Evaluation of Diesel Range Organics of Some Petroleum Polluted Remediated Soils within Rivers, South-East Region, Nigeria.

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ABSTRACT

This study evaluated the levels of Diesel range organics (DRO) in soil samples of some petroleum polluted remediated sites within the Rivers State South-East geographical region of Nigeria. The research provides insight into the level of contamination and the effectiveness of remediation efforts in the region. Forty soil samples were collected from eight remediated sites, and were analyzed using Agilent GC equipped with a Flame Ionization Detector. The mean DRO values obtained ranged from 36.10 to 84.75 mg/kg. The results of their mean values were compared to their control values which ranged between 10.96 and 19.32 mg/kg. The DPR, NOSDRA, and EGASPIN standard limits were used to compare the obtained results. The results showed varying levels of DRO in the soil samples, with some sites showing high levels of contamination and others showing successful remediation efforts in the region. It also buttressed

Keywords: (Diesel range organics, Petroleum pollution, Soil Remediation)

the importance of effective regulation of the petroleum industry in Nigeria.

INTRODUCTION

The Rivers South-East geographical region is a vital part of Nigeria economy and an important contributor to global energy markets due to its significant reserves of oil and gas with an estimated 23 billion barrels of oil and over 183 trillion cubic feet of natural gas reserves (Giwa *et*

al., 2016). However, the exploration and production activities have led to widespread environmental pollution, particularly in the form of hydrocarbon contamination of soil. Hydrocarbon contamination occurs when petroleum products are spilled or released into the environment, leading to the contamination of soil

and water resources (Adewuyi *et al.*, 2012). The region has experienced multiple incidents of hydrocarbon contamination, including oil spills and leaks from pipelines. These incidents have had severe consequences for the environment and the people who live in the area, leading to health problems, loss of biodiversity, and damage to the economy (Festus *et al.*, 2020).

To address this issue, a number of remediation efforts have been undertaken in the region to clean up contaminated sites and mitigate the environmental impact of hydrocarbon pollution. Remediation is the process of removing or reducing contaminants in the environment to acceptable levels (Okparanma et al., 2018; Stroo and Ward, 2010; Reddy and Camselle, 2009; John, 2007; USEPA, 1987). The objective of remediation efforts is to reduce the levels of hydrocarbon contamination in soil and water resources to acceptable limits set by regulatory bodies, in order to protect human health, restore ecological balance, and support sustainable development. economic However, the effectiveness of remediation efforts in the Rivers State, South-East geographical region has been the subject of debate. While some studies have

reported significant reductions in hydrocarbon contamination levels following remediation efforts, others have suggested that the efforts successful have not been in reducing contamination levels to acceptable levels (Gbarakoro and Bello, 2022; Okoro et al., 2011). There are several factors that can impact the success of remediation efforts, including the type and extent of contamination, the methods used for remediation, and the environmental conditions at the site (Mmom and Deekor, 2010).

Total Petroleum Hydrocarbon (TPH) is the gross amount of petroleum hydrocarbon compounds that are present in an environment. It has a detrimental effect on human health and plant growth when present in the soil (Festus et al., 2020; Paiga et al., 2012). Diesel range organics is a complex mixture of hydrocarbons ranging from C_{10} – C_{28} or C_{32} derived from crude oil (API, 1994). It is often referred to the group of hydrocarbons in the diesel fuel range, which includes alkanes. alkenes. and aromatic hydrocarbons with carbon chain lengths ranging from C₉ to C₂₈ (API, 1994). The carbon ranges for diesel range compounds using

chromatography with flame ionization detector (GC/FID) typically includes hydrocarbons with carbon numbers between C₉ and C₂₈. However, the exact carbon range can vary depending on the specific method and regulatory agency classification. The US Environmental Protection Agency (USEPA, 2000) specifies a carbon range of C₉ to C₃₆ for diesel range hydrocarbons analysis using GC/FID, while European Committee for Standardization specifies a carbon range of C₉ to C₄₀ (CEN, 2002). It is important to consult the specific method and regulatory agency for the appropriate carbon range related to DRO analysis in soil. The most hazardous combinations of organic chemicals that can be discovered in or generated from crude oil can be classified as Diesel range organics and they are very volatile in nature. Many of these products (gasoline, kerosene, and much more) have greasy odour (Gbarakoro and Bello, 2022; Okparanma et al., 2018). The DRO could enter the environment through a variety of sources, including crude oil spills, leaks from storage tanks, transportation accidents and human sabotage etc. When toxic organic matter such as DRO is released into the environment, it moves from the soil to the

groundwater. Some organisms may break down some of the contaminants into smaller fractions, while some may evaporate into the atmosphere (Okoro et al., 2015; ASTDR, 1999). DRO released into the environment, according to the United Nation Environmental Programme, public health endanger and safety contaminating drinking water, increasing the risk of fire and explosion, lowering the quality of the air and water, impairing agriculture, destroying recreational areas, destroying habitats, destroying food, and wasting non-renewable resources (UNEP, 2011).

