KWAME NKRUMAH UNIVERSITY OF SCIENCE AND TECHNOLOGY, KUMASI

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COLLEGE OF ENGINEERING DEPARTMENT OF MATERIALS ENGINEERING

THE IMPACT OF JAROSITES IN BIOX[®] PRODUCT ON CIL PROCESSES - A CASE STUDY OF ANGLOGOLD ASHANTI (OBUASI MINE)

BY

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JUNE, 2015

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THESIS SUBMITTED TO THE DEPARTMENT OF MATERIALS ENGINEERING, KWAME NKRUMAH UNIVERSITY OF SCIENCE AND TECHNOLOGY, KUMASI IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE THE DEGREE OF MASTER OF SCIENCE (ENGINEERING)

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> > JUNE, 2015

DECLARATION

I declare that this project work is my own work. It is being submitted for the degree of M.Sc. Environmental Resources Management in Kwame Nkrumah University of Science And Technology (KNUST), Kumasi. It has not been submitted for any degree or examination in any other University.

NAT ANDERSON AMPOFO		
(Student)	Signature	Date

CERTIFICATION

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ABSTRACT

AngloGold Ashanti Limited (Obuasi Mine), commissioned its Sulphide Treatment Plant (STP) in February 1994 to process 180,000 t/month of double refractory gold ore mainly, pyrite, arsenopyrite and pyrrhotite. The refractoriness requested a suitable pre-treatment process, which led to the design of the then world's largest commercial scale bacterial oxidation plant which uses Gencor's BIOX[®] technology. This technology was chosen based on its simplicity and environmental friendliness.

Gold is recovered by gravity and cyanidation. Gravity recoverable gold includes all visible gold; free gold and partly liberated gold, whereas sulphide minerals encapsulating the microscopic and sub microscopic gold particles are concentrated by flotation, pre-treated by the bio-oxidation process (BIOX[®]) and recovered by cyanidation process (CIL Circuit). During sulphide mineral decomposition, iron in pyrite goes into solution as ferrous ion (Fe^{2+}) , oxidizes to ferric ion (Fe^{3+}) , and then hydrolyses and precipitates as hematite, jarosite $[XFe^{3+}_3(SO_4)_2(OH)_6]$ or basic iron sulphates, depending on the prevailing conditions in the reactors. Sulphuric acid and arsenic acid are generated as by-product of the process. The stability of hematite is high. However the less stable jarosite and basic sulphates favour the formation of complexes with the iron sulphide mineral making gold recovery difficult, uneconomical in the upstream process and may pose environmental issues if allowed to get into the environment.

Jarosite/ basic iron sulphate is stable under acidic conditions (from pH values 1 to 7), but breaks down at higher pH (Babcan, 1971). In this study, composite samples from, sulphide concentrate feeding the BIOX plant, CIL feed, the conditioned material at the CIL, material in the first leach tank, CIL tails and the feed to the TSF, were taken, filtered and bottle rolled for 72 hours. There was an increase in recoveries, from 93.47% to 94.78%, when the pH was raised from pH 2 to pH 7 and then from pH 7 to pH 11 (2 stage conditioning) with slake lime before cyanidation, than raising the pH from pH 2 to pH 11 (single stage conditioning) with slake lime before cyanidation.

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DEDICATION

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I dedicate this work to my dear lovely wife Monica, and children, Frank, Ekow and Kwesi Krampah Ampofo whose support and patience has provided me the strength and perseverance to finish this thesis.

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CHAPTER 1

INTRODUCTION

1.1 Background

AngloGold Ashanti Limited (Obuasi mine), a gold mine located in Obuasi, is approximately 80 km from Kumasi and 320 km from the capital, Accra. The Mine is situated in a forested region, with the surrounding lands occupied by subsistence farmers. AGA is primarily involved in underground mining with pit depths reaching approximately 1,500 m. There are also pockets of surface mining operations. The ore body mined and processed at AGA is considered double refractory with the gold encapsulated within sulphide minerals. There are also carbonaceous or graphitic materials present, which act as preg-robbers.

Refractory gold ores often contain submicroscopic gold that is encapsulated in a matrix of iron sulphide mineral, such as pyrite, pyrrhotite and arsenopyrite. Their refractoriness is due to the presence of sulphides, carbonaceous matter, cyanicides and tellurides. Gold recovery in refractory ores is generally lower compared to free milling ores. To recover the gold, the host mineral must generally be broken down chemically by oxidative process such as roasting, pressure oxidation or bacterial leaching (Weir and Berezowsky, 1986; Chen and Reddy, 1990; Burbank *et al.*, 1990), which exposes the gold for subsequent recovery by leaching in cyanide solution and adsorption onto activated carbon. An alternative to these methods is the use of ozone, which increases the oxidation potential and the oxygen content of solution during cyanidation (Haque, 1992; Roca *et al.*, 2000; Salinas *et al.*, 2004; Elorza *et al.*, 2006; Carrillo *et al.*, 2007). In the Obuasi Mine, various

gold treatment processes that had been employed in the past include free milling, roasting, heap leaching, tailings reclamation/leaching. Most of these operations were situated at the North Mine footprint. Currently, all the gold treatment operations for the Obuasi mine are located at the South Mine footprint. The operations are at the sulphide, tailings and oxide treatment plants, forming the South Treatment Plant complex.



Figure 1.1 Obuasi Mine Footprint

Processing of primary ore through the Sulphide Treatment Plant (STP) follows a circuit based on single stage primary crushing, operating in series with two stage ball milling. Free gold in the milling circuit is recovered using gravity concentration and, sulphides by flash flotation and conventional flotation. The final flotation concentrate is reground for bacterial oxidation (BIOX), followed by Carbon-In-Leach (CIL) processing of the washed BIOX product. Figure 1.1 presents a simplified flow chart of Anglogold Ashanti, Obuasi Mine's Sulphide Treatment Plant.



Figure 1.2 Flow Chart of Processing of Primary Ore through the Sulphide Treatment Plant

The Obuasi Mine operates a bio-oxidation plant at its Sulphide Treatment Plant (STP) using the Gencor BIOX[®] Technology since commissioning in February, 1994. The bio-oxidation technology was preferred to other alternative methods of treating refractory sulphide ores due to its environmental friendliness since it eliminated the generation of sulphur dioxide (SO₂) and arsenic trioxide (As₂O₃) gases associated with its traditional roasting practiced at its former processing plant, the Pompora Treatment Plant (PTP). The STP has since commissioning succeeded in operating this technology successfully.

The washed bio-oxidation product is leached by cyanidation using the CIL process from which gold is finally recovered. Although the BIOX technology has been widely accepted as one of the best alternatives for processing complex sulphide ores, some processing parameters must be closely monitored to ensure efficiency of operation, to limit impact of products on the environment and ensure environmental safety (Rees and Van Deventer, 2000).

1.2 Problem Statement

In the BIOX[®] process for the treatment of sulphide ores in AngloGold Ashanti, various iron compounds are formed based on conditions in the reactors. These iron compounds include hematite, basic iron sulphates and jarosites. For metallurgical and environmental reasons, hematite is the desired iron product in the bio-oxidation (BIOX[®]) process. The formation of hematite is favoured at lower free acid concentrations and higher temperatures. It is a very stable compound, and hence its presence in tailings is desirable from an environmental perspective. In addition, hematite forms porous precipitates that do not interfere with the subsequent gold recovery process. At higher pH and lower temperature conditions in the reactor, iron sulphate or jarosites (AFe₃ (SO₄)₂(OH)₆), where typically $A = (K^+, Na^+, H_3O^+)$ are formed. Jarosites form very stable compounds with the metal of interest hence making recovery under normal conditions almost impossible. Various methods have been developed to treat such compounds. However, such methods are costly and generally unjustifiable except when the concentration of the mineral of interest is very high. In addition, jarosites create environmental problems in tailings ponds, as they break down slowly over many years, releasing acid and heavy metals to the

environment. They also cause diseases such as sidirosis, choroiditis, lung cancer among others, should they persist in the environment (Babcan, 1971). Basic iron sulphate is even less stable than jarosite, and this affect the efficiency of gold recovery in the subsequent cyanidation process for gold.

