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Determination of Polycyclic Aromatic Hydrocarbons and Total Petroleum Hydrocarbons in Crude Polluted Soil from Esaba, Ughelli South, Delta Nigeria

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ABSTRACT

The current research examined the levels and composition of Polycyclic Aromatic Hydrocarbons (PAHs) and Total Petroleum Hydrocarbon (TPH) at six locations affected by crude oil spills (E1, E2, E3, E4, E5, and E6) within the Esaba community in the Niger Delta region, which is notable for petroleum exploration. The samples were assessed for the 16 priority PAHs recognized by the USEPA utilizing GC/FID analysis. The concentrations of Σ16 PAHs and TPH in the soil ranged from 19.322 to 83.542mg/kg with (average of 45.562mg/kg) and 890.908 to 4393.094mg/kg (average of 2622.097mg/kg) respectively. The concentrations of Σ16PAHs US-EPA and TPH in all the studied locations far exceeded the safety value of 10mg/kg and 100mg/kg respectively, set by the soil quality guidelines of Switzerland and above which is regarded as being highly contaminated for Agricultural soils of Poland. The mean PAHs concentrations recorded in E1, E2 and E3 studied locations were significantly greater that the permissible limit of 40mg/kg set by Department of Petroleum Resources (DPR) for oil spill sites. The result from this study showed higher distribution of low Molecular Weight (LMW) PAHs than high Molecular Weight (HMW) PAHs indicating possible petrogenic source. The research found that Site E2 had highest level of PAHs than the other sampled locations. It is recommended that immediate intervention not only at studied locations of E1, E2 E3 but also E4 be carried out given that all individual PAHs in E4 are known carcinogens.

Keywords: Polycycic Aromatic Hydrocarbon, Total Petroluem Hydrocarbon, Aliphatic Esaba, soil

INTRODUCTION

In Nigeria, the Niger Delta is well-known for its severe environmental pollution resulting from crude oil exploration and production operations. The extensive processes of extraction, transportation, and utilization of crude oil have increased the likelihood of unintentional oil spills, which damage both terrestrial and aquatic ecosystems and present significant risks to the health of humans and animals (Saadoun, 2015). Between 2008 and 2018, a total of 7,581 oil spill incidents were documented on the Nigerian Oil Spill Monitor (NOSDRA) website, all occurring within the Niger Delta region. According to the data from the NOSDRA online oil spill monitor, approximately 418,414.57 barrels of crude oil were spilled during the 2008 to 2018-time frame, although 31.66% of the total incidents (2,400 incidents) had unavailable information on the volume of oil spilled.

The natural environment affected by oil spills is extremely challenging to clean up, particularly in marshes and mangroves (Wali *et al*., 2019). Oil spills that lead to contamination of environmental elements such as soil, water, and air have been associated with various health issues for local residents, including respiratory problems like cancer, skin conditions, and digestive disorders (Kuppusamy *et al.*, 2020; Onyena and Sam, 2020). If the spill contains a significant quantity of light aromatic hydrocarbons, it can cause toxic effects such as plant asphyxiation and organism fatalities (Linden and Jonas, 2013).

The primary constituents of crude oil are hydrocarbons, which are categorized into saturated, unsaturated, and

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polycyclic aromatic hydrocarbons; derivatives of petroleum products fall into two major classifications: aliphatics and aromatics (Mostert *et al*., 2010). The aliphatic compounds found in petroleum hydrocarbons are predominantly alkanes that can be either straight-chain or branched (Militon *et al*., 2010; Khan *et al*., 2018). Aromatic compounds vary from those containing a single benzene ring to complex substances formed by several fused rings, including polycyclic aromatic hydrocarbons (PAHs) (Khan *et al*., 2018), which are recognized as significant hazardous environmental pollutants resulting from oil spills (Dudhagara *et al*., 2016).

Total Petroleum Hydrocarbons (TPHs) primarily consist of both aliphatic and aromatic components (Khan *et al*., 2018). They are frequently introduced into the environment through unintentional spills and leaks during their transportation or storage. When such leaks happen, TPHs typically accumulate in the top layers of the soil (Varjani, 2017), where they impact physical characteristics like pH, nutrient bioavailability, and biodiversity (Varjani, 2017; Devatha *et al*., 2019).

