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Okanlawon, Olasehinde and Abdullahi (2018) Nigerian Mining Journal, (16) 1 - 11

Univariate and multivariate statistical analysis of heavy metal pollution in waters around Shikira, Kawo and Maigiro areas, Tegina sheet 142 SE, North central Nigeria

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Abstract

Hydrogeochemical studies were carried out in the parts of Tegina sheet 142 South East. This was as a result of reported lead poisoning that devastated Shikira, Kawo and Maigiro village in Rafi Local Government area of Niger State. The lead poisoning in these areas was due to illegal gold mining activities. Twenty eight water samples were analysed using Inductively Coupled Plasma- Mass Spectrometry (ICP-MS) for As, Pb and Sb. Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) was used for Fe and Mn while Fluorescence Spectrometry was used for Hg. Analysed results was subjected to univariate and multivariate (Correlation and Principal Component) analysis using Surfer 10 and SPSS package respectively. Correlation matrix analysis revealed a strong correlation between Hg-Pb and TDS-EC. Three components were extracted explaining 63.4% of overall total variance. The first PC explains 33.8% of total variance with a loading for Pb, Mn, Hg and pH. The second extracted component has 15.5% of the total variance with a loading for TDS while the third component explains 14.04% of the total variance with a loading for Fe and pH. Univariate analysis show a similar trend as Hg and Pb, TDS and EC has the same signature. The result of the study revealed that waters around the mining areas contain a significant concentration of heavy metals which exceeded WHO standard for drinking water quality. The miners and populace need to be educated of the implications of heavy metal pollution and best practice of mining the metals without polluting groundwater.

Keywords: Water, Heavy metals, multivariate, univariate and correlation analysis

1. Introduction

Mining has played an important part in the history of mankind from prehistoric to the present. It encompasses the extraction of naturally occurring mineral substances from the earth for utilitarian purposes. Mining activity and processing of metal ores are in one hand of great benefit to the society and on the other hand bring seriously contamination to the environment especially the host community and its environs if remediation and mitigations measures are not put in place. The mining area is related to disturbance of surrounding landscape, spilled mine tailing, emitted dust and acid mine drainage, exposing surrounding environment to toxic heavy metals (Johnson & Hallberg, 2005). It is well known that heavy metal pollution as a result of mining activities is a global phenomenon. The heavy metal pollution has a characteristics of high toxicity, and difficult to degrade and its migration bring about broader range of hazards (Liang *et al.,* 2011). Mine tailings and mining wastes are the major source of heavy metals such as mercury, lead, arsenic, manganese, copper, cadmium, zinc, iron in water and soil in areas of mining activities. Once water is polluted, its quality cannot be restored by stopping the pollutants from the source (Amadi *et al.*, 2012). Behavior of metals in the natural water is a function of the substance sediment composition, the suspended sediment composition, and the water chemistry (Mohiuddin *et al.,* 2012). Physicochemical characteristics and prevailing weather conditions are responsible for migration of these metals into environment (soils, streams, food crops, surface and groundwater) which are subsequently ingest and threatening human health (Okanlawon, 2018).

In Jos Plateau, Ogezi (2005) reported how the activities of artisanal miners degraded water quality through mining waste release into the water bodies which resulted into increased concentration of different elements above recommended standard coupled with changes in water colour. Also, in Nasarawa state unusually high lead concentration was found in the blood of general population living near lead-zinc mines in Adudu community (Olanrewaju *et al.*, 2016). According to him, 31% of children less than 7 years of age were found to have blood lead level exceeding 2µg/l and 11% exceeded 5µg/l while 68% of the adults have blood lead level exceeding 2µg/l and 14% exceeded 5µg/l respectively.

In Nigeria generally, it has been reported that mining at commercial or artisanal scale have resulted in pollution of air, water and soil and health risk for communities where such activities takes place (Ako *et al.,* 2014). The composition and nature of ore bodies always determine the nature of pollutants resulting from their processing. Activities of artisanal miners overtime have been described as lack of or very reduced degree of mechanization; low level of occupational safety and health care; inefficiency in exploitation and processing of mineral produce; low level of salaries and income; lack of social security and insufficient consideration of environmental issues hence significant impact on environment (Noetstaller, 1995; Charkravorty, 2001; Gunson and Yue, 2001; Jennings 2001; MMSD 2001). So, little do they know about the side effects of their activities on the environment and human health until recent outbreak of epidemics that claims lives of innocent children in part of Kawo and Maigiro area of Rafi Local Government area of Niger state (Olokor, 2015). The impact left by environmental pollution of mining activity may take a very long period to resolve. It is, therefore meaningful to study the concentration and associations of heavy metals in water around the mining areas to ascertain the level of contamination in the study area.

2.0 Study area

2.1. Location and accessibility

This study was carried out in parts of Rafi local government area, located in Niger State, North central Nigeria. The area was delineated using topographical map of Tegina sheet 142 bounded by 10°00'N to 10°15'N latitude and 6°15'E to 6°33'E longitude (Figure 1). The study area is prominent for Manganese and Gold mining. It is accessible through Kagara and Kukoki, Kagara is accessible through Minna-Birni Gwari road and Lagos- Kaduna highway. Footpaths leading to the various villages and harmlets ease access to different parts of the study area.

2.2 Climate and vegetation

The climate of the area lies within the tropical climate and its associated with high temperature (31.2°C-36.9°C) and marked with cloudless sky for most of the year with wet and dry season (NIMET, 2003). The dry season last for about six to seven months, from November to April with very low humidity accompanied by the North-East trade wind which is usually the harmattan periods. Relative humidity is low in the early March at around 40% and increase to over 94% around September. Wet season last for about five to six months usually begins from April to November (Elias, 2015). Highest mean monthly rainfall is September with almost 300mm. The vegetation of the area falls within the Guinea Savannah belt which comprises of different species of shrubs and also forest like along the stream channels (Kogbe, 1989; Ajibade, 1982). Generally, the climate, soil and hydrology of the area permits the cultivation of most of Nigeria's staple crops (yam, rice, guinea corn, millet,

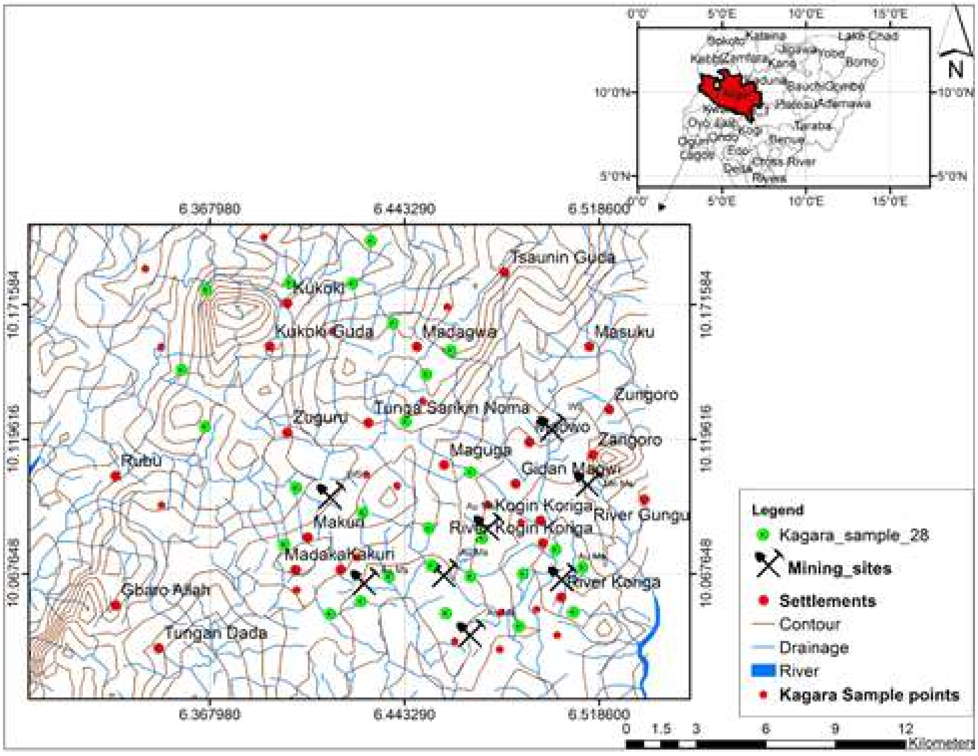


Figure. 1. Topographical map of parts of Rafi LGA, Niger State

cowpea and groundnut) and still allow sufficient opportunity for grazing, fresh water, fishing and other forestry development (Ayinde *et al.,* 2013)

2.3 Topography and drainage

The topography of the study area is gentle with major hill at Madaka which is granitic in nature while some portions of the area are rugged. Rivers in the study area are elevation and fractured controlled as they flow southward and mainly drained by river Kunukunu, Koro, Guleka, Ankawa, Gungu and Bikoro. Most of these rivers are seasonal; they flow during wet season and dry up during dry season.

2.4 Geology of the study area

The study area is part of Precambrian Basement Complex of Nigeria which comprises of migmatite-gneiss-quartzite complex, schist belts and older granite suites. The lithostratigraphic units recognized in the area are amphibolite, granite, gneiss and schist with quartz intrusions (Figure 2). Granite and granite-gneiss occupy the southwestern part of the area and vary in composition and texture. Granite in the eastern part was observed to have large grains with quartz intrusion. The northern and the eastern part are predominantly occupied by schist and quartzite with Feldsphatic rich granite. Dolerite dyke exposed along the river channel in the southern part with amphibolite schist.

3.0 Materials and Methods

3.1. Water sampling

A total of twenty-eight water samples were collected from hand dug wells, rivers, boreholes, mining site and washing site. All the samples were obtained using cleaned plastic bottles which were washed and rinsed with distilled water. At the site, the plastic containers were prewashed before final collection and then preserved with concentrated nitric acid in order to minimize precipitation and absorption of ions on the walls of the container. Elevation and Sampling locations were determined and

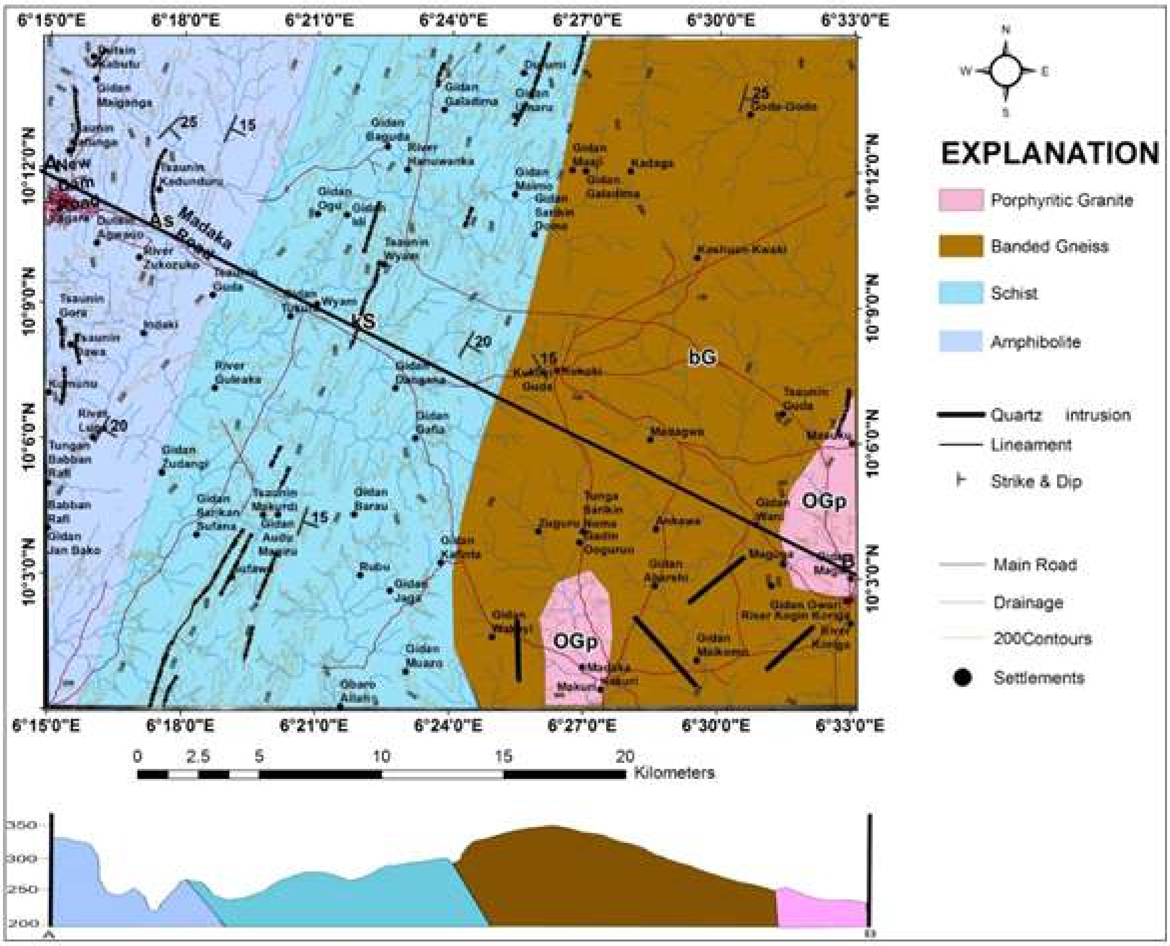


Figure. 2. Geological map of tparts of Rafi LGA, Niger State (Okanlawon, 2018)

recorded using a handheld Garmin e-trex vista Geographic Positioning System (GPS). Field parameter such as pH was measured with digital pH meter (pen type) model 8685 while Electrical Conductivity and Total Dissolved Solids and Temperature were determined using water quality meter (pen type) model 8361.

3.2. Sample analysis method

Analyses of heavy metals were determined using Inductively Coupled Plasma Mass Spectrometry (Agilent 7700X). The samples were prepared following Czech and USEPA standard (CZ-SOP-DO6-02-02, US EPA 200.8, CSN EN ISO 17294-2, US EPA 6020A) for arsenic (AS), Chromium (Cr), Lead (Pb) and Antimony (Sb). The content of metals were measured, after it was acidified by suprapure nitric acid. Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) (Agilent 725 radial type) was also used for determination of Iron (Fe) and Manganese (Mn) following Czech and US EPA standard (CZ-SOP-DO6-02-001,US EPA 200.7, ISO 11885, CSN EN 16192, US EPA 6010, SM 3120) while mercury (Hg) was determined by Fluorescence Spectrometry with Czeh and US EPA standard (US EPA 245.7, US EPA 1631, CSN EN ISO 17852, CSN EN 16192, CZ-SOP-DO6-02-096). The recoveries for these metals in the standard reference materials were found around 90.8%-103%. For quality control method blank were used, replicate samples were analyzed throughout the analysis. Invariably, the results indicate that there was no contamination during the analysis and measurement of uncertainty (expanded uncertainty) with coverage factor k=2, represent 95% confidence level.

4. 0 Results

Analytical results of physico-chemical data obtained from the study area are provided in Table 1 with their respective descriptive statistics (minimum, maximum, mean, variance, Standard deviation). These data were used to compute multivariate and individual chemical signatures of the area under study.

Table 1: statistical overview of water chemistry from Kawo and Maigiro area

s.u= Standard Unit; mg/l = Milligrams per liter; µs = Micro Second; RV = River; BH= Borehole; BDL= Below Detection Limit

4.1 Multivariate statistical analysis

Multivariate statistical analyses were generated from the analytical results in Table1. This analytical method is widely applied in earth and environmental sciences (Schuenemeyer & Drew, 2011). It assist in redefining description of signature by characterizing the extent at which chemical properties of two areas differ or similar to each other. It also involves simultaneous analysis of multiple variables instead of examining variables one after the other. Multivariate statistical analysis is good for identification of commonalities and differences between data set. Multivariate statistical analysis used for this study is Principal Component Analysis (PCA)

4.2 Principal components analysis

The hydrogeochemical data were subjected to Principal Component Analysis (PCA) and iteration of Principal Components yields a number of outputs that were interpreted. One of the yielded outputs is correlation matrix in Table 2. PCA as a mathematical procedure is used to transform correlated variable into a new uncorrelated set of variables in Table 3 with corresponding scree plots in Figure 2.

Table 2: Correlation Matrix

Table 3: Rotated Component Matrix[a]

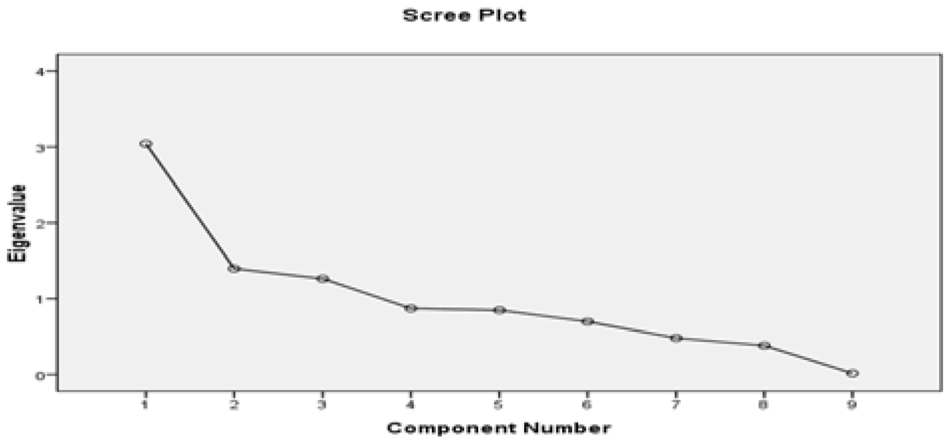
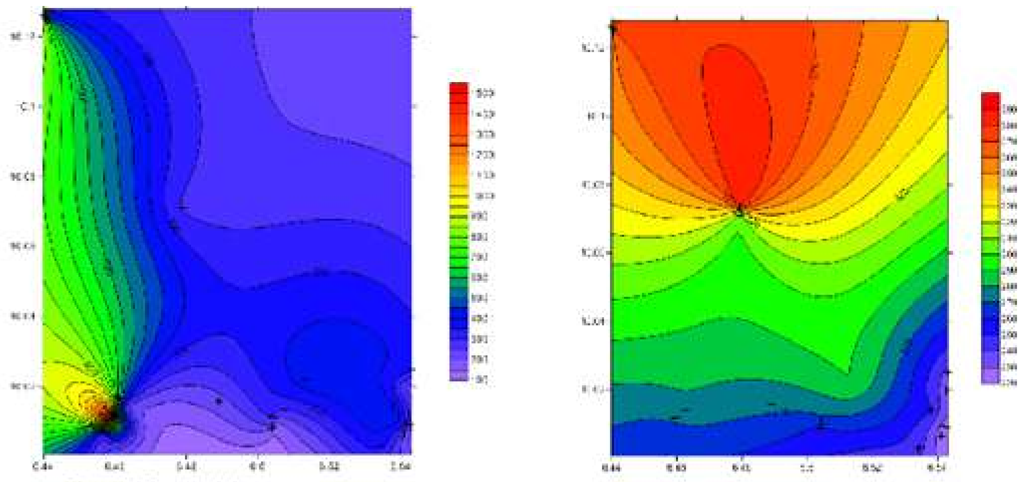


Figure. 3. Component Scree Plot

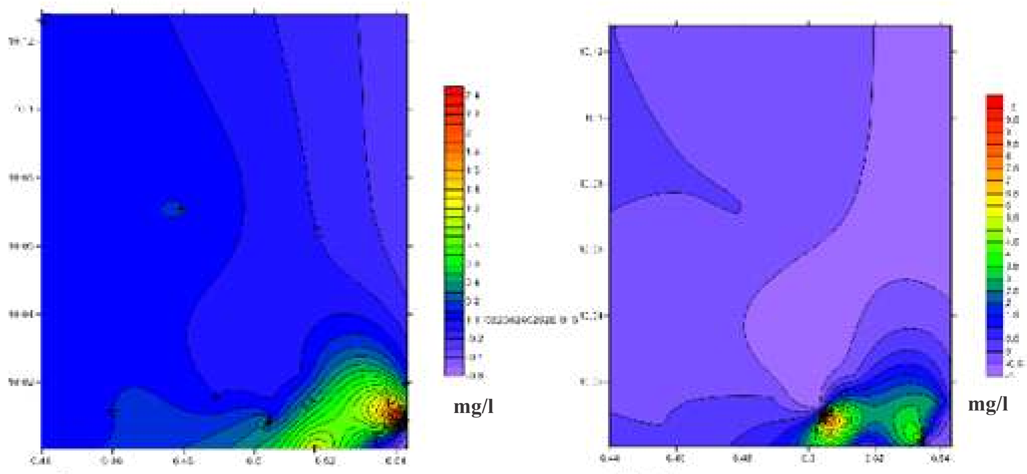
4.3 Univariate statistical analysis

Univariate statistical analysis was used to examine the distributions of measured parameters within the study area. To achieve this, univariate statistical plots were generated using surfer 10. The concentrations of each parameter are shown in Figures 4a-i in order to know the data spread within the study area.



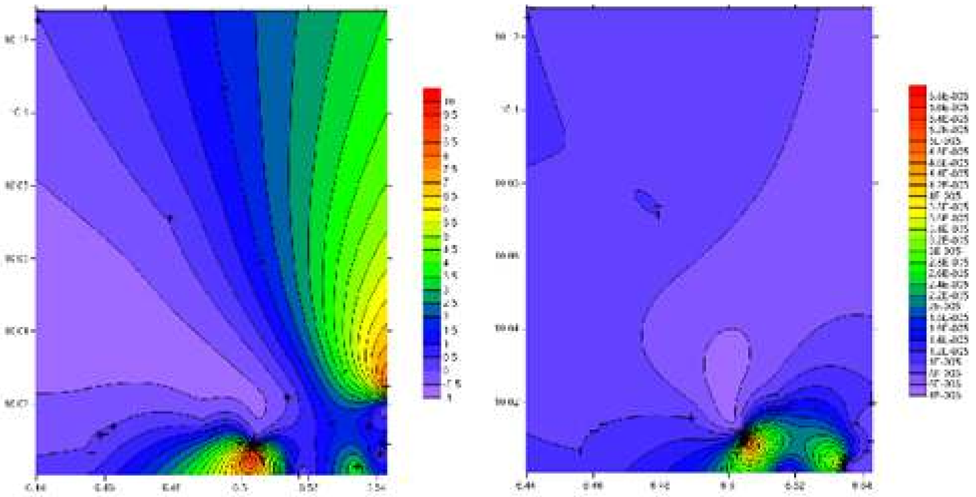
a. Electrical Conductivity

b. Elevation



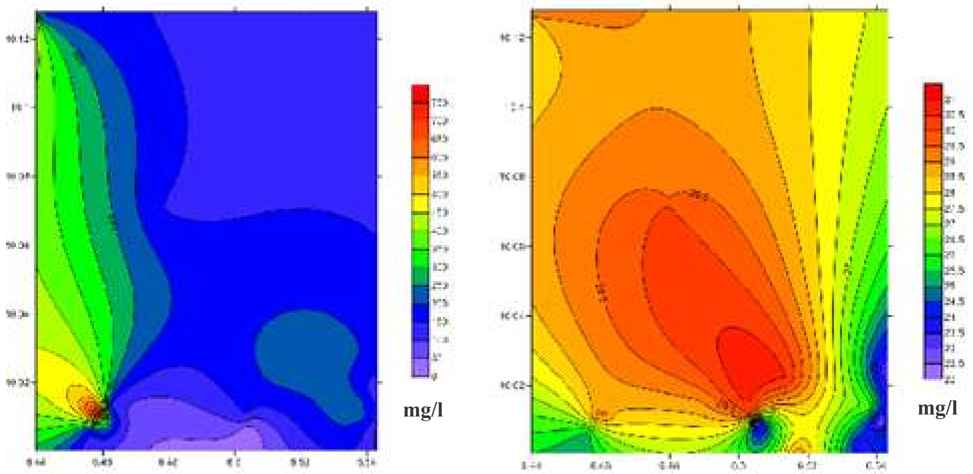
c. Iron

d. Lead



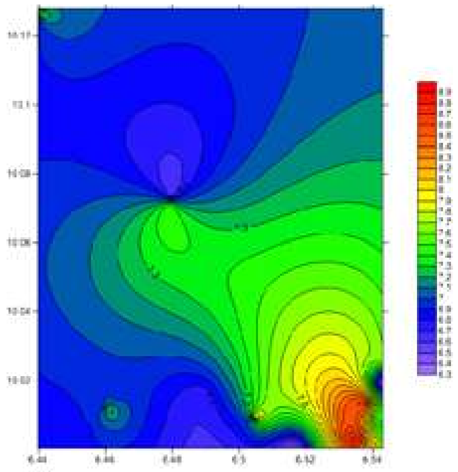
e. Manganese

f. Mercury



g. TDS

h. Temperature



i. pH

Figures 4a-i. Univariate analysis of parameters

5. Discussion

5.1 Correlation matrix

Correlation coefficients of heavy metals in waters from the studied area are listed in Table 2. It was observed from the result that EC-TDS and Pb-Hg are strongly correlated while Hg-Temp, Mn-Temp, pH-EC and pH-TDS are weakly inversely correlated. The remaining elements are less correlative. Presence of Pb and Hg in water is as a result of geogenic and anthropogenic processes respectively. The washing sites are places of anthropogenic processes. EC and TDS are function of dissolved ions in water and dissolved organic matters; it may be through natural or anthropogenic processes.

