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An Assessment of Surface Water Heavy Metal Background Properties: A Precursor to Baseline Pollution Control for Bituminous Sand Open Pit Mining

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Abstract: *The assessment of metals in water within the networks of surface water bodies in an area designated for future mine development is critical both for risk assessment, establishment of background reference and pollution control plans. In this study, samples of surface water bodies in the bituminous sand deposit area of Ondo State, Nigeria were obtained on rainy and dry seasons' basis to analyze the contents of nine heavy metals, seven of which belong to the priority pollutant metals (PPEs). The samples were analyzed for dissolved metal concentrations in both seasons and results obtained were analyzed using Statistical Package for Social Sciences (SPSS 20). Water quality indexes (WQI) of the water samples were also determined empirically. Statistical Levene t-test of equality of variances showed that there was a statistically significant difference in concentrations of dissolved heavy metals between rainy and dry seasons at P>0.05. The WQI of the rainy season indicated that 75% of sampled water was in unstable and 15% was classified to be of poor condition. Conversely, the dry season showed 52.94% of sample in excellent condition. The "poor" quality rating of most samples in the rainy season was largely due to the exceptional exceedances of two carcinogenic PPE metal (Cr, Cd) above the study reference limit which might be connected to increased precipitation-driven dissolution of initially stable adsorbed metals and the run offs/transport of sediments loadings into the water bodies.*

Keywords: *Heavy metal, surface water, bituminous sands, contaminants, pollutants, mining*

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I. Introduction

One of the challenges synonymous with mining of bituminous sand is the encroachment on natural water resources particularly surface water bodies[1,2]. This takes the form of undue incursion into the natural water systems, flow pattern and ecosystem functions. These may involve the introduction (disposal) of aesthetically unpleasant or toxicologically laden constituents into the natural body either directly or indirectly; abstraction of water for operational processes[3] or diversion; and alteration of groundwater gradient due to water influx within the perimeter of the pit into the cavity. Bituminous sand deposits are widely known to occur in sedimentary formations thus making the host environment water prone phreatic zone. Broadly speaking, impact on surface water could be grouped into two: (i) natural, such as direct precipitation, surface runoffs, flashfloods, material loadings from primary tributaries to secondary or tertiary ones, incursion of invasive plants and (ii) anthropogenic, these include all forms of human-induced inputs from domestic to industrial constituents [4]. The exploitation of bituminous sand deposit at or relatively close to the surface is usually carried out by open pit mining method [5]. The method entails the opening up of the earth surface by creation of open pits and in the process altering the landscape; which includes intersection and diversion of relatively small channels of water bodies. As these mining operations take place contaminants are introduced directly or indirectly into the water systems[6]. These inputs could be in the form of physical or chemical inputs, organic or inorganic constituents. Among the inorganic contaminants that mining introduces of the surface water, heavy metals are notorious for their prevalence and non-degradable nature[7]. Heavy metal contamination of the aquatic environment is a major international concern owing to its toxicity, abundance, and persistence in the environment[8].

The use of water is vital to the success of the mining and extraction processes just as it is to the sustenance of the ecosystems around the mine environment. Heavy metals released to surface water by mining activities may pose severe risks to human health[9] via drinking water and bathing and also deleterious effects to aquatic lives which sometimes could be lethal. Although, metals occur naturally both in ground and surface water bodies in very low quantities[8,10], the anomalous elevation of these metals above certain thresholds or limits portents serious threats to the aquatic system and other life forms including man and animals that depend on it. The health risk assessment is an efficient method for evaluating the relationship between the environment

and people's health, which can be quantitatively assessed in terms of hazard degree[4]. Concentration of trace metals in surface water systems could be influenced by weather conditions and water chemistry [11,12]. During wet season, trace metals level in water bodies can be influenced by surface runoffs from various land use activities in the catchment while in the dry season, the major factor is evaporation from water bodies, which can lead to an increase in the concentrations of contaminants as the dilution factor is removed.

