MINERALOGICAL AND DEPORTMENT STUDY OF GOLD USING DIAGNOSTIC LEACHING EXPERIMENT

SHAIK ABDUL RAHMAN BIN

SHAIK ABDUL WAHAB

UNIVERSITI SAINS MALAYSIA

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by

SHAIK ABDUL RAHMAN BIN SHAIK ABDUL WAHAB

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LIST OF ABBREVIATIONS

AARL	Anglo American Research Laboratory
AnalaR	Analytical reagents
AsPy	Arsenopyrite
BR	Bottle roll
BRR	Bottle roll residue
EDX	Energy Dispersive X-ray
FA	Fire assay
FAAS	Flame Atomic Absorption Spectrometry
FESEM	Field Emission Scanning Electron Microscope
Gn	Galena
ICP-OES	Inductive Couple Plasma - Optical Emission Spectrometry
ICSD	Inorganic crystal structure database
LOI	Loss on ignition
O	Opaque minerals
OM	Optical microscopy
PPL	Plane polarized light
PS	Polished section
PSA	Particle size analysis
PTFE	Polytetrafluoroethylene
Py	Pyrite
Qz	Quartz
R	Coefficient correlation value
S/L	Solid/liquid
TS	Thin section
V	Void
VO	Visual observation

WGC	World Gold Council
XPL	Cross-polarized light
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

LIST OF SYMBOLS

%	Percent
µm	Micrometer/micron
2θ	Angle of incidence
Ǻ	Ångström
$AgNO_3$	Silver nitrate
Al_2O_3	Aluminum oxide/alumina
As	Arsenic
As_2O_3	Arsenic trioxide
Au	Gold
$Au(CN)_2^-$	Aurocyanide complex
CaO	Calcium oxide
CN	Cyanide
d_{80}	80 % particle size passing
DIBK	Diisobutyl ketone
E_h	Oxidation potential
Fe	Iron
FeAsS	Iron arsenic sulphide/arsenopyrite
$FeCl_3$	Ferric chloride
FeS_2	Iron sulphide/pyrite
g/t	Gram per tonne
H_2O_2	Hydrogen peroxide
H_2SO_4	Sulphuric acid
HCl	Hydrochloric acid
HNO_3	Nitric acid
K_2O	Potassium oxide
KCl	Potassium chloride

M	Molarity/molar
MgO	Magnesium oxide
mm	Millimeter
mV	Millivolt
$Na_2B_4O_7$	Borax
Na_2CO_3	Sodium carbonate
NaCN	Sodium cyanide
NaOH	Sodium hydroxide
nm	Nanometer
NO_2	Nitrous oxide
ºC	Degree Celsius
Pb	Lead
PbO	Lead (II) oxide/litharge
PbS	Lead sulphide/galena
$PbSiO_3$	Lead silicate
ppb	Parts per billion
ppm	Parts per million
SiO_2	Silicon dioxide/silica
SO_3	Sulphur trioxide
wt%	Weight percentage
λ	Wavelength of the incident X-rays

KAJIAN MINERALOGI DAN DEPOTMEN EMAS MENGGUNAKAN EKSPERIMEN PELARUTLESAPAN DIAGNOSTIK

ABSTRAK

Sampel bijih emas dari Kuala Lipis, Pahang telah digunakan dalam menentukan taburan emas dalam permineralan bijih menggunakan eksperimen pelarutlesapan diagnostik. Pencirian mineralogi telah dijalankan ke atas sampel bijih emas sebelum rekabentuk eksperimen pelarutlesapan diagnostik. Kajian ciri fizikal terhadap sampel batuan menunjukkan permineralan telerang kuarza bersekutu dengan mineral sulfida. Kajian OM ke atas sampel keratan nipis dan keratan tergilap menunjukkan emas berpanca dengan pirit, arsenopirit, kuarza dan galena. Analisis XRF, XRD dan ICP-OES telah dijalankan ke atas sampel bijih emas terkisar. Dari analisis XRF, komponen utama hadir adalah SiO_2 (81.14 %), diikuti oleh Al_2O_3 (11.18 %). Sebatian signifikan lain yang hadir adalah Fe_2O_3, K_2O, SO_3, As_2O_3, MgO dan PbO. Fasa mineral yang hadir daripada analisis XRD adalah kuarza, pirit dan arsenopirit. Penemuan ini menyokong analisis XRF dan kajian OM yang memberikan kesimpulan bahawa sampel bijih emas yang dikaji mengandungi emas bersekutu dengan mineral-mineral sulfida dan silikat. Teknik cerakin api dan ujian "bottle roll" sianida memberikan kandungan emas total dan emas bebas masing-masing sebanyak 31.38 g/t dan 16.79 g/t. Analisis ICP-OES ke atas sampel bijih emas terkisar juga memberikan amaun As, Fe dan Pb yang signifikan. Seterusnya, daripada eksperimen pelarutlesapan diagnostik, sebanyak 18.48 % emas dijumpai berpanca dalam arsenopirit dan pirit, 14.18 % emas dalam logam biasa sulfida dan pirit labil, 10.07 % emas dalam konsentrat sulfida dan 3.41 % emas dalam galena, dengan baki akhir pepejal mengandungi sebanyak 1.80 g/t emas dalam silikat (kuarza).

