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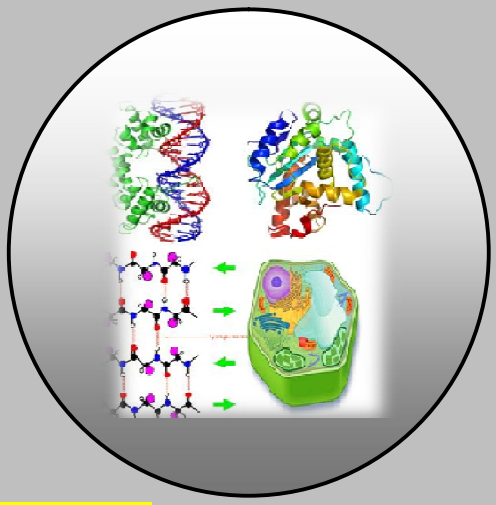

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RESEARCH PAPER

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Characteristic Assessment of the Levels of Hydrocarbon Contamination of Soil after Massive Crude Oil Spill

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ABSTRACT

57 contaminated soil samples from a crude oil spilled area were investigated about two months after a massive spillage from a petroleum Well-head in South-South Niger Delta. 400-600g of soil were collected at depths of 0-15 cm, 15-30 cm and 30-60 cm, prepared and analysed using gas chromatography equipped with a flame ionisation detector (GC-FID). Penetration and migration of C5-C9, C_{10}-C_{26} and C_{26} and above hydrocarbons through the soil layers were assessed by Principal Component Analysis (PCA) with cluster analysis to determine the distribution, penetration and similarity of these compounds over the contaminated area. The results indicated that total petroleum hydrocarbon concentrations varied from 12 – 401±3 mg kg^{-1} topsoil, 9 –325±1 mg kg^{-1} subsoil and 11 – 197±12 mg kg^{-1} at the greatest depth measured. The results also showed elevated levels of total hydrocarbon contents when compared with the reference sites. The paper recommends careful monitoring and sustainable remediation of the site by competent professionals.

Key Words: ***Liquid Hydrocarbon, Petroleum contamination, Remediation and Gas chromatography fitted with flame ionisation detector (GC-FID).***

INTRODUCTION

In recent time, there has been frequent contamination of soil and sediment from petroleum spills constituting an environmental problem globally. Crude oil contamination of soil by oil exploration activities has quickly become a considerable environmental issue significantly (Okop and Ekpo, 2012) ITOPF, 2012 (Jorge et. al 2012). Petroleum spillage from pipe lines, leakage from storage (surface and underground) tanks, and similar discharges associated with petroleum bring about environmental health risk and agricultural defects (Bosco, 2005), (Abrahams, 2002).

The economy of Nigeria, the most populous, black African country is largely dependent on crude oil tapped from the Niger Delta region though with the attendant hazardous effects meted on the environment (Iwegbue, 2007, Chukwujindu, 2008).

Not all the damaging effects of petroleum spillage are completely understood, despite effort to document such impacts on journals, scientific and other technical literature. There is continuous evidence of environmental harm though the analytical goal for each petroleum spill site is to access the level of contamination in the soil with the aim of returning the soil back to a useable form. The presence of these liquid hydrocarbon contaminants in the environment, will automatically incur natural weathering processes that act on the oil hydrocarbons and reducing the total concentration present as time progresses. These natural processes are often enhanced by addition of man-made fertilizers as part of the clean-up procedures (Garcia-Blanco et al, 2007). Attempts for complete removal may not be practically attainable either due to cost or source of contamination. The immediate objective is to remediate the soil to the concentration levels that will be harmless to plants, fauna, human health and the entire ecosystem (USEPA, 1990, NYS, 1991). The way to handle, dispose or reuse non-hazardous petroleum contaminated soils has received attention (Torres, 2005). *In situ* bioremediation proved to be an accepted mechanism to reduce the oil hydrocarbon load as it usually creates less significant disturbances than other the physical methods (Fingerman and Nagabhushanam 2005). A reasonably large proportion of the spilled oil is ultimately taken off from the environment by biological activity although the more resistant long chain and aromatic compounds may be persistent for many years (Short et al, 2007).