As a rule of thumb, all intending mining operators must carry out environmental impact assessment of the would-be mine environment to establish baseline environmental signatures for operational reference in terms of environmental compliance. Surface and ground water systems within the watershed of a mining project are a prime natural resource of economic and social importance; as such the quality of the water bodies in the catchment of mine must be protected in the course of mining. The acquisition of pre-mining data on surface water quality in the study area will serve as reference base for surface water pollution control and monitoring as well as anthropogenic related risk assessment. Among indicators frequently examined for surface water quality are heavy metals contents. In the study area, heavy metals studies reported in literatures mostly centered on sediments with very few addressing the dissolved metal concentrations in surface water bodies. Fagbote and Olanipekun[13,14] had examined the contents of heavy metals on the soil and stream sediments of Agbabu, an adjourning community to the study area. Ayandiran *et al.*[15] examined the domestic utility of a river transecting the neighborhood of the study area and noted that the river had higher levels of the examined metals recorded in some locations more than established permissible standard. The groundwater metal concentration of the study area was explored by Michael et al[16].

II. Materials And Methods

Geographical Location of Study Area

The study area is located in the bituminous belt of Southwestern part of Nigeria within latitudes 6° 38' 17.45" N and 6° 39' 6.69" N; and longitudes 4° 49' 48.27" E and 4° 53' 22.62" E respectively.

Geological Setting

The study area lies within the bituminous sand belt of Southwestern Nigeria in the Benin (Dahomey) basin but restricted to the eastern portion of the basin (the Okitipupa structure). The Benin Basin covers both the onshore and offshore parts[17] with the onshore part comprising the areas where both the Cretaceous and Tertiary sedimentary rocks are exposed along road cuts and quarries. The deposit is contained generally in the upper cretaceous sediments lying inland near the boundary between the coastal plain and the uplands[18]. In geologic terms, the bituminous sand spans the "Ilesha Spur or Okitipupa High" - a structural and slight topographic divide which is the highest upthrown block of the post-Santonian horsts in the basin[19].

The Drainage of the study Area

The drainage system of the study area is sub-dendrite, and characterized by irregular branching of tributary streams in many directions at almost any angle [20]. The temperatures range from 22-32°C while the average annual rainfall varies from 150-160 cm[21].

Sample collection

In this study, surface water bodies were selectively sampled during the dry season of the year 2016 and rainy season 2017. A total of thirty seven (37) grab samples were collected from surface water bodies (River, streams) at various locations both in the rainy and dry seasons. The sample collection procedure was in accordance with the United States Environmental Agency[22] protocol using uncontaminated 1- litre sized plastic bottles treated with 1:1 diluted hydrochloric acid and rinsed with distilled waters as containers. After collection, the sample containers were sealed, labeled, ice-packed and immediately taken to the laboratory for analysis.

Sample Analysis

All obtained water samples were filtered through 45μm membranes filters. Ten (10 mL) of each sample filtrate was acidified with 0.5 ml concentrated nitric acid (HNO_3). The solutions were digested accordingly as prescribed in the Methods for the Examination of Water and Wastewater by American Water Works Association/American Public Works Association/Water Environment Federation[23]. The samples were analyzed for heavy metal concentrations of nine elements out of which seven are listed among the thirteen priority pollutant elements (PPE) heavy metals by USEPA water Act, chromium (Cr), cadmium (Cd), manganese (Mn), copper (Cu), lead (Pb), nickel (Ni), zinc (Zn), iron (Fe) as dissolved (filterable) heavy metals and magnesium (Mg) as alkaline earth metal using nitric acid (HNO_3) digestion. The concentrations of metals in each sample digests were then analyzed by atomic absorption spectrometry (AAS) using Bulk Scientific 210 VGP Flame spectrometer. The pH and total dissolved solids (TDS) were analyzed using electronic pH probe Jenway 3510 and conductivity probe respectively.

