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Physicochemical Analysis of Petroleum Sludge Impacted Soils After Remediation with Organic Waste

Useh Mercy Uwem[1,3], Dauda Mary Sunday[3], Abdulrahman Funke Wosilat[3] and Useh Uwem Jonah[2]*

[1]Chemistry Advanced Research Centre, Sheda Science and Technology Complex, Abuja, Nigeria

[2]Department of Pollution Control, Ecological Fund Office, Federal Secretariat Phase 2, Abuja, Nigeria

[3]Department of Chemistry, University of Abuja, Abuja, Nigeria

*Corresponding author: *Useh, Mercy Uwem*, E-mail: usehmercy@gmail.com

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ABSTRACT

For optimization of effective bioremediation processes, it is essential to consider environmental factors affecting the process. Petroleum sludge impacted soil samples from around Warri refinery, Delta State, Nigeria were assessed for some physicochemical parameters before and after remediation with agro-waste from *Moringa* seed. The remediation process was carried out for 90 days and samples were taken for analysis at 30 days interval. The analysis revealed a sinusoidal pattern of results at the end of the study period which indicated that most of the degradation activities took place within the first 30 days. More so, from the analyses after treatment of the samples with *Moringa Oleifera* seed cake (MOSC), the results as compared to the control, indicated that the agro-waste did not only add the needed nutrients to the soil, it was also able to mop-up certain minerals that could further endanger the use of the soil.

Keyword: *Physicochemical, contamination, Moringa seed, bioremediation, Niger Delta*

INTRODUCTION

The increase in demand for petroleum products resulted in an increase in their production, refining and transportation which in turn has resulted in gross contamination of the environment [1]. The society today is increasingly concerned about soil degradation, the sustainability of soil productivity, and maintenance of biodiversity. Petroleum sludge as a result of refining operations which is one of the major problems in the Niger Delta region of Nigeria due to improper ways of disposal has brought about adverse effects on the soil microflora [2]. Poor waste disposal of the petroleum sludge affect the physicochemical properties of soils with sometimes also the build-up of high concentrations of heavy metals in the affected sites [3,4]. Overall, the sources of livelihood in impacted areas which include farmlands, rivers and forests have been negatively affected [1,5]. Eco-tourism and conservation suffer significant losses as the release of complex hydrocarbons results in high mortality rates including the loss of unique plant/microbial species whilst the survival rate of local wildlife diminishes [6,7]. The Niger Delta covers approximately 70,000 km^2 and is home to over 40 ethnic groups. Hundreds of thousands of these people are affected by the resulting oil contamination near their homes. Especially affected are some 80 % of the region's inhabitants who have little money and rely on fishing and agriculture to survive [2,8,9]. The devastating consequences of oil spill especially in the Niger Delta region of Nigeria together with its eventual hazards on aerial, terrestrial and aquatic environs manifest as an irreversible chain effect on both the biodiversity and human safety [10]. The adverse effects of crude oil on soil cannot be overemphasized, upon decreasing the nitrogen and phosphorus contents, it causes excessive hydrocarbon content which affects soil enzymatic activities due to the inability of soil microbes to degrade the excess hydrocarbons [11,12,13]. Several methods have been employed to tackle the menace of oily sludge contaminated sites, but bioremediation method, among other treatment options is the most cost effective and environment friendly way of restoring contaminated soils [14,15]. Organic manures as well as plants materials have been used over time to carry out bioremediation which also have proven to be effective in improving soil quality [16,17,18,19]. For optimization of effective bioremediation processes, it is essential to consider environmental factors affecting the process. In other words, to assess the soil quality and fertility at each stage and the success of bioremediation, there is need to monitor the indicators reflecting the physical and chemical components of the soil [20,21]. In the Niger Delta region of Nigeria, there is not enough literature regarding the effect petroleum sludge on soil quality after remediation with organic waste like *Moringa Oleifera* seed cake (MOSC). This study therefore is aimed at providing information on the physicochemical properties of petroleum sludge impacted soils after remediation with locally available agro-waste from *Moringa* seed.

