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Systematic Determination of Hydrocarbon Contamination of Shoreline Sediments in Taylor-Creek Sites Niger Delta Region of Nigeria

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Abstracts: *The Niger Delta region of Nigeria over the years have been ravaged with series of hydrocarbon contamination/pollution due to various anthropogenic activities related to oil and gas industries. The main aim of this research work was to investigate the total petroleum hydrocarbon (TPH) concentrations in various environmental matrix in the study area, and the efficacy of some alternative analytical techniques for the determination of hydrocarbon concentration in shoreline sediment. A total of fifteen (15) sediment samples were collected and analysed from fifteen (15) sampling locations and a replica sample analysis were carried out for all samples to obtain a sample mean and its standard deviation. The results indicated the presence of TPH concentration at various degree in sediment samples which ranged from 206.9 to 822.0 mg/kg. The TPH mean of 500.1±206.9 mg/kg of sediment sample was above the EGASPIN target value of 50 mg/kg. From the one sample T-test, sediment with TPH total mean concentration of 500.1 mg/kg with a standard deviation of 206.9 with a calculated t-value of 8.425 lies within the rejection region (critical value-1.761, df-14 one sided and a p-value-0.0007). Thus, the null hypothesis is rejected and concluded that the total TPH mean concentration for TC sediment samples of 500.1 mg/kg is greater than the EGASPIN target value of 50 mg/kg for sediment environmental quality standard. While majority of the analysed sediment samples were below the EGASPIN intervention value of 5,000 mg/kg, most of the sample TPH concentration were far above the 50 mg/kg target value. From the one sample T-test, TC sediment with TPH total mean concentration of 500.1 mg/kg with a standard deviation of 206.9j with a calculated t-value of -84.22 lies within the retention region (critical value-1.761, df-14 one sided and a p-value-1). Thus, the null hypothesis is accepted and concluded that the total TPH mean concentration for TC sediment samples of 500.1 mg/kg is less than the EGASPIN intervention value of 5,000 mg/kg for sediment safety limit. Hence, river sediments are contaminated with hydrocarbons in the study area. The study outcome shows that sediment with records of crude oil spill in the past are still contaminated with hydrocarbon which is as a result of poor or none remediation/clean-up of the affected sites, which is attributed to none compliance of extant environmental laws by International Operating Companies (IOCs) operating in the study area and the lack of government regulatory agencies in enforcing the established environmental laws.*

Keywords: systematic determination of hydrocarbon contamination of shoreline sediments in taylor-creek sites Niger Delta region of Nigeria

INTRODUCTION

Petroleum in the environment from either natural or anthropogenic sources, whether from pipeline corrosion, explosion, or from sabotage is seen as a vital environmental problem (Fent, 2004). At a global scale, major oil spill that have occurred received high level of attention and concern from the public because of the observable associated environmental damage (Farrel-Jones *et al.,* 2003). However, the release of small amount of oil into the ecosystem continuously over a long period of time, which results in chronic exposure of organisms to petroleum and its component chemical compounds also pose a serious threat to the ecosystem survival (Rodrigues *et al.,* 2009).

Petroleum is composed of several hydrocarbons, it causes severe environmental consequences when discharged into any ecosystem, whether through natural or chronic sources, which is due to their physical properties (e.g. smothering) or chemical properties (e.g. toxicity) (Umukoro, 2012). And owing to the large number of hydrocarbon compounds present, the health and environmental effects of all petroleum constituents' parts have not yet been completely studied or understood (NRC, 2003; Fent, 2004; Alrumman *et al.,* 2015). However, the harmful effect of oil pollution on the ecosystem is enormous; it leads to death of plants and animals in marine and terrestrial zones, it settles in beaches and kill organisms living on it, it kills benthic organisms in oceans, it causes algae poisoning. It disrupts major food chains and reduces the yields in the environment (Nwilo *et al.,* 2006; Omemu and Aderoju, 2008).

Several oil spills that have occurred of recent times have recorded severe damage to vulnerable ecosystems such as; the Gulf of Mexico - the Galapagos Island in France, the Sundarbans mangrove in Bangladesh, Ogoniland in Nigeria, and many other places (NRC, 2003; Labud *et al.,* 2007; Rodrigues *et al.,* 2009; Zabbey *et al.,* 2017). The associated ecological damages that are anchored with major oil spills that are devastating includes; the effect on living organisms such as; microorganisms, animals, plants, mammals and human, oil-coated shorelines, dead or moribund wildlife and etc (Orubu *et al.,* 2004; Akpabio and Akpan, 2010; Lim *et al.,* 2016). Economic and social impacts are also felt in major oil spills, as crude oil is a significant primary source of fuel, and the versatile applications in the downstream sector and other sectors (NRC, 2003).