Statistical Analysis

Data from laboratory analysis were statistically analyzed using IBM statistical Package for Social Science version 20 (IBM SPSS 20). Descriptive statistics of the obtained data were conducted and comparison of variance between the means of metal concentration in rainy and dry seasons were carried out using the Student's T-test to explore the differences between the seasonal values of measured parameters during the rainy and dry seasons. Also, the water quality index was determined in accordance to [24,25] in equations (1-4) using the United States Environmental Protection Agency (USEPA) reference standards of maximum contaminant limit (MCL).

$$Q_i = \left[\frac{C_i}{V_{si}}\right] \times 100 \quad (1)$$

$$K = \frac{1}{\sum_{i=1}^{n}\left(\frac{1}{V_{si}}\right)} \quad (2)$$

$$W_i = \frac{K}{V_{si}} \quad (3)$$

$$WQI = \frac{\sum_{i=1}^{n} Q_i W_i}{\sum_{i=1}^{n} W_i} \quad (4)$$

Where: C_i is the measured value, V_{si} is the standard reference value, Q_i is the quality rating, K is the proportionality constant, W_i is the relative weight, n is number of pollutant parameters, WQI is the water quality index. The evaluation of the water quality is assessed based on the following criteria.
WQI < 50: Excellent; 0 < WQI < 100 : Good; 100 < WQI < 200: Poor; 200 < WQI < 300: Very Poor; WQI > 300: Unstable

III. Results

Results of Water Samples Analysis

Results of the water samples analysis for the rainy season as presented in Table 1 showed that the pH ranged between pH 4.5 – pH 8.5 with a mean pH of 6.89 ± 0.193 se(Figure 1a,b). Total dissolved solids within the same period were within 11-910 (mg/L) range with a mean of 120.45 ± 56.54 se (mg/L) . The values of metal concentration of various sampled water during the rainy season were comparatively low. Cadmium concentration of sampled water recorded a maximum of 0.02 mg/L and while it was not detected (ND) in some samples during the rainy season. The mean value of Cd was 0.003 ± 0.001 se (mg/L). Chromium, Lead and Nickel were in the range 0.01-0.260 (mg/L), 0.01-0.100 (mg/L) and ND - 0.02 (mg/L) respectively. The mean values for chromium was 0.057 ± 0.013 se (mg/L); lead 0.027 ± 0.006 se (mg/L) and Nickel 0.009 ± 0.002 se (mg/L). Other heavy metal background concentrations analyzed for in the rainy season are shown in Table 1.

Table 1: Descriptive Statistics of Heavy Metal Contents of Water Samples in the Study Area (mg/L) (Rainy Season)

Statistics	pH	TDS	Cd	Cr	Mg	Mn	Cu	Pb	Ni	Zn	Fe
Statistics	pH	TDS	Cd	Cr	Mg	Mn	Cu	Pb	Ni	Zn	Fe
Min.	4.5	11.00	ND	0.010	0.920	0.020	0.010	0.010	ND	0.010	0.020
Max.	8.5	910.00	0.020	0.260	5.700	0.710	0.260	0.100	0.040	0.810	0.980
25 %tile	6.475	25.00	ND	0.020	1.130	0.048	0.010	0.010	ND	0.045	0.058
50 %tile	7.000	30.50	ND	0.040	1.885	0.090	0.030	0.015	0.010	0.150	0.295
75 %tile	7.400	53.25	ND	0.060	2.103	0.170	0.103	0.030	0.010	0.330	0.470
Mean	6.890	120.45	0.003	0.057	1.854	0.160	0.071	0.027	0.009	0.218	0.346
Var	0.748	63938.68	ND	0.004	1.042	0.035	0.006	0.001	ND	0.048	0.115
Std dev	0.865	252.85	0.006	0.060	1.021	0.188	0.077	0.028	0.010	0.218	0.339
Std err(mean)	0.193	56.54	0.001	0.013	0.228	0.042	0.017	0.006	0.002	0.049	0.076

ND: Not detected

The statistical results of water heavy metal analyses in the dry season are presented in Tables 2. Analysis of water for the dry season showed that the pH ranged from pH 4.45 – pH 7.19 (Figure 1c, d) with a mean pH of 5.906 ± 0.176 se. The value of the total dissolved solids for the period of sampling in dry season was between 14 and 162 (mg/L) (Figure 1f)with a mean of 37.29 ± 8.592 se (mg/L). The metal concentration of various sampled water during the dry season also varied slightly from those recorded during the rainy season indicating the effect of seasonal variations. From the analysis, cadmium concentrations in the samples ranged from non-detectable to a maximum of 0 0.02 mg/L with a mean value of 0.004 ± 0.001 (mg/L). Chromium

concentration ranged from less than detectable in some samples to 0.08 mg/L with a mean of 0.018 ± 0.006 se (mg/L).

