PRINT ISSN 1119-8362
Electronic ISSN 2659-1499

Full-text Available Online at
https://www.ajol.info/index.php/jasem
https://www.bioline.org.br/ja

J. Appl. Sci. Environ. Manage.
Vol. 28 (9) 2849-2854 September 2024

Heavy Metals, Polycyclic Aromatic Hydrocarbons, Total Petroleum Hydrocarbons and Total Hydrocarbon Contents in Drilling Mud Effluents From Eastern Obolo Oilfield In The Niger Delta Region Of Nigeria

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ABSTRACT: The objective of this paper is to evaluate the concentrations of heavy metals, polycyclic aromatic hydrocarbons (PAHs), total petroleum hydrocarbons (TPH) and total hydrocarbon content (THC) in drilling mud effluents from Eastern Obolo Oilfield in the Niger Delta region of Nigeria using appropriate standard methods. The TPH and THC of OBM and WBM analyses with UV-Vis spectrophotometer indicated 1.60 mg/kg for OBM and 1.30 mg/kg for WBM. A value of 5.35 mg/kg was obtained for OBM compared to 5.15mg/kg for the OBM in the THC. Generally, the result indicated that TPH<THC and was found to be far lower than 50 mg/kg allowable for drilling mud in the environment. GC-MS analysis for TPH indicated a range of *n*- alkanes with a higher concentration (46118.28 ug/l) in the OBM compared to WBM concentration (17185.95 ug/l) compared to the UV-Vis spectrophotometer. The analysis of TPH using GC-MS revealed a more comprehensive range of *n*-alkanes present in the OBM and WBM. The heavy metals indicated considerable differences of the concentration in OBM and WBM. The concentrations viewed significantly with the range for the WBM; Fe>Zn>Cr>Pb>Cu>Ni>Cd. The concentrations range in the OBM were; Fe>Cr>Zn>Ni>Cu>Pb>Cd. The evaluation of the concentrations of these elements in drilling mud are of environmental significance from pollution viewpoint especially ferrochrome lignosulphate (Chromium pollution) and lead compounds (lead pollution). The results from this study which indicates the presence of varying concentrations of heavy metals and petroleum hydrocarbons in the drilling mud is valuable and significant when making the choice for subsequent disposal of drilling mud effluents and, particularly in the study area where extensive oil/gas operation is ongoing.

DOI: https://dx.doi.org/10.4314/jasem.v28i9.30

Cite this Article as: AWAKA-AMA, J. J; UDO, G. J; NYONG, A. E; UMANAH, I; BASSEY, M. E (2024). Heavy Metals, Polycyclic Aromatic Hydrocarbons, Total Petroleum Hydrocarbons and Total Hydrocarbon Contents in Drilling Mud Effluents From Eastern Obolo Oilfield In The Niger Delta Region Of Nigeria. *J. Appl. Sci. Environ. Manage.* 28 (9) 2849-2854

Dates: Received: Received: 07 July 2024; Revised: 15 August 2024; Accepted: 19 August 2024 Published: 05 September 2024

Keywords: Drilling mud effluents; heavy metals; petroleum; hydrocarbon; environment

Oil and gas exploration and production usually have a variety of impacts on the environment. The highest impact results from waste discharges into the environment in concentrations that were not naturally found in such locations. The disruption of ecological balance through drilling operations occurs through surface discharge of pollutants from various activities affecting the environment. Environmental management of drill sites is a serious problem to combat the pollution both at onshore and offshore (AlBajalan and Haias, (2021; Apaleke *et al*., 2012). Drilling fluids are a set of complex chemical mixtures generally used in the upstream oil and gas industry and consist of solids and liquids, as well as

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base oil and brines. Typical drilling fluids generally is a combination of water, clay, weighting materials and various chemicals with a function to adjust the properties of the fluid to meet the requirement of each well in both onshore and offshore drilling processes (Jingen *et al*., 2022). Drilling fluids may be water-based mud (WBM), oil-based mud (OBM) or synthetic. Some of the critical roles of the drilling fluid include cooling and lubricating of thedrill bits and conveying a variety of chemicals down the borehole, and removing drill cuttings (Amin *et al.*, 2018; Araka *et al.*, 2019). Drilling fluids are associated with changing concentrations of hydrocarbons and heavy metals (Udosen *et al*, 2010; Udo *et al*, 2021; Awaka-ama, 2012). Many additives used in its composition are not toxic-free and are therefore regulated. The use of drilling fluids have widely been connected to some environmental problems due to a variety of wastes including drill cuttings and used drilling fluid (mud) generated during drilling process (Arpornpong *et al*, 2020; Ball *et al*, 2011; Awaka-ama, 2012). This wastes has also been a major source of heavy metal pollutants from barium, mercury, cadmium, diesel (from lubricants, spotting fluids and oil-based mud cuttings), arsenic and formaldehyde (biocides).

