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Assessment of Heavy Metal and Hydrocarbon Contamination in Wastewater from Warri Refinery and Petrochemical Company, Delta State, Nigeria, and the Remediation Potential of Kaolinite-Based Nanomaterials

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Abstract: *This study evaluated the pollution levels of industrial wastewater discharged from the Warri Refinery and Petrochemical Company (WRPC) in Delta State, Nigeria, and investigated the efficacy of kaolinite-based nanomaterials for contaminant removal. Wastewater samples were collected from five strategically selected sites and analyzed for physicochemical parameters and heavy metal content. Results revealed high levels of biochemical oxygen demand (BOD: 128.4 ± 6.5 mg/L), chemical oxygen demand (COD: 562.3 ± 14.6 mg/L), total dissolved solids (TDS: 1880 ± 44.7 mg/L), chromium (2.10 ± 0.09 mg/L), copper (1.78 ± 0.07 mg/L), nickel (1.43 ± 0.05 mg/L), total petroleum hydrocarbons (TPHs: 22.4 ± 1.6 mg/L), and phenols (3.8 ± 0.4 mg/L), all exceeding WHO and NESREA permissible limits. Pollution indices confirmed severe contamination, with Pollution Load Index (PLI) values exceeding 10.0 and Heavy Metal Pollution Index (HPI) reaching up to 6220.82. Adsorption studies using synthesized kaolinite nanomaterials showed high removal efficiencies: 91.2% for Cr, 88.7% for Cu, 86.3% for Ni, and 79.5% for TPHs. Adsorption capacities ranged from 1.04 to 3.98 mg/g, and the process followed the Freundlich isotherm model (R^2 > 0.98), indicating multilayer adsorption on heterogeneous surfaces. The findings highlight the environmental threat posed by untreated refinery effluents and demonstrate the potential of kaolinite nanomaterials as a cost-effective solution for industrial wastewater treatment.*

Keywords*: Kaolinite nanomaterial, Heavy metals, Warri Refinery, Adsorption, Water pollution*

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1.0 Introduction

Petroleum refining is indispensable to Nigeria's economy, yet its environmental footprint—particularly in the Niger Delta—is severe. Each barrel of crude processed generates 0.4–1.6 barrels of wastewater rich in refractory organics, phenols, oils, grease and trace metals (Cr, Cu, Ni), all of which are hazardous, persistent and prone to bio-accumulation (Awomeso et al., 2010; World Health Organization [WHO], 2017). The Warri Refinery and Petrochemical Company (WRPC) in Ekpan, Delta State, has operated since 1978 with an installed capacity of 125,000 barrels day^{-1}; studies around the Iffie, Ubeji and Ughoton creeks show effluent-driven elevations in turbidity, total dissolved solids, heavy metals and sulfides beyond national and WHO thresholds, rendering local water supplies unsafe for domestic and agricultural use arpejournal.com.

Multiple investigations corroborate the contamination profile of WRPC effluent. Atubi (2011) documented chromium and copper concentrations several-fold above permissible limits in adjacent surface waters arpejournal.com. Chukwu, Odubo and Iwuoha

(2021) reported mean COD of 560 mg L^{-1} and total petroleum hydrocarbons (TPH) up to 25 mg L^{-1} in refinery discharge canals—values that exceed NESREA standards. Igbagara (2023) found that although some parameters occasionally meet local regulations, periodic spikes in Cr, Ni and Zn still pose chronic ecological risks journal.fupre.edu.ng. Region-wide assessments of produced-water handling highlight systemic treatment shortfalls: COD, oil-and-grease and TDS often remain above Nigerian Upstream Petroleum Regulatory Commission targets despite conventional physico-chemical trains sciencepublishinggroup.com.

Concurrently, low-cost clay minerals—kaolinite, bentonite—have shown promise for simultaneous adsorption of metals and organics. Acid-activated or thermally modified kaolinite exhibits surface areas >80 m^2 g^{-1} and can remove >90 % of Cr(VI) in batch systems (Ekpete, Horsfall, & Tarawou, 2017). Yet, peer-reviewed data specifically coupling kaolinite-based nanomaterials with complex petroleum-refinery effluents remain scarce, especially for Nigerian installations.