The Optimum extraction time for the soil samples was established using dichloromethane (DCM) by comparing the extraction efficiencies of other solvents. A gas chromatography equipped with flame ionisation detector (GC-FID) capable of split injection with a Varian CP-Sil-GC capillary column and Combi Pal was employed. Gas chromatography is one of the most powerful, popular, unique and readily versatile analytical techniques used for the separation, identification, and quantitative assay of compounds in the vapour state. It still remains the most important single technique for oil spill identification partly because the equipment is relatively available, easy to operate with small amount of operator time and considerable amount of information can be gathered on using a high resolution (capillary) column. FID limitations (Draft, 1991) include ability to destroy the sample, detect volatile hydrocarbons from non-petroleum matter and organic material such as methane and peat.

The ultimate aim is to efficiently and safely remove the spilled petroleum products from the soil with the aim of returning the soil to its useful and natural condition.

It is believed that this work will offer useful informative data to oil companies and the Government to adopt in each petroleum spill site to access the level of contamination.

MATERIAL AND METHODS

Site selection and Sampling

The sampling site was located in Ikot Ada Udo, Ikot Abasi in Akwa Ibom State, South-South Niger Delta, Nigeria and covered about 250 x 350 m^2 and situated within longitude 7°41'-7.43'E and latitude 4°41' - 4°49' N.

At this site, soil and sediment have been frequently subjected to petroleum spillages and crude oil leakages from a Shell marginal oil pipeline called ''Ibibio I'' - a Well head established in 1954. A sample is an informative representative of a population and hence sampling is considered a vital and one of the most crucial steps in the procedure of analysis of organic pollutants in soils and sediments of our environment (Tadeusz and Jacek, 2002; Alain et al, 2006). An initial survey was carried out on the site to gather information about the sampling area and to establish any possible source of obstacle that may arise during sampling. The surveillance also enabled the researcher to determine the soil type, the terrain and the feasibility of using the hand soil auger for sampling as well as recognise the correct first aid kit and personal protection equipment (PPE) to use.

Sample collection and preservation

All sampling points were measured in meters from the well head as a reference point (Figure 1). Approximately 500 g of soil was collected at each sample depth. Four control site samples were taken from the same geographically uncontaminated soil to determine the background levels of petroleum hydrocarbons for comparison with the contaminated soil. A hand soil auger (Nickel-plated carbon steel, 3´´ diameter) was used to collect soil samples from the site by taking about 5-10 auger borings at random to the depths of 0 to 15 cm at the top soil, (TS) 15 to 30 cm at middle soil (MS) and bottom layer (BL) of 30 to 60 cm. Samples were collected into zip type plastic bags and placed in a 1 L glass jar with Teflon lined cap and seal. The sampling average ambient temperature was 28°C. All the samples were carefully labeled during sampling, separated from other sampling points during storage. The samples placed in icebox and transported to the laboratory for storage at 4°C until analysis was completed. Sampling took in one day. The auger was cleaned with water and rinsed with methanol after every sampling point.

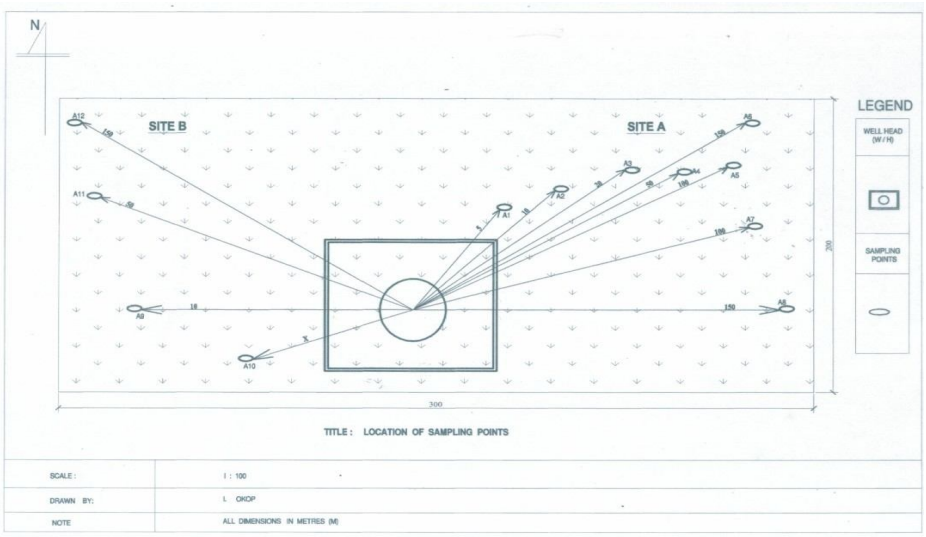

Figure 1. Sampling points randomly taken with reference from the Well Head. Measurements in metres (m).

