Effect of Oxygen and Micro-Cracking on the Flotation of Low Grade Nickel Sulphide Ore

Edison Muzenda and Ayo S Afolabi

Abstract**—**This study investigated the effect of oxygen and micro-cracking on the flotation of low grade nickel sulphide ore. The ore treated contained serpentine minerals which have a history of being difficult to process efficiently. The use of oxygen as a bubbling gas has been noted to be effective because it increases the pulp potential. The desired effect of micro cracking the ore is that the nickel sulphide minerals will become activated and this activation will render these minerals more susceptible to react with potassium amyl xanthate collectors, resulting in a higher recovery of nickel and hinder the recovery of other undesired minerals contained in the ore. Higher nickel recoveries were obtained when pure oxygen was used as a bubbling gas rather than the conventional air. Microwave cracking favored the recovery of nickel.

Keywords**—**Flotation, Conventional air, Oven micro-cracking, Recovery.

I. INTRODUCTION

LOW grade nickel-sulphide ore typically contain predominantly serpentine, pentlandite, millerite, antigorite and chrysotile. Other significant gangue minerals include talc, chlorite, brucite, magnesite, magnetite and pyrrhotite. These are types of minerals that could possibly and typically be associated or contained in the ore treated in this study. These minerals can affect the flotation process, and the typical visible problems of serpentine mineral in the flotation are: air bubbles tend to coalesce, flotation rate is slow and the mechanical recovery of sulphide and the metal content of the concentrates remain low. The gangue minerals of the ore are mainly members of serpentinite group, such as chrysotile and lizardite; talc in minor amount, as well as iron sulfides with pyrite and pyrrhotite as the main contributors. Pyrrhotite is generally much more abundant in nickel sulfide ore deposits than pentlandite. One of the difficult challenges for the nickel sulfide ores processing is the separation of pentlandite from pyrrhotite [1]. One of the difficult challenges for the nickel sulfide ore is the presence of violarite and magnetite

. The conditioning of sulphide ore flotation begins with a grinding process and ends in a flotation cell at the moment a gas bubble catches a mineral grain.

This kind of conditioning affects not only particle size distribution, but also the chemical properties of the mineral surface and the suspension. These chemical properties control, among other things, the degree of oxidation of particle surfaces, pH, conductivity, the concentration of dissolved oxygen and the temperature of the ore suspension [2].

The alkaline pH (6-9) is highly favorable with the concentration of dissolved oxygen. If xanthate is absorbed by an electrochemical mechanism, the important variable affecting the kinetics of conditioning are both mixed potential and concentration of oxygen at the interface. To quickly attain good recovery and grade with a lower consumption of collectors agent, both the pulp potential and the concentration of oxygen must be at optimum, as it is known that the pulp potential has been used to control the flotation process for a number of years.

By varying the dissolved oxygen concentration from extremely low values to a saturated concentration, different pulp potential and different xanthate absorption rates are achieved. If the effect of oxygen concentration is known and the optimum value for the pulp potential and the oxygen concentration is chosen, it is possible to improve the selectivity of minerals [2].

Serpentinites cause difficulties in particular with low grade ore if sulphides are finely disseminated in the matrix. The ore treated was initially subjected to microwave cracking in a kitchen type microwave with a variable power output of 1100 W and 50Hz. Ores that have a consistent mineralogy and contain a good absorber of micro wave radiation in a transparent gangue are the most responsive to microwave treatment. Under microwave irradiation, selective and differential heating of different mineral phases of the ore resulted in thermal stress cracking and made the ore more amenable to size reduction resulting in a decrease in the work index.

A significant number of studies have been reported on the application of microwave for improving size reduction and mineral liberation. Percentage metal extraction for the microwave treated sample was generally higher than the un-micro waved treated sample. Micro-waved samples display high reactivity, dissolution rates, dissolution current, current densities and a decrease in polarization resistance. Improved metal recovery observed in the microwave sample is attributed to an increase in electrochemical sites which resulted from an increase in the number of cracks. In this study microwave treating will then promote the ore to react quicker.