Drilling wastes can have different potential impacts depending on where it is discharged (Getliff *et al*., 2000; Neff, 2008; Iloms *et al*, 2020; Udo *et al*, 2023a). Therefore, discharge of untreated drilling effluents into seas, rivers and land constitutes a serious hazard and is an important concern due to the detrimental effects and degradation of land, air and water around the oilfield drill site. This may lead to the elevations of the concentrations of heavy metals, PAHs, TPH and THC in the environment, posing a potential risk to the ecosystem and human health. Therefore, the objective of this paper is to evaluate the concentrations of heavy metals, polycyclic aromatic hydrocarbons (PAHs), total petroleum hydrocarbons (TPH) and total hydrocarbon content (THC) in drilling mud effluents from Eastern Obolo Oilfield in the Niger Delta region of Nigeria.

MATERIALS AND METHODS

Location of the Study Area: The oilfield wells are located in Eastern Obolo, in the Niger Delta fringe between Imo and Qua Iboe Rivers estuaries in Nigeria, and lies between latitudes 4° 28' and 4° 53' and longitudes 7° 50' and 7° 55' East. It is a coastal local government under great tidal influence from the Bight of Bonny and is bounded in the south by the Atlantic Ocean.

Sample Collection: Drilling mud samples were collected from the mud tank at the samples site using a cup and were transported to the laboratory and stored in the refrigerator for 2 days prior to the analyses in the laboratory. A total of ten (10) water-based mud (WBM) and oil-based mud (OBM) from the production wells storage tanks were collected for this study and the composite samples collected were analyzed for the total petroleum hydrocarbon (TPH) using Gas chromatography- Mass Spectrophotometer method. The samples were also analyzed for heavy metal concentration in the WBM and OBM, using Atomic Absorption Spectrophotometer (AAS) method.

Digestion of WBM and OBM samples for heavy metal analysis: 5 ml of the WBM sample was digested by the addition of 10 ml of concentrated HNO_3 and 10 ml of hydrogen peroxide, (H_2O_2).This was heated on a hot plate to about half the original volume (Digestate).The flask was allowed to cool, and its content was then filtered into a 25 ml standard volumetric flask and made up to the mark with distilled water.Triplicate digestion of WBM samples was carried out in order to rule out experimental bias or random error.A portion of the solution was used for heavy metal analysis AAS.

Total Petroleum Hydrocarbon (TPH) Analysis using GC-MS: 10 g each of the WBM and OBM samples were collected into solvent (dichloromethane) rinsed beakers. 50ml of 50:50 mixture of acetone and dichloromethane was added. The samples were shaken gently but vigorously for 20-minutes while being heated at 20°C. 10g of anhydrous sodium sulphate was added to the samples and allowed to stand until a clear extract were developed. The extract were decanted, the solvent was then concentrated and exchanged with HPLC grade hexane, using column chromatography method, and then re-concentrated to 3ml. The extracted samples were transferred into Teflon-lined screw-cap vials. These were well labeled, corked and taken to the GC-MS (GC-MS model HP5890 PLUS II with AGILENT CHEMSTATION quantification software) for TPH analysis.

Determination of TPH using UV-Vis Spectrophotometer: A 5 g sample was weighed. It was mixed with xylene (50ml) then stirred using a magnetic stirrer for 2 minutes. A chromatographic column was parked with anhydrous sodium sulphate and then separation using xylene, DCM and chloroform. The Absorbance (Abs) was read using the UV-Vis spectrophotometer (SpectroLab 752Pro). The spectrophotometer was set at 425 nm wavelength:

$$\text{TPH and THC}\left(\frac{\text{mg}}{\text{kg}}\right)=\frac{V*Ab}{MF*W_s} \quad (1)$$

Where: TPH = total petroleum hydrocarbon; THC = total hydrocarbon; V = volume of xylene; Ab = absorbance, Ws = weight of sample; M.F (Multiplication Factor) = Swampy Area/Sea = 0.0004 M.F for Upland = 8103

Silica gel has the ability to adsorb polar materials. If a solution of hydrocarbons and fatty materials in a nonpolar solvent is mixed with silica gel, the fatty acids are removed selectively from solution. The materials not eliminated by Silica gel adsorption are designated hydrocarbons.