Sample Preparation, Extraction and Clean-Up.

All samples and controls were prepared and shipped the same day by air to United Kingdom for analysis by the chemical shipping agent in Nigeria with adherence to full special shipping procedures for transporting and handling of the samples (ASTM, 2005). The soil samples were homogenized at ambient temperature using mortar and pestle to obtain finer texture and to remove sticks, pebbles and rock particles. Soxhlet extraction using a Brinkmann Büchi 461 automated extraction apparatus was employed in this work because it really ensures intimate contact of the sample matrix with the extraction solvent and a reasonably large amount of 5-20g could be used to allow quantitative extraction. Soxhlet technique is usually the adopted reference and most often used method for a long time (Draft International Standard, 1995; Berst et al, 1991). All samples were extracted using this procedure as outlined in U.S. EPA method 3540 (U.S. EPA, 1996) and ASTM method D5369 (ASTM Standards, 2005) with slight modifications in the solvent choice and volume, extraction time and size of extraction flasks. About 2.0 hours optimum extraction time (Figure 2) was established using dichloromethane (DCM) after comparing with other extraction solvents such as acetone, toluene, methanol, hexane, ethyl benzene and the mixtures of these solvents. DCM proved to be the most suitable solvent over hexane, acetone, toluene, ethyl benzene or the mixtures for this extraction due to its consistency, efficiency and ability of not interfering with BTEX retention time window (RTW) at C_5-C_9.

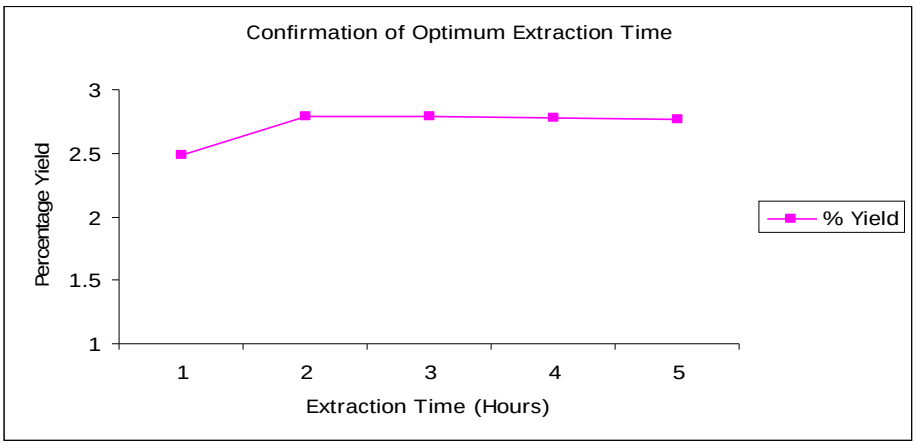

Figure 2. Plot of average percentage yield of extract obtained with time of extraction using DCM as solvent.

Each of the sample extracts were cleaned to remove moisture, polar hydrocarbons, colour interferences and any impurities before subjecting them to GC column analysis. The clean-up procedure effectively removed hydrocarbons of natural origin and did not have any significant effect on the amounts of petroleum hydrocarbons present but aid in column performance. This was achieved by filtering the extract under applied pressure through dual layer 6 mL glass, 2g/2g Florisil®/Na_2SO_4 SPE Tube (Figure 3) supplied by Fluka Analytical, Sigma Aldrich, Switzerland.