Flotation of sulphide minerals from their ores is achieved by a process in which two or more conditioning steps are carried out prior to a froth flotation step. The first step comprises conditioning a pulp of the ore in presence of at least one alkaline agent selected. The pulp is then further conditioned in the presence of xanthate of flotation reagents and sufficient dispersing agent for the effective dispersant of

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the mineral. The resulting pulp is then subjected to froth flotation [3].

Amongst several methods, a suitable approach to ensure that the metal content of the ore comes in contact with the flotation reagents is to thoroughly grind the ore fine enough to liberate the constituent mineral phases prior to flotation [4].

An attempt to float the ore without some form of size separation invariably gave disappointing results. Nickel recovery that exceeds 80% is between about 15μm and 60μm failed to reach 90% in any size fraction. Below 15μm and above 60μm recovery fell sharply. These size fraction results were obtained using a combination of a rod mill and a ball mill. Large amount of nickel at coarse size in the feed (+106μm) clearly gives poor response. A better approach is to ensure that as far as possible a minimum of 65% of the nickel in the feed is in the range of 10-106μm. Fine particles response float readily provided that the pulp density is less than 20 % solids [5].

The use of microwave heating for processing minerals is not a new concept [6]. Its applications in mineral processing and extractive metallurgy have been of great and particular research interest for over the past three decades [7].

A significant number of studies have been reported on the application of microwaves for improving size reduction and mineral liberation. While mineralogy is critical to the behaviour of ores during flotation processes, it is observed however, that not much attention has been given to studying the interplay between mineralogy and microwave processing, and their corresponding influences on hydrometallurgical processing of ores. As there are limited reports in literature on the means by which microwave processing improves the recovery of base metals from sulphide ores [8].

This investigation is relatively be related to the study of ref [2] where pure oxygen was used as the bubbling gas instead of the conversional compressed air. This paper presented discusses the effect of oxygen, conventional air and microwave cracking on the low grade nickel sulphide ore.

II. FLOTATION REAGENTS

Reagents usage and type are important factors in froth flotation. The recovery and purity of the final product obtained depends on various parameters such as the type and the amount of reagent, the nature and the particle size of solid, the absorption of reagent on solid particle surface.

A. Frothers

In pure water the air bubbles will burst when they reach the surface, and the particles adhering to them will sink back. By the addition of certain surface-active organic compounds, commonly called frothers, a stable froth is formed, which may be skimmed off together with the floated particles [9].

B. Collectors

Most ore minerals are hydrophilic, possible exceptions are graphite, sulfur, and molybdenite. The addition of collectors which are adsorbed to the mineral surface they may make them hydrophobic. Collectors are organic compounds, and different types of collectors are active for different types of minerals, e.g., xanthate for metal sulfides, fatty acids for metal oxide, hydroxides and carbonates, amines for silicates [9].

C. Activators

These are the compounds which make the mineral surface more responsive to the collectors. The addition of small amounts of copper sulfate to the pulp, however easily float the sulfide minerals. It is believed that copper ions in the solution react with sulfide to give a mono-molecular coating of copper sulfide. The latter is readily made floatable by addition of xanthates [9].

D. Depressors

These act in the opposite manner to the activators and counteract the collectors. Typical examples are cyanide and hydrosulfide ions, which depress various metal sulfides [9].

E. Conditioners

These are chemicals added to adjust the pH of the solution. The commonest one is lime which is used to make the solution slightly alkaline.

Sulphide minerals are semiconductors and oxidize in the presence of water and oxygen via a coupled electrochemical mechanism with the reduction of oxygen. Therefore the controlling mechanism for the electrochemical mechanism is the mineral solution interface potential [10].

