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CONCENTRATION OF HEAVY METALS AND PETROLEUM HYDROCARBONS IN PREVIOUSLY REMEDIATED SITES IN NIGER DELTA, NIGERIA

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ABSTRACT

This study was aimed at assessing the soil conditions of previously remediated oil spill sites in the Niger Delta region. Ninety-six (96) soil samples were collected over two seasons from remediated sites from three states (Rivers, Abia, and Imo). Gas Chromatographic (GC) Analysis was used in analyzing for Total Petroleum Hydrocarbon (TPH) content while heavy metals analysis for Lead (Pb), Zinc (Zn), Copper (Cu), Chromium (Cr) and Iron (Fe) was done using absorptive spectrophotometric analyses. The data generated was subjected to ANOVA and multiple comparisons, Post-Hoc Tukey HSD test using IBM SPSS version 26.0. Results showed TPH range of 0.85±0.72mg/kg to 1.76±0.42mg/kg in samples from Owaza site in Abia state. Concentration of heavy metals in soil samples was below the standard permissible levels of the World Health Organization and other regulatory bodies such as Department of Petroleum Resources (DPR) intervention values, United States Environmental Protection Agency (USEPA), and Environmental Guidelines and Standards for Petroleum Industry in Nigeria (EGASPIN 2002), accepted values for soil samples. There were no effects of season variation observed. Enrichment factor of (323.20- 508.64) and (298.37-567.26) indicates anthropogenic activities as sources of pollution. The result of the current studies signifies that, the soils of previously remediated sites have safe levels of the contaminants; however, we recommended a regular routine check on the levels of contaminants.

Keywords: Gas Chromatography, Heavy Metals Contamination, Hydrocarbon Contamination, Physicochemical Analysis, Soil Pollution, Spectrophotometry, TPH Concentrations.

INTRODUCTION

In the quest for industrialization and more significant economic empowerment, several developing countries, Nigeria inclusive, have tampered with the environment to the extent that there is widespread environmental degradation and devastation with attendant climatic, economic and health effects (Imoobe & Iroro, 2009).

From the early days of petroleum operations in the Niger Delta region, which dates back to the 1950's, Crude oil extraction and processing activities have resulted in massive land contamination (Sam et al., 2016; Zabbey et al., 2017). In addition, there have been several cases of oil spills resulting from the failure of pipes and storage units due to corrosion and vandalization, equipment failure and human errors in the region (Clewell et al., 2004). Oil spillage is the discharge of a liquid petroleum hydrocarbon into the environment due to natural or anthropogenic activity (Akhionbare & Osuji, 2013), and land affected by oil spills has been identified as a significant challenge to livelihood, human and environmental health in the Niger Delta region of Nigeria (Sam et al., 2016; Zabbey et al., 2017).

Soil contamination is a result of harmful chemicals or other alterations in the natural soil environment. Hydrocarbon contaminated soils are soils that the presence of petroleum hydrocarbon has polluted. Many technologies exist to treat contaminated soils; however, biological treatment offers the best environmentally friendly method for remediating hydrocarbon contaminations. Bioremediation is the application of microorganisms or plants to remove organic and inorganic contaminants from the environment. The types of bioremediation techniques are bio stimulation, bioaugmentation and phytoremediation (Bôto et al., 2021).

Once oil is spilt, land and groundwater resources are contaminated, and the option for their use becomes both limited and costly. Petroleum hydrocarbon has been indicted for introducing organic pollutants and heavy metals such as Zn, Cu, Cr, Hg, Ni, Cd and Pb into soils, which toxically damages the ecosystem. Spills alter the soil's physical and chemical properties soil, such as temperature, structure, nutrient, status and pH (Alabi et al., 2019; Logeshwaran et al., 2018; Medallon & Garcia, 2021; Nisha et al., 2018). Petroleum hydrocarbons and heavy metals may find their way into many environments through agricultural crops, soil surface and groundwater, where they underwent a redistribution process and are detected at different levels of concentration in the food chain (Hart et al.,, 2005). The effects of hydrocarbon pollution and contamination may be immediate if crops planted in spill sites that have accumulated the metals get consumed by man and livestock (Ekundayo & Obuekwe, 2000).

Numerous studies have also indicated that metal pollution (e.g. Pb, Cd and Ni) is responsible for certain diseases of humans and animals, and there is a need for cleaning up oil-contaminated soil (Fatoba et al., 2016; Young, 2005). Thus, it is crucial assessing areas of contamination, remediating and monitoring clean-ups, and final quality evaluation of the remediated soil (Ahmad et al., 2017). Hence, the need for regular re-assessment of oil spill areas for impacts on the soil and vegetation concerning their hydrocarbon and heavy metal loads is necessary for adequate environmental restoration and human health concerns after remediation.

Gas chromatography (GC) and spectrophotometry methods are widely recognized and used as analytical techniques for evaluating organic and heavy metals contaminants. Tanee and Albert (2011) have worked on post-remediation assessments of crude oil polluted sites at Kegbara-Dere towns in Gokana Local Government Area, Rivers State of Nigeria. The study employed methods including gas chromatography and spectrophotometry to analyze soil parameters like conductivity, pH, phosphate, nitrate, total organic carbon (TOC), total hydrocarbon content (THC) and total exchangeable cations (TEC); the

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obtained and interpreted results helped to ascertain the degree of success of the remediated soils.

This study assesses the heavy metals and TPH concentrations of soils from previously remediated sites in parts of the Niger Delta, Nigeria. It aims to investigate ex-post soil conditions, determine concentrations of residual organic and heavy metals contaminants in the soil, assess the effect of seasonal variations on soil properties, and compare results obtained from the different sites and WHO internationally accepted soil limits. The results were compared to the Department of Petroleum Resources (DPR) intervention values, United States Environmental Protection Agency (USEPA), and Environmental Guidelines and Standards for Petroleum Industry in Nigeria (EGASPIN 2002) limits for soils in the Niger Delta to determine the effectiveness of remediation.

LOCATION OF STUDY AREA

Niger Delta area is a Nigerian delta situated in the Gulf of Guinea on the Atlantic Ocean. The Niger Delta is located within nine southern Nigerian costal states , 6 of which are from south southern Nigeria, including Rivers, Delta, Edo, Bayelsa, Akwa-Ibom, Cross River, one state (Ondo) from South-Western Nigeria, two (Abia and Imo) from South Eastern Nigeria. The region extends over about 112,000 Square Km and makes up about 12.0 per cent of Nigeria's land mass. It was previously a primary producer of palm oil; the region is densely populated, with about 31 million people in 3000 communities and 186 Local Government Areas making it Africa's most densely populated regions (Steiner, 2010).