2 MATERIALS AND METHODS

2.1 Description of Sampling Sites

Delta State which is being nicknamed "The Big Heart of the Nation" lies approximately between Longitude 5°00 and 6°.45' East and Latitude 5°00 and 6°.30' North of the equator. It is located in the southern Nigeria with an area of 17,698 km^2 (6,833 sq mi) and a population of 4,112,445 as at 2006 [1], [22]. It is made up of 25 LGAs and comprising mainly five major ethnic groups: Urhobo, Isoko, Anioma and Ukwani, Ijaw and Itsekiri. Warri is the biggest commercial city in the state where the refinery is located. The major people in Warri comprise the Urhobos, Ijaws and Itsekiris [1],[16]. The oil spill impacted communities (Itsekiri) are situated between Latitudes 5^{0}30'N and 5^{0}33'N of the Equator and Longitudes 5^{0}45'E of the Prime Meridian, in Warri South Local Government Area of Delta State

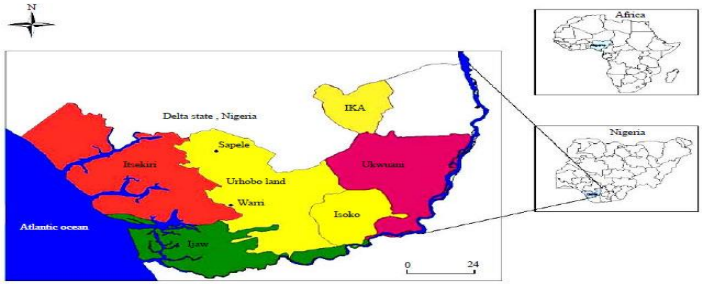

Figure 1: Map of Delta State showing the Study Area

2.2 Sample Collection, Handling and Preservation

US EPA (SW-846) guidelines were applied, using composite sampling for collecting sediment samples where sub-samples were collected from randomly selected locations in an area. Five (5) petroleum sludge samples were collected from the discharge pit of WRPC with core sampler in a 500 ml wide-mouth glass jar and pooled. Also, fifty (50) soil samples were randomly collected using soil auger from the depth of 0-15 cm from five selected oil-impacted communities (Ubeji – 500 m, Ekpan – 1.5 km, Aja-Etan – 2.5 km, Ifie-Kporo – 3.0 km, Ijala-Ikenren – 3.8 km from WRPC and were coded A, B, C, D and E respectively) and stored in sealed polythene bags. There were ten (10) replicates for each sampling site and the sub-samples were thoroughly mixed to obtain a representative sample of each. A control sample was also collected 8.5 km away from WRPC. These were stored in well-labeled amber glass bottles with teflon-lined screw cap, held at 4°C immediately in a cooler of ice and transported to the laboratory for pre-treatment and analyses [1],[23],[24]. On reaching the laboratory, stones and debris were removed and the samples were used as arrived for the treatment with agricultural waste from *Moringa Oleifera* seed. All analyses were carried out in triplicates to minimize error.

2.3 Preparation of Moringa Oleifera Seed Cake (MOSC)

The *Moringa Oleifera* (MO) seed pods were purchased from Kubwa market, FCT, Abuja, Nigeria. They were dehusked and pulverized. The oil in the seed was extracted by hexane using soxhlet extraction method. About 100 g of MO seed powder were poured into an extraction thimble. 1 L of hexane solvent was poured into a round bottom flask. After setting up the soxhlet apparatus, it was heated for 1 hr and the oil was extracted. After extraction, the seed cake was sun dried and pulverized. It was then stored in a polyethylene container [25],[26].

2.4 Sample Treatment

The samples used for the study were field-moist soil samples, no air-dried material was used. With the soil samples, the agro-waste from *Moringa* seed used as organic fertilizer was added to enhance biodegradation of the contaminant, and the whole mixture was mixed using a mixer. The ratio of organic fertilizer to contaminated soil was one part (20 g) of fertilizer to three parts (60 g) of soil [27],[28]. The treated soils were kept under controlled humidity 60 % of F.C. (field capacity), in the ambient laboratory conditions with temperature (28 ± 4 °C), under subdued light to serve as abiotic factors. The rate of biodegradation was studied as a function of time. The contents of the experiment were manually mixed twice a week to allow aeration and homogenous mixture of the materials [29],[30].

At 30 days interval, samples were collected and air-dried for two weeks at ambient temperature, rolled manually with a steel roller, sieved to remove stones and debris. These were further grounded with mortar and pestle until very fine fraction was achieved, it was sieved through a 2-mm stainless steel mesh to get a test sample of <2 mm fraction. Both devices were cleaned after each sample had been processed to avoid cross-contamination. These were properly stored in well-labeled air-tight containers until analysis [1].