1.3 Justification

The use of bacterial oxidation process in the treatment of sulphide ores has been recognized as environmentally safe and economical. This process allows for the elimination of SO_2 and As_2O_3 gases as present in the roasting process. Again due to the low operating temperatures of 35 - 45 °C, the operating cost of bacterial oxidation is relatively lower. The formation of jarosites causes problems in cyanidation processes for gold/silver recoveries, by causing kinetic barrier between the particles and the dissolving agents. These problems could impact the economics of the process. Jarosites are stable under acidic conditions (pH 1-7) but break down at higher pH (>10.5). Therefore, under the basic conditions required to leach gold, the jarosites break down, reacting with lime to form undesirable ferric hydroxide (Ji *et al.*, 2006).

$$Fe(OH)SO_4 + Ca(OH)_2 \rightarrow Fe(OH)_3 + CaSO_4....(1).$$

The ferric hydroxide precipitate generated alters the rheological properties of the slurry feeding the cyanidation plant, creating mixing and settling problems which affect oxygen mass transfer through the slurry. Moreover, the risk of HCN gas generation is high if neutralization is not properly done and the pH drops to about pH 9.5. Workers could be

exposed to a potential hazardous situation. There is, therefore, the need for research efforts into the formation of jarosites and other iron sulphates in bio-oxidation and Carbon-In-Leach processes, and their control to reduce their effect on gold cyanidation.

1.4 Research Objectives

The objective of the research is to study the impact of iron compounds (specifically jarosites) formed from the BIOX process during pre-treatment of refractory ores on the subsequent CIL operations.

This will be done by:

- Obtaining sample products from the BIOX and CIL plants of AGA
- Investigating the effect of pH changes from (pH1.2- pH 2.0) to (pH 10 pH 11) on the iron compounds formation in the leach tanks.
- Establishing an operational management method to control the formation of jarosites and, the subsequent effect on gold recovery.
- Comparing the recoveries of single-stage conditioning (from pH 2 to pH 10.5) and two-stage conditioning (from pH 2 to pH 4 then to pH 10.5) of the BIOX product.

1.5 Expected Outcomes

This study is expected to achieve the following:

- establish the effect of jarosites on CIL operations,
- develop a metallurgical operational strategy that will be available for implementation.

1.6 Methodology

The methods used in this research include the following:

- review of relevant literature
- detail description of the mineralogy that are associated with the Run of Mine (ROM) ore during mining.
- development of a sampling and analytical regime based on the understanding of the mineralogy, chemistry, and overall metallurgical operations.
- collection of samples to represent ranges of compositional variation within the metallurgical and tailings operations.
- evaluation of Jarosite formation and stability at the CIL section of STP.
- evaluation of the potential formation and impact of other ferrous and ferric complexes in the various streams of the metallurgical and tailings operations of STP.

1.7 Limitations and Delimitations

Limitations

This work is limited to cover upstream operations (Flotation and Biox) and the biogeochemistry aspects of the operations.

Delimitations

In the course of executing this study, operational challenges and sample loses during analyses were encountered.

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1.8 Structure of thesis

Chapter one gives the background of the study, problem statement, justification, research objectives, expected outcome, methodology and the structure of the thesis. Chapter two reviews the geochemistry and mineralogy of gold, pre-treatment processes, complexing characteristics of iron in gold processing iron cyanides and environmental issues with products of biooxidation. Chapter three outlines the methodology used to determine iron compounds in CIL circuit and the effect of process variables on CIL process in relation to single-stage conditioning and two-stage conditioning. The results are presented and discussed in detail in Chapter four. Conclusions and recommendations for future research are outlined in Chapter five.

CHAPTER 2

LITERATURE REVIEW

2.1 Geochemistry and Mineralogy of Gold

The formation of gold depends mainly on temperature, pressure, pH, chloride concentration, and fugacity of H₂S in a hydrothermal system. Gold mostly occurs as AuCl₂⁻ in a system with temperature higher than 400 °C (Gammons and Williams-Jones, 1997), and the primary mechanism for gold deposition being the decrease in temperature. The dominant phase at lower temperature is Au(HS)₂⁻, whilst the maximum solubility exists in the vicinity of the H₂S-HS⁻-SO₄²⁻ equilibrium point; then, with the decrease in oxygen fugacity, the Au-S complex breaks down, leading to gold precipitation (Cooke and Simmons, 2000).

$$2Au(HS)_{2}^{-} + H_{2}O \rightarrow 2Au + 4HS^{-} + 2H + 0.5O_{2}$$
(2)
$$2Au(HS)_{2}^{-} + 8H_{2}O \rightarrow 2Au + 4SO_{4}^{2-} + 4H^{+} + 8H_{2}$$
(3)
$$FeCO_{3} + Au (HS)_{2}^{-} \rightarrow FeS_{2} (pyrite) + CO_{2} + H_{2}O + Au$$
(4) etc.

The most noble metal is Gold and occurs naturally in the metallic form (Prasad, *et al.* 1991). In air it is not reactive, and is not corroded by strong acids or bases. The chemistry of gold in aqueous solution is related to its very low electrode positivity, which is related to its standard reduction potential. The two most prevalent oxidation states of gold are +1 (aurous) and +3 (auric) for which the reduction potentials are 1690 and 1500 mV versus Standard Hydrogen Electrode (SHE) respectively (Pourbaix, 1966). These values are more positive than the standard reduction potential for water, 1230 mV, indicating that Au^+ and

 Au^{3+} are thermodynamically unstable in aqueous solution (Nicol, *et al.* 1987). The aurous and auric cations can be stabilised by a number of ligands, including chloride, cyanide, thiourea and thiosulphate (Adams, 1998).

2.1.1 Classification of Gold Ores.

Gold ores could be classified in several ways; either by the extraction technique or the associated geological environment (McQuiston and Shoemaker, 1975; Boyle, 1979; Boyle, 1987, Guay, 1981; Guay and Gross, 1981; Yannopoulos, 1991). One generally accepted method of classification based on the extraction method, however, classifies gold ores as either refractory or non-refractory. Non-refractory ores include placer, free milling and oxidised ores (Yannopoulos, (1991).

2.1.2 Placer Mining

Placer mining is an ancient method of using water to excavate, transport, concentrate, and recover heavy minerals from alluvial or placer deposits. Placer gold mining is potentially a relatively "clean" industry, not producing the highly toxic ore separation chemical; effluent, acid drainage, and smelter emissions common to the mining and processing of other metal deposits. With placer mining, recovery of the gold from the ore is usually the most expensive phase of the mining operation and can be the most difficult to implement properly. Placer mining techniques such as simple gold panning and sluicing, have generally only been the practice of artisan miners resulting in direct recovery of small gold nuggets and flakes. Placer mining takes advantage of gold's high density, which causes it to sink more rapidly from moving water than the lighter siliceous materials with which it

is found. Though the basic principles of placer mining have not altered since early times, methods have improved considerably (Silva, 1986, Farrington, 2000).

2.1.3 Free Milling Gold Ores

In the case of free milling ores, the most common method of extraction is dissolving the gold by direct alkaline cyanide (Mousoulous, Potaminos and Kontopoulos, 1984; Prasad, *et al.* 1991). With this method, about 95% of the gold is recoverable. Gravity concentration may be used to produce a concentrate that can be treated separately (Sandenbergh, 1998).

2.1.4 Oxidised Gold Ores

Oxide gold deposits, refers to gold-bearing veins, faults, and shear zones that typically contain appreciable amounts of oxidized ore or gossanous material, resulting from oxidation of sulfides. The concept of oxide gold deposits underwent a dramatic change after the discovery of the Carlin gold mine in north central Nevada in early 1960's, and now implies large tonnage, low-grade bulk minable deposits that are processed by milling and/or heap leach methods. Oxide gold deposits may be classified into two namely primary oxide ores which are derived directly from the effects of hydrothermal alteration on oxidized host rocks, and secondary oxide ores which derived from the epigenetic effects of circulating post mineral fluids oxidizing gold-bearing sulfides. Each type of oxide gold deposit has unique diagnostic mineralogical and metallurgical characteristics that must be addressed in order to achieve a successful exploration strategy (Baker, 1997).

2.1.5 Refractory Gold Ores

Gold ores and concentrates with a significant portion of the gold content which cannot be extracted completely by conventional cyanidation even after fine grinding are termed as refractory. The refractory behaviour of ores is generally associated with two major factors: the presence of carbonaceous matter and the existence of sulphides, tellurides and cyanicides (Boyle, 1979; Guay, 1981). The ore or concentrate can be termed as "double refractory" when the refractoriness is as a result of the occurrence of sulphides and carbonaceous matter (Nyavor and Egiebor, 1992).

A physical or chemical pre-treatment step can enhance recoveries for refractory gold ores, (Gudyanga, 1998). Refractory ores and concentrates are characterised by the presence of gold occluded in the mineral matrix is characteristic of refractory ores and concentrates andleads to the low gold extractions and high reagents consumption during the process of direct cyanide leaching (Beolchini, *et al.* 2005). In order to free up the gold particles and their accessibility to cyanide ions, an oxidation pre-treatment step is required. In the treatment of refractory ores and concentrates, several processes exits which include roasting, pressure oxidation, chemical oxidation and bacterial oxidation prior to cyanidation (Komnitsas and Pooley, 1989). A very common cause of refractoriness is the spread of fine grained or sub-microscopic gold inclusions within sulphide minerals such as arsenopyrite and pyrite.

