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EXTRACTABLE HYDROCARBONS, NICKEL AND VANADIUM CONTENTS OF OGBODO-ISIOKPO OIL SPILL POLLUTED SOILS IN NIGER DELTA, NIGERIA

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Abstract. An oil spill polluted site at Ogbodo-Isiokpo in Ikwere Local Government Area of Rivers State in southern Nigeria, was identified for study following three successive reconnaissance surveys of oil fields in the Agbada west plain of Eastern Niger Delta. A sampling area of 200 m × 200 m was delimited at the oil spill impacted site using the grid technique and soils were collected at surface (0–15 cm) and subsurface (15–30 cm) depths from three replicate quadrats. A geographically similar, unaffected area, located 50 m adjacent to the polluted site, was chosen as a control (reference) site. Total extractable hydrocarbon contents of the polluted soils ranged from 3.02–4.54 and 1.60–4.20 mg/kg (no overlap in standard errors) at surface and subsurface depths respectively. The concentrations of two "diagnostic" trace heavy metals, nickel (Ni) and vanadium (V), which are normal constituents of crude oil, were also determined in the soils by atomic absorption spectrophotometric method after pre-extraction of cations with dithionite–citrate carbonate. Ni varied from 0.15 to 1.65 mg/kg in the polluted plots and from 0.18 to 0.82 mg/kg in the unpolluted plots; vanadium varied from 0.19 to 0.70 mg/kg in the polluted plots and from 0.14 to 0.38 mg/kg in the unpolluted plots. Ni and V were more enhanced ($p < 0.05$) in the oil-polluted soils, especially at subsurface depth. Whilst the oil spillage could be said to be indirectly responsible for the enhanced concentrations of nickel and vanadium via the injection and availability of the petroleum hydrocarbons that might have increased the activities of biodegradation on site, the physico-chemical properties of the soils and inherent mobility of metals, as well as the intense rainfall and flooding that characterized the period of study, may have also contributed, at least in part, to these enhanced concentrations. Such levels of Ni and V may result to enhanced absorption by plants, which may bring about possible bioaccumulation in such plants and the animals that depend on them for survival and all of these may lead to toxic reactions along the food chain.

Keywords: crude oil, hydrocarbon, nickel, Niger Delta, oil spill polluted soils, vanadium

1. Introduction

In recent years, studies on impact of trace heavy metals have continued to assume increasing relevance especially in the areas of petroleum chemistry and toxicology. Trace metals may be defined as metals occurring at 1000 mg/kg or less in the earth's crust (Duffus, 1990). Such metals may be classified as 'heavy' or 'light' with respect to density. Trace 'heavy' metals have densities greater than 5 g/cm^3 (e.g., vanadium) whereas 'light' metals have densities less than 5 g/cm^3 (e.g., beryllium) (Duffus, 1990). Excessive levels of trace heavy metals (as well as trace light metals) may occur in the biosphere as a result of normal geological phenomena such as ore formation, weathering of rocks and leaching or degassing (in the case of mercury). Other activities that could contribute to excessive release of these metals into the environment include burning of fossil fuels, smelting, discharges of industrial, agricultural, and domestic wastes and deliberate application of pesticides. Anthropogenic contributions or human activities such as petroleum prospecting and mining are also major sources of these metals (Osibanjo and Jensen, 1980; Duffus, 1990; Hunt, 1996; Obute and Osuji, 2002).

Oil spillage, as it is widely referred to, is the incidental discharge of crude oil onto the environment as a result of equipment failure, operational mishap and/or intentional damage to oil production facilities, otherwise known as sabotage (Osuji, 2002). In Nigeria, the area that is worst hit by oil spillage is the region geographically designated as the Niger Delta. This region, which covers a landmass of over 70,000 square kilometers, cuts across about 800 oil-producing communities. With an extensive network of over 900 oil producing wells, 100 flow stations/gas plants, over 1500 km of trunk lines and some 45,000 km of oil and gas flow lines, the 'Niger Delta' has become synonymous with oil pollution, recording an average of 221 oil spills per year (Oremade, 1986; Okoko and Ibaba, 1999; Osuji, 2001, 2002). When such oil spillages occur on-shore, they affect soil ecosystems and in environments that are completely aquatic, oil sometimes floats on water surfaces, where it is dispersed to shorelines by wind and wave actions, also affecting the soil environment (Amadi *et al.*, 1995).