In the Niger Delta region, environmental impacts on the ecosystem have been documented (Orubu *et al.,* 2004; Akpabio and Akpan, 2010). These impacts include; damage of mangrove, adverse surface and groundwater contamination, damage of vegetation and farmlands in the region (Kadafa *et al.,* 2012). Hence, petroleum hydrocarbon pollution in the Niger Delta region is posing a threat to food security, and as well a serious source of ecotoxicological impact in the region (Zabbey *et al.,* 2017).

In terms of environmental impact assessment records, the Niger Delta region lacks up-to-date monitoring, and a comprehensive data base prior to oil spills, have exacerbated the difficulties associated in determining the effects of oil spills in the ecosystem accurately (Akpabio and Akpan, 2010; Kadafa *et al.,* 2012). Also owing to the absent of an existing data base of species diversity and abundance in many ecosystems have made it difficult to obtain the severity of most oil spills especially the Niger Delta region of Nigeria (Omemu and Aderoju, 2008). However, the impacts on various sectors in the ecosystem in the region through research are documents at different level (Omemu and Aderoju, 2008; UNEP, 2011; Kadafa *et al.*, 2012; Zabbey *et al.*, 2017).

MATERIALS AND METHOD

Brief Geology and Hydrogeology of the Study Area

The topography of the study area is fairly gentle with an average elevation stands of about 50 m above sea level. Figure 1 shows the map of the study area. It has an average annual rainfall of about 1,450 mm to 2,400 mm which occurs mostly in the rainy season which also provide adequate groundwater recharge (Udom *et al.*, 1998). The area has quite a few numbers of perennial rivers and streams which forms a network that empties water into the Atlantic Ocean. Hence, most of the area is marshy and in some cases forms beaches.

Figure 1: Map showing the study area and sampling location in upstream and downstream areas. Source; www.bayelsagis.gov.ng (Accessed, 12/07/2019)

The study area is situated in the coastal Niger Delta sedimentary basin which is endowed with the core characteristics of the Niger Delta sedimentary rock. The geology and hydrogeology of the area has been reported by several literatures such as Etu-Efeotor and Akpokodje (1990), Amajor (1991), Edet (1993), and Udom *et al.*, (1998). For example, a study carried out by Abam (1999), on the dynamics and quality of water resources in the Niger Delta stated that the area is underlain by sedimentary formation (Benin formation). The author reported that the rocks were divided into the oldest Palaeocene (Akata formation), Eocene (Agbada formation) and the Miocene (youngest Benin formation). They consist of fluvial and lacustrine deposits. Abam (1999) concluded that the sediment of the coastal plan was about 2,000 m thick and it comprised of lenticular and unconsolidated coarse to medium-fine grained sands with interbreeding localised clay/shale. According to Amajor (1991), the Benin formation has a lithology which is made up of sands, silts, gravel and clayey intercalation. According to Nwankwoala *et al.*, (2014), the study area is reported to have an average depth of borehole of 15 to 50 m. The main source of groundwater recharge in the area is through rainfall in the area (Udom and Acra, 2006).

Climate of the Study Area

The Niger Delta region is located within the sub-equatorial region of Nigeria as shown in Figure 2 the study map. Temperature of the study area ranges from 20°C to 33°C, while the hottest months are from February to March. Figure 2 showing the mean monthly rain fall in the study area. Humidity is high with a mean value of about 75%rh. Basically, the study area is characterized by two distinct seasonal patterns known as, the wet season and the dry season.

The wet season commerce's from April to October while the dry season is from November to March. In the period of the wet season, the rainy season starts in March and ends in October with maximum in June and July. The rains are brought in by the southwest trade winds. A break, that is close to two weeks is observed within the period of the wet season, it occurs in the month of August and is popularly referred to as August break (it a close to two weeks non rainful period experienced during the raining season, after which the raining season continues till October). Annual mean rainfall in the area is more than 3000 mm. Within this period there is a short hamartan in the months of December and January. The dry season is brought in by north eastern trade winds.

