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SOIL AND WATER CONTAMINATION DUE TO ILLEGAL ARTISANAL REFINERY ACTIVITIES: A CASE STUDY OF OKARKI COMMUNITY, NIGER DELTA AREA, NIGERIA

Kwatamshuwa Mwada GWARY[1], Usman Abubakar ZARIA[2], Muhammed Sani GALADIMA[3], Bashir Hasan DIYA'UDDEEN[4]

Research article

Abstract: Using a combination of field visits, laboratory experiments and analyses, this study examined the impacts of illegal artisanal crude oil refineries on the environment. Total Hydrocarbons (THC) and heavy metals in the soil and water bodies as well as pr were determined and compared with both national international standards. The study found THC and heavy metals such as Iron and Manganese to be significantly high in both soil and water samples. It was found that the artisanal crude oil refining activities in the Niger Delta Area of Nigeria generate and discharge environmental safety hazards that are far beyond the statutory and intentional acceptable limits.

Keywords: Heavy metals, environmental safety, artisanal refineries, total hydrocarbon content, crude oil.

Introduction

The extraction of crude oil in the Niger Delta Area of Nigeria is made possible through a diverse network of onshore oil wells, pipelines and flow-stations located in the creeks of the mangrove forest. These infrastructures are mostly exposed to constant exploit through vandalism by breaching of pipelines and siphoning crude oil for sale in the 'international black market' or to local unconventional refineries, found within the swamps of the regions mangrove forest, for illegal refining (Onuoha, 2007; Borris, 2015).

Iwegbue et al. (2013) examined the concentrations, contamination/pollution index, anthropogenic input and enrichment factors for metals in soil in the vicinity of cassava processing mills in sub-urban areas of Delta State of Nigeria. The concentration levels of metals at the study sites were below the Nigerian Department of Petroleum Resources (DPR) target values for metals in soils except for Cadmium. A significant fraction of these metals arose from anthropogenic sources. Similar investigation was reported by Akudo (2016).

Soil toxicity poses health and safety risks to the ecological system. Most of the physical and chemical qualities of the soil in the Niger Delta are contaminated by petroleum hydrocarbons and heavy metals. Nigeria's petroleum crude oil is known to contain heavy metals in varying proportion (Kenneth and Saviour, 2016); these heavy metals when introduced to the environment in excessive amounts constitute a source of great danger to the health of both man and animals. Illegal refining and oil spills can cause random fire breakout, killing vegetation and creating a crust over the land, making remediation or revegetation difficult.

According to the UNEP (2011) investigation, surface water throughout the creeks of the study areas contained hydrocarbons floating layers of oil vary from thick black oil to thin oil layers. Frederick (1993) reported that external contamination of feathers is the most common form of exposure to birds. The bird feathers absorb the oil, thereby, becoming matted and leading to the loss of its ability to repel water, serve as insulation and flight abilities. Deaths then results from combinations of hypothermia, starvation and drowning. Also oil, as little as 1-10 μL, can also be lethal to embryos during the first half of incubation, thereby leading

[1] Ahmadu Bello University, Zaria, Kaduna State, Nigeria, kshuwa@gmail.com
[2] Ahmadu Bello University, Zaria, Kaduna State, Nigeria, augayya@yahoo.com
[3] Ahmadu Bello University, Zaria, Kaduna State, Nigeria, galadimams@yahoo.com
[4] Ahmadu Bello University, Zaria, Kaduna State, Nigeria, diyauddeen.bh@gmail.com

to high mortality rate of birds in the areas of crude oil pollution (Gordon and John, 2012; Yabrade and Tanee, 2016; Gijo et al., 2016).

Increased concentration (contamination) of heavy metals and hydrocarbons in the soil and water bodies could affect the environmental health and safety in terms of soil nutrients bearing capacity and biochemical oxygen demand. These in turn could affect crops production and aquatic lives respectively (Klamerus-Iwan, 2015).

This study seeks to investigate the heavy metal and hydrocarbon contamination levels from a destroyed illegal refinery site found in Okarki Community of Ahoada West Local Government Area of Rivers state, through a combination of field visits, laboratory experiments and analyses. The study also investigates the water and soil Physicochemical parameters of the study area.