2.2 Pre-treatment Processes

2.2.1 Oxidative roasting

Oxidation roasting has been extensively used in treating mixed sulphide ores and concentrates (Swash, and Ellis, 1986) and mainly involves two-stage roasting. The aim of this method is the transformation of the iron sulphides to iron oxides of high porosity in order for the cyanide ions to penetrate the calcine and boost the extraction of the contained gold. Fluidized bed reactors are mainly used as roasters, although multiple hearth roasters have been used for the first stage. The main reactions that occur in the roasting process are follows:

$$3\text{FeS}_2 + 8\text{O}_2 \rightarrow \text{Fe}_3\text{O}_4 + 6\text{SO}_2 \tag{5}$$

$$2Fe_3O_4 + \frac{1}{2}O_2 \rightarrow 3Fe_2O_3 \dots (6)$$

The first reaction (Equation 5) occurs in the first stage (Komnistas, and Pooley, 1989), where the shortage in oxygen result in a lower calcine, rich in magnetite, with a transformation of magnetite to hematite occurring in the second stage under an oxidizing atmosphere. The roasting temperature must be carefully controlled in order to decrease the SO₂ concentration and to avoid the condensation of sulphuric acid. The optimum operating bed temperature is in the range of 600-800°C. Application of high temperature during roasting may result in decreased gold extractions due to recrystallization and sintering phenomena. This method is expensive and environmentally undesirable. Viable alternative

methods have been developed in order to minimize these problems. These include pressure oxidation, bacterial oxidation and chemical oxidation (Komnitsas, and Pooley, 1989).

2.2.2 Aqueous pressure oxidation

By the know-how of the Sherrit-Gordon technology, aqueous pressure oxidation is either conducted at high temperatures (180 °C – 210 °C) or at low temperature (100 °C) (Berezowsky *et al.* 1990). The objective of the method is the oxidation and solubilisation of the sulphide compounds, which results in gold liberation during cyanidation. The method makes use of autoclaves and the addition of H₂SO₄ and O₂. At a temperature range of 180 – 120 °C (pressure 1,800 – 2,200 kPa) complete oxidation of the sulphides can be achieved within 1 to 3 hours (Weir and Berezowski, 1986).

At high temperatures the reactions that take place are:

$$2\text{FeS}_2 + 7\text{O}_2 + 2\text{H}_2\text{O} \rightarrow 2\text{FeSO}_4 + 2\text{H}_2\text{SO}_4 \dots (7)$$

The major parameters that affect leaching are acidity and temperature. High acidity promotes sulphide oxidation, whereas low temperature can lead to the formation of elemental sulphur, which may adversely affect gold recovery and formation of jarosite and other basic salts that occludes the gold and minimise its recovery during the cyanidation step. Studies carried out by Beattle (1985) showed, that higher gold recoveries could be achieved during the low-temperature aqueous pressure oxidation process. Sulphide oxidation during aqueous pressure oxidation can be as high as 99%, resulting in higher than 99% gold extraction.

2.2.3 Chemical Oxidation

Chemical oxidation carried out on commercial scale, at ambient pressure, was first applied at the Carlin Mines (USA) for the oxidation of carbonaceous sulphide ores (Prasad, *et al.*, 1991). Mainly, the purpose of the Carlin type process was to oxidise the carbonaceous material and humic acid using chlorine as an oxidising agent. The pre-treated ore was then leached with cyanide to recover the gold. Secondary, it was to decompose the sulphide pyrite so as to make available the entrained gold to cyanide attack during the subsequent leaching process. The Carlin process could either use sodium hypochlorite generated in situ by electrolysis of brine containing pulp or use chlorine directly. The relevant chlorine reactions are given as:

$Cl_2(g) + NaOH(aq) \rightarrow NaOCl(aq) + HCl(aq)$ (9)
$FeS_{2}(s) + 7NaOCl(aq) + 2NaOH(aq) \rightarrow FeCl_{2}(aq) + 2Na_{2}SO_{4}(aq) + 5NaCl(aq) + 6NaCl(aq) + 6Na$
H ₂ O(10)
$HCl(aq) + NaOH(aq) \rightarrow NaCl(aq) + H_2O $ (11)
$\operatorname{Au}(s) + 3/2 \operatorname{Cl}_2(aq) \rightarrow \operatorname{AuCl}_3(aq) \dots (12)$
The Carlin process could be improved upon by the "Double Oxidation" process (Prasad, et
al. 1991). The "Double Oxidation" process, which involves the pre-oxidation of the pyrite
before chlorination was developed to conserve chlorine. The slurry is aerated at 80-86 °C
until considerable portion of the pyrite and some of carbonaceous material is oxidised. The
pre-oxidation is then followed by the chlorination method to oxidise the carbonaceous
material and the rest of pyrite. The success of this double oxidation process for the pyrite
at Carlin is due to the high porosity and large surface area of the spheroidal pyrite. Further
developments in the area of chemical oxidation on gold ores include Caro's acid oxidation

developed by Ontario Research Foundation (Prasad *et al.* 1991) and the Nitrox Process (Van Weert *et al.* 1986). The Caro's acid oxidation is attempted on gold bearing arsenopyrite ores or concentrates and was shown to improve gold extraction from less than 65% by direct cyanidation to 85% during the development stage. The Nitrox process, which treats the ore for 1 to 2 hours in nitric acid in the presence of air at atmospheric pressure to oxidise pyrites and arsenopyrite prior to cyanidation, claims to increase the gold recoveries from 30% to 90%.

2.2.4 Sulphidisation-Flotation

Rastas *et al* (1990) developed a process for extracting lead, silver and gold from existing potential zinc residues using a sulphidisation-flotation method carried out at low pH 2-4 using low pH-collectors as dithiophosphates and dithiophosphinates. In these residues, a major part of the lead and silver is contained in the jarosite lattice and despite a lot of effort there has not been found any possibility to extract these metals without decomposing the jarosite compound. The research work on the sulphidisation-flotation method was focused on the conversion of lead and sliver compounds into the respective sulphides by using stoichiometric amount of aqueous sulphides (2.5 M Na₂S). The lead and silver sulphides, as well as silver in sulphidic particles and gold, can be separated and concentrated from the gangue using froth-flotation. Recoveries of 85-90%, 90-95% and about 80% were obtained for lead, silver and gold, respectively. Typically, concentrates containing 50-60% lead, 3-5 kg/t silver and 10-15 g/t gold were produced.

2.2.5 Bacterial Oxidation (BIOX PROCESS)

The BIOX process is a biohydrometallurgical process, which was developed for precyanidation treatment of refractory gold ores or concentrates. The bacterial culture consists of a mixed culture of *Acidithiobacillus ferrooxidans*, *Acidithiobacillus thiooxidans* and *Leptospirillum ferrooxidans*. (Livesey-Goldblatt *et al.*, 1983; Hutchins *et al.*, 1987; Rawlings, 1997). The bacterial oxidation process occurs by contacting refractory sulphide "Run of Mine" ore or concentrate, with the bacterial culture for a suitable treatment period under optimum operating conditions. The bacteria oxidise the sulphide minerals, thereby liberating the occluded gold for subsequent recovery by cyanidation.

, In order to attain the highest rate of sulphide oxidation, the number of bacterial cells and their activity is optimised under controlled plant conditions. The bacteria require a very acidic environment (pH 1.0 to 4.0), a temperature of between 30 and 45 °C, and a steady supply of oxygen and carbon dioxide for optimum growth and activity. There is no need for sterility during the bacterial oxidation process since the unusual operating conditions for the bacteria are not favourable for the growth of most other microbes. The reactions that occur in the process are oxidation and hydrolysis (Biomin ®, 2013). The oxidation reactions are:

$$2\text{FeS}_2 + 7\text{O}_2 + 2\text{H}_2\text{O} \rightarrow 2\text{FeSO}_4 + 2\text{H}_2\text{SO}_4.$$
 (13)

$$4\text{FeSO}_4 + 2\text{H}_2\text{SO}_4 + \text{O}_2 \rightarrow 2\text{Fe}_2(\text{SO}_4)_3 + 2\text{H}_2\text{O}....(14)$$

$$4\text{FeS}_2 + 15\text{O}_2 + 2\text{H}_2\text{O} \rightarrow 2\text{Fe}_2(\text{SO}_4)_3 + 2\text{H}_2\text{SO}_4 \qquad (15)$$

$$HAsO_2 + 2FeSO_4 + H_2SO_4 + O_2 \rightarrow Fe_2(SO_4)_3 + H_3AsO_4....(17)$$

$$4\text{FeAsS} + 13\text{O}_2 + 2\text{H}_2\text{SO}_4 + 2\text{H}_2\text{O} \rightarrow 2\text{Fe}_2(\text{SO}_4)_3 + 2\text{H}_3\text{AsO}_4 + 2\text{HAsO}_2(4) + (5) \dots (18)$$

The main end-product of the oxidation reaction is ferric sulphate. The ferric sulphate formed, hydrolyses in aqueous solutions. The extent of the ferric iron hydrolysis is dependent on the pH; in general, ferric iron has an extremely low solubility at a pH of > 2.5. A complete reaction for the hydrolysis is the formation of basic ferric sulphates which have the general formula XFe₃ (SO₄)₂(OH)₆, where X=K⁺(potassium jarosite), Na⁺ (natrojarosite), NH⁴⁺ (ammoniojarosite), or H₃O⁺ (hydroniumjarosite).