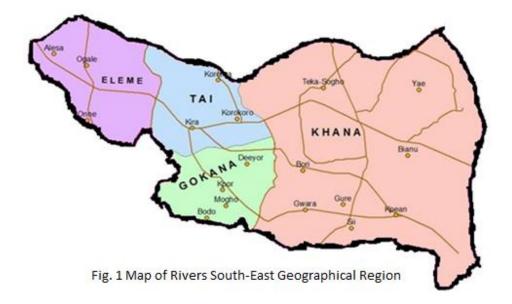
In recent years, there has been growing concern about the impact of hydrocarbon contamination on the health and livelihoods of people living in the Rivers South-East geographical region. The contamination has led to the loss of crops and fish stocks, which has had a significant impact on the economy of the region. It has also been linked to health problems, including respiratory illnesses, skin diseases, and cancer (Adewuyi *et al.*, 2012). This has led to calls for more effective remediation efforts in the region, in order to protect the health and livelihoods of local

communities and support sustainable economic development. The DRO compounds particularly harmful to plant and animal life and could contaminate the groundwater, air, and soil. The United Nations Environmental Programme (UNEP) conducted an assessment report on some communities affected by hydrocarbon pollution Rivers South-East geographical region, Nigeria between 2009 and 2011 (UNEP, 2011). Recommendations from the report led to the establishment of the Hydrocarbon Pollution Remediation Project (HYPREP) by the Federal Government of Nigeria (FGN). The body established aims to oversee the environmental remediation process and the restoration of livelihoods in the Niger Delta region of Nigeria, starting with Rivers South-East geographical region.

This research aims to fill a significant gap in the current literature by evaluating the concentrations

STUDY AREA

of DRO in petroleum polluted remediated sites in the Rivers South-East geographical region. While previous studies discussed TPH in general, none have specifically address Diesel range fraction of TPH in the region, making this study relatively. Furthermore, the study provided insight into the effectiveness of remediation approaches in reducing contamination levels to acceptable limits set by regulatory bodies in Nigeria. This is crucial for ensuring the safety of both the environment and human health. By focusing on several remediated sites in the region that have experience hydrocarbon contamination due to oil spills or pipeline leakages, this research had provided valuable data that would be used to improve future remediation methods. The results of this research have the potential to significantly impact current practices in the field of environmental remediation in Nigeria and beyond.



Along the Rivers State coast in the Niger Delta region of Nigeria, the Rivers South-East geographical region area is located between Latitude 4° 40'5" N and 4° 43' 19.5" N and Longitude 7° 22' 53.7" E and 7° 27' 9.8" E of the equator (Nkpaa et al., 2017). It is located in the Gulf of Guinea's Niger Delta basin, covering an area of 1,000 square kilometres (UNEP, 2011). There are four Local Government Areas (LGA) in Rivers South-East geographical region, namely Eleme, Gokana, Khana, and Tai (see Fig.1). It is a tropical rainforest belt with light shrubs and farmlands that are home to a variety of species as secondary vegetation, most of the land is covered in dense bush and bogs (Nkpaa et al., 2017). The East-West highway also passes through the

neighbourhood, which results in major vehicular traffic congestion. The region also produces oil and is home to numerous significant domestic and international oil and gas industrial facilities, refineries, petrochemical facilities, and fertilizer factories among others (Giwa et al., 2016). Local livelihoods, ecology, fish breeding grounds, and public health have all been impacted by oil contamination in Rivers South East geographical region. In addition, the spills had wiped out almost 1,000 hectares of mangroves, a vital habitat for fish nidification and reproduction as well as a very valuable carbon sinks (UNEP, 2011). The study area consists of eight remediated sites located in four LGA impacted by crude oil spills over the years. Akpajo and

Okuluegu are located in Eleme LGA, Gbogozor and Vuruvuru are located in Gokana LGA, Wiiboora and Wiikaragu are located in Khana

MATERIALS AND METHODS

The effectiveness of the remediation efforts was evaluated by measuring the levels of DRO in soil samples collected from the remediated sites, as well as from nearby control sites where no hydrocarbon contamination has occurred and remediation has not been undertaken.