Most WRPC studies focus on point-source characterisation or regulatory compliance rather than on integrated remediation solutions. There is limited empirical evidence on (i) combined heavy-metal and hydrocarbon removal efficiencies using readily available Nigerian clays engineered to the nanoscale, and (ii) isotherm/kinetic behaviour of such adsorbents under real wastewater matrices. Addressing these gaps is critical for formulating sustainable, locally sourced treatment options.

This work aims to (i) quantify present-day levels of key contaminants (Cr, Cu, Ni, TPH, phenols, COD, BOD) in effluents from WRPC discharge points; and (ii) evaluate the adsorption performance of acid-activated, thermally treated kaolinite nanomaterials for simultaneous removal of heavy metals and hydrocarbons under batch conditions.

Outcomes will (a) provide an updated pollution baseline indispensable for regulatory oversight and risk assessment in the Niger Delta, (b) deliver proof-of-concept data for a cost-effective, locally sourced remediation technology, and (c) inform scale-up possibilities—such as fixed-bed columns or constructed wetlands—for refinery operators and policy makers striving to meet Nigeria's revised National Environmental (Petroleum Refining Operations) Regulations. Ultimately, the study supports Sustainable Development Goals 6 (clean water) and 14 (life below water) by proposing a tangible pathway to reduce industrial effluent toxicity in coastal communities.and Petrochemical Company is located in Ekpan, near Effurun in Uvwie Local Government Area of Delta State, southern Nigeria. Situated in the Niger Delta, the area is characterized by a tropical wet climate and is home to several rivers and creeks that support biodiversity and human livelihood. WRPC has a refining capacity of 125,000 barrels per day and has been operational since 1978. The refinery is surrounded by residential communities and aquatic environments that are vulnerable to pollution.

2.1 Study Area Description

The study was conducted around the Warri Refinery and Petrochemical Company (WRPC), located in Ekpan, a town within Uvwie Local Government Area, Delta State, in the Niger Delta region of southern Nigeria. Commissioned in 1978, WRPC is one of Nigeria's four government-owned refineries and has a refining capacity of 125,000 barrels per day. The refinery is strategically situated adjacent to the Warri River and the Forcados Terminal, which facilitates crude oil import and refined-product export operations.

Geographically, the WRPC is positioned at approximately latitude 5°31'N and longitude 5°45'E, within the humid tropical belt characterized by heavy rainfall (1,500–3,000 mm/year), mean annual temperature of 27–

30°C, and high relative humidity (70–90%). The region lies within the lower Niger Delta floodplain, an ecologically sensitive wetland area interlaced with creeks, rivers, and estuaries.

The local water bodies—including the Iffie River, Ubeji Creek, Ughoton Creek, and Esisi River—serve as natural receptors for refinery effluents, stormwater runoff, and illegal discharges. These rivers are integral to the livelihoods of surrounding communities, supporting domestic use, artisanal fishing, irrigation, and small-scale aquaculture. However, reports of fish kills, oil sheens, and strong chemical odours around these water bodies underscore the urgency for rigorous pollution assessment and mitigation.

2.2 Sampling Locations

To comprehensively evaluate the contamination profile and remediation potential, effluent and water samples were collected from five key locations based on proximity to WRPC discharge points and downstream flow patterns. The specific locations are contained in Fig. 1 and described in Table 1