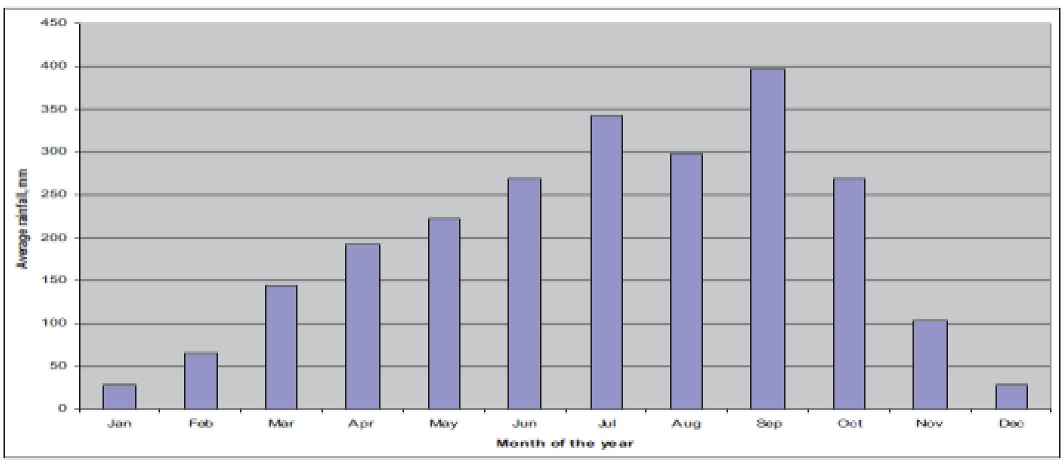

Figure 2: Mean monthly rain fall in the study area (UNEP, 2011)

Vegetation

The vegetation of the area under study is characterized by fresh water swamp forest, mangrove swamp and tropical rain forest.

The Fresh Water Swamp forest are areas that have fresh water vegetation and consist mainly of raffia palms and Africa mango, which are both economically very important due to their uses. The sap of the raffia produces palm wine which is processed into local gin. The Africa mango produces ogbono (irvingia gabonensis), other plant species commonly found in the area include Cuspiodala, Vossia, Nymphet, Lotus grasses, sedges such as Mariscus sp, Paspum Veginatum and climbers.

The Tropical Rain Forest vegetation is known for its three-layer canopy structure. It is well drained but experiences heavy rain falls with high temperature which supports the growth of tall and big trees in the region. The vegetation is dense and thick thus consist of both hard wood and soft wood. These trees are of greater economic value. They are used to produce timber through lumbering (for building, furniture, use for industrial work and can be converted into charcoal).

Taylor Creek (TC) - Kilama Community

Kilama community located in Yenagoa LGA of Bayelsa state, Nigeria, with a GPS coordinate of N05°14.433' E006°32.009' is a host community to SPDC. It serves as a transit community to NAOC as pipeline passes through the community to other major oil and gas facilities like flowing station and manifold. Kilama community has been a host to SPDC for over three decades and has experienced several degrees of oil spill incidents in the community.

Figure 3. Taylor Creek in the Study Area, were surface water and sediment samples were collected TPH analysis. (Picture captured during field exercise of this research work by author, 24/04/2019).

Taylor creek is amongst the fishing creeks owned and used by the Kilama community and members of its neighbouring community, it also serves as source of drinking water and route to farm lands. The Taylor creek like other creeks in the Niger Delta region, has been faced with several incidents of oil spill over the years. In 29th to 30th April and 14th to 15th May, 2013 respectively, the Taylor creek experienced two incidents of crude oil spill within a time frame of one month as a result of equipment failure. Figure 3 showing Taylor creek sampling location of the study area. Several barrels of crude oil is released to the creek and it's environ. Surface water, sediment and soil samples were collected analysed from this station. Findings from relevant sources have shown that clean-up has been carried out in the various petroleum hydrocarbon polluted sites. Hence, the site is considered as a remediated site for the purpose of this research work.

Sample Collection

For sediment, samples collection was carried out in six different stations (Rivers), and in each location, fifteen (15) samples were collected at different sampling points with a locally manufactured hand auger as shown in Figure 4.

Figure 4 showing hand auger locally made for the collection of soil samples in the study area

Analytical Technique

In-field Determination of TPH with - ELISA (Immunoassay Techniques)

Standard Sample Preparation; Calibration Curve Standard Sample Preparation

Standard samples preparation for diesel was carried out based on the linear range of the instruments following the manual book. Although six standard concentration of total BTEX of (0.00, 0.09, 0.35, 1.00, 2.00 and 3.00 mg/L) standards in buffered saline containing preservative and stabilizers were supplied with the portable TPH test kit by ModernWater Inc.

