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# Influence of mineralogy on biohydrometallurgical processing of complex sulphide ore

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# CHAPTER THREE

## INVESTIGATIONS PUBLISHED FROM THIS STUDY

### 3.1 Synopsis

The role of ore mineralogy in the biohydrometallurgical processing of low-grade complex sulphide ores, using a low grade ore from Nigeria as a case study, is investigated in this study. The study is approached through an applied mineralogical study of the ore and its influence on developing an optimal route for the microbial leaching of the low-grade complex ore under varying process parameters, and an investigation on the interaction between microwave processing, mineralogy and bioleaching. The experimental stages involve the mineralogical characterization of the low-grade ore and comminution at various rod mill parameters to determine an optimal comminution route. The bioleaching process involved sub-culturing of the mixed cultures of mesophiles; bioleaching at varying process parameters, characterization of bioleached residues; a study of the mechanisms of the bioleaching process using electrochemical and scanning electron microscopy techniques. Lastly, an investigation on the interaction between microwave heating and mineralogy, and their influence on bioleaching were carried out using an electrochemical technique. At the core of all these studies was the investigation of the interaction and influence of ore mineralogy on the process development and the subsequent products generated.

### 3.2 Summary of the published articles

A compilation of published articles from the investigations of this study in peer-review and reputable ISI journals assembled for this thesis are presented in this Chapter. Relevant literature cited, materials used for the study, experimental methodology and results in these studies, thoroughly presented and discussed in these publications make up this chapter and forms the body of the thesis. A summary of each of the articles and their interrelationship is provided in the first section of this chapter. These articles are

sequentially arranged (although not according to date of publication) to coherently accomplish the objectives of the research.

**Paper I:** **Process mineralogy as a tool for improving hydrometallurgical recovery of complex sulphide ores: an overview.** *Minerals Processing and Extractive Metallurgy Reviews. (Manuscript under review).*

Process mineralogy is one of the most important and fundamental tools for obtaining optimum economic recovery, but is often overlooked during minerals processing and extraction. This article therefore provides information on process mineralogy as applied to the processing of complex sulphide ores as well as mineralogical characteristics affecting the processing behaviour of ores during size reduction and recovery processes. It reviews the usefulness of process mineralogy and how it can be applied in improving the recovery of sulphide ores. Mineralogical characteristics of the major sulphide minerals in which principal base metals (copper, zinc, and lead) occur are discussed in this article. The need for characterization of sulphide ores is reviewed, while relevant techniques amongst the various types of analysis suitable for sulphide ores processing and for obtaining useful information that could aid effective understanding of their recovery processes are briefly discussed. Process mineralogical steps for improving sulphide ores are also highlighted.

It was found from the study that process mineralogy is an integral part of all the various processing stages and for improving efficiency during ore processing and metal recovery. Mineralogical studies prove to be very important in choosing a suitable flow-sheet and for optimizing and improving plant performance. Information obtained from this article, formed the fundamental foundation for this study.

**Paper II: Mineralogical characterization of Ishiagu (Nigeria) complex sulphide ore.** ***International Journal of Minerals Processing,*** 87, 83-89

The processing and extraction routes for non-ferrous metals from complex sulphide ores is largely influenced by mineralogical factors such as the origin and type of the ore deposit, type and amounts of the constituent minerals, their associations and intergrowths with both sulphidic and non-sulphidic minerals, morphologies, grain size, etc. Since the goal of every mineral processing operation is to effectively separate the valuable material from the gangue with minimum metal loss in the tailings, the need to have a full understanding of mineralogical characteristics of the ore deposit is imperative to developing and employing a sustainable, effective and relatively economical recovering route. The aim of this paper was therefore to characterize the complex sulphide ore deposit located at Ishiagu in the South Eastern part of Nigeria that was used for this study, with a view to provide relevant background mineralogical information on which the processing of the ore could be effectively achieved.

An initial, semi-geological study of the mineral deposit, including a visit to the mine sites of the two small scale mining companies, was carried out. Observations on the mineralization were made to understand the origin of the deposit, mode of occurrence and the type of host rock. Physical examination of the ore deposits at different locations and different points within the mine sites was also done to determine the trends of mineral association and assemblage. A combination of SEM (Scanning Electron Microscopy) combined with Energy-Dispersive X-ray analysis (EDX), X-ray diffractometry (XRD), X-Ray Fluorescent (XRF) analysis and Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) were thereafter used to characterize the ore.

