

## Assessment of TPH, PAH, Some Inorganic Anions and Heavy Metal Concentration of Arable Soil Around Crude Oil Spilled Sites in Rivers State, Nigeria

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**Abstract:** Oil spillage has always been a major source of pollution which releases harmful substances into the environment. The concern over pollution of farmland has constituted environmental issues over the year. Most remediation effort is aimed towards reducing hydrocarbon content of soil at the point of impact. In this study, the concentration of TPH, PAH, some inorganic anions and heavy metals (cadmium, nickel, zinc, lead) were determined in soil collected from farm land located around the point of impact of crude oil. The soil TPH ranged from  $123.92 \pm 3.26$  to  $934.79 \pm 5.26$  mg kg<sup>-1</sup>. The soil PAH ranged from  $12.18 \pm 1.10$  to  $86.21 \pm 1.80$  mg kg<sup>-1</sup>, chloride concentration ranged from  $24.70 \pm 0.00$  to  $399.37 \pm 5.77$  ppm in surface soil while the subsoil concentration ranged from  $29.37 \pm 5.77$  to  $221.03 \pm 14.43$  ppm, nitrate concentration ranged from  $7.40 \pm 0.36$  to  $59.67 \pm 1.53$  ppm in surface soil while the subsoil concentration ranged from  $10.00 \pm 0.01$  to  $43.33 \pm 1.53$  ppm, sulphate concentration ranged from  $16.00 \pm 0.00$  to  $160.00 \pm 10.00$  ppm in surface soil while the subsoil concentration ranged from  $16.00 \pm 1.00$  to  $126.67 \pm 5.77$  ppm, phosphate concentration ranged from  $16.00 \pm 0.00$  to  $28.57 \pm 0.12$  to  $393.33 \pm 3.06$  ppm in surface soil while the subsoil concentration ranged from  $14.37 \pm 0.15$  to  $282.33 \pm 2.08$  ppm, cadmium concentration ranged from  $0.24$ - $0.47$  ppm in surface soil while the subsoil concentration ranged from  $0.23$ - $0.35$  ppm, nickel concentration ranged from  $2.20$ - $2.97$  ppm in surface soil while the subsoil concentration ranged from  $1.07$ - $1.98$  ppm. Zinc concentration ranged from  $11.58$ - $24.78$  ppm in surface soil while the subsoil concentration ranged from  $6.00$ - $24.36$  ppm. Lead concentration ranged from  $0.61$ - $4.25$  ppm in surface soil while the subsoil concentration ranged from  $0.41$ - $3.73$  ppm. The study, therefore showed that the primary method of remediation by petroleum industries in Rivers State may achieve environmental partial clean-up at the oil-impacted site but contaminate a much wider area away from the oil-impacted site.

**Key words:** Crude oil, spillage, TPH, PAH, anion, heavy metals

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### INTRODUCTION

The environment of the Niger Delta areas of Nigeria has been disturbed by petroleum exploration and exploitation, refining and other oil related industrial activities which have led to a wide scale pollution of its arable land (Odokuma and Okpokwasili, 1992).

Some of the hydrocarbons from crude oil and related materials are very complex mixtures of several thousand of hydrocarbons and other indeterminate structures and additives (Okieimen *et al.*, 2011), the presence of which adversely affects the quality of soil (NCF, 2006).

Over 6,000 spills have been recorded in the 40 years of oil exploitation in Nigeria with an average of 150 spills per annum (DPR, 1997), it is recorded that terrestrial ecosystem are affected by the interaction of factors from natural or anthropogenic origin. Both of them

have direct or indirect impact on the environment causing deterioration of soil (Bradley *et al.*, 1994).

The entire Niger Delta is crisscrossed by thousands of kilometers of pipelines, punctuated by wells and flow stations. Poor maintenance of oil infrastructure equipment, sabotage of oil infrastructures, theft of oil and illegal refining all contribute to oil pollution in Rivers State (Anonymous, 2006).

One of the greatest problems associated with oil pollution is the constant exposure to high concentration of heavy metals from oil. Also inorganic anionic pollutants, such as chlorides, phosphates, nitrates and sulphates are found to be greater in soil disturbed by oil drillings and pollution (Powell and Florence, 1993).