Total petroleum hydrocarbons (TPH) are the primary components of crude oil, making them one of the most prevalent pollutants in soils contaminated by oil. When hydrocarbon pollution persists over an extended period, the compounds become tightly bound, resulting in the predominance of a recalcitrant fraction of hydrocarbons that are not easily bioavailable (Semple *et al*., 2007). While it may be seen as less toxic than an area that has been recently contaminated, the impact of lasting compounds in a chronically contaminated location is significant (Jonker *et al*., 2006). As different petroleum components diminish due to weathering, polycyclic aromatic hydrocarbons (PAHs) remain trapped within the soil matrix, posing substantial health risks to both humans and the environment.

Polycyclic aromatic hydrocarbons (PAHs) comprise a group of thousands of hazardous and widespread organic pollutants present in the environment. These compounds are naturally occurring, unsubstituted organic molecules made up of two or more fused benzene rings arranged in linear, angular, or clustered formations, typically found as complex mixtures instead of isolated substances (Lee and Vu, 2010). They pose health risks to humans (Fetzer, 2000; Tiwari *et al*., 2015, 2017). PAHs are recognized as persistent organic pollutants, categorized as organic micro-pollutants that are notably resistant to biodegradation and have detrimental effects on the environment (Boisa *et al*., 2019; Gao *et al*., 2019). Monitoring of these substances in the environment began over four decades ago, highlighted by a list released by the U.S. Environmental Protection Agency (EPA) in 1976 (Keith and Telliard, 1979; Keith, 2015).

Physically, polycyclic aromatic hydrocarbons are primarily characterized as colorless, white, or pale-yellow solids, exhibiting a range of boiling and melting points (Abdel-Shafy and Mansour, 2015). They are typically categorized based on their melting and boiling points, vapor pressure, and solubility, which are influenced by their structural composition. PAHs possess low vapor pressure, minimal solubility in water, sensitivity to light, resistance to heat, thermal conductivity, emission capabilities, and corrosion resistance. They are also highly lipophilic and exhibit physiological effects (Akyuz and Cabuk, 2010). In aquatic environments or when adsorbed onto particulate matter, PAHs may undergo photodecomposition when exposed to ultraviolet light from solar radiation. In the atmosphere, PAHs can react with ozone, nitrogen oxides, and sulfur dioxide, resulting in the formation of diones, nitro- and dinitro-PAHs, and sulfonic acids, respectively (WHO, 1987; ATSDR, 1994).

The existence of polycyclic aromatic hydrocarbons (PAHs) in crude oil is well documented. As noted by Gao *et al*. (2019), these compounds are regarded as the primary toxic constituents of crude oil, with many identified as carcinogenic or mutagenic. They have been classified as priority pollutants by both the European Union and the United States Environmental Protection Agency (US EPA). PAHs can be categorized into two principal groups: Light Molecular Weight PAHs (LMW), which consist of two or three aromatic rings and include compounds such as naphthalene, anthracene, fluorene, acenaphthene, and phenanthrene, are recognized for their acute toxicity. In contrast, High Molecular Weight PAHs (HMW), characterized by four or more rings and comprising substances like chrysene, pyrene, benzo(a)pyrene, and fluoranthene, are predominantly viewed as genotoxic (ATSDR, 1995; Ghosal *et al.*, 2016). Boisa *et al*. (2019) highlighted that HMW are particularly concerning due to their stability, high toxicity, and lipophilic nature, which facilitates their bioaccumulation in biological tissues. Furthermore, according to the Comprehensive Environmental Response, Compensation and

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Liability Act (CERCLA), PAHs were ranked 7th in 2005 in the biennial assessment of hazardous substances that pose the most significant risk to human health (Christopher, 2008).

Low-molecular-weight (LMW) polycyclic aromatic hydrocarbons present in contaminated soils exhibit a half-life ranging from 5 to 7 years, whereas high-molecular-weight (HMW) PAHs have a half-life of 9 to 10 years (Wild *et al*., 1991). The removal of PAHs from contaminated soils is essential due to the negative health impacts on humans linked to their ingestion (Haritash and Kaushik, 2009).