MINERALOGICAL AND DEPORTMENT STUDY OF GOLD USING DIAGNOSTIC LEACHING EXPERIMENT

ABSTRACT

The as-received gold ore samples from Kuala Lipis, Pahang was used in the deportment study of gold using diagnostic leaching experiment. Prior to the design of diagnostic leaching experiment, mineralogical characterization were conducted on the gold ore sample. Physical properties study on rock samples using visual observation showed the mineralization of quartz veins associated with sulphide minerals. OM study on the thin and polished sections samples showed that the gold was found interlocked with pyrite, arsenopyrite, quartz and galena. XRF, XRD and ICP-OES analysis were conducted on the ground gold ore samples. From XRF analysis, the major component present was SiO_2 (81.14 %), followed by Al_2O_3 (11.18 %). Other significant compounds present were Fe_2O_3, K_2O, SO_3, As_2O_3, MgO and PbO. Mineral phases detected by XRD analysis were quartz, pyrite and arsenopyrite. These findings compliment XRF and OM study which led to the conclusion that the gold ore sample received contained gold in association with sulphide and silicate minerals. From fire assay technique and cyanide bottle roll test, the total gold and free milling gold were about 31.38 g/t and 16.79 g/t, respectively. ICP-OES analysis on the acid digested samples gave a significant amount of As, Fe and Pb. The diagnostic leaching experiment that follows gave about 18.48 % of gold found interlocked in arsenopyrite and pyrite, 14.18 % gold in base metal sulphides and labile pyrite, 10.07 % gold in sulphide concentrate and 3.41 % gold in galena, with the final residue carrying about 1.80 g/t of gold in silicate (quartz).

CHAPTER ONE

INTRODUCTION

1.1 Introduction to gold ore processing

Gold is a metallic element with a chemical symbol of Au. Physically, gold is yellow in colour and has a high metallic lustre. Gold, the 79th element on the Periodic Table of Elements, is a metal of high degrees of malleability and ductility, higher than any other metals at all temperatures. In addition, gold is an excellent conductor of thermal and electricity. Chemically, gold is the most inert metallic element due to its great stability and resistance to corrosion (Yannopoulos, 1991). Gold is unaffected by most acids, except aqua regia solution (mixture of hydrochloric and nitric acid) and selenic acid. Thus, gold is a very stable natural occurring element. Gold also dissolves in solutions containing free chlorine and bromine and alkaline cyanides. Strong sulphuric acid, and polysulphides of the alkaline metals, also dissolves finely divided gold.

Malaysia is a country blessed with various natural and mineral resources such as palm oil, petroleum, tin, and also gold. A 2001 World Gold Council (WGC) report mentions Malaysia as one of the countries that could benefit significantly from an increase in gold mining activity (Monument Mining Limited, 2016). In Malaysia, gold occurs mainly in the Central Belt of Peninsular Malaysia. Figure 1.1 shows the geological map of Peninsular Malaysia with primary gold deposits. Major production of gold came from the states of Pahang and Kelantan within the Central Belt. In Malaysia, gold mineralization is typically associated with hydrothermal quartz vein system, skarn and volcanogenic massive sulphides (Kamar, 2012). Gold mineralization in the Central Gold Belt is generally categorized as a low mesothermal lode gold deposit due to its tectonic and geological setting.