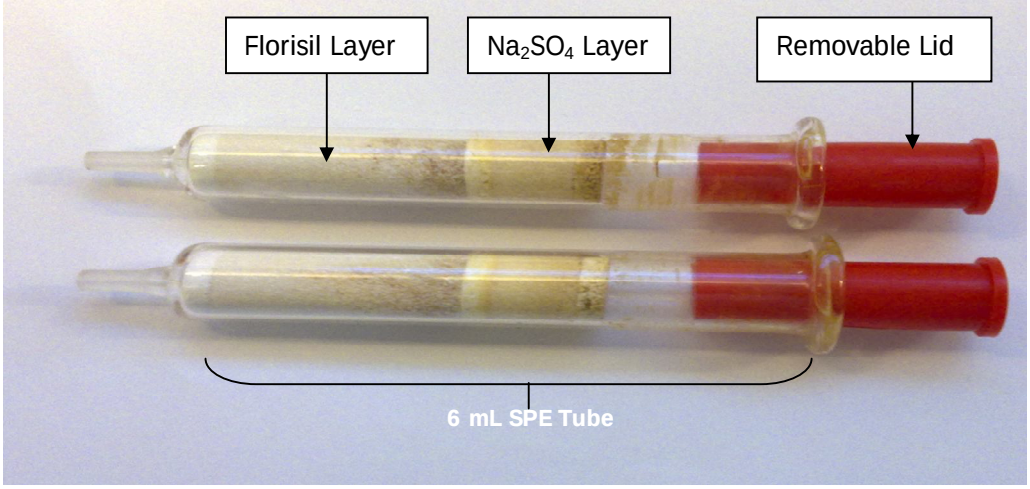

Figure 3. A pictorial representation of used Dual layer Florisil®/Na_2SO_4 tube for sample clean-up aimed at removing moisture, polar hydrocarbons, colour interferences and impurities before injection into the GC column.

Instrumentation Gas chromatograph fitted with the flame ionisation detector (FID) equipped with an automatic sampler CTC Analytics CombiPAL and the 1177 split/splitless front injector was used. All samples were taken into 2 mL chromatographic vial, injected and separated on a Varian Chrompack capillary column CP 5860 with coating of 95% methyl and 5% phenyl-polysiloxane phase, (oven max tempt 350°C), WCOT fused silica, 30m long, 0.25mm inside diameter(id), 0.39mm outside diameter (od) and 0.25µm film thickness with CP-Sil 8 CB low bleeds/MS coating. Carrier gas was helium 26 cm sec^{-1}. Temperature programme during the chromatographic analysis was 50°C for 3 min; 8°C/min to 320°C hold 15 min. Detection at 320°C. The carrier gas was helium (99.99 % pure) at velocity of 26 cm sec^{-1}. Sample injection volume of 1µL, 1:25 split ratio and column flow rate of 1.0 mL min^{-1} were applied.

RESULTS AND DISCUSSION

The samples analysis was carried out along with the certified reference and laboratory standards. Each sampling point/hole yielded three grab samples taken at different depths of 0-15 cm (top soil, TS), 15-30 cm (mid soil, MS) and 30-60 cm bottom level, BL). Samples were analysed and chromatograms overlaid with the reference standards to confirm the samples identity and retention times as parts of the validation and operational checks. The average peak values of all the samples were recorded and their standard deviation and % RSD calculated at 95% confidence level. Laboratory standards (C10, C11, C14, C15, and C16) were prepared, analyzed and overlaid with sample and confirm with certified standards purchased.

The overall concentration of total petroleum hydrocarbon (TPH) of each sample depth at the site were established and presented in table 1. The investigation of the site revealed that sample number 6 had the highest concentration of 401±3 mg kg^{-1} of total hydrocarbon recorded at the top soil (TS) level of depth 15-30 cm. Sample number 9 had a higher concentration of TPH (325±1) at the MS level, while the last level measured had a high TPH of 213±9 mg kg^{-1}. Least concentration level of total hydrocarbon was observed in all the soil strata of sample 2 location. Sample points were chosen randomly and recorded serially as sampling proceeded for the purpose of sample identification.

Control samples were taken from four points with geographical similar but non-spilled areas. Trace concentration of total hydrocarbons was recorded in these areas compared to the high level of contamination recoded in the real samples (table 1). High concentration levels of hydrocarbons present in contaminated sites could pose a health risk to humans, plants and animal lives and the entire ecosystem.

The samples showed elevated concentration of TPHs when compared with control samples in all the sites. The high levels of TPH contamination observed in this study for the crude oil contaminated soils far exceeded the fifty parts per million (50 $mgkg^{-1}$) compliance baseline limit (DPR, 1991) set for petroleum industries in Nigeria. The concentration of TPH at the top soil (0-15 cm) depth was higher than the concentration range reported by (Ekudanyo and Obuekwe, 2004; Iwegbue et al (2007) for oil spilled soils of other parts of Niger Delta and other areas (CCME 2001). The observed inflexible standards enforced around the world are in the range of 100 – 200 mg kg^{-1}.

