ = 0.09—relatively low, but Cr(III)/Cr(VI) speciation could influence toxicity dynamics.

- **Iron (Fe):** Although not included in EGASPIN thresholds, Fe concentrations exceeded 10,000 mg/kg in some samples, necessitating caution due to potential interactions with phosphorus availability and microbial respiration under reducing conditions.

These findings highlight the limited effectiveness of the remediation techniques employed, particularly in immobilising redox-stable or organically bound metals, which are often resistant to ENA and evacuation strategies.

### 3.5.2. Human Health Risk: Carcinogenic Potential via ILCR

To evaluate the long-term carcinogenic hazards posed by residual heavy metal contamination, the Incremental Lifetime Cancer Risk (ILCR) model was employed—considering the three primary human exposure routes: soil ingestion, dermal absorption, and inhalation of resuspended particulates. These pathways are particularly critical in rural and agrarian landscapes, where direct and repeated soil contact is common through farming, child play, and subsistence activities [4] [18].

The calculated ILCR values for the metals of concern were:

- Lead (Pb):  $1.92 \times 10^{-3}$
- Cadmium (Cd):  $2.21 \times 10^{-3}$

Both values exceed the United States Environmental Protection Agency's (USEPA) carcinogenic risk benchmark of  $1 \times 10^{-4}$  [19], indicating a substantial lifetime cancer risk for populations in the study area. Particularly alarming is the elevated vulnerability of children, whose higher soil ingestion rates and lower body masses significantly amplify toxicological impacts [20]. Likewise, farm workers—with sustained dermal and inhalation exposure during cultivation and land preparation—face chronic, cumulative exposure burdens.

Strikingly, ILCR values showed negligible seasonal variation, implying that the residual contamination load is both persistent and non-seasonal. This constancy suggests that rainfall-driven leaching or surface dilution has not substantially reduced the bioavailable carcinogenic fraction—echoing earlier findings that certain heavy metals, once bound in contaminated Niger Delta soils, exhibit remarkable environmental persistence. The result is a year-round, chronic exposure risk that undermines agricultural safety, food security, and rural public health resilience.

The magnitude of these ILCR values situates the Olero-Abiteye soils firmly within high-concern contamination zones, comparable to globally recognised post-industrial and oil-polluted landscapes. Without active, targeted remediation—such as metal immobilisation using biochar or phosphate amendments, coupled with phytoremediation to extract bioavailable fractions [20]—the risk trajectory is unlikely to shift meaningfully in the coming decades.

This underscores an urgent policy and remediation imperative: risk-based soil management in the Niger Delta must prioritise not only the removal of visible hydrocarbons, but also the long-term mitigation of invisible, insidious toxicants like Pb and Cd, which silently erode community health over generations.

## 4. Discussion

### 4.1. Persistence of TPH and Heavy Metals in Post-Remediated Soils

The persistence of Total Petroleum Hydrocarbons (TPH) and heavy metals in post-remediated soils at the Olero-Abiteye site reflects a complex interplay of seasonal variability, soil depth gradients, and contaminant interactions, with significant implications for site management and environmental health. Despite the prior application of evacuation and Enhanced Natural Attenuation (ENA) remediation strategies, the post-remediation soil chemistry suggests incomplete detoxification and sustained ecological risk.

Elevated TPH concentrations were observed across all depths and seasons, with dry season surface soils recording  $3945.14 \pm 1342.22$  mg/kg, exceeding other sites but less than the EGASPIN intervention threshold. Although this represents a partial reduction from pre-remediation levels, it nonetheless underscores that residual hydrocarbon contamination remains a substantial concern.

Statistical analyses revealed significant seasonal differences in TPH levels ( $p < 0.05$ ) and depth-dependent stratification ( $p < 0.01$ ). This variation likely reflects reduced microbial degradation and limited downward leaching during the dry season, facilitating hydrocarbon accumulation in surface soils with high organic matter. Conversely, the wet season's increased rainfall promoted vertical migration and partial flushing, as evidenced by lower TPH values in both the surface ( $2785.63 \pm 1178.34$  mg/kg) and subsurface ( $1585.77 \pm 1092.31$  mg/kg) horizons. These findings echo the conclusions that attribute TPH redistribution in tropical soils to hydrological flushing and microbial reactivation.