This region is characterized by the magnitude of petroleum activities due to the petroleum-rich nature of the region. These developments have earned the primary region interest from international concerns regarding pollution levels, among others. We selected three significant locations in three states in the Niger Delta area for this study due to the history of oil spillages occurrences recorded in these locations, accessibility, and security.

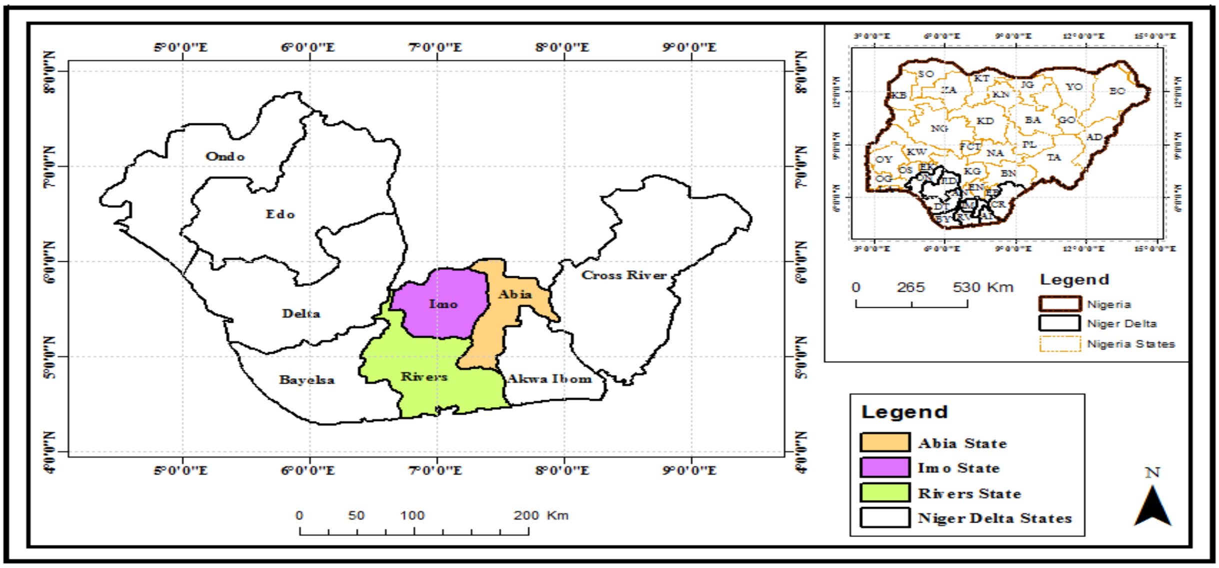

Figure 1: Map Showing Niger Delta States and Outlining the Study Location

Ukwa West local government area (L.G.A.) is the only oil producing local government in Abia State. Ukwa West is north bounded by Osisioma local government area, to the north-east by Ugwunagbo local government area and the east by Ukwa East local government area, all in Abia State. It is also west-bound by Omuma L.G.A. and by Etche L.G.A. to the south-west, and the south by Oyigbo L.G.A., all in Rivers State. The local government is the only crude oil producing area in Abia state.

Owaza is a community in Ukwa East, local government area of Abia State, Nigeria; it is located 4° 57' 47" North, 7° 11' 15" East. Owaza clan seats along the east bank of Imo River at a point not quite far from where it empties into Opobo Creek, emptying into the Atlantic Ocean. The clan consists of four autonomous communities: Isi Etitioha, Ipu West, Igiri-Ukwu and Etitioha, all oil-bearing communities. Reports have it that Owaza's OML 11 (Oil Mining Lease) is the largest oil producing oil mining lease on the land where Shell Petroleum Development Company SPDC operates. Owaza people are predominantly farmers, with subsistence agriculture being the primary practice, while small forms of commercial farming are also done (Okonkwo, 2011).