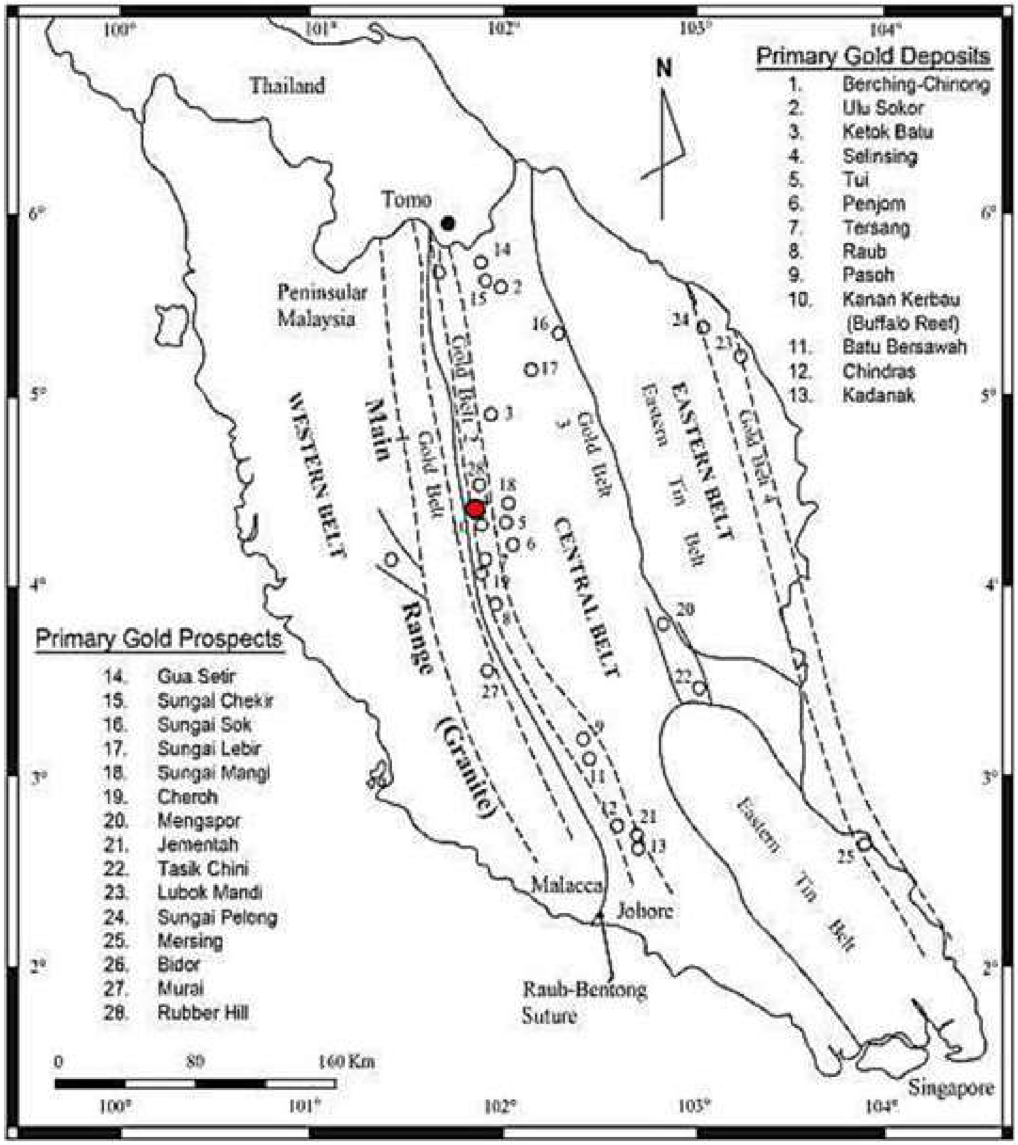

Figure 1.1 : Geological map of Peninsular Malaysia with primary gold occurrence (after Yeap, 1993)

Gold is basically formed from the transportation of hydrothermal fluid through cracks, fissures and faults, as a result of pressure and temperature gradient within ground subsurface. The hydrothermal fluid contains gold and other minerals which are originated from magma. There are some types of gold deposit all around the world. The most common deposits are quartz vein deposited from hydrothermal fluid in fault zones at medium (mesothermal) or shallow (epithermal) depths in crust (Kamar, 2012). Other common secondary deposit is the placer or alluvial deposit. Placer gold has been washed away by flowing water or carried by blowing wind (in case of gold dust) and deposited along with soil, sand, gravel and other transported matter (Yannopoulos, 1991). The high density and chemical stability of gold enables it to be mechanically concentrated in rivers and beaches, forming placer deposits.