When petroleum is spilled, its components undergoes various physical and chemical modification, evaporation, biodegradation and photo oxidation are

among the major factors contributing to the removal of the hydrocarbon molecules from the geosphere (Douglas *et al.*, 1996; Garrett *et al.*, 1998). As the hydrocarbons are consumed, less complex hydrocarbons are formed. When the process is complete, the remaining waste or by-product are mainly carbon dioxide, water, fatty acid and paraffin. However, these processes are very slow to bring about non-toxic and immobile hydrocarbon (Apajalahti and Salkinoja-Salonen, 1986). This process can be so slow that hydrocarbons may remain in the soil enough time for heavy metals, inorganic anion pollutants and other contaminants are run off into nearby farms. In this research, researchers assessed the concentration of TPH, PAH, some inorganic anions and heavy metals in arable soil of farm lands located around crude oil spilled sites in Rivers State, Nigeria.

## MATERIALS AND METHODS

**Soil sample collection:** The soil samples were collected from farmland around previously oil spilled sited in selected oil producing communities in Rivers State. These communities include Rukpokwu, Bomu, Ogale, Umusoya and Omagwa while selected non oil producing communities in Imo State viz, Owerri, Mgbidi, Orlu, Mbaise and orlu were used as control.

**Determination of Total Petroleum Hydrocarbon (TPH) and Polyaromatic Hydrocarbon (PAH) by gas chromatograph:** The soil samples were cleaned of roots, thoroughly mixed, 2 g of each soil sample was weighed into a clean extraction container. Then, 10 mL of extraction solvent (pentane) was added into each sample, mixed thoroughly and allowed to settle. The mixtures were carefully filtered into a clean solvent extraction vessel, using filter paper fitted into buchner funnels. The extracts were concentrated to 2 mL and then transferred for cleanup/separation. The concentrated aliphatic fractions were transferred into labeled vials with teflon caps for Gas Chromatograph (GC) analysis. About 1.0 mL of the concentrated sample was injected by means of hypodermic syringe through a rubber septum into the column. Separation occurs as the vapour constituent partition between the gas and the liquid phases.

The sample was automatically detected as it emerges from the column (at a constant flow rate) by the detector whose response is dependent upon the composition of the vapour.

**Determination of heavy metals by Atomic Absorption Spectrophotometer (AAS):** The soil samples were air-dried, ground and sieved through 2 mm Nylon sieve. The soil samples were pre-treated using acid digestion. Here, 20 mL of 1.0 M HCL was added to 5.0 g of air-dried

and sieved soil samples in 100 mL Teflon beakers. The content of the beakers were digested by heating vigorously to near dryness. The acid digest was filtered into 50 mL measuring cylinders via a filter paper-laced filter funnel. The residue was rinsed thoroughly and the volume of the filtrate made up to 50 mL using distilled water. The resulting solutions were then aspirated into the atomic absorption spectrophotometer for determination of heavy metals of interest present in the soil extracts. The spectrophotometer (GBC AVANTA A6600) was standardized using the standard solution of the element being analyzed.

### **Determination of inorganic anion in soil samples using reagent spectrophotometer powder pillows**

**Sample pre-treatment:** Soil samples were prepared by weighing 50 g of air-dried soil (passed through a 2 mm sieve) into a 100 mL beaker. About 100 mL of distilled water was added and allowed to stand for 30 min to 1 h and stirred occasionally with a glass rod. The solution of extract was filtered through a filter paper laced filter funnel into a receiving container and then samples are ready for analysis.

**Nitrate (NO<sub>3</sub>):** The method used in analysing nitrate in the samples is Nitrate/DR890 colorimeter method using Nitra ver 5 Reagent Powder Pillow (RPP)/cadmium reduction and the procedure is as follows: Nitrate in the samples was analysed by the colorimetric method using the Nitrate/DR890 colorimeter and Nitra ver 5 Reagent Powder Pillow (RPP)/cadmium reduction procedures.

**Sulphate (SO<sub>4</sub>):** This was determined using the sulphate direct reading method the procedure is as follows: The store program number for sulphate was entered. A sample cell was filled with 25 mL of sample, the content of 1 sulfa ver 4 Reagent Powder Pillow (RPP) was added and capped and inverted several times to mix. After the timer beeped a second sample cell was filled with 25 mL of sample and this serves as the blank and this blank was placed and the instrument was turned to zero and within 5 min after the timer beeped the sample was placed and the reading taken.