5.2 Principal components analysis

In order to compliment the correlation analysis by the Spearman correlation matrix, the data set was further subjected to Principal Component Principal Analysis (Table 3). This method extracts the eigenvalue and eigenvectors from the covariance matrix of original variables, thus reducing the dimensionality of data set (Amadi *et al.,* 2012). Varimatrix orthogonal rotation with Kaiser Normalization was applied to the Principal Component Analysis in order to minimize the effect of non-normal data (Gong *et al.,* 2010). The Principal Components with eigenvalues greater than 1 were retained in order to get a clear picture of the PCA structure. Kaiser – Meyer Olking measure of sampling adequacy was applied to know the adequacy of the samples and its meets suggested minimum value of 0.6. Three principal components were extracted explaining 63.4% of the overall total variance. Component 1 is associated with high loadings of Pb, Hg and pH, explain 33.8% of the total variance. The presence of Pb and Hg in water is associated with the Pb content of the ore and processes involved in gold winning that is mercury amalgamation. The dominant rock unit in parts of the studied area is granite and the major cations and anions in water are likely to be from the lithology via weathering/dissolution processes and it is a function of pH (Amadi *et al.*, 2012). Component 2 explain 15.5% of the total variance with a loading of Temperature. The third component accounts for 14.04% of the total variance with loadings of EC and TDS. The presence of EC and TDS is as a result of inorganic salts and small amount of organic matters that are dissolved in water. The graphical representation of the extracted components is shown in Figure 3.

5.3 Univariate statistical analysis

Univariate statistical analysis (Figure 4) shows the trend and concentration of both physical and chemical parameters in the study area. Hg and Pb concentration were found to be higher with similar signature around southern end of the study area. TDS and EC also have higher concentration at southwestern part around Madaka. High concentrations of Fe and Mn with low temperature were observed around Kawo and Maigiro while low pH is only observed in Kawo.

5.4 Metal concentration in water

It was observed from Table 1 that the concentration of heavy metals followed a decreasing order of Mn>Pb>Fe>As>Hg and physical parameters in order of EC>TDS>Temp>pH. Concentration of As and Hg are well below WHO (2011) standard for drinking water quality in all sampled locations. Higher concentration of Mn are far above WHO (2011) recommended limit of 0.4 mg/l in Mag 1, Mag 2, Mag 3, Mag 4, Mag 7, Mag 7B, Kaw 1, Kaw 2, Kaw 3 and Min ST. Health effects of Mn above the said limit leads to neurological disorder, manganism, “Parkinson-like syndrome”, and damage central nervous system (Bhattacharya *et al.,* 2008; WHO, 2011; Bada *et al.,* 2012). The observed Pb values in Mag 7, Mag 7B, Kaw 2, Kaw 3 and Min ST is far higher than the recommended WHO (2011) value of 0.01 mg/l. Its health effects include cancer, interference with vitamin D metabolism, poor mental development in infants, hearing loss, anemia, miscarriage, still birth, irritability, infertility, kidney and brain damage, disorderliness in the human nervous system and impair intellectual capability in children (WHO, 1995; Canfield *et al.,* 2003; Piantone, 2009; NIOSH, 2018). High concentration of Fe above WHO (2011) recommended limit of 0.3mg/l was observed in Mag 3, Mag 5, Mag 8, Kaw 1 and Min ST. Excessive iron in the body causes genetic disorder (hemochromatosis), skin cell damage, gene mutation and neurodegenerative diseases (Bothwell et al., 1979; WHO, 2003; Abbaspour *et al.,* 2014)

6. Conclusion

The result of this work revealed that waters downstream the mining areas contain a significant concentration of heavy metals as indicated by correlation analysis, Principal Components Analysis and Univariate Analysis. The dominant heavy metals were found to occur in association with gold and some of these heavy metals exceeded WHO (2011) recommended value for drinking water quality; therefore, Fe, Pb and Mn poses a serious health risk in the area. Continuous mining activities will further increase concentration of tested heavy metals which will in turn aggravate health condition of people living within the vicinity of study area. There is urgent need to educate inhabitants of the area about the danger associated with illegal mining and to educate them on proper handling of mining and gold wining process. There is need for further study in other to appraise heavy metal concentration in soils and rocks.

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Failure Analysis of Conveyor Belt System in Gbose Quarry Tool for Effective Monitoring and Maintenance

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Abstract

Frequent failures at the conveyor system of a processing plant reduces production rate, increases production cost and in the long run may lead to the abandonment of the plant. This paper analyses the various failuresat the conveyor system of Gbose Quarries Limited over certain period of time. The paper focuses on estimation of the scale of a problem such as: the most frequent failures, types of failures, the location of failures and their importance in the context of maintenance of a conveyor belt transportation system. The failure report data collected over a period of time was analyzed using graphs, bar charts and pie charts. The analysis showed that the conveyor belt system alone accounted for 51% of the total failure in the quarry; with 29.9% failure of gearbox and chain, 25.4% pulley failure, 20.9% coupling failure and 23.8% electric motor failure respectively. Also, the study shows that most secondary failures are due to unnoticed primary failures. After adopting condition monitoring in the quarry, the failure frequency of the conveyor belt system reduced. The study therefore suggested further effective monitoring of the frequently failing components. Also, the study recommends effective maintenance (both preventive and corrective) for the quarry.

Keywords: Failure Analysis, Conveyor System, Quarry, Monitoring and Maintenance.

1.0 INTRODUCTION

There are a lot of different mechanical systems in a quarry but one of the most important is a transportation system. One may consider two types of transportation systems: Type 1 that make use of dump trucks to convey either overburden or materials from place to place, and Type 2 that makes use of the conveyor belt. The conveyor belt transport system can further be classified into: (a) the conveyor system transporting materials directly from the pit to a processing plant site(mostly used in a coal mine) and (b) the conveyorsystem transporting materials within a crushing plant circuit. Belt Conveyors are available in a complete range of types and sizes to suit every material handling requirement. Standard and specially engineered units and various combinations of both, permit Engineers to select the system best suited to a particular job from the industries broad selection of conveyor components and accessories. It is often advisable to purchase both materials handling and processing equipment from the same manufacturer, the buyer is assured of greatest efficiency and economy from single-source responsibility for design, manufacture, erection and proper operation of the entire plant. The resulting balanced design eliminates bottlenecks and assures peak capacity and efficiency from every unit in the system.

1.1 CONVEYOR BELT SYSTEM

The belt conveyor system (BCS) consists mainly of:

a. drive unit (electric motor, coupling multistage gearbox), pulley (drive pulley and others);
b. belts (textile with steel cords or with their joints); and
c. idlers (belt cleaning systems, control system, etc.)

These are shown in figure 1. Belt conveyor may be viewed as a part of bucket wheel excavator or dumping machine. In such case CBS is rather short in length (<100 m).

It should be noted, that, although relatively simple design BCS are very important part of mechanical systems and failures of them are quite costly (Batelmus, 1998). Moreover because of their dispersion and number of moving/rotating part it is difficult to maintain it. Figure 2 shows view of Gbose quarries conveyor belt system.

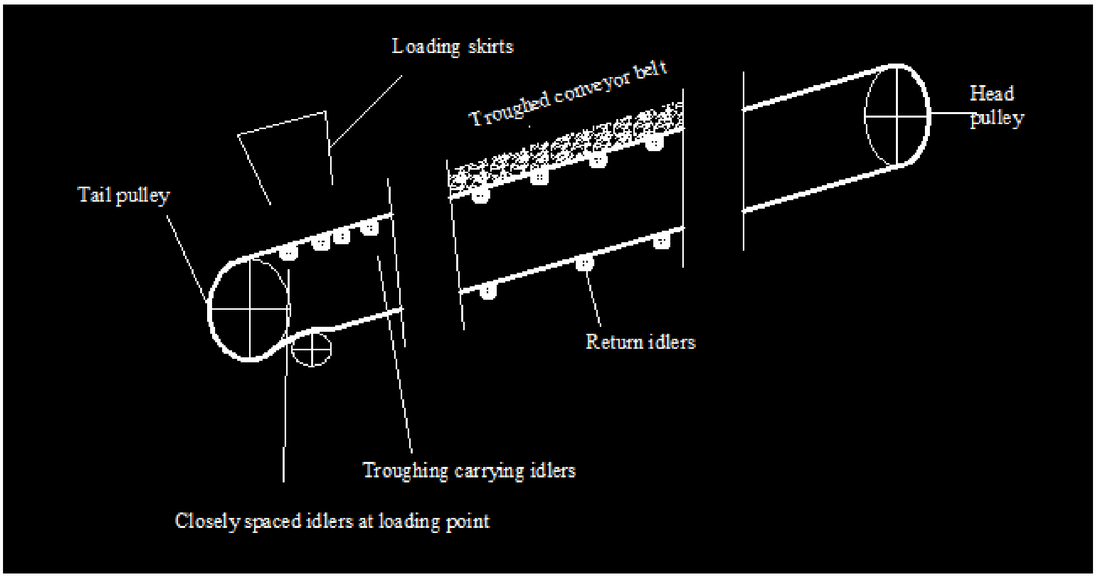

Figure 1. Scheme of belt conveyor (Bartelmus, 1998)

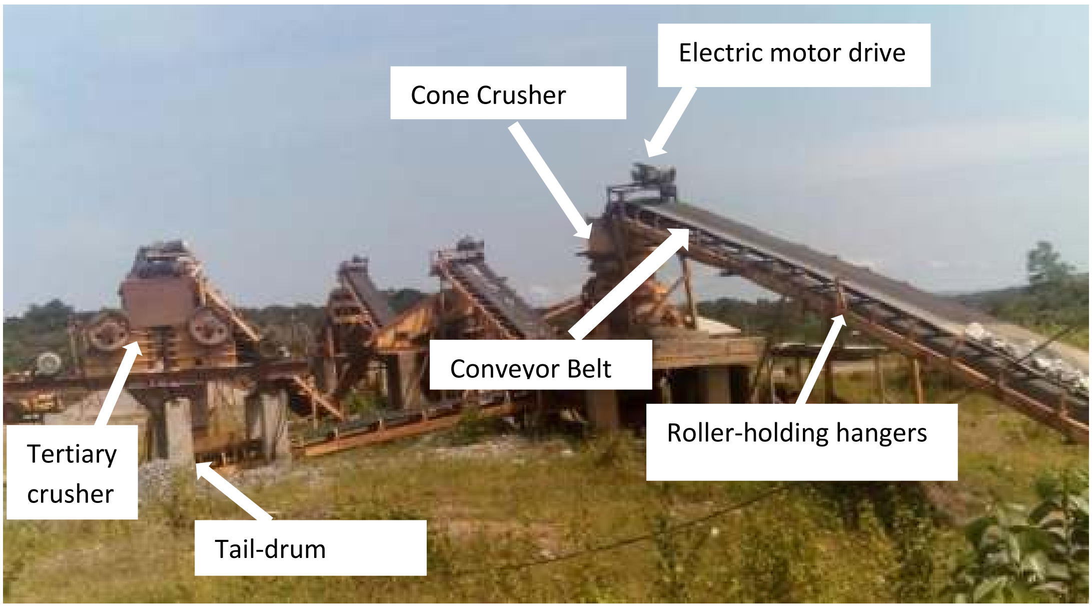

Figure 2: A view of Gbose quarries conveyor belt system

1.2 TYPE OF FAILURES IN CONVEYOR BELT SYSTEM

This section considers the type of faults that had occurred and may still appear in belt conveyor systems with reference to conveyor components. Focus will mainly be on the drive units, pulleys, idlers and belts as the most significant components. It should be noted that, while some failures take a short time to correct, others may cause a long production downtime.

1.2.1 Failures in the Drive Unit

The drive unit consist of electric motor, damping coupling, two or three stage gearbox and coupling that connect output shaft with pulley (as shown in figure 3). A crucial object in this subsystem is gearbox. It has been observed that the gearbox may fail due to unchecked primary failures (corrosion, excessive loading, etc.). The records at Gbose quarry in 2017 showed that 10% of gearboxes may be replaced each year due to unexpected failures; these failures are related to the geared wheel wear or damages (broken tooth) and bearings (mainly over limit backlash due to environmental impact, also typical failures like outer/inner race, rolling element).

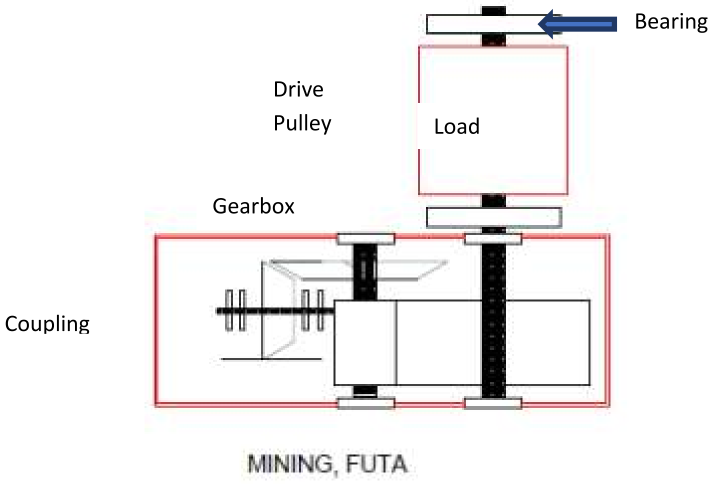

Figure 3: Scheme of drive unit used in CBS (Radoslaw, 2009)

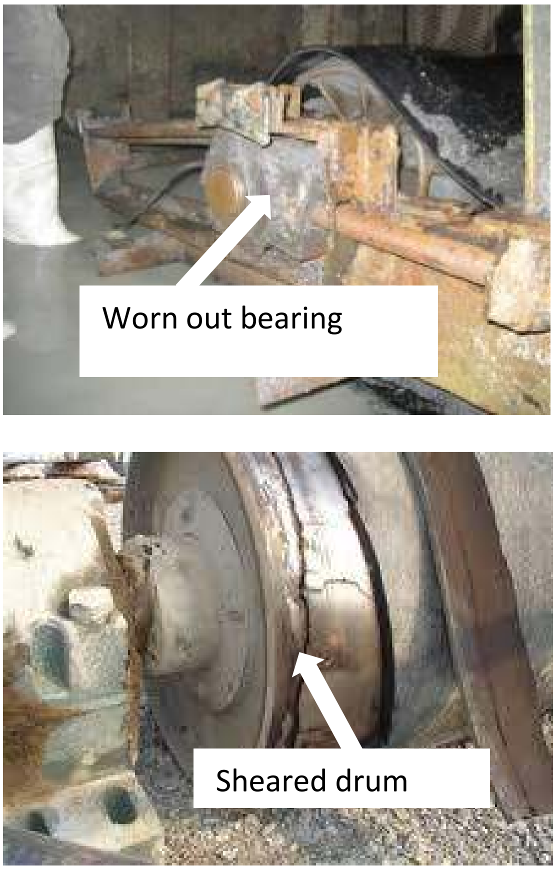

Figure 4: Damaged bearing and pulleys due to torsional stress

1.2.2 Failure of Pulleys

Mining pulley basically consist of two bearings, shaft, and drum. Two drums (head drum and tail drum) are used for the system. Without proper condition monitoring, pulleys used in aggregate quarries are prone to frequent failures. This is because stones trapped in the pulley component can increase friction and the excess friction can later cause wear, tear, and shear.The number of pulley failures may reach over 12% of used pulleys yearly(Matuszewski, 2007). One may notice some sources of primary problems, namely: bearings misalignment, roll back of stones, corrosion and components quality. Figure 4 shows pictures of damaged bearing and pulley

1.2.3 The Electric Motor

The electric motor drives the gearbox, and provides the needed rotational motion for the entire conveyor system. However, failure of the electric motor may lead to lengthy downtime since the coil of the motor may need rewinding. Most of the failures in electric motor are culminating results of:

a. Poor Earthen of electric cables on the mine site
b. Short circuiting
c. Water access into electric-motor coil which can be through rain or other means
d. Overloading
e. Excessive current from source, etc.

1.2.4 Idlers and Belts

The failure analysis of idlers and belts is a bit different issue. Idlers are used for supporting belts with transported materials. In some sense, idlers are similar to pulleys and consist of bearings and shells. One may expect similar types of failures. The support system for belt consists of three idlers. Usually because of different load for each idler, side idlers are more subjected to damage. Worn bearings in idlers will significantly increase external load for drive units andthereby increases the power consumption. Damaged idlers and pulleys may be the reason of damage for belts.Expected problems for belts are related to belt tear, puncture, cut of belt and abrasion of bottom/top covers and its joints (connected using glue, vulcanized or mechanical joint). Figures 5a to 5d showdamaged belts and idler.

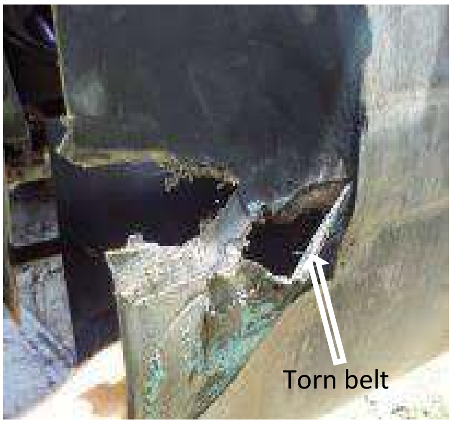

Figure 5a: Tear in a belt due to damaged idler.Figure 5b: Worn out idler due to rusting and friction.

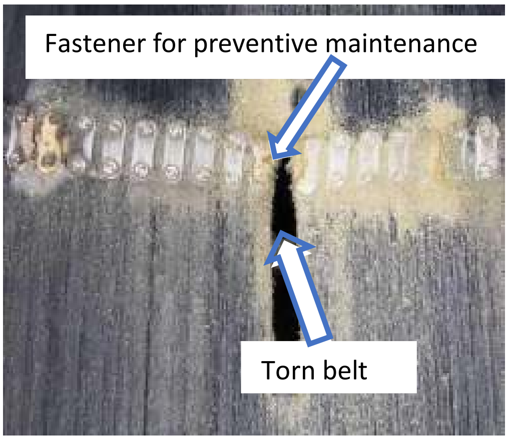

Figure 5c: Aged belt due to abrasive action.Figure 5d: Belt failing across the repaired regions.

1.3 MAINTENANCE

Maintenance involves functional checks, service, repairing or replacing of necessary devices, equipment, and machinery. In the material handling industry, there are basically four threetypes of maintenance carried out in the quarry, this includes:

a. Routine maintenance : this describes simple, small scale activities (usually requiring only mininmal skill or training) associated with regular (daily, weekly, monthly, e.t.c) and general upkeep of an equipment, machine, plants or system against normal wear and tear. Example of routine maintenance in the conveyor belt system includes daily clearance of the spilled aggregate between the tail drum and the floor.

b. Preventive maintenance: this is done to maintain a level of certain service on equipment. It ensures that the equipment is inspected even if it has not given any symptoms of having problem. Example includes fastening a slack bolt and nut, lubrication of bearings, e.t.c

c. Corrective maintenance; this task is designed to rectify the defects to be found in the different equipment. Examples includes: replacement of a ruptured roller, rewinding of a damaged electric motor, e.t.c

2.0 METHODOLOGY

The method employed for this work involves collecting failure report data for a period of time, the failure report contained description and determination of: failed components, mode of failure, mid-time between failure, mid-time to repair, numbers of failures, and predicted reasons for failure. The failures data over the period of time were quantitatively analyzed and compared using graphs, bar charts, and pie charts;identifying the most frequent failures, and reasons for the failures were discussed. Also, field one-on-one interview was conducted to the plant operators and the operators of the various sections of the conveyor belt system in Gbose quarry.

3.0 RESULTS AND DISCUSSION

This section presents the results in graphs, figures, and were discussed. It can be observed from figure 6 that, the belt conveyor system has the highest number of breakdown time (420 hours) between July and December, 2017. This pointed that the conveyor belt system needs more technical intervention.

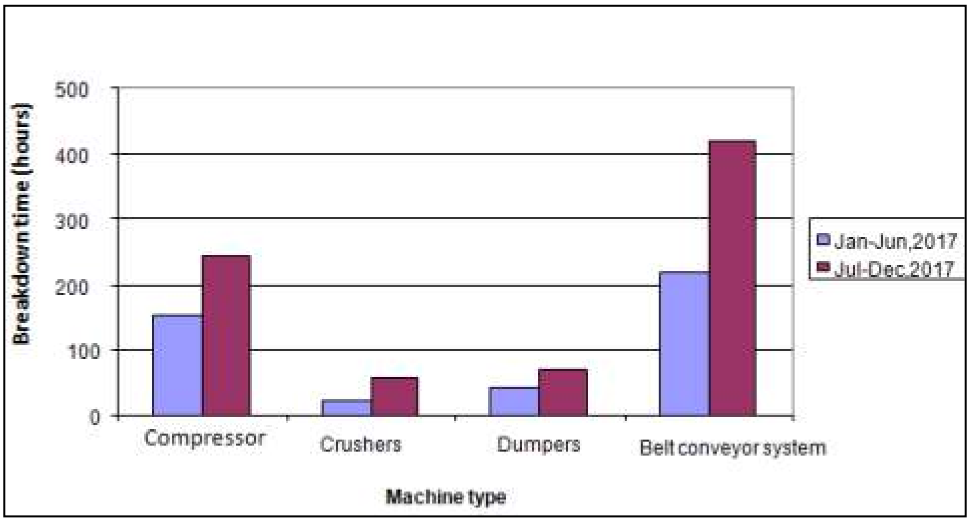

Figure 6: Comparison of breakdown time for different machines in Gbose Quarry.

As shown in figure 7 below, one may easily notice that cracking of the gearbox case has the highest failure. This may be due to the fact that the case is the directly exposed to adverse weather conditions (rain, sun, harmattan, etc.) or the manufactures low quality sales.

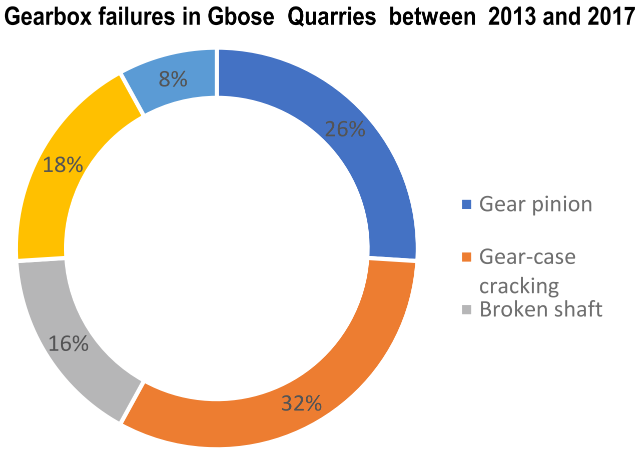

Figure 7: Percentage of failures for gearboxes used in Gbose Quarry

The figure 8 below shows that the bearing failed the most number of times in 2017.Figure 8 below also shows that failure of the bearing account for about 50% (on average) of the total failure of pulley components. However, the bearings need daily inspection and monitoring. During the study, the problem of the bearing failure was traced to poor routine (frequent lubrication) maintenance and was corrected.

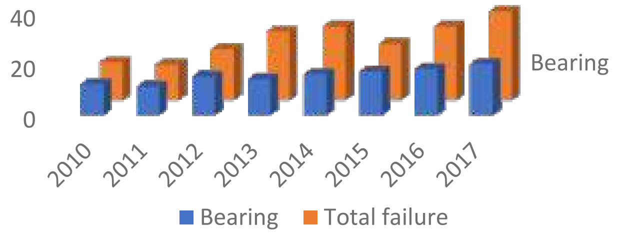

Figure 8: Failure analysis for pulleys (number of total failures and with respect to bearings)

From figure 9 below, the percentage of unnoticed primary failures is higher than the once noticed. This is a result of poor condition monitoring. This suggests that good condition monitoring will help to notice most primary failures before they culminate into greater problems. During the study, the quarry had to improve on preventive maintenance before the problem could be alleviated.