Hardrock gold deposits are commonly mined using open pit mining and/ or underground mining methods. At the earlier age of mining, manpower was used to dig lands into pits and recover gold ores. Nowadays, machines and explosives are used as an alternative to manpower and to increase the efficiency of gold mining. In underground mining, shaft and tunnels are built to extract gold ore allocated deep in the subsurface. As compared to the mining of hard rock gold deposits, mining of placer deposits are very simple and easy. Since it is the secondary gold ore deposits, the materials are relatively loose and the means of extracting it involve the use of water. The most common manual technique of placer deposit mining is the gold panning. Wide, shallow pans are filled with sand and gravel containing gold, submerged in water and shaken. As gold is denser than rock, it settles to the bottom of the pan quickly. Many recent machines are build based on the concept of panning, such as the Knelson concentrator. Besides panning, placer gold deposits can be extracted using sluice box and dredging.

In the history of gold ore processing, from the year 1700 to early 1900’s, most of the gold deposits found by miners were of alluvial or placer gold deposits (Paulo et al., 2009). The techniques used to extract gold during this time are by using gold pans, sluice boxes and followed by mercury amalgamation. Mercury is used since it amalgamates with gold easily and gold can be recovered in a short time. In the late 1800's to mid-1900, cyanidation was invented. Cyanidation is very useful and effective in extracting free gold from ores. During this period of time, alluvial or placer gold deposits has started to deplete and hardrock gold mining was increasing all over the world. The processing of gold has evolved towards cyanidation, although mercury is still used in some large scale operations. Later on 1970's to present, heap cyanidation was developed. The use of mercury in large scale mines had almost completely disappeared, due to more recent developed gold processing methods. New technologies are created and developed in treating refractory gold ores, which cannot be extracted using conventional cyanidation technique. Centrifugal gravity concentration was also developed during this time.

1.2 Problem statement

Gold ores can be classified into two types that is free milling and refractory (Vaughan, 2004). Free milling gold ores are gold ores which are leachable by using cyanide solution. This type of gold ores produce good gold recoveries of about 90%. On the contrary, refractory gold ores are ores which have low gold recoveries when subjected to direct cyanide leaching. The refractoriness of gold ores are dependent on mineralogical features with reference to the mode of presence and association of gold with other minerals (Celep et al., 2008). These other minerals tend to interfere with the cyanidation process and thus cause the sample to be refractory.

Refractory gold usually co-exist with preg-robbing minerals (preg-robbers) such as sulphides and carbonaceous minerals which are very difficult to be processed in cyanide leaching. Preg-robbers tend to consume the cyanide before it attacks the gold particles. This causes the cyanidation process of gold becomes less effective. Many pre-treatment processes have been developed in overcoming the problem of extracting gold from refractory ore. Recently, roasting, pressure oxidation, bio-oxidation, ultrafine grinding and modified cyanidations are being practiced as the pre-treatment processes for gold recoveries from refractory ores (Celep et al., 2009).

Serious cyanide consumption can arise from the reaction of some oxides or sulphides. These side-reactions cause excessive reagent consumption which increases the cost of production as well as decreasing gold recovery. In gold mines with significant sulphide mineralization, gold recovery by cyanidation is usually uneconomic. Sulphides also react with cyanide to give thiocyanate. Preg-robbing can also occur at low cyanide concentrations with some sulphides, for example pyrrhotite and pyrite. Under these conditions, metallic gold can cement out onto the sulphide minerals (Brooy et al., 1994).

The processing of problematic gold ore as discussed above involve excessive usage of cyanide due to the presence of other gangue elements or preg-robbers which would decrease the recovery rate of gold. Problematic gold ore refers to gold ore which is difficult to be processed due to its refractoriness. Thus, the gold processing would be uneconomical and unprofitable that would lead to an increase environmental hazards in the disposal of cyanide waste water.