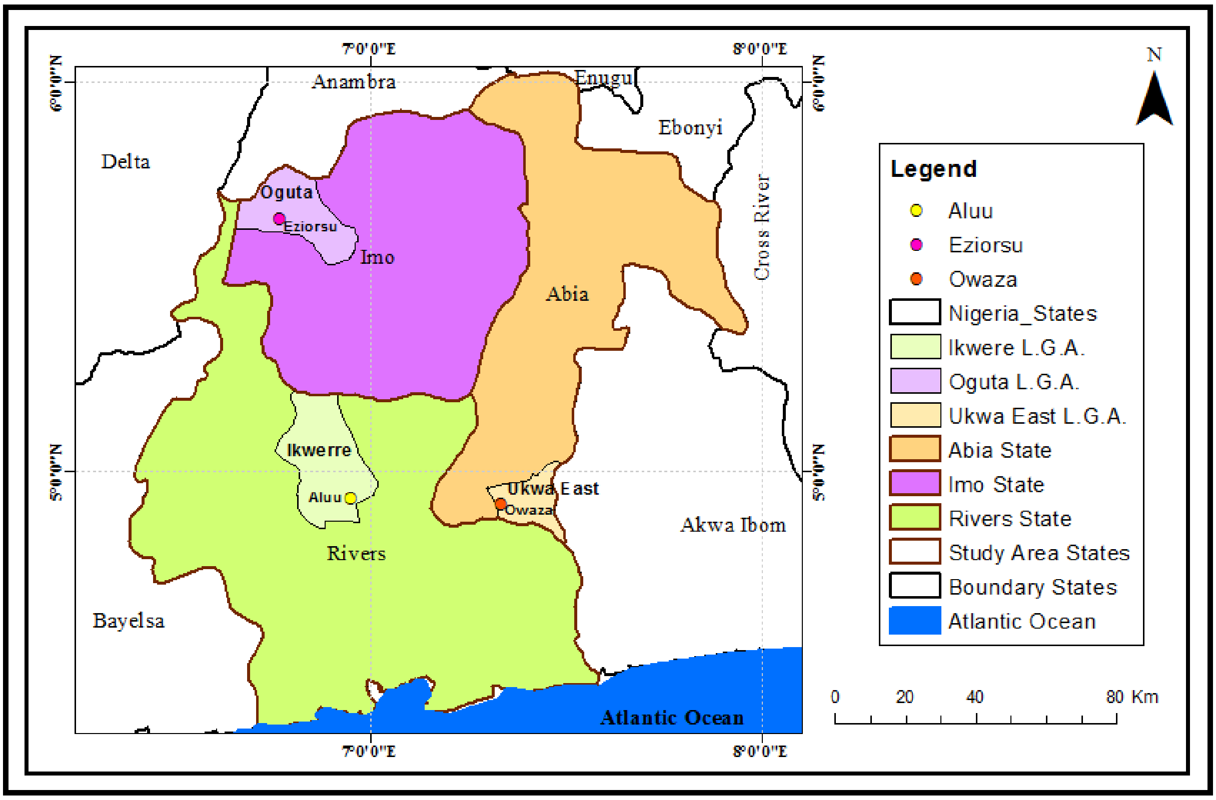

Figure 2: Map Showing Study Area and Sampling Points

Eziorsu is a South-bound community of Oguta Lake with a population estimated at 14,560 according to the 1991 census. It is one of the Oil and Gas producing communities in Imo State, the second largest producing community in Oguta L.G.A. In addition, the Eziorsu community is the major stable food producer in the state, with produce including yam, cassava, maize and palm oil (Okonkwo, 2011).

Ikwerre Local Government Area is located in Rivers State, Nigeria; its headquarters is located at Isiokpo. The local government

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area is blessed with many petroleum hydrocarbon deposits; it contains 92 oil wells producing an estimated 100,000 barrels daily. The local government plays host to several multinational oil-producing and servicing companies, in addition to many other industries and establishments. Aluu is a community in Ikwerre Local government area that hosts different oil and gas pipelines, including the Rumuekpe-Nkpoku flowline close to the Aluu community, Obio-Akpor Local Government Area in Rivers State.

MATERIALS AND METHODS

Sample Collection

A randomized sampling technique was used for sample collection after a global positioning system (GPS) was used to take the coordinates of the various sampling locations. A total of 96 soil samples (N=6) were collected over two seasons using a hang auger at a depth of 0 to 15 cm for topsoils and 15 to 30 cm for subsoils, respectively. Field reconnaissance and sampling were carried out in order to delimit the area to be sampled. Soil sampling was done twice (2 times), first during the dry season and then during the rainy (wet) season, with a 90 days sampling interval. This was done to help obtain the effects of seasonal variations on soil properties.

Control soil samples were taken from the Omuokiri community in River's state, an unaffected area by hydrocarbon contaminations for analytical correlations. The top surfaces of the soils at the previously remediated spill sites were cleared off to remove all stones, vegetation and organic debris. After sample collection, the samples were packaged in clean sampling bags, appropriately labeled, transferred into coolers and transported to the laboratory for analysis.

Sample Preparation for heavy metal analysis

The heavy metals concentration in the samples were determined using the atomic absorption spectrophotometry (Varian AA 240 AAS) (American Public Health Association (APHA 3030) and American water works association (Association, 1995). Soil samples were air-dried and grinded to reduce the moisture content. The heavy metals examined were Lead (Pb), Zinc (Zn), Copper (Cu), Chromium (Cr) and Iron (Fe).

Determination of TPH Concentration In samples

A petroleum hydrocarbon standard solution (20 mg/ml) was prepared for TPH quantification, and selected volumes of this solution were diluted with n-heptane to produce a series of working standards with TPH concentrations of 1.0, 2.0, 5.0, 10.0, and 15.0 mg/l. Between the retention durations of n-decane and n-tetracontane, the amount of total petroleum hydrocarbons (TPH) was calculated as a sum parameter of resolved and unresolved components eluted from the GC capillary column. With unweighted ordinary least squares regression, the calibration data were fitted to a straight line and the unknown sample TPH concentrations were derived from this standard line.

Data Analysis Plan

The data generated from spectrophotometer analysis and gas chromatography were presented as Mean ±SD, with statistical analysis by the SPSS version 26.0 (statistical software package for social sciences) for qualitative and inferential analysis. In addition, IBM, Post-Hoc Tukey HSD test was carried out to verify statistically significant differences among individual means at P≤0.05.

Contamination Factor (CF)

Contamination Factor (CF) is mainly used to assess the level of contamination by heavy metals in a sample. It is the ratio of the concentration of the heavy to the background value. The background value used in this work is the standard value stipulated by the Department of Petroleum Resources (DPR) (DPR, 2011), as shown in Table 1 below.

$$CF = \frac{Cmetal}{Cref} \text{ -------------------------1}$$

Where:C *metal* is the metal concentration in polluted media, and C-background is the background value of that metal.

Table 1: Pre-industrial reference level (mg/kg) and toxic response factor by Håkanson (1980)

Parameter	Cr (mg/kg)	Cu (mg/kg)	Pb (mg/kg)	Zn (mg/kg)	Fe (Mg/kg)
Bn	100.000	36.000	85.000	140.000	3800.000
Tr	2	5	5	1	

Enrichment factor (EF)

The enrichment factor (EF) was used to assess the enrichment degree of specific contaminants in different environmental media to facilitate comparison between the contamination of different environmental media (Benhaddya & Hadjel, 2014).

In calculating the enrichment factor of a media, a reference metal such as Aluminum (Al), Iron (Fe), total organic carbon, etc., are used for normalization (Ackerman, 1980). For this study, Fe was chosen as our reference metal.

The Enrichment factor was calculated using the relationship:

$$EF = \frac{Cn/Cref}{Bn/Bref} \text{ ------------------2}$$

Where: Cn – metal concentration in a sample, Cref – reference metal concentration in the sample, Bn – background value for metal and Bref – background value for reference metal.

Enrichment factor values greater than 1.5 suggest that the sources of pollution to the media under study are more likely to be anthropogenic.

RESULTS PRESENTATION

Sample Heavy Metals Concentration in Surface and Sub-surface Soils during the Wet Season

Chromium (Cr)

Cr at the surface level across the various stations ranged between 0.05-0.81mg/kg. Mean surface chromium across the stations was highest at Control with 0.35±0.35mg/kg and lowest at Owaza with 0.19±0.09 mg/kg as shown in Table 2. Analysis of variance (ANOVA) for surface chromium showed no significance difference at P<0.05 (P=0.377). The data generated for sub-surface mean value and standard deviation indicates that chromium values ranged from 0.09±0.03 mg/kg for Aluu to 0.51±0.34mg/kg as the highest mean and standard deviation value. Analysis of variance for sub-surface chromium concentration indicated a significant difference at P> 0.05.