Certified Reference Standards were used in calibrating, identifying and validating the compounds in the analyzed samples. The standard with even numbers of hydrocarbons from C_{10} to C_{40} and instrument sensitivity made separation up to C_{36}.

This work showed types, distribution, and movement pattern and penetration levels of the petroleum hydrocarbon contaminants in the study area. Principal Component Analysis (PCA) using multivariate statistical package MINTAB (Minitab, 2003) was adopted to interpret and classify chemical characteristics of the sample. The aim was to fuse the huge chromatographic data into a simple plane graph projection, thereby reducing the amount of data or number of dimensions without losing the integrity and relevant information of the samples.

In table 1, the TPH concentration for the top soil (0-15 cm depth) range from (2 ±5 to 401 ±3) mg kg^{-1}. The mid or sub-soils (15-30 cm depth) had a concentration range of (9 ±21 to 325 ±1) mg kg^{-1} and a range of (11±17 to 213 ±9) mg kg^{-1} was recorded for the 60 cm depths measured. The highest TPH concentration (401 ±3 mg kg^{-1}) in the top soil (15-30 cm) depth was observed in sample No. 6, followed by 325 ±1 mg kg^{-1} for the mid soil of sample No. 9 in site investigated. The greatest depths measured (60 cm) in most of the samples recorded had significantly low value of TPH though concentrations of total petroleum hydrocarbons did not decrease generally with depth.

However, no significant level of TPH was recorded for the control soil samples taken from similar geographical non-spilled areas. The real samples showed elevated concentration of TPHs when compared with control samples in all the sites.

The high levels of TPH contamination observed in this study for spilled soils far exceeded the fifty parts per million (50 $mgkg^{-1}$) compliance baseline limit (DPR. (1991) set for petroleum industries in Nigeria.

PCA used combined concentration and sample-discrete-identity information while related techniques like Principal Component Regression (PCR) and Partial Least Square (PLS) could only limit its quantification to concentration. Assessment of the penetration capability and distribution of the hydrocarbon contaminants were carried out. This aspect of investigation classified the hydrocarbons into groups based on their degree of penetration within the soil strata. Basically, three major groups of petroleum hydrocarbons are known, classified and adopted in this work.

Table 1. Overall average Total Hydrocarbon content (mg kg^{-1}) in the samples and controls.

Sample Location	**No of depths**	**Hydrocarbon Concentration** (mg kg^{-1}) TS	MS	BL
1	3	67±11	103±7	97±12
2	3	**2±5**	**24±8**	**17±7**
3	3	135±12	75±9	53±9
4	3	45±16	86±13	17±8
5	3	94±3	123±13	52±4
6	3	**401±3**	179±7	**213±9**
7	3	116± 2	123±7	89±7
8	3	215±4	141±3	127±3
9	3	138±21	**325±1**	91±15
10	3	143±6	67±17	49±2
11	3	53±8	44±13	172±11
12	3	14±24	9±21	13±19
13	3	134±11	217±5	197±12
14	3	67±9	41±13	11±17
15	3	74 ±9	82±8	123±11
16	3	213±3	75±3	46±8
17	3	79±2	67±12	58±8
18	3	312±9	173±5	101±4
19	3	213 ±2	212±6	193±4
Control samples				
O	3	0.00	0.00	0.00
P	3	0.00	0.00	0.00
Q	3	0.00	0.00	0.00
R	3	0.00	0.00	0.00

TS= Top soil; MS = Mid soil; BL = Bottom level

These are: (i) The Gasoline Range Organics (GRO), generally eluting at window C_5-C_9.(ii) The Diesel Range Organics (DRO) elutes from C_{10}-C_{26} (iii) The Waste Oil Organics (WOO), eluting above C_{26}. The presence and the concentrations of C_5-C_9, C_{10}-C_{26}, C_{26} and above had been identified and computed. The penetration, percentage distribution and migration of these groups of hydrocarbons in the samples were considered. The sample chromatograms representing TS, MS and BL and bar graph representing the percentage penetration is shown side by side. About four distribution and penetration patterns were observed in all the samples as represented below (figures 4-8). The first group has six samples (1, 2, 3, 5, 8 and 11) exhibiting similar spatial penetration and distribution pattern. This group showed both DRO (C_{10}-C_{26}) and WOO (C_{26} and above) with competitive penetration capacity as represented in Figures 4 with DRO having much penetration concentration and no significant presence of GROs in any of the soil levels.