Most of the terrestrial ecosystems and shorelines in the oil producing communities of the said region are, however, important agricultural lands and are under continuous cultivation. Oil spillages may, therefore, have far reaching implications on the agricultural productivity of an affected area and multiplier effects on the socio-economic well being of the people. It is based on the foregoing premise that we decided to empirically estimate the hydrocarbon contamination and status of nickel and vanadium, the two 'substantial' metals in crude oil. The choice of these metals was informed by their peculiarity as gross indices of the biodegraded oil. The objective was to know the levels of hydrocarbon contamination occasioned by the oil spillage and its effects on these indicator metals, which are also micronutrients necessary for nutritional requirements of the soil and maintenance of agricultural productivity.

2. Materials and Methods

2.1. Location of Study Sites, Oil Spill and Date of Occurrence

The study site is Ogbodo-Isiokpo oil spill polluted site (Figure 1), located in Ikwerre Local Government Area of Rivers State in the southern part of Nigeria. The site

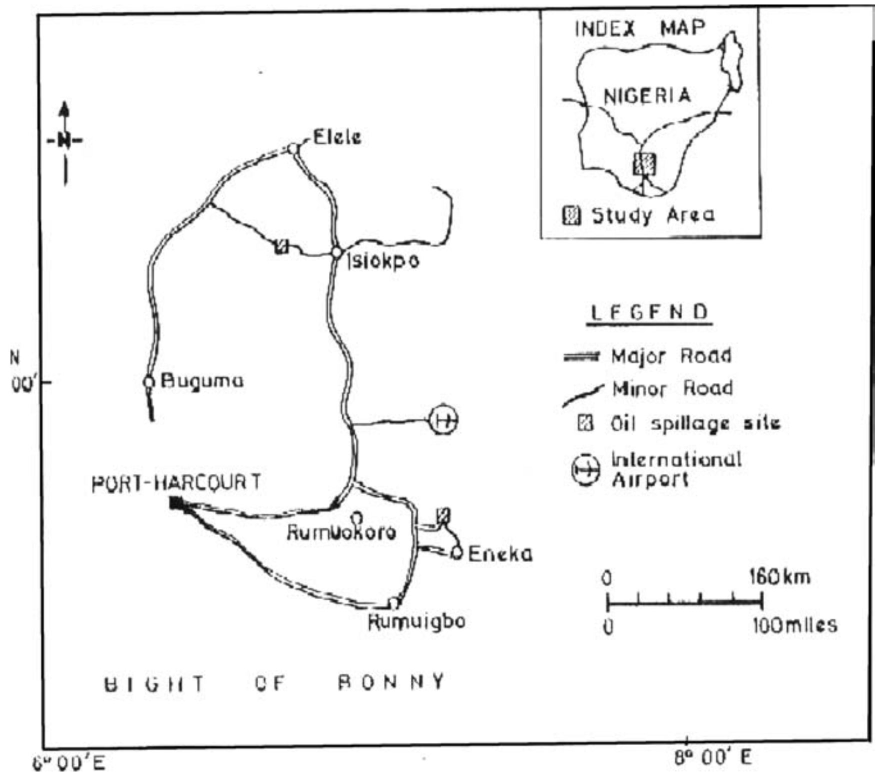

Figure 1. Schematic map of area showing the oil spillage site at Ogbodo-Isiokpo in Niger Delta, Nigeria.

slopes slightly towards the west and drops sharply on the valley of the Ogbodo stream. The spillage emanated from a high-pressure pipeline leakage at Ogbodo-Isiokpo (Plate 1). The incidence occurred on the August 21, 1997, spilling an estimated 12,000 barrels (approximately 1.9 million liters) of crude oil, covering an area of fifteen thousand nine hundred square meters (15,900 m^2). Sampling was carried out approximately six hour after the incident.