Water quality is a critical factor to be considered during flotation calcium ions improves the absorption of xanthate, especially on the nickel and copper sulphide. The quality of process water can have a significant role in flotation of nickel ores. In the study conducted by [11], it was found that the calcium and thiosulphate ions, usually present in process water, activate or depress the flotation of sulphide with xanthate collector depending on the type of milling equipment used in the grinding. In this study tap water was used because the water quality is not of great importance to our investigation.

Nadium ethyl xanthate or Potassium ethyl xanthate are some of the recommended collectors that were used in this study as they were found to improve the recovery of nickel. A good depressant for mainly talc and other minority minerals is IMP4 [10]. On the other hand, rich nickel extraction is feasible in alkaline pulp with lime, adding diethylenetriamine (DETA) as the pyrrohotite depressant and the usage of the amine depressant has simplified and improved nickel processing significantly.

III. MATERIALS AND METHODS

A. Samples

The 25kg sample from a mine was brought in sizes fractions of 30 to 25mm. It was then ground to size fraction of 1000μm using a Cone crusher. From that 25 kg only 10 kg was then milled for 50 min to -75µm with a 70% pass, using a rod mill with 13 rods inside.

B. Experimental procedure

250g of milled product was measured and emptied into the Denver Flotation cell. 2 liters of water was then added to the cell to the correct level. The agitator was then lowered into the cell and the speed adjusted to 1200 rpm. The bubbling gas was air. The pH was adjusted to 9.5 by adding lime. 125g/T of CuSO4 was added and conditioned for 5minutes. 125g/T of Potassium Amyl Xanthate (PAX) was added and conditioned for 2 minutes. Lastly 1 drop of aero froth 466 was added and conditioned for 30 seconds. The floating froth was scraped filtered and then taken for analysis. The same procedure was repeated again 4 times but this time the bubbling gas was pure oxygen from an oxygen cylinder and the rest of the parameters (pH, density, reagents, agitator speed, etc) were kept constant.

C. Microwave treatment of the ore

The same procedure was repeated again 4 times but this time the milled product was first subjected to micro-cracking using a 1000W kitchen house microwave before flotation. The bubbling agent was pure oxygen and the other parameters were also kept constant.

After flotation, the water in the concentration (froth) was removed using a filter press and then the samples were dried using a lab oven. When the samples were dried, they were then blended and put into a spinning rifle so that they were evenly mixed and then the densities were measured using a pycnometer.

D. Reagents

TABLE 1
REAGENT USED

Reagent	Description	Condition time (min)
$CuSO_4$	Copper sulphate	5
PAX	Potassium amyl xanthate	2
Froth	Aerofroth 466	0.5

IV. RESULTS AND DISCUSSION

Nickel is presented in the form of pentlandite in fig 2 as pentlandite contains (Fe and Ni). Fig 2 indicates the dry analysis of the low nickel-sulphide ore prior to flotation. These results were obtained using the XRD machine. The nickel that is contained in the ore is about 200 counts (20%) of the total composition as shown in fig 1.