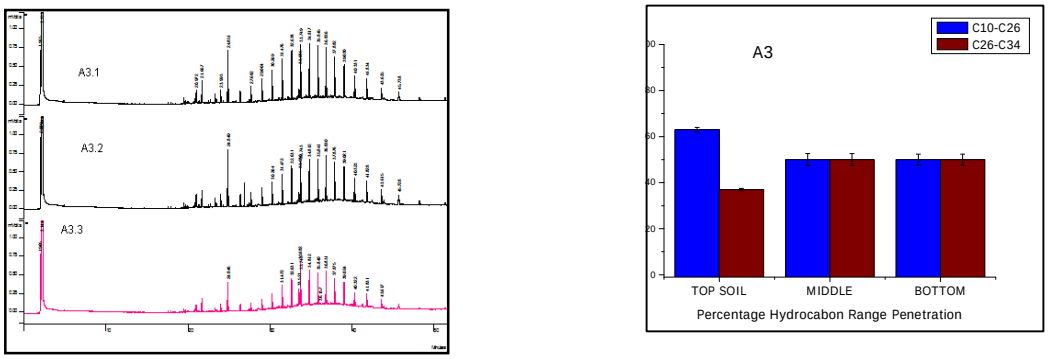

Figure 4. Chromatogram of sample 3 showing DRO and WOO penetration in TS, MS and BL on a bar graph. No presence of GRO within the measured depths.

The second pattern was demonstrated by three (3) sample locations (13, 14 and 15), each comprising of three (3) sampling depths. This group had DRO (100%) as the major contaminant through TS (0-15 cm), MS (15-30 cm) and the greatest depth measured with no significant contribution from Gasoline range organics and Lubricating or waste oil hydrocarbon (WOO) range - C_{26} and above (Figure 5).

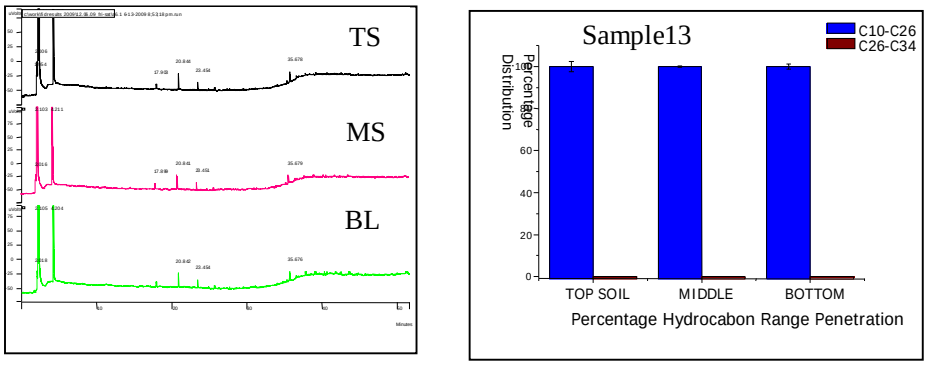

Figure 5. Chromatogram of sample 13 showing DRO penetration in TS, MS and BL on a bar graph no presence of GRO and WOO.

The third depth penetration pattern of the contaminants within the site of investigation had seven samples (6, 7, 9, 10, 12, 16, and 19) in WOO predominates in the soil levels (Figure 6). Representative sample 10 recorded 100% BL and over 80% at the MS level. DRO was not detected in these samples at the BL while its presence at the top and mid soil levels was 18% and 65% respectively.