Metal Analysis: One g of each of the sample type was digested using mixture of perchloric acid $HClO_4$, nitric acid HNO_3 and sulphuric acid H_2SO_4 in the ratio 1:2:2. (Adewole *et al.*, 2010; Neff et *al.,* 1988b). The prepared solution was analyzed for heavy metals / elements of interest using atomic absorption spectrometer (AAS). The results obtained were compared with both Nigeria Upstream Production and Regulatory Commission (NUPRC) guidelines and United State Environmental Protection Agency (USEPA) standards for drilling waste disposal.

RESULTS AND DISCUSSION

The results of the heavy metals analyzed in the WBM and OBM are shown in Table 1. The heavy metals analyses indicated marked differences in the concentrations in WBM and OBM. In terms of abundance, as shown in the Table 2 the OBM contained high concentration of Fe (24.4mg/l), Zn (0.53mg/l), Cr(0.13mg/l), Pb(0.08mg/l), Cu(0.06mg/l).

Table 1: Heavy metals concentration (mg/l) in the samples

Sample	Cr	Fe	Ni	Cu	Pb	Cd	Zn
OBM	0.13	24.40	0.15	0.06	0.08	0.02	0.53
WBM	0.11	12.40	0.05	0.02	0.01	0.00	0.06
NUPRC Standard	0.05	0.005	0.10	1.00	0.006	0.05	1.00

Note: NUPRC (Nigeria Upstream Production and Regulatory Commission, formerly, DPR)

In comparison, the concentration of Fe (12.4mg/l) in the WBM was found to be half the concentration (24.4mg/l) obtained in the OBM. The concentration of Cr (0.11mg/l) was nearly similar to that obtained in the OBM. The Chromium concentration in both the WBM and OBM were above the 0.05mg/l recommended by WHO (1993) and USEPA (1992) for drinking water and also for fishery by water quality criteria. Chromium pollution resulting from ferrochrome lignosulphate (a constituent of water-based mud) has been identified as a source of environmental pollution (USEPA, 1992; Shadizadeh and Zoveidavianpoor, 2010; Offiong *et al*, 2023). The abundance in other metals in the WBM were Zn (3.52mg/l), appreciably higher than the concentration in OBM (0.53mg/l). Ni (0.77mg/l), Cu (0.08mg/l), and Pb (0.01mg/l). Cadmium was found absent (0.00mg/l) in the WBM compared to the OBM (0.02mg/l). Pb content was appreciably higher in the OBM. For OBM: TPH =1.60mg/kg and THC = 5.35mg/kg while, for WBM: TPH = 1.30mg/kg and THC = 5.15 mg/kg

Table 2: Total Petroleum Hydrocarbon (TPH) for water-based mud (WBM) and oil-based mud (OBM)

TPH	WBM(μg/L)	OBM(μg/L)
n-octane	3392.652	723.413200
n-nonane	9063.970	4730.77200
n-decane	352.0269	143.632300
n-undecane	1981.834	658.161400
n-dodecane	71.11008	10170.6500
n-tridecane	37.29415	1190.78400
n-tetradecane	69.39721	1418.04000
n-pentadecane	403.6161	435.518100
n-hexadecane	67.09979	10530.1700
n-heptadecane	491.2397	1209.47200
Pristane	140.3726	1666.23100
n-octadecane	249.1221	1584.50700
Phytane	847.3272	859.757500
n-nonadecane	3.028081	5320.17900
n-eicosane	6.197512	160.588100
n-heneicosane	2.693108	114.099100
n-docasane	2.617612	15.5074400
n-tricosane	<0.01	8.70715100
n-tetracosane	0.182448	3.50437800
n-pentacosane	<0.01	1.23685400
n-hexacosane	<0.01	<0.01
n-heptacosane	<0.01	0.50350500
n-octacosane	0.211653	<0.01
n-nonacosane	<0.01	<0.01
n-tricontane	<0.01	<0.01
n-hentriacontane	<0.01	<0.01
n-dotriacontane	<0.01	5170.20400
n-tritriacontane	3.962289	2.58869500
ΣTPH	**17185.95**	**46118.2300**

Table 3 Showing Total Petroleum Hydrocarbon (TPH) and Total Hydrocarbon Contents in OBM and WBM Using UV-Vis Spectrophotometric analysis