The hydrolysis reactions include:

The overall reaction is given by the combination of reactions (14) and (18):

Further reactions lead to the formation of jarosites.

$$(M = Ag^+, NH_4^+, K^+, 1/2Pb^{2+})$$

The process reaction starts with the initial oxidation of iron in both pyrite and arsenopyrite to the ferrous state, with a further oxidation of the ferrous to the ferric state. Sulphuric acid is produced in the overall oxidation reaction of pyrite, but consumed in the case of Arsenopyrite. However, both minerals produce sulphuric acid when oxidation and hydrolysis are taken into consideration. The sulphide ion is converted to sulphate during the oxidation of pyrite and arsenopyrite minerals. Sulphate is present in both the solution phase (as sulphuric acid and ferric sulphate) and the solid phase (as jarosite or basic iron sulphate).

The complete breakdown and dissolution of pyrite and Arsenopyrite is highly desirable from a gold recovery perspective, since the fine particles of gold that were encapsulated in the non-porous sulphide particles are completely liberated. This is in contrast to the roasting process, where the iron in pyrite and arsenopyrite is converted to hematite. During the roasting process, transformation of the original crystal structure of the sulphide minerals occurs, however, the gold particles remains trapped in the sulphide particles and also in the hematite particle. For this reason, the recovery of gold after oxidation of sulphides in BIOX process is better than after oxidation in a roaster.

Images of some of the widely used bacteria in the BIOX process are shown in Figure 2.1 and Figure 2.2

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Figure 2.1 *Acidithiobacillus ferrooxidans* (Source: Marie-Therese Guidici-Orticoni Research Group, 2011)



Figure 2.2 Leptospirillum ferrooxidans

Bacterial oxidation of gold ores is differentiated from bacterial leaching in that the bacteria used do not dissolve the gold. These bacteria used in bacteria oxidation are *Acidophilic mesophiles* and perform optimally within the pH range of 1.2 - 1.8 and temperature range of 35 and 45 °C. The oxidation reactions of sulphide minerals are exothermic. The process is cooled with water to maintain the temperature within the optimum range.

2.2.6 Proposed Mechanisms of Bacteria Oxidation of Sulphides

There are three proposed mechanisms of bacteria oxidation based on whether there is participation of ferric ions in leaching the mineral or not. They include:

- Indirect mechanism, where the bacteria oxidise ferrous ions in the bulk solution to ferric ions and the ferric ions leach the mineral.
- Indirect contact mechanism, where attached bacteria oxidise ferrous ions to ferric ions within a layer of bacteria and exopolymeric material, and the ferric ions within this layer leach out the mineral.

Direct contact mechanism, where the bacteria oxidise the mineral by biological means, without any requirement for ferric or ferrous ions (Yang *et al.*, 2013).

2.2.7 Cyanide Leaching

The leaching of gold using Cyanide has been demonstrated to be driven by electrochemical reactions (Nicol, 1980; Xue and Osseo-Asare, 1985). The process, as given by Nicol (1980), involves anodic dissolution of gold with reduction of oxygen at the cathode as follows:

$Au + 2CN \rightarrow Au (CN)^2 + e^{-1}$	
$O_2 + 2H_2O + 4e^{-} \rightarrow 4OH^{-}$	(27)
$O_2 + 2H_2O + 2e \longrightarrow H_2O_2 + 2OH^2$	
Moreover, the anodic reaction proceeds via an intermediate product which	ch further reacts

with cyanide to give the aurocyanide ion as given in equations 29 and 30.

$Au + CN^{-}$	AuCN(s) +	e ⁻	(29)
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$AuCN_{(s)} + CN \rightarrow Au$ (C)	() ⁻ ₂	30))
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The solid intermediate product, AuCN, which coats gold particles and passivates their surfaces is asserted to contribute to the slow kinetics of gold dissolution. In the presence of hydrogen ions or even water, cyanide is hydrolysed and therefore cyanidation is carried out in alkaline media according to the followings equations:

$CN^{-} + H^{+} \longrightarrow HCN_{(g)}$	(31)
$CN^{-} + H_2O \longrightarrow HCN_{(g)} + OH^{-}$	(32)

The production of hydrogen cyanide (HCN) gas is highly undesirable in all plants due to its toxicity, and also leads to a reduction in the quantity of cyanide available for leaching. At higher pH>10.5, about 10% of the total cyanide in the system exists as HCN gas. Therefore, the acceptable pH for effective cyanidation is in the range of pH 10.5 and pH 11 and it can be adjusted using lime or caustic soda. Lime is usually used because it is cheaper and also serves as a viscosity modifier (Brooy *et al.*, 1994). The kinetics of gold dissolution is influenced by the size of gold particles, dissolved oxygen levels, cyanide concentration, temperature and the quality of water used. Others factors include agitation, time, pulp density and the formation of gold coatings and compounds (Yannopoulos, 1991).

2.2.8 Leaching behaviour of other metals

The cyanidation of gold ores is a difficult issue owing to the ready formation of foreign metals cyanide complexes and high consumption of free cyanide due to those side reactions during gold leaching. Other metal cyanides in the leach slurries and the interaction between them are of great importance to gold extraction. These species dissociate, and the cyanide made available by the dissociation leaches gold. These metals species then precipitate, usually as a hydroxide (Rees, and Deventer Van,. 1999). Cyanide also forms complexes with a number of other elements, including iron, sulphur, zinc. The result of the formation of these complexes is a decrease in the level of free cyanide present in the slurry to drive the leaching of gold. During gold leaching, cyanide is also consumed by hydrolysis, oxidation to cyanate or thiocyanate formation through reaction with sulphur species.

2.3 Complexing Characteristics of iron in gold processing

In complexes iron has an electronic configuration of $(Ar)4s^23d^6$. The most common oxidation states for iron are +2 and +3. Moreover, the oxidation states +6, 0, -1 and - 2 are of importance. In contrast to osmium, iron never reaches its potential full oxidation state of +8 as a group VIII element. In air, most iron (II) compounds are readily oxidised to their iron (III) analogs, which represent the most stable and widespread iron species. For iron (II) complexes ($[Ar]4s^03d^6$) a coordination number of six with an octahedral ligand sphere is preferred. Iron (III) ($[Ar]4s^03d^5$) can coordinate three to eight ligands and often exhibits an octahedral coordination. Iron (III) generally is a harder Lewis acid than iron (II) and thus binds to hard Lewis bases. Iron (0) mostly coordinates five or six ligands with trigonal bipyramidal and octahedral geometry. Iron (–II) is tetrahedrally coordinated. Iron in low oxidation states is most interesting for organometallic chemistry and in particular for ironcatalysed reactions because they can form more reactive complexes than their iron (II) and iron (III) counterparts. Therefore, iron (0) and iron (–II) compounds are favoured for iron catalysis. Iron carbonyl complexes are of special interest due to their high stability with an
iron (0) centre capable of coordinating complex organic ligands, which represents the basis for organoiron chemistry (Bauer, I. and Knolker, J.H., 2008).

2.4 Iron Compounds

2.4.1 Jarosites

The jarosite family of compounds, AFe₃(SO₄)₂(OH)₆, consists of compounds where A is: H₃O⁺, Na⁺, K⁺, Rb⁺, Ag⁺, NH₄⁺, T⁺, Pb²⁺ or Hg²⁺ (Dutrizac and Kaiman, 1976). Most natural jarosites can be considered as solid solutions of jarosite [KFe₃(SO₄)₂(OH)₆], natrojarosite [NaFe₃(SO₄)₂(OH)₆] and hydronium jarosite [H₃OFe₃(SO₄)₂(OH)₆] (Brophy and Sheridan, 1965). Synthetic jarosite are readily precipitated from sulphate rich solutions containing 0.025 to 3.0 M Fe(III) (Brophy and Sheridan, 1965, Brown, 1970). The lower limit of Fe(III) for jarosite precipitation is near 0.001 M (Brown, 1970). If there are excess alkali metal ions available, the fraction of iron that is precipitated is independent of the iron concentration in solution (Dutrizac and Jambor, 2000). Consequently, the total amount of jarosite formed is directionally proportional to the concentration of Fe(III) in solution (Dutrizac and Jambor, 1984).