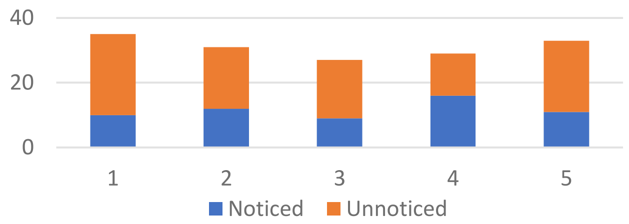

Figure 9: Response of workers in selected aggregate quarries regarding noticed and unnoticed primary failure.

The figure 10 below shows the number of failures related to elements of a drive unit. As one may see, the number of failures is similar for each element. However, the number of failures of gearboxes and chain has the highest value followed by failure of the pulley and bearing unit. During the study, investigations showed that the rate of of feeding the conveyor belt was too high and it resulted into frequent failure of the gearbox and chain.

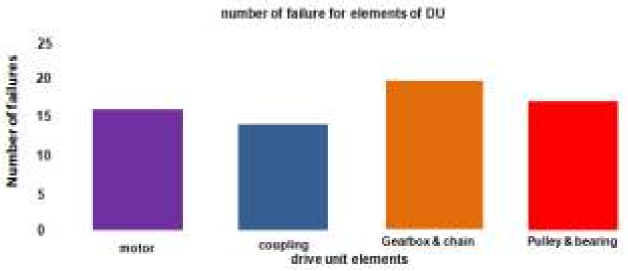

Figure 10: Comparison of the various failures of the drive unit elements.

In Figure 11 below, analysis showed that overloading had caused 45% of electric motor failures. The problem of failure due to overloading or overfeeding of materials into the conveyor belt can be avoided if the operators improved on effective monitoring and attentiveness when feeding the conveyor. The operator should not at any time be carried away or distracted when operating the plant.

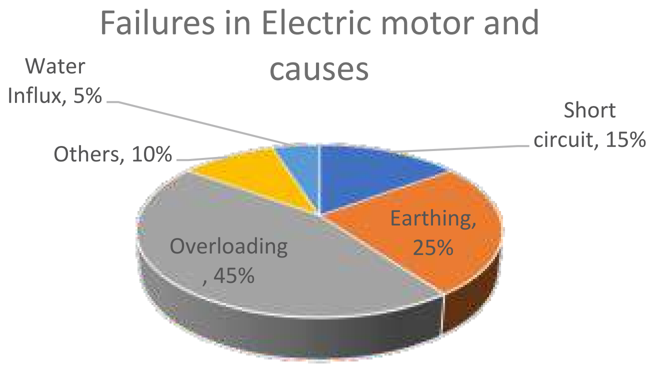

Figure. 11: Various causes of failure of electric motor in Gbose Quarries over certain period of time

From figure 12 below, it can be observed that the higher the failure of the idlers, the more it extrapolates into failure of the conveyor belt. This shows that unchecked failed idler (primary failure) will always shear the conveyor belt, and eventually cause it to fail (secondary failure). It is worthy to be noted that some damaged idlers were observed yet to be replaced, indicating poor corrective maintenance. The operators were then informed and enlightened during the study and the problem abated for the period.

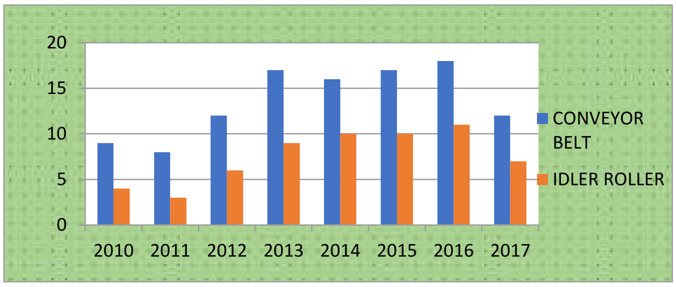

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Figure 12: Comparison of failure frequencies of conveyor belt and idler rollers.

3.1 CONDITION MONITORING OF BELT CONVEYOR SYSTEM

Since it has been shown that different components of conveyors may be damaged in different ways, it is therefore technically advisable to monitor their conditions (before, during and after operations). For gearboxes (geared wheel and bearings) and pulleys, one may use a vibration based condition monitoring, CM (real time or according to schedule) (Bartelmus 1998). It may be also useful to use temperature measurements for bearing condition monitoring. For pulley coating a shell damage detection modal analysis may be used. For idlers condition thermography measurements or noise may be used (Kostanski, 2008). In order to detect cut of covers it was proposed by one of belt produce to place kind of electromagnetic field transmitter. If belt is cut, electric circuit will be damaged and transmitter will stop working (Szczygielska *et al*., 2001). Very often similar approaches like speed measurement (for rotating shaft or moving belt) may provide sufficient information about conveyor condition Monitoring of consumed power/current also may be useful; one of the simplest (technically) is temperature monitoring (Radoslaw, 2009).

Also, physical inspection of the conveyor system is a good condition monitoring approach; damaged idlers can easily be sited and replaced before further complications arise. During condition monitoring of operations, minor worn out parts of belts and other components (primary failures) can easily be corrected.

3.2 EFFECTIVE MAINTENANCE

One of the usefulness of the analysis of the various failures in the conveyor system is to identify components that need more attention in terms of monitoring and maintenance. The component with most number of failures obviously needs effective preventive maintenance i.e periodic check, periodic adjustment to control slack, removal of foreign matter, frequent inspection and against any interference.

Similarly, any observed failure of any conveyor belt system component should be immediate rectified i.e corrective maintenance should be applied before it accumulate to greater problems.

4.0 CONCLUSION

This paper has analyzed the various failures that occurred in the conveyor system of Gbose Quarry. The parts that was observed to have failed often during the study analysis includes the electric motor, the chain drive,the bearings, the conveyor belt, the idler rollers and the roller drums. The maintenance majorly practiced in the study area is the corrective maintenance. It was observed that: damaged conveyor belt was a result of unchecked damaged idlers, failure of bearing was a result of inconsistent lubrication, damaged electric motor was a result of overloading or overfeeding of the conveyor belt. It was also observed that the quality of maintenance spare parts affects frequency of failure.Furthermore, the study identified that some failures are results of uncorrected primary failures.The analysis helped also to identify areas that need effective monitoring and appropriate maintenance; the bearings needed routine maintenance of lubrication, the gearbox and chain drive needed effective preventive maintenance, the conveyor belts also needed effective preventive maintenance. Therefore, condition monitoring and appropriate effective maintenance will help reduce the number of failures in the conveyor system.

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Calcareous Nannofossil, Foraminifera Biostratigraphy and Paleoenvironmental Analysis of D1 Well, Offshore Eastern Niger Delta, Nigeria

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Abstract

Calcareous nannofossil and foraminifera biosratigraphic analysis was carried out on one hundred ditch cutting samples from the D1 well, offshore Niger Delta Basin. The studied interval ranges from 3440 - 9950 feet. The samples were studied for their lithology, calcareous nannofossil and foraminiferal contents with the aim of subdividing the sequence into foraminifera and calcareous nannofossils biozones, determining the age and the paleoenvironment of the strata penetrated by the well. The standard foraminifera and calcareous nannofossil recovery techniques were employed. The lithology of the penetrated sequence is charaterised by the alternating sand and shale sequence of the Agbada Formation at the lower section, while the predominantly sandy upper section of the well corresponds to the Benin Formation. Richly diverse calcareous nannofossils and foraminifera assemblages were recovered. Four foraminifera biozones established in this study are the *Globorotalia tumida/Valvulina flexilis* zone, *Neogloboquadrina dutertrei/Cyclammina minima* zone, *Sphaeroidinella dehiscens/Haplophragmoides narivaensis* zone, *Globorotalia merotumida/plesiotumida/Ammobaculites agglutinans* zone. Two calcareous nannofossils zones established in this study are the *Discoaster* spp. and *Discoaster quinqueramus*/*Discoaster berggrenii* zones. The age assigned to the studied interval of D1 well ranged from Late Miocene to Early Pliocene based on the recovered assemblages. Outer neritic to Upper bathyal depositional environment is inferred for the entire studied interval of D1 well because of the frequent occurrence of the diagnostic species of the following genera: *Uvigerina, Globocassidulina, Eponides, Bulimina, Pullenia, Oridosalis, Sphaeroidina, Cibicidoides, Gyroidinoides* and deep water arenaceous forms such as *Cyclammina, Kareriella* and *Recurvoides*.

Keywords: Foraminifera, Calcareous Nannofossil, Biostratigraphy, Paleoenvironment, Offshore, Niger Delta.

1.0 Introduction

The Niger Delta oil province over the years has become a key place of interest and research as its importance lies in its hydrocarbon resources and is among the world's most productive oil provinces. Over the years, thousands of wells have been drilled across the delta penetrating the sediments, in which petroleum generation, migration and accumulation have occurred. The studied D1 well is located in the offshore depobelt of the eastern Niger Delta (Figure 1). The concentration of this study is on the calcareous nannofossil and foraminifera biostratigraphy, dating and environment of deposition of D1 well.

Fadiya (2014) stated that some biostratigraphic studies have been carried out in the offshore area of the Niger Delta basin with only few of such works on calcareous nannofossils been documented in the literature due to proprietary reasons. Ogunjobi (1996) discussed the advantages of calcareous nannofossils in the recognition of Marine Flooding Surfaces in the Niger Delta most especially in the Late Miocene to Late Pliocene. He recognized four delta wide flooding surfaces based on the *Discoaster quinqueramus*, *Ceratholithus* species and *Gephyrocapsa* species and *Sphenolithus* species. This was confirmed by Oyebamiji (1997) who also observed the appearance of *Sphenolithus abies* in the Late Miocene of the Niger delta. Fernacci *et al.* (1996, 2000) also published valuable information on calcareous nannofossils biostratigraphy of some wells in the Niger Delta basin.

Ozumba (1999) carried out high resolution foraminiferal biostratigraphy of four wells (Kanbo-5, Egbedicreek-1, Angalalli-1 and Opukushi-5) located in the coastal and central swamp of the western Niger Delta. Six foraminiferal zones (Assemblage/Partial range zones) were defined for the middle to late Miocene Niger Delta namely; *Globigerina cf ciperoensis* zone, *Nonion centrosulcatum / Chiloguembelina victoria* zone, *Eponides eshira* zone, *Uvigerina sparsicostata* zone, *Spirosigmoilina oligoceanica* zone, and *Florilus ex. gr. costiferum* zone.

Boboye and Adeleye (2009) dated some sequences in the deep offshore Niger delta Late Miocene, based on the presence of *Eggerella bradyi* and the first downhole occurrence of *Cyclammina cf. minima*. Chukwu (2012) established a planktic *Praeorbulina glomerosa* zone and a benthic *Poritextularia panamensis* zone in Oloibiri-1 well, Eastern Niger delta.

Okosun *et al.* (2012) revealed that the planktic foraminiferal preservation in the wells from Akata field, onshore south-eastern Niger delta is poor, they identified four planktic zones, namely: *Globorotalia continuosa* zone, *Globorotalia mayeri* zone, *Praeorbulina glomerosa* and *Globorotalia peripheroroacuta* zone. They also identified the following benthic zones: *Spirosigmoilina oligocaenica, Uvigerina sparsicostata*, and *Eponides eshira/Brizalina mandorovensis*, and *Poritextularia panamensis*. With these the interval studied was dated Miocene based on foraminiferal assemblage.

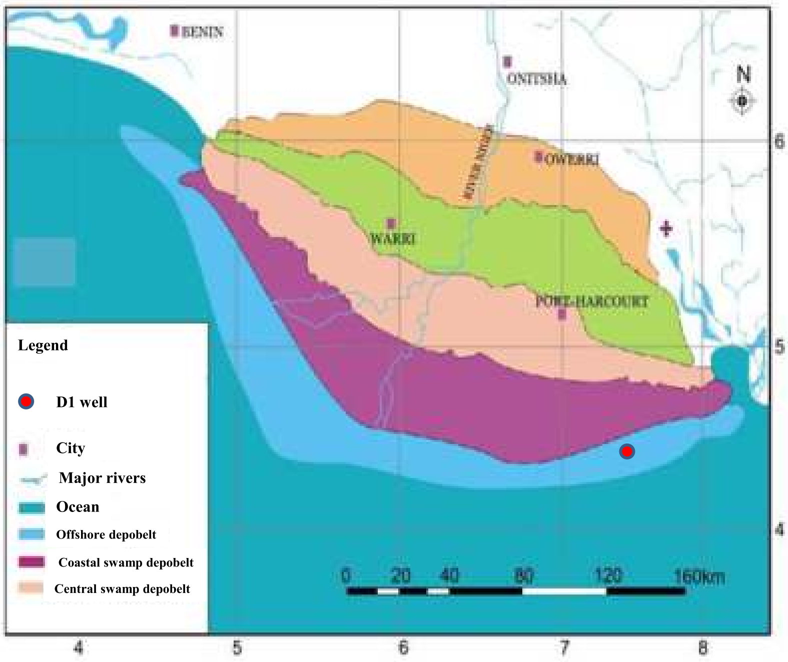


Figure 1: Location of D1 well, Niger Delta

Fadiya (2014) assigned Middle to Late Eocene age to the stratigraphic interval studied from AM-2 well, Niger Delta due to the occurrence of Middle to Late Eocene foraminfera age diagnostic marker species such as *Globigerina eocaena*, *G. bagni*, *G. cryptomphala*, *G. inaequispira*, *Chiloquembelina cubensis, C. martini*, *Pseudohastigerina micra*, *P. wilcoxensis*, *Turborotalia cerroazulensis cerroazulensis*, *T. griffinae*, *T. pseudomayeri* and *T. cerroazulensis pomeroli*.

A great challenge to petroleum exploitation has been the precise biozonation and high resolution biostratigraphic correlation of hydrocarbon bearing units within a field or basin for the purpose of directing well trajectory. This study presents biostratigraphic analysis which will enable mapping of time significant surfaces. The aim of this study is to subdivide the studied interval (D1 well) into calcareous nannofossil and foraminifera biozones and also deduce it paleoenvironment of deposition.

2.0 Materials and Method

One hundred samples from D1 well have been studied, and the samples within the depth interval of 3440 ft. - 9950 ft. were analysed for foraminiferal and calcareous nannofossils. Some of the materials used during sample preparation and analyses include distil water, kerosene, aluminium foil, liquid detergent, hot plate, 63 micron sieve size, filter paper, sample bags, marker pen for labelling the sample bags, picking brush, picking tray, binocular microscope, slides and cover slides and gum.

The kerosene method of preparing samples for foraminifer's recovery was employed because it is economical and could disaggregate the samples. 30 g of each sample was weighed and crushed to loosen the bounded particles. The samples were soaked using distilled water and kerosene in a beaker over night for complete digestion. Samples were then washed with tap water using 63 micron mesh sieve.

The residues from the washed samples were then dried both on hot plate and in an oven. The samples were packed in well labelled sample bags for picking and observation under the binocular microscope. The prepared samples were placed on a picking tray and viewed under a reflected light binocular microscope for any preserved foraminifera content. The foraminiferal specimens were picked out with either a fine brush or wet toothpick and dropped in the micro paleontological slide cavity. Cover slips were used in covering the slides and arranged serially according to their depths in slide tray for analysis. The picked foraminifera were subjected to identification and abundance or diversity counts. During identification, relevant published manuals were utilized, such as Stainforth *et al.* (1975), Bolli *et al.* (1985), Petters (1982, 1983, and 1995), Okosun and Liebau (1999). The biozonation and age determination of the studied well were carried out using the age diagnostic foraminiferal species.

Twenty eight (28) ditch cutting samples were selected from foraminifera-rich depth intervals (5330 ft. - 9170 ft.) from D1 well which indicate periods of high marine influence in the area penetrated by the well. The samples were processed for calcareous nannofossil recovery using the standard preparation technique of Haq *et al.* (1987). The simple smear slide method was routinely applied to process all the samples. The prepared slides were examined for their calcareous nannofossil content under a high power light microscope in cross-polarized and transmitted lights. Detailed abundance counts of the assemblages were made at x1000 magnification. Identification of species was made by consulting the works of Blow (1969), Backman (1980), Perch-Nielsen (1985), Fernacci *et al.* (1996) and Fadiya (1999). Lithological description of the ditch cutting samples was carried out to support the interpretation. The ditch cutting samples were studied with a magnifying hand lens for lithologic description and preparation of lithologic log. The roundness, colour, average grain size and sorting of the sand particles was noted. The lithology of the ditch cutting samples was then calibrated and depth matched with corresponding wireline logs.

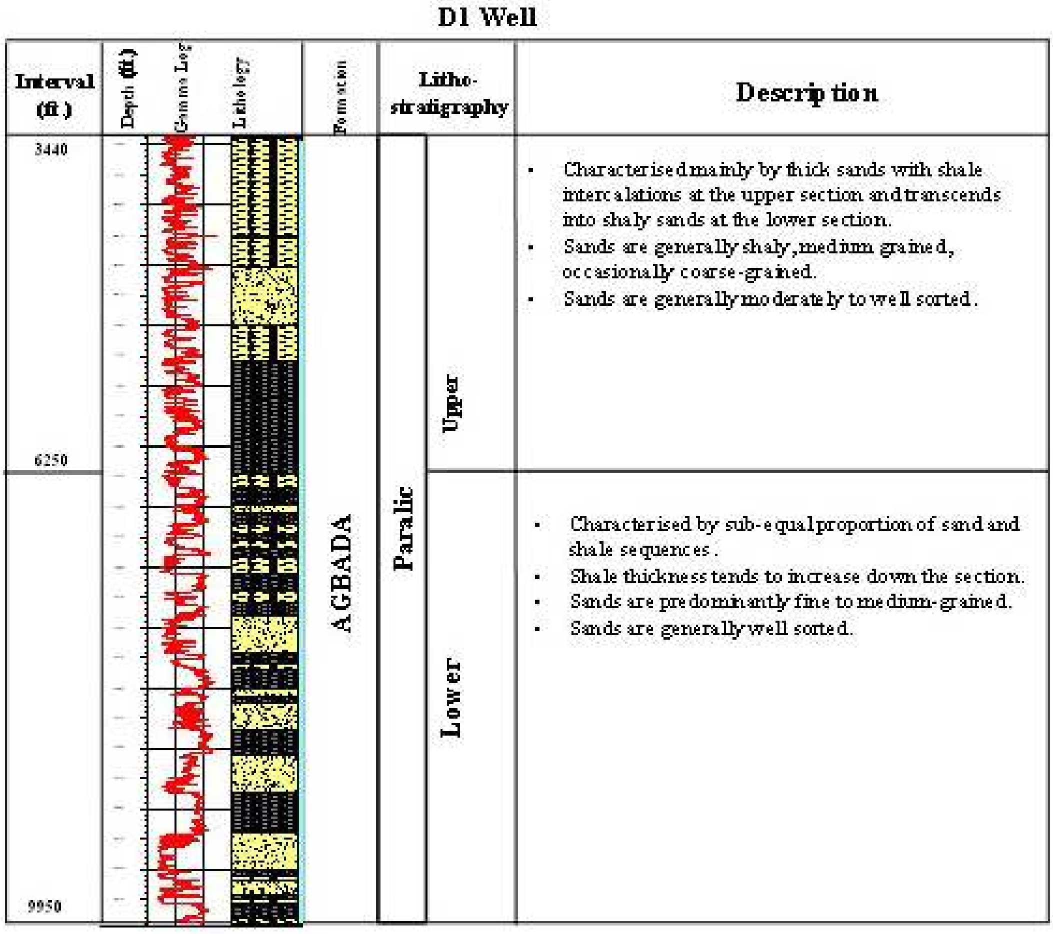
3.0 Results and Discussion

3.1 Lithostratigraphy of D1 well

Lithostratigraphy and subdivision of the D1 well is presented in Figure 2. The studied depth interval ranges from 3440 ft. - 9950 ft. The formation delineated within this interval is the Agbada Formation (3440 ft. - 9950 ft.). Two (2) lithofacies sequences delineated within the studied interval are the Upper paralic sequence and the Lower paralic sequence.

The Upper paralic sequence (3440 ft. - 6250 ft.) is characterised mainly by thick sands with shale intercalations at the upper section and transcends into shaly sands at the lower section. Sands are generally shaly, medium grained, occasionally coarse-grained. Sands are generally moderately to well sorted.

The Lower paralic sequence (6250 ft. - 9950 ft.) is characterised by sub-equal proportion of sand and shale sequences. Shale thickness tends to increase down the section. Sands are predominantly fine to medium-grained. Sands are generally well sorted (Figure 2).



- Characterised mainly by thick sands with shale intercalations at the upper section and transcends into shaly sands at the lower section.
- Sands are generally shaly, medium grained, occasionally coarse-grained.
- Sands are generally moderately to well sorted.

- Characterised by sub-equal proportion of sand and shale sequences.
- Shale thickness tends to increase down the section.
- Sands are predominantly fine to medium-grained.
- Sands are generally well sorted.

Figure 2: Lithostratigraphic description and subdivision of D1 well

3.2 Biostratigraphy of D1 Well

The results of the foraminifera and calcareous nannofossil analysis of the studied interval are presented in the foraminifera distribution chart, calcareous nannofossils distribution chart, foraminifera and calcareous nannofossils synthesis chart of D1 well (Figures 3, 4, 5, 6 and 7). A lithologic log was prepared by integrating lithologic description data with the available well log data which include gamma ray and resistivity logs. Some photomicrographs of the recovered foraminifera and calcareous nannofossils species are presented in Plates 1 and 2.

3.2.1 Foraminifera Biostratigraphy of D1 Well

Foraminiferal analysis of D1 well presented below was carried out using One hundred (100) ditch cutting samples and the biozonation of the well was based largely on sequence stratigraphic principles with precise age dating of zonal boundaries.

The statistical data obtained was computerized using the StrataBugs software. Bar plots of the abundance and species diversity were made, from which candidate Maximum Flooding Surfaces (MFS) were selected. Their positions were later confirmed on the log. The complete micropaleontological data is plotted in colour using StrataBugs at a scale of 1:5000. Faunal associations including benthic, planktic, benthic/planktic ratio (normalised) agglutinated/calcareous foraminiferal ratios etc., are plotted in Figure 3. The ditch cutting samples were analysed at 60 ft. intervals for foraminifera and richly diverse assemblages of planktonic and benthonic foraminifera species with a total of 93 species recorded. 70 species (75%) are calcareous, while 23 (25%) are arenaceous. Among the calcareous forms, benthics accounted for 66% (46 species), while the remaining 24 species (34%) are planktics. The foraminiferal distribution chart and the plots of the peaks of species diversity and population abundance are presented in Figure 3, while the zones are shown in Figure 4.

A good number of diagnostic marker species such as *Cyclammina minima*, *Karreriella bradyi, Haplophragmoides narivaensis, Valvulina flexilis, Neogloboquadrina dutertrei, Sphaeroidinella dehiscens, Globorotalia plesiotumida/merotumida, Globigerinoides bulloideus, Turborotalia acostaensis, Globorotalia tumida tumida* and *Globigerinoides extremus* were recovered from D1 well. This assemblage is typical of N16 - N19 foraminiferal zones of Berggren *et al.* (1998) of the Late Miocene – Early Pliocene age and permitted the zonal subdivision of the well section based on the zonation scheme of Berggren *et al.* (1998) and Gradstein *et al.* (2012).

Four (4) foraminifera zones are recognized in D1 well namely: *Globorotalia tumida/Valvulina flexilis* zone, *Neogloboquadrina dutertrei/Cyclammina minima* zone, *Sphaeroidinella dehiscens/ Haplophragmoides narivaensis* zone and *Globorotalia mero/plesiotumida/Ammobaculites agglutinans* zone, based on the critical evaluation of the key bioevents, particularly the First Downhole Occurrence (FDO) and Last Downhole Occurrence (LDO) of chronostratigraphically important foraminifera markers. Characteristic benthic markers such as *Cyclammina minima*, *Karreriella bradyi* and *Haplophragmoides narivaensis*, whose LAD's / FDO's mark Late Miocene ages confirm the age of the well.

Zone: *Globorotalia tumida/Valvulina flexilis* zone

Stratigraphic Interval*:* 3440-4430 feet.