**Phosphate (PO<sub>4</sub>):** Phosphate contained in the sample was measured using its programme number with the sample cell filled with 25 mL of sample. The content of 1 phos ver 3 Reagent Powder Pillow (RPP) was added and another sample cell was filled with 25 mL of sample which serves as blank and set too zero and the reading was taken.

**Chloride (Cl):** Chloride in the samples was determined using the Argentometric method as described in American Public Health Association in 1992. About 10 mL of the

sample was titrated against silver nitrate solution using potassium chromate solution as indicator until a pinkish yellow endpoint was achieved.

**RESULTS AND DISCUSSION**

Figure 1 showed the result of soil total petroleum hydrocarbon from polluted and non-polluted areas. The TPH ranged from 123.92±3.26 to 934.79±5.26 mg kg<sup>-1</sup> in polluted areas to 2.40±0.01 to 8.59±0.13 mg kg<sup>-1</sup> in the non-polluted areas.

Figure 2 showed the result of soil polycyclic aromatic hydrocarbon from polluted and non-polluted areas. The

PAH ranged from 12.18±1.10 to 86.21±1.80 mg kg<sup>-1</sup> in polluted areas while in the non-polluted areas PAH was below detectable level.

Figure 3 showed the result of chloride concentration of surface and subsoil from polluted areas. The chloride concentration ranged from 24.70±0.00 to 399.37±5.77 ppm in surface soil while the subsoil concentration ranged from 29.37±5.77 to 221.03±14.43 ppm.

Figure 4 showed the result of nitrate concentration of surface and subsoil from polluted areas. The nitrate concentration ranged from 7.40±0.36 to 59.67±1.53 ppm in surface soil while the subsoil concentration ranged from 10.00±0.01 to 43.33±1.53 ppm.

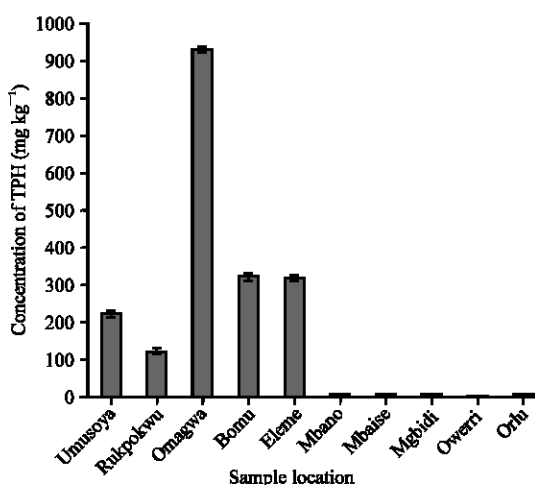


Fig. 1: Concentration of total petroleum hydrocarbon in polluted and non-polluted soil

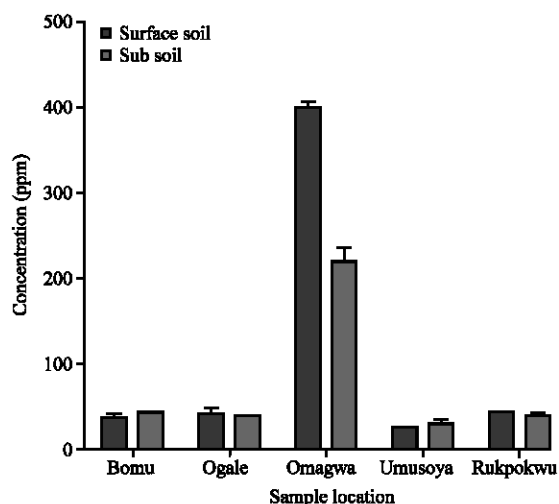


Fig. 3: Chloride concentrations (ppm) of surface and sub soil samples from polluted areas

