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Physicochemical Parameters and Heavy Metals Characterization of Soil from Okpella Mining Area in Edo State, Nigeria.

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Abstract
The present study examines the physicochemical parameters and heavy metal contents of soil from Okpella Mining Area in Edo State. Top soil samples and control samples from about 200m from the mining area were analyzed for their heavy metals content using Atomic Absorption Spectrometer (AAS) techniques. The pH, electrical conductivity and organic matter were determined using standard procedures. The soil pH ranged from 6.8 to 7.9 which show slightly acidic to alkaline conditions. The concentrations of the metals in mg/kg were as follows: Cu (23.03 to 170.83), Pb (3.50 to 10.59), Cd (0.70 to 1.93), Cr (0.75 to 8.74), Ni (5.0 to 34.52), Zn (3.29 to 25.46), Mn (3.49 to 22.50), Fe (22.03 to105.67), Co (9.99 to 64.43) and As (0.35 to 1.20). Inter elemental correlation showed that most of the metals investigated were of common origin. The mean concentrations of the metals were in the general order: Cu>Fe> Co> Ni> Zn>Mn>Pb>Cr>Cd>As. The concentrations of the metals at mining area were generally higher than control. With the exception of Cd, the concentration of other metals analyzed were within safe limit based on WHO standard. Results from pollution index assessment also show that Cd has higher degree of contamination. There is therefore need to monitor agricultural activities going on in this area so as to guard the safety of man and organisms in the area.

Keywords: Mining, heavy metals, Pollution, Okpella, Limestone and Soil.

1. INTRODUCTION

The disruption of heavy metals from their geochemical state through anthropogenic activities and the inherent health implications has called for concern in recent times (Kabir *et al.*, 2017). Contributions from various sources such as mining, agriculture, industrialization amongst others constitute the major source of soil pollution in the environment (González *et al.*, 2014). Thus soil pollution by heavy metals has become a wide spread problem globally.

Soil is a natural sink for various pollutants as well as metals from different points in the environment, thus these metals are absorbed by plants and passed across the food chain (Fonge *et al.*, 2017).The trace element content of soil depends on the nature of its parent rocks and also the amount of sewage effluents, industrial wastes and fertilizer impurities entering the soil (Nnabo, 2015). These metals have become issue of concern in recent times due to their deleterious effect on health. They also have a unique tendency to bio-accumulate along the food chain and are not biodegradable (Chao *et al.*, 2014).

The major routes through which man gets in contact with these metals are food, air, water, etc. Various heavy metals show varying level of toxicity to life. Metals such as cadmium, lead and mercury have been reported to have no physiological functions in organism and yet are highly toxic even at minute concentrations (Yu *et al.*, 2017). Copper is known to be vital to health at a low concentration but very toxic at high level. Exposure to high dose of chromium can result in lung cancer, inflammation of skin amongst others, while lead is associated with severe damages of vital body organs such as brain and kidney (Rana *et al.*, 2010).

Considering the lethal effects of high concentrations of these metals in soil, it becomes pertinent to continually monitor their contents as a critical measure for environmental management and remediation (González *et al.*, 2014).

There are available studies on the extent of environmental contamination by heavy metals due to mining activities in different parts of the world. There is however scarcity of documented studies on heavy metal content in Okpella Mining area of Edo State, Nigeria, where the majority of people are rural farmers and locally employed workers in the cement factories.

The present research therefore aims at studying the heavy metals characterization of soil from Okpella mining area in Edo State, Nigeria.

2. MATERIALS AND METHODS

Description of study area

Okpella is located in Etsako East Local Government area of Edo State, Nigeria as shown in Figures 1.1 and 1.2. It is situated along Benin-Abuja federal high way. It falls within latitude 7.2721° and longitude 6.3465°. It is known for its limestone mining activities. In view of the abundance of other solid minerals, it is a home of granite- and marble-making industries (Okoson, 2010).