Age: Early Pliocene

Diagnosis: This is the topmost zone identified in the studied section of the well. The zonal top is tentatively placed at 3440 ft, the depth of the first sample analyzed, while the base is placed at the observed FDO of *Cyclammina minima* recorded at 4430 feet which marks the top of the next zone. The interval is characterized by the LDO of *Globorotalia tumida*, FDO of *Valvulina flexilis* and occurrence of associated forms such as *Globigerina bulloides, Globigerina praebulloides, Globigerinoides extremus, Globigerinoides immaturus, Globigerinoides sacculiferus, Globoquadrina altispira, Globorotalia plesiotumida/ merotumida, Globorotalia scitula, Globorotalia tumida tumida, Turborotalia acostaensis, Globorotalia scitula gigantea* and *Globorotalia scitula praescitula*. The zone is correlated with the Lower N19 and Upper N18 foraminifera zone of Berggren *et al.* (1998) and Gradstein *et al.* (2012). The age is Early Pliocene.

Zone: *Neogloboquadrina dutertrei / Cyclammina minima* zone

Stratigraphic Interval*:* 4430-6980 feet.

Age: Late Miocene

Diagnosis: The top of this zone is placed at the observed FDO of the zonal marker *Cyclammina minima* recorded at 4430 ft. while the base is marked by the FDO of *Haplophragmoides narivaensis* recorded at 6980 feet. The other zonal markers in this zone are *Neogloboquadrina dutertrei and Karreriella bradyi.* The associated species in this zone include *Globigerina bulloides, Globigerinoides bolli, Globigerinoides immaturus, Globorotalia plesiotumida/ merotumida, Globorotalia scitula, Turborotalia acostaensis* and *Orbulina universa*. The 5.47Ma MFS; Gradstein *et al.* (2012) recognized at 5390 ft. occur within this zone. The zone correlates with the Upper N18 foraminifera zone of Berggren *et al.* (1998) and Gradstein *et al.* (2012). The age for this zonal interval is **Late Miocene**.

Zone: *Sphaeroidinella dehiscens / Haplophragmoides narivaensis* zone

Stratigraphic Interval*:* 6980-9410 feet

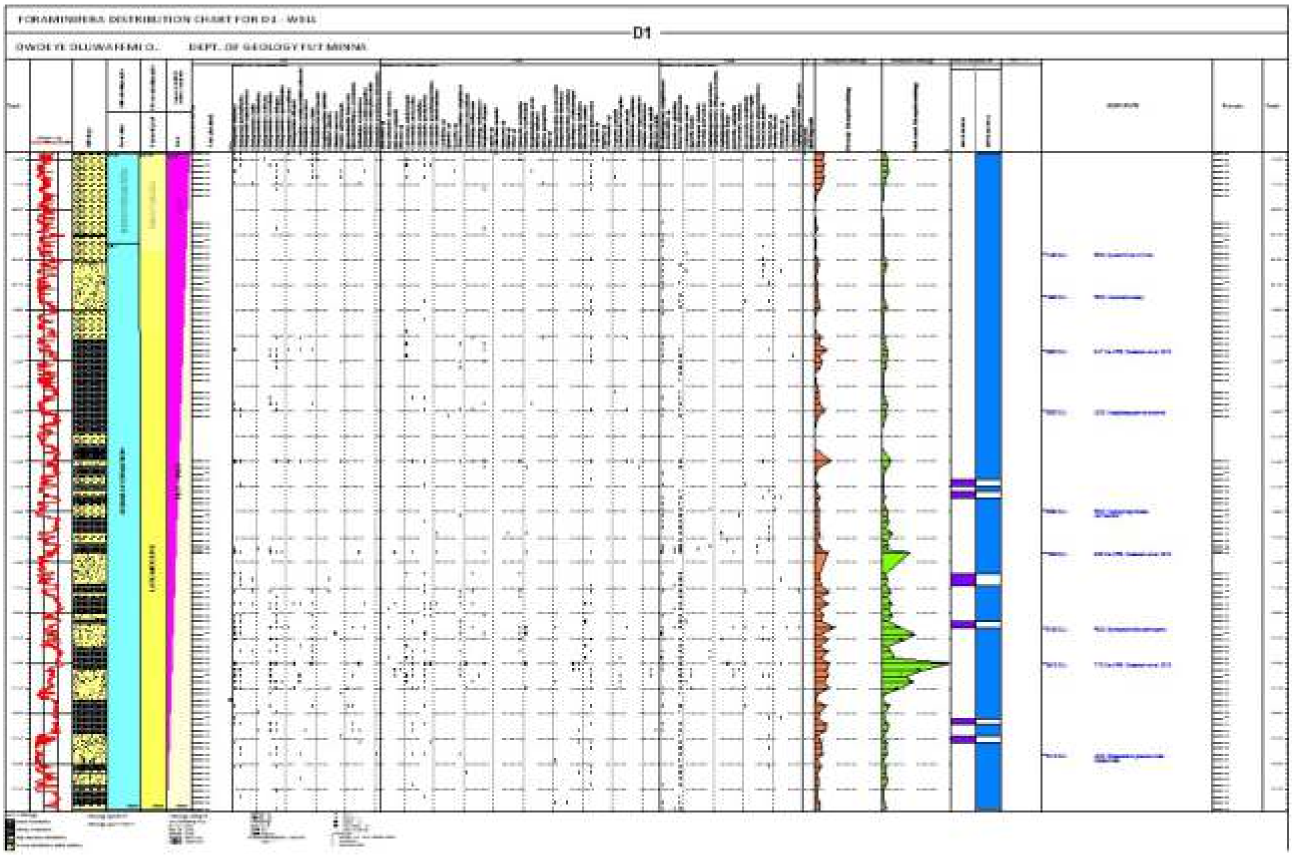


Figure 3: Foraminiferal distribution chart of D1 well.

Age: Late Miocene

Diagnosis: The zonal marker, *Haplophragmoides narivaensis* whose FDO marks the top of the zone was recorded at 6980 feet while the base is marked by the LDO of *Globorotalia mero/plesiotumida* recorded at 9410 feet which marks the top of the next zone. The other zonal marker in this zone is *Sphaeroidinella dehiscens.* The associated species in this zone include *Globigerina bulloides, Globigerina praebulloides, Globigerinoides obliquus, Globigerinoides quadrilobatus, Globorotalia mero/plesiotumida, Globorotalia scitula, Turborotalia acostaensis, Globorotalia scitula gigantea* and *Globorotalia acostaensis trochoidea*. The 5.99Ma MFS; Gradstein *et al.* (2012) recognized at 7395 feet occur within this zone. The zone correlates with the Upper N17 - LowerN17 foraminifera zone of Berggren *et al*. (1998) and Gradstein *et al.* (2012). The age is Late Miocene.

Zone: *Globorotalia mero/plesiotumida / Ammobaculites agglutinans* zone

Stratigraphic Interval*:* 9410 ft. - 9950 ft.

Age: Late Miocene

Diagnosis: The LDO of the zonal marker *Globorotalia mero/plesiotumida* recorded at 9410 ft. marks the top of this zone. *Ammobaculities agglutinans*, another zonal maker whose FDO, marks the top of the zone was not recorded in the well. The base of the zone is tentatively placed at 9950 feet, the depth of the last sample for the studied section of the well. The associated species in this zone include *Globigerina bulloides, Globigerina praebulloides, Globigerinoides obliquus, Globigerinoides quadrilobatus* and *Globorotalia acostaensis trochoidea*. The zone correlates with the “Upper” N16 Planktic Foraminifera zone of Berggren *et al.* (1998) and Gradstein *et al.* (2012). The age for this zonal interval is Late Miocene.

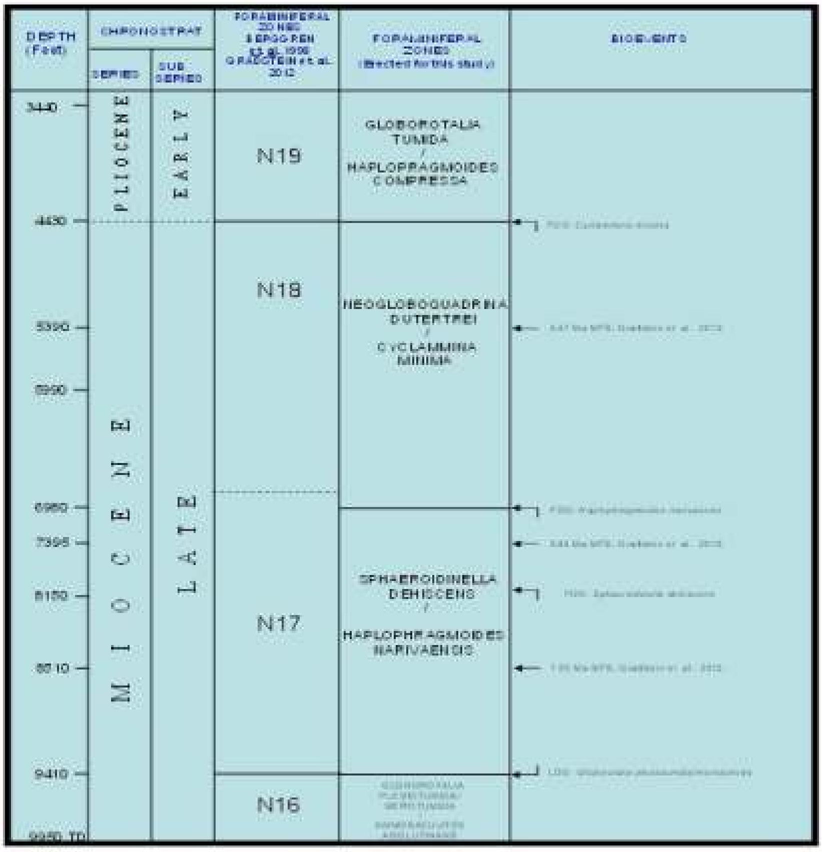


Figure 4: Foraminiferal zones recognised in D1 well.

3.2.2 Calcareous Nannofossil Biostratigraphy of D1 well

A total of twenty-eight samples were analysed for calcareous nannofossils. Calcareous nannofossils are studied under a transmitting light microscope in polarised light. For this, a binocular microscope was used with work being done at 1250x magnification with immersion oil. Then standard counts of 12 traverses were carried out followed by an extensive search of the slide for rare marker fossils. This method was standardised for each slide in the well. The D1 well is moderately rich and diverse in calcareous nannofossils. A total of fifteen nannofossil species were recovered. The biozonation and age dating of the wells was based largely on calcareous nannofossils assemblages, abundance and diversity.

The assemblage comprises mainly of *Discoaster quinqueramus, Discoaster pentaradiatus, Discoaster brouweri, Discoaster beggrenii, Pontosphera japonica, Pontosphera multipora, Coccolithus pelagicus, Sphenolithus moriformis, Reticulofenestra haqii, Helicosphera cateri*.

The chronostratigraphic scheme adopted follows the usage of the worldwide zonation schemes of Martini (1971), Okada & Bukry (1980) and Gradstein *et al.* (2012). Considerable effort was made to identify and define zonal tops with the First Down-hole Occurrence (FDO's), Last Down-hole Occurrence (LDO's) of diagnostic marker species, abundance, and species diversity peak as these form the most reliable events.

The highest nannofossil peaks were dated using important marker species such as *Discoaster quinqueramus* and *Discoaster berggrenii*. The stratigraphic distribution of the recorded species along with the significant datum, suggested Maximum Flooding Surfaces, Nannofossil zones and age interpretations. The result of this analysis is ~~been~~ presented in the nannofossils distribution chart and the summary of biozones shown in Figure 5 and 6.

Nannofossil zones of D1 well

Interval: 5330 – 6860 feet

Zone: ?NN12

Age: Late Miocene

Diagnosis: This interval is NN12 zone of Martini, 1971 based on superpositioning and it is characterized by few diagnostic calcareous nannofossils. There are records of *Discoaster spp., Discoaster brouweri, Reticulofenestra minuta, Reticulofenestra hagii, Helicosphaera multipora.*

Interval: 6860 – 9170 feet

Zone: NN11

Age: Late Miocene

Diagnosis: This interval is fairly rich in calcareous nannofossils. The FDO of *Discoaster quinqueramus* at 7100 ft. marks the top of this NN11 zone of Martini (1971). The base of the zone is tentatively placed at 9170 ft. the depth of the last sample for the studied interval. The highest peak at 7395 feet is dated 5.99 Ma MFS (Gradstein *et al.,* 2012) condensed section.

The presence of *Discoaster berggrenii* at 8390 ft. marks the 7.72 Ma MFS (Gradstein *et al.,* 2012).

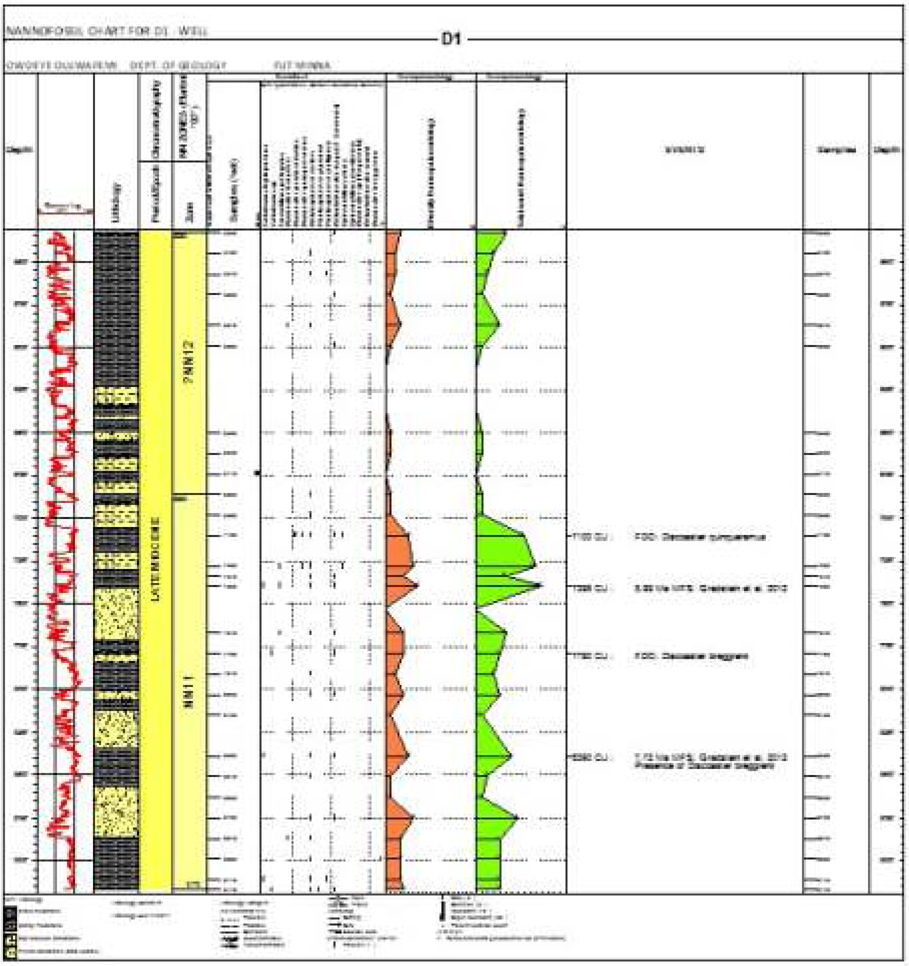
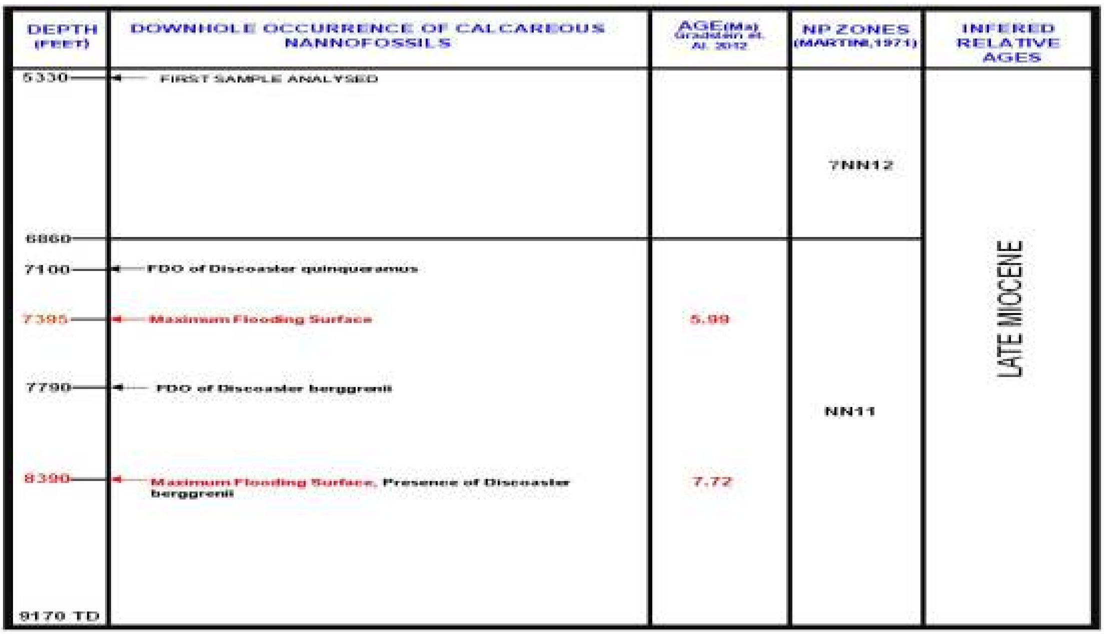


Figure 5: Calcareous nannofossil distribution chart of D1 well.



DEPTH (FEET)	DOWNHOLE OCCURRENCE OF CALCAREOUS NANNOFOSSILS	AGE(Ma) Gradstein et. Al. 2012	NP ZONES (MARTINI,1971)	INFERED RELATIVE AGES
5330	FIRST SAMPLE ANALYSED		?NN12	LATE MIOCENE
6860			NN11	
7100	FDO of Discoaster quinqueramus			
7395	Maximum Flooding Surface	5.99		
7790	FDO of Discoaster berggrenii			
8390	Maximum Flooding Surface, Presence of Discoaster berggrenii	7.72		
9170 TD				

Figure 6: Calcareous nannofossil zones recognised in D1 well.

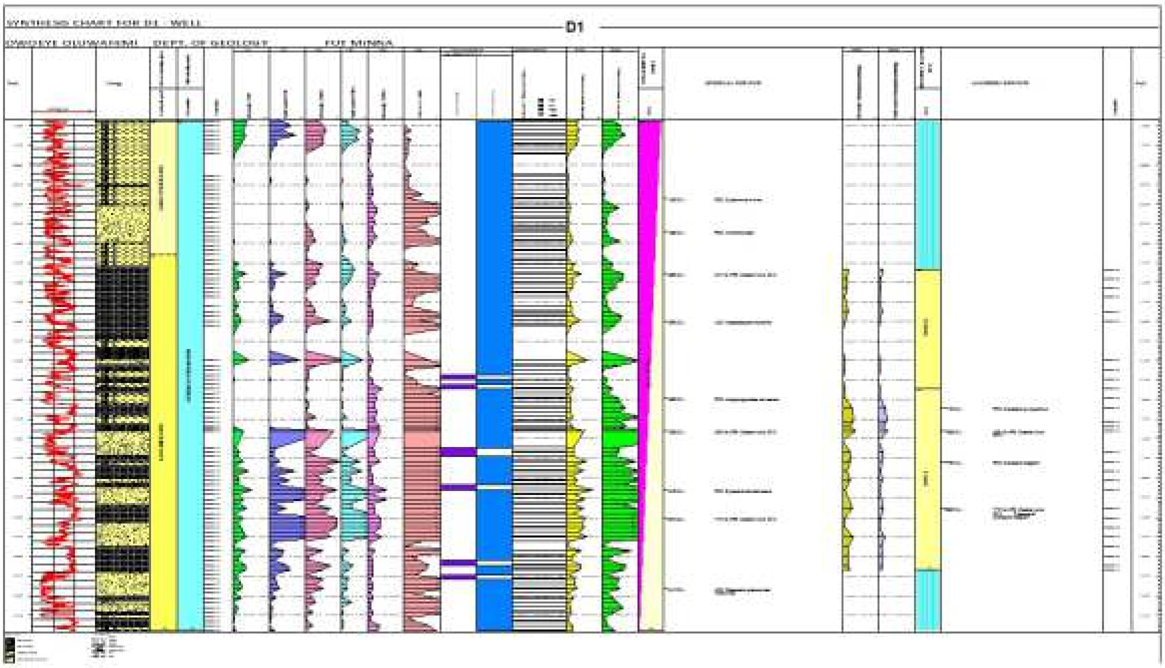


Figure 7: Biostratigraphic Synthesis Chart produced for D1 well.

A biostratigraphic synthesis chart was produced for the studied interval (Figure 7). This chart represents the summary of all the analysis carried out in this study as it aim is to integrate all findings from the foraminiferal and calcareous nannofossil analysis.

4.0 Paleoenvironment

Inference on the paleodepositional environment of the studied well was based on the biofacies information interpreted from the qualitative and quantitative evaluation of the benthic foraminiferal assemblages and integration of the lithologic description of the section. The parameter considered in the interpretation of the paleoenvironment is the presence/absence of environmental diagnostic marker species (Phleger 1960 and Murray 1991).

The depositional environment of D1 well is predominantly Upper Bathyal, shallowing to Outer Neritic at some horizons. Details of the paleoenvironments and the associated taxa are presented below on interval basis:

i. Stratigraphic Interval*:* 3440-5270 feet.

Common occurrence of deep water foraminifera such as *Cibicidoides pseudoungerianus, Cibicidoides pachyderma, Uvigerina proboscidea, Alveolophragmium subglobosum, Cyclammina minima, Eggerella bradyi, Trochammina proteus, Recurvoides deformis* and *Valvulina flexilis* confirm an environment of deposition that is predominantly Upper Bathyal (Phleger, 1960 and Bandy, 1967; Stanley and Adegoke 1972).

ii. Stratigraphic Interval*:* 5270-7395 feet.

The abundant record of Upper Bathyal foraminifera such as *Cibicidoides pachyderma, Cibicidoides pseudoungerianus, Globocassidulina subglobosa, Gyroidinoides girardanus, Heterolepa bellincioni, Hoeglundina elegans, Planulina wuellerstorfi, Uvigerina proboscidea, Alveolophragmium subglobosum, Eggerella bradyi, Trochammina proteus* and *Recurvoides deformis* confirm an environment of deposition that is predominantly Upper Bathyal, shallowing to Outer Neritic at some horizons (Phleger, 1960 and Bandy, 1967).

iii. Stratigraphic Interval: 7395-9950 feet.

The abundant record of Upper Bathyal foraminifera such as *Bulimina marginata, Anomalinoides alazanensis, Cibicidoides incrassatus, Cibicidoides pachyderma,* *Cibicidoides pseudoungerianus, Globocassidulina subglobosa, Gyroidinoides neosoldanii, Heterolepa bellincioni, Heterolepa dertonensis, Hoeglundina elegans, Planulina wuellerstorfi, Uvigerina proboscidea, Sigmoilopsis schlumbergerii, Sphaeroidina bulloides, Alveolophragmium subglobosum, Eggerella bradyi, Trochammina proteus* and *Recurvoides deformis* confirm an environment of deposition that is predominantly Upper Bathyal, shallowing to Outer Neritic at some horizons (Phleger, 1960 and Bandy, 1967).

Photomicrographs of some of the foraminifera species recovered from D1 well are shown in Plate I and calcareous nannofossils species recovered from the studied interval are also shown in Plate 2.

5.0 Conclusion

One hundred (100) ditch cutting samples from D1 well within the depth interval of 3440 - 9950 ft. yielded richly diverse assemblages of planktonic and benthonic foraminifera species and twenty-eight (28) ditch cutting samples selected from foraminifera-rich depth intervals from D1 well which were also analysed yielded moderately rich and diverse assemblages of calcareous nannofossils species. The foraminifera biozones established in this study are the *Globorotalia tumida/ Valvulina flexilis zone*, *Neogloboquadrina dutertrei/Cyclammina minima zone*, *Sphaeroidinella dehiscens/Haplophragmoides narivaensis zone*, *Globorotalia mero /plesiotumida/ Ammobaculites agglutinans* zone. These zones correspond to the Upper N16 - Lower N19 zones of Berggren *et al*. (1998) and Gradstein *et al*. (2012). Calcareous nannofossil biostratigraphic analysis was also carried out in D1 well, and the *Discoaster* spp. and *Discoaster quinqueramus* / *Discoaster berggrenii* zones were established. These zones correspond to the NN12 and NN11 zones of Martini (1971). The age assigned to the studied interval of D1 well ranged from Late Miocene to Early Pliocene.

The age assigned to the stratigraphic surfaces delineated within the studied interval (5.47 Ma - 8.5 Ma) established the fact that the well is located in the ofshore depobelt of the Niger Delta Basin.