Sampling and Collection

The sampling methodology adhered to the standard guidelines outlined in (ASTM D1452 – 09; D4547-98 USEPA 2020; NESREA B 796, Part III: 27; Popek, 2003). The sample points were located using a Garmin etrex10 GPS device (model GS284), and 40 points were mapped out across various locations, with 5 points chosen from each community, consisting of 4 points from the impacted site and 1 point from the control site. The selected points were drilled to a depth of 5 meters using a manual drilling auger, in line with the excavation depth during the remediation process. Discrete samples were collected at each drilled point, and control

LGA while Korokoro and Kporghor are located in Tai LGA.

samples were obtained from a location 120 meters away from the remediated sites. Sample collection was performed immediately after removing the auger head from the hole and the samples were deposited in a 100 ml widemouthed amber-colored glass bottle, filled to the brim to avoid headspace and minimize volatilization. The amber-colored glass bottle was used to prevent photochemical reactions with the soil sample. The samples were transferred to an insulated cooler containing ice blocks at a temperature of 4°C to maintain sample integrity. Prior to sampling at each depth, the auger head was washed with distilled water and rinsed with methanol before collecting the sample. Each sample was labelled and identified by community name and point number as follows; AJO-1 to AJO-4 denote samples from Akpajo, with the corresponding control point labelled as AJO-C. OBU-1 to OBU-4 represent samples from Okuluegbu, accompanied by OBU-C as the control point. Similarly, GOR-1 to GOR-4 signify samples from Gbogozor, with GOR-C as

the control point. VRU-1 to VRU-4 denote samples from Vuruvuru, linked to VRU-C as the control point. WRA-1 to WRA-4 represent samples from Wiiboora, accompanied by WRA-C as the control point. WGU-1 to WGU-4 signify samples from Wiikaragu, with WGU-C as the control point. KRO-1 to KRO-4 represent

Sample Extraction

The samples were extracted according to the ASTM D5765 procedures, whereby 10 grams of each sample were weighed with Denver analytical balance (model TP-214) and transferred to a 250 ml conical flask. A 50:50 mixtures of Dichloromethane (DCM) and Acetone, with 99.8% 99.9% and assay respectively, both procured from Sigma-Aldrich, was employed as the extracting solvent. The samples were then left to stand overnight, followed by agitation for 2 hours using a Jinotech HY-4 flask shaker (model H033AU) to ensure thorough mixing.

Subsequently, a No.41, 150 mm Whatman filter paper containing 5 grams of anhydrous Na₂SO₄ (99%) procured from Loba Chemie was utilized to remove moisture content from the extracts. A clean-up process involving the addition of 5 ml of

samples from Korokoro, linked to KRO-C as the control point. Lastly, KOR-1 to KOR-4 signify samples from Kporghor, with KOR-C as the control point. After the sampling process, a total of 40 soil samples were collected and promptly shipped to the laboratory for analysis.

the extracting solvent to the filter papers was also performed. The extracts were concentrated to 1 ml in a fume cupboard and then transferred to a 2 ml vial, which was sealed to preserve the sample integrity and avoid volatizing. This rendered the extracts ready for instrument analysis.

Sample Analysis and Instrument Condition

The analysis of samples for Total Diesel range hydrocarbons quantification was carried out using an Agilent GC 6890N (model G150N) equipped with a Flame Ionization Detector. The method used was in accordance with (USEPA Method 8015C; API, 1994; Pikoyskii, 2017). The GC/FID was equipped with an HP-519091J-413 capillary column, with a length of 30 m, a diameter of 0.32 mm, and a film thickness of 0.25 μ m. The injection mode was set to split less, with an injection volume of 1.0 μ l. Nitrogen gas was

used as the carrier gas at a flow rate of 1.2 ml/min while Hydrogen flame was used for the ionization. The injection temperature was maintained at 290°C, while the oven temperature was programmed to ramp up from 50°C to 300°C at a rate of 6°C per minute, and then held at 300°C for 2 minutes. The flame ionization detector temperature was set at 300°C.

Following the analysis of the samples, the chromatogram results were generated, providing a comprehensive profile of the concentrations of a wide range of non-halogenated volatile organic compounds, semi-volatile organic compounds, and petroleum hydrocarbons. In summary, the Agilent GC 6890N with Flame Ionization Detector was found to be an efficient and reliable method for the quantification of Diesel range hydrocarbons.