Figure 1.1: Map of Nigeria Showing the Location of Edo State

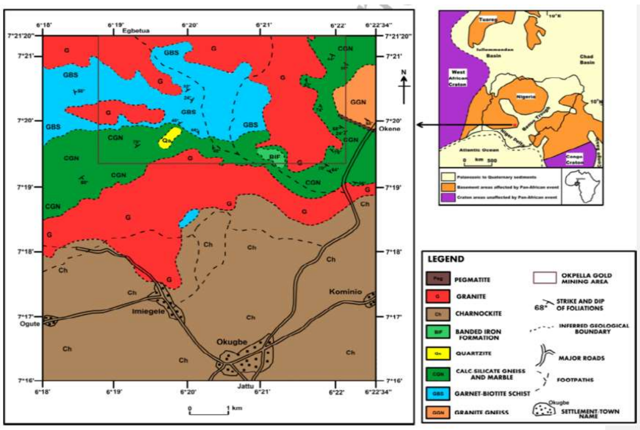

Figure 1.2: Map of Edo State Showing the Location of the Study Area (Okpella)

Soil sampling and Pre-treatment

A total of 50 soil samples were collected at different sampling points from the vicinities of the mining areas. The samples were collected using a soil auger, with proper decontamination after each point so as to avoid cross contamination. The top soils (0-15 cm) collected were homogenized so as to obtain 10 composites in all. Two control samples were also taken from about 500 m radius away from the study area.

The samples were air-dried for two days and then ground using mortar and pestle. They were then sieved using 2 mm and 0.5 mm mesh in line with Rana *et al.*, 2010. The samples were kept in polythene bag and further analysis carried out in the Chemistry Laboratory of Edo University Iyamho, Nigeria.

Determination of physicochemical properties of the soil

The organic matter content of the samples was determined using dichromate wet oxidation method (Walkley and Black, 1934). The pH of soil was determined using glass electrode pH meter in KCl in line with Jones (2001). The electrical conductivity (EC) was measured using a conductivity meter as reported by Carter and Gregorich, (1994).

Determination of metals

Digestion of soil samples was done using three different acids (Nitric, Sulphuric and Hydrochloric acid) in line with Ekwue *et al.*, 2012. Heavy metal content was determined using Atomic Absorption Spectrometer (AAS: model solar 969 Unican series). Results obtained were presented as mean ± SD (Standard deviation) for triplicated determinations, analysis of variance (ANOVA) was also done to check for significant differences. Interelemental correlation was verified using Pearson's Correlation.

Computation of Limit of Detection (LOD) and Limit of Quantification (LOQ)

Limit of detection (LOD) and limit of quantification (LOQ) were computed using linear regression method in line with Shrivastava and Gupta 2011. LOD and LOQ were expressed as:

$$LOD = 3Sa / b \quad (1)$$

$$LOQ = 10Sa / b \quad (2)$$

where S_a is the standard deviation of the response and *b* is the slope of the calibration curve. The standard deviation of the response can be estimated by the standard deviation of either *y*-residuals, or *y*-intercepts, of regression lines.

3. RESULTS AND DISCUSSION

Tables 1 and 2 show the result of the metals analyzed values in quantification and Organic matter (OM) obtained from the study. There were variations in the organic matter content among the sampling areas. Soil organic matter is a key factor in determining water retention ability, cation exchange capacity as well as the mobility of heavy metals in soil (Fonge *et al.*, 2017). According to Olatunji and Osibanjo, (2012), the sorption capacity of soils is related to the soil organic matter and clay minerals. The OM content obtained varied from 1.01 to 4.20%. The mean OM content was 4.14%. The electrical conductivity (EC) values differ significantly among the various sampling areas. The EC ranges from 745 to 1408µs/cm, which shows that there is variation in the dissolve salts present in the various sampling areas. The highest EC was at sampling point 6. The mean EC from the study was 1163.9 µs/cm.