2.2. Historical antecedence and geo-characteristics of soils

Over 28 soil types from various soil zones of the Niger Delta have been identified. Soils from the study sites, which fall within the Agbada-1 and Agbada-2 prospect areas of the Niger Delta Basin, are believed to have been derived from the Upper Delta Plain built up from soil sediments from the Upper Cretaceous times (Ilaco, 1966). In this plain, fluviatile sedimentation is one of the major agents of land formation. This fluvio-deltaic environment is composed mainly of alluvial sand with clay and mud deposits in depressions and along valley bottoms.

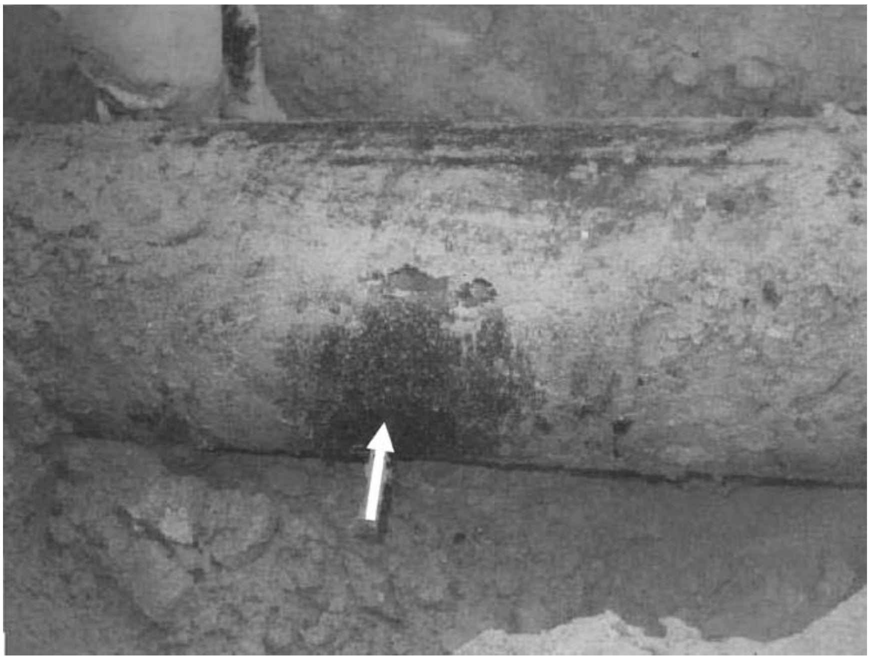

Plate 1. High pressure oil pipeline at Ogbodo-Isiokpo showing point of oil leakage.

2.3. CLIMATE

The study area lies mainly in the wet equatorial climatic region, with high cloud cover. Sunshine hours are low and air is damp for most of the year because of the very high relative humidity of the air. Temperatures are moderated by the cloud cover and by the generally damp air. Still, mean annual temperatures are as high as 24–32 °C. It rains in every month of the year with a short dry spell in the months from January to March (NDES, 1999).

2.4. VEGETATION AND LAND USE

The study area, like most part of the Niger Delta region of Nigeria, cannot be said to have a completely pristine natural vegetation. The vegetation is made up of a mosaic of arable farmlands, tree crop plantations and patches of natural vegetation. The arable crops include *Manihot esculanta* (cassava), *Dioscorea sp.* (yam), *Zea mays* (maize), *Annas comosus* (pineapple), *Capsicum sp.* (pepper), *Solanum lycoperscium* (tomatoes), *Solanum melogenia* (garden egg), leafy vegetables such as *Telfera occidentalis* (fluted pumpkin) and spices such as *Piper guineensis* (African black pepper, uziza). The tree and fruit crops include the *Elaeis guineensis* (oil palm), *Musa sp.* (plantain), *Cocos nucifera* (coconut) *Caraca papaya* (pawpaw), *Dacryodes edulis* (native pear, ube), *Chrysophyllum perpulchrum* (white star apple, udala) and *Citrus spp*.

2.5. Soil fauna

Soil fauna found in the study area are chiefly oligochaetes, diplopods (millipedes and centipedes), *Lumbricus terrestis*, adult/larval beetles and nematodes like *Panagrolaimus spp*. The insect fauna are mostly hymenoptera.