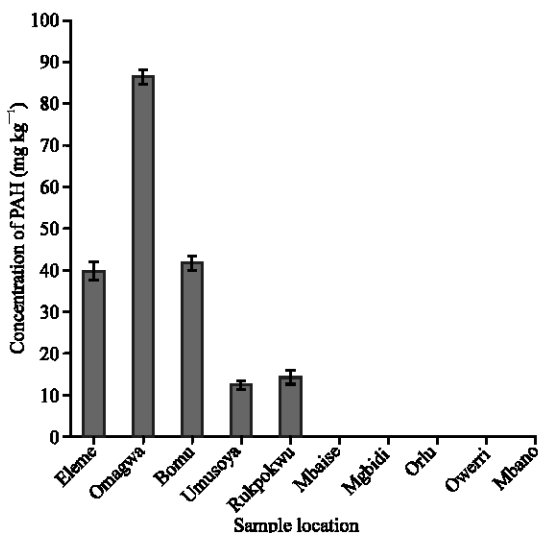


Fig. 2: Concentration polycyclic aromatic hydrocarbon in polluted and non-polluted soil

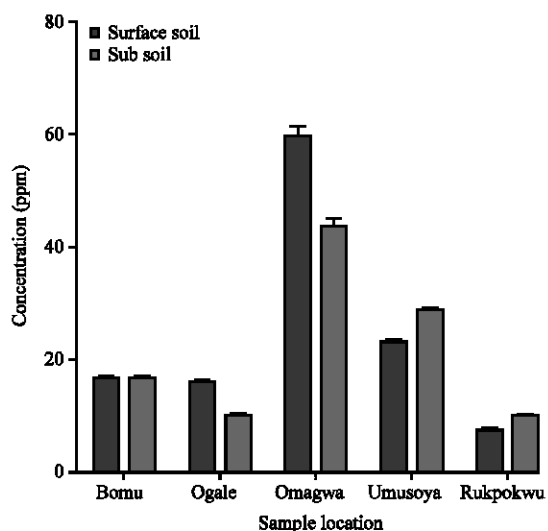


Fig. 4: Nitrate concentrations (ppm) of surface and sub soil samples from polluted areas

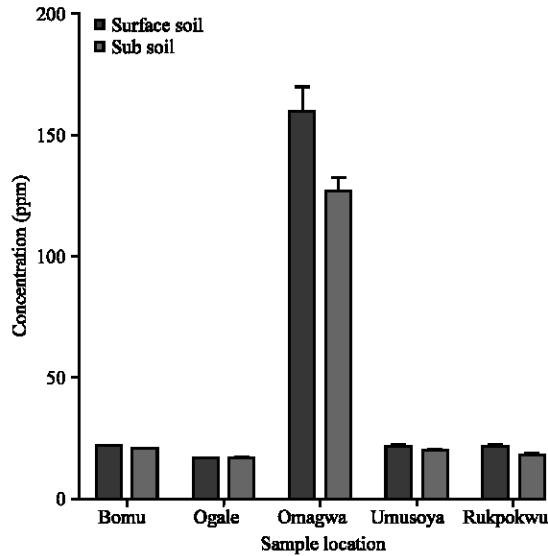


Fig. 5: Sulphate concentrations (ppm) of surface and sub soil samples from areas

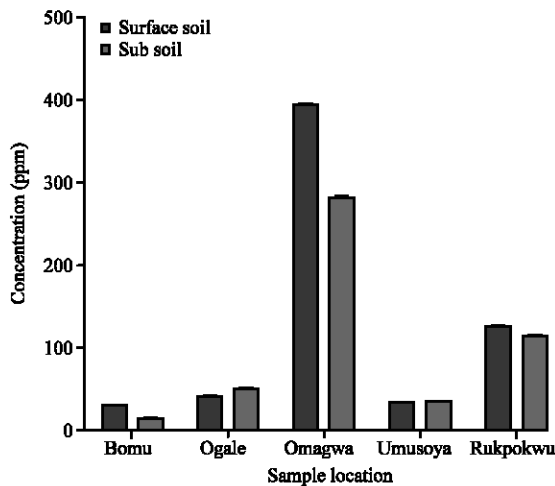


Fig. 6: Phosphate concentrations (ppm) of surface and sub soil samples from polluted areas

Figure 5 showed the result of sulphate concentration of surface and subsoil from polluted areas. The sulphate concentration ranged from  $16.00 \pm 0.00$  to  $160.00 \pm 10.00$  ppm in surface soil while the subsoil concentration ranged from  $16.00 \pm 1.00$  to  $126.67 \pm 5.77$  ppm. Figure 6 showed the result of phosphate concentration of surface and subsoil from polluted areas. The phosphate concentration ranged from  $28.57 \pm 0.12$  to  $393.33 \pm 3.06$  ppm in surface soil while the subsoil concentration ranged from  $14.37 \pm 0.15$  to  $282.33 \pm 2.08$  ppm.