Table 1: Operating condition and other parameters of the metals analyzed values in quantification of metals

Metal	Maximum current (mA)	r^2	LOD (mg/kg)	LOQ (mg/kg)
Copper	15	0.99	0.05	0.10
Cadmium	15	0.99	0.10	0.11
Lead	10	0.97	0.05	0.08
Iron	25	0.99	0.09	0.10
Zinc	15	0.95	0.03	0.05
Manganese	20	0.99	0.05	0.09
Nickel	30	0.91	0.02	0.05
Chromium	20	0.95	0.01	0.10
Cobalt	10	0.95	0.04	0.09
Arsenic	15	0.97	0.02	0.05

Table 2: Physicochemical parameters of soil samples

Samples	pH (water)	OM (WB)	EC (µs/cm)
1	7.0±1.01	2.89	1016.4±6.20
2	6.9±0.90	3.05	775.1±5.00
3	6.8±1.00	3.11	1361±1.01
4	7.9±1.02	3.00	1221±4.10
5	7.2±0.80	4.12	1258.4±1.98
6	6.8±0.01	2.11	1408.1±2.11
7	7.1±0.23	4.20	1337.8±0.90
8	7.3±1.00	3.89	1144.8±4.09
9	6.9±0.40	1.01	744.9±1.90
10	7.3±0.10	2.91	1371.5±2.14
Mean	7.1	3.03	1163.9±3.24
Con1	7.2±0.80	2.90	845.0±2.11
Con2	7.0±0.20	3.00	483.8±3.10

Results for some parameters are expressed as mean ±SD for triplicate determinations.

Table 3: Heavy metal content in soil (mg/kg)

Sample	Cd	Cr	Ni	Zn	Pb	Cu	Mn	Fe	Co	As
SP 1	2.31±0.89^{g}	8.74±0.65^{i}	23.98±0.50^{h}	22.70±0.90^{e}	10.97±0.98^{e}	88.09±0.90^{d}	15.25±0.89^{d}	69.07±0.75^{c}	51.03±0.90^{d}	0.89±
SP 2	0.99±0.10^{b}	1.24±0.09^{b}	10.10±1.08^{b}	9.19±0.78b	4.03±1.00^{b}	30.06±0.65^{b}	4.72±0.65^{b}	33.46±0.45^{b}	18.49±1.03^{b}	0.35±
SP 3	2.08±0.01^{f}	4.45±0.23^{h}	32.10±0.89^{i}	18.60±0.27^{c}	10.48±0.56^{d}	81.05±1.01^{c}	13.51±1.02^{c}	70.47±0.89^{e}	51.43±0.89^{d}	1.06±
SP 4	1.59±1.09^{c}	3.74±0.90^{f}	22.49±1.06^{g}	22.35±0.67^{e}	10.97±0.35^{e}	99.08±0.93^{g}	18.17±0.90^{e}	79.53±1.11^{g}	55.10±0.02^{h}	0.79±
SP 5	2.48±0.67^{h}	3.99±0.23^{g}	21.96±0.34^{f}	25.46±0.78^{f}	15.43±0.99^{i}	91.90±1.00^{f}	19.19±0.67^{f}	76.03±0.97^{f}	49.01±1.08^{c}	0.90±
SP 6	1.85±0.80^{e}	2.76±0.67^{d}	34.52±0.25^{j}	19.84±0.98^{d}	13.98±1.02^{h}	90.39±1.20^{e}	20.17±1.11^{g}	105.67±0.56^{i}	55.04±1.22^{h}	0.86±
SP 7	3.11±0.23^{i}	3.26±0.09^{e}	16.03±1.22^{c}	19.89±024^{d}	10.05±0.43^{c}	90.99±0.34^{e}	22.50±1.92^{h}	105.63±1.21^{i}	49.01±0.98^{d}	1.00±
SP 8	1.69±0.09^{d}	0.75±0.09^{a}	19.51±0.65^{d}	23.33±0.79^{f}	13.99±0.35^{h}	133.00±1.29^{h}	13.43±0.09^{c}	66.48±0.01^{d}	53.46±0.67^{e}	1.20±
SP 9	0.70±0.78^{a}	3.24±0.78^{e}	5.00±0.45^{a}	3.29±0.89^{a}	3.50±1.00^{a}	23.03±0.99^{a}	3.49±0.23^{a}	22.03±0.56^{a}	9.99±1.21^{a}	0.40±
SP 10	2.50±0.89^{h}	2.65±0.04^{c}	21.07±1.02^{e}	18.13±0.01^{c}	12.53±0.35^{g}	170.83±1.23^{i}	15.43±1.01^{d}	85.23±1.10^{h}	64.43±1.03^{f}	1.00±
mean	1.93	3.48	20.68	18.28	10.59	89.84	14.92	71.36	45.77	0.84
Con1	0.68±0.43	0.67±0.19	7.32±1.03	2.98±0.18	2.10±0.34	24.10±0.99	2.10±0.18	15.10±1.11	6.92±1.01	0.21±
Con2	0.94±0.12	1.01±0.45	4.17±0.90	3.60±1.13	0.90±0.64	15.20±0.98	3.10±0.45	10.33±0.78	10.12±0.18	0.15±

