



Physicochemical and Bacteriological Analysis of Sludge and Water Contaminated by Oil Spillage in Some Coastal Communities of Akwa Ibom State, Nigeria

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Abstract: Crude oil contaminated sludge and water samples from four coastal communities (Ibendo, Onna, EsitEket and Eket) in Akwa Ibom State, Nigeria were analyzed to investigate the bacterial load and physicochemical parameters using standard pour plate method and standard analytical methods respectively. The pH values of both (sludge and water) samples as determined ranged from 5.14 – 5.22 and 4.23 – 6.15 respectively which indicated that the samples are acidic in nature. Other physicochemical parameters determined were: temperature, electrical conductivity, moisture content, organic matter, total alkalinity, TDS, TSS, total hardness, DO, BOD, sulphate, chloride, oil and grease. The bacteriological analysis of the water and sludge samples showed mean total bacterial count of 3.57×10^4 and 1.77×10^3 cfu/ml respectively. The mean coliform counts of water and sludge samples were 1.5×10^2 and 8.4×10^1 cfu/100ml respectively. The result of these analyses showed that most of these parameters did not meet the WHO recommended standards. Hence, the environment should be remediated with immediate effect to avoid further disaster.

Keywords: Crude Oil, Akwa Ibom, Bacteriological Analysis, Water, Sludge, Physicochemical Parameters

1. Introduction

Since commercial exploration of petroleum started in Nigeria in 1958, the oil industry in Nigeria has completely restructured the country's economy. However, this exploration has led to the pollution of lands and waterways [1]. Crude oil contamination from drilling processes creates problems that disrupt the lives of people living in close proximity to oil camps, wells, pumping stations, and pipelines. People living on oil-rich sites around the world are subjected to contamination of drinking water, topsoil, and livestock due to toxic pollution that results from the oil extraction process. In some contaminated sites, serious illnesses resulting from exposure have been documented. Multiple studies have been done that compared cancer rates

of people living in close proximity to oil contaminated sites to those who live in unaffected regions. Most of these studies conclude that there is a correlation between the proximity of the subject to the contamination and an increased cancer rates. The water, soil and air have been severely tainted by petroleum pollutants [1, 2].

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. The Niger Delta covers approximately 70,000 km² 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. The agricultural lands have become less productive, the creeks and fishing waters have become extremely vulnerable to damaging effects of oil pollution. Some of the major factors associated with accelerating pace of oil spill are accidental damage of pipes and tankers (50%), sabotage (28%), oil production operations (21%) and poor maintenance of infrastructure (1%) [3].

Many locals in the region attest to environmental damage that allegedly developed as a result of the leak. Oil spills in the delta's creeks have been left to fester for decades, polluting the air, soil, and water of impoverished communities. Nigeria sees its future output growth largely in offshore fields and does not want spills there to compound its environmental woes. The Nigerian Environmental Rights Action group issued a demand for N51 billion (\$100 Million) from Exxon Mobil in Nigeria for their failure to compensate fisher men within the coastal areas who suffered devastating losses due to the oil company's exploration activities and major oil spills. Thick balls of tar have also been sighted washed upon the shore line as well as oil slicks. The spill has only exacerbated the already growing problem of pollution in the Delta. The Nigerian government estimates there were over 7,000 spills, large and small, between 2000 and 2012, according to the BBC. Vast swathes of the Delta have been seen covered with tar and stagnant lakes of crude due to oil spills of the past [4].

Environmental Rights Action / Friends of the Earth Nigeria (ERA/FoEN) visited Ibene Community on the Atlantic coastline of Akwa Ibom State in 2014, following reports of an oil spill from the Qua Iboe Oil Fields operated by Mobil Producing Nigeria (MPN). Two major oil spills were said to have also occurred within the Qua Iboe Oilfield. It has been a string of woes for the coastal communities. Though MPN, an affiliate of Exxon Mobil had in a statement admitted its facility caused the spill which it put at some 200 barrels of crude oil, community members told ERA/FoEN field monitor that the latest incident is one among several spills that they said, was injurious to aquatic life which they depend on for their livelihood. During the visit, members of the community were seen in groups at the coastline bemoaning what had befallen them and its effect on their fishing vocation. ERA/FoEN monitor observed massive crude oil deposits on the shoreline as well as fishing boats and nets stained with crude oil deposits [5].

