



# GEOCHEMICAL ANALYSIS OF PETROLEUM CONTAMINATION IN SELECTED AREAS IN THE CALABAR RIVER MUDFLATS, SOUTHEASTERN NIGERIA

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## ABSTRACT

This study investigates the contamination of sediments along the Calabar River mudflats in southeastern Nigeria, based on hydrocarbon pollution from anthropogenic activities, such as industrial waste, untreated sewage, oil spills, and agricultural runoff. The aim is to assess the extent of contamination by examining the levels of total hydrocarbon content (THC), soluble organic matter (SOM), and petroleum-derived biomarkers at three locations: Adiabo Beach, Esuk Utan, and Nsidung Beach. Sediment samples were collected from these sites and analyzed for THC, SOM, and specific biomarkers using gas chromatography. Results indicate high levels of THC, with Esuk Utan exhibiting the highest concentration of 512.8 mg/kg, followed by Nsidung Beach (365.4 mg/kg), and Adiabo Beach (278.9 mg/kg). SOM values were similarly elevated, especially at Esuk Utan (15.4%), and the analysis of biomarkers revealed significant petroleum contamination, particularly at Esuk Utan, with a pristane/phytane ratio of 4.5, indicative of petroleum-derived hydrocarbons. The study shows that the sediment quality in the Calabar River mudflats is significantly impacted by various pollution sources, with Esuk Utan being the most affected. The results underscore the need for enhanced monitoring and management of industrial, sewage, and oil-related discharges in the region. A key recommendation is the need to implement strict regulations on industrial and sewage waste management in order to mitigate further ecological degradation and protect aquatic life.

**KEYWORDS:** Geochemical analysis, petroleum contamination, Calabar River, mudflats, southeastern, Nigeria.

## INTRODUCTION

Petroleum contamination has emerged as one of the most pressing environmental concerns worldwide, given its significant impact on ecosystems and human health. The pollution emits from various sources, including industrial activities, transportation, and natural or other anthropogenic events such as oil seeps. Petroleum products, which consist of hydrocarbons, have the potential to disrupt aquatic ecosystems, degrade water quality, and harm biodiversity (Mustafa *et al.*, 2024; Al-Robai *et al.*, 2023; Inyangetoh & Udoekpo, 2024). The global reliance on oil extraction and consumption has exacerbated the release of petroleum pollutants into the environment, often leading to long-lasting ecological damage (Luswet *et al.*, 2022; Ite *et al.*, 2018).

Industrial processes, such as oil drilling and refining, often result in the accidental discharge of petroleum hydrocarbons as oil seeps or discharge of untreated wastes into the surrounding environment. Additionally, transportation of oil through pipelines and ships poses a risk of spills, leading to widespread contamination of water bodies (Mishra *et al.*, 2023; Bashir *et al.*, 2020). Naturally occurring oil seeps, though less frequent, also contribute to petroleum pollution (Mohsenpour, 2020). The environmental degradation caused by petroleum contamination in aquatic ecosystems includes the destruction of habitats, disruption of nutrient cycles, and long-term toxicity to aquatic organisms (Ifon & Asuquo, 2021; Ameh *et al.*, 2023). Petroleum pollutants, primarily hydrocarbons, can persist in the environment for extended periods, accumulating in sediments and impacting the health of marine and freshwater species.

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(Al-Robai *et al.*, 2023; Nabebe *et al.*, 2024).

The Calabar River mudflats, located in the southeastern Niger Delta, play a crucial role in the local ecosystem. Mudflats serve as critical habitats for wildlife, particularly fish, invertebrates, and birds. The Calabar River mudflats provide a habitat for a wide range of species, such as shrimp, crabs, mollusks, and various finfish, as well as migratory birds that rely on the nutrient-rich sediments for feeding and breeding (Asuquo & Ifon, 2019). Additionally, they contribute to nutrient cycling, which supports the food web in the estuary. These areas are also vital for nutrient cycling, and shoreline stabilization. The region's mudflats act as a buffer zone, protecting the coastal areas from erosion and supporting the productivity of the entire estuarine ecosystem (Andem *et al.*, 2013; Agi-Odey *et al.*, 2024). The mudflats play an essential role in carbon sequestration by capturing organic carbon from plant and animal remains, thus mitigating the impacts of climate change (Asuquo *et al.*, 2018; Asuquo *et al.*, 2024).