Results expressed as mean±SD for triplicate determinations. Values with same superscript on the same column do not differ significantly at $p < 0.05$. SP: Sampling Point. Con: control

There were variations in the physico-chemical properties and heavy metals content in the studied area The pH ranged from 6.8 to 7.9. This shows that the soil in this area is slightly acidic to alkaline in nature. Generally, heavy metals tend to be mobile and easily leached at lower pH. As the soil becomes alkaline, the metals are retained and accumulated on the surface soil making them more readily available for plant uptake (Fonge, et *al*., 2017). The pH of soil in this area could be attributed to the additive input from agricultural activities, pesticide use among others (Wei *et al*., 2005). The pH gives a measure of the acidity or exchangeable hydrogen ions (H^+) present in the soil. Rana *et al*., (2010), reported that pH has major influence on solubility of metals and soil anion exchange capacity and thus the comparative abundance of heavy metals in soils.

The results of the heavy metals in the soil samples were shown in Table 3. The results were compared to permissible limits based on international standards. The concentration of Cadmium varied from 0.70 to 2.50 mg/kg. There was a significant difference in the concentration of Cd among the sampling areas. Sampling point 7 had the highest concentration; which may be related to the pH of soil in this area since heavy metal accumulate more in soil surface with higher pH. The minimum concentration of Cd obtained is higher than 0.63 mg/kg reported by Sitil *et al*., (2014) in a similar study. It is also higher than the WHO permissible limit of 0.3mg/kg for Cd in soil. It is however lower than 12.62-20.70 mg/kg recorded by Kabir *et al*., (2017) and 6.57 mg/kg reported by Alonge, *et al*., (2017) in a related study. High content of Cd can affect the metabolism of calcium, which can result to calcium deficiency, cartilage disease and bone fractures (Modaihsh *et al*. 2004; Chao *et al*., 2014). Cd has no known function in soil and bodies of living organisms. Even at a low concentration it is toxic to animals causing cardiovascular diseases, renal abnormalities among others (Kabata-Pendias, 2000).

The IARC has classified Cd compounds as carcinogenic to humans (Yu, *et al*., 2017). Arsenic had the least concentration among all the metals analyzed. There was no significant difference in the concentration of As between sampling area 1, 3 and 5. The mean concentration was 0.84 mg/kg. The As content of the studied soil was also higher than that of the control. The mean concentration obtained for arsenic in this study is lower than 89.03 mg/kg reported by Yu *et al*., (2017) in a related study. As-containing compounds, such as potassium arsenite, are highly toxic and carcinogenic to humans. Antwi-Agyei *et al*., 2009 reported the concentration of As in Ghana mining area as 581mg/kg which is higher than the value from this study. The concentration of Chromium varied among the different sampling areas. The highest amount of Cr obtained was at sampling point 1 (8.74 mg/kg), while the mean concentration in the study was 3.48 mg/kg.

The control showed the least level of contamination by Cr. The lower content at this point may be related to the distance away from the mining site (Kitula, 2006). Olatunji and Osibanjo, (2012) documented the concentration of Cr as 8.18–14.89 mg/kg in a related study, which is higher than the mean concentration of 3.28 mg/kg from this study. Zinc is a component of superoxide dismutase enzyme, and also takes part in neurotransmission and immune functioning (Yu, *et al*., 2017). The mean content of Nickel was 20.68 mg/kg. The concentration among the soil investigated ranged from 5.00 to 34.52 mg/kg. There were significant differences in the content of Ni among the various sampling areas. The mean concentration of Ni (20.68 mg/kg) is comparable to 11.99±2.71 to 20.84±2.09 mg/kg reported by Olatunji and Osibanjo, (2012); Ni and Cu have tendencies to induce tumors in human and their carcinogenic effect has attracted global concerns.