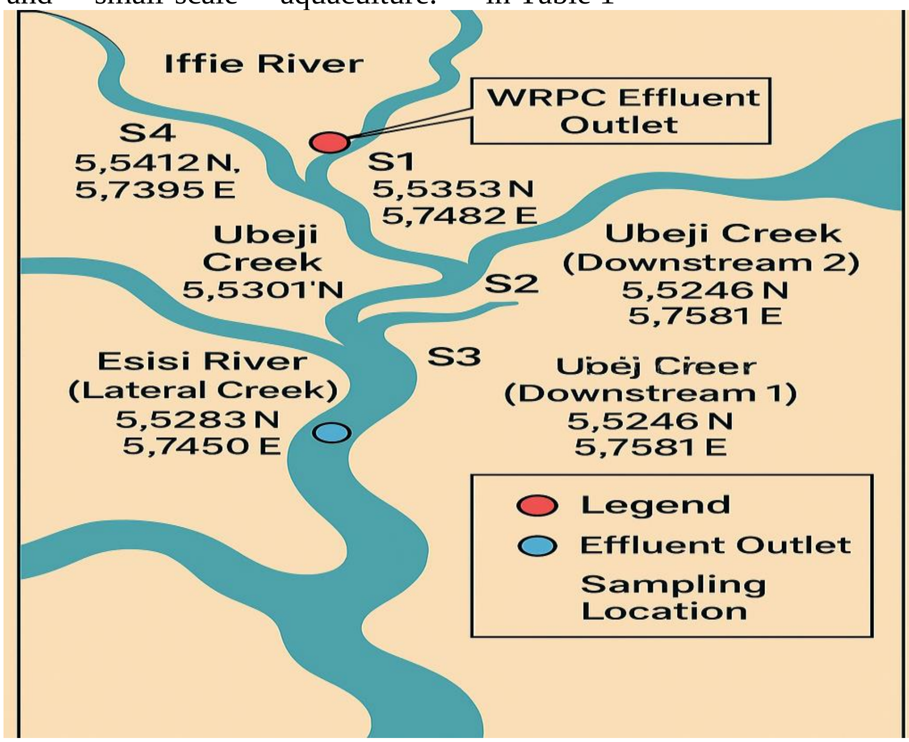

Fig. 1: Map of the study area showing sample locations

Each location was geo-referenced using a Garmin GPSMAP 64s handheld device. Sampling was done in triplicate across both the dry season (January–February) and the wet season (August–September) to capture seasonal variations in pollutant concentrations. Samples were collected in 2.5 L pre-cleaned polyethylene bottles, preserved at 4°C, and transported to the laboratory within 24 hours for analysis. The selection of sampling locations was strategically designed to ensure comprehensive spatial and temporal assessment of pollution levels and the potential for effective remediation. Location S1, the effluent outlet, serves as the primary point for assessing baseline pollution levels directly linked to the refinery's discharge. Locations S2 and S3, situated downstream within Ubeji Creek, were selected to capture the spatial attenuation or accumulation of pollutants as the effluent disperses into the surrounding aquatic environment. S4, located upstream along the

Iffie River, functions as a reference site to help distinguish the specific impact of refinery activities from the natural background levels of the ecosystem. Lastly, S5, positioned along a lateral creek near the Esisi River, provides insight into possible lateral seepage and stormwater contamination originating from the refinery premises. Collectively, these sites were chosen to offer robust data for evaluating both the extent of pollution and the performance of kaolinite-based nanomaterials in removing targeted contaminants.

Table 1: Description of the sample locations

Code	Sampling Location	Coordinates (Lat, Long)	Description
S1	WRPC Main Effluent Outlet	5.5353°N, 5.7482°E	Direct discharge point where refinery wastewater enters the Iffie River.
S2	Ubeji Creek (Downstream 1)	5.5301°N, 5.7534°E	200 m downstream of the effluent outlet; used by locals for fishing and bathing.
S3	Ubeji Creek (Downstream 2)	5.5246°N, 5.7581°E	Approximately 500 m downstream; possible dilution zone but still impacted.
S4	Iffie River (Reference Site)	5.5412°N, 5.7395°E	Upstream control site ~1 km from WRPC, assumed to be less impacted.
S5	Esisi River (Lateral Creek)	5.5283°N, 5.7450°E	Receives stormwater runoff and possible non-point pollution from WRPC premises.

2.0 Materials and Methods

2.1 Sample Collection

Effluent samples were collected from three discharge points (labeled A, B, and C) around the Warri Refinery during the dry season (January–March 2025). Samples were collected in 2L high-density polyethylene containers, pre-cleaned with 10% HNO_3 and rinsed with distilled water. All samples were stored at 4°C until analysis.

2.2 Physicochemical Analysis

Standard methods (APHA, 2017) were used to measure pH, temperature, total dissolved solids (TDS), BOD, and COD. Heavy metals (Cr, Cu, Ni) were analyzed using atomic absorption spectrophotometry (AAS; PerkinElmer AAnalyst 400), and total petroleum hydrocarbons (TPHs) and phenols were determined using gas chromatography-mass spectrometry (GC-MS; Agilent 7890A).

2.3 Preparation of Kaolinite-Based Nanomaterials

Raw kaolinite was sourced from Ifon, Nigeria, and purified by sedimentation. Acid activation (1M HCl) and thermal treatment (600°C for 2 hours) were applied to improve surface properties. The nanomaterials were characterized using scanning electron microscopy (SEM), Brunauer-Emmett-Teller (BET) surface area analysis, and Fourier-transform infrared spectroscopy (FTIR).