Polycyclic aromatic hydrocarbons (PAHs) in soil can become encapsulated within minerals and exist in non-aqueous liquids, which may pose a risk of human exposure (Tarafdar *et al*., 2018). These compounds can adversely affect soil functions and disrupt the soil microbiome due to their persistence and resistance to natural degradation processes (Zhao *et al*., 2021; Roslund *et al*., 2018). The ubiquitous presence of PAHs has sparked research into various remediation methods (Gan *et al*., 2009). Therefore, there is an immediate necessity for effective and environmentally friendly strategies to alleviate the detrimental effects of oil spills on the ecosystem. This study focused on assessing the concentration and distribution of total petroleum hydrocarbons (TPH) and PAHs in contaminated soil samples from the Esaba community in Delta State.

MATERIALS AND METHODS

Description of the Study Area

The host communities are located in the Ughelli south local government of Delta state. Esaba is located in the west of the region of Ughelli South, its situated near to Otutuama village as well as Okwagbe town. It has a tropical monsoon climate It is located on the Bight of Benin, with a shoreline spanning about 60 km with latitudes of 5° 24′ 52″ N and 5° 46′ 49″ E. The residents of Esaba primarily engage in farming and fishing, and the community is home to a Shell flow station. Unfortunately, the agricultural lands in this area have been adversely affected by crude oil pollution resulting from years of oil bunkering, illegal refineries activities, and environmental contamination.

Sampling collection

Six soil samples were collected from each sub-area of Esaba (E1, E2, E3, E4, E5 and E6) and homogenized into a composite sample. The samples were collected with an improvised soil augur (nitric acid sterilized PVC pipes) A total of eighteen batch soil samples made into six different composite samples were collected at depth of 0-15 cm, after the removal of the exposed surface.

A thin layer of the composite samples was sieved (212-μm) to remove fragments of plants debris and stone. The screened samples were thereafter air-dried in the dark. The dry soil samples were then ground into powder in a ceramic mortar and demagnetized with a magnetic rod and kept in sealed vials labeled. Soil samples were taken to the laboratory for analysis

Methods Soil Extraction

A solvent mixture consisting of acetone and methylene chloride in a 50:50 ratio was prepared. A 10g aliquot of the thoroughly mixed sample was transferred into a beaker that had been rinsed with solvent, followed by the addition of 50ml of the solvent mixture to the sample. Subsequently, 1ml of the surrogate mixture was introduced. The sample was then placed in a sonicator and subjected to sonication for approximately 10 to 15 minutes at around 70°C. Anhydrous sodium sulfate, weighing 10g, was added to the sample until a clear extract was obtained. The extract solvent was then transferred into a round-bottom flask. This process was repeated with an additional 50ml of the solvent mixture, and the beaker was allowed to settle before decanting into the same round-bottom flask. Finally, the solvent was concentrated to a volume of approximately 1 to 3ml. The sample was subsequently prepared for purification utilizing a silica gel column. The columns were filled with 10 grams of 100-200 mesh silica gel, which had been pre-conditioned by baking at 105°C overnight. The silica was combined with dichloromethane to create a slurry. Column chromatography was performed to separate the aliphatic hydrocarbons and polycyclic aromatic hydrocarbons (PAHs) fractions through successive

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elution with 20 mL of n-hexane followed by 70 mL of a n-hexane/dichloromethane (7:3 v/v) mixture. The PAH fraction was then concentrated using a rotary evaporator at 30°C to approximately 1 mL, transferred to a 1.5 mL vial, and reduced to 0.5 mL under a gentle nitrogen stream. The analysis was conducted using gas chromatography with a Flame Ionization Detector (GC/FID).

Statistical Analysis

Descriptive (mean and standard deviation) statistical analysis was used to present data in numerical forms

RESULTS

Table 1: Polyclclic Aromatic hydrocarbon concentration of the different sampled sites