2.5 Determination of Soil Physicochemical Properties

pH was determined for all samples by using 1:2 slurry of 10 g sediment samples with 20 ml deionised water. After 10 minutes, pH was determined using a digital pH meter (Jenwaymodel 3015) with a glass-calomel electrode combination. Conductivity measurements were determined on fresh sediment samples using a conductivity meter (Systronics-304) at 25°C. The moisture content was determined by the gravimetric method. Soil organic carbon was determined using a modified dichromate wet oxidation method (Walkley-Black (WB) procedure) which measures the active, or decomposable organic matter in the soil samples. The organic matter content in the soils was determined by multiplying the organic carbon content from the procedure above by 1.729 (using the assumption that organic matter contains approximately 58% carbon) [31]. The apparatus for "0 Bar" water holding capacity method was used to determine the water holding capacity. Sulphate was determined by a gravimetric method which involved the use of an excess amount of barium chloride solution. Total nitrogen in the soil samples was determined using the macro Kjeldahl's method. Available phosphorus was determined by Bray No. 1 method. Sodium and Potassium were determined using a flame photometer (Sherwood Model 410) after due calibration. Calcium and Magnesium were determined using atomic absorption spectrophotometer (AAS) iCE 3000 Series 3000 at their respective wavelength. Oil and grease by gravimetric method also. All these were done following the standard protocols and methods of American Public Health Organization (APHA) [31,32,33]

2.6 Statistical Analysis

Data analysis involved simple descriptive and univariate summary statistics such as mean, standard deviation and percentage. The physicochemical parameters were the main index for evaluating success of biodegradation in the different soil samples. Hence, the data were subjected to analysis of variance (ANOVA) to compare the variability in the parameters in the different soil samples over time. All the statistical analyses were performed using statistical software SPSS Windows version 16.0 [1].

2.7 Quality Assurance

To ensure that the results were accurate, reliable and reproducible, strict adherence to the standard operating procedures and precautions were ensured at all levels. Also, reagent blank determinations were used to correct the instrument readings.

3. RESULTS AND DISCUSSION

The results of the physicochemical properties (Table 1) of the agro-waste [*Moringa Oleifera* seed cake (MOSC)] showed a total nitrogen content of 10.28 ± 0.15 %, potassium content of 12.87± 1.13 mg/kg while the phosphorus content was 19.15 ± 0.01 %. The presence of these limiting nutrients (N and P) in the agro-waste sample analyzed in this study is in consonance with the earlier reports of [17,21,28]. There were also appreciable levels of other trace elements. The physicochemical properties of the impacted soil samples before and after amendment with the MOSC for the 90 days period are recorded in Tables 1 – 4. The results showed a rise in pH at the on-set of the remediation process. The pH maintained a steady increase up to day 60, after which there was a decline at the end of the study period. Similar observations have been documented [34,35]. The pH of all the treated samples ranged

from 6.20± 0.05 to 6.57±0.07 at day 30, 6.50±0.08 to 6.65±0.01 at day 60 and 6.50±0.02 to 6.63±0.01at day 90 while that of the control ranged from 7.81 at day 1 to 7.95 at the end of the study period. However, this pH range fell within the optimum pH required for effective bioremediation process [37,38].This is suggested to be one of the conditions that increased the rate of biodegradation of PHC in amended soil since crude oil degrading microbes grow and make perfect use of hydrocarbons at slightly alkaline pH. The soil electrical conductivity in the remediated samples were observed to reduce significantly with the extension of remediation time, indicating a reduction in the concentration of soluble salts present. The mean moisture contents recorded in the remediated