Figure 7 showed the result of chloride concentration of surface and subsoil from non-polluted

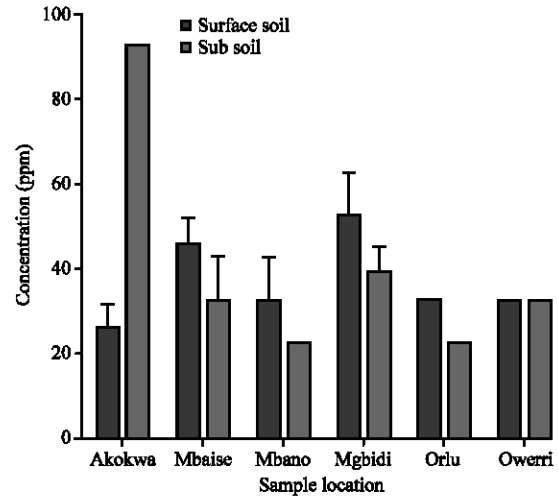


Fig. 7: Chloride concentrations (ppm) of surface and sub soil samples from non-polluted areas

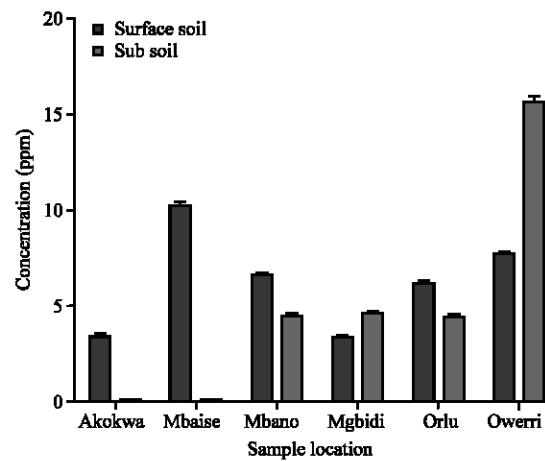


Fig. 8: Nitrate concentrations (ppm) of surface and sub soil samples from non-polluted areas

areas. The chloride concentration ranged from  $26.03 \pm 5.77$  to  $52.70 \pm 10.00$  ppm in surface soil while the subsoil concentration ranged from  $22.70 \pm 0.00$  to  $92.70 \pm 0.00$  ppm.

Figure 8 showed the result of nitrate concentration of surface and subsoil from non-polluted areas. The nitrate concentration ranged from  $3.33 \pm 0.15$  to  $10.20 \pm 0.17$  ppm in surface soil while the subsoil concentration ranged from  $0.10 \pm 0.00$  to  $15.63 \pm 0.25$  ppm.

Figure 9 showed the result of sulphate concentration of surface and subsoil from non-polluted areas. The sulphate concentration ranged from  $6.33 \pm 0.58$  to  $29.00 \pm 0.00$  ppm in surface soil while the subsoil concentration ranged from  $1.00 \pm 0.00$  to  $11.67 \pm 0.58$  ppm.

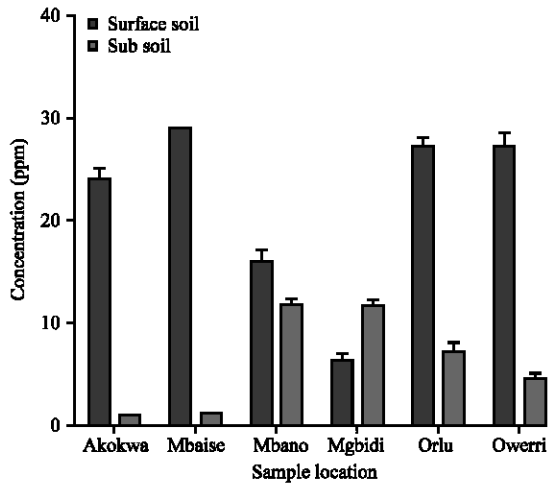


Fig. 9: Sulphate concentrations (ppm) of surface and sub soil samples from non-polluted areas