Standard solution samples of various volumes ranging from 0.1 µL, 0.3 µL, 0.5 µL, 0.8 µL, 1.0 µL, 1.5 µL and 2.0 µL of diesel stock solution which are concentration equivalent of 0.084, 0.254, 0.420, 0.672, 0.840, 1.680, 4.20 and 8.40 mg/L respectively were used to prepare the calibration. However, it is only six calibration standard that is used during the field exercise as the Rapid Assay instrument used have provision for only six calibration curve slots. For the field exercise for samples analysis, 0.00, 0.420, 0.84, 1.62, 2.80 and 4.2 mg/L were used for the calibration of the equipment.

BTEX Calibration Curve for Samples (Diesel)

The results of the six known BTEX standard solutions of 0.00, 0.09, 0.35, 1.00, 2.00 and 3.00 mg/L obtained from the Rapid Assay test kit are shown in Table 1. These results are used to plot the calibration curve for manually prepared standard samples of diesel analysis. Figure 5 shows the calibration curve. These calibration curves were used as predictive models to calculate the concentration of TPH in the samples (prepared standard samples of diesel). The analytical results of the diesel standard samples prepared for the test of precision of method for the eight (8) various concentrations are shown in Table 4.2. All samples were analysed three times and the average taken as a representative mean. Hence, three replicates samples results were produced and the average taken as the sample mean for each analysed sample.

Table 1: BTEX Standard readings from Rapid Assay Test Kit

Concentration (mg/L)	0.00	0.09	0.35	1.00	2.00	3.00
Absorbance	1.106	0.964	0.795	0.634	0.403	0.214

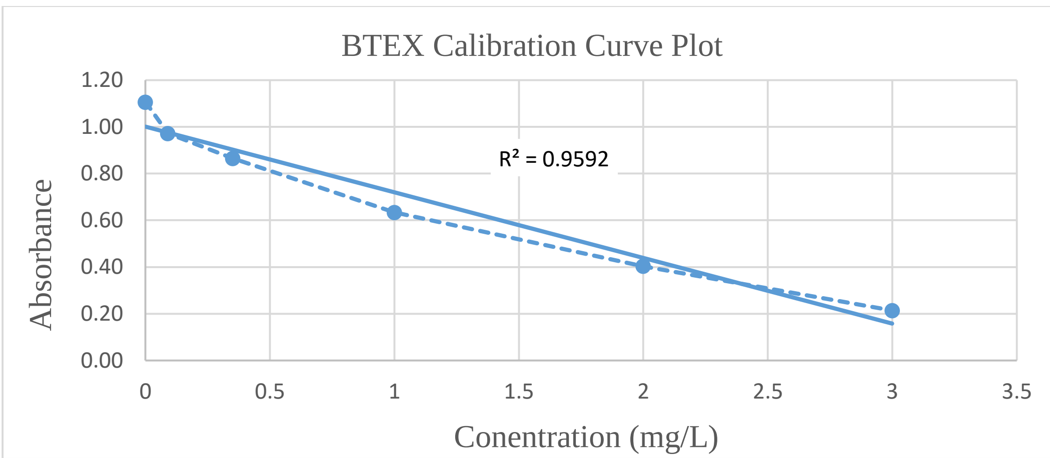

Figure 5: BTEX standard calibration curve plot used to calculate the diesel prepared samples. It shows the regression line and a coefficient of determination (R^2) value of 0.9592. This calibration curve plot is used to calculate the low range prepared standard samples of diesel.

RESULTS

Analysed Sediment samples

The results of the sediment samples analysed are shown in Table 2. For each of the fifteen sampling points, after the calibration of the Rapid Assay test equipment with the required prepared standard solution which were prepared according to the linear range of the equipment, each sediment sample (composite) was analysed three times to determine the random variability. The normality test carried out showed that results obtained are normally distributed, hence, a parametric test procedure where followed for the various data analysis.

Table 2: Descriptive statistic summary of sediment samples that were analysed three times each for TPH concentration from the fifteen various sampling points of the study area with the standard deviation as the error bar.