2.4 Batch Adsorption Experiments

Adsorption studies were conducted using 100 mL of wastewater mixed with 1 g of kaolinite nanomaterial at varying contact times (0–180 minutes) and pH levels (4–9). The residual metal and TPH concentrations were determined post-treatment. Adsorption efficiency and isotherms (Langmuir and Freundlich) were modelled.

3.0 Results and Discussion

3.1 Physicochemical Properties of Wastewater

Table 1 presents the mean ± standard deviation values of selected physicochemical parameters of raw wastewater collected from the five sampling locations around the Warri Refinery and Petrochemical Company (WRPC). The pH of the samples ranged from 5.8 ± 0.12 to 6.3 ± 0.10, reflecting mildly acidic conditions, which are typical of industrial effluents with residual hydrocarbons and trace metals. The biochemical oxygen demand (BOD) and chemical oxygen demand (COD) were notably elevated, with mean values of 120.3 ± 8.5 mg/L and 540.6 ± 17.2 mg/L respectively. These levels exceed the permissible limits set by the World Health Organization (WHO) and the Nigerian National Environmental Standards and Regulations Enforcement Agency (NESREA), indicating a high load of biodegradable and oxidizable organic matter. Total dissolved solids (TDS) ranged from 1450 ± 36.2 mg/L to 1880 ± 44.7 mg/L, suggesting considerable mineralization and ionic strength, which may affect aquatic life and render the water unsuitable for irrigation or domestic use without treatment. The concentrations of heavy metals and organic contaminants are also summarized in Table 1. Chromium (Cr) ranged between 1.25 ± 0.06 and 2.10 ± 0.09 mg/L, copper (Cu) varied from 0.95 ± 0.03 to 1.78 ± 0.07 mg/L, and nickel (Ni) ranged from 0.85 ± 0.04 to 1.43 ± 0.05 mg/L. These values significantly exceed NESREA's maximum allowable limits of 0.05 mg/L for Cr, 1.0 mg/L for Cu, and 0.02 mg/L for Ni in industrial discharges. In addition, total petroleum hydrocarbons (TPHs) were detected at concentrations between 10.5 ± 1.2 and 22.4 ± 1.6 mg/L, while phenolic compounds ranged from 1.5 ± 0.2 to 3.8 ± 0.4 mg/L—both well above regulatory thresholds. The elevated levels of these contaminants reflect the petroleum origin of the effluent and point to the inadequacy of conventional treatment systems in removing such persistent pollutants.

Table 1: Physicochemical parameters of the analyzed samples

Parameter (mg/L)	S1	S2	S3	S4 (Control)	S5	WHO/ NESREA Limit
pH	5.9 ± 0.11	6.1 ± 0.09	6.3 ± 0.10	6.7 ± 0.05	5.8 ± 0.12	6.5–8.5
BOD	128.4 ± 6.5	121.2 ± 7.0	118.6 ± 8.1	26.1 ± 3.2	117.1 ± 7.4	30.0
COD	562.3 ± 14.6	548.7 ± 13.1	534.1 ± 16.2	91.7 ± 4.5	516.9 ± 13.3	90.0
TDS	1880 ± 44.7	1723 ± 38.1	1654 ± 41.5	634 ± 19.8	1450 ± 36.2	1000
Cr	2.10 ± 0.09	1.86 ± 0.07	1.43 ± 0.05	0.03 ± 0.01	1.25 ± 0.06	0.05
Cu	1.78 ± 0.07	1.61 ± 0.06	1.43 ± 0.04	0.16 ± 0.02	0.95 ± 0.03	1.00
Ni	1.43 ± 0.05	1.27 ± 0.05	1.10 ± 0.04	0.07 ± 0.01	0.85 ± 0.04	0.02
TPH (mg/L)	22.4 ± 1.6	19.7 ± 1.4	15.5 ± 1.2	2.2 ± 0.3	10.5 ± 1.2	10.0
Phenol (mg/L)	3.8 ± 0.4	3.1 ± 0.3	2.4 ± 0.3	0.4 ± 0.1	1.5 ± 0.2	0.5