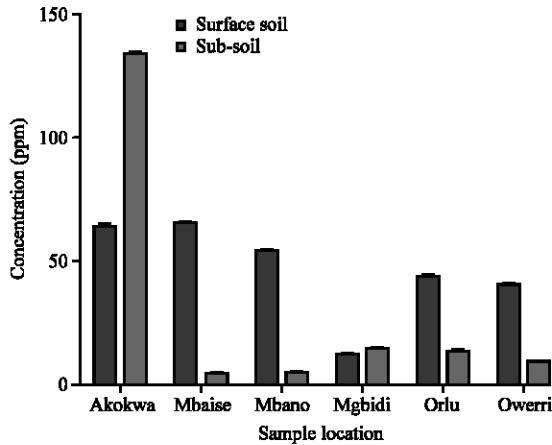


Fig. 10: Phosphate concentrations (ppm) of surface and sub soil samples from non-polluted areas

Figure 10 showed the result of phosphate concentration of surface and subsoil from non-polluted areas. The phosphate concentration ranged from  $12.33 \pm 0.58$  to  $65.47 \pm 0.25$  ppm in surface soil while the subsoil concentration ranged from  $4.47 \pm 0.25$  to  $133.50 \pm 1.00$  ppm.

Figure 11 showed the result of heavy metal concentration of surface soil samples from polluted areas. The heavy metal concentration ranged from 0.36-24.76 ppm.

Figure 12 showed the result of heavy metal concentration of sub soil samples from polluted areas. The heavy metal concentration ranged from 0.23-24.36 ppm. Figure 13 showed the result of heavy metal concentration of surface soil samples from non-polluted areas. The heavy metal concentration ranged from 0.16-6.80 ppm.

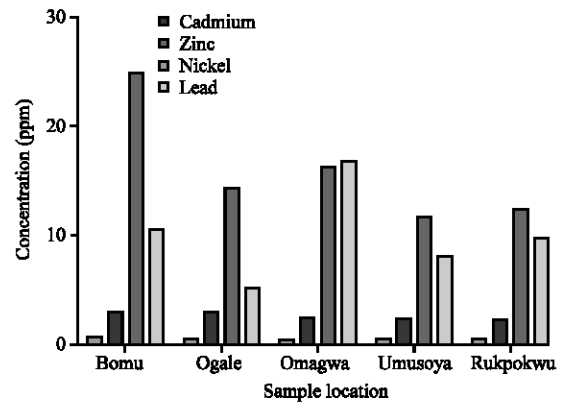


Fig. 11: Heavy metal concentrations (ppm) of surface soil samples from polluted areas

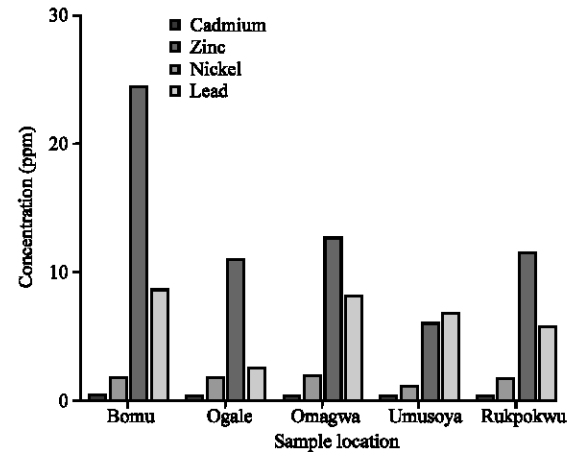


Fig. 12: Heavy metal concentrations (ppm) of sub soil samples from polluted areas

