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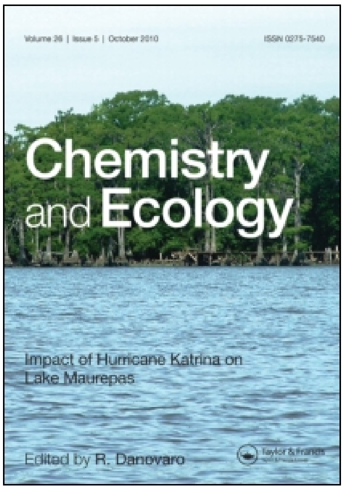

Chemistry and Ecology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455114

Chemical reclamation of crude-oil-inundated soils from Niger Delta, Nigeria

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To cite this Article Osuji, L. C. , Egbuson, E. J. and Ojinnaka, C. M.(2005) 'Chemical reclamation of crude-oil-inundated soils from Niger Delta, Nigeria', Chemistry and Ecology, 21: 1, 1 — 10
To link to this Article: DOI: 10.1080/02757540412331335988
URL: http://dx.doi.org/10.1080/02757540412331335988

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Chemistry and Ecology
Vol. 21, No. 1, February 2005, 1–10

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Chemical reclamation of crude-oil-inundated soils from Niger Delta, Nigeria

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(Received 31 August 2004; in final form 26 November 2004)

Crude-oil-inundated soils were collected from the Agbada oil field in the Niger Delta region of Nigeria 2 months after the recorded incidence of oil spillage. The soils were taken on the second day of reconnaissance from three replicate quadrats, at surface (0–15 cm) and subsurface (15–30 cm) depths, using the grid sampling technique. The total extractable hydrocarbon content (THC) of the polluted soils ranged from 1.24×10^2 to 3.86×10^4 mg/kg at surface and subsurface depths (no overlap in standard errors at a 95% confidence level). Greenhouse trials for possible reclamation were later carried out using 10–100 g of $(NH_4)_2SO_4$, KH_2PO_4 and KCl (NPK) fertilizer as nutrient supplements. Nitrogen as NO_3-N and potassium were optimally enhanced at 2% (w/w) and 3% (w/w) of the NPK supplementation, respectively. Phosphorus, which was inherently more enhanced in the soils than the other nutrients, maintained the same level of impact after treatment with 20 g of NPK fertilizer. Total organic carbon (%TOC), total organic matter (%TOM), pH, and percentage moisture content all provided evidence of enhanced mineralization in the fertilizer-treated soils. If reclamation of the crude-oil-inundated soils is construed as the return to normal levels of metabolic activities of the soils, then the application of the inorganic fertilizers at such prescribed levels would duly accelerate the remediation process. However, this would be limited to levels of pollution empirically defined by such THC values obtained in this study.

Keywords: Crude-oil-inundated soils; NPK fertilizer; Niger Delta; Oil spillage

1. Introduction

The soil ecosystem is usually inundated by crude oil when it is incidentally discharged into the environment, due to various reasons ranging from failure of production equipments to operational mishaps, or intentional damage to production facilities, otherwise known as sabotage. In Nigeria, the area worse hit by this ugly menace is the region geographically designated as the "Niger Delta". The Niger Delta covers a landmass of over 70,000 km^2, which cuts across about 800 oil-producing communities. With an extensive network of over 900 oil-producing wells, 11,000 km aging flow lines and over 160 flow stations, the region has become synonymous with pollution of aquatic and terrestrial ecosystems [1]. In environments that are completely

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Chemistry and Ecology
ISSN 0275-7540 print/ISSN 1029-0370 online © 2005 Taylor & Francis Ltd
http://www.tandf.co.uk/journals
DOI: 10.1080/02757540412331335988

aquatic, oil sometimes floats on water surfaces, where it is dispersed to shorelines by wind and wave actions, thus affecting the soil environment [2]. The spilled oil on site may also sometimes lead to serious conflagration that may consume several acres of arable land, as in the case of the Oyakama oil spillage of 10 May 1980 [3].

Most of the terrestrial ecosystems and shorelines in the oil-producing communities, however, encompass important agricultural land and are under continuous cultivation. Oil has adverse effects on soil conditions, micro-organisms and plants [1, 4, 5]. Any contact with oil may result in damage to soil and plant communities [4]. Beyond 3% concentration, oil has been reported to be increasingly deleterious to soil biota and crop growth [6].