Location	**Taylor Creek**
Sample point 1	751±1.528
Sample point 2	764±2.082
Sample point 3	648±7.638
Sample point 4	353±1.667
Sample point 5	822±25.66
Sample point 6	642±2.887
Sample point 7	527±25.17
Sample point 8	245±2.887
Sample point 9	652±2.082
Sample point 10	213±2.887
Sample point 11	410±3.341
Sample point 12	562±4.213
Sample point 13	323±1.241
Sample point 14	274±0.837
Sample point 15	316±0.654
Ranges	213.0 – 8220
Mean	500.1
SD	206.9
Skewness	0.091

Sediment Regulatory Standard for TPH Intervention and Target Values

Results in Table 3 and 4 presents the calculated one sample t-test parameters of sediment TPH concentration obtained from the study area against the EGASPIN for target value and intervention value respectively. Table 4.16 shows the summary statistics of the total mean concentration and pooled standard deviation for sediment samples TPH concentration analysed from the six sites including the control site in the study area. Table 2.5 shows the EGASPIN sediment intervention and target value for TPH pollution.

Table 3: One sample t-test parameters calculated for the EGASPIN target values standard (50 mg/kg) comparison for sediment samples TPH total mean concentration.

Parameters	**Taylor Creek**
Average mean	500.1
Test statistics	8.425
Critical value	1.761
df	14
p-value	0.0007

For TC, the H_o; μ_{OY} < EGASPIN target value of 50 mg/kg against the alternativeH_1; μ_{OY} > EGASPIN target value was determined with a ProUCL. From the data analyses, TC sediment with TPH total mean concentration of 500.1 mg/kg with a standard deviation of 206.9 with a calculated t-value of 8.425 lies within the rejection region (critical value-1.761, df-14 one sided and a p-value-0.0007). Thus, the null hypothesis is rejected and concluded that the total TPH mean concentration for TC sediment samples of 500.1 mg/kg is greater than the EGASPIN target value of 50 mg/kg for sediment environmental quality standard.

Table 4: One sample t-test t-parameters calculated for the EGASPIN intervention values standard (5,000 mg/kg) comparison for sediment samples TPH total mean concentration.

Parameters	**Taylor Creek**
Average mean	500.1
Test statistics	-84.22
Critical value	1.761
df	14
p-value	1

For TC, the H_o; μ_{OY} < EGASPIN intervention value of 5000 mg/kg against the alternativeH_1; μ_{OY} > EGASPIN intervention value was determined with a ProUCL. From the data analyses, TC sediment with TPH total mean concentration of 500.1 mg/kg with a standard deviation of 206.9 with a calculated t-value of -84.22 lies within the retention region (critical value-1.761, df-14 one sided and a p-value-1). Thus, the null hypothesis is accepted and concluded that the total TPH mean concentration for TC sediment samples of 500.1 mg/kg is less than the EGASPIN intervention value of 5,000 mg/kg for sediment safety limit.

Histogram Plot of Sediment Sample Sites

The histogram plot of the sediment sampling location is shown in Figure 6. From the plots, the concentration frequency and the concentration ranges of the analysed samples are indicated pictorially.

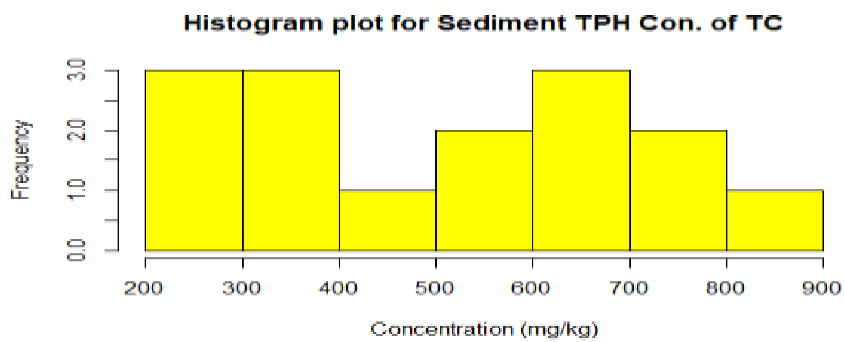

Figure 6: Histogram Plot of Sediment TPH Concentration of Taylor Creek (Kilama Community)

The data in Figure 6 illustrated the TPH concentration frequency of analysed sediment samples of Taylor Creek in Kilama community of the study area. From the data, TPH concentration ranges from 213 to 822 mg/kg. From the histogram plot, TPH concentration values between 213 to 499.9 mg/kg comprises of 46.7% of the total analysed samples and is more dominant within the 15 analysed sediment samples, while TPH concentration values between 500 to 699.9 mg/kg comprises of 33.3% of the total analysed samples, and TPH concentration range between 700 to 900 mg/kg comprises of 20% respectively out of the total analysed sediment samples of Taylor Creek of Kilama Community. Histogram plot was obtained with the R/Studio package.