Table 1: Selected physicochemical parameters of the samples at day 1

PARAMETERS	SITE A	SITE B	SITE C	SITE D	SITE E	CONTROL	MOSC
pH	5.31± 0.25	5.35±0.62	5.42±0.03	5.47±0.51	5.54±0.01	7.81±2.01	7.90± 0.03
Conductivity (µs/cm)	0.57± 0.02	0.56±0.02	0.48±0.08	0.50±0.01	0.44±0.00	0.26±0.03	ND
Moisture content (%)	1.85± 0.03	3.22±0.83	2.63±0.01	3.41±0.01	3.75±1.30	5.45±0.02	35.00± 0.01
Organic carbon (%)	18.22± 0.13	16.51±0.37	15.53±0.05	13.29±0.16	10.02±1.92	4.25±1.04	1.08± 0.31
Organic matter (%)	31.50±0.01	28.55±0.05	26.85±1.41	22.98±0.03	17.32±0.17	7.35±0.02	1.87±0.10
Sulphate (mg/l)	463.94 ± 1.06	465.13±1.25	429.76±0.01	420.65±0.06	412.73±1.00	56.73±1.13	NA
Water holding Capacity (%)	38.00 ± 0.15	45.47±0.01	50.28±0.04	57.10±0.26	58.62±1.91	65.10±0.43	NA
Available Phosphorus (%)	8.72± 0.01	10.59±1.33	10.86±0.20	12.28±1.37	12.40±1.85	15.62±1.11	19.15 ± 0.01
Sodium (mg/kg)	58.39 ± 0.00	54.20 ±1.00	47.65 ±0.01	45.17 ±2.01	36.15 ±0.01	21.76 ±0.01	6.03 ± 0.10
Potassium (mg/kg)	1.36 ± 0.00	1.59 ±0.10	1.84 ±0.01	2.35 ±0.01	2.78 ±0.00	5.00 ±0.20	12.87± 1.13
Calcium (mg/kg)	ND	ND	ND	1.62±0.08	ND	3.68±0.01	128.56±0.01
Magnesium (mg/kg)	231.24±0.11	193.62±0.03	158.71±1.00	160.45±1.03	137.28±0.02	65.94.±1.02	26.14±0.06
Total Nitrogen (%)	0.08± 0.00	0.26±0.01	0.35±0.01	0.37±0.02	0.44±0.00	0.87±0.01	10.28 ± 0.15
Oil and grease (mg/kg)	587642.0± 0.27	247698.0±2.33	194532.0±0.04	175072.4±1.16	96077.2±0.45	1032.4±0.18	NA

NA = Not applicable, ND = Not detected. The results are means of triplicate determination ± standard deviation

Table 2: Selected physicochemical parameters of the Soil samples after 30 days

PARAMETERS	SITE A	SITE B	SITE C	SITE D	SITE E	CONTROL
pH	6.20± 0.05	6.21±0.15	6.25±0.01	6.53±0.32	6.57±0.07	7.87±0.12
Conductivity (µs/cm)	0.42± 0.01	0.41±0.01	0.38±0.00	0.35±0.03	0.34±0.00	0.22±0.02
Moisture content (%)	23.24± 0.06	23.57±0.01	23.31±0.05	23.48±2.01	23.82±1.02	26.48±0.00
Organic carbon (%)	10.52± 0.10	9.76±1.31	9.08±0.02	8.24±0.10	6.92±0.01	2.43±1.00
Organic matter (%)	18.19±0.00	16.88±1.00	15.70±0.25	14.25±0.01	11.96±0.66	4.20±0.73
Sulphate (mg/l)	1.62±0.00	1.54±0.00	NA	NA	NA	NA
Water holding Capacity (%)	41.00 ± 1.03	50.83±1.19	54.63±0.59	59.32±0.00	60.88±1.36	68.47±0.43
Available Phosphorus (%)	10.45± 0.06	11.92±0.00	12.82±0.01	14.85±1.81	15.17±0.05	18.84±0.01
Sodium (mg/kg)	41.72± 0.13	38.89±0.74	32.42±0.01	32.18±0.01	24.35±0.00	14.78±0.01
Potassium (mg/kg)	3.45± 0.32	3.72±0.11	3.87±0.09	5.53±0.02	5.48±1.00	16.21±0.04

Calcium (mg/kg)	3.00±0.01	3.11±0.00	3.39±0.01	6.23±0.00	5.10±0.00	12.47±0.00
Magnesium (mg/kg)	187.06±0.07	152.58±0.45	121.13±0.00	120.39±0.01	117.18±0.76	42.61±0.01
Total Nitrogen (%)	1.13± 0.00	1.30±0.01	1.38±0.00	1.43±0.01	1.52±0.00	2.94±0.05
Oil and grease (mg/kg)	245307.8± 0.06	128573.1±0.18	82745.2±0.01	54371.0±1.05	25468.3±0.00	418.0±0.02