Over the years, there has been a dearth of reliable data on the remediation or reclamation of soils typically inundated by crude oil. However, Amadi *et al.* [4] attempted to reclaim soils artificially spilled with crude oil using different organic (poultry manure, peptone water, sawdust and yeast extract) and inorganic (NPK, KNO_3) nutrient supplements. Attempts have also been made at liming highly acidic soils, arising from oil inundation, by the application of calcium and magnesium compounds to raise the soil pH [7]. Other previous reports available on chemical remediation using chemical techniques, had been in relation to the aquatic ecosystem where chemical dispersants and emulsifiers had been employed. For instance, emulsifiers were used after the Torrey Canyon oil spillage that spilled approximately 106,000 tonnes (117,000 tons) of Kuwait crude oil in Cornwall (UK) and the Seestern tanker spillage that spilled an estimated 11,000 tonnes (12,000 tons) of Nigerian Bonny Light crude oil in the Medway estuary of south-east Britain [6,8].

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Our hypothesis is that chemical remediation of oil-spill-polluted soils can be accomplished *in situ* by selection and optimization of known conditions that promote the metabolic activities of the soils. It is based on this premise that we set to formulate this methodology of chemical reclamation using soil macronutrient status as an index of remediation. Acceleration of the remediation process will reduce the level of pollution and thus minimize its impact on agricultural productivity and community structure.

2. Materials and methods

2.1 *Description of study site, oil spill and date of occurrence*

The study area, i.e. the oil spill site (figure 1), is located in the Agbada community in the Niger Delta region of Nigeria. The incident occurred on 21 August 2003, approximately 2 months prior to sampling, and was attributed to intentional damage to one of the facilities on site. An estimated 28,000 barrels (bbl) of crude oil (approximately 4.45 million litres of crude oil) was spilled, covering over 1.2 ha (3 acres) of a third-party farmland close to the well head at the Agbada-1 oil field (figures 2 and 3).

The physico-chemical properties of the spilled oil, Bonny Light crude oil, are summarized in table 1.

2.2 *Field reconnaissance and sampling technique*

Soil samples were collected on the second day of the field-reconnaissance survey from the two sites, while geographically similar, uncontaminated areas located 50 m adjacent to each of the oil-contaminated site, were selected as the control sites. At the sites from where soil samples were collected, a sampling area of 200 m $\times$ 200 m was delimited around the epicentre of the spillage. The area was divided into 100 grid plots, and 33% of these (i.e., 33 grid plots)

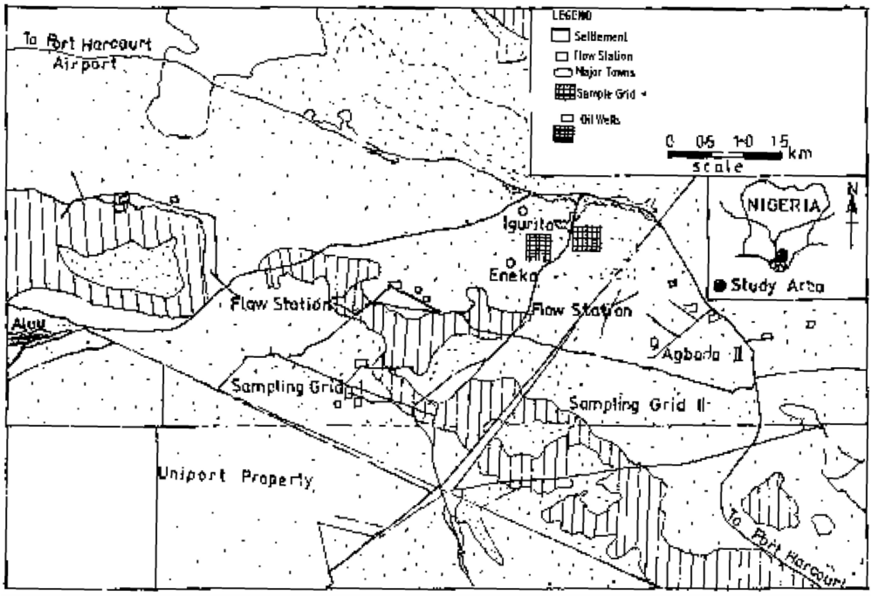

Figure 1. Map of the study area showing sample grids within the Agbada oil field in the Niger Delta, Nigeria.