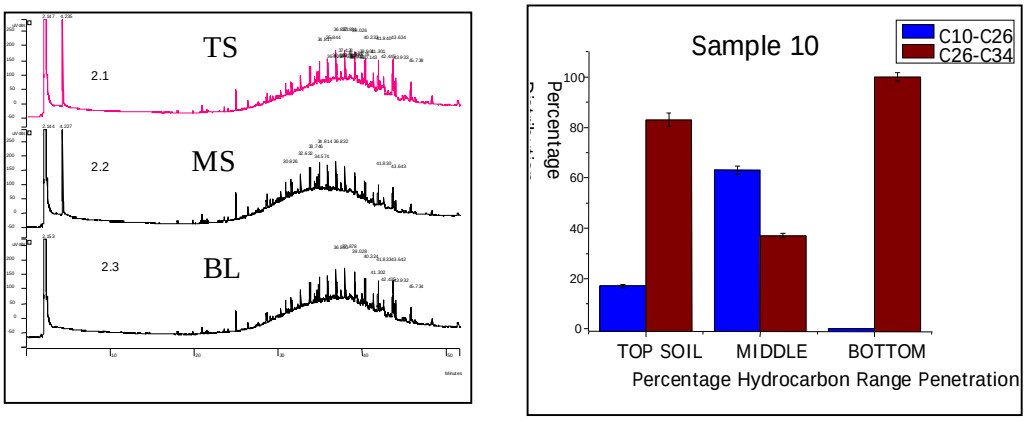

Figure 6. Chromatogram of sample 10 showing DRO and WOO penetration in TS, MS and BL on the bar graph. No significant presence of GRO within the greatest depth (60 cm) measured.

The fourth group of four samples (4, 17, and 18) had the penetration and migration pattern represented by sample 4 (Figure 7). This cluster recorded no significant petroleum hydrocarbon contaminants (GRO, DRO and WOO) at the 60 cm depth (BL) measured. 100% and 85% of DRO was observed at top and mid soil respectively. Only 20% presence of WOO was seen at the MS level.

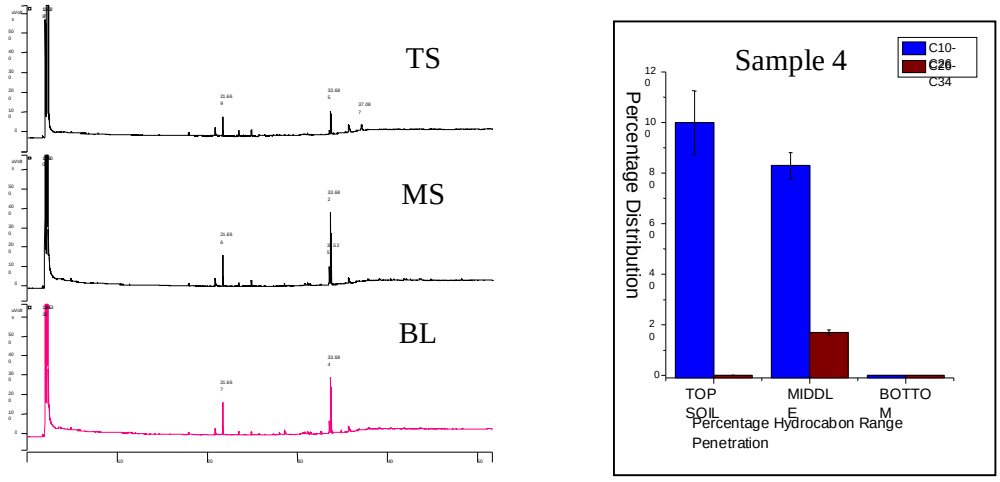

Figure 7. Chromatogram of representative sample 4 showing the dominance of DRO penetration in TS and MS on a bar graph. No significant presence of GRO, DRO and WOO at the greatest depths (60 cm) measured.

The non detection of elevated levels of Gasoline Range organics (GRO), C_5- C_9 in all the sample points could be attributed to their solubility in soil water, atmospheric temperature, type and extent of such contamination (Hokstad et al, 2000) and evaporation (Mohammed 2004) of the light crude oil on exposure for long time before sampling and analysis. This result is consistent with (Craig Adams, et al 2007) that very low concentrations of organic pollutants (gasoline components) were found in soil and water after Katrina.

CONCLUSION

The characteristic assessment of liquid hydrocarbon contaminants in the soil of Niger Delta under the tropical weather conditions was carried out. The concentration range of these contaminants in the spilled site and the type of penetration patterns of the hydrocarbon contamination at different depths at each location were established and recorded. The results obtain in this work revealed that the TPH concentration in the all the levels of soil strata measured ranged from 2±5 to 410±3 mg kg^{-1}. The concentrations and penetration ranges for the four groups of TPHs – C_5-C_9 (Gasoline Range Organics), C_{10}-C_{26} (Diesel Range Organics) and C_{26}-C_{34} (Waste Oil Organics) were recognised.

Despite limited information on the migration and depth penetration of hydrocarbons in soils, data from this study showed the types, distribution, migration and penetration capability of the petroleum hydrocarbon contaminants in the study area.

The spatial distribution and penetration pattern of petroleum contaminants at the investigated site were established as an informative guide to the Government and oil industries during remediation process by competent professionals.

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