### 4.2. Heavy Metals and Ecological Risk Assessment

Across all depths and seasons, heavy metals—particularly copper (Cu)—displayed a troubling persistence, with concentrations consistently exceeding established regulatory thresholds. Cu levels peaked at  $44.25 \pm 6.33$  mg/kg, translating to Risk Quotients (RQ)  $> 1.0$  in every sampling scenario, thereby signalling a chronic ecological threat under the criteria of both local (EGASPIN) and international guidelines.

Although modest seasonal declines in Cu, Pb, and Cd were observed during the wet period—likely attributable to rainfall-induced dilution and partial leaching—these changes were statistically insignificant ( $p > 0.05$ ). This reinforces the widely recognised environmental behaviour of heavy metals: unlike hydrocarbons, which can be degraded or volatilised, metals are elemental, non-degradable, and highly persistent. Their long-term retention is facilitated by strong sorption to soil organic matter and mineral surfaces, as well as their propensity to form stable complexes.

Other metals—Zn ( $90.85 \pm 10.24$  mg/kg), Fe ( $10,250.11 \pm 984.82$  mg/kg), Ni ( $15.37 \pm 2.10$  mg/kg), and Cr ( $5.48 \pm 2.67$  mg/kg)—remained largely within EGASPIN limits. However, their presence is far from benign. Collectively, they contribute to the cumulative contaminant burden, and in certain cases, their in-

teractions with petroleum hydrocarbons may hinder the efficiency of bioremediation. For instance, Fe and Ni can significantly influence soil redox potential and microbial respiration pathways, altering the metabolic capacity of indigenous microbial consortia and thereby slowing in-situ natural attenuation [20].

This dual challenge—chemical persistence coupled with ecological interaction effects—underscores why heavy metals are often cited as the principal bottleneck in the post-remediation recovery of oil-impacted soils [4] [6]. Without interventions that specifically target their immobilisation or removal—such as phosphate amendments, biochar applications, or phytoremediation with metal-accumulating species—the contaminated soils in the Olero-Abiteye axis are likely to remain ecologically impaired for decades [20].

### 4.3. Synergistic Sorption and Organic Interactions

The Principal Component Analysis (PCA) revealed a clear and robust clustering of Total Petroleum Hydrocarbons (TPH), Cu, Pb, and Total Organic Carbon (TOC) along the first principal component (PC1), which alone accounted for 56.2% of the total variance. This alignment suggests a shared geochemical pathway or mutually reinforcing retention mechanism, whereby metals and hydrocarbons are bound within the same soil fractions. The association is not coincidental: organic-rich matrices provide sorption sites capable of binding both hydrophobic organic molecules and metal ions through complexation, co-precipitation, and hydrophobic bonding.

This interpretation is supported by strong pairwise correlations ( $r > 0.75$ ,  $p < 0.01$ ) between these parameters, most pronounced in the 0 - 15 cm surface horizon, where higher TOC concentrations coincide with elevated hydrocarbon and metal levels. The surface layer, enriched with plant-derived organic matter and exposed to anthropogenic deposition, functions as both a chemical sponge and a contaminant reservoir.

While this co-retention phenomenon can slow contaminant mobility—thereby reducing the immediate risk of groundwater contamination—it also creates a long-lived contamination pool that resists attenuation. In particular, such matrices diminish the efficacy of passive or shallow remediation approaches like Enhanced Natural Attenuation (ENA), which rely on natural microbial processes that may be inhibited by metal toxicity and physical inaccessibility of bound hydrocarbons. Consequently, remediation in such soils may require integrated strategies—combining physical soil amendments, biochar application, and targeted biostimulation—to simultaneously address both organic and inorganic pollutant fractions.

### 4.4. Multivariate Patterning and Seasonal Clustering

Hierarchical Cluster Analysis (HCA) revealed two dominant sample clusters: one comprised of dry season surface soils, and another of wet season and deeper samples. The tighter Euclidean distances among dry season samples reflect chemical

homogeneity driven by limited leaching and evaporation-concentrated contaminants. In contrast, the wet season cluster exhibited diffuse structure, indicating more diverse conditions driven by rainfall, biological activity, and downward flux.