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were selected at random. From these plots, three replicates of pre-determined quadrats were established, and soil samples were taken from each. This was done by removing litter from the pre-determined area inside each quadrat and thereafter using a trowel to take the soil from the surface (0–15 cm) and subsurface (15–30 cm). Soil samples were placed into polyethylene bags, labelled, and brought to the laboratory where they were kept in a refrigerator until they were required for analysis.

Figure 2. Photographic plate of an oil-spill-polluted site in Agbada in the Niger Delta region of Nigeria.

Figure 3. Cross-section of the Agbada study site in the Niger Delta, Nigeria, showing crude-oil-inundated soils and vegetation.

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2.3 *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 site, which falls within the Agbada-1 and Agbada-2 prospect areas of the Niger Delta Basin, are believed to have been derived from the quartenary Warri–Sombreiro plains, which also constitute the major underlying bedrock of the area [9]. The physical and chemical properties of soils in the study area are summarized in table 2. This plain, which appears on either side of the recent alluvial plain, was laid in the Late Pleistocene to Early Holocene time and occupies an area similar to the present-day delta, but was mostly eroded away during the ice ages when the sea level was lower. The sediments occur as grey to dark grey/brown clayey silty sands. The shallow nature of the aquifer around the Agbada flow stations would naturally pre-dispose groundwater to contamination. However, the relatively low permeability of the clayey sands separating the topsoil from the aquiferous sands retards the rate of vertical infiltration, resulting in limited downward migration of surface contaminants. Thus, there is a likelihood of the retention of contaminants in the near-surface horizon that could have adverse effects on soils. The study area has a mean daily temperature of 26 °C and rainfall of 180 mm; rain falls every month of the year, with a short dry spell in the months of January to March [10].

Table 1. Physico-chemical properties of spilled Bonny Light crude oil.[a]

Sulphur content	0.14%
Specific gravity	0.8398
API gravity 15.5 °C[b]	37
Viscosity (cSt) at 25 °C[c]	4.09
Wax content	3.8%
Pour point (−18 °C)	23
Surface tension (/Nm)	0.02041
Refractive index	1.472

[a]Source: Nigerian National Petroleum Corporation (NNPC).
[b]API: American Petroleum Institute.
[c]cSt: centistokes.

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Table 2. Some physicochemical properties of soils from study area.[a]

Texture	Sandy loam
pH	4.8–5.2
Ca^{2+}	0.18–0.33 meq/100 g of soil
Mg^{2+}	0.09–0.20 meq/100 g of soil
K^{+}	0.30–0.46 meq/100 g of soil
Na^{+}	0.13–0.29 meq/100 g of soil
PO_4^{3-}	4–80 mg/kg
Mn	0.02–0.08 mg/kg
Fe	40–54 mg/kg
Organic C	0.27–0.63%
Total N	0.08–0.12%
C/N	18–20
CEC	1–2 meq/100 g of soil

[a]Source: NDES [10].

2.4 *Vegetation and land use*

The study area, like most parts 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 microflora and fauna*

Organisms involved in microbial activities in the study area include the following microflora: *Micrococcus* spp., *Streptococcus* spp., *Fuarium* spp., *Aspergillus* spp., *Azobacter* spp., and *Nitrosomonas* spp. Most of these organisms are known to also utilize petroleum hydrocarbons. Soil fauna found in the study area are chiefly oligochaetes, diplopods (millipedes and centipedes), *Lumbricus terrestis* and adult/larval beetles. The insect fauna are mostly Hymenoptera [10].

2.6 *Total extractable hydrocarbon content*

Five grams of each soil sample was weighed out and transferred into a 500 ml volumetric flask and then 50 ml of xylene was added to this. The xylene/soil mixture was shaken vigorously for 5 min and filtered into a 400 ml cylinder. The volumetric flask and solid materials were rinsed properly with 500 ml xylene and filtered again into the cylinder. The xylene-oil extract was thereafter placed in cuvette wells, and its absorbance was determined using a Hack DR/2010 Particle Data Logging Spectophotometer. A calibration curve was obtained by measuring the absorbance of dilute standard solutions of lease oil (Bonny Light/Bonny Medium crude oils), prepared by diluting 2.5, 5.0, 10.0, 20.0, 25.0, and 30.0 μl of the lease oil with 50 ml of xylene solution. The total hydrocarbon content (THC) was calculated after reading the absorbance of the extract from the spectrophotometer, extrapolating from the calibration curve and multiplying by an appropriate dilution.