These mudflats are vital for local livelihoods, are essential for aquaculture, as they provide ideal conditions for fish farming activities and agriculture (Inyang-Etoh *et al.*, 2024). Additionally, the sustainability of the mudflat ecosystem is thus intimately linked to the socioeconomic activities of the region's residents. The Calabar region has a long history of petroleum exploration, with both offshore and onshore drilling activities contributing to the region's economic development. However, these activities have resulted in the release of petroleum products into the environment, particularly in the Calabar River. The increasing frequency of oil spills and seepage in the region has raised concerns regarding the potential ecological damage and long-term impacts on local communities (Ekpo *et al.*, 2021a, Ekpo *et al.*, 2021b).

Several studies have explored petroleum contamination in other regions of the Niger Delta, highlighting the negative impacts of oil exploration and exploitation on water quality, sediment composition, and aquatic life (Ekpo *et al.*, 2023; Ola *et al.*, 2024a;

Ola *et al.*, 2024b). However, there remains a gap in the research specifically focused on the Calabar River mudflats, particularly regarding the extent of petroleum contamination in the area's sediments. Although studies have documented petroleum pollution in the Niger Delta, few have focused on the geochemical analysis of sediments in the Calabar River mudflats. This study aims to provide an in-depth organic geochemical analysis of surface sediments, using advanced techniques such as biomarkers, carbon isotope analysis, and Rock-Eval pyrolysis. These methods will provide valuable data on the extent of contamination and its ecological implications.

## MATERIALS AND METHODS

### Study Area

The Calabar River, a tributary of the Cross River in southeastern Nigeria, is situated between 04°57'N and 05°03'N latitude and 08°18'E longitude (Fig. 1). Originating from the hilly regions of northern Cross River State, the river traverses agricultural lands, villages, and urban areas before emptying into the Cross River estuary. The river is characterized by a combination of crystalline basement rocks from the Oban Massif and Cretaceous sediments of the Calabar Flank, with sediment dispersal influenced by tidal action and seasonal changes. Its depth varies between 1 meter to 10 meters during high tide and up to 8 meters during low tide (Asuquo, 1989).

The region experiences a tropical climate with annual rainfall exceeding 2,000 mm, most of which occurs between May and October. Temperatures range between 25°C and 32°C. Anthropogenic activities along the Calabar River include oil exploration, industrial discharges, agricultural runoff, and domestic sewage disposal, making it a hotspot for environmental contamination.

The Calabar River mudflats are ecologically significant, serving as habitats for diverse aquatic species and supporting local livelihoods through fishing and farming. Their proximity to oil exploration and industrial zones makes them vulnerable to petroleum contamination, necessitating this research to understand the extent of pollution and its ecological impacts.