Statistical analysis and data processing

Utilizing Microsoft Excel 2016, a comprehensive statistical analysis was executed to derive both mean concentration values and mean standard

deviations for each individual sample. The mean concentration values effectively encapsulate the hydrocarbon average DRO concentration prevailing distinct at each site under consideration. Concurrently, the mean standard deviation values serve to elucidate the magnitude of concentration variance from the established mean.

Noteworthy is sample AJO, which prominently exhibits the highest DRO concentration level, manifesting an average concentration value of 84.74 mg/kg (Table 1). Complimenting this concentration, the mean standard deviation of ±1.13 unveils a considerable degree of variance around the mean, thus underscoring substantive heterogeneity in the concentration spread. Conversely, sample GOR emerges with the lowest DRO concentration, denoting an average value of 36.19 mg/kg (Table 1). Notably, the accompanying mean standard deviation of ±0.50 signifies a relatively constrained concentration spectrum, indicative of a more tightly clustered distribution around the mean.

RESULTS AND DISCUSSION

Table 1. The DRO concentration, their permissible limit and intervention values

S/N	Sample ID	∑DRO Conc.mg/kg	Mean Values mg/kg	Regulatory Bodies DPR, NOSDRA, EGASPIN	
				50 mg/kg	5000 mg/kg
1	AJO-1	78.15			
2	AJO-2	84.62	AJO		
3	AJO-3	93.78	84.75 ±1.13		
4	AJO-4	82.45			
5	AJO-C	10.96			
6	OBU-1	33.40			
7	OBU-2	38.11	OBU		
8	OBU-3	48.78	39.23 ±1.19		
9	OBU-4	36.62			
10	OBU-C	14.10			
11	GOR-1	39.04			
12	GOR-2	35.71	GOR		
13	GOR-3	32.51	36.10 ±0.50		
14	GOR-4	37.15			
15	GOR-C	17.07			
16	VRU-1	76.17			
17	VRU-2	85.49	VRU		
18	VRU-3	83.47	83.01 ±0.85		
19	VRU-4	86.91			
20	VRU-C	19.32			
21	WRA-1	40.71	WRA		
22	WRA-2	38.53	38.86 ±0.36		
23	WRA-3	36.32			
24	WRA-4	39.88			
25	WRA-C	15.42			
26	WGU-1	83.49			
27	WGU-2	79.88	WGU		
28	WGU-3	82.99	83.10 ±0.42		
29	WGU-4	86.06			
30	WGU-C	12.60			
31	KRO-1	82.85			
32	KRO-2	78.01	KRO		
33	KRO-3	87.43	84.62 ±1.05		
34	KRO-4	90.22			
35	KRO-C	14.47			
36	KOR-1	37.88			
37	KOR-2	40.87	KOR		
38	KOR-3	38.21	40.18 ±0.53		
39	KOR-4	43.75			
40	KOR-C	13.47	2011; EGASPIN, 2022]		

[DPR, 2018; NOSDRA, 2011; EGASPIN, 2022]

The results of this research provide important insights into the level of DRO concentration in soil samples from various sites within the Rivers South-East geographical region. Table 1 showed the various limits for DRO as well as values in which it is presume in excess and requires remediation actions. In Nigeria, these values are set by regulatory bodies. The regulatory bodies include: Department of Petroleum Resources (DPR), National Oil Spill Detection and Response Agency (NOSDRA), Environmental Guidelines and Standards for the Petroleum Industry in Nigeria (EGASPIN). DRO limits can vary depending on the region where they are studied. This is because different regions may have different environmental conditions, cultural practices, and regulatory frame works which DRO can be managed and regulated.

The results revealed that the DRO mean concentrations in the soil samples for the remediated sites ranged from 36.10 to 84.75 mg/kg (Table 1). The highest concentration was observed at AJO with a value of 84.75 mg/kg while the lowest concentration was observed at GOR with a value of 36.10 mg/kg. The second highest concentration was observed at KRO with a value of 84.63 mg/kg while the second lowest concentration value was observed at WRA with a value of 38.86 mg/kg. The third highest concentration was observed at WGU with a value of 83.10 mg/kg while third lowest concentration was observed at OBU with a value of 39.23 mg/kg. The results for the control sites ranged from 10.96 to 19.32 mg/kg.