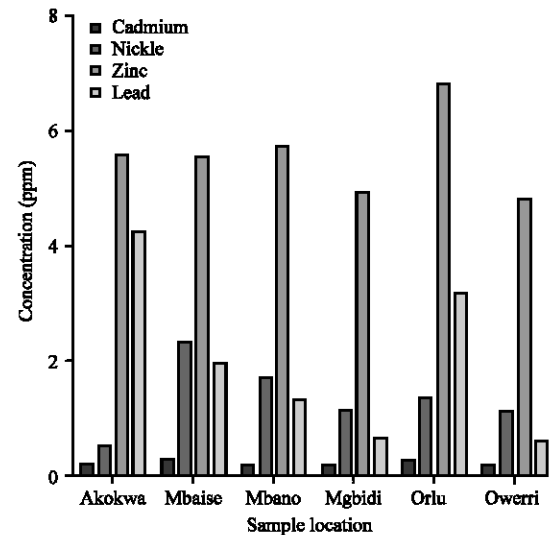


Fig. 13: Heavy metal concentrations (ppm) in surface soil samples from non-polluted areas

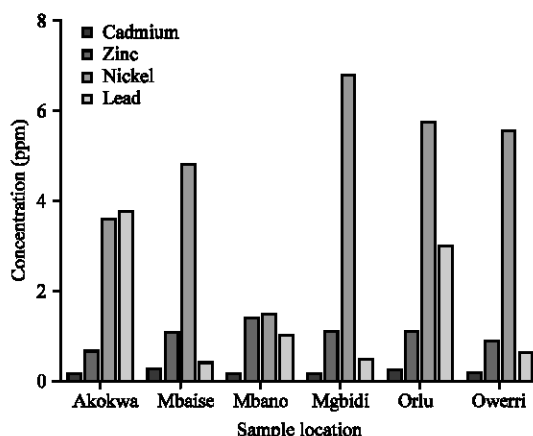


Fig. 14: Heavy metal concentrations (ppm) in sub soil samples from non-polluted areas

Figure 14 showed the result of heavy metal concentration of sub soil samples from non-polluted areas. The heavy metal concentration ranged from 0.14-6.80 ppm.

From the results, the arable soils around the crude oil polluted sites under study are vulnerable to contamination by hydrocarbon contaminants resulting from oil spillage. The TPH, PAH and soil inorganic anion usually indicate the extent of damage caused by crude oil spillage while iron (Fe), copper (Cu), zinc (Zn), cadmium (Cd), chromium (Cr), mercury (Hg), nickel (Ni), lead (Pb) and vanadium (V) are some of the heavy metals found associated with crude oil. In case of ecological perturbation arising from incidents such as oil spillages, their new levels in the post impact assessment will indicate the extent of environmental pollution.

The soil TPH ranged from  $123.92 \pm 3.26$  to  $934.79 \pm 5.26$   $\text{mg kg}^{-1}$  in study areas to  $2.40 \pm 0.01$  to  $8.59 \pm 0.13$   $\text{mg kg}^{-1}$  in the non-polluted control areas. TPH have been used in various studies to assess the level of petroleum pollution (Ebuehi *et al.*, 2005; Douglas *et al.*, 1996; Aiyesanmi *et al.*, 2004). Although, the value is much lower than the range of 126-52,200  $\text{mg kg}^{-1}$  investigated by UNEP (2011), along SPDC pipeline rights of way in Ogoniland. The result also showed that soil TPH concentration in areas around oil impacted sites is considerably higher than value of  $75.33 \pm 38.42$   $\text{mg kg}^{-1}$  obtained after phytoremediation of a crude oil contaminated soil as reported by Edwin-Wosu and Albert (2010). A much lower value of  $45.7$   $\text{mg kg}^{-1}$  was also reported by Wegwu *et al.* (2010) after 14 days of application of the RENA technique in the recovery of crude oil polluted farm lands, their study however did not look at the TPH level in soil of the surroundings. The higher TPH value of  $934.79 \pm 5.26$   $\text{mg kg}^{-1}$  at Omagwa

could be due to the fact that it was a relatively newer spill. TPH concentrations in the soil samples from the polluted areas were above regulatory limits target values of  $50$   $\text{mg kg}^{-1}$ . In the study conducted by UNEP (2011) a TPH value of  $4.14$   $\text{mg kg}^{-1}$  was observed at a distance of 290 km from a contaminated site at Gokana.