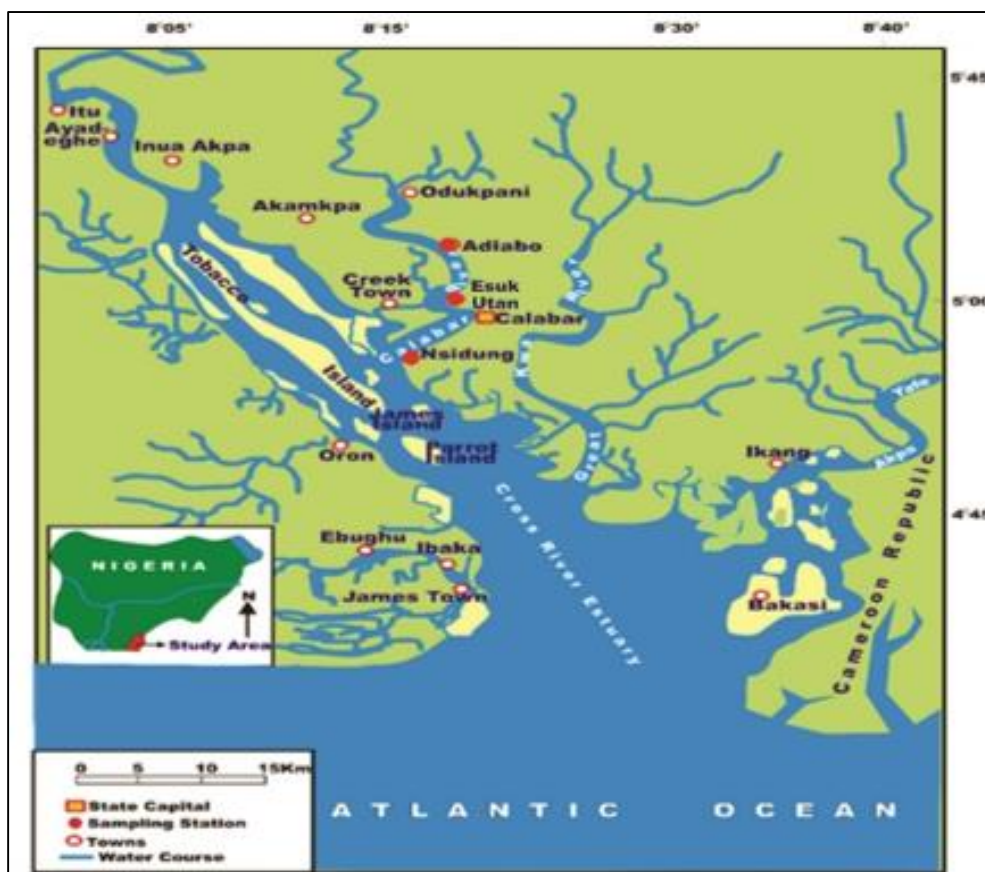


Figure 1: Map of the study area

### Sampling Procedures

Three locations along the Calabar River were selected for sediment sampling: Adiabo Beach (05°03'18.34"N, 008°18'19.25"E), Esuk Utan (05°00'37.05"N, 008°18'53.86"E), and Nsidung Beach (04°57'27.74"N, 008°18'32.23"E). Selection criteria included proximity to sources of pollution, such as untreated sewage, industrial waste, and petroleum activities, ensuring coverage of diverse contamination gradients (Table 1). Surface sediments (top 10 cm) were targeted for analysis due to their high interaction with contaminants and relevance to bioavailability studies. A modified Van Veen grab sampler (0.1m<sup>2</sup>) was employed to collect sediments. At each site, triplicate samples were taken from the left, middle, and right sections of the river to ensure spatial representation. Sediment samples were placed in pre-cleaned glass jars, labeled, placed on ice and transported to the laboratory for further analysis.

### Laboratory analysis

In the laboratory, samples were powdered, freeze-dried and sieved through a 63 µm mesh to isolate the fine fraction for analysis.

### Total Organic Carbon (TOC) Determination

TOC serves as an indicator of organic pollution and helps estimate the extent of petroleum hydrocarbon contamination.

The TOC content was measured using a LECO CS-444 analyzer after carbonate removal with 2N HCl. Rock-Eval pyrolysis (Peters, 2006) was conducted to characterize the composition of organic matter. The TOC percentage was calculated using the difference in pre- and post-combustion weights, providing insight into the organic content across samples.

### Extraction and Fractionation of Organic Matter

Organic compounds were extracted using a Soxhlet system with a hexane-acetone mixture (1:1) over 18 hours. Extracted organic matter was separated into saturated hydrocarbons, aromatic hydrocarbons, and polar compounds using silica-gel chromatography. Elution was performed with hexane for saturated fractions, dichloromethane for aromatics, and a methanol-dichloromethane mixture for polar compounds.

### Kerogen Isolation

Kerogen, the insoluble fraction of organic matter, offers insights into the origin and maturity of organic deposits. Powdered samples were treated sequentially with HCl and HF to remove carbonates and silicates, respectively. The resulting kerogen was purified using methanol and dichloromethane and dried for further analysis (Zhang *et al.*, 2023).

The isolated kerogen was visually examined under a microscope to assess its composition and structural features.

#### Carbon Isotope Determination

Carbon isotopic analysis differentiates between biogenic and anthropogenic sources of organic matter. A Finnigan Mat 251 mass spectrometer was used to measure the  $\delta^{13}\text{C}/^{12}\text{C}$  ratio, referencing the Pee Dee Belemnite (PDB) standard (Remigi *et al.*, 2023). Organic carbon was converted to  $\text{CO}_2$  via combustion at  $750^\circ\text{C}$ , ensuring accurate isotope measurement.  $\delta^{13}\text{C}$  values were analyzed to identify the contamination sources, distinguishing petroleum-derived from natural organic inputs.