Crude oil if spilled into the water and soil spreads over a wide area forming a slick and the environment immediately begins to undergo a variety of physical, chemical and biological changes including evaporation of high volatile fractions, dissolution of water-soluble fractions, photochemical oxidation, emulsification, microbial degradation and sedimentation. Water pollution occurs when crude oil spills directly or indirectly into the water body resulting in unfavorable condition in which safety and welfare of any living organism is endangered. Several civil unrests due to environmental degradation from oil exploration is reported in the tropics, especially in Ibene,

Onna, Esit Eket and Eket LGAs of Akwa Ibom State, Nigeria, therefore the release of crude oil into the environment by oil spill is receiving worldwide attention [6]. Since the commencement of oil exploration activities in these areas, only scanty data are available for levels of chemical pollution brought about by these activities. Secondly, in spite of various efforts to bring development to all parts of the LGAs by the government, there are immense difficulties arising from the absence of a coherent body of information and data on which to base intervention decisions. Thus the main aim of this research is to investigate the physicochemical properties and bacterial load of crude oil contaminated water samples and sludge collected from Ibene, Onna, Esit Eket and Eket LGAs of Akwa Ibom State, Nigeria.

2. Materials and Methods

Sample Collection, Preparation and Preservation

A total of twenty four (24) samples were used for the present study. Twelve (12) sludge samples were randomly collected using soil auger from four (4) oil-impacted areas; Stubbs Creek in Ibene LGA, Ikot Ebidang in Onna LGA, Etebi in Esit Eket LGA and Esuk Odio in Eket LGA all in Akwa Ibom State, Nigeria. The samples were taken from three (3) locations in each site, stored in sealed polythene bags properly labeled and transported to the laboratory for pre-treatment and analyses. The sludge samples were air-dried for two weeks, rolled manually, mixed and sieved with 2 mm mesh to remove stones and debris [7]. Twelve (12) water samples were collected in the said areas in well-labeled polyethylene containers which were prewashed and soaked in 1 M HNO₃ for 24 hours and later rinsed with deionised water. The samples were transported to the laboratory in a cooler of ice to ensure rapid cooling and were protected from direct sunlight during transportation. Uncontaminated samples were used as control. They were further preserved in the refrigerator prior to analyses [8].

3. Physicochemical Analysis

Soil pH was determined for all samples by using 1:2 slurry of 10 g sludge sample with 20 ml deionised water. After 10 minutes, pH was determined using a digital pH meter and electrical conductivity measurement was determined at 25°C using conductivity meter (Systronics-304). The sludge moisture content was determined by gravimetric method in which 2.0 g of each sample was dried in an oven at 105°C to constant weight. The difference in weight gives the moisture content. Organic matter was determined using Loss-On-Ignition (LOI) method which involved the heated destruction of all organic matter in the sludge sample. A known weight of sample was placed in a ceramic crucible which was then heated to 400°C overnight. The sample was then cooled in a desiccator and weighed. Organic matter content was calculated as the difference between the initial and final sample weights divided by the initial sample weight times

100%. Total alkalinity was determined by titrimetric method in which 100 ml of distilled water was added to 20 g of the sludge sample and stirred for one hour at regular intervals using magnetic stirrer. The suspension so obtained was filtered and the filtrate was titrated with a standard solution of HCl using phenolphthalein and methyl orange as indicators. Sulphate was determined by gravimetric method which involved the use of excess amount of barium chloride solution. Sulphate was precipitated as barium sulphate and the precipitate was filtered, washed, dried, ignited and weighed. Chloride was determined by Mohr's method in which the filtrate was titrated with a standard solution of silver nitrate using potassium chromate as indicator. Then oil and grease by gravimetric method also.

The temperature of the water samples were determined in-situ using the mercury-in-glass thermometer. pH was determined using a Pocket Digital pH Meter and electrical conductivity was measured at 25°C using conductivity meter (Systronics-304). All these were recorded at the site of sample collection [7, 8]. Other parameters carried out in the laboratory include: chloride by Argentometric method as stated above, total hardness was determined by titrimetric method in which 1 ml of a buffer solution was added to 50 ml of each of the water samples using Eriochrome black T-NaCl mixture as indicator. When the sample turned red, it was titrated with EDTA solution until the solution turned blue, then hardness was calculated in terms of mg of CaCO_3/L . Total dissolved solids was determined by gravimetric methods, alkalinity by titrimetric method in which 100 ml of the sample was used following the same procedure as stated above, total suspended solids was determined by filtration method, dissolved oxygen was determined by Winkler's method in which 100 ml glass-stoppered conical flask each was filled with the water sample and was treated with 1 ml each of alkaline KI and MnSO_4 solution. The contents were thoroughly shaken and the precipitates were allowed to settle for sometimes. 1 ml of concentrated H_2SO_4 was added and titrated with sodium thiosulphate solution using starch as indicator. Biochemical oxygen demand was determined by APHA method which involved the measurement of the oxygen concentrations of the samples immediately and that after incubating it for five days [8].