The soil PAH ranged from  $12.18 \pm 1.10$  to  $86.21 \pm 1.80$   $\text{mg kg}^{-1}$  in study areas while in the non-polluted control areas, PAH was below detectable level. This value is also above regulatory limits target values of  $1$   $\text{mg kg}^{-1}$ . The PAH value in soil is comparable to a value of  $12$   $\text{mg kg}^{-1}$  obtained from 60 samples of superficial soils from urban locations in 3 new England cities which were analyzed for PAH (Bradley *et al.*, 1994). Another study instituted by Mmon and Deekor (2010) to assess the effectiveness of land farming (Enhanced Natural Attenuation) in the remediation of hydrocarbon polluted sites in the Niger Delta in which soil samples from 10 sites polluted and remediated shows a value of  $16.01$   $\text{mg kg}^{-1}$  after land farming.

For chloride, the mean was significantly different ( $p < 0.05$ ) between the study areas and the non-polluted control but within the study areas. These differences in chloride level maybe attributable to chloride overload due to oil pollution. The difference between the chloride value of surface soil and subsoil was not significantly different. The chloride value of the study areas was however smaller than values obtained from another study on assessment of hydrocarbon content of contaminated soil reported by Onojake and Osuji (2012) where the results for surface and subsurface chloride value was  $973.94 \pm 55.63$  and  $366.06 \pm 17.29$  ppm, respectively.

For nitrate, the mean was significantly different ( $p < 0.05$ ) between the study areas and the non-polluted control. This value is indicative of effective mineralization and contribution to the fertility of the soils and compares to value of  $82.45$  ppm reported by SPDC (2004) in an environmental impact assessment of the  $20'' \times 37$  km Kolo Creek-Rumuekpe Trunkline Replacement Project.

For sulphate, the mean was significantly different ( $p < 0.05$ ) between the study areas and the non-polluted control. Petroleum pollution must have influenced the sulphate level of soil as the values obtained were higher than the range reported for similar ecosystems in the Niger Delta.

The high phosphate value could be because the phosphate ions are strongly adsorbed on clay particles and not easily leached into the receiving waters (FRN, 2008). Phosphorus plays a vital role in plant metabolism. Also, organisms involved in biological activities in the soil require phosphorus for the production and synthesis of new cell tissues. This anion is also known to stimulate root growth in plants.

The cadmium concentration in the study areas are within acceptable range because the background Cd level in soils should not exceed 0.5 ppm (Kabata-Pendias and Pendias, 1984). In a study reported by Etchie, the cadmium level of surface soil in a settlement located close to a refinery was observed to be above safe limit range of 0.04-0.48 ppm.

The fact that nickel is present in crude oil as metallo-porphyrin complex and its concentration increases with increasing hydrocarbon heavy fraction content could explain the high value observed in the study areas.

The zinc concentration falls within the value of 2.13-32.6 mg kg<sup>-1</sup> reported by SPDC in an environmental impact assessment of the 20"×37 km Kolo Creek-Rumuekpe Trunkline Replacement Project. The relatively high values of zinc an essential metal do not represent pollution.

The Lead concentration ranged from 0.61-4.25 ppm in surface soil while the subsoil concentration ranged from 0.41-3.73 ppm. This finding do not correlate with Holmgren *et al.* (1993) finding of 0.01-2.7 mg kg<sup>-1</sup> of lead concentration in soils and could be as a result of runoff from the polluted sites.

Results indicated that the heavy metals were more concentrated in the upper sections of the soil profile probably due to the complexation effect of organic matter on these heavy metals. However, these metals were also found in the sub soil sections of the profile. The movement of these metals down to the depth may be a threat to the underground water bodies within this area.

## CONCLUSION

The study has shown that the study areas are impacted by hydrocarbon, this is indicated by the high concentration of TPH and PAH above biogenic level, suggesting migration and transport of potential crude oil constituents as runoff from the nearby spilled sites.

It has already been demonstrated by previous studies that oil trapped in soil persist longer than that in water, this explains the physicochemical changes observed in the soil of the study areas.

The presence of anions (chloride, nitrate, sulphate and phosphate) in soil was detected. Petroleum pollution must have influenced anion level in soil going by the higher levels of anions in the study areas compared to the non-polluted control areas. The anion level was however within acceptable range.

The level of heavy metals in soil of the study areas was generally higher than the control areas. Cadmium,

nickel, zinc and lead must have come from the land run off since their level on the soil of the study areas were relatively high.

Heavy metals were more concentrated in the upper sections of the soil profile probably due to the complexation effect of organic matter on these heavy metals.

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