#### Gas Chromatographic-Mass Spectrometric (GC-MS) Analysis

GC-MS was utilized to identify hydrocarbon compounds and biomarkers indicative of petroleum contamination. A Hewlett-Packard 5890 gas chromatograph equipped with a DB-5MS capillary column and helium as the carrier gas was employed (Steen, 1986; Okon *et al.*, 2021). The temperature was programmed from  $60^\circ\text{C}$  to  $295^\circ\text{C}$  at a rate of  $3^\circ\text{C}$  per minute. Sediment extracts were injected into the GC-MS system, and hydrocarbons were separated and identified using characteristic fragment ions (e.g.,  $m/z$  191 for tricyclic terpanes). Diagnostic ratios, such as pristane/phytane and hopane/sterane, were calculated to determine the sources and maturity of hydrocarbons.

#### Quality Assurance and Control

Certified reference materials were used to calibrate instruments, ensuring measurement accuracy. Duplicate samples were analyzed to validate the consistency of results. Procedural blanks and spiked samples were included to detect and correct potential contamination or analytical errors.

#### Statistical Analysis

TOC, isotope, and hydrocarbon data were analyzed using Python (ver. 3.12.6) to detect spatial and temporal contamination trends. Correlation analysis

was carried out to reveal the relationship between Total Organic Carbon (TOC) and Soluble Organic Matter (SOM)

## RESULTS AND DISCUSSION

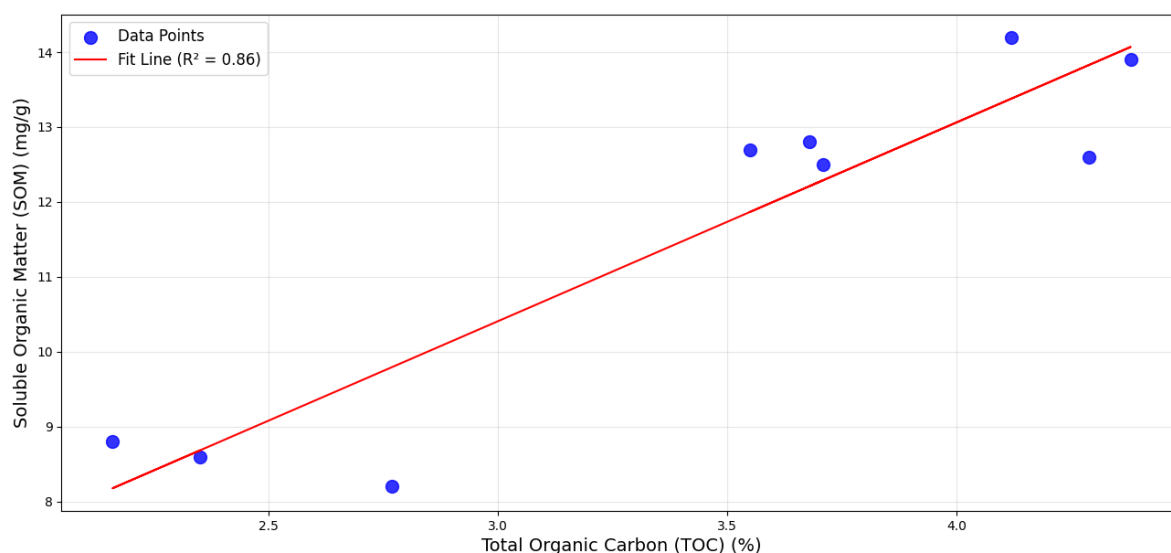
### Total Organic Carbon (TOC) and Soluble Organic Matter (SOM)