Sample	OBM (mg/kg)	WBM (mg/kg)
TPH	1.60	1.30
THC	5.35	5.15

In general, the lead concentration in both the WBM and OBM were higher than the recommended 0.006mg/l by NUPRC (2022) and the established threshold level of 0.05mg/l. These elevated concentrations potentially will contribute to increased lead accumulation in the aquatic environment where the drill wastes are disposed. Generally, the results indicated heavy metals of higher concentration in the OBM compared to the WBM. Several studies (Neff *et al.,* 1988a, 1988b; Bascom, 1983; Kay, 1984) have shown that with the exception of organomercury compounds, which are not found in drilling wastes discharges, concentration of most metals in natural marine food webs show either no relation or an inverse relation to trophic level, indicating that food chain biomagnifications of inorganic metals does not occur. Drilling muds have been found to contain heavy metals such as barium, chromium, mercury, and lead, posing significant environmental concerns (Udosen and Awaka-ama, 2005; Shadizadeh and Zoveidavianpoor, 2010). However, the exceptionally high values of Iron (Fe) in both the OBM and WBM are of great concern when compared with the WHO (1993) and USEPA (2011) drinking water standard and water quality criteria for irrigation and fishery (Okop *et al,* 2018). Though these elements are likely to be of nutritional importance to the aquatic animals, the associated metals will constitute a problem to aquatic lives even in very small concentrations. Elevated heavy metal in the environment indicates the consequences of oil-related activities as well as other sources of anthropogenic contaminants (Nwadinigwe *et al*, 2015, Udo *et al*, 2017, Udo *et al*, 2018, Nyong *et al*., 2020)

Total Petroleum Hydrocarbon (TPH) Using GC-MS analysis: It was observed that the concentration of TPH in WBM to the OBM was in the ratio of 1:4. The TPH analysis indicated a range of *n*-alkanes with a higher concentration (46118.23µg/l) in the OBM compared with the WBM concentration (17185.95 µg/l). The limit for THC disposal into the marine environment is regulated by Nigeria Upstream Production and Regulatory Commission (NUPRC) guidelines.

The TPH value is higher in the OBM. However, TPHCWG ,1998b; Udo *et al*, 2020; Udo *et al, 2023b*; Awaka-ama *et al*, 2024 stated that most petroleum hydrocarbon mixtures contain very low concentration of THC. The major concern regarding THC is the potential carcinogenicity of some molecules (0ffiong *et al.,* 2023; IPCS, 1998a). Oil-field drilling fluids, especially oil-based mud, contain hazardous constituents (Martin, 1991; UKOOA, 2000). It is important to identify the origin and potential sources of PAHs in the environment to assess the environmental risk (Nyong *et al.,* 2020; Akanimo *et al*, 2021).

Total Petroleum Hydrocarbon (TPH) and Total Hydrocarbon Contents Using UV-Vis Spectrophotometric analysis: The total petroleum hydrocarbon (TPH) and Total Hydrocarbon Contents of the oil-based mud (OBM) and water-based mud (WBM) were analyzed using Ultraviolet–Visible Spectrophotometer. This is shown in Table 3. The TPH for OBM was 1.60 mg/kg. For the WBM, the TPH was 1.30 mg/kg. The results indicated a THC of 5.35mg/kg for OBM and a slightly close value of 5.15mg/kg for the WBM.

The TPH and THC concentrations were found to be higher in the OBM compared to WBM. In general, the results indicated that TPH < THC. However, the results obtained for the TPH and THC were far lower than 50mg/kg allowable for drilling mud in the environment recommended by the NUPRC (1991). Recent investigations of TPH and THC using UV-Vis spectroscopy (Dumka and Kingdom, 2018; Adewole *et al.,* 2010) have shown similar low concentrations for drilling mud. The results from this investigation also indicated that the analyses of TPH using GC-MS revealed a more comprehensive outline of the range of *n*-alkanes present in OBM and WBM respectively as shown in Table 2 compared to the analyses using UV-Vis Spectrophotometer.

Declaration of Conflict of Interest: The authors declare no conflict of interest

Data Availability Statement: Data are available upon request from the corresponding author.

Conclusion: The investigation revealed considerable variations in the concentrations of heavy metals and hydrocarbon profiles, indicative that drilling mud effluents are laden with these contaminants. The discharge of contaminated drilling mud into the environment without pre –treatment may result in pollution with adverse environmental impacts on aquatic, edaphic, and groundwater systems, if it permeates through aquifers and run-off. Hence, the findings from this study are critical when making the choice for subsequent disposal of drilling mud effluents. It is recommended that drilling mud resulting from drilling operations should be properly treated and managed before discharge to the recipient ecosystem.

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