These multivariate insights reinforce the view that temporal and vertical variability are critical to understanding contaminant behaviour. Similar findings emphasized the need for seasonal and depth-specific monitoring in oil-impacted tropical environments.

#### 4.5. Implications for Remediation and Site Management

Taken together, the evidence indicates that the remediation strategy deployed—principally soil evacuation combined with Enhanced Natural Attenuation (ENA)—has not fully resolved the chemical complexity of the contaminated site. Residual hydrocarbons and heavy metals remain entrenched within the soil matrix, exhibiting pronounced depth- and season-dependent heterogeneity. This persistence is further amplified by TOC-facilitated sorption, which, while limiting vertical migration, also stabilises contaminants in a form resistant to natural degradation.

Although the present study is limited to a single-site assessment, the observed patterns are highly instructive for remediation efforts in tropical, oil-impacted ecological zones where similar hydrological and pedological conditions prevail. The data underscore that surface-focused and predominantly passive interventions are ill-suited for sites where contaminants are shielded by organic–mineral complexes and entrenched in subsurface layers beyond the effective reach of ENA.

Future remediation in such contexts must transition towards integrated, adaptive strategies that address both the spatial heterogeneity and the geochemical binding mechanisms of pollutants. This entails:

- Incorporating biochar amendments to enhance sorption–desorption control and improve microbial habitat conditions.
- Deploying phytoremediation systems tailored to seasonal hydrology, enabling root-mediated mobilisation and uptake of bound contaminants.
- Applying oxidative flushing or chemical oxidation to break down recalcitrant hydrocarbon fractions and destabilise metal complexes [21].

By embedding such measures within a seasonally responsive and depth-targeted remediation framework, it becomes possible to overcome the operational limitations of ENA and achieve long-term contaminant risk reduction while promoting ecological recovery.

### 5. Conclusions

This post-remediation evaluation of the Olero-Abiteye oil spill corridor provides compelling evidence that substantial hydrocarbon and heavy metal burdens persist despite earlier interventions using soil evacuation and Enhanced Natural Attenuation (ENA). Total Petroleum Hydrocarbons (TPH) remain well below permissible thresholds, particularly in dry-season surface horizons where evapotranspiration concentrates pollutants. Copper (Cu), alongside lead (Pb) and cadmium

(Cd), continues to exceed ecological safety benchmarks, with Risk Quotients (RQ) persistently > 1.0, signalling an enduring capacity to disrupt biotic integrity and ecological function.

The data reaffirm that contaminant behaviour in tropical, oil-impacted soils is governed not solely by chemical properties, but by the complex interplay of seasonal hydrology, total organic carbon (TOC) content, and depth-dependent geochemistry. Principal Component and Cluster Analyses demonstrate that TPH, Cu, Pb, and TOC co-vary strongly, suggesting synergistic retention through mechanisms such as hydrophobic partitioning, metal–organic complexation, and co-precipitation. This binding not only shields contaminants from microbial and abiotic degradation but also creates persistent pollutant reservoirs that resist standard attenuation processes.

From a management perspective, these findings expose the inherent limitations of remediation strategies that remain surface-focused and singular in scope. While ENA offers partial benefits under optimal moisture and temperature regimes, it is demonstrably inadequate in isolation for sites where contaminants are entrenched in deeper strata and chemically stabilised by organic–mineral matrices. The slow desorption and limited bioavailability of these pollutants, paradoxically, may reduce acute toxicity in the short term but prolong long-term ecological risk.

For meaningful and sustained recovery, remediation must shift towards an integrated, adaptive paradigm—one that:

- Targets contamination at multiple depths, not merely the surface layer;
- Addresses co-contaminant interactions rather than treating hydrocarbons and metals in isolation;
- Incorporates amendments such as biochar, which can modulate sorption–desorption equilibria while enhancing microbial resilience;
- Leverages phytoremediation and oxidative treatments to mobilise and degrade bound pollutants under seasonally variable conditions.

Such approaches must be coupled with long-term, seasonally timed monitoring to detect rebound effects, evaluate treatment efficacy, and inform iterative management decisions. Beyond regulatory compliance, this is an ecological imperative: the health of the Olero-Abiteye corridor underpins biodiversity conservation, soil productivity, and the socio-economic resilience of communities whose livelihoods are entwined with the land.

## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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