2.7 *Physico-chemical analyses*

The percentage moisture content and pH were determined using standard methods [11]. The total organic carbon (TOC) and total organic matter (TOM) were determined by the titrimetric method of Walkley and Black [12] as follows: 1 g of soil sample was weighed into a 500 ml flask, and 10 ml of $K_2Cr_2O_7$ and 20 ml of concentrated H_2SO_4 was added. Then, 200 ml of distilled water, 10 ml H_3PO_4 and five drops of diphenylamine indicator were added to the mixture, before titrating with 0.5 N $(NH_4)_2SO_4Fe$. A blank titration (without 1 g of soil) was then carried out, and the percentage TOC was calculated as:

$$\begin{aligned} \%\text{TOC} &= \frac{\text{Blank titre} - \text{sample titre} \times 0.003 \times 100}{\text{sample weight}} \\ \%\text{TOM} &= \text{TOC}\ (\%) \times 1.724, \end{aligned} \qquad (1)$$

where 1.724 = conversion factor (i.e. %TOM = %TOC × 100/58, since TOC is 58% of TOM).

2.8 *Greenhouse trials with chemical nutrients*

Reclamation trials with chemical nutrient supplements were carried out in the greenhouse using varying concentrations of nitrogenous, phosphatic and potassic fertilizers obtained from Bayelsa State Ministry of Agriculture, Yenegoa. Fifty grams (50 g) of soils from polluted and reference sites was placed into black polyethylene bags and placed in the greenhouse according to a method modified after Jackson [13]. The soils were thereafter treated with 10–100 g of $(NH_4)_2SO_4$, KH_2PO_4 and KCl. Treatments were displayed in a randomized complete block (RCB) design with three replications per treatment. The soils were left to stand for 30 days before subsequent laboratory analyses.

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2.9 *Post-treatment analyses*

Samples were analysed for nitrogen and potassium using the methods of Jackson [13]. Available phosphorus was analysed using the molybdate blue colour method of Murphy and Riley [14] after extraction with Bray P-1 extractant [15].

2.10 *Statistical analysis*

Standard error (±SE) was given as: SE = $SD^{1/n}$, where SD is the standard deviation, and n is the number of replicates. SE was estimated at the 95% confidence limit by multiplying by 1.96.

3. Results and discussion

The results of the total extractable hydrocarbon content (THC) for each sampled plot are given in table 3, while tables 4 and 5 summarize the physico-chemical parameters (pH, moisture content, TOC, TOM) and nitrogen, potassium and phosphorus contents, respectively. Particle size, estimated by the diameter of individual soil fragments, and the proportional amounts, or distribution, of the different sized particles within the soil sample, determine its texture. The study soils are classified as sandy loam (table 2), which usually consists of 21–50% clay

Table 3. Total extractable hydrocarbon content (THC) of crude-oil-inundated soils from the Agbada oil field in Niger Delta, Nigeria.

	Total hydrocarbon content, THC (mg/kg)	
Sample location	Surface (0–15 cm)	Subsurface (15–30 cm)
AA-06	7.725×10^3	3.452×10^4
AA-10	3.031×10^4	3.856×10^4
AA-15	3.849×10^4	9.451×10^3
AA-23	4.111×10^2	3.992×10^2
AA-28	3.005×10^2	1.240×10^2
AA-35	3.450×10^3	2.452×10^3
AA-48	7.660×10^3	9.402×10^3
AA-66	3.110×10^2	1.350×10^2
AA-68	3.520×10^4	2.564×10^3
AA-75	3.041×10^4	4.001×10^2
AA-82	4.661×10^3	3.758×10^4
AA-94	3.902×10^2	3.450×10^4
Mean ± SE[a]	$2.00 \times 10^4 \pm 8.53 \times 10^3$	$2.13 \times 10^3 \pm 8.99 \times 10^2$
Control plots		
AA-15	0.02	0.00
AA-28	0.00	0.01
AA-48	0.02	0.01
AA-75	0.01	0.02
Mean ± SE	0.03 ± 0.00	0.02 ± 0.01

[a]SE: standard error for the means of three replicate quadrats (at the 95% confidence level).