Table 2: Descriptive Statistics of Heavy Metal Contents of Water Samples in the Study Area (mg/L) (Dry Season)

Statistics	pH	TDS	Cd	Cr	Mg	Mn	Cu	Pb	Ni	Zn	Fe
Min.	4.45	14	ND	ND	0.210	ND	ND	ND	ND	0.010	0.010
Max.	7.19	162	0.020	0.080	63.000	0.260	0.060	0.060	0.030	0.120	0.560
25 %tile	5.700	22.00	ND	ND	0.620	0.020	ND	0.010	0.010	0.020	0.020
50 %tile	5.890	28.00	ND	ND	0.890	0.030	ND	0.010	0.010	0.030	0.050
75 %tile	6.300	32.00	0.010	0.030	1.130	0.050	0.010	0.020	0.020	0.050	0.070
Mean	5.906	37.29	0.004	0.018	11.537	0.046	0.009	0.018	0.013	0.038	0.079
Var.	0.529	1255	ND	0.001	571.067	0.003	ND	ND	ND	0.001	0.016
Std dev.	0.728	35.42	0.006	0.027	23.897	0.059	0.018	0.013	0.010	0.028	0.128
Std err (mean)	0.176	8.592	0.001	0.006	5.796	0.014	0.004	0.003	0.003	0.007	0.031

ND: Not detected

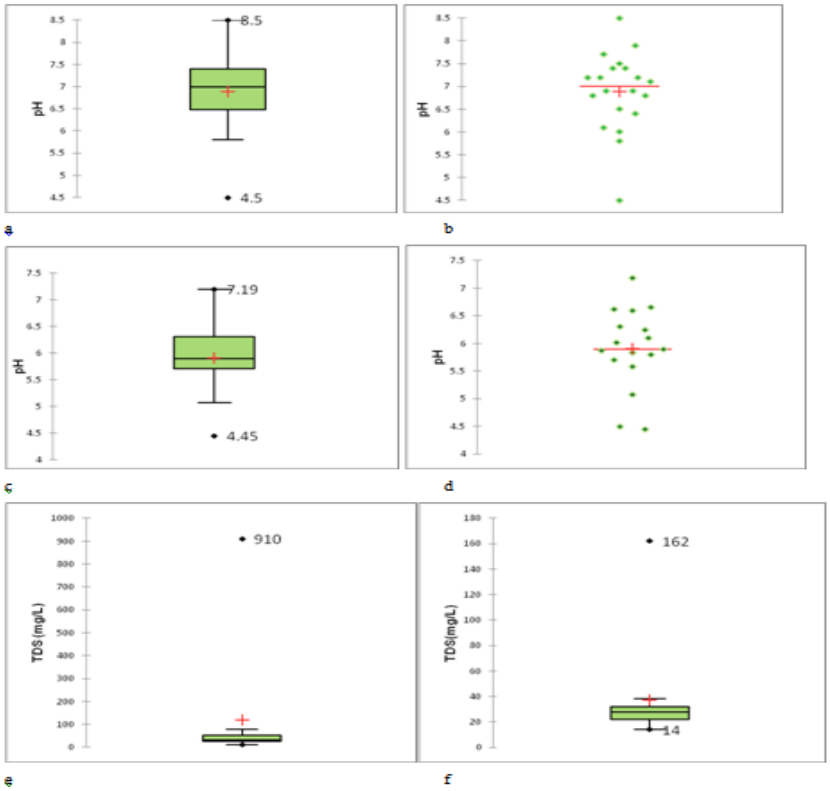

Figure 1: Indicator Chemical Parameters of Surface Water Bodies in Study Area in both Seasons. a) Boxplot and b) Scattergram of Water pH in Rainy Season; c)Boxplot and d) Scattergram of Water pH in Dry Season; (e and f) Boxplots of Dissolved Solids in Water in Rainy and Dry Seasons respectively

Lead and Nickel values ranged between ND to 0.06 (mg/L) and ND - 0.03 (mg/L) respectively. The mean value of lead was 0.018 ±0.003 se (mg/L) and that of nickel 0.013 ± 0.003 se (mg/L).