4. Bacteriological Analysis

The total bacterial count was determined by pour plate technique using standard methods. Nutrient agar medium was used for the enumeration of bacteria in the samples. Total coliform count was determined by MPN index method by employing 3-3-3 regimen using macConkey broth and negative result was indicated since there was no acid and gas production on incubation at 37°C for 48 hours [9].

5. Statistical Analysis

All the determinations were conducted in triplicates and

data generated were analyzed statistically by one-way analysis of variance (ANOVA) technique using (SPSS) 16.0.

6. 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.

7. Results and Discussion

The results of the analyses are presented in tables 1 to 3. From tables 1 and 2, the pH of the sludge samples ranged from 5.14–5.22 with a mean value of 5.18 ± 0.04 while that of water samples ranged from 4.23–6.15 with a mean value of 5.08 ± 0.84 which are below the WHO standard range of 6.50 to 8.50. These pH values indicated that the samples are acidic which is not good for human consumption. It equally showed a slight decrease from acceptable fish/aquatic lives survival limit [10]. From Table 1, the moisture content ranged from 0.2 to 1.0% with an average value of $0.60 \pm 0.34\%$. Therefore, the weight of the sludge samples used for the digestion was taken as absolute value, since percentage moisture content makes practically no difference [11]. From table 1, the organic matter is relatively high ranging between 5.17 and 8.24%. This could be attributed to the presence of the crude oil samples available in the sorption process. Also, the presence of crude oil on the soil medium created an anaerobic condition within the medium. This condition led to the death of most aerobic micro organisms which formed part of the organic substances that added to the organic matter content of the soil. The % organic matter obtained here was within the ranges observed for some tropical soil type [12, 13, 14, 15].

The electrical conductivity recorded for the sludge samples ranged from 0.23–0.41 $\mu\text{S}/\text{cm}$ with an average value of $0.31 \pm 0.08 \mu\text{S}/\text{cm}$ while that of the water samples ranged from 0.24–0.57 $\mu\text{S}/\text{cm}$ with an average value of $0.45 \pm 0.14 \mu\text{S}/\text{cm}$. These values were below the recommended WHO standard [16]. The total alkalinity indicates the capacity of the sample to neutralize acids. A sample may be alkaline due to the presence of free OH^- ions and also certain salts like carbonates, bicarbonates, borates and silicates which produce alkalinity because of hydrolysis. However, the contribution of carbonates and bicarbonates is a major source of alkalinity. The average alkalinity values of 0.83 ± 0.16 and $0.50 \pm 0.12 \text{ mg/l}$ were recorded for the sludge and water samples respectively. These values were below the highest desirable level of 200 mg/l recommended by WHO [16].

Sulphates are found appreciably in all natural waters, particularly those with high salt content. Besides industrial pollution and domestic sewage, biological oxidation of reduced sulphur species also adds to sulphate content. Soluble in water, it imparts hardness with other cations. Sulphate causes scaling in industrial water supplies, and

odour and corrosion problems due to its reduction to hydrogen sulphide. The presence of chlorides in natural waters can mainly be attributed to dissolution of salt deposits in the form of ions (Cl^-). It is the major form of inorganic anions in water for aquatic life. High chloride content has a deleterious effect [17, 18]. The concentrations of major anions (sulphate and chloride) in the sludge samples obtained

ranged from 10.48–15.13 mg/l and 264–318 mg/l with mean concentrations of 12.21 ± 2.02 mg/l and 295.75 ± 23.75 mg/l respectively. The chloride concentration of the water samples ranged from 191–285 mg/l with an average value of 252.25 ± 42.2 mg/l. It is obvious that the chloride concentration at each site exceeds the WHO permissible limit except the water sample from Eket site.

Table 1. Results of some physicochemical parameters of the sludge samples.

PARAMETERS	IBENO	ONNA	ESITEKET	EKET	MEAN \pm S.D	RANGE
pH	5.16	5.19	5.22	5.14	5.18 ± 0.04	5.14–5.22
Conductivity ($\mu\text{S}/\text{cm}$)	0.25	0.23	0.41	0.35	0.31 ± 0.08	0.23–0.41
Moisture content (%)	0.70	1.00	0.20	0.50	0.60 ± 0.34	0.20–1.00
Organic matter (%)	6.83	8.24	5.17	6.62	6.72 ± 1.26	5.17–8.24
Total alkalinity (mg/l)	0.72	1.06	0.75	0.80	0.83 ± 0.16	0.72–1.06
Sulphate (mg/l)	11.54	15.13	10.48	11.67	12.21 ± 2.02	10.48–15.13
Chloride (mg/l)	309	292	318	264	295.75 ± 23.75	264–318
Oil and grease (mg/l)	15869	15524	16231	15745	15842 ± 295.8	15524–16231

Table 2. Results of some physicochemical parameters of the water samples.