Outer neritic to bathyal depositional environment is inferred for the entire studied interval of D1 well because of the frequent occurrence of the diagnostic species of the following genera: *Uvigerna, Globocassidulina, Eponides, Bulimina, Pullenia, Oridosalis, Sphaeroidina, Cibicidoides, Gyroidinoides* and deep water arenaceous forms such as *Cyclammina, Kareriella* and *Recurvoides* known to inhabit outer neritic to bathyal environment. Based on the lithologic, foraminiferal and paleoenvironmental analysis, it is inferred that the intervals penetrated by the well corresponds to Benin Formation and Agbada Formation, and they are of Late Miocene to Early Pliocene age.

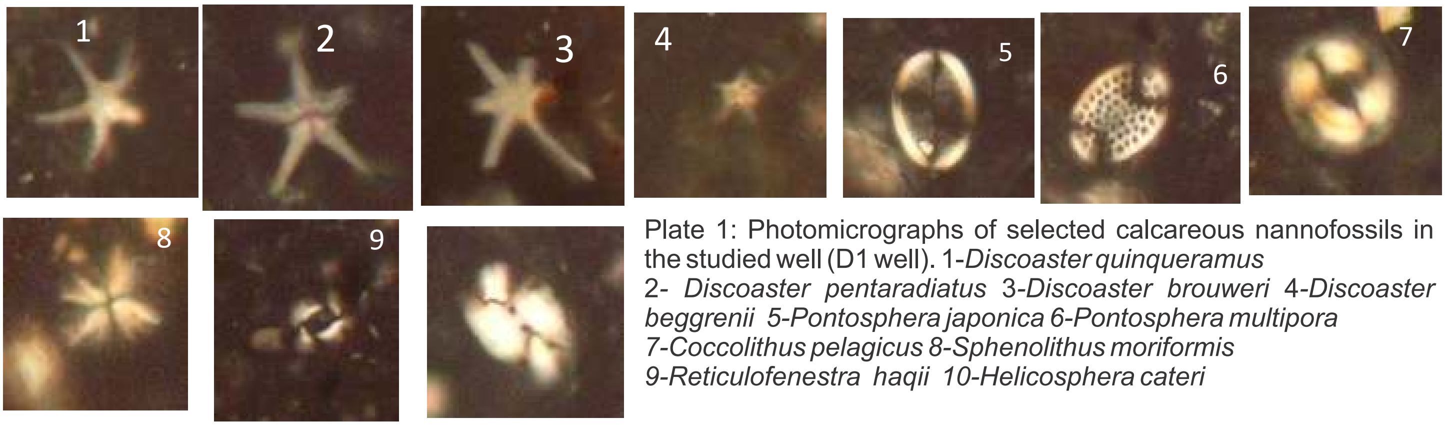
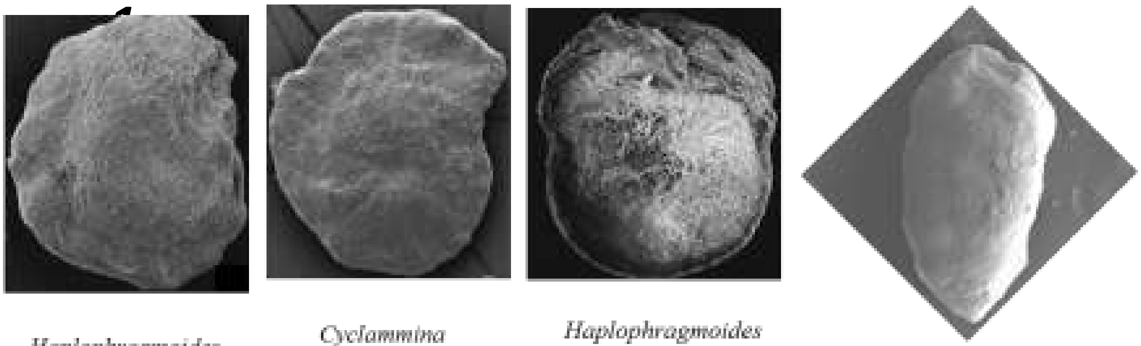


Plate 1: Photomicrographs of selected calcareous nannofossils in the studied well (D1 well). 1-*Discoaster quinqueramus* 2- *Discoaster pentaradiatus* 3-*Discoaster brouweri* 4-*Discoaster beggrenii* 5-*Pontosphera japonica* 6-*Pontosphera multipora* 7-*Coccolithus pelagicus* 8-*Sphenolithus moriformis* 9-*Reticulofenestra haqii* 10-*Helicosphera cateri*

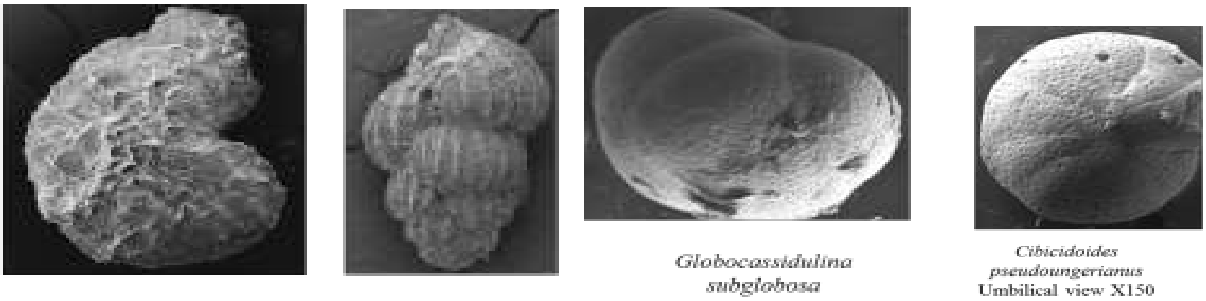


Haplophragmoides compressa
Umbilical/Side view X100

Cyclammina minima
Umbilical/Side view X100

Haplophragmoides narivaensis
Side view X100

Karreriella bradyi
Apertural/Side view X150



Ammobaculites agglutinans
Side view X100

Uvigerina peregrina
Side view X100

Globocassidulina subglobosa
Side view X100

Cibicidoides pseudoungerianus
Umbilical view X150

Plates 2: Photomicrographs of selected foraminifera species within the studied wells.

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Mineralogical Characterisation of Enyigba Lead Ore, Ebonyi State, Nigeria

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Abstract

Galena ore deposits' in Enyigba, Ebonyi Stata is being exploited in small-scales and in a crude method that needs attention for sustainable mining development. In an attempt to characterize the ore, A research was carried out using optical microscopy, XRD, and scanning electron microscopy (SEM) in order to delineate the mineralogy and chemical composition of the ore. The result of the analyses revealed that galena sulfides and quartz are the major while chalcopyrite and pyrite are the minor mineral phases in the ore matrix. The ore can serve as another potential sources of lead and copper minerals and generate the sought foreign exchange if fully explored and exploited. where copper, silver and gold can be recovered as byproducts.

Key words: Galena, Mineralogy, Characterization, Deposit.

1.0 Introduction

It has been established that Nigeria is endowed with mineral resources ranges from metallic, non-metallic, industrial, as well as energy generation varieties. However, despite the abundance of mineral resource potentials in the country, exploration and exploitation still remain abysmally low and have failed to contribute substantially to the growth of the economy due mainly to poor mining and characterization practices (Anon, 2012).

Mineralogical characterization of an ore deposit has dual importance as it (i) provides genetic information i e., about the source and physicochemical environment of formation of a deposit and (ii) plays a critical role in optimum utilization of scarce natural resources. It helps in the selection of suitable recovery method and dictates the process flow sheet and plant flow sheet optimization (Xiao and Laplante, 2004).

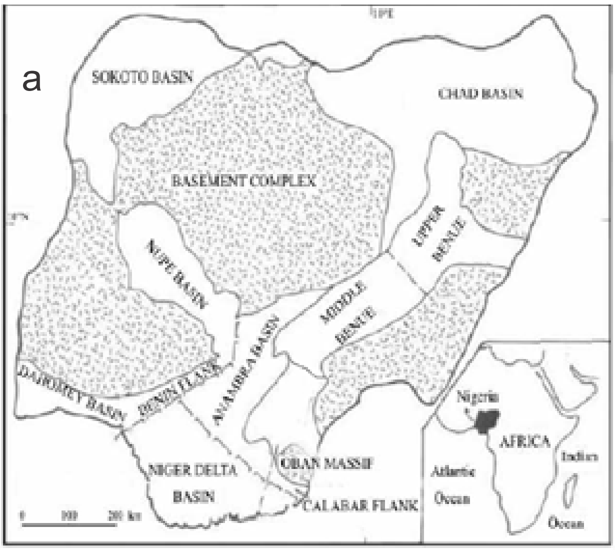
According to Cook (2000), the growing need for detailed information about the mineralogical composition of a mineral deposit therefore form an integral and often critical part of investigations of deposits. Knowledge of mineralogical/chemical composition, size, morphology and association with other minerals is therefore expected to provide insights and information on the characteristics, type, nature and amount of minerals and elements present within the ore at different locations that would permit an assessment and determination of the optimal processing route for its constituent minerals/metals.

Since different minerals from different locations within an ore deposit have different mineralogical compositions with different characteristics and complexities, their qualitative and quantitative distributions vary from location to location within an ore deposit, and from deposit to deposit, which could lead to variations in their recovery processes (Eymery and Ylli, 2000; Kahn et al., 2001; Hope et al., 2001).

Further to this, the prevalent illegal mining syndrome especially in Zamfara State that led to massive lead poisoning that killed scores of people made up of mainly women and children, who were "scavenging or pig cropping" for gold, a few years ago, may not be unconnected with poor characterization and lack of sufficient knowledge of ore composition. Hence, the needs to carry out the characterization of Ebonyi ore deposit.

2. Brief Geology of Lead-Zinc Mineralization in Nigeria

A brief look at the geology of Nigeria is important to give an idea of the geological composition of the country in terms of the ore bearing rock groupings, while the review of Pb-Zn mineralization is intended to give credence to findings in previous studies that the Benue Trough houses some of the complex sulphide minerals including the lead-Zinc deposits under study (MMSD, 2010). The Geology of Nigeria is composed of four main groups known as: the Basement Complex, the Younger Granites, the Sedimentary series and the Tertiary-Recent Volcanic rocks. Precambrian rocks of the "Basement Complex", including gneisses, amphibolites, marbles and the "Older Granites" underlie large parts of Nigeria, while the post-tectonic tin-tungsten-bearing "Younger Granites" of late Paleozoic to Mesozoic age exhibit ring structures. The southwest-northeast striking Benue Trough is part of a down-faulted "failed arm" of a triple junction that formed during the separation of the African plate and the South American plate, is a NE-SW elongated Trough of subsidence which extends from the Gulf of Guinea to Lake Chad. It is considered as a part of the Pan African Mobile Zone of West Africa. The Benue Trough is largely covered by Cretaceous continental and marine sediments. Figure 1(a) is a map of Nigeria showing the Benue Trough divided into three: the Upper, Middle and Lower Benue Trough which constitute the major Pb-Zn mineral bearing feature in Nigeria. While Figure 1(b) shows the lead-zinc mineralization in Nigeria, which is associated with saline water intrusion in the sedimentary basins or fractured/shear zones in crystalline rocks with Lead-Zinc mineralization occurring in almost the entire 800 km length.



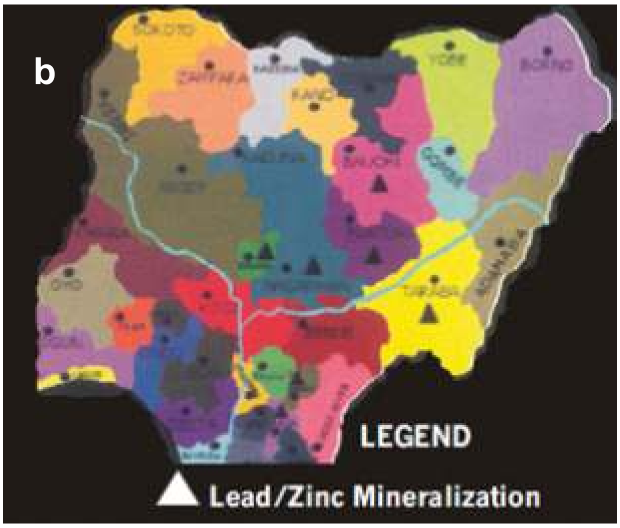


Figure1:(a) The Benue trough and (b) Lead/Zinc mineralization in Nigeria.

The general geology of lower Benue Trough in Abakaliki area is made up of thick sequences (500 m) of slightly deformed cretaceous sedimentary rocks made up of essentially of albian shales, subordinate siltstones of the Asu River Group. There is also the presence of Volcanic and Pyroclastic materials forming elongated conical hills in the cores of the anticlinal Structures. The Lower Benue Trough houses the Ebonyi State Pb-Zn mineralization shown in Figure 2 is emplaced at a low temperature of about 140˚C and it is made up of primarily four lodes namely Ishiagu, Enyigba, Ameri and Ameka all in the present day Ebonyi state. In the Middle Benue

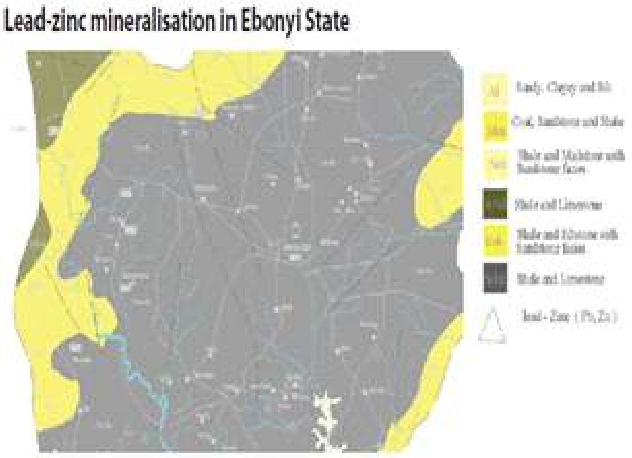


Figure 2. Map of lead/zinc mineralization in Ebonyi State. Source: Nigerian geological survey agency of the Ministry of Mines and Minerals Development (MMSD/NGSA, 2010).

Trough, veins are located mainly in Akwana and Arufu in Nassarawa State, while the Upper Benue Trough composed of the Zurak, Isimiya, Digi, Biu locations, etc in Taraba and Borno States (Nwachukwu, 1974; Maurin and Benkheli, 1990). The Ebonyi State Lead-Zinc are localized in the Enyingba area along the Northeast-Southwest trending belt of slightly deformed volcanic and sedimentary Cretaceous sequences (Albian Asu River Group) which is about 500 m thick, and they occur in the form of veins and veinlets associated with the host rock. Figure 2 shows map of Pb-Zn mineralization in Ebonyi State, while Figure 3(a) and Figure 3(b) show Sketch maps of the Benue Trough locating the main Pb-Zn occurrences and map of the Enygba area showing the distribution of the mineralized fractures, respectively. The Lead-Zinc is often associated with other gangue minerals such as marcasite, pyrite, quartz, barites etc

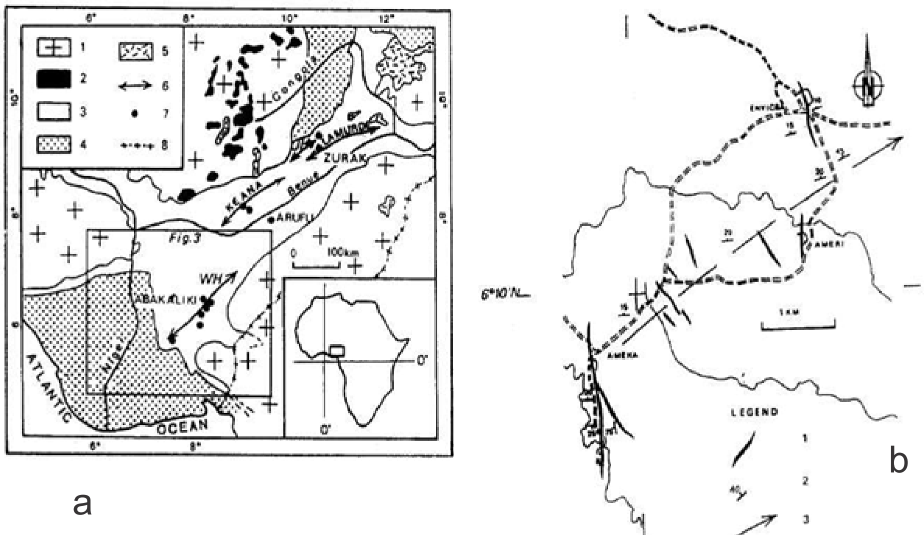


Figure 3. (a) Sketch map of the Benue Trough and Location of the main Pb-Zn occurrences. (1) Precambrian basement; (2) Jurassic younger granites; (3) Cretaceous; (4) Undifferentiated tertiary; (5) Neogene to recent volcanic; (6) Anticlinal axis; (7) Location of mineralization; (8) International border. WH = Workum Hills, JH = Jawara Anticline; (b) Map of the Enygba area showing the distribution of the mineralized fractures. (1) Mineralized fractures; (2) Strike and dip of strate; (3) Anticlinal axis (Maurin & Benkhelil, 1990).

3.0 Materials and Methods

Five (5) kg of samples collected in lumps size from different locations in the Benue Trough (Enyigba lode) with mineralized body stretching about 2km length and approximately 30m wide were broken manually with sledge hammer to provide a required size acceptable to laboratory jaw crusher. Crushed and pulverized, then coned and quartered to yield a representative sample. The samples were massive (Fig. 1) and physically appear to be dominated mainly by galena.

Plate 1: Photograph of Samples from Enyingba (Ebony State)

An integrated instrumental method of mineral characterization using optical microscope, X-ray diffraction (XRD), X-ray fluorescence (XRF) and Scanning Electron Microscopy (SEM) was used to characterize the sample. Polished mounts of the lumps were prepared and studied under polarized reflected light using Leica-make optical petrological microscope and under a JEOL-make EPMA (JXA-8230) having attachments of both EDS and WDS. X-ray image for elemental mapping was carried out. Quantitative mineral compositions were determined at a voltage of 20 kV, ~20 nA sample current and keeping the probe diameter 2μm. Powder samples were investigated by X-ray diffraction model panalytical ADX700 (EMPYRIAN) with Cu-target, carried out at Nigerian Geological Survey Agency (NGSA), Kaduna.

4.0 Results and Discussion

X-ray diffraction analysis of the sample confirmed the dominance of galena with minor association of sphalerite. While quartz is the major associated gangue in the sample. In addition, optical microscopy revealed the trace presence of chalcopyrite, pyrite, framboidal pyrite, marcasite veins in the sample. Semi-quantitative volume% estimates from the polished mounts indicate that petrology of Enyigba is very straightforward and simple (galena + minor other sulfides + quartz). Random point analysis by EPMA on different galena grains of the Enyigba lode analyzed trace quantities of iron, cobalt, nickel, copper and zinc (Table 1). However, germanium and cadmium contents are significant which occur up to the first decimal level. Presence of strontium, arsenic, antimony and silver was also recorded in galena.

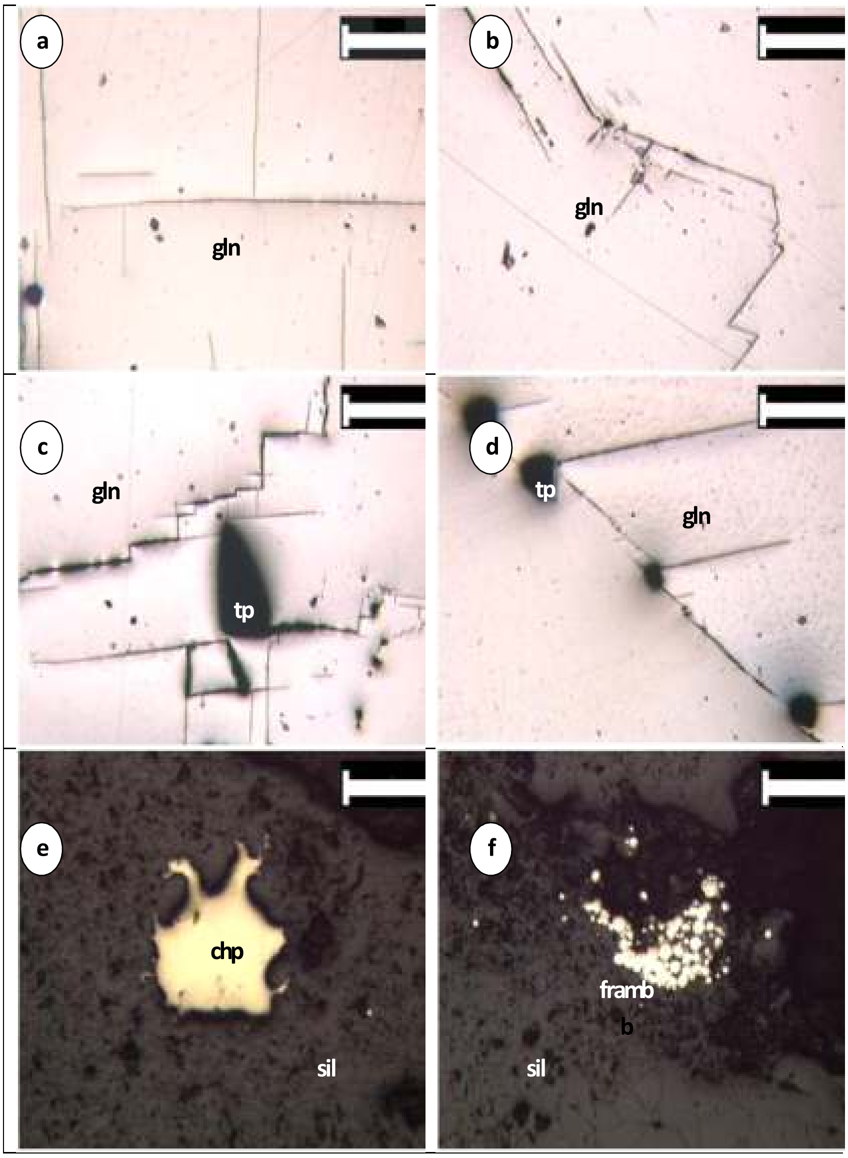


Plate4.1: Optical photomicrographs showing the characters of minerals in Ebonyi sample. (a) and (b): Galena (gln) showing cleavage and fracture planes; (c) and (d): Galena (gln) showing triangular pits (tp); (e): Chalcopyrite (chp) occurring within a silicate matrix (sil); (f): Pyrite framboids (framb) occurring within a silicate base (sil). Photographs are taken in plane polarized reflected light and the bar-scale in the photomicrographs equals 200μm.