Methods

The study considered an illegal refinery site located in Okarki, Ahoda West Local Government Area. Okarki community is a town found on the border of Bayelsa and Rivers state, which lies within Latitude 4.9871 and Longitude 6.4213. The study site accommodates one of the illegal refinery facilities destroyed by the Nigerian Army under 'Operation Delta Safe' in February 2017, and is located in a forest area found in the community (between Latitude 4.9412 and Longitude 6.4309).

Site visitation

Access to the site was both by foot and canoe as seen in Fig. 1. Security personnel were involved for safety and security reasons. Access to the sites was only possible with security 'pass' because Okarki site was destroyed and under tight security.

Crude oil was found in makeshift reservoirs. Waste discharge from the 'fractionation column' was found in the waste pit dug into the ground. On reaching the site it was found empty with the crude oil reservoir abandoned. A layer of oil was observed covering the soil and water ways, indicating illegal refining activity as an anthropogenic source of contamination of the area. Soil and water sampling were drawn randomly onsite. Control samples were also taken from the nearest uncontaminated farmlands.

Fig. 1 Arrival at Study Area

Soil sampling

A systematic random sampling method was used to ensure proper coverage of the study area. The sampling area was classified into zones and the subsurface depth 0-15 cm was sampled using a hand trowel and ruler (Keith, 1996). Five replicates were collected from each sampling zone/location and mixed thoroughly to form homogenous compost. Non-decomposed surface litters were removed before sampling so as to ensure effective soil sampling. The samples were labelled with masking tape using tempo marker, the labels contained date of sampling, sample ID, parameters required and location of sampling. Then all samples were packed and stored in ice chest before taken to the laboratory for analysis.

Laboratory analysis - soil samples

Heavy metals analysis

Each soil sample was oven dried, grinded and passed through a 2 mm mesh sieve to remove debris. The weighted out gram of the dried and sieved soil sample was transferred into a 100 mL beaker, 10 mL of concentrated nitric acid (HNO_3) and 5 mL of concentrated per chloric acid ($HClO_4$) were added and the mixture was placed on a hot plate under fume cupboard and heated to near dryness until its colour turned white. This residue was allowed to cool and then dissolved with 20 % nitric acid (5 mL). The mixture was then filtered and made up to 20 cm^3 with distilled water. So also the blank mixtures and the various concentration mixtures

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were equally prepared. The preparation samples were then analysed using an Atomic Absorption Spectrophotometer (AAS). A standard solution of each of the metals determined was used in calibrating the AAS.

Total hydrocarbon content analysis

This was determined by shaking 5 g (oven dried weight basis) soil samples with 100 ml Methyl Isobutyl Ketone (MIBK), analar grade known weight of the extract was taken and spiked with an appropriate internal standard of 1-chlorooctadecane. A Gas Chromatograph was used, with the peak area analysis done using a Perkin Elmer Recorder Interfaced to a Computer. Tab. 1 gives a summary of the analyses, principle and equipment used for the soil samples study.

Water sampling

Water samples were collected both upstream and downstream of the study area. At each sampling location five samples were collected and mixed into a single sample. Each sample consisted of five separate portions appropriately collected, subdivided and stored. For Heavy Metal Analysis the samples were collected in clean glass bottles after rinsing with portions of the samples. Each sample was spiked with analar grade nitric acid to pH $\leq$ 2.0 as preservative. While for the Total Hydrocarbon Content (THC) analysis, the samples were collected into clean glass bottles, pre-rinsed with portions of the samples. Each sample was spiked with sulphuric acid to pH $\leq$ 2 to serve as preservative. All water samples were properly labelled based on sample ID, sampling location, date of sampling, parameters to

Tab. 1 Summary of Equipment used for Chemical Analysis of Soil Samples

Analysis/Test Item	Unit	Principle	Equipment
pH	pH unit	Electrometric	pH meter
Organic Carbon	%	Titrimetric	Walkley-Black method
Conductivity	µS/cm	Electrometric	Conductivity meter
Ca	mg/kg	Atomic Absorption	AAS*
Mg	mg/kg	Atomic Absorption	AAS*
Na	mg/kg	Atomic Absorption	AAS*
HCO_3	mg/kg	Distillation/Titrimetric	KCl Extraction method
NO_3	mg/kg	Distillation/Titrimetric	KCl Extraction method
NH_4	mg/kg	Distillation/Titrimetric	KCl Extraction method
SO_4	mg/kg	Filtration	KH_2PO_4 Extraction method
Heavy metals	mg/kg	Atomic Absorption	AAS*
THC	mg/kg	Gas Chromatography	Gas Chromatograph

* Atomic Absorption Spectrophotometer (AAS).