Table 4. Mean concentrations (±SE) of some physico-chemical properties of oil-spill-polluted and unpolluted (control) soils from Agbada in Niger Delta, Nigeria.

Soil sample	pH	Moisture (%)	TOC (%)	TOM (%)
Control	5.6 ± 0.1	17.0 ± 2.1	3.4 ± 0.9	5.9 ± 1.6
Surface	5.2 ± 0.2	17.6 ± 1.8	4.3 ± 1.4	7.4 ± 2.5
Subsurface	5.1 ± 0.2	18.0 ± 0.8	9.6 ± 4.3	7.9 ± 1.5

and/or silt. Soil texture affects how well nutrients and water are retained in the soil. When nutrients leach into the soil as a result of its inability to hold water, they become unavailable for plants to use [17]. THC of the soils gave an empirical insight into the level of hydrocarbon pollution on site, which had been difficult to estimate from field reconnaissance surveys. Although the concentration of hydrocarbon at the time of spillage barely 3 months prior to sampling was not known, the paucity of data obtained for the THCs ranging from 1.24×10^2 to 3.86×10^4 mg/kg at surface and subsurface depths (table 3) presupposes high levels of hydrocarbon on site and far exceeds the 50 ppm compliance baseline limit set for petroleum

Table 5. Mean concentration of nutrients (±SE) of NPK-treated soils and their untreated control.

%(w/w) of NPK supplement	Mean concentrations of various nutrients ± SE (mg/kg)		
	NO_3-N	PO_4-P	K
Control	23 ± 3	0.8 ± 0.2	30 ± 6
1	1600 ± 76	1.5 ± 0.2	640 ± 42
2	2303 ± 143	1.2 ± 0.3	480 ± 35
3	2162 ± 184	1.6 ± 0.2	550 ± 79

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industries in Nigeria [16]. Usually, such levels of petroleum hydrocarbons on the site create anoxic conditions in surface and subsurface soils because the oil film reduces gaseous diffusion and increases the presence of anaerobic organisms, which deplete available oxygen. Depletion of oxygen in the affected soil environment increases the stress to living organisms (both surface and subterranean biota), some of which may eventually die of asphyxiation [2].

3.1 *Addition of nutrients*

A high hydrocarbon content increases the population density of heterotrophic organisms that utilize the hydrocarbons as the sole source of carbon for their metabolism [17]. Oil-degrading or hydrocarbon-utilizing microbes such as *Azobacter* spp. normally become more abundant, while nitrifying bacteria such as *Nitrosomonas* spp. become reduced in number [18]. This probably explains the relatively lower values of NO_3-N obtained for the pre-treated polluted soils (table 5). The process of nitrification might have been reduced following oil spillage. Conversely, the agronomic addition of nitrogen via the fertilizer input, as limiting nutrient, may indeed have facilitated the level of nitrification, thus explaining the observed increase in nitrogen content of the treated samples.

Thus, the mean NO_3-N concentration of $2.16 \times 10^3 \pm 1.84 \times 10^2$ in the treated soils, which was significantly higher ($P < 0.01$) when compared with the 23.25 ± 3.34 mg/kg concentration in the untreated control soils (no overlap in standard errors at a 95% confidence level), clearly suggests enhanced mineralization in the treated samples. Although a lower but comparatively higher mean value ($P < 0.05$) was obtained for potassium, this also suggests that the potassic fertilizer enrichment must have enhanced mineralization. That the study area is known to have an inherently high level of available phosphorus [10] might explain the exceedingly high mean phosphorus values obtained for the treated samples.