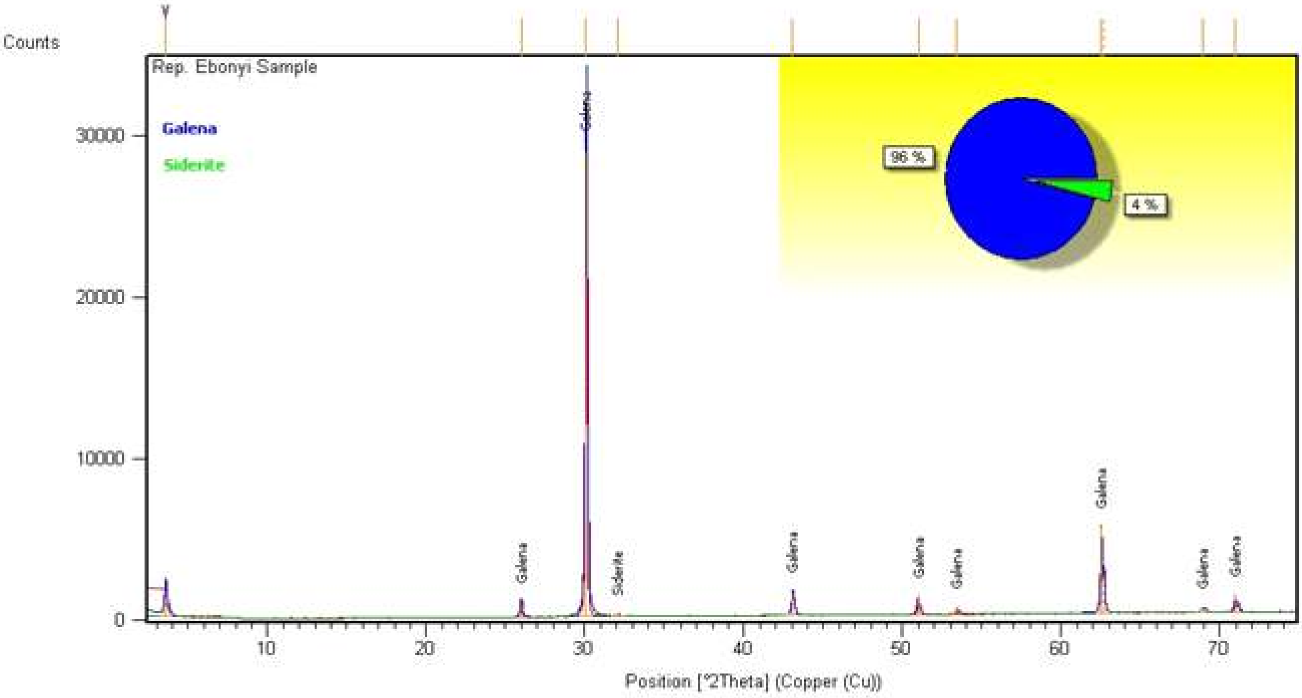


Figure 4: XRD Pattern of the Sample

Table 1: Chemical composition of Enyigba Lead samples by EPMA

Element	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Sb	0.01	0.02	0.06	0.03	0.02
S	13.22	13.77	13.34	13.11	13.11
Fe	0.00	0.02	0.00	0.02	0.03
Zn	0.00	0.02	0.00	0.00	0.00
Pb	86.03	85.97	85.59	86.18	86.19

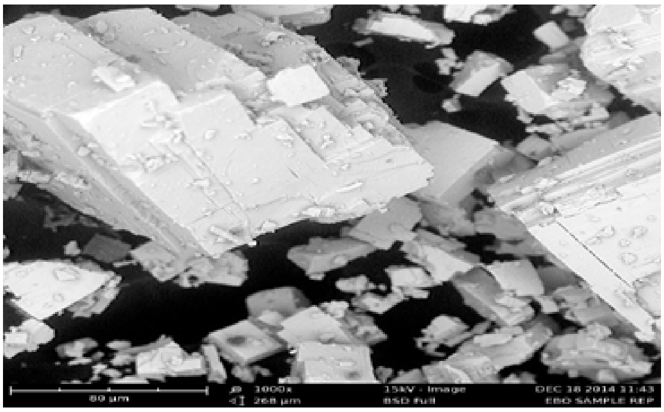


Plate 4.2; scanning electron micrograph showing majorly cubical shape with other shape such as rectangular etc as the surface morphology of Ebonyi sample. Photomicrograph taken at the bar-scale of 80µm on magnification of 1000x at full back scatter detector.

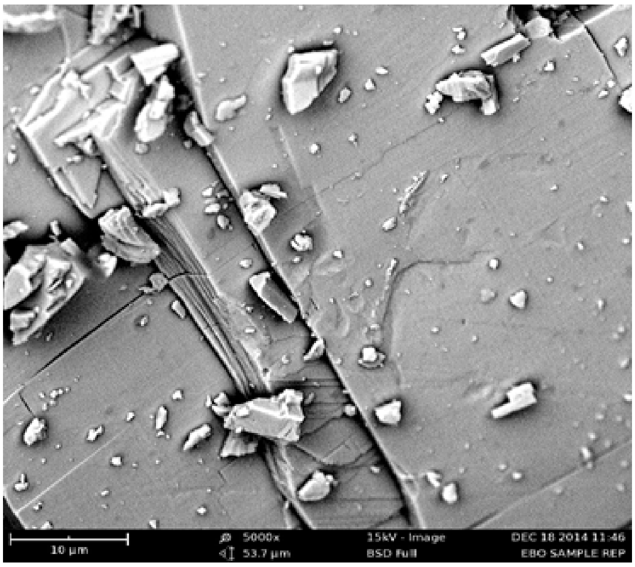


Plate 4.3: scanning electron micrograph showing some level of failure (cracks) with identifiable strata/layers indicating brittleness of Ebonyi sample. Photomicrograph taken at the bar-scale of 10µm with magnification of 5000x.

5.0 Conclusion

It is imperative that the key to successful beneficiation of an ore is greatly attached to the amount of information available on the nature and properties of the various components making up the ore. The work sample confirmed to be galena-rich sulfides cognizant to the results obtained from all analyses carried out on Enyigba Lead ore. It can be conclusively stated that since these sulfides contain toxic contaminants like Pb, Cd, As, Sb etc., their large-scale mining and beneficiation operations would warrant systematic approach following environmental stipulations.

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Mill Sizing and Selection in the Grinding Circuit of Azara Copper Ore

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Abstract

Comminution processes constitute a significant proportion of capital and operating costs in mineral processing plants and are critical to the economic performance of most mining operations. Indeed, in general, power is the most critical item in any comminution circuit owing to the fact that the comminution circuit is well known to be the major power consumer in any mineral processing plant. To be energy efficient, a circuit needs not only to have the right equipment but also have that equipment optimized. The mill sizing and selection of appropriate equipment in the grinding circuit for Azara copper ore are designed to ensure effective particle size and cost reduction as relating to energy estimated from the plant operating and mill grinding parameters. The work index was used and the result obtained after the selection and calculations of all factors showed that throughput was 60tph, specific energy consumption (E) was 16.9064 kW/tonne, net mill power demand was 1017.624kW. The grinding efficiency with increasing mill diameter was 3.8m inside the liners, the optimum feed size was found to be 4001.7microns and the power consumption (W) indicated 1045.238kW.

Keywords: mill, sizing, selection, factors, reduction, grinding.

Introduction

With the increase in demand of copper and its alloy, it has become necessary to explore and exploit low grade copper deposits. Nigeria is endowed with many low-grade copper deposits with copper minerals in association with other valuable minerals. In order to enjoy and fully utilise Azara copper ore, proper design of the grinding circuit with respect to the mill sizing and selection is paramount. This in turn will help in reducing comminution cost which usually takes about 60% of the cost of power consumed in ore processing (Fuerstenau and Han, 2003).

In mineral processing, the design of grinding circuit is very important to the efficient operation of the process because it provides information on the mill sizing and selection of adequate equipment required. Thus, it is very important for a mineral engineer to be thoroughly furnished with the various operations involved in liberation of the ore and processes to select the required equipment (Fuerstenau and Han, 2003; Mills, 2014). The selection of the types and sizes of equipment for mineral processing plant needs adequate experience and care to ensure overall cost reduction, particularly when the process is partially or completely new. If the process is an established one or in operation elsewhere or it is the case of expansion of identical unit processes, then the task mainly consists of comparative calculations, scaling the equipment and accessories up or down. The conventional mills such as rod and ball mills are sized based on the Bond's work index obtained from the grindability test of the ore. To achieve this, it is therefore imperative that as a first step, a competency test of the ore is made to determine the rock competence.

All equipment has a service life, after which it becomes uneconomical and should be replaced. For some items the service life may be equal to the mine life, in which case replacement is unnecessary. Generally, major items such as headframes, winders (hoist), crushers, ball mills and so on are designed to last the life of the mine and in practice, may outlast several mines (Weiss, 1985).

Also, a mineral processing plant typically involves a large number of unit operations within the comminution unit. Each of these units targets the reduction of ore to achieve liberation size. The comminution system must be designed to ensure that liberation is achieved and prevent waste of energy (Metso Minerals, 2006). The main design factors to consider when mill sizing and selecting of grinding circuit are as follows:

- Mill dimension
- Type of mill
- Mill speed
- Mill loading
- Required particle size (final product)
- Feed size
- Work index of the ore
- Mill shaft power
- Specific gravity of the ore

Furthermore, the determination of the work index of an ore is crucial in determining the mill sizing and selection of equipment. It was found out that the Azara copper ore contains 3.3% copper (Oyeladun et al, 2015). According to Oyeladun et al (2015), the Azara chalcopyrite copper ore can be described as having hardness of 3.5 using Mohs hardness scale, the specific gravity of 4.40, the Work Index of 12.989kWh/tonne and it is graded as type B with soft/medium texture.

Once the Work Index has been determined, the power requirement and mill size can be calculated from Bond's equation which is expressed as follows:

$$W = 10W_i\left(\frac{1}{\sqrt{P_{80}}} - \frac{1}{\sqrt{F_{80}}}\right)$$

Where W is power consumption in kWh/ton of feed, F_{80} **=** diameter in microns which 80% of the feed passes, P_{80} **=** the diameter in microns which 80% of the product passes (Wills and Napier-Munn, 2006). This calculation gives the power at standard reference points:

- Rod milling: wet open-circuit grinding in a 2.4m diameter (inside liners) rod mill
- Ball milling: wet closed-circuit grinding in a 2.4m diameter (inside liners) ball mill
- The calculated power is the power required at the pinion shaft of the mill and includes mill trunnion bearing and gear and pinion losses, but does not include motor losses or losses in other drive component reducers and clutches.

In studying plant mill performance, a number of factors have been found that cause the work indices calculated from the plant operating data to vary from the work indices obtained from Bond grindability tests. These factors are as follows:

CF$_1$: Dry grinding
CF$_2$: Open-circuit ball milling
CF$_3$: Mill diameter
CF$_4$: Oversized feed
CF$_5$: Grinds finer than 80% passing 74 microns (200 mesh)
CF$_6$: Too small or too large reduction ratio, rod mill
CF$_7$: Too small a reduction ratio, ball mill
Where CF = Correction Factor

The basic power W (i.e. Work Index) for ball mill should therefore be adjusted in accordance with correction factors (CF$_1$ to CF$_2$) as follows:

Correction Factor 1 – CF$_1$(dry grinding) = 1.3

Correction Factor 2 – CF$_2$ (open – circuit ball milling) see values in Table 1

Correction Factor 3 – CF$_3$ (mill diameter) = $(8/D_1)^{0.2}$ (or 0.914 minimum value) where D$_1$ is the mill diameter inside liners. The gain in grinding efficiency with increasing mill diameter ends at about 3.8m inside the liners. The use of diameter efficiency factor beyond this point does not give the correlation between W_{io} and W_r the diameter efficiency factor for diameter larger than 3.8m inside the liners should be the same as used for mills 3.8m inside the liners; namely, 0.914

Correction Factor 4 – CF$_2$ (Oversized feed)
Optimum Feed Size F$_o$

$$F = 4000\sqrt{(13/W_i)}$$

If F80>F,

then $CF_4 = [R_r + (W_i - 7)\times(F_{80} - F_o)/F_o]/R$

Where $R_r = F_{80}/P_{80}$

Correction Factor 5 – CF5 (fineness of grind)
If P_{80}<74µm, then CF5 = (P_{80}+10.3/(1.145×P_{80})
If P_{80}>74µm, then CF5 = 1.000

Correction Factor 6 – CF_6 (low ratio of reduction, ball milling)
Correction Factor 7 – CF_7 (low ratio of reduction, ball milling)
If $R_r < 3$,
then $CF_7 = [20(R_r - 1.35) + 2.6] / [20(R_r - 1.35)]$
If $R_r \geq 3$, then CF_7 =1.000 (Gupta and Yan, 2006)

Materials and Methods

To determine the relative hardness of any mineral based on the Moh's hardness scale, it was necessary to find which of these minerals, the unknown mineral can and cannot scratch. Minerals of known hardness from the Moh's Hardness Kits were used. The sample was scratched using the mineral from the kit continuously with harder or softer minerals from the kit until the hardness was determined.

The Modified Bond's method called Berry and Bruce method of determining the work index of an ore was used in this research work because of its simplicity and speed. The method is also called the comparative method of determining grindability parameter (Wills and Napier-Munn, 2006). This method requires the use of a reference ore of known work index (granite of known work index 15.14kWh/tonne).

Calculations

From mill dimension design;
Bond expressed mill capacity as a function of mill shaft power (P_M, kW) and energy required for particle breakage (kWh/f):
$Q = P_M/E$
Ore: Azara Copper ore
Throughput: 60tph

F_{80}: $8500\ \mu m$
P_{80}: $50\ \mu m$
E=10x12.989(1/√50 – 1/√8500) = 16.9064
P_M= 60x16.9604= 1017.624kW
The basic power P_M is corrected as follows:
Correction Factor 1 – CF_1 and Correction Factor 2 – CF_2 are not applicable.
The diameter inside the shell is 3.5m and the diameter inside the D_1 is 3.4m.

Correction Factor 3 – CF_3 (mill diameter) = $(8/D_1)^{0.2}$
Correction Factor 4 – CF_2 (Oversized feed)
Optimum Feed Size F_o

$$F_0 = 4000\sqrt{(13/W_i)} = 4000\sqrt{\left(\frac{13}{12.989}\right)}$$
$$= 4001.7$$

If $F_{80} > F_0$, $F_0 = 4001.7; R_r = \frac{8500}{50} = 170$

$$\text{then } CF_4 = \frac{\left[R_r + (W_i - 7)\times\frac{F_{80} - F_o}{F_o}\right]}{R_r}$$

$$CF_4 = \frac{\left[170 + (12.989 - 7)\times\frac{8500 - 4001.7}{4001.7}\right]}{170}$$
$$= 1.0396$$

Correction Factor 5 – CF_5 (fineness of grind)
If P_{80}<74μm, then CF_5 = (50+10.3/(1.145× 50)=1.0533
$W = 1017.64\times0.938\times1.0396\times1.0533$
$= 1045.238\ kW$

Critical Rotational Speed of the Mill

At Θ=0° and cos Θ = 1, the gravitational force pulling the media off the mill was maximized.

Mill Throughput Capacity

The expression for mill shaft power developed by Bond incorporates a number of operating parameter,
$P_M = 7.33 J_B \Phi_C (1 - 0.931 J_B) \rho_B L D^{2.3} (1 - 0.1/2^{9 - 10\Phi C})$
The energy required for breakage
i.e. $E = 10W_i\left(\frac{1}{\sqrt{P_{80}}} - \frac{1}{\sqrt{F_{80}}}\right)$

Based on a large number of observations, power draw was determined to be:
directly proportional to the mill length
a function of mill speed
a function of the total mass of the grinding media and rock charged
a function of the feed characteristic
a function of the work index of the material

Bond developed an expression of quantify mill shaft power based on data from a number of laboratory and industrial ball mill:

$$P_s = 7.33 J_B \Phi_C (1 - 0.931 J_B) \rho_B L D^{2.3} (1 - 0.1/2^{9 - 10\Phi_C})$$

P_s= Mill Power
D = Mill diameter in meters
L = Mill length in meters
ρ_B = Ball density in t/m^3

Result and Discussion

From the mill size tables, the power requirement for an overflow ball mill corresponds to a MQG 3645, Diameter 3600cm, Length of mill = 4500cm and Speed =17.3.

In single – stage, run – of – mine milling closed – circuit with hydrocyclone classification is the simplest form of comminution.

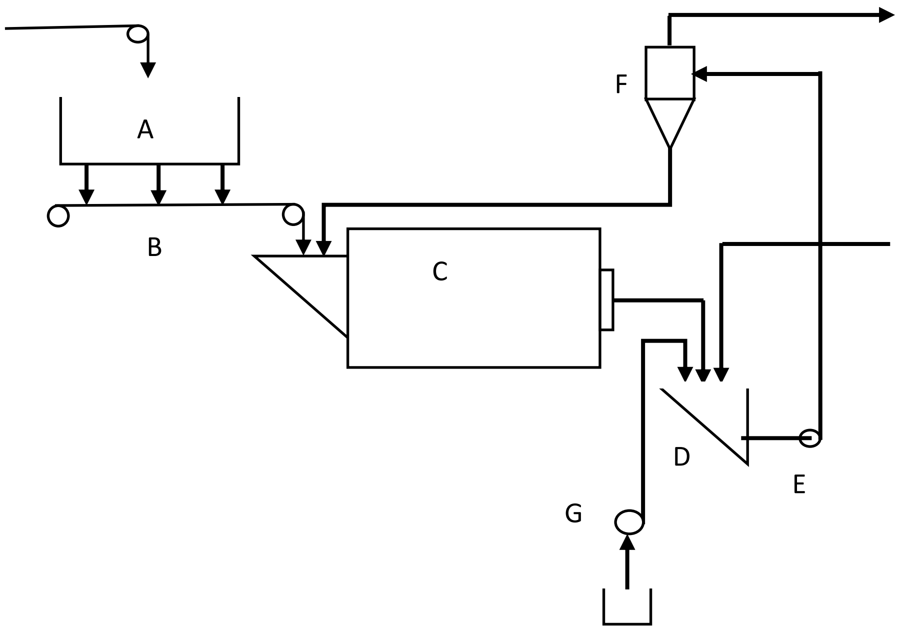

Figure 1: Run – of – mine milling
A; Silo, run – of – mine mill feed storage; B: Conveyor, mill feed; C: Ball mill; D: Sump, mill discharge; E: Pump, mill circuit; F: Hydrocyclone, mill circuit; G: Spillage pump

Conclusion

Mill sizing and selection in the grinding circuit of Azara copper ore was carried out and it was concluded that at a throughput of 60tph, specific energy consumption (E) required was 16.9064 kW/tonne and net mill power demand was 1017.624kW. The grinding efficiency with final mill diameter was 3.8m inside the liners, the optimum feed size was found to be 4001.7microns and the power consumption (W) indicated 1045.238kW.

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Table 1: Open–circuit Ball Milling Correction Factor

Reference % Passing	Correction Factors 2 (CF_2)
50	1.035
60	1.05
70	1.10
80	1.20
90	1.40
92	1.46
95	1.57
98	1.70

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Determination of Optimum Grind Time for the Comminution of Anka Copper Ore using Ball Milling Method

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Abstract

The purpose of this research work was to determine the optimum grind time for the comminution of Anka Copper Ore using ball milling method Systematic sampling was employed where copper ore samples were collected from six different points from the site. Grinding time was carried out for 10 to 30 minutes at an interval of 5 minutes. Thereafter, channel analysis was carried out to determine the effect of increased residence time on the grinding performance. However, 25minutes grind time was the optimum mesh of grind with corresponding copper grade of 1.49%. It is recommended that the optimum mesh of grind should be 25 minutes and test works should be carried out t a decreased grind time.

Keywords: Optimum, Comminution and Grinding.

INTRODUCTION

The importance of comminution in any mineral processing facility is often misunderstood or, at the very least, not fully appreciated. Grinding, because it is normally the final stage of this comminution, takes on a special importance and is often described as the key to successful plant operation and production.

The main purpose is liberating minerals for economic recovery. With metal prices down, long term challenges of declining ore grades and increasing ore hardness, and climate change causing new constraints and costs on energy, the mining industry needs to stay focused on minimizing energy use in metal production, including comminution .

Optimal comminution time use is generally determined by economics, while operating within acceptable environmental boundaries (e.g. safe tailings disposal practice). This requires understanding relationships between crushing, grinding and subsequent separation. In flotation, recovery of the valuable mineral generally falls in the coarser particle size ranges which results in lower recovery at coarser grinds

Copper is obtained from its ore by series of metallurgical operation. Copper is widely distributed and it is also formed in the native state. The marble copper has a specific gravity of about 3.9 and melts at about 1,100°C. It is comparatively soft but extremely tough metal, very ductile and malleable when pure and next to silver the best conductor of electricity.

Copper is a major raw material for the power (electricity) industry among many of it uses. But copper cannot be used directly for such purpose in its ore form. It has to be mined and undergo comminution (crushing and grinding) before it can be used for both industrial and commercial purpose. Copper ore is finely and intimately associated with gangue and must be initially liberated, before separation can be done. Grinding serves the purpose of increasing the surface area of a solid, manufacturing a solid with a desired grain size and pulping of resources.

In general, grinding process require a relatively large amount of energy. For this reason, it is necessary to determine the optimal grinding time of copper ore so as to conserve energy and resources during the comminution process hence the research study which is a separation process in the processing of copper ore using ball mill.

To optimise grind, both revenue and operating costs need to be considered. Recovery will increase as grind size decreases, and operating costs (power, grinding media) will also increase. Incremental revenue (through head grade, metal price) needs to be compared with incremental operating costs (through power and media unit costs and specific consumption rates). For low metal prices and/or low head grades, the optimum will shift towards coarser grinds and lower operating costs. Sites that filter tailings also favour coarser grinds, as these will increase throughput capacity and lower specific power consumption in filtration. In most concentrators, a low-moderate grade rougher-scavenger concentrate is produced that is treated in a regrind and cleaner circuit to make final concentrate. The grind sizes in the primary-secondary-tertiary and regrind circuits, and recoveries in the rougher-scavenger and cleaner blocks can be determined for an overall economic optimum (Wills, 2006).

Grinding

This is the last stage in the process of comminution where particles are reduced in size by a combination of impact and attrition either wet or dry. It is performed in rotating cylindrical vessels mainly made of steel and are called tumbling vessels. These comprises of loose grinding media, which are free to move inside the mill, thus grinding the particles. The grinding media may be steel rods or balls hard rock or in some cases the ore itself (Wills, 2006).

Naturally, most minerals are finely disseminated and intimately associated with the gangue. Before separation can be undertaken, the mineral must be initially “unlocked or liberated”. While, this is achieved by crushing and grinding, and to be specific grinding prior to flotation size reduction is up to some extent. It has been observed that grinding should be done at the optimum mesh of grind which is the particle size at which there is maximum profitability (Deelder, 1996).

Grinding is the most energy intensive operation in mineral processing and accounts for about 50% of the concentrators' energy consumption. Therefore, over grinding and under grinding should be avoided to reduce difficulties faced in the flotation of slimes and coarse material respectively. Hence, there is a lower and upper size limit for the particles to be floated. Size reduction to a relatively coarse size does not liberate the mineral particle, flotation recoveries in this case will be poor as the particle size will not be small enough to be lifted by the air bubbles during flotation (Flavel,1978).

Grinding with a tumbling mill is influenced by a variety of factors which includes speed of the mill load of grinding media and material, pulp level, size distribution of grinding media and solid-liquid ratio and circulating load.

An optimum mesh of grind is important for several reasons, with the most basic being achievement of liberation of the mineral species contained in the ore. If the mill is not grinding fine enough, or under grinding the ore, it produces a product which is coarse and may have a low degree of liberation, which will result in poor recoveries, poor concentration ratios and uneconomical operation. Other factors which contribute to the importance of this optimum mesh of grind are consideration of downstream processing and possible customer size specifications imposed on the product (Klimpel, 1998).

Speed of Mill

The speed must not be too high because this brings about centrifuging of the charge and this grinding does not take place. At the same time the speed must not be too low because this will promote the production of slimes. An action called cateacting action must result from the speed of the mill accompanying a free fall of grind media for favourable impact giving a high capacity and efficiency.

Load of Grind Media and Material

Increasing these will require more power until the maximum value is achieved and the power requirements decreases with increasing load as the centre of the load approaches the axis of rotation.

Pulp Level

This will require high power if the pulp level is high. The lower pulp level entails greater freedom of movement of grinding media hence more effective grinding. Therefore, the ball mills should be filled slightly more than half full with the materials and balls.

Circulating Load

In order to achieve grinding to size in a single step might result in wasteful over grinding. To avoid over grinding and giving an effective grinding, the residence time for material to be in the mill must be short. Generally, increased circulating load results due to the average size of entering feed becoming lower, interstitial loading is improved with closed adjustment of solid-liquid ratio.

In practice, ores are ground to an optimum mesh of grind determined by laboratory and pilot scale test work to produce an economic "degree of liberation" which is the percentage of the mineral occurring as the particle in the ore in relation to the total content. This can be high if there are weak boundaries between mineral and gangue particles. The optimum mesh of grind is the particle size at which the most economic recovery can be obtained. This depends not only on the grindability which is the case with which the ore can be comminuted but also the ease with which the comminuted ore can be floated (Gupta, 2006)

Materials and methods

Sample Preparation

The technique employed during sampling was called a systematic sampling. Copper ore samples collected from six different points each feeding the ball mills. Samples were taken to the laboratory for crushing and homogenization of the ore was done manually before grinding.

For this work, the Anka required sample was obtained from Anka Local Government Zamfara State. The representative sample was crushed using the jaw crusher to prepare a feed for the grinding process. The grinding process was carried out in the rod mill. The ground product was fed into the sieve shaker and sieve into different sizes and analyses to determine the liberation size of the one. Grinding time evaluation was carried out at the following times 10, 15, 20 25 and 30 minute for every 500g respectively and their size distributions are shown in Figure 2. After grinding the ball mill is charge was analyses to determine the percentage of copper present in each.