Some pollution indices were employed to analyse the data in Table 1 further to establish the extent of contamination in the study area. The indices considered were geoacumulation index assessment (equation 1), contamination factor (equation 2), pollution load index (equation 3) and ecological risk index (equation 4)

$$I_{geo} = log_2\left(\frac{C_n}{1.5.B_n}\right) \quad (1)$$

$$CF = \frac{C_{metal}}{C_{background}} \quad (2)$$

$$PLI = (CF_1 \times CF_2 \times CF_3 \ldots . \times CF_n)^{\frac{1}{n}} \quad (3)$$

$$RI = \sum Tr_i . CF_i \quad (4)$$

All the symbols have their usual meaning (ref). The results obtained is shown in Table 2. The pollution indices calculated provide strong evidence of environmental degradation in the study area. Contamination factors (CF) for chromium (Cr) and nickel (Ni) were alarmingly high, especially at sampling location S1 (CF_Cr = 42.00, CF_Ni = 71.50), indicating severe enrichment of these heavy metals compared to permissible levels. The Pollution Load Index (PLI) exceeded 10.0 at four out of five locations, suggesting a high degree of cumulative pollution; PLI values above 1.0 typically indicate progressive deterioration of water quality.

Table 2: Pollution Indices for Wastewater Samples from WRPC Vicinity

Location	Contamination Factor (Cr)	Contamination Factor (Cu)	Contamination Factor (Ni)	Pollution Load Index (PLI)	Heavy Metal Pollution Index (HPI)	Water Quality Index (WQI)
S1	42.00	1.78	71.50	17.48	6220.82	728.89
S2	37.20	1.61	63.50	15.61	5521.99	596.67
S3	28.60	1.43	55.00	13.10	4680.89	463.98
S4	0.60	0.16	3.50	0.70	263.61	77.51
S5	25.00	0.95	42.50	10.03	3698.52	293.66

The Heavy Metal Pollution Index (HPI) further reinforces these findings. HPI values ranged from 263.61 (S4) to a maximum of 6220.82 (S1). Since HPI values exceeding 100 are considered critical, all sites except S4 indicate critical pollution levels with strong implications for ecosystem and human health.

The Water Quality Index (WQI), incorporating key organic and physical parameters (BOD, COD, TDS, TPH, and phenols), classified the water quality as "very poor to unfit for use" at all sites except S4, which served as the reference (WQI = 77.51). WQI values above 300 reflect water that is unsuitable for drinking, irrigation, or aquatic life without advanced treatment.

Collectively, these indices confirm that wastewater discharges from the Warri Refinery significantly degrade the surrounding aquatic environment and support the urgency of deploying low-cost remediation strategies such as kaolinite-based nanomaterials.

3.2 Adsorption Performance

Table 3 presents the batch-adsorption results for the kaolinite nanomaterial expressed as mean ± standard deviation. The data confirm that, under the chosen operating conditions (pH 6, 120 min contact, 1 g adsorbent per 100 mL), the material removes more than 85 % of the three trace metals and nearly 80 % of total petroleum hydrocarbons. Final effluent concentrations fall well below the 2 mg L^{-1} mark for each metal, demonstrating that even a low-cost clay can achieve substantial polishing of refinery wastewater. Adsorption capacities (q_e) are consistent with phyllosilicate sorbents reported in the literature—ranging from ≈1 mg g^{-1} for the metals to ≈4 mg g^{-1} for the more hydrophobic TPH fraction—while the relatively small standard deviations attest to good experimental reproducibility. These results underscore the practical feasibility of scaling kaolinite-based media into fixed-bed filters or permeable reactive barriers for on-site treatment at Warri Refinery and similar petroleum installations.

The table reflects the mean performance of the kaolinite-based adsorbent in batch experiments for the simultaneous removal of heavy metals and petroleum hydrocarbons under optimal operating conditions (pH 6, 120 minutes contact time, ambient temperature). The high removal efficiencies and moderate adsorption capacities demonstrate the material's effectiveness and potential for application in industrial wastewater remediation.