The Total Organic Carbon (TOC) content in the sediment samples varied significantly across the three locations, with Adiabo Beach recording the lowest levels ( $2.35 \pm 0.18\%$ ), followed by Nsidung Beach ( $3.68 \pm 0.21\%$ ), and Esuk Utan ( $4.12 \pm 0.25\%$ ) (Table 1). These findings are consistent with studies that observed high TOC concentrations in areas subjected to higher anthropogenic activity, such as industrial and port activities (Osuji & Adesiyun, 2005; Ikhajagbe & Chijioke-Osuji, 2015). Result shows relatively low TOC levels from Adiabo Beach which can be attributed to the predominant organic input from agricultural waste and untreated sewage. This is consistent with findings by Anh *et al.* (2023) who reported similar trends in coastal areas impacted by agriculture. Result also revealed very high TOC values in samples obtained from Esuk Utan, likely due to the influx of industrial waste, oil spills, and petroleum depot activities (Elenwo *et al.*, 2024). Similarly, high levels of TOC were also reported from Nsidung beach possibly due to oil spills and waste from port activities. Soluble Organic Matter (SOM) concentrations followed a similar trend, with Esuk Utan showing the highest values ( $14.2 \pm 1.3$  mg/g), followed by Nsidung Beach ( $12.8 \pm 1.1$  mg/g), and Adiabo Beach ( $8.6 \pm 0.9$  mg/g), which correlates with the findings of Ozigis *et al.* (2019) that reported elevated SOM levels in areas with increased industrial and oil pollution.

The correlation between TOC and SOM ( $R^2 = 0.86$ ) (Fig. 2) further supports the hypothesis that SOM is a reliable indicator of organic pollution. Previous studies have similarly found that high organic matter concentrations often correlate with increased contamination in coastal environments (Yang *et al.*, 2020; Yang *et al.*, 2023).

**Table 1: Organic geochemical parameters of sediments from three locations along the Calabar River mudflat**

| Location      | TOC (%)         | SOM (mg/g)     | THC (mg/kg)     | HI (mgHC/gTOC) | Tmax ( $^\circ\text{C}$ ) | CPI |
|---------------|-----------------|----------------|-----------------|----------------|---------------------------|-----|
| Adiabo Beach  | $2.35 \pm 0.18$ | $8.6 \pm 0.9$  | $58.2 \pm 3.1$  | 124            | 432                       | 3.2 |
| Esuk Utan     | $4.12 \pm 0.25$ | $14.2 \pm 1.3$ | $176.4 \pm 6.7$ | 178            | 445                       | 1.1 |
| Nsidung Beach | $3.68 \pm 0.21$ | $12.8 \pm 1.1$ | $153.7 \pm 5.4$ | 153            | 440                       | 1.5 |



**Figure 2: Correlation Between Total Organic Carbon (TOC) and Soluble Organic Matter (SOM)**

### Hydrocarbon Content

Total Hydrocarbon Content (THC) varied significantly between sites, with Adiabo Beach recording the lowest THC levels ( $58.2 \pm 3.1$  mg/kg), Esuk Utan exhibiting the highest THC values ( $176.4 \pm 6.7$  mg/kg), and Nsidung Beach showing moderate levels ( $153.7 \pm 5.4$  mg/kg) (Table 1). These results align with those of Dumka and Kingdom (2018), who found higher THC concentrations in areas with significant industrial waste and petroleum inputs. The elevated THC levels in Esuk Utan are directly attributed to the influence of industrial waste, oil spills, and petroleum depot activities, while THC levels observed in Nsidung Beach could reflect the combined impact of port activities and oil spills. The relatively low THC at Adiabo Beach suggests that agricultural runoff and untreated sewage may contribute less to hydrocarbon contamination, similar to findings by Rusănescu *et al.* (2022) in regions dominated by agricultural pollution.

### Pyrolysis Parameters

Rock-Eval pyrolysis results further highlighted the differences in organic matter composition and maturity across the sites (Table 1). The Hydrogen Index (HI) ranged from 124 to 178 mgHC/gTOC, with Esuk Utan and Nsidung Beach showing higher values, indicating a mix of biogenic and petroleum-derived hydrocarbons. Adiabo Beach, on the other hand, had lower HI values typical of terrestrial organic matter (Nady & Harb, 2015). Tmax values, which reflect organic matter maturity, ranged from 432°C to 445°C, with Esuk Utan showing slightly higher maturity due to

the higher presence of petroleum contamination (Stepanova *et al.*, 2022). These results are consistent with previous studies that showed Tmax values correlate with increased petroleum input (Evenick, 2021).