2.6. Heavy metals in Nigerian crude oils

Major Nigerian crude oil brands such as Bonny Light, Forcados Blend and Qua Iboe Light, have on the following heavy metal contents: Vanadium (1.0 part/million); nickel (4.0 parts/million); cadmium (0.1 part/million); copper, lead, etc. are <0.1 part/million.

2.6.1. *Field Reconnaissance, Sampling Technique and Soil Collection*

Field reconnaissance surveys were carried out to assess the extent of pollution on soil. Based on these, the position and direction of sampling grids and quadrats were determined. On the second day of reconnaissance, soil samples were collected according to the following method: a sampling area of 130 m $\times$ 60 m was delimited around the epicenter of each of the oil spill polluted sites. The area was divided into sixty grid plots and 30% of these (i.e., 18 grid plots) were selected at random. From each of the grid plots were taken three replicate soil samples. This was done by removing litter from the pre-determined area inside each quadrat and then using a trowel to take the soil from the surface (0–15 cm) and subsurface (15–30 cm). An uncontaminated area located fifty metres (50 m) adjacent to each of the oil spill polluted sites was selected as a control (reference) site. A total of fifty-four (54) samples were collected from surface and sub-surface depths of the polluted sites and twelve (12) from the control sites. The samples were put into polyethylene bags, labeled accordingly and taken to laboratory for subsequent analyses.

2.6.2. *Oil Extraction and Estimation of Total Hydrocarbon Content*

One gram (1.0 g) of each soil sample was put into a 500 mL volumetric flask and to this was added 200 mL of xylene. The xylene/soil mixture was shaken vigorously for five minutes and filtered into 400 mL cylinder. The volumetric flask and solid materials were rinsed properly with 500 mL xylene and filtered again into the cylinder. Total hydrocarbon content (THC) in the xylene/hydrocarbon mixture was thereafter determined by photometric method using Fisher Electrophotometer-II at a wavelength of 425 nm. THC was estimated from a calibration curve, obtained by measuring absorbance of a standard prepared by diluting 2.5, 5.0, 10.0, 20.0, 25.0, and 30.0 microliters of Bonny Light crude oil with 50 mL xylene solution.

2.6.3. *Spectrophotometric Analyses*

Spectrophotometric analyses of replicate soil samples were carried out after pre-extraction of cations with dithionite–citrate carbonate according to the method of Hesser (1977) as follows: two and a half grams (2.5 g) of each soil sample was weighed into a beaker and the same quantity of sodium dithionite was added. This was prepared by adding 88.23 g of sodium citrate to 21.02 g of citric acid in a 2 L flask and made to mark with distilled water to give exactly 0.15 M sodium citrate and 0.05 M citric acid essential for this extraction. The beaker was shaken over night in a shaking machine and later filtered with Whatman no. 42 filter paper. Twenty-five milliliters of the extract was pipetted into a 200 mL beaker and 5 mL of 30% H_2O_2 was added after which the beaker was covered with watch glass. The sample was then allowed to cool. At this stage, 10 mL of HNO_3–H_2SO_4 acid mixture was added in a fume chamber with the sample again digested for 3.5 h until the extract became clear. The extract was allowed to cool and diluted with distilled water and made to 100 mL in a volumetric flask. Concentrations of nickel, vanadium, and copper in the extract were determined thereafter using Perkin Elmer model 2280/2380 Atomic absorption spectrophotometer. Readings were taken at 316.3 μm for nickel, 228.90 μm for vanadium, 422.7 μm for copper, and 438.9 μm for cadmium.

2.6.4. *Statistical Analysis*

Means of metal concentration of the various replicate samples and standard deviation (S.D.) were calculated using conventional statistical formulae. Standard error (S.E.) was calculated as follows:

$$\text{S.E.} = \text{S.D.}/N^{1/2},$$

where N is the number of replications.

Standard errors (±S.E.) were inserted onto the representative bar charts using vertical bars.