The semi-geological study revealed characteristic hydrothermal vein deposits of the complex sulphide ore. Characterization studies showed that the complex ore is made up of sulphide, carbonate and oxide typically consisting of 42% siderite, 35% sphalerite, 11% galena, 8% quartz and traces of copper bearing mineral. The ore was typically made up of fine to coarse grain intergrowths of the constituent crystalline phases both at the interstitials and the boundaries. The galena content of the ore occurred as medium to

coarse crystalline masses, while sphalerite occurred as crystalline masses and was characterized by a dark grey colour. Sphalerite was observed to occur as ferrous sphalerite with a variation in the percentages of zinc and iron within the various size fractions.

**Paper III: Influence of applied mineralogy in developing optimal hydrometallurgical processing route for complex sulphide ores.** ***Mineral Processing and Extractive Metallurgy Review, 27, 143–158 (2006)***

There is need to understand hydrometallurgical recovery processes of low-grade ores, clarify reasons for unexpected or poor metal recovery, and process effectiveness. This need necessitates a full background and useful mineralogical information for understanding, and interpreting the data, and solving problems that could be encountered during processing. This is because the extraction route applied for optimum technical and economical processing for metal recovery is influenced by chemical and mineralogical compositions, relative amounts, grain size distribution, and its chemical and mineralogical distribution amongst size fractions. In this paper, useful mineralogical data on size reduction characteristics, size distribution pattern, degree of mineral liberation, mineralogical and elemental distribution among different size fractions are interpreted and used for predicting parameters for which optimal hydrometallurgical recoveries of constituent metals could be obtained.

The ore was sequentially crushed in a jaw crusher and a cone crusher, and ground in a rod mill. Size analysis of the ground products from each of the varied rod mill parameters using the sieve analysis method in a laboratory test sieve was done to determine the effectiveness of each grinding parameter. Mineral phases distributed within varying size fractions were determined by XRD, while their liberation was determined by microscopy, using SEM/EDX to produce Backscattered Images (BSI). The elemental distribution

within each size fraction was determined by X-ray Fluorescence and Inductively Coupled Plasma- Optical Emission Spectrometry.

Comminution results showed that grinding time improved the effectiveness of size reduction while the amount of feed ore had a negative effect. Optimal grinding where the particle size fractions were most evenly distributed was observed to be grinding 500 grams of the ore for 15 minutes using 10 rods of 460 g each. The ore displayed a very good mineral liberation characteristic, as the constituent minerals were freely separated even at a particle size fraction of 75 µm. The ease and free release of the minerals was attributed to the less complex nature of the intergrowths of the associated minerals with little penetrations at the grain boundaries, coupled with the differences and a combination of both transgranular and intergranular fracturing liberation. Variations in elemental and mineralogical distributions within the different particle size fractions were observed. The concentrations of zinc, copper, and iron reduced as the particle size decreased while silicon, sulphur and lead contents increased.

**Paper IV: Effects of ore mineralogy on the microbial leaching of low grade complex sulphide ores.** ***Hydrometallurgy, 86, 96–104 (2007).***

Particle size plays a very important role in mineral recovery, and it is a key design variable in minerals beneficiation. It is of great importance in the biohydrometallurgical processes, and a significant factor that determines bacteria-mineral attachment and detachment effectiveness. Its relevance on the bioleaching process has drawn the attention of many researchers and several investigations have been reported. Nevertheless, the interrelationship between particle size and bioleaching microbes is yet a matter of controversy as there is still no strong agreement on the effects of particle size on the overall bioleaching behaviour. Owing to the differences in the mineralogical compositions in the different particle sizes, variations may exist in the microbial-mineral interaction that might lead to differences in dissolutions at varying particle sizes. Mineralogical data on the variations in mineral and phase distribution within particle sizes

of -53, 53, 75 and 106 µm reported in paper III was therefore utilized in this paper to provide an understanding of the influence of particle size and ore mineralogy on the microbial dissolution behaviour of the ore with respect to zinc and copper recoveries.

Bioleaching was conducted in mechanically stirred glass reactors using 6% vol/vol clean cell suspension of mixed cultures of Acidithiobacillus ferrooxidans, Acidithiobacillus thiooxidans and Leptospirillum ferrooxidans of an initial population of 1.5 x $10^6$ cells/ml. The bioleaching medium was kept at 32-35 $^0$C and agitated at 150 rpm. Pulp density was kept at 10% wt/vol, while the initial pH was 2.0, and the change in pH was measured every two days. The morphologies of the particles were observed by SEM analysis before and after microbial attack, while the mineral phases of the bioleached residues were also identified by XRD.

It was observed that mineralogical and elemental distribution within the various particle sizes both affected the mineral-microbe interaction, galvanic interaction, as well as precipitate formation on the surfaces. Highest bioleaching recoveries were obtained at a particle size of 75 µm, while particle sizes of 106 µm gave the least recoveries. Mineral phases and morphologies of the bulk ore and the leached residues showed differences both in phase and morphological changes, with the 75 µm having the highest transformation and attack.