In many cases, refractory gold ore specifically sulphidic ore appears to be hosted by pyrite, arsenopyrite, stibnite and sometimes pyrrhotite (Badri & Zamankhan, 2013). The major problem with sulphidic refractory gold ore is that very fine grain nature of some of the arsenical pyrite and arsenopyrite, mostly associated with silicate gangue minerals. Pre-concentrate or pre-treatment of refractory sulphide gold ore is very important for further process, such as cyanide leaching (Allan & Woodcock, 2001).

Most refractory gold ore cannot be directly treated using cyanidation. This is due to the characteristics of the ore such as passivation due to the presence of sulphide minerals, which are cyanide and oxygen consumers. Destruction of sulphide matrix to liberate the gold requires pre-treatment by oxidation where the sulphide matrix has to be pre-oxidized either by roasting or autoclaving. Both of these methods have proven to produce high gold recovery especially from refractory gold ore, however, these techniques involved high capital cost. Roasting tend to cause environmental pollution by emission of toxic gaseous while autoclave leaching having the drawback of yielding undesired precipitates due to the gold ore mineralogy (Afenya 1991).

The forgotten diagnostic leaching experiment which was introduced by Lorenzen in 1995 was being utilized in this research work to study the deportment of gold in the ore, as well as functioning as a pre-treatment to improve the recovery of gold in a problematic ore. Deportment of gold refers to the distribution of gold in an ore, based on its specification, grain size and mode of occurrence (liberation, exposure and mineral association with gold) (Coetzee et al., 2011a). As highlighted earlier, refractory or problematic gold ore is not economical to be processed by direct cyanidation, hence diagnostic leaching is proposed as a pre-treatment prior to cyanidation. This is important to increase the gold recovery as well as improving the efficiency of cyanide consumption which in return will reduce environmental and health issues due to excessive use of cyanide.

In Malaysia, gold ore processing was done using cyanide leach with some modification such as carbon-in-leach or carbon-in-pulp in which activated carbon is used for the recovery of gold. However, the problem with refractory or problematic ore will still rise if the deportment of gold is not fully understood thoroughly. Therefore in the future, diagnostic leaching experiment is still very relevant and important in the processing of problematic or refractory gold ore, especially in Malaysia.

1.3 Significant of research

Gold is a valuable mineral with various applications. Some ores can be processed directly with cyanide and some need certain pre-treatment in removing gangue minerals before recovering the gold. The significant of this research is to study the deportment of gold using diagnostic leaching experiment, in which its design is dependent largely on the mineralogical data of the ore. Mineralogical data can be obtained from a thorough mineralogical characterization. Diagnostic leaching was developed by Anglo American Research Laboratories (AARL) during the mid-1980's (Lorenzen, 1995). The concept of diagnostic leaching is to eliminate the least stable mineral in sample matrix by using selective oxidative acid leach, after which cyanidation is used to extract the gold liberated by the destruction of this mineral. The residue is then further leached in a more oxidative acid leach to eliminate other specific minerals, and the process repeated (Lorenzen and Van Deventer, 1992). At the end of this research, it is expected that diagnostic leaching experiment can be utilized to understand the deportment of gold in ore, and thus increase the recovery of gold by eliminating gangue minerals.

1.4 Research objectives

There are some objectives that this research is concerned. The objectives are listed below:

1) To conduct physical and chemical mineralogical characterization on gold ore samples obtained from Pahang, Malaysia.
2) To determine the amount of total gold, free milling gold and gangue elements in the gold ore using fire assay technique, cyanide bottle roll test and aqua regia acid digestion, respectively.
3) To apply the diagnostic leaching experiment on refractory gold ore of Pahang, Malaysia for the improvement of cyanidation process as well as understanding the deportment of gold.

1.5 Scope of research work

The gold ore sample used in this research was obtained from a local gold mine located in Kuala Lipis, Pahang. The early stages of this research involve the thin and polished sections preparation using selected rock samples. Comminution using crushing and grinding were conducted for the preparation of ground gold ore samples. Particle size distribution of the ground sample was determined to ensure the desired particle size was achieved prior to the following experimental works. Mineralogical characterization including optical microscopy (OM) study using polarizing and reflected light microscopes, Field Emission Scanning Electron Microscopy (FESEM) equipped with Energy Dispersive X-ray (EDX), X-ray Diffraction (XRD) spectroscopy and X-ray Fluorescence (XRF) spectroscopy were also conducted.