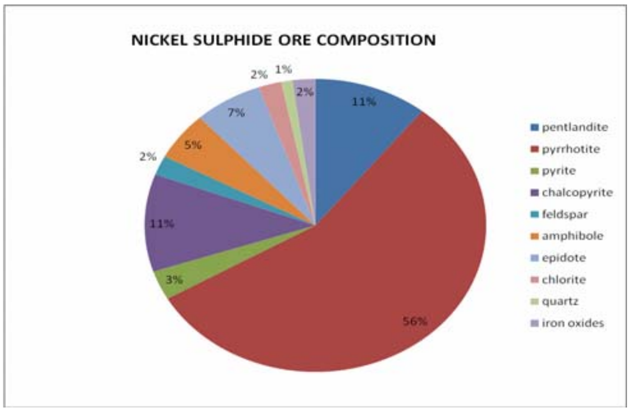

Fig. 1 Mineral composition of nickel-sulphide ore

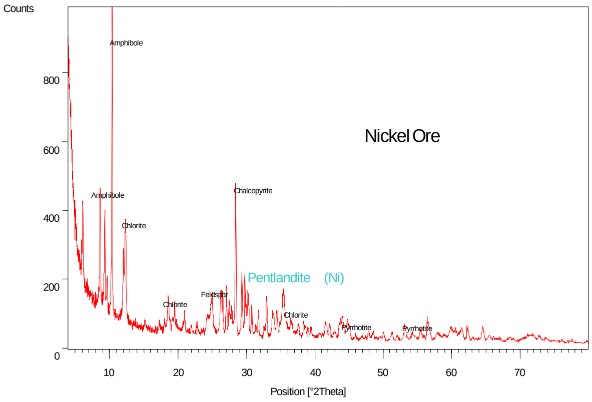

Fig. 2 Nickel ore analysis prior flotation

A. Effect of air and oxygen flotation at 4 minutes

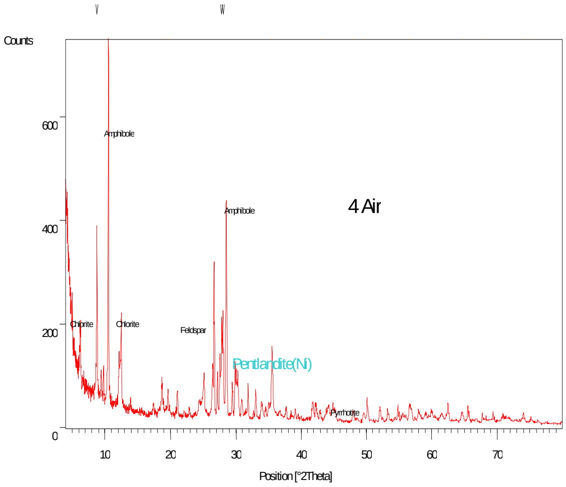

Fig. 3 Floating using air (froth)

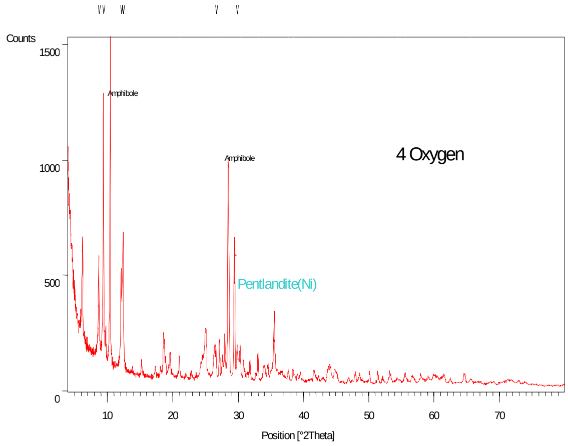

Fig. 4 Floating using oxygen (froth)

Figs 3 and 4 indicate a higher recovery in the oxygen floating. The oxygen shows a recovery of about 500 counts while that of the air floating content shows a recovery of 150 counts.

B. *Effect of air, oxygen and micro-cracking at 10 minutes*

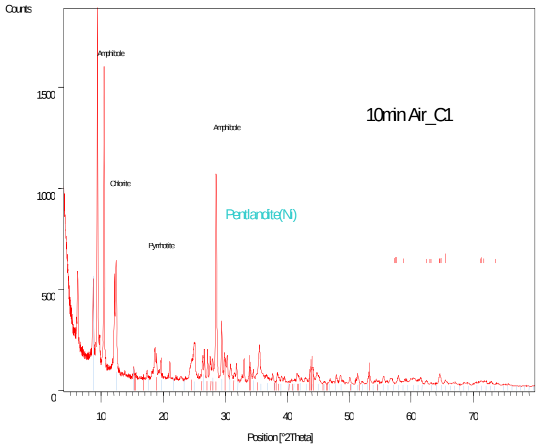

Fig. 5 Micro-waved & floated sample (air froth)