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Table 2: Mean ± standard deviation and mean range of chromium (mg/kg) in wet season at the sampling points

	SURFACE					SUBSURFACE		
Site	Mean	±	Std. Dev	Min	Max	Mean ± Std. Dev	Min	Max
CONTROL	0.35	±	0.35	0.05	0.81	0.17 ± 0.06	0.08	0.22
EZIORSU	0.32	±	0.08	0.18	0.42	0.51 ± 0.34	0.18	1.03
OWAZA	0.19	±	0.09	0.09	0.33	0.24±0.05	0.2	0.31
ALUU	0.21	±	0.06	0.16	0.32	0.09 ± 0.03	0.06	0.15

Copper (Cu)

The result of Cu at the surface level across the various stations ranged between 1.04-5.03mg/l. Mean surface Copper across the stations was highest at Eziorsu with 3.85 ±0.72mg/l and lowest at Control with 1.59±0.13mg/l., as shown in Table 3. Sub-surface data showed the mean value and standard deviation for copper to range from 2.10 ±0.24 mg/kg from the control site to 4.13±0.10 mg/kg for Eziorsu. Analysis of variance for sub-surface copper concentration indicated a significance difference at P> 0.05

Table 3: Mean ± standard deviation and mean range of copper (mg/kg) in wet season at the sampling points

	SURFACE					SUB-SURFACE		
Site	Mean	±	Std. Dev	Min	Max	Mean ± Std. Dev	Min	Max
CONTROL	1.59	±	0.13	1.46	1.83	2.10 ± 0.24	1.65	2.28
EZIORSU	3.85	±	0.72	3.17	5.03	4.13 ± 0.10	4.01	4.31
OWAZA	2.14	±	0.65	1.04	3.01	2.74 ± 0.44	2.14	3.15
ALUU	2.32	±	0.55	1.76	3.04	2.44 ± 0.49	1.96	3.21

Iron (Fe)

The surface level result for Fe across the various stations ranged between 1.72-12.10mg/l. Mean Iron at surface level across the stations was highest at Eziorsu with 9.39±1.76mg/l and lowest at Control with 3.60±1.09mg/l., as shown in Table 4. Analysis of variance for surface Iron showed a significance difference at P<0.05 (P=0.001). Data from the analysis of variance for mean and standard deviation for Iron content in sub-surface soil from different sampling points and the control site indicated the control site with mean and standard deviation of 4.59 ±0.55 mg/kg recorded the lowest while the highest mean range of 8.57±0.95 mg/kg for Eziorsu as the site with the highest mean and standard deviation value.

Table 4: Mean ± standard deviation and mean range of Iron (mg/kg) in wet season at the sampling points

SURFACE						SUB-SURFACE		
Site	Mean	±	Std. Dev	Min	Max	Mean ± Std. Dev	Min	Max
CONTROL	3.60	±	1.09	1.72	4.65	4.59 ± 0.55	4	5.51
EZIORSU	9.39	±	1.76	7.21	12.10	8.57 ±0.95	7.71	10.24
OWAZA	6.87	±	1.02	6.11	8.54	5.94 ±0.86	4.52	7.01
ALUU	7.59	±	0.73	6.51	8.36	6.31 ±0.84	5.14	7.32

Lead (Pb)

The result for surface Pb across the various stations ranged between 0.32-0.81mg/l. The mean of surface Pb across the stations was highest at Eziorsu with 0.62±0.13mg/l and lowest at Owaza with 0.52±0.12mg/l., as shown in Table 5. No significance difference was recorded at $P<0.05$ ($P=0.414$). Analysis of variance for a mean and standard deviation for Lead (Pb) content in sub-surface soil from different sampling points and the control site indicated that Owaza with mean and standard deviation of 0.42 ±0.30 mg/kg recorded the lowest while the highest mean range of 0.65±0.15 mg/kg for Eziorsu as the site with the highest mean and standard deviation value.

Table 5: Mean ± standard deviation and mean range of Lead (mg/kg) in wet season at the sampling points

SURFACE						SUB-SURFACE		
Site	Mean	±	Std. Dev	Min	Max	Mean ± Std. Dev	Min	Max
CONTROL	0.60	±	0.08	0.49	0.71	0.49 ± 0.09	0.39	0.63
EZIORSU	0.62	±	0.13	0.42	0.73	0.65 ± 0.15	0.44	0.83
OWAZA	0.52	±	0.12	0.32	0.64	0.42 ±0.30	0.07	0.82
ALUU	0.60	±	0.13	0.42	0.81	0.55±0.06	0.5	0.66

Zinc (Zn)

The result for surface Zn across the various stations ranged between Control (2.15±0.0.29mg/kg) and Eziorsu with 6.18±0.86mg/l, as shown in Table 6. Data from the analysis of variance for a mean and standard deviation for zinc content in sub-surface soil from different sampling points and the control site indicated that the control site with mean and standard deviation of 2.03 ±0.23 mg/kg recorded the lowest while the highest mean range of 5.35±0.39 mg/kg for Eziorsu.

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Table 6: Mean ± standard deviation and mean range of Zinc (mg/kg) in wet season at the sampling points

	SURFACE					SUBSURFACE		
Site	Mean	±	Std. Dev	Min	Max	Mean ± Std. Dev	Min	Max
CONTROL	2.15	±	0.29	1.76	2.61	2.03 ± 0.23	1.7	2.21
EZIORSU	6.18	±	0.86	5.21	7.52	5.35 ± 0.39	5.11	6.04
OWAZA	4.20	±	0.55	3.56	5.10	3.10 ± 0.87	1.97	4.11
ALUU	4.22	±	0.52	3.63	5.17	3.20 ± 0.64	2.61	4.13

Mean Concentration of Heavy Metals in Soil of the Study Area in Dry Season

Chromium (Cr)

Chromium mean concentration and standard deviation in the surface soil during dry season ranged between 0.29 ± 0.07 and 0.39 ± 0.14 mg/kg for the Control and Aluu sites, respectively. On the other hand, the mean concentration and standard deviation of Cr in the subsurface soil during dry season ranged between Aluu 0.22 ± 0.08 mg/kg and Eziorsu, 1.26 ± 0.91 (Table 7).