3. Results and Discussion

Extractable hydrocarbon ranges of 3.02–14.54 and 1.60–4.20 mg/kg (no overlap in standard errors) obtained from surface and subsurface polluted soils, respectively (Table I) provide evidence of hydrocarbon contamination at the study site. Although clean up measures had been effected about six months prior to the sampling period as part of the contingency program (cf. Plate 2), results obtained herein presuppose that there are still contaminable percolates of the spilled-oil at surface and subsurface depths of the polluted site. Usually, such hydrocarbon ranges deplete available oxygen and reduce gaseous diffusion in surface and subsurface soils, thereby stressing the organisms trapped beneath, some of which eventually die of asphyxiation. Such hydrocarbon levels may also discourage plant growth, thereby reducing the

TABLE I

Mean levels (±Standard Error at 95% confidence level) of total extractable hydrocarbons at surface and subsurface depths of oil spill polluted soils from Ogbodo-Isiokpo in Niger Delta Nigeria

	Total hydrocarbon content (mg/kg) ± S.E.	
Sampled plots	Surface (0–15 cm)	Subsurface (15–30 cm)
Polluted site		
2	3.14 ± 0.133	2.82 ± 0.166
5	3.20 ± 0.088	3.10 ± 0.112
7	3.13 ± 0.154	1.96 ± 0.104
10	3.02 ± 0.104	1.60 ± 0.116
14	3.18 ± 0.104	3.06 ± 0.105
18	3.39 ± 0.165	1.95 ± 0.108
20	3.55 ± 0.132	2.90 ± 0.096
22	2.90 ± 0.127	3.20 ± 0.160
27	3.07 ± 0.046	3.46 ± 0.102
28	4.15 ± 0.102	2.54 ± 0.114
30	3.60 ± 0.084	1.85 ± 0.164
33	3.15 ± 0.114	2.90 ± 0.112
37	3.48 ± 0.126	3.00 ± 0.090
41	4.13 ± 0.107	4.20 ± 0.111
44	3.82 ± 0.180	2.00 ± 0.145
46	3.95 ± 0.104	3.05 ± 0.110
57	3.20 ± 0.122	2.46 ± 0.136
58	4.54 ± 0.106	2.80 ± 0.126
Mean ± S.E.	3.48 ± 0.055	2.71 ± 0.076
Control plots		
5	0.2 ± 0.00	0.0 ± 0.00
28	0.1 ± 0.06	0.2 ± 0.18
41	0.0 ± 0.00	0.2 ± 0.20
57	0.1 ± 0.04	0.0 ± 0.00
Mean ± S.E.*	0.1 ± 0.22	0.1 ± 0.26

*S.E. is standard error for the means of four replicates at 95% confidence limit.

population density and species diversity of plant cover and other vegetations found in the affected area. In a recent reconnaissance survey of the Agbada west plain of Niger Delta, Osuji *et al.* (in press) gave an empirical decimation of flora and fauna by oil spillage. Odu *et al.* (1985), NDES (1999) and several other past studies also corroborate this evidence.

Results obtained from the spectrophotometric analysis of the "diagnostic" trace heavy metals (nickel and vanadium) showed that there were no significant differences in the amounts of nickel and vanadium in the subsurface polluted soils when