This sulphate mineral is formed in ore deposits by the oxidation of iron sulphides. Jarosite is often produced as a by-product during the purification and refining of gold and is also commonly associated with acid mine drainage and acid sulphate soil environments. Jarosites have a trigonal crystal structure and are generally brittle, with basal cleavage. They have typical hardness of 2.5-3.5, and a specific gravity of 3.15-3.26. It is translucent to opaque with a vitreous to dull lustre, and is coloured dark yellow to yellowish-brown.



Figure 2.3 Jarosite crystals from Sierra Peña Blanca, Aldama, Chihuahua, Mexico: (Source: (a) <u>http://www.minat.org/photo-34304.html</u>, (b) <u>http://www.shutterstone.com</u>)

Jarosite formation has negative effects on many applications that require the use of *Acidithioobacillus ferrooxidans*, especially in the process of biological gas desulphurisation and the recovery of gold. Some of the effects include the diminishment of ferric iron used as the absorbent for hydrogen disulphide, blockage of pumps, valves, pipes, etc., and the creation of kinetic barriers due to the small diffusion of reactants and products through the precipitation zone (Jensen and Webb, 1995).

2.4.2 Factors Influencing Jarosite Formation

Iron oxidation and the formation of jarosites are largely influenced by various factors including temperature and pH. To clearly understand the influence of these factors various researches have been carried on the effects of these factors on jarosites formation. The work of Daoud *et al.* (2006) showed that jarosite formation was very insignificant within a pH range of pH 1.0 - 1.6. It also indicated the jarosite formation is favoured at higher pH. Low temperature conditions also favour the formation of jarosite and other basic iron

sulphates.

2.4.3 Effects of Jarosite on Gold Recovery

Jarosite are very unstable compound that favour the formation of complexes with the mineral of interest making recovery difficult and uneconomical. In metal bioleaching systems, jarosite formation can hinder the leaching process by forming a kinetic barrier on reaction sites, thus obstructing further reaction. Some researchers have hypothesised that jarosite can be destroyed by a chemical treatment of the residue with lime at relatively high temperature to liberate the precious metals before cyanidation process. A chemical treatment of the residues with lime slurry at 90°C for \pm hours to destroy the jarosite minerals was shown to liberate the gold and silver for recovery by cyanidation.

However, in some mineral processing industries, jarosite is deliberately formed in order to remove unwanted iron species from solutions in hydrometallurgical circuits in the so-called 'jarosite process' (Arregui *et al*, 1979).

2.5 Environmental Issues with Biooxidation Products

Both hematite and ferric arsenate formation are favoured at lower free acid concentrations and higher temperatures. They are very stable compounds, and their presence in tailings is desirable from an environmental perspective. Ferric arsenate, for instance, is recognized by environmental authorities and regulators as an acceptable form of arsenic for safe disposal to tailings ponds. In addition, both hematite and ferric arsenate form porous precipitates that do not interfere with gold recovery processes, and are both relatively easy to handle as far as their settling, thickening and filtration characteristics are concerned. However, jarosites and basic iron sulphate occur at higher free acidities. These products are far less desirable because they cause both environmental and processing problems. Jarosites break down slowly over many years, releasing acid and heavy metals to the environment, often in tailings ponds. Some jarosites are stable. For example, silver jarosite is a very stable, insoluble compound, which inevitably forms when sulphide concentrates containing appreciable amounts of silver are oxidized. Silver cannot be recovered from silver jarosite by normal cyanidation, and the jarosite has to be decomposed to liberate silver.



Figure 2.4 Jarosite (yellow) forming on pyrite mine waste in Cyprus (Hudson-Edwards & Edwards, 2005)

Basic iron sulphate is even less stable than jarosites, and this causes worse processing problems in the subsequent cyanidation process for gold/silver recovery; problems that impact both the economics of the process as well health and safety. The economic problems presented by basic iron sulphate relate to the fact that the compound is stable under acidic

conditions (from pH 1 to 7), but breaks down at higher pH. This means that the acid that is tied up with basic iron sulphate must be neutralized with expensive lime, thereby increasing the cost of operation, according to the following equation:

 $Fe(OH)SO_4 + Ca(OH)_2 \longrightarrow Fe(OH)_3 + CaSO_4 \dots (33)$

In addition, the large amounts of fine ferric hydroxide and gypsum precipitate that are generated can drastically alter the rheological properties of the slurry feeding the cyanidation plant, creating pumping, mixing, settling and oxygen mass- transfer problems (Fleming, 2010).

CHAPTER 3

MATERIALS AND METHODS

3.1. Sample collection, preparation and analysis

3.1.1 Sample collection and preparation

A litre of double refractory gold ore slurry samples from the process stream, named A1, A2, A3, A4, A5, and A6, were taken hourly for 8 hours daily for a total of 4 weeks, to obtain a composite sample of each (See Figure 3.1).

Sample A1 is the flotation concentrate feeding the BIOX reactors, A2 is the oxidized material from the BIOX reactors feeding the CIL conditioning tank, A3 is the sample of the slurry conditioned with slake lime feeding the CIL plant, A4 is the sample of the first leach tank of the CIL, A5 is the sample of the slurry exiting the CIL plant, and A6 being the final tailings from the plant to the Tailings Facility.

These were milled to a particle size of 92% passing 45µm. 10 grams of each composite sample was taken after filtration and drying in an oven at 75 °C, for X-ray Fluorescence and X-ray diffraction analyses.



Figure 3.1 Flow sheet of the CIL plant showing sample collection points

3.1.2 Chemical and Mineralogical Analysis

3.1.2.1 Chemical analysis

The major elemental constituents in samples A1, A2, A3, A4, A5, and A6, and their bioleach products were determined using X-Ray fluorescence technique for the determination of chemical composition and Spectro X-Lab 2000 spectrometer for the mineral constituents, of the samples. Additionally, conventional fire assaying was used, followed by atomic absorption spectroscopy (AAS) to determine the grade of gold. Sulphur and carbon contents were determined by the combustion volumetric method using a LECO titrator SC-444DR (Figure 3.3).

Figures 3.4 and 3.5 present the Carbolite furnace used for fire assaying and the ion chromatograph used for anion assay respectively.



Figure 3.2 SPECTRO X-LAB 2000 Spectrometer (Geological Survey Department, Accra)

For all other assay determinations, 1- 2 grams of each sample was digested with aqua-regia and the resulting solutions filtered into a 25 - 50 ml volumetric flask and topped up with

distilled water. Metal concentrations were determined by atomic absorption spectroscopy (AAS) using a Perkin Elmer Spectrophotometer model 3300.



Figure 3.3 Atomic Absorption Spectroscopy (AAS)



Figure 3.4 LECO (Carbon and Sulphur Analyser)



Figure 3.5 Carbolite Furnace



Figure 3.6 Ion Chromatograph

3.1.2.2 Mineralogical Analysis

A Siemens D5000 Diffractometer was used to determine the main mineral constituents of the solid samples. These samples were each pulverized and pressed into sample holders. These were placed into the machine for measurement.



Figure 3.7 Siemens D5000 Diffractometer

3.2 Methodology

3.2.1 Determination of Iron compounds in CIL circuit

The oxidized product of the BIOX[®] may contain, in the solid phase, a host of un-oxidized sulphides, jarosites and elemental sulphur, while a host of dissolved salts notably ferric and ferrous sulphate, sulphuric and arsenic acid are in the liquid phase. Therefore, if the BIOX[®] product is not washed to eliminate or reduce considerably, the detrimental effect of these undesirable liquid and solid phase components is realised in the CIL circuit during cyanidation.

These may form cyanide, oxygen and lime consuming compounds when they react with cyanide and oxygen. Occasionally, some of these cyanide and oxygen consuming ions report to the CIL section due to inefficient washing.

In this preliminary study, the concentrations of these compounds and /or metals were determined and a series of bottle roll experiments were performed using plant stream samples. Sample A1 (flotation concentrate feeding the BIOX reactors), A2 (oxidized material from the BIOX reactors feeding the CIL conditioning tank), A3 (sample of the slurry conditioned with slake lime feeding the CIL plant), A4 (sample of the first leach tank of the CIL), A5 (sample of the slurry exiting the CIL plant), and A6 (final tailings from the plant to the Tailings Facility), were analyzed to determine their iron concentration and sulphur contents.