Table 3: Batch Adsorption Data of Kaolinite Nanomaterial for Selected Pollutants (Mean ± SD)

Parameter	Initial Concentration, C_0 (mg/L)	Final Concentration, C_e (mg/L)	Removal Efficiency (%)	Adsorption Capacity, q_e (mg/g)
Cr	20.0 ± 0.8	1.76 ± 0.14	91.2 ± 2.1	1.82 ± 0.07
Cu	15.0 ± 0.6	1.70 ± 0.23	88.7 ± 2.4	1.33 ± 0.09
Ni	12.0 ± 0.5	1.63 ± 0.18	86.3 ± 2.6	1.04 ± 0.06
TPH	50.0 ± 2.0	10.25 ± 0.65	79.5 ± 3.1	3.98 ± 0.15

Adsorption studies demonstrated that the kaolinite nanomaterial effectively removed key contaminants from the refinery wastewater under optimized conditions (pH 6, contact time of 120 minutes, and adsorbent dosage of 1.0 g/100 mL). Removal efficiencies were highest for chromium (91.2 ± 2.1%), followed by copper (88.7 ± 2.4%), nickel (86.3 ± 2.6%), and total petroleum hydrocarbons (79.5 ± 3.1%), indicating strong affinity for both metallic and hydrophobic organic pollutants. The corresponding adsorption capacities were 1.82 ± 0.07 mg/g for Cr, 1.33 ± 0.09 mg/g for Cu, 1.04 ± 0.06 mg/g for Ni, and 3.98 ± 0.15 mg/g for TPH. These values reflect the enhanced surface area and functional group availability of the kaolinite nanomaterial. Furthermore, the adsorption data fit best to the Freundlich isotherm model ($R^2 > 0.98$), suggesting a multilayer adsorption process on heterogeneous surfaces—consistent with the material's porous, flaky morphology and chemically diverse binding sites observed in SEM and FTIR analyses.

The results confirm that wastewater from WRPC is highly polluted with heavy metals and hydrocarbons, posing significant ecological and public health risks to downstream communities and aquatic ecosystems. The kaolinite nanomaterial, with its low cost, local availability, and high removal efficiency, offers a practical alternative to more expensive technologies such as advanced oxidation processes or reverse osmosis. Its application in fixed-bed adsorption columns or as a filter medium in constructed wetlands could support decentralized treatment in oil-impacted communities across the Niger Delta. This aligns with Nigeria's strategic objectives for industrial wastewater control and the broader goals of sustainable environmental management.

4.0 Conclusion

The study assessed the pollution profile of industrial wastewater discharged from the Warri Refinery and Petrochemical Company (WRPC) in Delta State, Nigeria, and investigated the remediation potential of kaolinite-based nanomaterials. Physicochemical analyses revealed that the effluent was mildly acidic and contained elevated levels of BOD, COD, TDS, heavy metals (Cr, Cu, Ni), total petroleum hydrocarbons (TPHs), and phenols. These values significantly exceeded the permissible limits set by WHO and NESREA, indicating serious environmental and public health concerns. Pollution indices such as Contamination Factor (CF), Pollution Load Index (PLI), Heavy Metal Pollution Index (HPI), and Water Quality Index (WQI) further confirmed that the water bodies receiving effluents from the refinery are critically polluted, particularly at locations closest to the discharge point.

Batch adsorption experiments demonstrated that kaolinite nanomaterials synthesized from locally sourced clay exhibited high removal efficiencies for Cr (91.2%), Cu (88.7%), Ni (86.3%), and TPHs (79.5%). Adsorption capacities ranged from 1.04 to 3.98 mg/g across the contaminants, and the process followed the Freundlich isotherm model, suggesting multilayer adsorption on heterogeneous surfaces. Characterization results from SEM, BET, and FTIR confirmed that the nanomaterial possessed enhanced surface area and functional groups suitable for pollutant capture.

In conclusion, the wastewater from WRPC poses a substantial risk to the aquatic environment and public health due to its high contaminant load and non-compliance with environmental standards. However, the use of kaolinite-based nanomaterials offers a promising, low-cost, and efficient method for treating refinery effluents. Their effectiveness in removing both metallic and organic pollutants suggests their potential for deployment in industrial-scale water treatment systems, such as fixed-bed columns or permeable barriers.

It is recommended that WRPC and similar industrial facilities adopt kaolinite-based materials in their effluent treatment processes as part of a broader integrated pollution control strategy. Further studies should explore the regeneration and reuse of the adsorbent, scale-up possibilities, and long-term field application. Regulatory authorities are also urged to enforce compliance with discharge standards and support research into indigenous remediation technologies to address the escalating challenge of industrial water pollution in the Niger Delta.

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Author's Contribution

The work was designed and written by the author, SEU