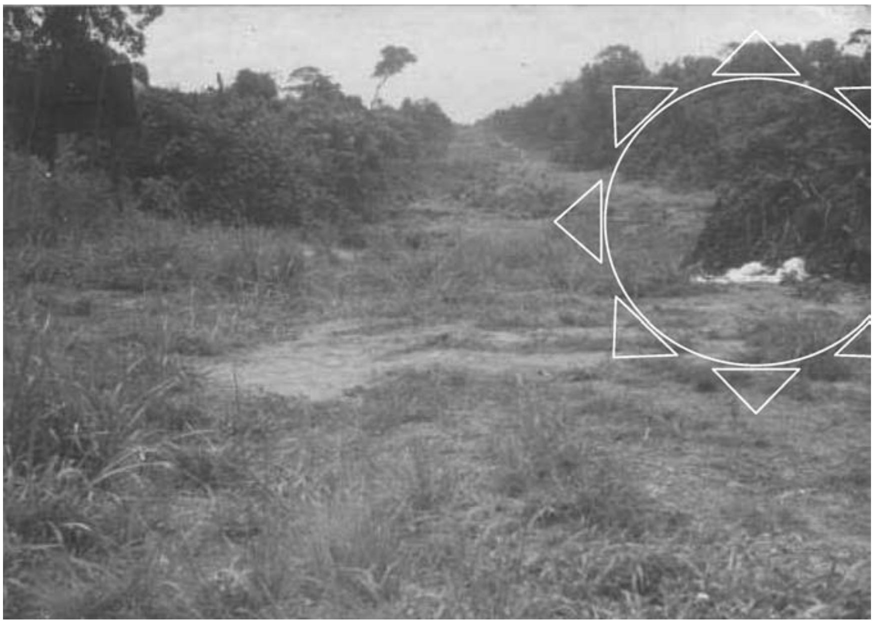

Plate 2. A section of the oil spill polluted at Ogbodo-Isiokpo showing excavated top soil.

compared with the unpolluted reference (control) soils (Table II). The amounts of nickel in surface and subsurface soils varied from 0.15 to 1.65 mg/kg in the polluted plots and from 0.18 to 0.82 mg/kg in the unpolluted plots; vanadium varied from 0.19 to 0.70 mg/kg in the polluted plots and from 0.14 to 0.38 mg/kg in the unpolluted plots. The slightly enhanced levels of nickel and vanadium in the soils may result to enhanced absorption by plants which may bring about possible bioaccumulation by such plants and the animals which depend on them for survival and all of these may lead to toxic reactions along the food chain (Blummer *et al.*, 1970; Duffus, 1980; Okonya *et al*., 1988). The implication of an enhanced level of nickel and vanadium may not be as significant in a non-arable land as it would be in the study area where the sites were previously used as farmlands for the cultivation of crops like cassava and maize (as earlier observed from field reconnaissance). That there were higher concentrations of these metals at the subsurface depth may be attributable to the fact that metal profiles in polluted soils usually penetrate a little below the 10-cm region even after many years (Smith *et al.*, 1999). Additionally, it is possible that with the intense weathering activity of the Niger Delta, where records of annual rainfall exceed 1200 mm, the excess water which may have accumulated over the rainy season that preceded sampling, might have informed possible accumulation of the metallic oxides which probably increased mineralization by strains of microbial genera some of which increased in population due to availability of excess hydrocarbon. Increased population densities of hydrocarbon utilizing microbes in the study sites had earlier been reported (Osuji, 1998). Such increases in Ni and V

TABLE II
Mean concentration (±Standard Error at 95% confidence level) of nickel and vanadium in polluted and control (reference) soils from Ogbodo-Isiokpo