Table 7: The mean Concentration, standard deviation of CHROMIUM in soils of the study area

	Surface			Sub-surface		
Site	Mean ± Std. Dev. (Mg/kg)	Min	Max	Mean ± Std. Devi. (Mg/kg)	Min	Max
Control	0.29 ±0.07	0.20	0.35	0.46 ± 0.10	0.32	0.56
Eziorsu	0.63 ±0.23	0.38	0.90	1.26 ±0.91	0.43	2.63
Owaza	0.35 ±0.23	0.12	0.73	0.56 ±0.12	0.45	0.78
Aluu	0.39 ±0.14	0.23	0.63	0.22 ±0.08	0.13	0.34

Copper

Cu's mean concentration and standard deviation in the surface soil during dry season ranged between 2.21 ± 0.11, to 5.41 ± 0.93, mg/kg for the Control and Eziorsu (Table 4.16). On the other hand, the mean concentration and standard deviation of Cu in the subsurface soil during the dry season was between 2.72 ± 0.16, 5.92 ± 0.56, mg/kg for the Control and Eziorsu (Table 8).

Table 8: The mean concentration, the standard deviation of COPPER in soils of the study area

	Surface			Sub-surface		
Site	Mean ± Std. Dev. (Mg/kg)	Minimum	Maximum	Mean ± Std. Devi. (Mg/kg)	Minimum	Maximum
Control	2.21 ± 0.11	2.00	2.32	2.72 ± 0.16	2.45	2.91
Eziorsu	5.41 ± 0.93	4.68	7.01	5.92 ± 0.56	5.03	6.72
Owaza	2.73 ± 0.62	1.79	3.65	3.99 ± 1.13	2.50	5.00
Aluu	3.03 ± 0.48	2.52	3.63	3.31 ± 0.59	2.52	4.11

Iron

Fe's mean concentration and standard deviation in the surface soil during dry season ranged between 5.49 ± 0.60, 11.51 ± 2.68 for control, and Eziorsu (Table 4.17). On the other hand, Fe's mean concentration and standard deviation in the subsurface soil during dry season ranged between 6.99 ± 0.54, 11.02 ± 1.52, control, and Eziorsu (Table 9).

Table 9: The mean concentration, the standard deviation of IRON (Fe) in soils in the dry season of the study area

	Surface			Sub-surface		
Site	Mean ± Std. Dev. (Mg/kg)	Min	Max	Mean ± Std. Dev. (Mg/kg)	Min	Max
Control	5.49 ±0.60	4.9	6.1	6.99 ±0.54	6.42	7.63
Eziorsu	11.51 ± 2.68	7.77	15.07	11.02 ±1.52	9.33	13.03
Owaza	9.65 ±0.90	8.41	10.89	8.15 ±1.18	6.88	10.32
Aluu	10.21 ±1.09	8.22	11.2	7.98 ±1.68	5.66	10.11

Lead

During the dry season, the mean concentration and standard deviation of Pb in the surface soil ranged between 0.54 ± 0.12 Owaza to 0.72 ± 0.19 Eziorsu (Table 4.18). On the other hand, the mean concentration and standard deviation of Pb in the subsurface soil ranged between 0.43 ± 0.29, Owaza mg/kg to 0.70 ± 0.15 mg/kg, Eziorsu (Table 10).

Table 10: The mean concentration, the standard deviation of LEAD (Pb) in soils in the dry season of the study area

	Surface			Sub-surface		
Site	Mean ± Std. Dev. (Mg/kg)	Min	Max	Mean ± Std. Dev. (Mg/kg)	Min	Max
Control	0.65 ±0.04	0.59	0.72	0.58 ±0.05	0.52	0.63
Eziorsu	0.72 ±0.19	0.48	0.99	0.70 ±0.15	0.49	0.85
Owaza	0.54 ±0.12	0.40	0.69	0.43 ±0.29	0.11	0.80
Aluu	0.65 ±0.12	0.52	0.88	0.56 ±0.04	0.50	0.60

Zinc

Zn's mean concentration and standard deviation in the surface soil ranged between 3.63 ± 0.39, to 7.69 ± 0.60, mg/kg for the Control to Eziorsu. Zn's mean concentration and standard deviation in the sub-surface soil ranged between 2.77 ± 0.33, to 6.72 ± 0.75, mg/kg for the Control to Eziorsu (Table 11).

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Table 11: The mean concentration, the standard deviation of ZINC (Zn) in soils in the dry season of the study area

	Surface			Sub-Surface		
Site	Mean ± Std. Dev. (Mg/kg)	Min	Max	Mean ± Std. Dev. (Mg/kg)	Min	Maxi
Control	3.63 ±0.39	3.29	4.13	2.77 ±0.33	2.18	3.11
Eziorsu	7.69 ±0.60	6.86	8.33	6.72 ±0.75	6.10	8.01
Owaza	5.80 ±0.70	4.91	6.66	4.32 ±0.94	3.01	5.11
Aluu	5.31 ±0.61	4.61	6.41	4.36 ±0.86	3.10	5.33

TPH Concentration in Surface and Sub-surface Soils during the Wet Season

Surface TPH results across the stations throughout the period sampled as presented in (Tables 12) showed that the variation in TPH values ranged from 0.00-2.18mg/kg. The lowest mean value of 0.85±0.72mg/kg was recorded in control, while the highest mean value of 1.76±0.42mg/kg was recorded in Owaza. One way analysis of variance revealed that Total Petroleum Hydrocarbon (TPH) was not significantly different at $p>0.05$ ($P=0.052$). ANOVA result indicated the mean ± standard deviation for TPH for sub-surface soil in the wet season from different sampling points and the control site. The control site was the lowest (1.18 ±0.27 mg/kg) while Owaza was (1.38±0.48 mg/kg).