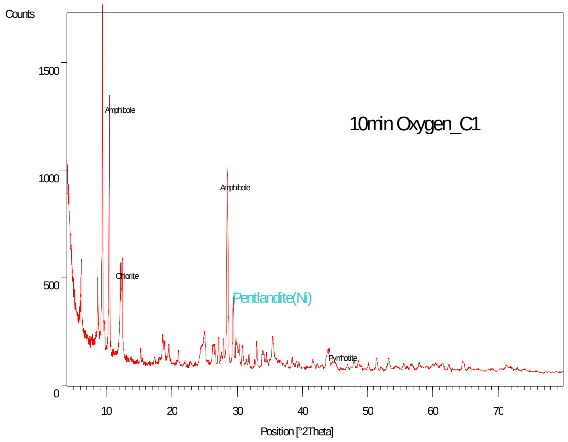

Fig. 6 Micro-waved & floated sample (oxygen froth)

Figs 5 and 6 represent the samples that have been subjected to pretreatment of microwave-cracking using a domestic 1000W max power, kitchen microwave for specified period of time. Microwave-cracking was used for activating the ore before flotation.

More nickel (pentlandite) was recovered in the 10min microwaved oxygen float than in the 10 min microwaved air float. About 400 counts is recovered in the oxygen float and ±270 counts is recovered in the air float, which is fairly below the recovery levels of the samples that were un-pretreated. Analysis of the concentrate and the tailings showed that more of the nickel is contained in the air tailings than in the oxygen tailings.

Analysis of the micro-waved ore in figs 5 and 6 shows that microwave-cracking of the low nickel-sulphide ore activates other minerals (especially amphibole) more than it activates the nickel.

C. *Effect of air, oxygen and micro-cracking at 15 minutes*

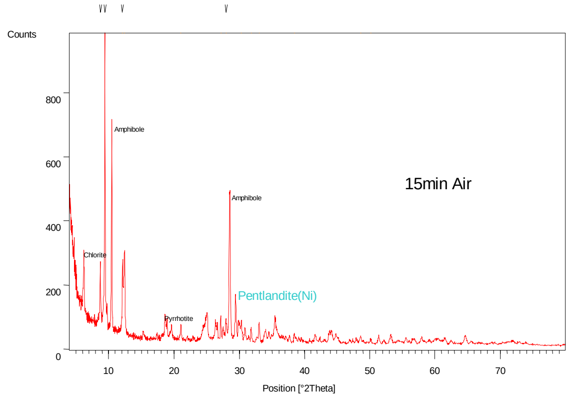

Fig. 7 Micro-waved & floated sample (air froth)

Figs 7 and 8 after 15 minutes of floating indicate a higher recovery of nickel in the oxygen float (±300 counts) than in the air float (150 counts). The amphilobe mineral has the highest counts of all the minerals recovered and this is due to the activation of the ore using microwave.

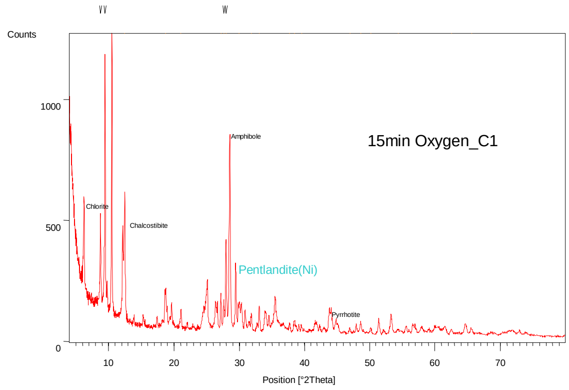

Fig. 8 Micro-waved & floated sample (Oxygen froth)