### Gas Chromatographic Analysis

Gas chromatographic analysis of the hydrocarbon components revealed distinct patterns across the sites. Samples from Adiabo Beach exhibited a dominance of long-chain n-alkanes (C27–C33), with a high Carbon Preference Index (CPI) of 3.2, which is characteristic of terrestrial organic matter (Herrera-Herrera *et al.*, 2020). Esuk Utan, which experienced high petroleum input, showed a significant unresolved complex mixture (UCM) and a low CPI of 1.1 with highest peak intensities at Esuk Utan (Fig. 3), indicating petroleum contamination. Nsidung Beach had a mixed profile with moderate UCM presence and a CPI of 1.5, suggesting both biogenic and petroleum inputs (Gao *et al.*, 2018). The biomarker analysis shows that pristane was highest ( $>5$  mg/g) in sediment samples from Esuk Utan and lowest ( $<3$  mg/g) in Adiabo (Fig. 4). This also confirmed the result showing pristane/phytane ratios of  $<1$  for Esuk Utan and Nsidung Beach, indicative of petroleum-derived hydrocarbons (Fig. 5), a trend that is consistent with previous research on port and industrial areas (Mohsenpour, 2020). Adiabo Beach had ratios  $>1$ , suggesting biogenic sources, which supports the finding that less industrial and petroleum contamination leads to higher ratios (Gao *et al.*, 2018).

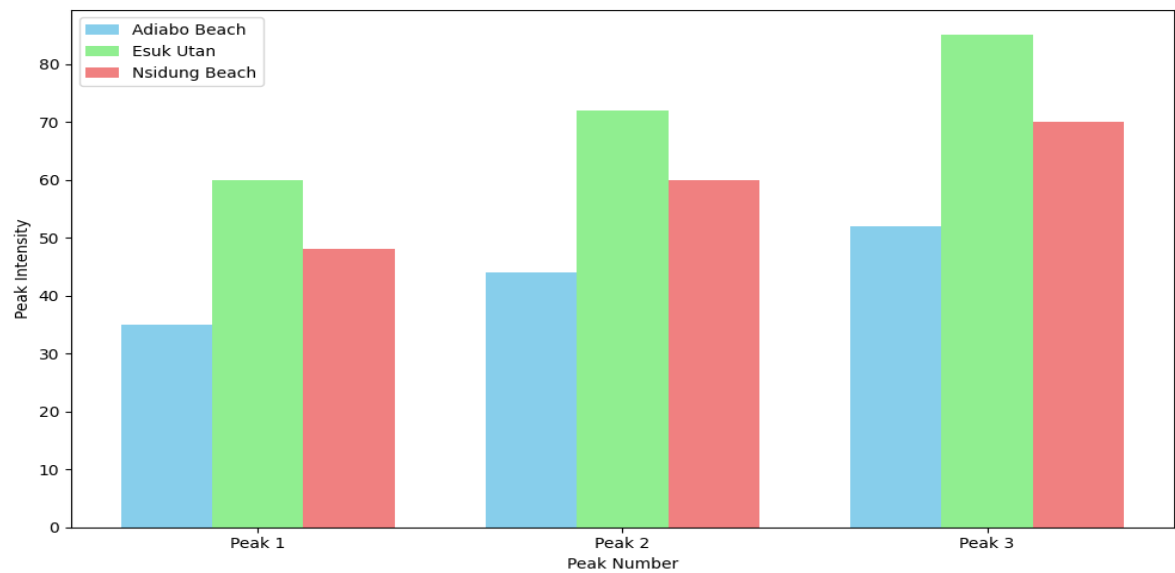


Figure 3: Gas chromatography analysis of hydrocarbons in sediments from the Calabar river mudflat