Fire assay technique was performed on the ground gold ore sample to determine the total gold content in the ore. Following this, cyanide bottle roll test was also conducted with the aim of determining the free milling gold content in the ore. From these two processes, the amount of interlocked gold can be determined and thus the refractoriness of the ore can be studied. The refractoriness of the ore is dependent largely on the mineralogical data of the ore in addition to information from fire assay technique and cyanide bottle roll test. Analysis on the residues after cyanide bottle roll test was also conducted using fire assay to determine the amount of interlocked gold remaining after the extraction of free gold from the ore. Another method used in studying the refractoriness of the ore was the aqua regia acid digestion which was conducted to determine the gangue elements content in the ore. For gold determination, the filtrate samples from cyanide bottle roll test and fire assay technique were analyzed using Flame Atomic Absorption Spectrometry (FAAS), while for filtrate samples from aqua regia acid digestion were analyzed using Inductively Coupled Plasma - Optical Emission Spectrometry (ICP-OES). ICP-OES can be used for multi-element determination at lower concentration of elements from ppm to ppb.

Based on the mineralogical data obtained from the mineralogical characterization, diagnostic leaching experiment was conducted on the ground gold ore sample to study the deportment of gold in the ore as well as utilizing it as a pre-treatment process to improve the recovery rate of refractory gold from Pahang, Malaysia. In diagnostic leaching, selective oxidative acid leach was used to solubilize the least stable minerals associated with gold in the sample matrix, before being eliminated. This was followed by a subsequent cyanidation to extract free gold from the eliminated minerals. About 50 g of ground gold ore samples were used in each run of diagnostic leaching experiment.

1.6 Thesis outline

This thesis contains five chapters. **Chapter 1** includes the introduction of the research background and also provides the objectives and problem statements related to the research, as well as its significances and the scope of the research work. **Chapter 2** reviews literature on the previous studies done related to this research, including the gold ore types, geology and mineralogy of gold, characterization techniques for mineralogical analysis and diagnostic leaching experiment. In **Chapter 3**, the materials, apparatus, equipment and experimental procedures related and conducted in the research work are explained in details. These include sample preparation and sampling method, mineralogical characterization of samples, experimental setup and diagnostic leaching experiment. **Chapter 4** discusses the data analysis and their interpretation. Experimental data were obtained from the rock and ground gold ore samples using mineralogical characterization, fire assay technique, cyanide bottle roll test, aqua regia acid digestion and diagnostic leaching experiment. Finally, **Chapter 5** gives an overall conclusion on the research work and provides recommendations and suggestions for the improvement step that can be taken in future works.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

This chapter provides information on subjects regarding gold ore based on previous research works. Firstly, gold ore classification is discussed in detail followed by the properties of gold physically and chemically. The next section of this chapter reveals the studies on mineralogy and deportment of gold in ore, which are the most important subjects in this research. After the mineralogical characterization, it was followed by chemical and instrumental analysis technique to determine the total gold content and free milling gold using and fire assay technique and cyanide bottle roll test respectively. Finally, literature reviews on the design of diagnostic leaching experiment are presented in this chapter, which includes selective oxidative acid leach and cyanidation of gold ore. All these techniques and processes are discussed in detail in relation to the work conducted by earlier researchers.

2.2 Gold ore classification

Gold ores can be broadly categorized as “free milling” and “refractory” depending on their response to cyanide leaching. In a gold mining industry, as the pits grow deeper, readily-leached oxidized gold-ore is gradually being replaced by primary ore that may be refractory, i.e. may not give economically acceptable gold recovery by conventional cyanidation. Pre-treatment of the refractory material will raise processing costs and will need to be offset by higher gold grades (Brooy et al., 1994).

2.2.1 Free milling gold ore

Ore deposits with predominantly free gold that is easily amenable to cyanide extraction have previously been defined as free-milling ores. Free milling ores (80 % of passing 75 µm size) would give a higher gold recovery with a conventional 20 to 30 hours cyanidation leach where in the same process, sufficient cyanide is added to leave a concentration of 100 to 250 ppm at about pH 10, at the end of the leach. A gold recovery of more than 90 % (Brooy et al., 1994 and Goodall et al., 2005) can be readily achieved with a conventional cyanide leaching of free milling ores.