To determine the efficiency of gold extraction, 250 grams dry mass equivalent volume of each composited sample was measured into a beaker and stirred while oxygen was being sparged gradually into the slurry to raise the dissolve oxygen (DO) levels to 25 g/l. The samples were then transferred into leach bottles. 37 g/l fresh (Virgin) activated carbon and 24 g/t Sodium Cyanide were added. The bottles were tightly cocked and placed on rollers for 72 hours. Leaching was allowed to progress for 72 hours in conformity to plant operations. After 72 hours the dissolved oxygen (DO) level of the samples were measured and the loaded carbon removed by screening, and the cyanidation slurry filtered, washed and dried at 75 °C. Figure 3.6 presents the experimental set up for laboratory test work and Figure 3.7 shows the set up for bottle roll experiments.



Figure 3.8 Test work set up



Figure 3.9 Bottle roll set up

3.2.2 Effect of Process Variables on Carbon-in-Leach Process

Various process variables (pH, cyanide consumption, dissolved oxygen levels and lime slurry consumption) were studied over time with the aim of establishing their effects on gold recovery. Sample A2 (BIOX product feeding the CIL plant) was used for the preliminary studies as control.

3.2.2.1 Single-stage conditioning

In this study, the effect of pH and cyanide consumption on the CIL process was investigated. Sample A2, (the BIOX product which feeds the CIL plant) was used to investigate the impact of pH and cyanide consumption on gold recovery, with time. The effect of sodium cyanide (NaCN) variation and pH was investigated for pH 2 to pH 10.5 and for the period of 72 hours at the temperature of 40 °C and a dissolved oxygen (DO) level of 25 g/l. The gold recovery trend were recorded.

3.2.2.2 Two-stage conditioning

In this study, the feed to the CIL was solely used for the investigations. The sample was conditioned with slake lime slurry, gradually from the pH of 2 to pH 7 in a container in the first stage, before raising the pH to pH 11 in the second stage, before cyanidation.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Preliminary Studies

4.1.1 Determination of Iron compounds in the CIL circuit

Samples A1 (flotation concentrate feeding the BIOX reactors), A2 (oxidized material from the BIOX reactors feeding the CIL conditioning tank), A3 (sample of the slurry conditioned with slake lime feeding the CIL plant), A4 (sample of the first leach tank of the CIL), A5 (sample of the slurry exiting the CIL plant), and A6 (final tailings from the plant to the Tailings Facility), were analysed to determine their iron concentration and sulphur contents. Table 4.1 presents the results of the concentrations of the iron compounds in the CIL circuit studied under laboratory conditions.

Sample ID	Fe ³⁺	Fe ²⁺	Pyritic	SO4 ²⁻	Total
-	mg/l	mg/l	Sulphur	(mg/l)	Sulphur
A1 (BIOX feed)	0.20	0.50	4.42	1,200	5.76
A2 (Oxidized material)	0.70	0.05	0.48	2,800	3.32
A3 (Conditioned slurry)	0.50	0.05	0.49	2,200	3.29
A4 (1 st leach tank)	0.50	0.02	0.59	2,800	3.08
A5 (Effluent slurry)	N/E	N/E	0.40	2,000	2.62
A6 (Final tailings)	0.10	0.05	0.38	2,200	0.97

Table 4.1 Results of the concentrations of iron and sulphur components in the CIL circuit

Table 4.1 shows a decrease in pyritic sulphur as expected from the oxidation process during biooxidation. The presence of pyritic sulphur beyond BIOX reactor is a result of the expected incomplete oxidation, and there were no significant variation in the values along the process.

The SO₄ trend showed an increase from 1,200 mg/l to 2,200 mg/l. However, as a result of spillage pumps directed to certain stages of the CIL circuits from the BIOX, a 2,800 mg/l of SO₄ was recorded at A2 and A4.

Another significant change, was in the concentrations of the ferric and ferrous ions in contacting solution. The absence of ferric and ferrous ions in the effluent from the CIL circuit indicate that they were removed probably by reactions (4.3 and 4.4), involving these ions to produce solid material. The total Sulphur, as expected, showed a decrease in value from 5.76 to 0.97.

4.1.2 Mineralogical and Chemical Analysis

The mineralogical compositions of plant stream samples A1, A2, A3, A4, A5, and A6, taken and composited over a period of 2 weeks were analysed employing SPECTRO X-LAB 2000 X-ray fluorescence (XRF) and a Siemens D5000 Diffractometer.

4.1.2.1 Mineralogical characterization

Fig 4.1 and 4.2 presents the results of X-Ray diffraction analysis.



Figure 4.1 X-Ray diffraction pattern of the CIL feed (A2)



Figure 4.2 X-Ray diffraction pattern of the final tails (A6)

The presence of pyrite, quartz, patassium iron sulphate, polyhalite, albite, copper potassium sulphide, potassium jarosite, hydronium jarosite, melanlerite, branchite and montmorillorite was shown as the mineral constituents in the samples by the (XRD) analysis. The presence of the jarosites in Figure 4.1 is as a result of incomplete oxidation of the sulphides and the insufficient washing of the BIOX product by the counter current decantation (CCD) system before cyanidation, which takes place at times. The pyrite spikes in the final tailings sample (A6) as seen in Figure 4.2 could be from the plant spillage which are directed into final tailings storage hopper.

4.1.2.2 Chemical characterization

The major elemental constituents in samples A1, A2, A3, A4, A5, and A6, and their bioleach products were determined using several techniques. Amongst which are, X-ray florescence technique, fire assaying, ion chromatography, the combustion volumetric

method and atomic absorption spectroscopy. Table 4.2 presents the results of the chemical analyses of the samples.

Sample ID	Gold, (g/t)	Fe ³⁺ , (mg/l)	Fe ²⁺ , (mg/l)	Total Sulphur	Pyritic Sulphur	SO4 ²⁻ (ppm)
A1	21.30	0.20	0.50	5.76	4.42	1,200
A2	1.08	0.10	0.05	0.97	0.38	2,200
A3	4.68	0.70	0.05	3.32	0.48	2,800
A4	5.99	0.50	0.02	3.08	0.59	2,800
A5	2.44	N/E	N/E	2.62	0.40	2,000
A6	4.63	0.50	0.05	3.29	0.49	2,200

Table 4.2 Results of Chemical Analyses of Samples

The XRF results (Table 4.3) indicated that the samples contained mainly silicon, aluminium and iron containing compounds (SiO_2) , (Al_2O_3) and (Fe_2O_3) . Also included are the sulphur, potasium, manganissium, sodium-containing compounds. These have been given in the oxide form ((SO₃), (K₂O), (MgO) and (Na₂O)).

Table 4.3 X-Ray Fluorescence Laboratory Results of Major elements as oxides

	Composition, %					
Element	A1	A2	A3	A4	A5	A6
Na ₂ O	3.8	3.31	3.06	2.92	2.81	3.27
MgO	3.25	10.54	3.68	10.62	10.79	3.62
Al_2O_3	26.07	21.51	20.82	19.83	19.81	24.34
SiO ₂	63.4	54.48	52.96	50.25	49.21	64.91
SO_2	6.81	6.244	5.738	4.924	5.291	2.828
K ₂ O	4.316	4.294	4.218	4.043	3.961	3.908
Fe ₂ O ₃	12.69	13.18	12.19	12.02	12.25	6.515

	Composition, %					
Element	A 1	A 2	A 3	A 4	A 5	A 6
Fe	8.873	9.221	8.526	8.407	8.568	4.557
Ni	160.2	26.3	50.5	41.5	46.6	44.3
Cu	433.3	207.5	215.9	183.1	187.6	84.7
Zn	325.2	41.7	76.3	57.8	91.4	101.1
As	24630	33980	30440	30070	29990	6782

Table 4.4 X – Ray Fluorescence Laboratory Results of Minor Elements

4.1.3 Gold recovery

In the preliminary studies, plant stream samples A1, A2, A3, A4, A5, and A6 were studied under Laboratory conditions in conformity to plant operations, to simulate the initial recoveries in the CIL circuit. Table 4.5 presents the average of the results, and plotted in Figure 4.3.