	Concentration (mg/kg) ± S.E.			
	Surface (0–15 cm)		Subsurface (15–30 cm)	
Plots	Ni	V	Ni	V
Polluted site				
2	0.25 ± 0.004	0.21 ± 0.011	0.34 ± 0.020	0.36 ± 0.020
5	0.43 ± 0.034	0.39 ± 0.006	0.75 ± 0.013	0.44 ± 0.016
7	0.30 ± 0.027	0.20 ± 0.006	0.20 ± 0.016	0.70 ± 0.013
10	0.71 ± 0.019	0.22 ± 0.010	1.22 ± 0.005	0.30 ± 0.013
14	0.80 ± 0.041	0.28 ± 0.008	0.23 ± 0.004	0.35 ± 0.009
18	0.30 ± 0.002	0.29 ± 0.005	0.30 ± 0.018	0.52 ± 0.016
20	0.30 ± 0.010	0.35 ± 0.002	0.16 ± 0.018	0.45 ± 0.021
22	0.75 ± 0.005	0.30 ± 0.014	0.45 ± 0.036	0.25 ± 0.001
27	0.15 ± 0.022	0.20 ± 0.009	0.30 ± 0.018	0.20 ± 0.010
28	1.65 ± 0.038	0.18 ± 0.015	0.30 ± 0.022	0.31 ± 0.005
30	0.31 ± 0.010	0.24 ± 0.010	0.28 ± 0.009	0.28 ± 0.008
33	0.34 ± 0.008	0.20 ± 0.012	1.50 ± 0.022	0.20 ± 0.025
37	0.28 ± 0.002	0.19 ± 0.006	0.30 ± 0.015	0.30 ± 0.028
41	0.31 ± 0.013	0.38 ± 0.040	0.16 ± 0.006	0.28 ± 0.004
44	0.40 ± 0.029	0.27 ± 0.002	0.22 ± 0.004	0.46 ± 0.030
46	0.30 ± 0.008	0.28 ± 0.005	0.24 ± 0.018	0.34 ± 0.010
57	1.02 ± 0.060	0.28 ± 0.008	0.25 ± 0.020	0.26 ± 0.008
58	0.38 ± 0.025	0.35 ± 0.016	0.30 ± 0.004	0.24 ± 0.008
Mean ± S.E.	0.50 ± 0.040	0.26 ± 0.008	0.42 ± 0.040	0.38 ± 0.105
Control site				
10	0.82 ± 0.009	0.28 ± 0.012	0.32 ± 0.018	0.20 ± 0.005
22	0.41 ± 0.010	0.22 ± 0.015	0.26 ± 0.011	0.18 ± 0.012
33	0.36 ± 0.014	0.14 ± 0.004	0.90 ± 0.012	0.38 ± 0.010
46	0.28 ± 0.014	0.18 ± 0.004	0.20 ± 0.016	0.38 ± 0.010
57	0.44 ± 0.059	0.26 ± 0.002	0.32 ± 0.081	0.26 ± 0.013
58	0.18 ± 0.007	0.16 ± 0.017	0.25 ± 0.020	0.22 ± 0.019
Mean ± S.E.	0.42 ± 0.062	0.21 ± 0.018	0.36 ± 0.080	0.27 ± 0.028

contents have, in deed, been reported as one of the gross properties of biodegraded petroleum (Hunt, 1996). This implies that the enhanced concentration of Ni and V may not have resulted directly from the spilled-oil, but indirectly through the enhanced activities of biodegradation by such hydrocarbon-utilizing organisms, which had responded to the presence of the petroleum hydrocarbons on site.

Flooding of soils, particularly when readily oxidizable organic nutrients are available, leads to significant mobilization of most trace heavy metals (Alexander, 1961). The amounts of soluble nickel and vanadium, in the submerged soils might have increased following flooding, more so since the quantity of the elements mobilized in this manner increases with added organic carbon (Alexander, 1961). Therefore, in appraising the potential hazards of such metals found in crude oil, it is necessary to consider their mobility, chemical form as well as the physico-chemical characteristics of the soil. Though nickel is a micronutrient, an excessive level of the metal in the soil might be toxic to some soil fauna, like earthworms, which are adjuncts to the microflora in organic matter decomposition and may also reduce heterotrophic activity of the microflora (Osuji *et al.*, 2002). In the same vane, vanadium is essential for green algae and stimulates higher green plants in small amounts, but can be fatal even to rats at concentrations of 10 mg/kg. Although results obtained in this study are sub-lethal compared to the afore-mentioned concentration, the high pH and organic matter content of the soils as reported by Osuji (2001), might enhance the concentrations of these metals to a potentially hazardous level.

4. Conclusion

Given the results obtained in this study (the mean levels and standard errors), there were no significant differences in the concentrations of nickel and vanadium in polluted and control soils. However, the oil spillage at Ogbodo-Isiokpo can still be said to be indirectly responsible for the slightly enhanced concentrations of these metals through the injection and availability of the petroleum hydrocarbons that might have increased the activities of biodegradation on site through increased activities of hydrocarbon utilizing organisms. Physico-chemical properties of the soils, mobility of metals, as well as the intense rainfall and flooding that characterized the period of study may have also contributed, at least in part, to the enhanced concentrations of soil nickel and vanadium. The higher concentration of metals at subsurface depth is attributable to the fact that metal profiles of polluted soils penetrate a little below the 10-cm region. However, such higher than normal levels of Ni and V as detected in the polluted subsurface soil are capable of impairing plant growth and causing possible bioaccumulation of the metals that are supposed to be present in such micro-concentrations as were detected in the control (reference) soils.

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