PAHs component (mg/kg)	**E1**	**E2**	**E3**	**E4**	**E5**	**E6**	**∑ sum of the total PAH COMPONENT**	**Mean of the ∑PAH component**
Naphthalene	10.094	14.098	-	-	-	0.605	**24.797**	8.265
Acanaphthalene	13.358	22.826	-	-	2.323	0.861	**39.368**	9.842
Acenaphthene	-	9.525	-	-	-	-	**9.525**	9.525
Florene	17.602	9.147	6.021	-	18.777	1.193	**52.740**	10.548
Phenathrene	5.351	14.119	42.547	-	2.632	0.957	**65.606**	13.121
Anthracene	8.311	2.745	-	-	13.889	2.957	**26.908**	6.727
Fluoranthene	-	-	4.112	-	-	1.667	**5.776**	2.888
Pyrene	-	-	-	-	-	17.248	**17.248**	17.248
Benzo(a) anthracene	-	5.895	-	2.203	-	-	**8.098**	4.049
Crysene	-	5.182	-	3.860	-	-	**9.052**	4.526
Benzo(b) fluoranthrene	-	-	-	4.762	-	-	**4.762**	4.762
Benzo(a) pyrene	-	-	-	4.553	-	-	**4.553**	4,553
Benzo(k) fluoranthracene	-	-	-	3.944	-	-	**3.944**	3.944
Indeno(123)perylene	-	-	-	-	-	-		Mean of the ∑PAH component
Dibenzo(a,h) anthracene	-	-	-	-	-	-		
Benzo (g,h,i) perylene	-	-	-	-	-	-		

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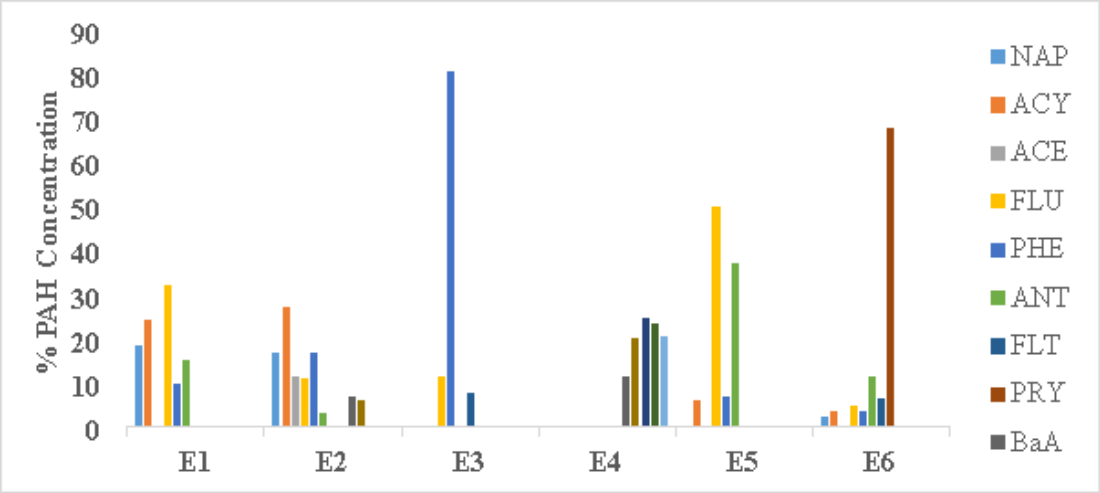
Total (mg/kg)	**54.716**	**83.542**	**52.680**	**19.322**	**37.620**	**25.491**		

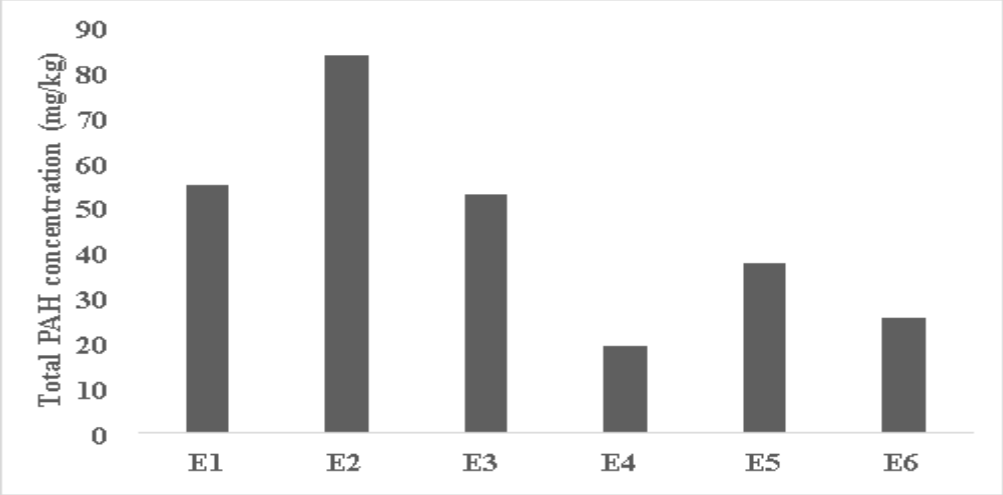
E1, E2, E3, E4, E5 and E6 are the different sampled locations at Esaba,