In Figures 2 the comparative charts of the log means of water heavy metal contents is shown. These results show that the heavy metal concentrations were slightly higher in rainy season than in dry season except for Mg and Ni which had higher values recorded in the dry season. The group statistical figures for each of these metals are presented in Table 3.

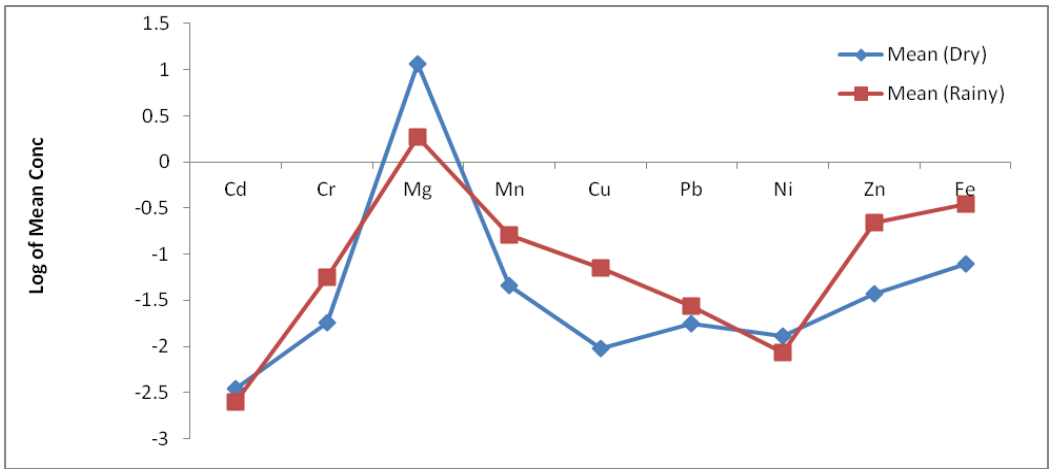

Figure 2: Comparative Log Plots of Mean Metals Concentrations in Rainy and Dry Seasons

The result of the comparable analysis of metal concentrations in both seasons as reflected by the independent samples t-test (Table 4) shows that the null hypothesis stating no statistically significant difference exists between the means of water samples metal concentrations for both seasons cannot be maintained. Hence, the acceptance of alternative hypothesis indicating statistical significance differences between the seasonal means of metals concentrations. According to the Levene test for equality of variance in Table 4, concentrations of heavy metals Cd, Cr, and Ni are not significantly different in both seasons as p is higher than the alpha level i.e. $p>0.05$. However, the remaining metals Mg, Mn, Cu, Pb, Zn and Fe are significantly different at $p < 0.05$. Based on the t-test for equality of means in the table, mean concentrations of Mn, Cu, Zn, and Fe were significantly different at $P<0.05$. The negative t- values indicate that the mean values of respective metal concentrations in the dry season are higher than those of the rainy season. This applies to element Mg and Ni as earlier stated and shown in Figure 2. Conversely, the values of dissolved Cu, Pb, Zn, and Fe are higher in the rainy season than in the dry season.

Table 3: Group Statistics of Metal Concentrations in Surface Water (Rainy and Dry seasons)

Group		N	Mean	Std. Deviation	Std. Error Mean
Cd	Rainy Sn	20	0.00250	0.005501	0.001230
	Dry Sn	17	0.00353	0.006063	0.001471
Cr	Rainy Sn	20	0.05650	0.059760	0.013363
	Dry Sn	17	0.01824	0.026513	0.006430
Mg	Rainy Sn	20	1.85350	1.020894	0.228279
	Dry Sn	17	11.53706	23.897004	5.795875
Mn	Rainy Sn	20	0.15950	0.187770	0.041987
	Dry Sn	17	0.04624	0.058906	0.014287
Cu	Rainy Sn	20	0.07050	0.076741	0.017160
	Dry Sn	17	0.00941	0.017843	0.004328
Pb	Rainy Sn	20	0.02700	0.028116	0.006287
	Dry Sn	17	0.01765	0.013477	0.003269
Ni	Rainy Sn	20	0.00850	0.009881	0.002209
	Dry Sn	17	0.01294	0.010467	0.002539
Zn	Rainy Sn	20	0.21750	0.217954	0.048736
	Dry Sn	17	0.03765	0.028180	0.006835
Fe	Rainy Sn	20	0.34550	0.338673	0.075730
	Dry Sn	17	0.07882	0.127568	0.030940