Results and Discussion

The chemical composition of the ore

Table 1: Chemical Analysis of Anka Copper Ore (Head Sample)

Element	Cu	Fe	Pb	Ca	Mn	Ti	Zr	Sr	W
Composition (%)	1.67	4.0	2.51	0.62	0.3	0.021	0.03	0.02	0.04

Table 2: Sieve Analysis Result

Sieve fraction(μm)	Weight retained (g)	Weight retained (%)	Nominal aperture size (μm)	Cumulative over size	Cumulative under size
+355μm	65.5	14.55	355	14.55	85.45
-355+250μm	30.7	6.82	250	21.37	78.63
-250+180μm	40.8	9.07	180	30.44	69.56
-180+125μm	68.8	15.28	125	45.72	54.28
-125+90μm	99.6	22.13	90	67.85	32.15
-90+63μm	60.5	13.44	63	81.29	18.71
-63	84.1	18.69		100	
	450	100			

Table 3: Chemical Composition of Size Fraction of Anka Copper Ore

Element	+355µm	+250µm	+180µm	+125µm	+90µm	+63µm
	%	%	%	%	%	%
Fe	1.66	1.27	1.33	2.23	2.25	2.08
Cu	**0.87**	**0.80**	**0.69**	**1.24**	**1.20**	**0.92**
Pb	0.34	0.41	0.54	0.96	1.11	1.19
Ca	0.27	0.32	0.80	0.91	1.02	0.85
Ti	0.06	0.11	0.16	0.22	0.18	0.20
Cr	0.05	-	-	-	-	-
Sb	0.02	0.02	0.01	0.02	0.02	-
Mn	0.02	0.01	0.02	0.02	0.02	0.02
Sr	0.01	0.01	0.02	0.02	0.02	0.02
Zr	0.008	0.01	0.01	0.02	0.002	0.02
Zn	0.003	0.003	0.004	0.006	0.005	0.008
Mo	0.002	0.002	0.003	0.004	0.004	0.005
Nb	_	0.002	-	0.003	0.003	0.003

Table 4: Result of Comminution Investigation Study of Anka Copper Ore (XRF ANALYSIS)

	10min		15min		20min		25min		30min	
Element	Ppm	%	ppm	%	ppm	%	ppm	%	ppm	%
Fe	43177	4.32	36224	3.62	37483	3.75	38048	3.80	33404	3.34
Ti	4283	0.43	2552	0.26	2618	0.26			2051	0.21
Zr	387	0.04	364	0.04	362	0.04	379	0.04	381	0.04
Nb	86	0.009	26	0.003	_	_	_	_	30	0.003
Ca	7218	0.72	6014	0.60	6371	0.64	6808	0.68	5872	0.59
Mn	607	0.06	416	0.40	304	0.03	320	0.03	251	0.03
Cu	14873	**1.49**	12518	**1.25**	13491	**1.35**	13362	**1.34**	12044	**1.20**
Pb	29922	2.99	2583	2.59	27204	2.75	28675	2.87	25031	2.50
Bi	_	_	53	0.005	49	0.005	_	_	45	0.005
Sr	279	0.03	238	0.02	258	0.03	281	0.03	244	0.02

Table 5: Grind time Versus Percentage Assay

S/NO	Grind time (minutes)	Assay (%)
1	10	1.20
2	15	1.25
3	20	1.35
4	25	1.49
5	30	1.20

DISCUSSION

Table 1 revealed the result of XRF analysis of head sample which indicates the percentages of the following elements 4.00% Fe, 0.03% Mn, 0.62% Ca,0.21% Ti,0.02% Sb,1.67% Cu,and 0.03% Zr respectively. This revealed that Anka copper ore attained the cut-off grade for copper exploitation (more that 0.5% metal content).

Table 2 shows the sieve fractions of the ground ore. The sieve fractions on 355µm, 180µm, 125µm, 90µm, and 63µm respectively were 65.5g, 30.7g, 40.8g, 68.8g 99.6g, and while -63µm was 84.1g respectively. The 80% passing was obtained graphically to be 184µm.

Table 3 shows that for the assay at +355µm, the metal content was 0.87%, at +250µm 0.80%, at 180µm 0.69%, at +125µm 1.24%, at +90µm 1, 20% and at +63µm 0,92% respectively. From the result obtained, +125µm gave the highest assay of 0.68 %. According to literature, the liberation size of Anka copper ore was established to be +125µm. Therefore, to liberate the copper value form it associated waste or gangue, Anka copper ore need to be liberated to less than +125µm (-125µm).

Generally, copper recovery increased with increase in grind time up to 25 minutes of grinding with a corresponding grade of 1.49%. However, beyond 25minutes grind time, copper grade begins to decrease with increase grind time and it also occurred at 30minutes grind time, with a grade of 1.26%. This result in high cost of energy consumption hence slimes production which has a deleterious effect on the concentration performance. The 25minutes grind time gave the highest grade indicating that the degree of liberation is highest at this time having a grade of 1.49%.

CONCLUSION AND RECOMMENDATION

CONCLUSION

The result of XRF (X-ray fluorescence) analysis on the head sample reveals that copper ore contained 1.67% of copper.

From the result of Comminution carried out on the sample using different grind times the optimum or economic grind time for Anka copper ore was found to be 25 minutes

RECOMMENDATION

Since the optimum grind time is dependent on the speed of the mill, amount and size distribution of the grinding media, they must be a consistence in replacing the grinding media in order to improve the efficiency of grind and also on the reduction of power consumption.

Mineralogical examination should be carried out on the Anka copper ore. This will aid not only in the positive identification of mineral but also the behaviour of the same constituent minerals or how they will respond to ore treatment.

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Chemical and Mineralogical Analysis of Maru Copper Ore

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Abstract
The chemical and mineralogical analysis of Maru copper ore was carried out by chemical analysis using X-ray fluorescence, X-ray diffractometer and thin sections under petrological microscope. Chemical analysis showed that the head sample (10g) contains 2.43% Cu, 45.31% Si, 3.08% Fe, 3.46% K and 2.42% K with 0.025% Ag, and 0.086% Au. Mg of 3.46%, Manganese at 0.035%, (0.33%), Sulphur (0.15%) and Chromium of 0.0087%). The X-ray diffractograph indicated the presence of Cuprite (Cu_2O 2.2%) as the copper variety with quartz (47%) and albite (23.1%) as the major impurities in the ore. The petrographic studies of the thin section equally confirmed the presence of feldspar and quarts present in the ore (state major and trace minerals observed). Further investigation was recommended on the deposit as the copper content showed a lot of prospect having surpassed the minimum economic grade of 0.5% Cu element in an ore.

Keywords: Copper, Maru, Cuprite, Diffractometer, Thin-section.

Introduction

The task of a Minerals Engineer is to characterize each deposit quantitatively and systematically and then to select the economically optimum combination of process steps to suit the characteristics.

Characterization of mineral deposits is the first step in the exploitation of any mineral endowment. This will usually involves some distinct steps stated below as reported by Thomas (2008)

1. Chemical analysis of the ore: This is to establish the chemical composition of the ore and also confirm the result of geological investigation.
2. Size/assay analysis: This is to establish the distribution of both valuable and gangue minerals in the various size fractions.
3. Mineralogical characteristics determination of the ore: Is carried with the aim of establishing the major, minor and trace minerals in the ore, the grain size of each mineral in the ore and probably the degree of association of the valuable and gangue minerals.
4. Liberation studies: This is to establish the liberation size of the individual mineral in the ore, with emphasis is placed on the grain size of the valuable minerals and also the gangue which may likely affect the subsequent separation process.
5. Determination of the work index: This is carried out to determine the energy required for the comminution of the ore from a certain size to any size and for selection of the appropriate comminution equipment for the ore.
6. Concentration tests: This is carried out to determine the grade and quantity that the valuable mineral can be recovered into the concentrate and at the same time into the gangue. The method of concentration test to be used in the beneficiation of the ore will depend on the information gathered from 1 to 5 above.

The above fact makes chemical and mineralogical analyses of Maru copper ore a very important step in the development and eventual exploitation of the deposit.

Nigeria has been established to be endowed with mineral resources that are metallic, non-metallic, industrial, as well as energy generation varieties (Anon., 2012). However, the nature of these minerals type in a deposition ought to be established in order to facilitate the downstream enrichment processes. These processes are no doubt relative to the nature of the composition, for example copper's existence aschalcopyrite ($CuFeS_2$), chalcocite (Cu_2S), covellite (CuS), malachite ($CuCO_3.Cu(OH)_2$), cuprite (Cu_2O), etc. is a typical case as reported in (Usaini *et al*, (2014).

For most metalliferrous ore produced by mining operations, beneficiation is an important intermediate step in the transformation of natural ore to pure metal (Craig and Vaughan, 1981). Hence, it is imperative that the key to successful beneficiation of an ore is greatly attached to the amount of information available on the nature and properties of the various components making up the ore. Cognisant of the fact that achieving the aforementioned stepis usually relative to the chemical and mineralogical studies of anore; this work was conducted with the purposeof investigating such parameters of Akiri copper ore as demand copper concentrate is in the increase. The justification by Yue (2009) indicated that, the sharp increase in copper consumption in the world due to the rapid development of Chinese economy, make it one of the most important materials that are widely used in industries. Furthermore, the global demand-growth rate is estimated at 3.3% per year as reported in ~~(~~Alvarado, (1999).

Materials and Methods

Field Sampling

Representative samples were obtained from Karakai village of Maru Local Government Area of Zamfara State at three random points located on latitude N 12 22 53.5, E 6 29 11.3 and Elevation of 1392ft. Grab method of sampling was adopted to obtain the sample at an interval of 20m apart to be able to have a good representative.

Chemical Analysis

Energy Dispersive x-ray fluorescence (EDXRF) spectrometer of model “Minipal 4” was used for the analysis. The samples were pulverized (ground to fine powder) using agate pulverizing machine (planetary micro mill pulverisette 7). The ground samples were ensured to pass 150 micro mesh sieves. This was to ensure homogeneity of the samples. The powdered samples were them packaged and labeled, ready for the analysis.

Twenty (20) gram of the prepared sample were weighed each into a sample cup. The cups with the contents were carefully placed in their respective measuring positions on a sample changer of the machine. The following condition sets were made as the machine was switched on:

- Elemental composition determination
- Nature of the samples to analyzed as loose powder
- The current used was 14kv for major oxides, 20kv for the trace elements/rare earth metals.
- Selected filters were used for major oxides, Ag/Al-thin for the trace elements/rare earth metals. The selection of filters was guided by a given periodic table used for elemental analysis. Time of measurement for each sample was 100 seconds and the medium used was air throughout. The machine was then calibrated by the machines gain control, after which the respective samples were measured by clicking the respective positions of the sample changer.

Loss on ignition (LOI) was determined gravimetrically by heating 1g of the powdered sample in a cleaned weighed crucible at 1000^{o}c. After which the crucible and the content was weighed to get the difference in weight before and after heating.

LOI = (a-b/1) x 100%

Wherea = weight of crucible + 1g of the sample before heating

B = weight of crucible + 1g of the sample after heating.

Phase Determination of the ore

1. The analyzed material was finely ground, homogenized, and average bulk composition is determined. The powdered sample was then prepared using the sample preparation block and compressed in the flat sample holder to create a flat, smooth surface that was later mounted on the sample stage in the XRD cabinet.
2. The sample was analyzed using the reflection-transmission spinner stage using the Theta-Theta settings. Two-Theta starting position was 4 degrees and ends at 75 degrees with a two-theta step of 0.026261at 8.67 seconds per step. Tube current was

40mA and the tension was 45VA. A Programmable Divergent Slit was used with a 5mm Width Mask and the Gonio Scan was used.

4. The intensity of diffracted X-rays is continuously recorded as the sample and detector rotate through their respective angles. A peak in intensity occurs when the mineral contains lattice planes with d-spacings appropriate to diffract X-rays at that value of θ. Although each peak consists of two separate reflections ($K\alpha_1$ and $K\alpha_2$), at small values of 2θ the peak locations overlap with $K\alpha_2$ appearing as a hump on the side of $K\alpha_1$. Greater separation occurs at higher values of θ. Typically these combined peaks are treated as one. The 2λ position of the diffraction peak is typically measured as the center of the peak at 80% peak height.
5. Results are commonly presented as peak positions at 2θ and X-ray counts (intensity) in the form of a table or an x-y plot (shown above). Intensity (*I*) is either reported as peak height intensity, that intensity above background, or as integrated intensity, the area under the peak. The relative intensity is recorded as the ratio of the peak intensity to that of the most intense peak (*relative intensity* = $I/I_1 \times 100$).
6. The d-spacing of each peak is then obtained by solution of the Bragg equation for the appropriate value of λ. Once all d-spacings have been determined, automated search/match routines compare the *d*s of the unknown to those of known materials. Because each mineral has a unique set of d-spacings, matching these d-spacings provides an identification of the unknown sample. A systematic procedure is used by ordering the d-spacings in terms of their intensity beginning with the most intense peak. Files of d-spacings for hundreds of thousands of inorganic compounds are available from the International Centre for Diffraction Data as the Powder Diffraction File (PDF). The peaks obtained from these analyses were matched with the minerals phases from the PDF 2 ICDD database which is attached to the XRD processing software, XPert High score Plus also from Panalytical. National Geological Survey Agency of Nigeria, Kaduna as well as National Steel Raw Materials Exploration Agency, Malali, Kaduna.

Mineralogical Analysis

Mineralogical investigations of ore thin sections were conducted with petrological microscope under plane polarized light and cross polarized light. The aim was for the identification of different constituents of the copper ore to reveal the major and minor constituting minerals. The following procedures were adopted in the preparation of the thin section in accordance with (Hibbard, 2002, and Gribble, 2004).

1. The ore specimens were cut using an electric cutting machine to a small thickness of about 2mm to 3mm to avoid unnecessary grinding.
2. After cutting, the sample was shaped rectangular on a rotating plate lubricated with water.
3. A hot plate was used to mount the sample on a glass slide. The sample was placed on the hot plate with the polished surface upward. Canada Balsam which melts between 100 ^{0}c-120 ^{0}c was placed on the glass slide, the slide , with the help of pair of tweezers was lifted and mounted over the rock chip gently from one end to get rid of excess Canada Balsam and expelled air bubbles which, could hinder proper observation if present. The mounted rock was allowed to cool.
4. Grinding followed mounting which reduced the thickness of the sample

to about 30 microns. The grinding was done in gradual stages, using silicon carbide, at each stage, using different grades of the chemical, of 120, which was coarsest and finished off with a grade of 1,000 which was the finest. After each stage, washing, drying and examined under the petrological microscope to note the progress.

5. After satisfying that the thin section is perfectly thin, covering of the sample was done, with a very thin glass cover. An appropriate size was laid down on top of the section and Canada balsam was poured. Again tweezers was used to pick the cover by one end turn over, with the edge first, gradually lowered on the rock section. After cooling, the remaining Canada balsam was scrapped along the edges and washed thoroughly with methylated spirit and then water.

Results and Discussion

The results of the chemical and mineralogical tests carried out on the sample of the ore were presented in plates 1a to 1d, figure 1 and table 1 & 2.

Chemical analysis using X-ray fluorescence machine was conducted on the sample of the ore which comprehensively revealed all the constituting elements as shown in table 2.

A prepared thin-section was viewed under petrological microscope and the following features were seen: The sections occur in dendritic form and host mineral is haematite, banded into lamellar.

Petrographs from thin sections of Maru ore

Plate 1a to d are the petrographs obtained from the prepared thin sections of Maru ore observed under the petrographic microscope.

Petrograph of the ore under plane polarized light (plate 1a) showing microcline as colourless to cloudy in appearance traversed by opaque mineral in veuns, with quartz as colourless less relief anhedral in shape and inclusions in the microcline. Plate 1b was viewed under cross polarized light depicting reddish dark to greenish dark in colour of copper and microcline with colourless to grey quartz inclusion. Plate 1c is another view under plane polarized light showing colourlessanhedral quartz with inclusion of muscovite and an opaque mineral and a cloudy to colourless feldspar. Plate 1d is the view of the thin section under cross polarized light showing copper as reddish dark and greenish dark and colourless grey; quartz, and grey white feldspar.

Table 1: Mineralogical composition of Maru ore using X-ray diffractometer (XRD)

Valuable Mineral	Other mineral	Major Gangue mineral
Cuprite (Cu_2O)	Feldspar/Albite ($KAlSi_3O_8$)	Quartz (SiO_2)

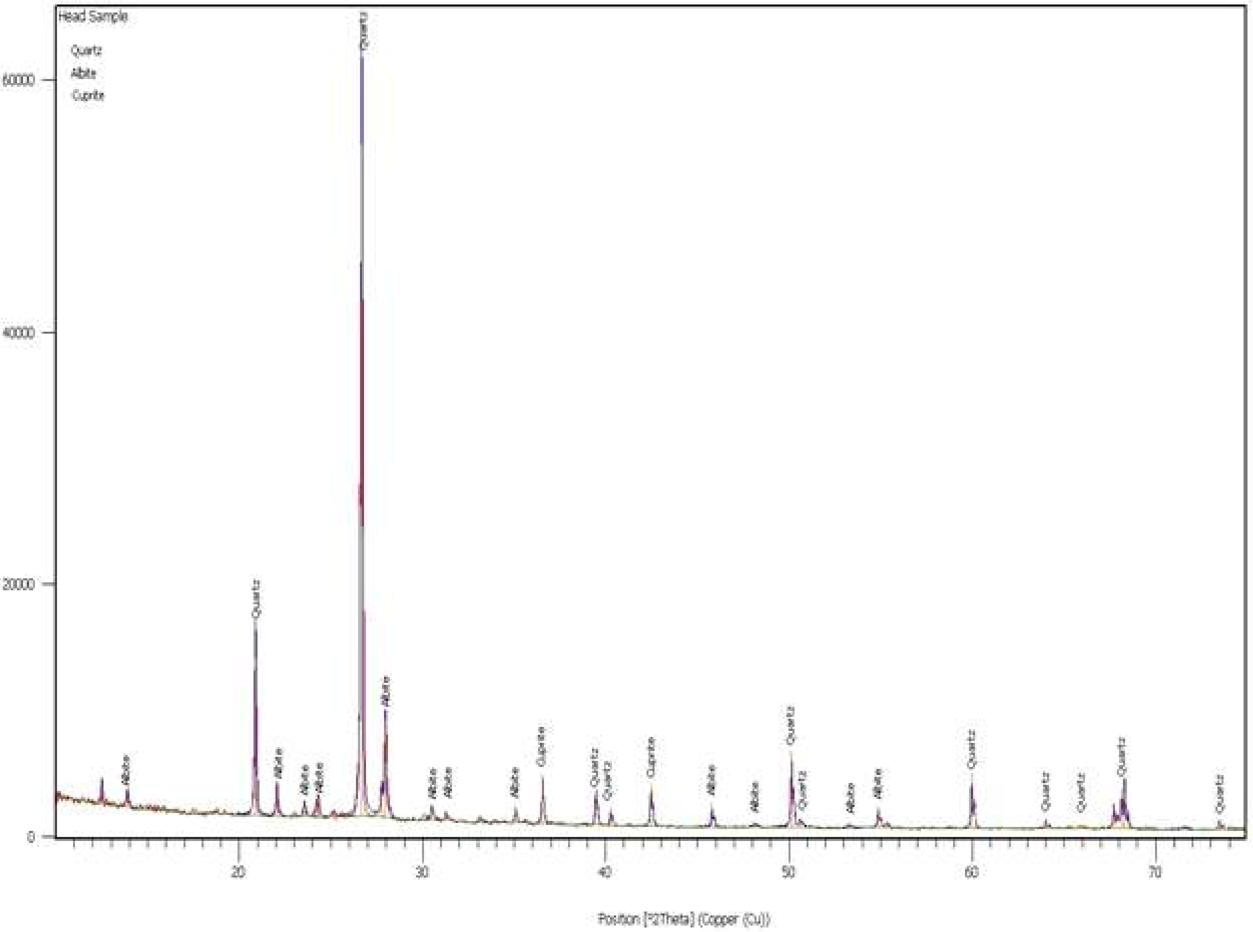

Figure 1: Peak matched diffractograph of maru ore

The X-ray diffractometer result as shown in table 1 and figure 1 (peak matched diffractograph), revealed the presence of three major components. These are Cuprite, (Cu_2O), as the valuable mineral, quartz (SiO_2) and albite, ($NaAlSi_3O_8$) as major and minor mineral gangue phases. The XRD results has agreed significantly with the results of thin section which established the presence of plagioclase feldsapar, (albite), opaque minerals; (copper mineral) and large quantity of quartz, as corroborated from the peak denoting quartz on the diffractograph.The head sample was obtained after conducting the laboratory assay sample by putting the entire sample from different pits together, mixed properly and a representative sample generated as head sample.

Table 2: Chemical Analysis of the Head Sample of the Ore

S/No.	Element	% Composition
1	Cu	2.4337
2	Al	5.8595
3	Si	45.5930
4	Ag	0.0057
5	Au	0.0867
6	K	3.4623
7	Fe	3.0808
8	P	0.1021
9	S	0.1550
10	Mg	3.4623
11	Ca	0.2463
12	Ti	0.0000
13	V	0.0127
14	Cr	0.0087
15	Mn	0.0356
16	Co	0.0168
17	Ni	0.0000
18	Zn	0.1691
19	As	0.0257
20	Pb	0.1530
21	W	0.0447
22	Rb	0.2510

The head sample was obtained after conducting the laboratory assay sample by putting the entire sample from different pits together, mixed properly and a representative sample generated as head sample.

Tables 2, presents the results of chemical analysis of the head sample of the copper ore. The results revealed that the composition of copper element to be 2.43%, with about 39% Fe and a variety of other elements including, Aluminium (1.7%), Silicon (6.11%), Sulphur (13.6%), Nickel (14%), Molybdenum (3.1%) and a host of other minor constituents of less than 1% each. The percentage composition of copper in the head sample of 2.43% assay grade gave a very good copper composition to technically warrant exploitation according to Wills (2006), which gave 0.5%-2% as a good grade for the feed to copper extraction.

Conclusions

The research study had established that:

- The Copper Ore is an oxide copper – Cuprite (Cu_2O) and contains albite, and quartz as the major impurities.
- The elemental composition of Copper in the sample is 2.43% Cu showing very good prospect for the development and subsequent extraction of the copper metal.

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Dephosphorization of Koton-Karfe Iron Ore by Leaching with Citric and Hydrochloric Acids.

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Abstract

Dephosphorization of Koton-karfe iron ore Kogi State Nigeria has been studied. Hydrochloric and citric acids were used to leach -355 +250μm and -180 +125μm size fractions. The analysis was carried out using X – Ray fluorescence Spectrometer (XRF) and Inductively Coupled plasma optical emission spectroscopy(ICP-OES). The leaching was conducted under varying time, concentration, agitation and temperature. The optimum leaching condition developed for hydrochloric and Citric acid were: 120 and 20 minutes, 2.0 and 0.5M concentration, 300 and 400rpm respectively at a common temperature of 30°C. Leaching of -355 +250μm gave 33.84 and 27.27% while -180 +125μm gave 35.13 and 29.73% dephosphorization for hydrochloric and citric acid respectively. Hydrochloric acid gave the best leaching at the optimum condition. With both acids, only phosphorus in $AlPO_4$ is leached while phosphorus in solid solution with Goethite could not be leached. ICP-OES reveal minimal loss of iron to the leach liquor.

Keywords: Dephosphorization, iron ore, leaching, X – Ray fluorescence Spectrometer.

INTRODUCTION

Large deposits of iron ores have been discovered in the country making Nigeria one of the richest countries in the world as far as mineral resources are concerned, with workable iron ore reserve estimate of 2.5 billion tons as reported in MMSD (1987). Agbaja has the largest reserve in Nigeria as reported in MMSD (1987). It is closely followed by the Koton-karfe and the Bassa Nge deposit which lie in the same region as noticed in the research report by Amadi *et al*., (1983). BRGM (1983) reported that the Agbaja-Lokoja-Koton-karfe-Bassa Nge ore body is located in the Lokoja confluence ($6^0$45E; $7^0$47N), and covers an expanse of land approximately 400Km^2 in the geographical area of Nigeria now known as Kogi State. The confluence divides the deposit into three major areas: (i) The Agbaja-Mount Patti-Lokoja area (approximately 150Km^2), located west of the river Niger; (ii) The Koton-karfe area (approximately 170Km^2), located north of the confluence; and the Bassa Nge area (approximately 80Km^2), located south east of the confluence as reported by Uwadiale (1984).