Table 3: Selected physicochemical parameters of the Soil samples after 60 days

PARAMETERS	SITE A	SITE B	SITE C	SITE D	SITE E	CONTROL
pH	6.52± 0.00	6.50±0.08	6.56±0.01	6.58±0.02	6.65±0.01	7.92±0.31
Conductivity (µs/cm)	0.37± 0.01	0.38±0.02	0.34±0.00	0.33±0.00	0.30±0.00	0.20±0.00
Moisture content (%)	23.29± 0.00	24.05±0.03	23.57±0.01	23.86±0.01	24.21±1.00	26.77±0.20
Organic carbon (%)	7.43± 0.17	6.28±0.02	7.35±0.12	6.50±0.15	3.01±0.01	2.11±0.00
Organic matter (%)	12.85±0.42	10.86±1.10	12.71±0.00	11.24±0.61	5.20±0.30	3.65±0.01
Sulphate (mg/l)	NA	NA	NA	NA	NA	NA
W H C (%)	46.36 ± 1.00	52.07±1.07	55.81±0.42	61.77±1.00	63.03±1.20	68.59±0.34
Available Phosphorus (%)	10.48± 0.04	11.97±0.10	12.89±0.22	14.88±1.00	15.26±0.15	18.86±0.41
Sodium (mg/kg)	35.83±0.01	33.10±0.01	30.17±0.30	27.92±1.00	21.01±0.01	10.65±0.03
Potassium (mg/kg)	5.63±1.04	5.88±0.01	5.92±0.00	6.84±0.03	7.73±0.02	19.23±0.01
Calcium (mg/kg)	3.25±0.01	3.54±0.12	4.86±0.00	8.21±0.01	7.03±0.12	15.05±0.03
Magnesium (mg/kg)	155.85±0.46	136.94±0.44	114.95±0.49	110.19±0.10	108.71±0.06	28.34±0.04
Total Nitrogen (%)	1.20± 0.00	1.35±0.07	1.44±0.20	1.47±0.00	1.57±0.30	3.96±0.01
Oil and grease (mg/kg)	51643.2± 0.01	33592.1±2.05	26484.0±0.01	1522.4±1.00	1193.5±0.31	137.2±0.10

Table 4: Selected physicochemical parameters of the Soil samples after 90 days

PARAMETERS	SITE A	SITE B	SITE C	SITE D	SITE E	CONTROL
pH	6.50± 0.02	6.50±0.01	6.53±0.11	6.59±0.02	6.63±0.01	7.95±0.03
Conductivity (µs/cm)	0.32± 0.00	0.30±0.00	0.30±0.01	0.28±0.00	0.27±0.00	0.13±0.00
Moisture content (%)	23.30± 0.01	24.03±0.00	23.58±0.00	23.88±0.01	24.24±0.04	26.82±0.21
Organic carbon (%)	5.48± 0.10	5.13±0.01	4.99±0.04	4.38±0.12	2.47±0.01	1.94±0.00
Organic matter (%)	9.47±0.08	8.87±1.00	8.63±0.00	7.57±0.42	4.27±0.80	3.68±0.00
Sulphate (mg/l)	ND	ND	ND	ND	ND	ND
Water holding Capacity (%)	46.27 ± 1.40	51.85±1.02	55.56±0.22	61.73±1.90	60.00±1.10	68.50±0.44
Available Phosphorus (%)	10.45± 0.01	11.90±0.05	12.82±0.10	14.87±0.00	15.25±0.13	18.89±0.25
Sodium (mg/kg)	35.85±0.10	32.97±0.00	30.14±0.01	26.87±0.02	17.03±0.01	10.48±0.00
Potassium (mg/kg)	5.60±0.01	5.87±0.00	5.90±1.00	6.85±0.14	7.75±0.00	20.15±0.10
Calcium (mg/kg)	3.28±0.00	3.60±0.01	5.02±0.20	8.25±0.33	7.24±1.00	26.11±0.00
Magnesium (mg/kg)	148.64±0.48	131.89±0.46	109.83±0.26	107.06±1.47	103.39±0.31	17.15±0.48
Total Nitrogen (%)	1.18± 0.00	1.34±0.01	1.40±0.00	1.45±0.00	1.52±0.00	3.98±0.16
Oil and grease (mg/kg)	1015.4± 0.01	895.6±2.01	320.4±0.01	104.7±1.10	88.6±0.05	72.5±0.10