2.2.2 Refractory gold ore

Ores that do not provide economic gold recovery with conventional cyanidation are termed refractory. Ores may not respond to conventional cyanidation for three basic reasons. Firstly, in highly refractory ores, the gold can be locked up in the mineral matrix so that leach reagents are unable to reach it. Secondly, in complex ores, reactive minerals in the ore can consume the leach reagents in side reactions and there may be insufficient cyanide and/or oxygen in the pulp to leach the gold. Thirdly, components of the ore may adsorb or precipitate the dissolved gold cyanide complex so that it is lost from the leach liquor. Some ores may have contributions from each of these factors, which will influence the processing strategy. Ore mineralogy determines the degree of refractoriness and can be graded and classified as shown in Table 2.1. (Brooy et al., 1994).

Table 2.1 : Classification of ore refractoriness (Brooy et al., 1994)

Percentage recovery	Degree of refractoriness
< 50 % recovery	Highly refractory
50 – 80 % recovery	Moderately refractory
80 – 90 % recovery	Mildly refractory
90 – 100 % recovery	Non-refractory (Free milling)

Refractory gold ores are often characterized by low gold extractions (< 80 %) in cyanide leaching. The refractoriness of gold ores and concentrates stems primarily from the inherent mineralogical features with reference to the mode of presence and association of gold and carbonaceous matter present. In practice, the refractoriness has been reported to be caused by some reasons. One of the reason is very fine dissemination (< 10 μm) or presence of gold particles as solid solution in mostly pyrite and arsenopyrite. Secondly, association and presence of gold with tellurides and base metal sulphides of lead, copper and zinc can also cause an ore to be refractory. Thirdly, refractoriness of ore can be caused by carbonaceous matter and silicate minerals present in the ore and finally, it could be due to ultrafine gold locked up in the gangue matrix (mostly quartz) or complexes with manganese oxides.

Deposits where gold is not amenable to a direct cyanide leach and requires a pre-treatment stage to liberate the gold are termed as refractory. The refractory nature of gold deposits can be attributed to a number of different factors. The most common cause of metallurgical problems is the encapsulation of gold by sulphide minerals. This problem can often be overcome by fine grinding of the ore to liberate macroscopic gold, however, in some cases the particles of gold may be too small to be effectively liberated by fine grinding or may even be in solid solution. When this occurs, gold is termed as ‘‘invisible gold’’, which has been defined by Boyle (1979) as colloidal or chemically bound gold that is undetectable by microscopic techniques (Atr and Swash, 1988). ‘‘Invisible gold’’ is generally seen in arsenopyrite or arsenic rich pyrite. A number of studies have determined the mechanism of occurrence of ‘‘invisible gold’’. ‘‘Invisible gold’’ will only occur in pyrite once a certain bulk concentration of arsenic has been reached, and also be concentrated in arsenic rich rims of pyrite (Dunn and Chamberlain, 1997). It has been suggested that in arsenopyrite, depletion of Fe with gold concentration suggests Au substitutes for Fe in the sulphide matrix (Zhou and Cabri, 2004) and in pyrite the incorporation of As into the matrix could lead to distortion of the lattice allowing the incorporation of gold (Goodall et al., 2005).

Refractory gold and silver ores yield low gold and silver extractions in cyanide leaching. A suitable pre-treatment process such as roasting, pressure oxidation, bio-oxidation, alkaline or alkaline sulfide leaching and, to a limited extent, ultrafine grinding, is often required to overcome refractoriness and render the encapsulated gold and silver accessible to the lixiviant action of cyanide and oxygen (Celep and Serbest, 2015). Gold deposit is known to be problematic in its beneficiation properties, because most of the gold is present in the ore as "invisible" gold in pyrite and arsenopyrite (Labtium Ltd, 2016). Hence, the recovery of gold in a pilot cyanide leaching test was only 7% from a feed of 10g/t Au. Because of the problematic nature of the gold distribution, the exploration was stopped at Suurikuusikko for several years (Kojonen and Johanson, 1999). From the work done by all these researchers as discussed earlier, the processing of refractory gold ore would require high operating cost in the recovery of gold.

2.3 Geology, mineralogy and deportment study of gold

Gold occurs primarily in quartz veins and as placers in soil (eluvium) and stream sediments (alluvium). The quartz veins containing gold occur in association with metamorphosed rocks ranging in composition from semipelitic to pelitic and mafic. Pelitic refers to the characteristics of a metamorphosed fine-grained sedimentary rock, for example mudstone while semipelitic refers to pelite rocks having similar chemical composition but being of a crystalloblastic nature. Mafic on the other hand refers to the characteristics of a silicate mineral or igneous rock that is rich in magnesium and iron.