MMSD (1987) reported that the percentage iron contents in Nigerian iron ores, were found to be: Itakpe (36.88%Fe, 0.18%P_2O_5) and Ajabanoko hills (37.22%Fe, 0.10%P_2O_5) between Okene and Lokoja in Kogi State and were considered suitable for iron and steel making after beneficiation because of the acceptable level of impurities such as phosphorus, sulphur, arsenics, zinc, and alkali metals, etc. Baba et. al, (2005) reported that deposits such as Koton-karfe iron ore (48.125%Fe; estimated at 428 million tons) and Agbaja iron ore (47.80%Fe; estimated at 2 Billion tons) containing high proportion of phosphorus (2.14%P_2O_5 for Koton-karfe iron ore and 2.08%P_2O_5 for Agbaja iron ore) and fine grain texture are not suitable for use in iron and steel making in Nigeria

Elements such as phosphorus (P) and sulphur (S) when contained in large amount in iron ores have detrimental effects on the steel produced as reported in Serdar, (1999). Phosphorus impacts to steel cold shortness as reported in Kudrin, (1985). Large quantity of phosphorous reduces the ductility of steel, thereby increasing the tendency of the steel to crack when cold worked as reported in Serdar, (1999). The depletion of the richer iron ore deposits as a result of advancing global technologies and civilization necessitated the processing of lower quality iron ores as reported in Jian and Sharma (2004).

The Koton-karfe iron ore deposit is found in the northern part of Koton-karfe town in Kogi State. The iron ore deposit was discovered by the geological survey of Nigeria as reported in (Famuboni, (1990). Koton-karfe iron ore deposit is mainly of the sedimentary type with uncertain basal bed thickness due to poor exposure. These basal beds are conformable over-lain by a lithology comprising mainly sandstones and subsidiary claystones, fine conglomerates and siltstones. Both beds and the underlying ores are probably about 300m thick. They are known by various local names, Bida sandstone around Bida, and Lokoja basal sandstone around Niger/Benue confluence. Massive appearance and flat beds are most common in the basal area of the sandstones in Bida area, while the upper shows widely developed large scale across stratification as reported in Thomas (2008). Famuboni, (1990) reported that the Koton-karfe iron ore contain siderite, magnetite and goethite as the major iron bearing minerals while hematite and other associated minerals are the minor constituents. The ore has low silica content, is oolitic and pisolitic in nature and formed ore of the meta-sedimentary iron ore deposit. Asuke et al.~~,~~ 2018 reported in line with Famuboni (1990) and added that the phosphorus is uniformly distributed in the iron ore in the form of berlinite ($AlPO_4$) and in solid solution with goethite.

Over the past couple of decades, there have been reports of increasing levels of phosphorus in the raw materials used in steel making as reported in Lenintov et al. (2007), due to a gradual shortage of low – phosphorus iron ores and coking coals. Thus the steel makers are facing stiffer challenges in achieving the desired phosphorus levels in the finished products and increased cost of production. Several steel plants have adopted the practice of external dephosphorization where a part of the phosphorus is removed before the main steel making process as reported in Somnath (2007).

Various attempts have been made by researchers to make possible the utilization of the high phosphorus iron ore deposits. Among such attempts is the use of Hydrometallurgy.

Williams and Cloete 2008 reported an attempt to investigate the most effective acids to remove phosphorus and potassium from Sishen iron ore concentrates by chemical leaching using a range of acids and oxidative chemicals, the acids used are, acetic acid, citric acid, ferric sulphate, hydrochloric acid, lactic acid, oxalic acid, propanoic acid and sulphuric acid. 1molar concentrations of all leaching acids were used for five days at room temperature. They reported that citric acid proved to be best leaching agent for removing potassium from the ore, while sulphuric acid was detected to be the most effective to remove phosphorus and most corrosive on iron.

Xia Weng et al.,(2011) studied the removal of phosphorus from high phosphorus iron ores by selective hydrochloric acid leaching. The ore used in their research is from Wushan, Chongquing, China. They reported that hydrochloric acid concentration is a very important parameter for dephosphorization. On the condition of high concentration of hydrochloric acid, the phosphate can convert into soluble phosphate salts easily, quickly and completely. But if the concentration of hydrochloric acid was too high it could lead to loss of iron, and the selectivity of the leaching process would be reduced. Their optimum experimental parameter were reaction time 30 – 45 minutes, particle size <0.147mm, hydrochloric acid concentration 2.5 mol/liter, reaction temperature 25^0C and stirring strength 5.02 – 12.76 s^{-1}, with these conditions the hydroxyapatite was converted into soluble phosphate during the process of HCl leaching.

Forssberg and Adolfsson (1981) investigated the economic feasibility of acid leaching of iron ore in which apatite, $Ca_5(PO_4)_3(F, Cl, OH)$, was the phosphorous containing mineral. Nitric and hydrochloric acid leaching agents were used. They reported that the economy of leaching with hydrochloric or nitric acid depend very much on the extent to which the phosphorus can be recovered as phosphoric acid.

Biological processes for phosphorus removal have also been evaluated based on the use of several fungi, some being oxalic acid producing. Anyakwo and Obot (2008) worked on the use of Aspergillus niger and their cultural filtrates for removing phosphorus from Agbaja iron ore, and concluded that phosphorus removal efficiency at the end of the 49days of the leaching are 81, 63, and 68% for 5, 100, and 250 mesh grain size respectively.

William and Cloete (2008) worked on the production and use of citric acid for the removal of phosphorous and potassium from the iron ore concentrates of the Sishen iron ore mine, South Africa. In their studies submerged fermentation of Aspergillus niger for the production of citric acid was used, and the citric acid produced used for the leaching of the iron ore. They reported that 1 molar citric acid leaching solution at 60^0C proved to be the most efficient, removing 35.29% phosphorous and 23.53% Potassium contained within the iron ore concentrate.

In the last ten years, the situation of iron ore markets has changed dramatically due to an increase on the world steel consumption, pushed up mainly by the economic growth of China and other Asian emerging markets. Currently, prices of iron ore are increasing in real terms and the production of this commodity is increasing also observed by Poveromo (2005). This new scenario for the iron mining industry has revived the interest in upgrading the quality of phosphorus bearing iron ores by hydrometallurgical routes and for example at least one beneficiation plant to leach phosphorus from iron ores has been recently installed in kazakhstan as reported in Kokal et al. (2003).

Based on these backgrounds, little or no work has been done on the leaching of Koton-karfe iron ore with acids. It is for this reason that a chemical process of reducing the phosphorus content was considered. Hence an attempt will be made in this research to reduce the high amount of phosphorus (2.14% P_2O_5) of the Koton-karfe iron ore through chemical leaching using various acids.

MATERIALS AND METHODS.

Materials and Equipment

The materials used include; Koton-karfe Iron ore sample obtained from the iron ore deposit, hydrochloric acid, citric acid, and filter papers obtainable from the Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of technology, Pretoria, South Africa. The equipment used include: Denver pulverizing machine, Denver jaw crushing machine, Denver sieve shaking machine, X – Ray fluorescence Spectrometer (XRF) in Faculty of Natural and Agricultural Science, Geology Department, University of Pretoria, Pretoria, South Africa, Inductively Coupled plasma optical emission spectroscopy(ICP-OES), Conical Flasks and Beakers, Digital Weight Scale, Drier, magnetic stirrer and a pH meter available at Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of technology, Pretoria, South Africa.

Material Collection and Preparation

The sample of the iron ore used for this research work was obtained from the Koton-karfe Iron ore deposit in Kogi State, Nigeria. 80 kg of the Samples were collected from different points on the deposit by simple random sampling according to recommended sampling for iron ore by ASTM E877. The ore was crushed and ground using the Denver crusher (JC - 300) and ball milling machine (GMO85) respectively in line with procedures in Wills and Napier-Munn (2006). The ground ore was sieved to produce particle size fractions of -355 +250µm, and -180 +125µm according to procedure reported in Asuke et al. (2018).

Determination of the chemical composition of the head Sample.

The XRF analysis was done in the Faculty of Natural and Agricultural Science, Geology Department, University of Pretoria, Pretoria, South Africa. Thermo fisher ARL9400 XP+ Sequential XRF and uniquant software was used for the XRF analysis. Before the analysis, the ore was first milled to -75µm, the samples were prepared as pressed powder briquettes as required by the machine standard for proper mineral count and analysis. Each sample was placed in the machine for one hour thirty minutes (thirty minutes for major elements and one hour for trace elements). The uniquant software (WinXRF)was used to analyze for all elements in the periodic table between sodium (Na) and uranium (U), but only elements found above the detection limits were reported. All major elements were expressed as oxides and analyzed in percentage (%) composition.

The Leaching process.

Leaching reagents used.

The acids used for this research were Hydrochloric acid (HCl) and Citric acid ($C_6H_8O_7$). All leach solutions were prepared from analytical grade of acids by dissolution of calculated amount in deionised water. Hydrochloric acid (HCl) used had 32% concentration and density 1.159g/cm^3(batch no 350418), and the Citric acid ($C_6H_8O_7.H_2O$) assay 99.7%, density 1.5g/cm^3 (batch no 322400)

Leaching Procedures:

The leaching procedures in Mills (2006), was adopted or modified for this work. The leaching procedures that was carried out in four stages were as follows:

Stage I: 40g of -355 +250µm particle size was weighed, and then transferred into 500ml conical flask containing 200ml of 0.5M hydrochloric acid at temperature of 30^0. The mixture was covered with glass lid and agitated with the aid of magnetic stirrer at an initial rate of 100rpm. The mixture was leached for a constant time of 1hr after which the solution was filtered, the residue washed thoroughly and taken for analyses. The process was repeated varying agitation speed from 200 – 500rpm at an interval of 100rpm. The above procedure was repeated for citric acid.

Stage II: From stage I the best agitation was determined for the different acids and used in this stage. The leaching in this stage was carried out at constant time (1hr), acid concentration (0.5M) for hydrochloric acid, agitation of 300rpm and an initial temperature of 30^{0}C after which the solution was filtered, the residue washed thoroughly and taken for analyses. The stage II process was repeated with varying temperature of 40 - 80^{0}C at an interval of 10^{0}C. This process was repeated for citric acid at 0.5M and 400rpm.

Stage III: With the optimum parameters developed from stages one and two for agitation and temperature respectively, the effect of the variation of concentration and time were studied at this stage. For hydrochloric acid, an initial concentration of 0.5M, was used with varying time from 0 – 120minutes at an interval of 20minutes (at 300rpm and 30^{0}C), after every 20minutes of leaching, samples were taken, filtered and the residue washed thoroughly with distilled water, dried and analyzed and the leach liquor kept for further analysis. This process was repeated with hydrochloric acid but varying concentration from 1.0 – 2.0M at an interval of 0.5M; at each concentration varying time as stated above. The whole process as described is repeated with citric acid but with agitation of 400rpm. From the above procedures the best leaching parameters were attained.

Stage IV: In this stage the effect of the variation of particle size was investigated. The optimum conditions developed from stages I - III above were used to leach another particle size (-180 +125µm size fraction).

Determination of phosphorus content before and after leaching of the ore.

The phosphorus content of the ore before and after leaching was determined using XRF analysis. The samples were prepared using the same method as earlier described.

Determination of the Amounts of Phosphorus removed during Leaching with Acids.

Reduction in the contents of phosphorus as a result of the acid-leaching process was calculated using the results from the XRF analysis in equation (3.1);

$$\boldsymbol{R_P} = \frac{\%\boldsymbol{P_B} - \%\boldsymbol{P_A}}{\%\boldsymbol{P_B}} \times 100 \qquad (3.1)$$

Where R_P = Reduction in Phosphorus Content (%), P_A, P_B = phosphorus content after and before leaching (%) respectively.

Analysis of the acids before leaching and the leached liquor.

Analysis using Inductively coupled plasma optical emission spectrometry (ICP-OES).

The acid before leaching and the leach liquor were analyzed using Inductively coupled plasma optical emission spectrometry (ICP-OES) using standard procedure as described in Asuke, 2014

pH Measurement

The pH of the leach solution before and after leaching was measured. This was carried out using the hand-held pH/ORP meter (Model No. 8651). The pH meter was calibrated before use and the probe was always rinsed in fresh de-ionized water before use in order to remove any impurity adhering to the probe.

Determination of the Amounts of Iron dissolved in acid during Leaching.

The amount of iron, aluminium and silicon lost to the leaching process was calculated using the results obtained from the ICP-OES analysis by the equation (3.2);

$$\boldsymbol{R_{Fe}} = \frac{\%\boldsymbol{Fe_B} - \%\boldsymbol{Fe_A}}{\%\boldsymbol{Fe_B}} \times 100 \qquad (3.2)$$

R_{Fe} = dissolved iron content (%), Fe_B, Fe_A = iron content before and after leaching (%) respectively,

The principle applied in equation 3.2 was also used to determine the amount of aluminium, silicon, manganese, cobalt and nickel dissolved in the various acids after leaching.

RESULTS AND DISCUSSIONS

XRF analysis of the selected particle size -355 +250μm, and -180 +125μm before leaching are presented in Table 4.1. Based on the distribution of the iron and phosphorus within the particle sizes, -355 +250μm and -180 +125μm particle sizes were selected for leaching. Particle size of -355 +250μm containing the highest amount of P_2O_5(1.98%) is selected to allow maximum exposure of phosphorus to the leaching condition, while -180 +125μm was selected to investigate the effect of particle size on the leaching with respect to the highest distribution of iron (69.81%) which is also the liberation size as reported in Thomas (2008).

Table 4.1: XRF of selected particle sizes before leaching

	Percentage Composition	
Constituent oxides	**-355 +250**	**-180 +125**
SiO_2	15.81	14.06
TiO_2	0.24	0.22
Al_2O_3	12.20	12.25
Fe_2O_3	67.73	69.81
MnO	0.45	0.43
MgO	0.47	0.39
CaO	0.35	0.22
Na_2O	*<0.01*	*<0.01*
K_2O	0.08	0.08
P_2O_5	1.98	1.85
Cr_2O_3	0.02	0.02
NiO	0.02	0.02
V_2O_5	0.07	0.07
ZrO_2	0.01	0.01
SO_3	0.15	0.17
WO_3	0.07	0.05
BaO	0.07	0.07
Cl	0.06	0.05
CuO	*<0.01*	0.01
ZnO	0.05	0.05
Co_3O_4	0.07	0.07
SrO	0.05	0.05
MoO3	0.02	0.02
RuO4	*<0.01*	*<0.01*
Rh2O3	0.01	0.01

Note: All particle sizes are in μm.

Variation of Agitation on leaching of -355 +250μm particle size.

The dried ore was analysed using XRF from where equation 3.1 was used and the result presented in Table 4.2. For Hydrochloric acid from Table 4.2, the least amount of phosphorus leached was obtained at 100rpm (8.08%) while the best phosphorus leaching (20.70%) was obtained at 300rpm, while with citric acid the phosphorus leaching of (6.57%) was least at 200rpm and the highest leaching of phosphorus (19.7%) was obtained at 400rpm. From the result, hydrochloric acid gave the best leaching rate at 300rpm and citric at 400prm. This is in line with earlier reports by Jin Yong-shi et al (2006). They reported that when the speed exceeds the optimum revolution the phosphorus extraction decreases. That is because the iron ore samples are fine ground, and they could be easily driven by the whirlpool of the leaching solution at high speed of agitator, and in this way, the relative refresh velocity of the leaching solution on the surface of the ore particles decreases.

Table 4.2: The effect of agitation on the leaching of -355 +250μm particle size with the different acids used.

	AGITATION (rpm)/ PHOSPHORUS LEACHED(%)				
ACID	100	200	300	400	500
HCl	8.08	20.2	20.7	9.07	20.7
CITRIC	7.57	6.57	6.76	19.7	19.19

Variation of Temperature on leaching of -355 +250μm particle size.

Table 4.3 presents the result for the variation of temperature on the phosphorus leaching for the acids used. From Table 4.3 the acids perform better at 30°C with phosphorus leaching of 20.71% for hydrochloric acid and 19.7% for citric acid. The phosphorus leaching drops as the temperature increases. This is in line with initial reports (Xia et. al 2011).

Table 4.3: Variation of temperature with the amount of phosphorus leached with the different acids used.

	TEMPT (°C)/ PHOSPHORUS LEACHED (%)					
ACID	30	40	50	60	70	80
HCl	20.71	3.03	14.65	2.53	2.53	1.01
CITRIC	19.7	17.68	4.04	3.03	5.56	2.53

Variation of Concentration and time on leaching of -355 +250μm particle size.

The effect of the variation of concentration and time on the leaching of -355 +250μm particle size is the third stage of the leaching. It involved the variation of time from 20 – 120minutes on an interval of 20mins and concentration from 0.5 – 2.5M concentration for Hydrochloric and Citric acid. The leaching was carried out at constant temperature of 30°C, and agitation of 300, and 400rpm for hydrochloric and citric acid respectively as earlier established. Tables 4.4 and 4.5 presents the effects of variation of time and concentration on the selective leaching of phosphorus with hydrochloric and citric acid respectively from the iron ore.

Table 4.4: Variation of Hydrochloric acid concentration and time on the leaching of Phosphorus in the -355 +250µm particle size.

CONC.	TIME(Mins)/LEACHING RATE (%)					
(M)	20	40	60	80	100	120
0.50	6.57	20.71	20.71	22.73	18.69	3.54
1.00	8.59	21.72	7.07	21.72	6.06	3.54
1.50	22.22	7.58	7.07	19.19	21.21	4.54
2.00	8.58	7.57	21.72	22.73	23.23	33.84
2.50	22.22	8.08	7.07	7.58	21.72	19.67

Table 4.5: Variation of Citric acid concentration and time on the leaching of Phosphorus in the -355 +250µm particle size.

CONC.	TIME(Mins)/LEACHING RATE (%)					
(M)	20	40	60	80	100	120
0.50	27.27	25.25	19.67	6.06	17.68	3.54
1.00	7.07	6.57	6.06	18.69	5.56	4.55
1.50	7.58	24.74	6.06	5.56	4.55	15.66
2.00	10.61	18.18	22.73	23.23	11.62	7.07
2.50	8.59	9.09	10.1	8.59	21.21	6.06

From Table 4.4, the best leaching rate is obtained after 120minutes with 2.0M Concentration of Hydrochloric acid, while Table 4.5 reveals the best leaching condition at 0.5M concentration at a time of 20minutes when leaching with Citric acid. The results obtained were recorded as the optimum leaching conditions and summarized in Table 4.6. See Appendix Table A1 for XRF results for leaching at optimum developed conditions.

Table 4.6: The developed optimum leaching conditions and leaching rates for the selective leaching of Phosphorus from -355 +250 particle size of the Koton-karfe iron ore

ACIDS	CONDITIONS				Leaching rate (%)
	Temp. (^{o}C)	Conc. (M)	Time (mins)	Agitation (rpm)	
Hydrochloric	30	2.0	120	300	33.84
Citric	30	0.5	20	400	27.27

Leaching of -180 +125 particle size

The developed leaching conditions were used to leach -180 +125µm particle size with all the acids under investigation. Table 4.7 presents the amount of phosphorus leached, calculated from the XRF results in Appendix Table A2.

Table 4.7: Result of leaching of -180 +125µm particle size with optimum leaching conditions generated for the different acids

Acid	Phosphorus Leached (%).
Hydrochloric	35.13
Citric	29.73

Table 4.7 revealed that 35.13% and 29.73% leaching of phosphorus is obtained with hydrochloric and citric acid respectively. This shows an improvement from what was recorded from the leaching of -355 +250μm particle size (33.84% for Hydrochloric and 27.27% for citric acid). The improvement in selective leaching of phosphorus with the -180 +125μm particle size is attributed to the increase in surface area which aids the exposure of more mineral to the leach liquor and therefore increases the leaching of phosphorus.

Optimum phosphorus leached for the various particle sizes and the acids used.

From Tables 4.6 and 4.7 it is seen that citric and hydrochloric acid leached 27.27 and 33.84% P for -355 +250μm particle size and 29.73 and 35.13% P for -180 +125μm particle size respectively. Therefore, it is concluded that citric and hydrochloric acid could only leach phosphorus associated with the $AlPO_4$ phase present in the ore, while the phosphorus in solid solution with goethite is not leached by any of this acids.

DISSOLUTION OF IRON DURING LEACHING.

Tables 4.8 and 4.9 presents the ICPOES analysis for the -180 +125μm particle size which shows the dissolution of elements in the various acids before and after leaching. Using equation 3.2, the percentage loss after leaching is calculated.

Table 4.8: ICP-OES analysis of Citric acid before and after leaching at optimum leaching concentration.

	Concentration (mg/l)		Percentage (%)
	Before	After	After
Al	0.0257	85.774	0.0089
Fe	3.0625	14.4779	0.0011
Mn	1.1998	4.4109	0.0003
Si	232.91	540.411	0.0308
Pb	0.1416	0.5568	4.2×10^{-5}
Co	0.4384	0.5293	9.1×10^{-6}
Ni	0.334	0.3575	2.4×10^{-6}

Table 4.9: ICPOES analysis of Hydrochloric acid before and after leaching at optimum leaching concentration.

	Concentration (mg/l)		Percentage (%)
	Before	After	After
Al	0.0691	603.784	0.0604
Fe	3.3087	282.132	0.0279
Mn	1.2075	25.006	0.0024
Si	102.139	2995.57	0.2893
Pb	0.0696	1.6595	1.6×10^{-4}
Co	0.4388	1.0511	6.1×10^{-5}
Ni	0.266	0.3617	9.6×10^{-6}

The results revealed that the major elements dissolved in the acids are Si, Al, and Fe while other elements Mn, Pb, Co, and Ni are dissolved in negligible amount. For Citric acid on Table 4.8 it revealed the dissolution of 0.031%Si, 0.009%Al, and 0.001%Fe. The result for hydrochloric acid on Table 4.9 revealed that 0.29%Si, 0.06%Al, and 0.028%Fe is dissolved This indicates that the highest amount of Si and Fe dissolved are associated with Hydrochloric acid. The high dissolution of Silicon associated with hydrochloric acid is confirmed by the observation of the formation of a light grey jelly like substance which is suspected to be amorphous colloidal silicon in the course of leaching at higher concentration of hydrochloric.

pH VALUES OF ACID BEFORE AND AFTER LEACHING.

Tables 4.10 and 4.11 present the pH values of the acids before and after leaching. They reveal that the amount of acid used up for the leaching of the -180 +125µm is more than that used for the -355 +250µm. This is in line with the initial results obtained. From the pH values it can therefore be concluded that minimum amount acid is used up for leaching.

Table 4.10: pH values of acid before and after initial leaching at optimum conditions for -355 +250µm particle size. (19.3°C)

	Before	After
Citric	1.7	1.74
Hydrochloric	0.31	0.24

Table 4.11: pH values of acid before and after Initial leaching at optimum conditions for -180 +125µm particle size. (2.17°C)

	Before	After
Citric	1.45	1.51
Hydrochloric	0.48	0.42

Conclusions

From the work of leaching of the iron ore, the following conclusions are drawn:

Leaching of – 355 + 250µm, gave 33.84 and 27.27% while -180 +125µm gave 35.13 and 29.73% dephosphorization for hydrochloric and citric acid respectively.

Hydrochloric acid gave the best leaching, but only phosphorus in $AlPO_4$ is leached while phosphorus in solid solution with Goethite could not be leached.

ICP-OES reveal minimal loss of iron to the leach liquor. pH measurements reveal that minimal amounts of acid is used, therefore acids can be reclaimed and recycled.

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APPENDIX

Table A1: XRF Results for the leaching of of -355 +250μm particle size under optimum leaching conditions.

Constituent	Control	Hydrochloric	Citric
SiO_2	15.81	16.45	16.45
TiO_2	0.24	0.21	0.21
Al_2O_3	12.20	12.14	11.40
Fe_2O_3	67.73	67.95	68.85
MnO	0.45	0.36	0.40
MgO	0.47	0.34	0.41
CaO	0.35	0.12	0.15
Na_2O	*<0.01*	*<0.01*	*<0.01*
K_2O	0.08	0.02	0.03
P_2O_5	1.98	1.31	1.44
Cr_2O_3	0.02	0.01	0.02
NiO	0.02	0.02	0.02
V_2O_5	0.07	0.06	0.07
WO_3	0.07	0.04	0.06
BaO	0.07	0.05	0.05
Cl	0.06	0.40	0.01
MoO3	0.02	0.02	0.01

Table A2: XRF Results for the leaching of -180 +125μm particle size under optimum leaching conditions.

Constituent	Control	Hydrochloric	Citric
SiO_2	14.06	14.25	14.97
TiO_2	0.22	0.22	0.21
Al_2O_3	12.25	12.13	11.11
Fe_2O_3	69.81	70.13	70.96
MnO	0.43	0.35	0.40
MgO	0.39	0.26	0.38
CaO	0.22	0.12	0.13
Na_2O	*<0.01*	*<0.01*	*<0.01*
K_2O	0.08	0.02	0.02
P_2O_5	1.85	1.20	1.30
Cr_2O_3	0.02	0.02	0.02
NiO	0.02	0.02	0.02
V_2O_5	0.07	0.07	0.07
WO_3	0.05	0.03	0.03
Co_3O_4	0.07	0.06	0.06
SrO	0.05	0.04	0.04
MoO3	0.02	0.02	0.01

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