Workers who come in close contact with the nickel powder are more likely to suffer from respiratory cancer (Chen, 2011). The least content of Zn obtained in this study (3.29 mg/kg) was at sampling point 9. The highest concentration was 22.70 mg/kg. The mean concentration among the various sampling areas was 18.28 mg/kg. The mean content of Zn obtained here is lower than the permissible limit of 50 mg/kg reported by WHO, 2008; 185.56 to 373 mg/kg recorded by Kabir *et al*., 2017 in a related study as well as 498 mg/kg reported by Zhuang *et al*.,(2009) in Daba Oshan mines in South China. The value for Zn from this study is however higher than 14.15mg/kg reported by Fonge *et al*., (2017) in a related study. The mean content of Mn obtained in this study was 14.92 mg/kg. The value obtained here is lower than 111.04 to 247.62 reported by Afzal *et al*., 2013. It is also lower than the permissible level of based on international standard.

The concentration of Mn from this study is comparable to 6.39-20.31mg/kg reported by Olatunji and Osibanjo, (2012) in their study in Itakpe Fe mining region of Nigeria. High concentration of Mn causes hazardous effects on lungs and brains of humans (Jarup, 2003). Copper recorded the highest mean concentration among all the elements in this study. The concentration of Cu obtained in the sampling areas ranged from 23.03 to 170.83 mg/kg. The mean value of Cu obtained is higher than the permissible limit of 30 mg/kg based on WHO, 2008. This shows the high level of contamination due to mining activities in this area. The concentration of Cu is lower than 240.59 mg/kg documented by Sitil *et al*., 2014 in a related study. The maximum concentration of Cu is higher when compared to 42.0 mg/kg reported by Kabir, *et al*. (2017) in mining areas in Bauchi, Nigeria. The concentration of Cu in this area could also be attributed to the agricultural activities around this area, since copper compounds are commonly used as pesticides for agricultural pests (Zhou and Guo, 2015). Fonge *et al*., (2017) reported the content of Cu in soil as Cu 14.15 mg/kg in their study which is lower than the values from this study. Copper is essential for collagen synthesis in animals however at higher concentration it becomes toxic.

Iron had the second highest concentration in this study after Cu. The highest content was 105.67 which was at sampling point 6, while the least (22.03 mg/kg) was at sampling point 9. This may be due to the distance of this area from the mining site. The levels of heavy metals in the agricultural soils depend on the distance from the source of pollution (Escarre, 2011). The value obtained is higher 0.58 to 0.78 mg/kg reported by Kabir *et al*., 2017 in a related study. It is however lower than 235.53 and 341.90 mg/kg reported by Afzal *et al*., 2013 in soil.

The mean concentration of Co was 45.77 mg/kg. The concentration of Co ranged from 9.99 to 55.04 mg/kg. Co is relatively toxic to plants when given in high doses. Under reductive conditions, however, cobalt is also more readily soluble, as well as becoming more available for plants. The comparably lower value of Co obtained in the control sample indicates contamination from mining activities in the study area. The content of Pb in this study is from 3.50 to 13.99mg/kg. The mean content obtained is comparable to the range of 2.08 – 16.57 mg/kg reported by Afzal *et al*., 2013 in a soil. It is however lower than the permissible limit of WHO, 2008. This shows the area is safe in terms of Pb contamination. It is also lower than 65.80 – 291 mg/kg reported by Kabir *et al*., 2017. It is lower than 193.133 mg/kg reported by Yu *et al*., 2017 in mining areas in Central China.