Table 4: Independent Samples T-Test of Surface Water Samples in Study Area

		Levene's Test for Equality of Variances		T-test for Equality of Means						
		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	99% Confidence Interval of the Difference	
									Lower	Upper
Cd	Equal variances assumed	0.737	0.396	-.541	35	0.592	-0.001029	0.001902	-0.006210	0.004151
	Equal variances not assumed			-.537	32.730	0.595	-0.001029	0.001917	-0.006272	0.004214
Cr	Equal variances assumed	1.800	0.188	2.440	35	0.020	0.038265	0.015683	-0.004452	0.080981
	Equal variances not assumed			2.580	27.093	0.016	0.038265	0.014830	-0.002813	0.079342
Mg	Equal variances assumed	24.398	0.000	-1.815	35	0.078	-9.683559	5.335817	-24.217287	4.850170
	Equal variances not assumed			-1.669	16.030	0.114	-9.683559	5.800369	-26.618246	7.251128
Mn	Equal variances assumed	9.594	0.004	2.385	35	0.023	0.113265	0.047492	-0.016094	0.242624
	Equal variances not assumed			2.554	23.284	0.018	0.113265	0.044351	-0.011108	0.237637
Cu	Equal variances assumed	17.850	0.000	3.203	35	0.003	0.061088	0.019072	0.009139	0.113037
	Equal variances not assumed			3.452	21.391	0.002	0.061088	0.017697	0.011071	0.111105
Pb	Equal variances assumed	4.133	0.050	1.253	35	0.219	0.009333	0.007466	-0.010982	0.029688
	Equal variances not assumed			1.320	28.211	0.197	0.009333	0.007086	-0.010217	0.028922
Ni	Equal variances assumed	0.650	0.426	-1.326	35	0.193	-0.004441	0.003349	-0.013564	0.004682
	Equal variances not assumed			-1.320	33.320	0.196	-0.004441	0.003365	-0.013634	0.004752
Zn	Equal variances assumed	25.273	0.000	3.371	35	0.002	0.179833	0.053346	0.034548	0.325118
	Equal variances not assumed			3.655	19.746	0.002	0.179833	0.049213	0.039638	0.320028
Fe	Equal variances assumed	16.339	0.000	3.062	35	0.004	0.266676	0.087095	0.029447	0.503906
	Equal variances not assumed			3.260	25.044	0.003	0.266676	0.081808	0.038679	0.494674

Results of assessment of sampled water quality with respect to dissolved metals in the study area showed that there were marked differences in the qualities of respective samples especially on seasonal basis as shown in Tables 5 and 6. This is indicated by the WQI for which weighted concentrations of respective contributing metals were calculated. The quality or each of sample is expressed quantitatively and qualitatively in Table 5 while the aggregate quality of all samples in both seasons are expressed in Table 6.

Table 5:Water Quality Index of Sampled Surface water

Rainy season			Dry Season		
Sample ID	QWI	Classification	Sample ID	WQI	Classification
WR1	511.1289	Unstable	WD1	601.2673	Unstable
WR2	411.9813	Unstable	WD2	14.93841	Excellent
WR3	147.9509	Poor	WD3	39.24861	Excellent
WR4	634.9634	Unstable	WD4	622.1436	Unstable
WR5	354.6178	Unstable	WD5	29.50714	Excellent
WR6	112.5999	Poor	WD6	351.3511	Unstable
WR7	151.621	Poor	WD7	16.17171	Excellent
WR8	436.4864	Unstable	WD8	409.6868	Unstable
WR9	271.4453	Very Poor	WD9	271.4453	Very Poor
WR10	300.9722	Unstable	WD10	300.9722	Unstable
WR11	627.9971	Unstable	WD11	832.2436	Unstable
WR12	425.3268	Unstable	WD12	35.39345	Excellent
WR13	638.4346	Unstable	WD13	25.14049	Excellent
WR14	219.9891	Very Poor	WD14	59.50402	Good
WR15	1119.438	Unstable	WD15	11.26624	Excellent
WR16	1650.018	Unstable	WD16	16.70953	Excellent
WR17	524.7335	Unstable	WD17	13.46001	Excellent
WR18	2682.094	Unstable			
WR19	679.9683	Unstable			
WR20	419.9344	Unstable			