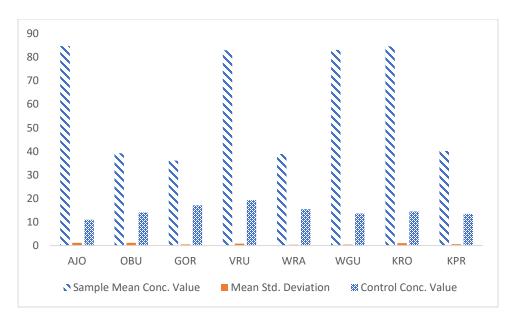


Fig. 2 Chart showing DRO mean values from the various sites

The results obtained in the remediated sites were compared to the concentrations obtained from their control values. The results showed that samples from AJO, OBU, GRO, VRU, WRA, WGU, KRO, and KOR where above their control values (see Figure 2). Also, comparing the results to the permissible limit set by regulatory bodies in Nigeria, it was observed that sample OBU, GOR, WRA, and KOR were below the permissible limit. The low concentration of DRO observed suggests that these sites may have undergone successful remediation efforts. The success of remediation efforts in these sites may be due to a range of factors, including the effectiveness of the remediation technique used,

the time taken to implement the remediation effort, and the level of support from stakeholders. However, it was observed that at AJO, VRU, WGU, and KRO the total DRO values were above the permissible limit but below the intervention value of 5,000 mg/kg. The high concentration observed suggests that these sites may have been heavily contaminated with oil spill. Also it indicates that the remediation efforts carried out in this sites may not have been effective in completely removing the DRO in the sites, since the aim of the remediation activity is to bring down these hydrocarbons below the permissible limit. According to DPR, NOSDRA, and EGASPIN, the maximum allowable limit for

Total petroleum hydrocarbon (TPH) including DRO in soil is 50 mg/kg. The contamination of these sites may be as a result of some factors, including the proximity of the sites from oil production activities, transportation routes and storage facilities. The contamination may also be due to inadequate storage and disposal of petroleum products. The high concentration results indicated that the study area is still contaminated with petroleum hydrocarbons despite remediation efforts.

It is important to note that the DRO concentrations observed in this study are consistent with those reported in other studies on TPH contamination in Nigeria and other parts of the world. A study by Samuel and Omoleomeo (2016) found TPH concentrations ranging from 4.51 to 76.68 mg/kg in soil samples from oil production facilities in Niger Delta region, Nigeria. Another study by UNEP (2011) reported TPH concentrations ranging from 2.88 to 1,040.00 mg/kg in soil samples from oil spill sites in the same region. The similarity in TPH concentrations reported in these studies suggests

that TPH contamination remains a significant environmental problem in the Niger Delta region.

In addition, the DRO concentrations observed in this study are also consistent with those reported in other studies on TPH contamination in other regions of the world. A study by Paiga *et al.*, (2012) found TPH concentrations ranging from 1.00 to 9,230.00 mg/kg in soil samples from different locations in north Portugal. Another study by Lin *et al.*, (2010) reported TPH concentrations ranging from 380.00 to 7,380.00 mg/kg in soil samples from oil-contaminated sites in Taiwan. These studies suggest that TPH contamination is a global environmental problem that requires urgent attention.

Several studies have reported that the use of bacteria and fungi can be effective in the remediation of hydrocarbon-contaminated soils. According to a study by Ahmad et al., (2022), bacteria and fungi have the potential to degrade hydrocarbons through enzymatic reactions. The study found that indigenous bacteria and fungi effectively degrade hydrocarbons in contaminated soils, and the use bio augmentation can further enhance the

remediation process. In another study by Lai *et al.*, (2014), it was found that a combination of bio augmentation and phytoremediation can significantly reduce the TPH concentration in contaminated soils. The results of these studies buttressed the use of bioremediation techniques as an effective way for remediating hydrocarbon-contaminated soils.

The research findings suggest that continued investment in effective remediation strategies can help to mitigate the negative impacts of hydrocarbon pollution in the Rivers South-East geographical region. This could include strategies such as bioremediation and phytoremediation, which have shown to be effective in reducing TPH concentrations in contaminated sites.

In addition, this study highlights the importance of ongoing monitoring and evaluation of remediated sites to ensure that the effectiveness of the remediation strategies is maintained over time. This could involve regular sampling and analysis of soil samples to assess DRO concentrations, as well as other indicators of soil health and ecosystem function.

CONCLUSION

This research provides important insights into the level of Diesel range hydrocarbons in soil samples from remediated sites within the Rivers South-East geographical region. The results of the research suggest that some of the sites may have been heavily contaminated with oil spill. The research also found that some of the sites may have undergone successful remediation efforts. The findings of this research highlight the need for continued monitoring and remediation efforts in the region. The results could also be used to inform policy decisions related to the regulation of the petroleum industry particularly in Nigeria. The study also highlights the importance of effective storage and disposal of petroleum products to prevent environmental pollution.

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