PARAMETERS	IBENO	ONNA	ESITEKET	EKET	MEAN \pm S.D	RANGE
pH	4.23	6.15	5.31	4.62	5.08 ± 0.84	4.23–6.15
Conductivity ($\mu\text{S}/\text{cm}$)	0.24	0.57	0.48	0.51	0.45 ± 0.14	0.24–0.57
Temperature ($^{\circ}\text{C}$)	29.5	29.8	29.6	29.9	29.7 ± 0.18	29.5–29.9
Cl^- (mg/l)	274	285	259	191	252.25 ± 42.2	191–285
Total hardness (mg/l)	147	152	135	139	143.25 ± 7.68	135–152
TDS (mg/l)	1659	1549	1582	1594	1596 ± 46.12	1549–1659
Total alkalinity (mg/l)	0.56	0.32	0.55	0.58	0.50 ± 0.12	0.3–0.58
TSS (mg/l)	1508	1476	1471	1485	1485 ± 16.39	1471–1508
DO (mg/l)	1.77	1.59	1.92	1.53	1.70 ± 0.18	1.53–1.92
BOD (mg/l)	194	188	190	197	192.25 ± 4.03	188–197
Oil and grease (mg/l)	956	563	748	759	756.5 ± 160.5	563–956

Table 3. Bacteriological analysis results.

SITES	SAMPLE	TOTAL BACTERIAL COUNT (cfu/ml)	TOTAL COLIFORM COUNT (cfu/100ml)
IBENO	Water sample	1.05×10^4	2.8×10^1
ONNA	Water sample	6.74×10^4	4.0×10^2
ESIT EKET	Water sample	2.50×10^4	3.5×10^1
EKET	Water sample	4.00×10^4	1.4×10^2
MEAN		3.57×10^4	1.5×10^2
IBENO	Sludge sample	1.25×10^3	1.7×10^1
ONNA	Sludge sample	2.11×10^3	1.35×10^2
ESIT EKET	Sludge sample	2.41×10^3	1.05×10^2
EKET	Sludge sample	1.30×10^3	8.0×10^1
MEAN		1.77×10^3	8.4×10^1

Total hardness is the sum of calcium and magnesium concentrations, both expressed as CaCO_3 in mg/L. The total hardness of the water samples ranged from 135 to 152 mg/l with a mean concentration of 143.25 ± 7.68 mg/l indicating that the water is hard [19]. Waters with high dissolved solids generally are of inferior palatability [20]. The total dissolved solids of the water samples ranged from 1549–1659 mg/l which is very high while the total suspended solids ranged from 1471–1508 mg/l with an average concentration of 1485 ± 16.39 mg/l. Water with high-suspended solids as seen here is unsatisfactory for bathing, industrial and other purposes.

Oxygen dissolved in water serves as an indicator of the physical, chemical and biological activities of the water body and it is considered to be the major limiting factor in water bodies with organic materials [21]. The average DO recorded

was 1.70 ± 0.18 mg/l which was very minimal and this could be attributed to the consumption of dissolved oxygen by bacteria feeding on the spilled hydrocarbons which also contributes to the death of fish. Biological Oxygen Demand (BOD) test is used to determine the pollution load of water samples [21, 22]. The BOD ranged from 188–197 mg/l with a mean concentration of 192.25 ± 4.03 mg/l which signified that water is greatly polluted. From table 1 and 2, the mean concentrations of oil and grease in the soil samples is 15842 ± 295.8 ml/l while that of the water samples is 756.5 ± 160.5 mg/l. This showed that the environment is highly contaminated. The bacteriological analysis of the water and sludge samples showed total bacterial count of 3.57×10^4 and 1.77×10^3 cfu/ml respectively. The mean coliform counts of water and sludge samples were 1.5×10^2 and 8.4×10^1 cfu/100ml respectively. These values are very

much higher than the WHO standards for drinking water.

8. Conclusion

Over the years, several statistical models have been used to make estimates of the burden of environmental degradation. Understanding the magnitude and future trends of the oil spillage is necessary prerequisite for proper planning and mobilization of resources for its clean-up and remediation. The results showed marked variation in characteristics in all the 4 locations or sites. The degree of contamination for the four identified polluted sites has been estimated following a rigorous quality control procedure. This work is recommended to all involved in the commendable work of environmental safety and sustainability especially cleaning up and remediation of oil spill polluted sites in the Niger Delta Region of Nigeria.

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