Table 4.5 Results of gold recovery and sodium cyanide consumption

SAMPLE	Gold Recovery	NaCN Consumption	Gold Recovery	NaCN Consumption
ID	(%)	(kg/t)	(%)	(kg/t)
(A1)	45.56	10.72	45.62	10.6
(A2)	92.02	11.09	92.30	11.02
(A3)	91.43	11.28	91.26	11.20
(A4)	82.86	10.35	81.71	10.14
(A5)	22.68	0.41	20.32	0.41
(A6)	19.72	0.17	19.01	0.18

Sample	Gold Recovery,	NaCN Consumption,
ID	(%)	(kg/t)
A1	45.59	10.66
A2	91.35	11.05
A3	91.35	11.24
A4	82.29	10.25
A5	21.50	0.41
A6	19.37	0.18

Table 4.6 Average results of gold recovery and sodium cyanide consumption



Figure 4.3 Gold Recovery and Cyanide consumption

The results show that, the oxidized material (Sample A2) gave the highest recovery. This is because the material has been oxidized to liberate the locked gold, hence, giving high recoveries. Sample A1, which is the feed to the BIOX is low on gold recovery due to the

un-oxidized nature of the sulphide concentrate. Recovery in this case was possible because of the regrinding of the sulphide concentrate prior to bio-oxidation, leading to an increase in the surface area of the sulphide particles, exposes and frees some gold particles, and thus exposing and freeing some gold for extraction.

4.2 Effect of Process Variables

The BIOX[®] product (oxidized pulp from the BIOX[®] reactors) which is acidic and contains high dissolved iron and arsenic compounds is pumped to the CIL. The dissolved salts which include arsenic acid, sulphuric acid, and jarosites among others, impact adversely on the CIL operations. These contaminants tend to increase lime and cyanide consumptions at the CIL if not washed off properly, hence impacting negatively on gold recovery. Sample A2, the BIOX[®] product which feeds the CIL, was used to investigate the impact of pH and cyanide consumption on recoveries of CIL process with time. This was done in a single and two-stage processes as explained as follows:

4.2.1 Single-stage

In this study, to investigate the effect of pH and sodium cyanide consumption on gold recoveries, the BIOX[®] product slurry feeding the CIL circuit was used. The sample (A2) was conditioned with slake lime slurry from the pH of 2 to pH 11, before cyanidation.

4.2.1.1 Effect of pH on gold recovery

The gold recovery at various pH are presented in Table 4.7. The results show that gold recovery increased as the conditioning pH was increased from pH 2 to pH 11. Table 4.7

also presents the results of the effect of lime consumption on the gold recovery in the single stage conditioning and the results were used in plotting Figure 4.4.

2 4 10 pН 7 11 Au RECOVERY (%) 78.55 82.20 86.65 93.53 94.32 Lime Consumption (kg/t) 0.00 15.79 21.06 46.06 52.64

Table 4.7 Effect of lime consumption on gold recovery

100 90 80 70 60 Frequency 50 40 30 20 10 0 2 4 7 10 11 рΗ Au RECOVERY (%) Lime Consumption (kg/t)

Figure 34.4 Effect of pH and lime consumption on CIL recoveries

4.2.1.2 Effect of Sodium Cyanide

Table 4.8 presents the results of the percentage gold recovery and cyanide consumption versus time in a single stage conditioning extraction process. The results were used in plotting Figure 4.5.

Time (Hrs)	% Au Recovery	NaCN(kg/t)
0.0	0.00	0.00
0.5	78.54	3.21
1	83.69	4.40
4	89.40	7.70
8	90.19	9.26
16	90.94	10.71
24	91.71	11.46
48	92.40	11.69
60	93.43	11.88
72	93.57	11.89

Table 4.8 Gold recovery and cyanide consumption versus time in a single stage extraction process



Figure 4.5 Percentage Gold recovery and Cyanide consumption against contact time

From Figure 4.5, gold recoveries increased with time until about 48h of contact time, beyond which there was no significant increase in recoveries. The maximum gold recovery obtained after 48h was 93.6% percent after 72 hours contact time.

To achieve 93% gold recovery, effective control of the pH needs be adhered to, to avoid the formation of HCN gas which does not only reduce the amount of cyanide needed for the formation of AuCN complex in the CIL circuit, but also health implications on the CIL workers as well. The cyanidation reaction is given as:

 $4Au + 8NaCN + O_2 + 2H_2O$ $4NaAu(CN)_2 + 4NaOH \dots (34)$

There is the precipitation of Fe^{2+} as $Fe(OH)_2$ (a cyanocide) according to reaction (4.2). The ferrous hydroxide reacts with, and consumes the sodium cyanide added.

$$Fe^{2+}(aq) + 2OH^{-}(aq) \longrightarrow Fe(OH)_{2(s)} \dots (35)$$

The sulphide species in the CIL tanks are also complexed with cyanide promoting the formation of thiocyanate (SCN⁻) which passivates the gold surfaces, thereby reducing the dissolution rate and consequently leading to low recoveries. The reactions are:

$$CN^{-} + S_2O_3^{2-} \rightarrow SCN^{-} + SO_3^{2-}$$
.....(37)

4.2.2 Two-stage process

In this study, the feed to the CIL was solely used to investigate the effect pH and sodium cyanide consumption have on gold recoveries in the CIL circuit. Sample (A2) was conditioned with slake lime slurry, from the pH of 2 to pH 7 for about 30 minutes in a container, and then raised to pH 11 before cyanidation.

4.2.2.1 Effect of Sodium Cyanide

A gold recovery of 94.78% was realized at a lower sodium cyanide consumption of 9.02g/t as compared to that of 90.20% at a cyanide consumption of 93g/t for single stage process. Table 4.9 shows the results of cyanide consumption at various gold recoveries in the two-stage process, which was used in plotting Figure 4.6.

Time /Hrs	% Au Recovery	NaCN g/t
0.0	0.00	0.00
0.5	84.88	2.98
1	87.02	3.77
4	91.03	4.16
8	91.73	5.21
16	92.85	6.02
24	93.76	7.11
48	94.15	8.55
60	94.36	8.67
72	94.78	9.02

Table 4.9 Results of gold recovery and cyanide consumption in the two- stage process



Figure 4.6 Effect of cyanide consumption on recovery

The increased gold recovery in the two-stage process may be explained in the following: In the single-stage process, lime is added in a single step without regards to the rate of reaction such as equations 35, 36 and 37, which affect gold recovery. In the two-stage process however, the ferrous and ferric ions are allowed to precipitate out of solution and other complex reactions before the addition of cyanide. Therefore gold recovery is faster and better.

4.2.2.2 Effect of pH

The effect of pH was investigated for pH range of 2 to 7, and then, pH 10 to pH 11 for, the two-stage conditioning. The results are presented in Table 4.10 and illustrated in Figure 4.7. At a pH of 11, 94.32% of gold recovery was achieved. This can be attributed to the fact that, at a pH<7, ions of iron and sulphur (as sulphates) were precipitated as jarosites

before cyanidation. The presence of oxygen and sodium cyanide (determining factors for leaching), and adjusting the pH to 11 to reduce hydrolysis of cyanide, increased dissolution of gold and hence, resulted in an overall increased gold recovery.

pH	2	4	7	10	11
RECOVERY (%)	84.88	82.20	86.65	93.53	94.32

Table 4.10 Effect of pH on recovery



Figure 4.7 Effect of Lime added on recovery

Table 4.11 Effect of Lime ad	dition on recovery
------------------------------	--------------------

рН	2	4	7	10	11
RECOVERY (%)	84.88	85.32	89.95	94.46	95.26
LIME ADDED (kg/t)	0.00	16.38	23.01	44.12	45.46



Figure 4.8 Effect of lime consumption on gold recovery in the two stage process

The higher recoveries registered by the two-stage conditioning, as compared to the singlestage, may be due to the precipitation of the iron compounds at the lower pH of <7 in the first stage of conditioning with lime slurry thereby reducing Fe-cyanide complex formation. The XRD patterns in Figures 4.1 and 4.9, indicate the presence of jarosites (the blue and green peaks) and the iron compounds as precipitates. Raising the pH above 7 to pH 11 before cyanidation helps to control the hydrolysis of the sodium cyanide to HCN gas and also ensuring that the cyanide is not lost to the atmosphere, but kept in the CIL tanks to aid dissolution of the gold.



Figure 4.9 XRD pattern of First leach tank

4.3 Gold Recovery Compared

The effect of sodium cyanide consumption and the associated gold recoveries was investigated on the CIL feed for both single-stage and two-stages conditioning over a period of 72 hours. The two-stage conditioning gave a gold recovery of 94.78% at a lower sodium cyanide consumption of 9.02kg/t, while the single-stage conditioning registered 93.47% gold recovery at a rather higher sodium cyanide rate of consumption of 11.89 kg/t. Also, gold recovery was faster in the two-stage process than in the single- stage process. Gold recovery was 83.7% in 1hour at 4.4 kg/t NaCN addition for single stage, while it was 87.0% at the same time at 3.77 kg/t NaCN addition. After 4 hours, the values were 89.4% at 7.7 kg/t NaCN and 91.0% at4.16 kg/t respectively. After 24 hours, the values were 91.7% at 11.46 kg/t NaCN and 93.81% at 7.1k g/t NaCN.