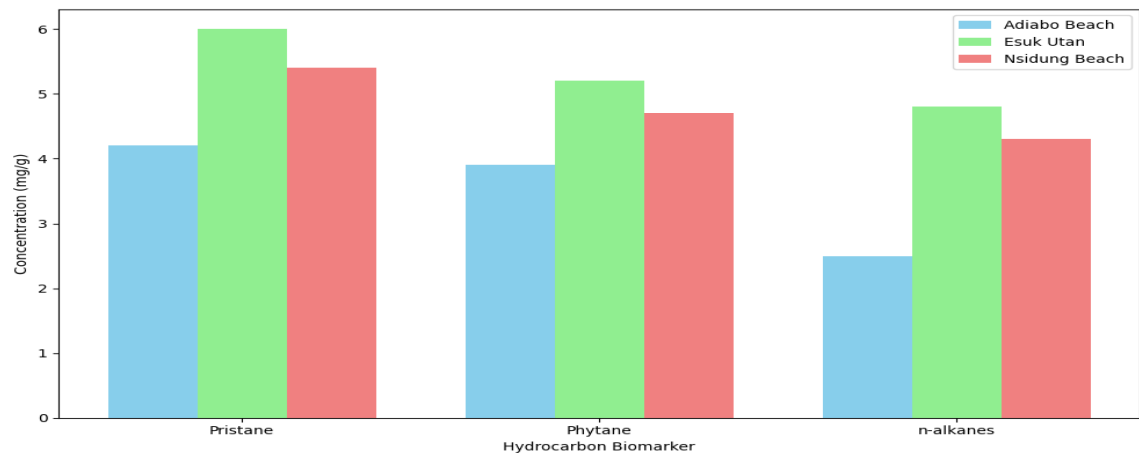


Figure 4: Biomarker analysis of petroleum-derived hydrocarbons in sediments from the Calabar river mudflat

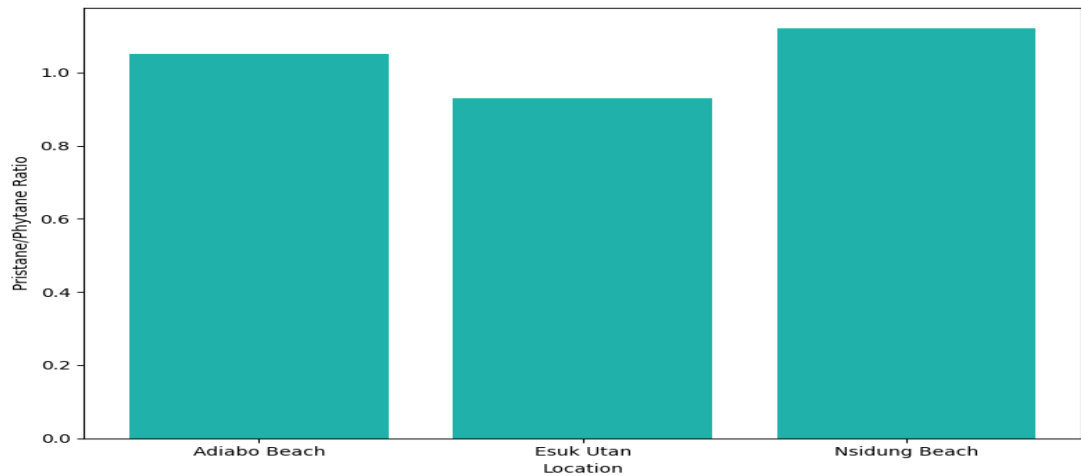


Figure 5: Pristane/Phytane ratio in sediments from Calabar River mudflat

## CONCLUSION

This study presents an in-depth analysis of the sediment quality across three locations along the Calabar River mudflats, focusing on various pollutants and hydrocarbon concentrations. The findings reveal significant differences in the levels of soluble organic matter (SOM), total hydrocarbon content (THC), and petroleum-derived biomarkers across the sampled locations. The sampled location at Esuk Utan showed the highest levels of THC and SOM, likely due to its proximity to industrial activities and untreated waste discharges. In contrast, sampled location at Adiabo Beach and Nsidung Beach exhibited relatively lower concentrations, though still indicative of contamination from various anthropogenic sources, including sewage, oil spills, and agricultural runoff.

Gas chromatographic analysis of hydrocarbons revealed distinct differences in the n-alkane profiles between locations, with higher peak intensities at Esuk Utan, further suggesting a significant petroleum influence. Additionally, the pristane/phytane ratios confirmed the presence of petroleum-derived hydrocarbons, with Esuk Utan showing the highest values, followed by Nsidung and Adiabo Beach. Biomarker analysis also indicated substantial contamination, reinforcing the impact of both point and non-point source pollution on sediment quality in the Calabar River mudflats.

These findings emphasize the urgent need for effective management strategies to address pollution sources in the study area. Given the potential environmental and ecological risks associated with hydrocarbon contamination, particularly its effects on aquatic life and local communities, it is crucial to implement both preventive and remediation measures. Monitoring and regulating industrial discharges, sewage management, and oil spill responses should be prioritized to mitigate further degradation of the Calabar River ecosystem.

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