soil samples experienced a drastic increase after the first 30 days (1.85± 0.03 to 3.75±1.30 from site A to E at day 1, 23.24± 0.06 to 23.82±1.02 from site A to E at day 30) which got reduced towards the end of the study, although these were within the optimum range for microbial activity. This may be due to regular addition of water to the experiment to retain 60-70 % moisture to make sure the moisture content was not below the recommended range for microorganisms. The organic carbon and organic matter was observed to consistently decrease upon addition of the bio-waste material over time. This could be attributed to the stimulation of microorganisms by the addition of nutrients to the contaminated soils. The microorganisms utilized the nutrients for their growth leading to the rapid depletion of the available pools of major inorganic nutrients while degrading the hydrocarbons by utilizing it as its energy source. This is in line with the observation by Jiang *et al.*,[39]. This observation also agrees with the findings of Ekundayo and Obuekwe [6] who noted increment of organic carbon content of crude oil contaminated soils in Southern Nigeria. It may also be related to slow decomposition rate of the amendment by soil organisms since contamination of the soil with petroleum sludge might have resulted in poor soil aeration. It was also observed that after addition of activated copper to the contaminated samples, sulphate was no more available in the subsequent analysis except minimal concentrations in site A and B at day 30, which showed that desulphurization had taken place. Total nitrogen and available phosphorus was seen to decrease as each site is closer to the refinery (Table 1). At day 30 after remediation, it was observed that the N and P contents of the samples were improved. This improvement was consistent at day 60, but at the end of the treatability studies, a decline in the values of N and P was observed (Tables 2, 3 and 4). Nitrogen is the most commonly limiting factor to biological degradation of hydrocarbon in soils according to Pond *et al.,* [40]. N and P addition could stimulate the microbial activity, and increase the degradation rate of hydrocarbon. The agro-waste (MOSC) which is rich in these limiting nutrients (N and P) was able to supply the needed ingredients to complement the already available carbon. This provided the optimum condition for the soil microbes, but with time, the low bioavailability of contaminant and the accumulation of recalcitrant compounds could inhibit the microbial biodegradation ability which led to a decrease in N and P values at day 90. This is in agreement with earlier researchers [29, 41] who noted that the addition of these limiting nutrients is a key factor in achieving effective biodegradation of hydrocarbons. When an oil spill occurs, the result is a vast increase in carbon and this also stimulates the growth of the already present oil degrading microorganisms. However, these microorganisms are limited in the level of growth and remediation that can occur by the available nitrogen and phosphorous. So by adding these supplemental nutrients in the proper concentrations, the hydrocarbon degrading microbes are capable of achieving their maximum rate of contaminant uptake. Also in an earlier report by Emmanuel *et al*., [42], it was stated that the MOSC has greater support for microbial growth. The oil and grease contents were observed to decrease consistently upon addition of the bio-waste material over time. The changes in the ionic contents of K, Na, Ca and Mg are recorded in Tables 2, 3 and 4. There were no calcium at day 1 in the tested soil samples except in site D (Table 1), however, the addition of organic fertilizer improved the calcium content of the soil as shown in Tables 2, 3 and 4. This is at variance with the work of another researcher Emmanuel *et al*., [42] who reported that MOSC scavenged the calcium content of the soil. At day 1 when no MOSC was added, potassium ion content in general decreased with increasing proximity of each site to the refinery. The reduction in potassium content may be due to nutrient immobilization consequent to the formation of complexes in the soil. After contaminant degradation, the potassium value was seen to increase in all the sites to day 60, which also became reduced at the end of the treatability studies unlike the control indicating that addition of organic fertilizer (MOSC) improved the potassium content of the soil which in turn was used up by the soil organisms [Table 4]. This is in agreement with Shukry *et al.,* [43] who reported that potassium ion content decreased with increasing crude oil concentration in the soil. There was an observed increase in sodium content at day 1 in sites closer to the refinery. This may be associated with instability of soil physical structures, caused by the petroleum sludge contamination. From the analyses after treatment of the samples with MOSC, the results as compared to the control, indicated a decrease in the sodium content of the samples which in turn improved the soil quality. In the same manner, the magnesium content was higher in day 1 according to proximity of each site to the refinery, but it was seen to decrease consistently after treatment with MOSC. This revealed that the agro-waste did not only add the needed nutrients to the soil, it was also able to mop-up certain minerals that could further endanger the use of the soil.

4 CONCLUSION

Over the years, several physical and chemical models have been used to make estimates of the burden of environmental degradation. Understanding the magnitude and future trends of the petroleum sludge impact is necessary prerequisite for proper planning and mobilization of resources for its clean-up and remediation. The results showed remarkable success in using *Moringa Oleifera* seed cake for remediation of such sites, and also resounding improvement in the soil quality which in turn will improve the yield of crops. This is shown in the level of the nutrients available in the remediated soils and the control soil and also the improved levels of the studied physicochemical parameters at the end of the study. This work is recommended to all involved in the commendable work of environmental safety and sustainability especially cleaning up and remediation of oil spill contaminated sites in the Niger Delta Region of Nigeria.

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