Tab. 2 Summary of Methodology and Equipment used for Chemical Analysis of Water Samples

Analysis/Test Item	Unit	Methodology	Equipment
pH	pH unit	Electrometric	pH meter
Turbidity	NTU*	Turbidimetric	Turbidity meter
Total Dissolved Solids (TDS)	mg/l	Gravimetric/ Electrometric	Oven-balance/TDS meter
Dissolved oxygen (DO)	mg/l	Titrimetric/ Electrometric	DO meter
Biological Oxygen Demand (BOD)	mg/l	Titrimetric/ Electrometric	Winkler's method
Chemical Oxygen Demand	mg/l	Titrimetric	Open reflux method with $K_2Cr_2O_7$ and FAS spectrometer
Salinity as chloride	mg/l	Titrimetric	Titration method ($AgNO_3$-K_2CrO_4)
Conductivity	µS/cm	Electrometric	Conductivity meter
Heavy Metals	mg/l	Atomic Absorption	Atomic Absorption Spectrophotometer
THC	mg/l	Gas Chromatography	Gas Chromatograph

* Nephelometric Turbidity Unit

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be analysed and type of preservation, then packed in ice chest and taken to the laboratory for analysis (Boulding, 1996).

Laboratory analysis - water samples

Heavy metals analysis

For 100 ml water samples, 20 ml HNO_3 solution was added in 125 ml conical flask and the solution heated gently for 20 minutes on hot plate. Another 5 ml HNO_3 solution was further added and heated for another 30 minutes until digestion was complete. The digested sample was allowed to cool, then it was filtered using Whatmann filter paper and made up to 100 ml with deionised water in a calibrated volumetric flask. The heavy metals were determined using AAS.

Total hydrocarbon content analysis

The total hydrocarbon in the sample was initially extracted with about 100 ml MIBK (Methyl IsoButyl Ketone) Analar grade known weight of the extract was taken and spiked with an appropriate internal standard of 1-chlorooctadecane. A Gas Chromatograph was used and the peak area analysis was done using a Recorder Interfaced through to a Computer. Tab. 2 gives a summary of the analyses, principle and equipment used for the water samples study.

Pollution index calculations

The study also involves pollution index calculations for hydrocarbon and heavy metal contamination of the study area. The work considered pollution index calculations of two types: Single and Integrated indices.

Single index

Single index, such as Contamination Factor (CF), is an indicator calculated based on concentrations of the individual contaminants (Hakanson, 1980).

$$CF=\frac{C_{metal}}{C_{reference\ value}} \quad (1)$$

The CF is the ratio obtained by dividing the concentration of each metal (C_{metal}) in the soil or water by the reference value. The reference values ($C_{reference\ value}$) are obtained from the requirements/guidelines developed by the Department of Petroleum Resources (DPR), Nigeria (Joseph and Ajienka, 2010) and Nigerian Standard Organization (SON) (SON, 2007). The following ranges are used to characterise the contamination factor (Turekian and Wedepohl, 1961).

Range	**Extent of Contamination**
$CF < 1$	Low contamination
$1 \le CF < 3$	Moderate contamination
$3 \le CF < 6$	Considerable contamination
$CF \ge 6$	Very high contamination

Integrated indices

Integrated indices are indicators used to calculate the collective contamination of more than one contaminant and are based on the single index. Integrated indices of interest here are Pollution Load Index (PDI) and Degree of Contamination (DC).

For a single site, the PLI is the nth root of the product of the n contaminants (see Equation 2). A PLI value of zero indicates perfection; a value of one indicates the presence of only baseline levels of pollutants, and values above one would indicate progressive deterioration of the site quality (Tomlinson et al., 1980). PLI value > 1 implies pollution whereas PLI value < 1 indicates no (acceptable) pollution (Chakravarty and Patigiri, 2009), (Seshan et al., 2010). For this study, n = 8.

$$PLI=(CF1\cdot CF2\cdot CF3\cdot\ldots\cdot CFn)^{1/n} \quad (2)$$

The DC is another index that can be derived from the CF values. It is defined as the sum of all contamination factors for a given site as shown in Equation 3 (Tomlinson et al., 1980).

$$DC = \sum_{i}^{n} CF \quad (3)$$

Where CF is the single contamination factor and n is the count of the contaminants considered.