Taylor Creek (TC) Sediment sample analysis

Results obtained from sediment samples from the study area are shown in Table 2, and the results indicates the presence of TPH in samples at various degrees. From the sediment samples summary statistics in Table 2, the TPH concentration from sediment samples of the selected locations in the study area ranged from 206.9 to 822.0 mg/kg of wet weight in various sampling points. TPH concentration of the various samples analysed were within the 0.02 mg/kg detection limit of ELISA test kit used for the field analytical exercise.

A total of 45 sediment samples in fifteen different sampling sites were analysed for the determination of TPH concentration in the study area.

Results from the one sample t-test carried out to determine if the sediment standard for TPH in the Taylor creek sampling location is up to the EGASPIN recommended standard for sediment sustainability and functionality for human, animal and aquatic life is shown in Table 3 and 4 respectively. From the one sample t-test, the TPH mean concentration of 500.1 mg/kg obtained from sediment samples analysed was above the EGASPIN target value of 50 mg/kg, while it was below the intervention value of 5000 mg/kg for TPH in sediment (t-value, 8.425, critical value 1.761, df, 14, one sided, p-value, 0.0007) and (t-value, -84.22, critical value 1.761, df, 14, one sided, p-value, 1) respectively. The histogram plot indicates the various concentration frequency range with 213 to 499.9 mg/kg comprised of 46.7% and was the dominant concentration interval in the study location as shown in Figure 6.

The results of the analysed river sediment samples from the study location shows TPH concentrations that were all above the target value of 50 mg/kg for sediment functionality, however they stand below the intervention value of 5000 mg/kg. The findings from the study location shows an environment that is moderately contaminated with petroleum hydrocarbon. Nonetheless, this TPH concentrations recorded are not up to the EGASPIN recommended value for a remediation or clean-up to be carried out in a crude oil contaminated site. Although, the confirmed results were far above the target value of 50 mg/kg, thus, the river sediment of the study location required an immediate consideration for remediation/clean-up to preserve aquatic lives and the ecosystem at large for ultimate utilization (UNEP, 2011; Kadafa *et al.*, 2012). No preceding studies relating to petroleum hydrocarbon pollution has been completed in this study location, though, the presence of the TPH concentration found in samples are attributed to petroleum hydrocarbon through crude oil spillages that has occurred in the study location overtime.

Subsequently, for the sediment in the study area, the results obtained has shown that the region sediments area contaminated with various degree of hydrocarbons. From the various sampling location data analysed, some sampling location TPH concentrations were as high as 10 times the regulatory standard as recommended by EGASPIN as target value. Although, the value obtained were below the EGASPIN intervention value for remediation to be carried out, but the TPH level obtained were far above the target value. Hence, there is a need for the relevant government agencies like NOSDRA, HYPREP and the ministry of environment to critically carry out a baseline study or and EIA in the study area with respect to hydrocarbon pollution for subsequent remediation actions. Rivers disturbance as boat movements and waves has the ability to shake the sediment and reintroduce hydrocarbons settling in the sediment to surface water and as such it is a serious threat to human and other aquatic life's. It has the ability to impact negatively on benthos and bottom dwellers.

CONCLUSION

For sediment samples, fifteen (15) sampling locations were selected and the results obtained from each sample analysed indicated the presence of TPH concentration at various degrees in sediment samples of the study area. In this research, a total of forty-five (45) sediment samples were analysed. From the analyses, sediment samples TPH concentration obtained ranged from 213 to 822 mg/kg with an average concentration of 500.1±206.9 mg/kg which were above the EGASPIN target value of 50 mg/kg, while they were below the EGASPIN intervention value of 5000 mg/kg for a sediment environmental quality standard.

1. Consequently, this research work has shown that the sites with records of crude oil spill in the past are still contaminated with hydrocarbon which is as a result of poor or none remediation/clean-up of the affected sites, which is attributed to none compliance of extant environmental laws by IOCs operating in the area and the lack of government regulatory agencies in enforcing the established environmental laws (UNEP, 2011; Kadafa *et al.*, 2012). Weakness in the enforcement of established laws and the reluctance on the part of the government to fully implement the laws have been suggested to be largely due to the fact that the government do not wish to scare major investors in the oil and gas sector that is important to the nation's economy (UNEP, 2011; Kadafa *et al.*, 2012; Anejionu *et al.,* 2015).

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