Table 12: Mean ± standard deviation and mean range of TPH (mg/kg) in wet season at the sampling points

	SURFACE				SUB-SURFACE		
Site	Mean ± Std. Dev		Min	Max	Mean ± Std. Dev	Min	Max
Control	0.85 ±	0.71	0.00	1.57	1.18 ± 0.27	0.82	1.46
Eziorsu	1.41 ±	0.51	0.65	2.11	1.22 ± 0.22	0.82	1.41
Owaza	1.76 ±	0.42	1.10	2.18	1.38 ± 0.48	0.88	2.17
Aluu	1.14 ±	0.48	0.72	2.03	1.29 ±0.29	0.89	1.7

TPH Concentration in Surface and Sub-surface Soils during the Dry Season

The mean concentration and standard deviation of TPH in the surface soil ranged between 2.22 ± 0.34, 2.07 ± 0.68, 2.78 ± 0.69, and 1.92 ± 0.73 mg/kg for the Control, Eziorsu, Owaza, and Aluu sites, respectively. Also, the lowest and highest concentrations recorded for TPH were 1.56 and 2.53 mg/kg, 0.98 and 2.89 mg/kg, 2.21 and 3.67 mg/kg, and 1.23 and 3.25 mg/kg in control, Eziorsu, Owaza, and Aluu sites, respectively.On the other hand, the mean concentration and standard deviation of TPH in the sub-soil ranged between 2.11 ± 0.54, 1.63 ± 0.34, 2.00 ± 0.65, and 1.99 ± 0.50 mg/kg for the Control, Eziorsu, Owaza, and Aluu sites, respectively. Also, the lowest and highest concentrations recorded for TPH were 1.22 and 2.69 mg/kg, 1.00 and 1.98 mg/kg, 1.00 and 2.78 mg/kg, and 1.24 and 2.62 mg/kg in the control, Eziorsu, Owaza, and Aluu sites respectively (Table 13).

Table 13: The mean Concentration, the standard deviation of TPH in soils in the dry season of the study area

	Surface			sub-surface		
Site	Mean ± Std. Dev.	Min	Max	Mean ± Std. Dev	Min	Max
Control	2.22 ±0.34	1.56	2.53	2.11 ± 0.54	1.22	2.69
Eziorsu	2.07 ±0.68	0.98	2.89	1.63 ± 0.34	1.00	1.98
Owaza	2.78 ±0.69	2.21	3.67	2.00 ± 0.65	1.00	2.78
Aluu	1.92 ± 0.73	1.23	3.25	1.99 ± 0.50	1.24	2.62

Contamination Factor of Heavy Metals of Study Area Surface and Sub-surface Soil and Control in Wet Season

The result of contamination factor as an index of heavy metal contamination showed that the Contamination factor of Cr (mg/kg) for surface soil in wet season ranged from 0.002 (Owaza and Aluu), while control and Eziorsu recorded a contamination factor of 0.003 (Table 14). The contamination factor of copper from the surface soil ranged from 0.044 from the Control site (lowest) to 0.107, the highest from Eziorsu (Table 14). The contamination factor of Pb (mg/kg) in the surface soil indicated that Owaza (0.006) was the least while the Control site, Eziorsu, and Aluu (0.007) recorded higher values. Zinc (mg/kg), CF ranged from 0.015 (control and Owaza) to (0.044) Eziorsu in surface soil in the wet season of the study area (Table 14).

The results of the contamination factor for sub-surface soil during the wet season from the study areas indicated that, for chromium Cr (mg/kg), subsurface soil contamination faction ranged from Aluu (0.001) to Owaza with the value of 0.005 wet seasons sub-surface while Cu (mg/kg) the range was 0.058 (control) to Eziorsu 0.115. For Lead, the range was from Owaza (0.005) to Eziorsu (0.008). The sub-surface contamination of wet season soil by Zn (mg/kg) revealed a range of 0.014 (control) to 0.038 (Eziorsu). All results are presented in Table 15.

Table 14: Contamination factor of heavy metals in surface soil from the study area during the wet season

CONTAMINATION FACTOR FOR SURFACE SOIL IN WET SEASON				
Parameter	CONTROL	EZIORSU	OWAZA	ALUU
Cr (mg/kg)	0.003	0.003	0.002	0.002
Cu (mg/kg)	0.044	0.107	0.059	0.065
Pb (mg/kg)	0.007	0.007	0.006	0.007
Zn (mg/kg)	0.015	0.044	0.030	0.030

Table 15: Contamination Factor for Sub-Surface Soil In Wet Season

Parameter	CONTROL	EZIORSU	OWAZA	ALUU
Cr (mg/kg)	0.002	0.005	0.002	0.001
Cu (mg/kg)	0.058	0.115	0.076	0.068
Pb (mg/kg)	0.006	0.008	0.005	0.007
Zn (mg/kg)	0.014	0.038	0.022	0.023

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Contamination Factor of Heavy Metals of Study Area Surface and Sub-surface Soil and Control in Dry Season

The result of contamination factor for heavy metal (chromium, copper, Lead and Zinc) for Surface soil samples from the study area and the control area in the dry season indicated that the contamination factor of Chromium Cr (mg/kg) in the surface soil range from control and Owaza (0.003) to Eziorsu (0.006) (Table 16). The range of CF for Copper Cu (mg/kg) was from control (0.061) to Eziorsu (0.150). The lowest value of contamination factor for Lead Pb (mg/kg) was recorded in Owaza (0.008), while the control, Eziorsu and Aluu (0.008), recorded the highest contamination factor. The surface soil contamination factor during the dry season of Zinc Zn (mg/kg) recorded the control site (0.026) to be the lowest while Eziorsu (0.055) had the highest CF value (Table 16).

Sub-surface soil contamination factor revealed Chromium Cr (mg/kg) contamination factor for Aluu (0.002), as the lowest while Eziorsu (0.013) was the highest chromium contamination factor. Dry season sub-surface soil contamination for Copper Cu (mg/kg) ranged from control (0.076) to Eziorsu (0.165), the contamination factor of Lead Pb (mg/kg) from sub-surface soil during dry season range from Owaza (0.005) to Eziorsu (0.008). Zinc Zn (mg/kg) contamination factor range from control sitee (0.020) Eziorsu (0.048) (Table 17).

Table 16: Contamination Factor For Surface Soil In Dry Season

Parameter	CONTROL	EZIORSU	OWAZA	ALUU
Cr (mg/kg)	0.003	0.006	0.003	0.004
Cu (mg/kg)	0.061	0.150	0.076	0.084
Pb (mg/kg)	0.008	0.008	0.006	0.008
Zn (mg/kg)	0.026	0.055	0.041	0.038

Table 17: Contamination Factor for Sub- Surface Soil In Dry Season

Parameter	CONTROL	EZIORSU	OWAZA	ALUU
Cr (mg/kg)	0.005	0.013	0.006	0.002
Cu (mg/kg)	0.076	0.165	0.111	0.092
Pb (mg/kg)	0.007	0.008	0.005	0.007
Zn (mg/kg)	0.020	0.048	0.031	0.031

Enrichment Factor of Heavy Metals for Wet Season in the Study Area

The enrichment factor of heavy metals in surface soil wet season from the study area and the control site was as follows: The enrichment factor of chromium (mg/kg) from calculated data revealed the lowest value at Owaza 10.35, while the control site had the highest value control (36.53) for wet season surface soil. Values of Cu (mg/kg) showed that Aluu (323.20) was the lowest at the control site (464.64) recorded the highest value. For Lead Pb (mg/kg), the range of Igoe in surface soil was from control (74.47) to Eziorsu (29.74). For Zinc Zn (mg/kg), the lowest Enrichment factor was calculated for surface soil from Aluu

(150.93) while Eziorsu (178.69) was the highest (Table 18).