Range	**Degree of Contamination**
$DC < n$	Low
$n \le DC < 2n$	Moderate
$2n \le DC < 4n$	Considerable
$DC > 4n$	Very high

With n = 8 in this study, the following criteria apply:

Range	**Degree of contamination**
$DC < 8$	Low
$8 \le DC < 16$	Moderate
$16 \le DC < 32$	Considerable
$DC > 32$	Very high

Results and discussion

Heavy metals and total hydrocarbon content - CF, PLI and DC

After the random sampling, the Heavy Metals (HMs) and Total Hydrocarbon Content (THC) values were determined for each of the samples. The average concentration of each parameter was calculated. Control samples were also collected and analysed.

It can be seen that the THC concentration in Okarki soil samples for both the test and control sites were above the DPR standard target value of 50 mg/kg, but well below the intervention level of 5000 mg/kg (DPR, 2002). The Okarki test and control site showed significant statistical difference ($p < 0.05$) between the sites and the average concentration of 432.50 mg/kg recorded in the test site can be related to the findings of (Moslen and Miebaka, 2017) who recorded mean values from 7.9-1144.6 mg/kg in sediment samples gotten from a crude oil polluted creek of the Azuabie Creek, the upper reaches of the Bonny Estuary in Rivers State. Mean THC values ranging from 400-6205 mg/kg were also recorded by (Wokoma, 2014) from sediment samples gotten from a tidal creek of the Bonny River in Rivers State. The (UNEP, 2011) environmental analysis of Ogoni land also recorded petroleum hydrocarbon concentration as high as 33200 mg/kg from samples taken from Bodo west artisanal (illegal) refinery site. The present study indicates a lower THC concentration when compared with past literature because of the nature of the test site being a destroyed and abandoned site that was subjected to runoff due to continuous rainfall leaving minimal hydrocarbon on the soil surface within the 0-15 cm depth.

The water samples results collected from Okarki. The values fell within the range of 270.0-336.0 mg/L with mean value of 308.3 mg/L which is comparable with results recorded by (Gordon and John, 2012) who reported THC values of 236.8 mg/L from borehole water samples of Bolo mainland and 986.1 mg/l from Aseminigo laka, Bodo west, which was subjected to artisanal (illegal) refining activities. THC values of 1881.79 mg/L, 1907.89 mg/L and 1810.43 mg/L where recorded from different sampling stations of the crude oil polluted areas of the Nun River Estuary by (Gijo et al., 2016).

The concentration of Fe as observed in Okarki test site is above the target value as specified by DPR and can be attributed to the rusting of the abandoned materials used by the Illegal refiners. This value is equally well below 20623.67 mg/kg as reported by (Ikpesu and Dickson, 2016) in samples collected at the pipeline location in Agip Oil Company Environs. In the same manner Pb-26.99 mg/kg, Cr-15.03 mg/kg and Cu-21.21 mg/kg were also recorded from the same Agip Oil company Environs. (Chukwujindu, 2011) reported Cd-1.12 mg/kg, Cu-11.20 mg/kg, Cr-28.80 mg/kg, Pb-25.00 mg/kg, Mn-201.80 mg/kg, Ni-31.70 mg/kg and Zn-29.30 mg/kg from soil samples collected from a crude oil contaminated site in the Niger Delta area.

For the water samples from Okarki site, the HMs such as Pb, Cd, Cr and Ni were below detection level of 0.001 mg/L in both test and control sites. The test site recorded values of Fe-8.57 mg/L, Zn-0.10 mg/L, Cu-0.02 mg/L and Mn-2.32 mg/L with lower values found at the control site. All HMs concentrations were below the standard for drinking water (SON, 2007).

The CF, PLI and DC were evaluated for both soil and water samples drawn from the study area, and results summarised in Tab. 3.

Tab. 3 OKARKI Soil and Water Samples- Summary of CF, PLI and DC values

Parameters	Soil Samples		Water Samples	
	Test Samples	Control Samples	Test Samples	Control Samples
Fe	682.344	0.481	28.576	0.189
Zn	0.449	0.047	0.033	0.001
Cu	0.314	0.047	0.023	0.013
Pb	3.377	0.778	0.000	0.000
Cd	1.232	0.930	0.000	0.000
Cr	0.085	0.001	0.000	0.000
Ni	0.693	0.004	0.000	0.000
Mn	21.642	0.430	11.586	0.020
PLI	**2.136**	**0.071**	**0.876**	**0.124**
DC	**710.137**	**2.717**	**40.217**	**0.223**