D. Effect of air, oxygen and micro-cracking at 20 minutes

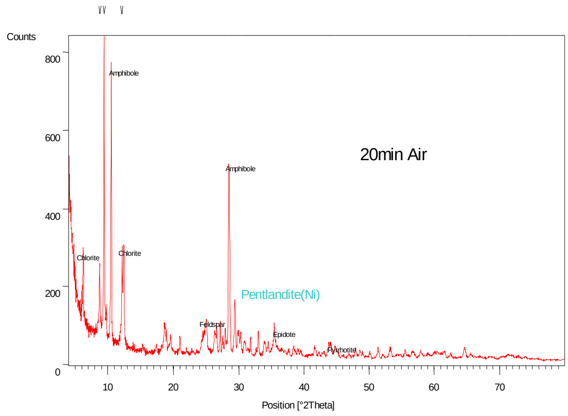

Fig. 9 Micro-waved & floated sample (air froth)

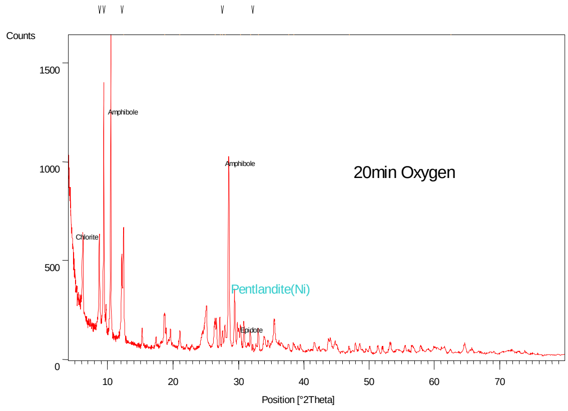

Fig. 10 Micro-waved & floated sample (oxygen froth)

In figs 9 and 10, after 20 minutes of floating recovery counts of 350 and 150 were obtained in oxyegen and air float respectively. This indicates that after a certain time of subjecting the ore to microwave cracking it reaches an optimum or saturation point. Microwave cracking improved the recovery of the amphilobe mineral because it is one of dominant mineral of the low grade nickel sulphide ore, so when subjected to microwave cracking they were activated more than any other mineral in the ore.

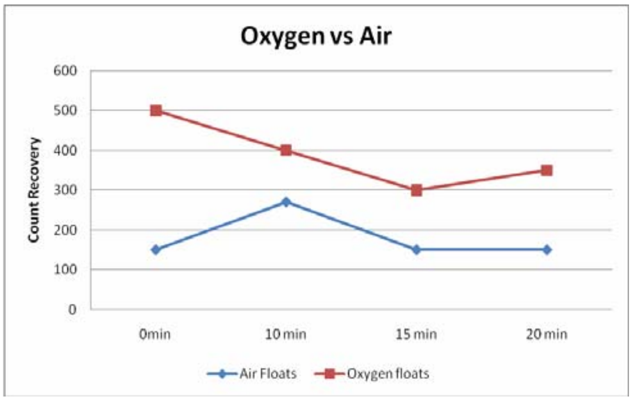

Fig. 11 Overall comparisons between oxygen vs. air

The recovery of nickel was higher when oxygen was used as a bubbling gas instead of conventional air as indicated in the overall comparison figure 11.

V. CONCLUSION

It can be concluded that a higher recovery of nickel is obtained by using pure oxygen as the bubbling gas on the ore that is below -75 µm and untreated. A recovery of 500 counts was achieved using oxygen as the bubbling agent and only 150 counts was obtained using air as the bubbling gas. Microwave cracking is favorable for Amphilobe because it activates the minerals in a way that higher recoveries are obtained to the un-microwaved sample. The Amphilobe recovery still remains consistently high in the tailings of the microwaved samples. The use of high power single-mode microwave of about 2-3 kW has to be investigated to assess the effect of microwave-cracking on the nickel (pentlandite) particles because the domestic kitchen microwave activates only the dominant particles because of their large surface area.

ACKNOWLEDGMENT

The authors are indebted to the investigators whose work was reviewed in this study. The authors gratefully acknowledge the financial support of the universities of Johannesburg and South Africa.

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