(-) = below detectable limit



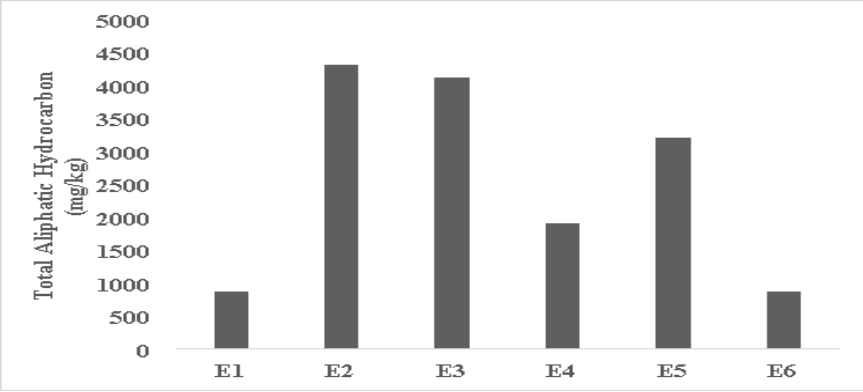
E1, E2, E3, E4, E5 and E6 are the different sampled locations at Esaba

Figure 1: % of Polycyclic Aromatic Hydrocarbon in the different sampled sites



E1, E2, E3, E4, E5 and E6 are the different sample locations at Esaba

Figure 2: Mean Total Polycylic Aromatic Hydrocarbon Concentrations of the different sampled sites



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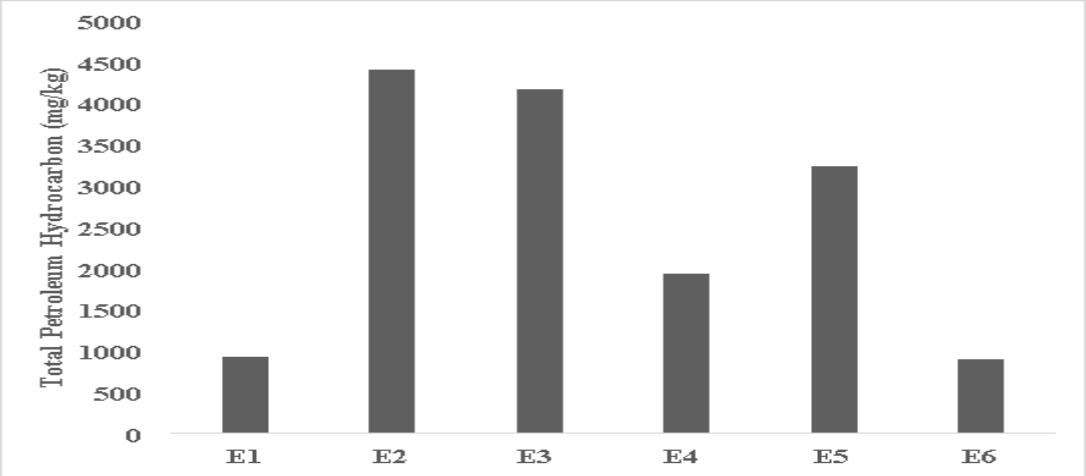
E1, E2, E3, E4, E5 and E6 are the different sampled locations at Esaba

Figure: 3 Total Aliphatic Hydrocarbon of the different sampled sites



E1, E2, E3, E4, E5 and E6 are the different sampled locations at Esaba

Figure 4: Total Petroleum Hydrocarbon concentration of the different sampled sites