Fe = Iron, Zn = Zinc, Cu = Copper. Pb = Lead, Cd = Cadmium, Cr = Chromium, Ni = Nickel, Mn = Manganese, PLI = Pollution Load Index, DC = Degree of Contamination and CF = Contamination Factor

The CF values, shown in Tab. 3, which were calculated for all HMs in both soil and water samples of Okarki test site showed very high contamination for Fe and Mn ($CF > 6$). Okarki soil samples also indicated very high Zn ($CF > 6$) contamination in both test and control sites. CF of Pb and Cd in soil samples of the test site was considerable ($3 \leq CF$

≤ 6) with Cu, Cr and Ni giving low contamination (CF < 1). In water samples Zn and Cu showed low contamination (CF < 1) while Pb, Cd, Cr and Ni were not detected in all water samples. Soil samples indicated PLI > 1 meaning the site was HM polluted with very high DC (DC > 32). The water samples of Okarki study area gave PLI < 1 indicating no HM pollution.

Soil particle size distribution

Soil particle sizes were also determined, summary of the finding are presented in Fig. 2.

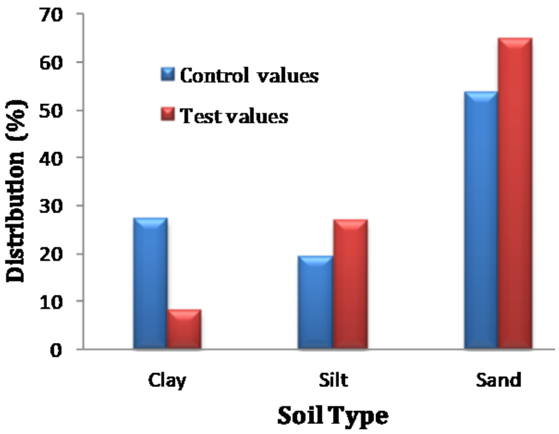

Fig. 2 Okarki Soil Particle Size Distribution

While percentage of the clay in Okarki soil dropped from about 27 % to 8 %, both silt and sand showed increase - with silt increasing from 19 % to 27 % and sand distribution jumping from 54 % to about 65 %. The high percentage of sand can be attributed to the constant burning of crude oil thereby disintegrating the soil and increasing the pore sizes.

Physicochemical characteristics

From Tab. 4, it can be seen that values of pH in both test and control site were below 7.0, indicating the soil is slightly acidic. This may be attributed to long term petroleum processing activity in the Niger Delta area, daily tidal flushing of mangrove soils, dredging activities, localised elevation, topographical heights among others (SPDC, 2004).

The conductivity values were mostly above 800 μS/cm for the test samples and below 700 μS/cm for the control soil samples. From Tab. 4, it can be seen that the organic carbon (O.C.) content of soils (with mean value of 1.21 %) was more than twice the control value (with mean value of 0.5 %). The findings compare well with the earlier work by Yabrade and Tanee (2016) which found that the organic carbon levels at an artisanal (illegal) refinery in south-west of Warri, Delta state Nigeria was in the range 1.74-3.00 %.

The levels of soil fertility indicators are expressed by the distributions of NO_3, SO_4 and NH_4 (Tab. 4) showed values typical of the Niger Delta soils (SPDC, 2004). The values for the test site were higher than that of the control samples. These values are normal and can support the usual fauna and flora in that ecosystem, except when affected by THC or other toxic elements.

The summary levels of exchangeable cations as expressed by the levels of Na and Ca are also given in Tab. 4. Generally, the values were high compared to other findings representing the natural concentration of the mangrove of the Niger Delta. Other findings such as that of Ikpesu and Dickson (2016) reported Ca levels of 776.75 ± 19.7 - 1625.1 ± 10.8 mg/kg. Yabrade and Tanee (2016) recorded Ca concentration ranging from 162.51 ± 10.8 -

Tab. 4 Physicochemical Characteristics of Soil Samples of Okarki Study Area

Parameters	Test Samples	Control Sample	p-Value	DPR Standard for Soil
pH	6.26 ± 0.04	6.35 ± 0.841	0.433	NA
E.C [μS/cm]	825.10 ± 22.84	672.00 ± 17.44	0.000	NA
Ca [mg/kg]	311.40 ± 12.03	720.79 ± 8.65	0.000	NA
Na [mg/kg]	116.29 ± 3.04	151.99 ± 9.88	0.058	NA
O.C (%)	1.21 ± 0.01	0.50 ± 0.08	0.008	NA
SO_4 [mg/kg]	83.36 ± 4.12	4.20 ± 0.33	0.000	NA
NO_3 [mg/kg]	0.12 ± 0.01	0.11 ± 0.02	0.059	NA
NH_4 [mg/kg]	0.00 ± 0.00	0.07 ± 0.01	0.009	NA