The enrichment factor for heavy metals in sub-surface soil during wet season from the study area and the control sites area presented chromium Aluu 5.34 as the lowest while Eziorsu 22.59 was the highest. For Copper (mg/kg) lowest value was from Aluu (407.25), while the highest value was from Eziorsu (508.64). EF of lead (mg/kg) ranged from Owaza 31.72 to control 47.29. Enrichment factor Zinc ranged from the control (119.83) to Eziorsu (169.59) (Table 19).

Table 18: Enrichment Factor for Heavy Metals in Surface Soil (Wet Season)

Parameter	Bn	Bref	Cref (Fe)	CONTROL	EZIORSU	OWAZA	ALUU
Cr (mg/kg)	100	38000	3.604	36.53	13.12	10.35	10.51
Cu (mg/kg)	36	38000	3.604	464.64	433.48	329.06	323.20
Pb (mg/kg)	85	38000	3.604	74.47	29.74	33.56	35.07
Zn (mg/kg)	140	38000	3.604	162.18	178.69	166.15	150.93

Table 19: Enrichment Factor for Heavy Metals In Sub-Surface Soil (Wet Season)

Parameter	Bn	Bref	Cref (Fe)	CONTROL	EZIORSU	OWAZA	ALUU
Cr (mg/kg)	100	38000	4.587	13.77	22.59	15.18	5.34
Cu (mg/kg)	36	38000	4.587	482.79	508.64	486.44	407.25
Pb (mg/kg)	85	38000	4.587	47.29	34.03	31.72	39.27
Zn (mg/kg)	140	38000	4.587	119.83	169.59	141.81	137.65

Enrichment Factor of Heavy Metals for Dry Season in the Study Area

Enrichment factor for chromium, copper, Lead in surface soil in the control sites during the dry season is presented below. Chromium recorded Enrichment factors range from Owaza (13.77) to Eziorsu (20.66). For copper (mg/kg) was from Owaza (298.37) to Eziorsu (495.98), Lead was from the lowest in Owaza (24.97) and highest in control (53.00). The enrichment factor for zinc ranged from Aluu (141.33) to Eziorsu (181.45) (Table 20 and 21).

Table 20: Enrichment Factor For Heavy Metals In Surface Soil (Dry Season)

Parameter	Bn	Bref	Cref (Fe)	CONTROL	EZIORSU	OWAZA	ALUU
Cr (mg/kg)	100	38000	5.493	20.11	20.66	13.77	14.66
Cu (mg/kg)	36	38000	5.493	423.86	495.98	298.37	313.44
Pb (mg/kg)	85	38000	5.493	53.00	28.06	24.97	28.32
Zn (mg/kg)	140	38000	5.493	179.49	181.45	163.24	141.33

Table 21: Enrichment Factor of Heavy Metals In Sub-Surface Soil (Dry Season)

Parameter	Bn	Bref	Cref (Fe)	CONTROL	EZIORSU	OWAZA	ALUU
Cr (mg/kg)	100	38000	6.992	25.03	43.56	26.17	10.42
Cu (mg/kg)	36	38000	6.992	410.87	567.26	516.80	437.38
Pb (mg/kg)	85	38000	6.992	37.11	28.41	23.72	31.18
Zn (mg/kg)	140	38000	6.992	107.61	165.40	143.80	148.38

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DISCUSSION

Mean Concentration of Heavy Metals in Soil of the Study Area in Wet and Dry Season

The mean concentration, standard deviation and range, of heavy metals (chromium, copper, lead, iron, and zinc) during the wet and dry season in selected three ex-post remediated sites (Eziorsu, Owaza, and Aluu) and a control site in the Niger-Delta is presented in Tables 2 – 6 and Tables 7 – 11 above indicate the following results:

Heavy metals were detected in varying concentrations in polluted surface and sub-surface soil. Many studies on the Niger – Delta, such as Unimke et al., (2014) and Numbere (2019), have shown that soils naturally have varying but trace amounts of heavy metals even in an undisturbed environment. Therefore, it is normal to have obtained this varying number of heavy metals at the study site.

Chromium (Cr)

The values obtained for Cr are similar to the one report by Onojake and Frank (2013) for hydrocarbon contamination in the Niger Delta. The concentration of chromium recorded below the permissible limit and target values for unpolluted soils set by WHO as 1.30 mg/kg and 100 mg/kg, respectively. This result is far below Olawoyin et. al, (2012) reports, who reported 50.6 ± 16.8mg/kg chromium levels.

The low level of chromium recorded could indicate that the remediation exercise on the previously spilt site may have helped lower chromium levels to safe limits. Except for Eziorsu 0.51 ±0.34mg/kg, the oxidation condition of chromium toxicity depends on the state of the metal (Cr occurs in two states in soils). The oxyanion chromate CrO42-, is highly mobile and more toxic in soils and groundwater. Based on the dose, exposure time and oxidation state, chromium can be helpful or harmful to health (Olawoyin et. al, 2012). The last is highly soluble, mobile, and toxic to humans, animals, and plants.

On the contrary, Cr (III) has relatively low toxicity and mobility, and it is one of the micronutrients needed by humans. In addition, Cr (III) can be absorbed on the surface of clay minerals in precipitates or complexes. The reduced ion Cr (III) forms either a weakly soluble hydroxide or stable complexes with soil minerals (Vodyanitskii, 2016).

Copper (Cu)

Copper, the mean total concentration of copper in the wet and dry soil samples (sub-surface and surface) was between 1.59 to 5.93 mg/l (as shown in Tables 3 and 8). These are slightly higher values than those reported by (C & Ezebuiro, 2006; Iwegbue et al., 2009). The concentration of copper in all the wet soil samples were below the permissible limit and target values for unpolluted soils set by WHO as 10 mg/kg and 36 mg/kg, respectively. Copper mainly occurs in its divalent state (Cu2+) and has a high affinity for binding to organic matter.