PAHs contamination of Agricultural soil by the exploration of crude oil and oil spill in Niger Delta region has been reported by many researchers, but study on PAHs contamination in Esaba crude oil community of Ughelli South Local Government of Delta State remain unavailable and that brought about this study. The analysis of the PAHs content and TPH on crude oil contaminated soil from Esaba at different sampled locations showed various contamination level and distribution of PAHs component and TPHs. From our result the concentrations of the sixteen PAHs identified by the United State Environmental Protection Agency (USEPA) as priority pollutants investigated in our study as shown in table1, indicated the presence of thirteen PAHs in all the sampled locations expect for indeno (123) pyrene, dibenzo (h g), anthracene and benzo (ghi) perylene that were below detection limit of the analytical equipment used in all the sampled points. The order of increase of total individual PAHs concentration is as follows: phenathrene > florene > acenaphthalene > anthracene > naphthalene > pyrene > acenaphthene > chrysene > benz (a) anthracene > fluoranthene > benzo (b) fluoranthen > benzo (a) pyrene > Benzo (k) fluoranthene with respective concentrations in mg/kg as (65.606, 52.740, 39.368, 26.908, 24.797, 17.248, 9.525, 9.052, 8.098, 5.776, 4.762, 4.553 and 3.944).

For the source identification and distribution of the PAHs, our result showed that both petrogenic and pyrogenic (that is the LMW and the HMW PAHs) sources were identified in all sampled locations studied. Soil that are polluted by petrogenic (oil spillage contamination) sources tends to have higher percentage of LMW PAHs in their PAHs composition while soil polluted by pyrogenic (coal and fossil fuel combustion) sources tend to have higher percentage of HMW PAHs in their PAHs composition (Wang *et al.,* 1999: Moore *et al.,*2015). The result from this study showed that all the study sampled locations had higher concentration of LMW PAHs indicating the possible contaminants of the surface soil sampled originated from petrogenic sources expect for E4 that only showed HMW PHAs concentrations. The LMW PAHs are very unstable and tends to evaporate in the soil when in contact or exposed to sunlight over time, and are highly violate (Sanches *et al*., 2011; Ukiwe *et al*., 2013). The evidence for the LMW PAHs in most of the sampled is due to the fact that there are ongoing crude oil activities on these sites resulting in continuous oil spillage.

The PAHs mean concentrations for surface soil exceeded the DPR Intervention limit of 40mg/kg for some of the sampled locations (E1, E2 and E3) while the other sampled locations (E4, E5 and E6) were below DPR intervention. In our study E2 sample location had the highest total PAHs concentration, while E4 had the lowest as shown in figure 2.

However the total PAH concentrations in all the sampled locations (54.716, 83.542, 52.680, 19.322, 37.062 and 25.491mg/kg for E1, E2, E3, E4, E5 and E6 respectively) were far higher than when compared with the

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maximum background limits of 15mg/kg for polluted soils set by Dutch Environment Ministries and E1, E2 E3 sampled points were higher than 50mg/kg set by Polish Environment Ministries in polluted soil , (Polish Environment Ministry, 2002). Also, the concentrations of Σ16PAHs in all sampled locations exceeded the precautionary value of 1000 ng/g 10mg/kg set by the soil quality guidelines of Switzerland (Dasaules *et al*., 2008. Based on these classifications, sampled soil from Esaba could be classified as heavily contaminated with PAHs

Naphthalene concentration in the soil ranged from 10.094 to 0.603mg/kg with an average value of 8.266 mg/kg. A lower naphthalene concentration was reported by (Ayedun *et al*., 2024) in a crude oil contaminated soil. Also, Al-Sad *et al.* (2019) reported a higher naphthalene concentration. Acenaphthylene ranged from 22.86 to 0.861 mg/kg with an average value of 39.368 mg/kg. Acenaphthene was only detected in E2 soil sample. Fluorene ranged from 18.777 to 1.193 mg/kg with an average value of 10. 548mg/kg. Phenanthrene ranged from 42.547 to 0.957 mg/kg with an average value of 13.121 mg/kg. Anthracene ranged from 13.889 to 2.748 mg/kg with an average value of 6,727 mg/kg. Floranthene was detected in E3 and E6 with the value of 4.112 and 1.6657 mg/kg respectively. Pyrene was only detected in E6 with the value of 17.248 mg/kg. Chrysene and Benz(a)anthracene was detected only in E2 and E4 with 5.895 and 5.182 for Chrysene and 2.203 and 3.860mg/kg for Benzo(a) anthracene respectively.