The results for NO_3-N (table 5) further highlight the significance of baseline references in the evaluation and application of such inorganic nutrient supplements. For instance, treatment with 20 g of NPK, which gave an optimum level of NO_3-N, would imply that 2% (w/w) of the fertilizer treatment might result in the significant improvement of the level of that macronutrient element in the oil-polluted soil. This, however, may depreciate as the concentration tends towards 3% (w/w), as seen in table 5. Conversely, the inherently enhanced soil phosphorus did not show such an optimal effect on the polluted soil but rather maintained the same impact level after the 2% (w/w) treatment with NPK. Therefore, although potassium shows an increasing trend in impact with the varying supplementation with NPK (table 5), it also shows a marked enhancement at 3% (w/w) concentration, thus buttressing the need for empirical baseline determination of peculiar nutrient supplements before application. Amadi *et al.* [5] corroborate the need for such baseline guidelines in the use of nutrient supplements for oil-polluted soils with particular reference to the performance of maize (*Zea may* L.). The authors agreed that while the adverse effects of crude-oil-inundation of soils could be ameliorated by nutrient supplementation, adequate care must be taken in the choice and application of such supplements, as several supplements rather than enhanced crop performance might impose toxicity problems.

3.2 *Effect of pH and moisture content*

The variation in soil acidity as typified in the pH values obtained in this study (cf. table 4) may have affected mineralization and the entire process of remediation. Several investigations have demonstrated a significant correlation between nitrate production and pH [19, 20]. In acidic environments, nitrification is said to proceed slowly, even in the presence of an adequate supply

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of nutrient substrate. Ibia *et al.* [5] and Amadi *et al.* [20] suggested the addition of lime to such acidic soils to bring them to the optimum pH values conducive for microbial metabolism (which would imply a shift from acidity to neutrality). Thus, the relatively lower pH values obtained in the pre-treated polluted soils must have deterred the rate of nitrification, though an exact limiting pH was not ascertained since a variety of other physico-chemical factors in the soils (such as moisture content) may have altered specific boundaries. Because moisture content affects the aeration regime of the soil, the water status of the soil would have a marked influence upon mineralization. Oxygen, an obligate requirement for the process of mineralization and remediation, is usually limited by waterlogging, and moreover in an oily environment where oil also limits gaseous diffusion of O_2 [2]. Therefore, a high moisture content might have a suppressed O_2 supply, which might have limited the oxidation of ammonium.

3.3 *Organic carbon and organic matter content*

Total organic carbon and total organic matter contents (%TOC and %TOM) are co-indices of soil fertility. The data obtained for these parameters for all treatments also provide evidence of enhanced mineralization in the treated samples, judging from the significantly higher concentrations of these nutrients when compared with the untreated control soils (cf. table 4). Thus, if bioremediation is construed as the return to normal levels of metabolic activities of the oil-spill-polluted soils, then an increase in such metabolic activities would presuppose an enhanced process of bioremediation. This posits that the inorganic-fertilizer treatment of affected soils, which enhanced the macro-nutrient status of the soils, duly accelerated the remediation process of the soils.

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4. Conclusion

Chemical reclamation of oil-spill-polluted soils from Agbada in the Niger Delta region of Nigeria was optimally enhanced at 2% (w/w) and 3% (w/w) supplementation with $(NH_4)_2SO_4$, KH_2PO_4 and KCl (NPK) fertilizer for NO_3-N and potassium, respectively. Phosphorus, which was inherently enhanced in the soils more than the other nutrients, maintained the same level of impact after 2% (w/w) treatment with NPK. Physico-chemical properties of the soils such as pH, percent moisture content, %TOC and %TOM, all provided evidence of enhanced mineralization for the inorganic nutrient-supplemented (NPK-fertilizer-treated) soils. Therefore, if reclamation of the crude-oil-inundated soils is construed as the return to normal levels of metabolic activities of the soils, the application of the inorganic fertilizers at such prescribed levels would duly accelerate the remediation process. However, this would be limited to levels of pollution empirically defined by such a THC range (1.24×10^2 to 3.86×10^4 mg/kg) as obtained herein by the present investigation. Though the parameters investigated in this study do not provide any evidence of imposed toxicity by the supplements, post-germination tests and possible toxic residue analysis need to be carried out, perhaps for further studies, to adequately demonstrate this.

Acknowledgements

The authors wish to express their profound gratitude to the Management of Shell Petroleum Development Company of Nigeria (SPDC) Port Harcourt for the provision of unclassified data and that of the Department of Petroleum Resources (DPR) Port Harcourt at whose request the fieldwork was carried out.

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