**Paper V:** **Electrochemical studies on the interplay of mineralogical variation and particle size on bioleaching of low grade complex sulphide ore.** *International Journal of Minerals Processing. (Manuscript under review).*

The bioleaching of a sulphide ore occurs via an electrochemical mechanism similar to corrosion phenomena, which involves electrochemical and chemical reactions of the mineral with the bioleaching solution. As galvanic interactions depend on the mineralogical association between mineral phases present, there would be variations in their electrochemical galvanic interactions. Useful information for understanding the

mechanism of the bioleaching process could thus be best obtained using electrochemical techniques. In this present paper, a further understanding of the effects of particle size on bioleaching of the low-grade complex sulphide ore initially reported in paper IV is provided by studying the interplay of ore mineralogy, particle size, and mineral phase distribution within varying particle sizes on bioleaching behaviour through bioleaching experiments, electrochemical techniques and scanning electron microscopy methods.

Bioleaching experiments were carried out similar to the method used in paper IV. However, for this present study, the pH was kept constant at 2.0 through adjustment with sulphuric acid when needed. The amounts of zinc, lead, copper and total iron dissolved in the solution were analyzed periodically by atomic absorption spectrophotometry (AAS). Electrochemical behaviour was studied using particulate electrodes from the four varying particle sizes and from massive electrodes prepared from the two major sulphide mineral rich phases (sphalerite-rich and galena-rich) and a complex mineralogical phase of the bulk ore. The morphologies of the four particle size fractions after bioleaching experiments and the surfaces of the massive electrodes after microbial attack were examined using SEM. The mineral phases of the bioleached residues of the particles sizes and the bioleached residues on mineral surface of the massive electrodes were also identified by XRD.

Bioleaching studies revealed highest recoveries at a particle size of 75 µm, while electrochemical investigations revealed highest dissolution at a particle size of 106 µm. Electrochemical results showed that the sphalerite rich phase had the highest dissolution rate while the galena-rich complex phase had the least. SEM studies confirmed that the highest bacterial attack occurred at the sphalerite rich phase. The discrepancies between the dissolutions within particle sizes obtained from bioleaching experiments and electrochemical studies were consistent with and attributed both to the physical and mineralogical influences. Electrochemical behaviour was influenced and controlled by galvanic interaction resulting from mineralogical variation, while bioleaching behaviour was influenced by mineralogical variation as well as the physical effects of particle size.

**Paper VI: Role of ore mineralogy in optimizing conditions for bioleaching low-grade complex sulphide ores.** ***Transactions of Nonferrous Metals Society of China (Accepted for publication)***

A major attempt towards overcoming the challenges of the low solubility of complex sulphide ores, and improving the effectiveness of the biohydrometallurgical process for recovering the valuable metals is to investigate accelerating parameters favouring mesophilic leaching. Apart from the effect of physical parameters on mesophiles which has been widely studied, the use of mixed cultures of mesophiles has been observed to be an effective accelerating parameter. In spite of the knowledge of the better bioleaching capacities of mixed cultures over pure cultures, the effect of ore mineralogy, which is a major contributing factor during mesophilic leaching, has not been adequately taken into consideration. The knowledge of mineralogical behaviour of the constituent minerals within the ore would be very useful to aid the understanding of the microbial dissolution process, thereby revealing vital information about a way to allow process optimization and thus guides decisions on processing parameters. The role that ore mineralogy plays in understanding and optimizing the conditions favouring the bioleaching of the ore using mixed cultures is reported in this paper.

Bioleaching experiments were carried out similar to the method used in paper IV. Apart from particle size effects, bioleaching influencing parameters including stirring speed, volume of inoculum, pulp density and pH were investigated. The experimental results were analyzed by the analysis of variance (ANOVA) using Stata software. Reliability tests were also conducted on the data using Cronbach's Alpha coefficient. Solution potential of the system and pH changes were measured every two days in some of the experiments. The morphologies of the particles were observed by SEM analysis before and after microbial attack, while the mineral phases of the bioleached residues were identified by XRD.

Results showed that zinc can be bioleached about two and a half times than copper over an equivalent period of time. Dissolution at varying pulp density, volume of inoculum, solution pH and the low solution potential observed were also influenced by ore

mineralogy. Bioleaching at reduced pH had a positive influence on copper, lead and zinc dissolution, but the effect was greater for copper recovery, due to the absence of jarosite formation. Keeping the ratio of pulp density to volume of inoculum at 5:6 up till 10% wt/vol pulp densities and 12% vol/vol inoculum addition, the results of this study revealed that 89.2% zinc and 36.4% copper can be recovered at optimal parameters of particle size of 75 μm bioleached at a stirring speed of 150 rpm at pH of 1.6 for 5% and 10% wt/vol pulp density. A separate one-way analysis of variance which was done on the three major base metals in the ore at 95% confidence to determine if the differences in dissolution at varying particle sizes were statistically significant, revealed a potential significance of particle size on the bioleaching behaviours of these metals.