PAH components detected in E2 and E4 falls within the category of PAHs with the highest health risk especially at prolonged exposure (ATSDR, 1999). Although E4 having the lowest total PAHs concentration of 19.322 mg/kg when compared to other sampled locations, but had five PAHs that are described as carcinogens according to the USEPA (California Environmental Protection Agency, 1994) namely benzo(a)anthracene, chrysene, benzo(b)fluoranthrene, benzo(a) pyrene and benzo (k) fluoranthrene. These PAHs pose a major threat in terms of its mutagenic and carcinogenic effects. Some studies have also shown that some of these PAHs can induce dioxin-like activity and weakened estrogenic responses (Villeneuve *et al.*, 2002).

Dudrikova *et al*. (2023) reported presence of naphthalene, floranthene, pyrene, chrysene, and benz(a)anthracene in natural and anthropogenically transformed coastal soils of Southern Russia. Lower than our present study is the report of Barrán-Berdón *at al.* (2012) who reported a total PAHs concentrations range of 0.0007mg/kg to 1.384 mg / kg with an average PAHs concentration of 0.22 mg/Kg in soil samples. They also noted that Naphthalene is more abundant followed by Fluorene, Chrysene, Benzo [a] Anthracene and Dibenzo [a, h] Anthracene in their study. Also, when compared to this present study, Ayedun *et al.* (2024) reported lower concentration of PAHs in crude oil contaminated soil. Aoeed *et al.* (2023) reported a higher total PAHs concentration of 609.77 ug/kg in soil from oil company visinity, with naphthalene Fluorene and Acenaphthylene showing high concentrations in all the seasoned. Abundant value of fluorene, followed by acenaphthylene and naphthlene was reported by Abed *et al.*, 2015 at North Biji City in Iraq, the total concentration of 16 polycyclic aromatic hydrocarbons ranged from 49.9 to 986.4 µg / kg and average of 587.2 µg / kg, which is also lower than our present study

Comparing our present result with other study from Niger Delta region, Faboya *et al*. (2023) reported a total PAHs range of 24330.68 - 40845.32 with average of 299523.47ng/g and 7361.66 -14141.49ng/g with average of 9819.96 ng/g. Also, Aedosu *et al*. (2013) have previously reported PAHs concentrations ranged of 23.8 to 120 and 7.40–78.3 ng/g, respectively in polluted soil from Niger Delta region. All this reports from Niger Delta regions where all below the concentration revealed in our present study, as such DPR has to intervene, and help reduce the level and future occurrence.

Total Petroleum Hydrocarbons (TPHs) are mainly composed by aliphatic and aromatic fractions (Khan *et al*., 2018). The concentrations of aliphatic hydrocarbons present in crude oil contaminated soil is presented in figure 3, the aliphatic hydrocarbons ranging from C8 to C40 range were detected in sampled locations as 872.969, 4309.574, 1907.506, 3194.193 and 865.417mg/kg for E1, E2, E3, E4, E5 and E6 respectively. Similar to our study is Martinez-Cuesta *et al.* (2023), that reported aliphatic value of 3944.4 at the initial concentration in polluted soil before ecopilcs remediation. The TPH concentration present in the contaminated soil samples were 927.685mg/kg for location E1, 4393.094 mg/kg for location E2, 4362.254mg/kg for location E3, 1926.828 mg/kg for location E4, 3231.813 mg/kg for location E5 and 890.908 mg/kg for location E6 as shown

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in Figure 4. The total petroleum hydrocarbons obtained at the six sampled locations in this study were all below the Department of Petroleum Resources (DPR) limit of 5000 mg/L.

Concentration of TPH higher than 10–100 mg/kg, indicates pollution (Adeniyi and Afolabi 2002), from above it can be said that all the soil samples collected at the polluted sites are polluted with THP. Akagbue *et al*., (2024) reported a high TPH concentration of 7829.23ppm in crude oil polluted soil from Souther Ijaw. Their concentration is far higher when compared with our present study and it could attribute to higher crude oil contamination level.

CONCLUSION

The high concentration and abundance of LMW PAHs gave evidence that the nature of the contamination was mainly from crude oil. The results obtained from this study reveals that the level of total petroleum hydrocarbons (TPH) obtained from all the samples were lower than the maximum recommended levels by the Department of Petroleum Resources (DPR). while the concentration of total PAHs in most of the sampled locations were higher than the maximum recommended level for DPR. Even at low concentration of PAHs it can exert some harmful effect on human, as such this study recommends remediation measures by the government given that PAHs in some of sampled locations were above the DPR limit for intervention. Though some of the PAHs and TPH level were below intervention limit.

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