Copper was found in association with the organic matter bound fraction. Surface soil samples both in the wet and dry seasons had smaller amounts of Cu in the organic fraction than sub-surface soil samples, probably because there are fewer organic substances in the subsoil and because biological activities mainly occur at the superficial horizons. The association of Cu with organic matter fraction is due to the high formation constant of the Cu-organic complex (Abollino et al., 2002). The metal complex with the organic matter is generally described by an ion exchange organo-metallic complexation. The copper in the oxidizable phase is not considered very available because it is thought to be associated with stable high-molecular weight humid substances that slowly release small amounts of metal. Therefore, adsorption forms the most crucial controlling mechanism in this fraction.

Iron (Fe)

Onojake and Frank (2013) found Fe values ranged from 6.4 to 8.7 mg/kg with a mean of 7.7 mg/kg (5.6 mg/kg for the uncontaminated soil sample) in a part of the Niger Delta. The values are inclined with the wet and dry soil samples (sub-surface and surface) between 5.94 to 11.51 mg/kg. These values show that the dry and wet soil samples are contaminated. The control result shows that it is not contaminated in both cases, as the values are less than 5.00 mg/kg. The concentration of Fe in all the wet and dry soil samples, mostly the surface soil samples, was above the maximum permissible limit set by WHO (5.0 mg/kg).

Lead (Pb)

Mean total concentration of Lead in the wet and dry soil samples (sub-surface and surface) are shown in tables 4.4, and 4.18, C and Ezebuiro (2006); Udoetok et al., (2011) recorded values of Lead between 0.32 to 0.76 mg/kg, in Niger Delta, Region, Nigeria. Their results are lower as compared to the values obtained in this study. This result is significantly lower than the reports of Olawoyin et al., (2012), who reported lead levels of 49.5 ± 2.0 in their studies on the Potential risk effect from elevated levels of heavy soil metals on human health in the Niger Delta. In almost all the collected wet and dry soil samples, the concentration of Pb was below the permissible limit and target values for unpolluted soils set by WHO as 2 mg/kg and 85 mg/kg.

Zinc (Zn)

Iwegbue et al. (2009) reported similar values in the Niger Delta. In almost all the wet soil samples, the zinc concentration was above the permissible limit and below target values for unpolluted soils set by WHO as 0.6 mg/kg and 50 mg/kg. Zinc is a trace element of vital role in many organisms' physiological and metabolic processes. Nevertheless, higher concentrations of zinc can be toxic to the organism. In addition, zinc is an essential component of various enzymes responsible for driving many metabolic reactions in all crops. Maximum zinc values as presented in the present study are significantly lower than the reports of Olawoyin et al., (2012), whose result on Zn levels in petroleum impacted soil was 185±3.

Concentration Total Petroleum Hydrocarbon (TPH) in Wet and Dry Season Soil from the Study Area

The concentration of Total hydrocarbon content in soil samples from the three post remediated sites and control yielded a range with a lower limit of 0.85±0.72mg/kg in the control site, while the highest mean value of 1.76±0.42mg/kg was recorded in soil samples from Owaza. During the dry season, the mean and standard deviation of TPH in the soil ranged from 1.63 ± 0.34 in Eziorsu to 2.22 ± 0.34 in samples from the control site. These results are far less than Olawoyin et al., (2012) reports, which reported a TPH value of 160.4 mg/kg.

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The results are also far lower than the reports of Beg et al. (2003), who reported a mean range of TPH value of 6.7 to 2066mg/kg during their study on the distribution of petroleum hydrocarbon in sediment from the coastal area receiving industrial effluents in Kuwait. This result is also in contrast to Osuji and Nwoye (2007) finding that reported a range of 3400 – 6800mk/kg from Owaza, which is in total contrast with the values from this study in the same study area (Owaza) of 1.032 to 3.671 mg/kg. This is as a result of the effects of remediation carried out on the area.

A sampling of the study areas showed a high level of contamination of total petroleum hydrocarbon (TPH). High hydrocarbon content causes deprivation and reduction in gaseous diffusion by the surface film of oil, and this usually has a high effect on flora and fauna of the affected area, hence the soil fertility (Egedeuzu & Nnorom, 2013). The result of TPH from the present study indicates that the level of contamination by total petroleum hydrocarbon from the study sites is very low.

CONCLUSION

This study aimed to assess the conditions of previously remediated sites in three states in the Niger Delta known for intensive oil and gas activities and records of oil spills caused by different factors, as highlighted earlier. The geographical information system was used to identify two post remediated sites in each identified location in the three states. (Owaza, Eziousu, and Aluu for Abia, Imo and Rivers state, respectively). The conditions of post remediated sites have been a source of concern due to reported risks associated with human exposure to contaminants. Assessing the conditions of the Ex-post remediated site was aimed at providing data to the scientific world on the actual state of the post remediated sites in the Niger Delta region in Nigeria.

Mean concentration and standard deviation of heavy metals in soil were all within safe limits of the World health Organization for Iron (Fe) =50000mg/kg Lead (Pb) 10mg/kg, Zinc (Zn) 300mg/kg and copper (Cu) is = 100mg/kg and chromium (Cr) =100mg/kg. The Enrichment factor of heavy metals in the study area indicated that copper recorded the highest Enrichment factor for wet and dry seasons (323.20 – 508.64) and (298.37 – 567.26). In the present study, all Enrichment factor values were above the value of 1.5, so we can conclude that the pollution sources for heavy metals in the study sites are highly from anthropogenic sources.

Total Petroleum Hydrocarbon (TPH) concentrations in soil samples from all particular sites were within stipulated permissible safe levels signifying non-contamination by TPH.. Heavy metal levels in soil samples were within permissible levels by the world health organization (WHO) and other regulatory bodies, which indicated the effectiveness of the remediated work carried out on the previously spilt sites.

Though the results of this study reveal low contamination of the heavy metals in soil samples, it is recommended that a routine check on the levels of contaminants be carried out regularly. Effective remediation should ensure that background levels of TPH and heavy metals are kept in check to prevent exposure to heavy metals and other pollutants. Enrichment indicates anthropogenic sources of pollutants, which we recommend conscious and deliberate steps be taken to curb environmental ills such as oil pipeline sabotage, which leads to spills that endanger our environment and the health of the people.

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