Pollution Index (PI)

Pollution index of heavy metals in the sampling areas was computed in order to account for the extent of contamination. The pollution index was determined in relation to the background metals content of the

Table 4: Pollution Index of Metals in Soil

Metal	Background Value (mg/kg)	Metal concentration			Pollution Index (PI)		
		min	max	mean	min	max	mean
Cu	26.70[b]	23.03	170.83	89.84	0.86	6.39	3.36
Pb	19.40[b]	3.50	15.43	10.59	0.18	0.79	0.55
Cd	0.12[b]	0.70	3.11	1.93	5.83	25.91	16.08
Cr	49.30[b]	0.75	8.74	3.48	0.015	0.18	0.07
Ni	26.60[b]	5.00	34.52	20.68	0.19	1.29	0.77
Zn	50.00[b]	3.29	25.46	18.28	0.07	0.51	0.37
Mn	459[b]	3.49	22.50	14.92	0.01	0.05	0.03
Fe	14.31[c]	22.03	105.67	71.36	1.54	7.38	4.98
Co	13.14[c]	9.99	64.43	45.77	0.76	4.90	3.48
As	14[b]	0.35	1.20	0.84	0.03	0.09	0.06

N.B: b Soil background value (Pan *et al*., 1998). c Agenor *et al*., 2016.

samples (Sitil *et al*., 2014). PI was computed and shown in Table 4 for each metal using the formula:

$$PI = C_n / B_n \quad (3)$$

Where C_n and B_n represent the determined concentration of the metals and the background concentration respectively. The *PI* is classified as follows:

- Pi ≤ 1: low contamination
- 1 < PI < 3: moderate contamination
- PI > 3: high contamination

The PI for Cr shows low contamination (0.015 to 0.18) with a mean value of 0.07. The mean value for Ni also revealed a low contamination. However one of the sampling areas was moderately contaminated. The values show that for copper, the extent of pollution varied from low contamination with PI value of 0.86 to high contamination with a value of 6.39 and mean value of 3.36. The values obtained for Cd ranged from 5.83 to 25.91, showing a very high degree of contamination by Cd in this area. The mean PI values for Mn, Fe, Co and As were 0.03, 4.98, 3.48 and 0.06 respectively. There is high contamination of soil with Co, and a low contamination by Mn and As based on pollution index assessment.

Cadmium shows a strong positive correlation with Mn and Fe. This shows a close association between these elements in soil. Cr did not give any significant association with the other metals investigated. Ni showed a strong positive correlation with Co. The high correlation coefficient obtained for Zn with Pb and Cu shows that they are closely associated in terms of their origin. With the exception of Cd and Cr, Pb associated strongly with the other elements in this study. There was a strong positive association of Cu with Co and As. Asernic also showed a closed association with Pb, Co and Cu as indicated in their values of correlation coefficient. Generally, the positive values for correlation coefficient between metal pairs show a common source of pollution.

Table 5: Inter-elemental Correlation Coefficient

	Cd	Cr	Ni	Zn	Pb	Cu	Mn	Fe	Co	As
Cd	1	0.333	0.448	0.705[1]	0.646[1]	0.609	0.830[2]	0.805[2]	0.734[1]	0.724[1]
Cr		1	0.291	0.275	0.097	0.059	0.222	0.110	0.186	0.099
Ni			1	0.676[1]	0.726[1]	0.456	0.642[1]	0.694[1]	0.766[2]	0.634[1]
Zn				1	0.909[2]	0.675[1]	0.831[2]	0.733[1]	0.866[2]	0.807[1]
Pb					1	0.765[2]	0.798[2]	0.754[1]	0.876[2]	0.825[2]
Cu						1	0.584	0.636[1]	0.884[2]	0.804[2]
Mn							1	0.957[2]	0.819[2]	0.696[1]
Fe								1	0.841[2]	0.702[1]
Co									1	0.867[2]
As										1

[1]Correlation is significant at the 0.05 level (two-tailed)
[2]Correlation is significant at the 0.01 level (one-tailed)

4. CONCLUSION

In this work, we examined the physicochemical parameters and heavy metal contents of soil of Okpella Mining Area in Edo State. Inter-elemental correlation showed that most of the metals investigated were of common origin. The concentrations of the metals at mining area were generally higher than those of the control samples. With the exception of Cd, the concentrations of other metals were within safe limit based on WHO standard. Results from pollution index assessment also show that Cd has higher degree of contamination.

5. ACKNOWLEDGMENTS

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6. AUTHORS' CONTRIBUTIONS

All the authors contributed to this work. Edema, O. G. and Obigwa, P. A. collected the samples. All the authors participated in the laboratory work. Inobeme, A. Olori, E. and Adekoya, M.A. analyzed the data.

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