Table 6: Seasonal Comparison of Water Quality Indices based on Background Dissolved Metals

Quality Index Class	Rainy Sn	Dry Sn
	Percentage sample Size	
Excellent	Nil	52.94%
Good	Nil	5.88%
Poor	15%	Nil
Very Poor	10%	5.88%
Unstable	75%	35.29%

IV. Discussion

In the study, it was observed that the overall surface water sampled had relatively low concentrations of dissolved metals in the study area with the exceptions of Cadmium and Chromium. By inference, the study area had not been seriously impacted by excessive extraneous loads of heavy metals particularly through anthropogenic activities. The pattern and level of dissolved metals in the water bodies reflect a dynamic geogenic source resulting from dissolved natural sediments that enter the water bodies as run offs or those mobilized by other forms of biologically driven mechanisms. According to the study few exceedances above USEPA minimum contaminant level occurred in individual metal cases. These occurrences include Cd in sample location WR which had 0.02 mg/L two folds the value of USEPA maximum contaminant limit (MCL). Similarly, the value of chromium in all the samples exceeded the MCL value with at least two folds in three locations why the highest was by as much as 56 folds in sample WR 18 in the rainy season. In the sample the value of tolerance (for drinking) was exceeded by Mn with about 11 folds the set limits. The MCL level of manganese (0.05mg/L) was also exceeded in the rainy season in samples WR5, WR9, WR 10-20. The reason for these exceedances might be due to greater erosive activities of rain during the rainy season which tends to sweep sediments from diverse overland sources within the water shed of the study area into these water bodies. In the dry season samples there were no occurrences of exceedance in threshold MCL values as compared to the rainy season. It must be noted here that the basis for the use of a stricter reference standard (the USEPA drinking water reference) for measuring the quality of the surface water bodies in the study was due to the fact that water is occasionally withdrawn from these sources for domestic consumption. Also activities of fishing are common especially on the main river that traverses the west bank of the study area.

With the exception of Cadmium and Chromium in the rainy season samples, all other metals were within the threshold limits. Since these metals hardly remain in solution due to their reactivity they are likely to be absorbed into the surfaces of particulate materials such as organic matter or colloidal suspensions in the water bodies. From the study statistical analysis, the pH records showed that the there were two extreme values pH 4,5 in the rainy season and 4.45 pH. These occurrences might be some anomalies occasioned by sample analysis or probably attributed to the sampling locations which were located close to receiving ends of local palm oil processing effluent discharge point and a rallying point for people in the community for washing and bathing respectively.

According to the study, water quality index shows poorer quality of water during rainy season (75%) compared to dry season (35.29%). It implies that higher dissolution of metals takes place during the rainy season as sediments are transported from ground surfaces through drainages and stream channels. It has been observed that metals exist in different forms in the surface water environment which include the colloidal, particulate or dissolved phases. The soluble form constitutes the lowest quantitatively compared to other forms and are generally in ion or unionized organometallic chealates. Judging from the exertive influence of pH on the solubility of heavy or trace metals in surface water, descriptive result of rainy season proves that mean of water samples pH (6.980) which is lower than the 50% percentile of the rainy season's samples pH distribution could be yet another indicator of why there is higher metal dissolution in the water samples in rainy more than in dry seasons. It is thus important to discreetly plan management strategy for surface water abstraction during the mining process in order to prevent further degradation of the water bodies beyond buffering capacity. This will ensure that the various water bodies maintain the ability to render their aquatic ecosystem functions.

V. Conclusion

This study concludes that heavy metal concentrations in surface water in the study area vary significantly from one season to another. The basis for this might be related to the influx of metal laden materials from the ground surface including eroded sediment that navigate into the water bodies and possibly greater dissolution and other activated dynamic chemical processes that take place more actively during the rainy season than the dry season.

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