NA = Not Available, DPR = Department of Petroleum Resource, EC = Electric Conductivity, Ca = Calcium, Na = Sodium, O.C = Organic Carbon, SO_4 = Sulphate, NO_3= Nitrate, NH_4 = Ammonia.

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776.75 ± 19.7 mg/kg. The current research reports Ca values of 311.4 ± 38.05 mg/kg in Okarki test site, which corresponds to values recorded from crude oil affected areas.

Okarki water samples recorded (Tab. 5) low salinity (14.20 ± 1.16 mg/L) corresponding to low EC (43.6 ± 0.70 µS/cm). Slightly acidic pH (6.08 ± 0.04) levels were observed, which is typical of the Niger Delta ecosystem having pH levels ranging from acidic to slightly acidic which is directly or indirectly affected by crude oil activities taking place in the environment (Gijo et al., 2016; Franscis and Nsifiok, 2010).

Levels of Dissolved Oxygen (DO) was observed to be low (5.83 ± 0.20 mg/L), with Biological Oxygen Demand (BOD) of 1.95 ± 0.10 mg/L and Chemical Oxygen Demand (COD) of 5.18 ± 0.12 mg/L. Oil-film (from crude oil spills during Illegal artisanal refining or transportation of stolen crude oil) formed at the surface of water bodies prevents oxygen diffusion from the air which could lead to low levels of DO. High oxygen depletion can be severe enough to affect aquatic life, thereby reducing the level of BOD as well. Chapman and Kimstach (1992) noted that dissolved oxygen concentration below 5.0 mg/L adversely affects the functioning and survival of biological communities and below 2 mg/L may lead to the death of most fish and aquatic life. Thus, the DO content (5.39 ± 0.80 mg/L) in the analysed samples record critical level and should be closely monitored to prevent death of aquatic life.

Conclusion

The study found THC levels to be above national target values, but were well below the emergency intervention levels. Fe and Mn concentrations were found to be significantly high in both soil and water samples - eliciting the need for closer checks and monitoring. Fe was found to have highest concentration while Cr has the least. Overall, The Contamination Factor (CF) values followed the ordering: Fe > Mn > Pb > Cd > Ni > Zn > Cu > Cr in soil samples. Both Pollution Load Index (PLI) (> 1) and the Degree of Contamination (DC) (> 700) suggest that the contamination levels are very high. For the water samples, the CF values were in the order: Fe > Mn > Zn > Cu with PLI (< 1) and DC (> 40) suggesting relatively high contamination. The soil samples were considered to be heavy metal polluted while the water samples did not appear to be significantly polluted. Overall, the study quantitatively demonstrates the potential of the artisanal crude oil refineries to cause considerable health, safety and environmental concerns in the Niger Delta Area of Nigeria.

Tab. 5 Physicochemical Characteristics of Water Samples of Okarki Study Area

Parameters	Test Sample	Control Sample	p-Value	SON Standard
pH	6.08 ± 0.01	6.08 ± 0.02	0.928	6.5-8.5
EC [µS/cm]	43.60 ± 0.22	112.36 ± 0.37	0.000	1000
TDS [mg/l]	241.84 ± 2.24	200.38 ± 0.17	0.000	500
Turbidity [NTU]	1.16 ± 0.06	1.07 ± 0.03	0.248	5
Salinity [mg/l]	14.20 ± 0.37	25.27 ± 0.19	0.000	600*
COD [mg/l]	112.30 ± 0.88	130.07 ± 0.04	0.000	10*
BOD [mg/l]	47.91 ± 0.10	64.06 ± 0.04	0.000	10*
DO [mg/l]	5.39 ± 0.25	1.55 ± 0.04	0.000	NA

NA = Not Available, SON = Standard Organization of Nigeria, EC = Electric Conductivity, TDS = Total Dissolve Solid, COD = Chemical Oxygen Demand, BOD = Biological Oxygen Demand, Dissolve Oxygen, *DPR Standard.

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