**Paper VII: Influence of microwave heating on the processing and dissolution behaviour of low-grade complex sulphide ores.** ***Hydrometallurgy, 89, 127–135(2007)***

The applications of microwave technology in mineral processing and extractive metallurgy have been of great and particular research interest for more than three decades. Despite the significant number of research studies in this area and the potential for achieving highly attractive benefits, there is still no agreement on the mechanism of interaction of microwaves with hydrometallurgical systems. Reported studies could not provide sufficient information from which industrial systems could be understood, and thus provide a basis for its industrial acceptability. While mineralogy is critical to the behaviour of ores during leaching, it is observed that attention has not been given to studying the effects of microwave pre-treatment on ore mineralogy and their influence on the dissolution mechanism of the treated ores.

This study provides a mineralogical basis for understanding the way in which microwave treatment affects the processing and recovery of a complex sulphide ore. In this study, the interplay of microwave irradiation and mineralogy on the heating characteristics, breakage response, and the mechanisms of dissolution of microwaved ore was

investigated in sulphuric and hydrochloric acid using electrochemical techniques. Microwave heating was carried out at varying process parameters, while the microwaved and un-microwaved samples were subjected to particle size analysis. Elemental distribution within the size fraction of the samples were carried out, while phase changes in the treated ore was also determined.

It was observed that the application of microwave heating has a beneficial effect on the processing and dissolution behaviour of the ore. Both ore mineralogy and microwave heating were observed to have a synergistic effect. The complexity in the mineralogical characteristics of the ore resulted in uneven heat transmission, which caused a build up of internal stresses within the ore. This gave rise to crack initiation, propagation, prolongation, and thus resulted in a positive effect of microwave irradiation on size reduction. Although microwave irradiation did not cause any significant mineral phase changes, the peak intensities of sphalerite and pyrite were slightly altered. An increase in dissolution observed in the microwaved treated samples as compared to untreated samples was reported to be attributed to an increase in electrochemical sites which resulted from an increase in the number of cracks and the increased pyrite phases.

**Paper VIII: Influence of microwave pretreatment on the bioleaching behaviour of low-grade complex sulphide ores. *Hydrometallurgy*** (in-press)**.**

Although a significant number of research studies have been reported on the application of microwave technology for improving size reduction and mineral liberation, it is however observed that not many studies have been reported on the bioleaching behaviour and mechanisms of microwave treated ores. Moreover, not much attention has been given to studying the interplay between mineralogy and microwave processing, and their corresponding influences on biohydrometallurgical processing. As there were limited reports in literature on the means by which microwave processing improves the recovery of base metals from sulphide ores, and little attention has been paid to studying effects of microwave irradiation on their biohydrometallurgical processing behaviour, it was

decided to investigate the interrelationship of mineralogy, microwave irradiation, and bioleaching behaviour of low grade complex sulphide ores.

An initial study on the influence of microwave heating on the processing and dissolution behaviour of low-grade complex sulphide ores was carried out and reported in paper VII. In this present study, the influence of microwave irradiation on the bioleaching behaviour and mechanism of a low grade complex sulphide ore is investigated in a mixed mesophilic bacterial culture through bioleaching experiments and electrochemical study. Bioleaching experiments were carried out on ground bulk ore and samples from microwave heating route where optimal size distribution at lower energy cost was obtained. Bioleaching behaviour was investigated using the mixed cultures similar to the method used in paper IV. The amounts of zinc, lead, copper and total iron dissolved in the solution were analyzed periodically by atomic absorption spectrophotometry (AAS). Electrochemical behaviour was studied using particulate electrodes from both microwaved and un-microwaved samples through electrochemical measurements such as Open Circuit Potential (OCP) measurement, potentiodynamic polarization, and chronoamperometric techniques.

Bioleaching results revealed similar dissolution trends for both samples. Percentage metal extractions for the microwaved sample were generally higher than the un-microwave treated sample; the pH of the microwaved ore was generally lower. Microwave treatment was observed to have more effects on copper and iron dissolutions than on zinc and lead. Both samples also showed similar electrochemical behaviour. However, microwaved samples displayed higher reactivity, dissolution rates, dissolution currents, current densities and a decrease in polarization resistance. Improved metal recovery observed in the microwaved samples was attributed to an increase in electrochemical sites which resulted from an increase in the number of cracks and the increased pyrite phases which promoted galvanic interactions within the bioleaching system.

## 3.3 THE PUBLISHED ARTICLES

The reprint versions of the articles as they appeared in the journals as well as the electronic submitted versions of articles under review are herewith presented without any alteration.