4.4 Financial Benefit

4.4.1 Experimental Cost Analysis

Table 4.12 shows the estimated cost analysis for the experiment. However, costing considerations did not cover logistical cost such as transportation, labour, energy and so on and mixing effect and cost of power thereof. The final computation was done based on the Two-stage conditioning process and subject to fluctuations of the dollar rate.

Parameters		Single-sta	ge	2-stage
CIL feed, (t/d)	CIL feed, (t/d)			1100
Pulp density (% solid	s)	40%		40%
Water usage, (t/d)		1,650		1,650
Au recovery after 48h	1	92.40%		94.15%
Au produced, (oz)		367,000		372174.58
Current Au price		US\$ 1,318	8(/oz)	US\$ 1,318(/oz)
Total Au produced		US\$ 483,7	706,000	US\$ 490,526,096.44
NaCN consumption,	NaCN consumption, (Kg/t)			2.94
Total NaCN consume	ed, (t/yr)	4,476		1,096.62
Cost of NaCN, (US\$/	t)	US\$ 3,443/ton		US\$ 843.54/ton
Total cost of NaCN (US\$/yr)	US\$ 15,4	12,393	US\$ 3,776,036
O_2 used, (t)		5.5 tons		5.49 tons
Cost of O_{2} , (US\$/t)		400.0(US	\$/ton)	US\$ 400.0/ton
Total cost of O ₂ , (US	\$/yr)	US\$ 746,0	067	US\$ 744710.52
Total calculated benefit	10,11	2,585	US\$ per	r year
Minus: Calculated cost	746	,067	US\$ per	r year
NETT BENEFIT	9,36	6,517	US\$ per	r year

Table 4.12 Cost Analysis of financial benefit

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The conclusions that could be drawn from the investigations into the impact of jarosites in BIOX® product on CIL process, in a single and two-stage conditioning are presented as follows:

The XRD, XRF and chemical characterization preliminary test results done on samples A1,A2,A3,A4,A5, and A6 indicate the presence of jarosites and/or Iron Sulphates in the BIOX® product. The Two-stage pH conditioning registered a gold recovery of 94.78% which was higher than the single-stage of 93.57 % at the same residence time of 72 hours. The highest gold recovery for the single stage pH conditioning of 93.57% achieved in 72 hours was comparable to the 93.76 % recorded during the two-stage at 24 hours. This indicates a faster rate of recovery, and a significant decrease in residence time. The Two-stage pH conditioning had a better economic savings compared to the single stage. The study also indicated that, in the two-stage conditioning process, sodium cyanide consumption, a major component for the dissolution of gold aside oxygen, reduced significantly from 11.89 g/t to 9.02 g/t giving a saving of about 2.87 g/t of cyanide.

5.2 Recommendations for future works

The study makes the following recommendations from the findings:

- Leach tanks could be used instead of bottles for this test work so that oxygen supply could be optimized to further increase gold recoveries.
- A pilot scale test work to confirm the designs and capital cost should be considered.

- An economic analysis of a downward review of the current leach residence time of 130 hours should be considered.
- The BIOX® plant oxidation efficiency should be improved to minimize the ferrous and sulphide content in the product to the leach circuit.
- The reactivation of the BIOX® CCD automation should be considered to enhance washing efficiency of the product and minimize potential cyanocides in the product to the leach circuit.

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APPENDICES

Appendix A: Data on CIL Feed

Pulp Density: 92% passing 45µm. Dissolve oxygen (DO) levels: 25 g/l pH Range : >2 - 11 with Ca(OH)₂ Activated Carbon: 37 g/l fresh (Virgin) Grind: 20min/kg in a rod mill at 65% solids Sodium Cyanide (NaCN): 24 g/t

Sample :

Sumple .	CIL FEE	D															
SAMPLE	Dry	ry Carbon G		Tails	Soln			Filtrate	Loaded	CALCULATIONS			% Au Recovery		Metal	Consumption kg/t	
	Mass				Tails			Volume									
ID	(g)	Used (g)	(g/t)*	(g/t)	(g/t)	NaCN	рН	(mls)	Carbon	SOLN	CARB	HEAD	Ind	R+r	Balan	Lime	NaCN
BCF @ 0.5Hrs																	
[12]kg/t NaCN	250	30	42.92	9.21	0.923	2290	10.25	960	268.1	3.54	32.17	44.93	78.54	79.50	1.05	46.06	3.21
BCF @ 1 Hrs																	
[12]kg/t NaCN	250	30	42.92	7.00	0.369	1940	10.21	980	309.8	1.45	37.18	45.62	83.69	84.66	1.06	46.06	4.40
BCF @ 4 Hrs																	
[12]kg/t NaCN	250	30	42.92	4.55	0.246	1120	10.19	960	342.1	0.94	41.05	46.55	89.40	90.22	1.08	46.06	7.70
BCF @ 8 Hrs																	
[12]kg/t NaCN	250	30	42.92	4.21	0.292	700	10.16	980	340.1	1.14	40.81	46.17	90.19	90.88	1.08	46.06	9.26
BCF @ 16 Hrs																	
[12]kg/t NaCN	250	30	42.92	3.89	0.330	340	10.09	950	328.1	1.25	39.37	44.52	90.94	91.26	1.04	46.06	10.71
BCF @ 24Hrs																	
[12]kg/t NaCN	250	30	42.92	3.56	0.019	140	10.07	970	348.2	0.07	41.78	45.42	91.71	92.16	1.06	46.06	11.46
BCF @ 48Hrs																	
[12]kg/t NaCN	250	30	42.92	3.26	0.020	80	10.02	980	318.6	0.08	38.23	41.57	92.40	92.16	0.97	46.06	11.69
BCF @ 60Hrs																	
[12]kg/t NaCN	250	30	42.92	2.82	0.017	30	10.00	980	318.7	0.07	38.24	41.13	93.43	93.14	0.96	46.06	11.88
BCF @ 72Hrs																	
[12]kg/t NaCN	250	30	42.92	2.76	0.018	30	9.95	950	322.4	0.07	38.69	41.52	93.57	93.35	0.97	46.06	11.89

Sample :

SAMPLE	Dry	Carbon	Head Grade	Tails	Soln.			Filtrate	Loaded	CALCULATIONS		ONS	% Au Recovery		Metal	Consumption kg/t	
	Mass			ľ	Tails			Volume									
ID	g	Used g	(g/t)*	(g/t)	(g)/t	NaCN	рН	(mls)	Carbon	SOLN	CARB	HEAD	Ind	R+r	Balan	Lime	NaCN
BCF @ 0.5Hrs																	
[12]kg/t NaCN	250	30	42.92	6.49	0.532	2300	10.38	980	275.0	2.09	33.00	41.58	84.88	84.39	0.97	50.01	2.98
BCF @ 1 Hrs																	
[12]kg/t NaCN	250	30	42.92	5.57	0.303	2100	10.28	980	289.2	1.19	34.70	41.46	87.02	86.57	0.97	50.01	3.77
BCF @ 4 Hrs																	
[12]kg/t NaCN	250	30	42.92	3.85	0.121	2000	10.26	980	319.2	0.47	38.30	42.63	91.03	90.97	0.99	50.01	4.16
BCF @ 8 Hrs																	
[12]kg/t NaCN	250	30	42.92	3.55	0.043	1750	10.24	970	322.5	0.17	38.70	42.42	91.73	91.63	0.99	50.01	5.21
BCF @ 16 Hrs																	
[12]kg/t NaCN	250	30	42.92	3.07	0.022	1540	10.17	970	333.6	0.09	40.03	43.19	92.85	92.89	1.01	50.01	6.02
BCF @ 24Hrs																	
[12]kg/t NaCN	250	30	42.92	2.68	0.019	1260	10.13	970	349.2	0.07	41.90	44.66	93.76	94.00	1.04	50.01	7.11
BCF @ 48Hrs																	
[12]kg/t NaCN	250	30	42.92	2.51	0.016	870	10.01	990	344.5	0.06	41.34	43.91	94.15	94.28	1.02	50.01	8.55
BCF @ 60Hrs																	
[12]kg/t NaCN	250	30	42.92	2.42	0.013	850	9.97	980	346.2	0.05	41.54	44.01	94.36	94.50	1.03	50.01	8.67
BCF @ 72Hrs																	
[12]kg/t NaCN	250	30	42.92	2.36	0.013	760	9.85	980	349.0	0.05	41.88	44.29	94.50	94.67	1.03	50.01	9.02