In general, gold mineralization can be classified into three types. Firstly, gold occurances in sheet and stockworks quartz -carbonate and tourmaline veins carrying significant amount of sulphides such as pyrite, arsenopyrite, and trace of pyrrhotite, galena and hematite. Secondly, gold disseminated within stockwork of quartz-carbonate veins affiliated with tonalite and with graphite-ankerite-quartz intrusive rock, and gold was complicated occurrence as very fine grain size which is associated with pyrite and arsenopyrite, and low amounts of galena and sphalerite. Thirdly, gold associated with arsenopyrite and pyrite in quartz-carbonate veins and stringers (Chye et al., 2001 and Ariffin & Hewson, 2007).

Primary gold mineralization produced chemical signature in the overburden and surrounding soil probably through weathering processes. Weathering processes provide samples (soils and stream sediments) that yield data on local hidden mineralization or on the potential existence of major or minor mineralization in a wide region. The residual soil is the geochemical sample that is often used to detect the location of hidden mineralization once a zone of economic interest is localized.

Migration of groundwater provided chemical response at the surface. This process produces elemental dispersion pattern. Most of these dispersed elements (e.g., Cu, Ag, Zn, Cd, As, Bi, Pb, Sb, Hg, W, Mo. and Se) are useful indicators or pathfinders for the presence of gold. Analyses of samples taken would enable the observation of patterns and concentrations in the distribution of metals in the soil and potentially would indicate enriched rock underneath (Oke et al., 2014).

Gold occurred in various grain size, and some gold particles were entrapped in very fine grained minerals (Petruk, 2000). In some quartz veins, gold was commonly found as coarse grain around 1 to 2mm, and small amount of gold occurred as disseminated particle range from 100 to 200µm infilling fracture of quartz-carbonate as well as sulphide minerals (Ariffin & Hewson, 2007; Alp et al., 2008; and Makoundi et al., 2014). In sulphide minerals, gold is mostly associated with pyrite in native form as inclusions in size range 1 to 50µm, which is also associated as sub-round inclusions in arsenopyrite and galena within the size range of 1 to 20µm. In addition, gold was also existed with pyrite along the boundaries of non-sulphide gangue. The average of invisible gold contents in pyrite and arsenopyrite varies from < 2.2 to 585 ppm, and in arsenopyrite from < 2.2 to 964 ppm (Kojonen & Johanson, 1999).

It is known that different minerals from various locations have unique mineralogical composition with varying characteristics and complexities. At this point, it is expected that the understanding of mineralogy, chemical composition, size, morphology and association with other minerals, through in-depth characterization, will provide an indication/basis on the mineral's behaviour during beneficiation and recovery processes (Makoundi et al., 2014). It is evident that from literature, the northern Pahang area is divided into Devonian metasediments, acid and intermediate intrusive structures and various sections of argillaceous, calcareous and volcanic

Permian facies (Ismail et al., 2016). These provide sufficient geological information to the formation of ore in Pahang to assist in the understanding of the mineralogy characterization of the gold ore received.

In terms of mineralogy, gold could be interlocking by other host mineral in a number of ways. Firstly, gold as physical locking in sulphides, oxides, silicates, etc. Secondly, chemical locking as gold alloys or compounds e.g. electrum, gold tellurides, $AuSb_3$ (aurostibnite), Au_2Bi (maidonite), etc. Another way is the gold substitution in the sulphide lattice e.g. "solid solution" gold in arsenopyrite and finally gold surface passivation due to formation of a chemical layer (Brooy et al., 1994).

Gold can be associated with a variety of minerals within an ore matrix in many different ways. Knowing these associations can be vital in the optimization of the gold recovery route. The occurrence of gold can be in the range from large nuggets of free gold generally found in alluvial deposits to sub-micron sized particles or even gold in solid solution with the ore minerals (Goodall et al., 2005).

2.4 Gold geology and mineralization in Pahang, Malaysia

Malaysia is a country blessed with a lot of gold deposits, especially in the Peninsular area. Gold production in Malaysia has come widely from the states of Pahang, Terengganu and Kelantan. Major gold production in the Central Belt cover deposits of Berching-Chinong, Ulu Sokor, Ketok Batu, Selinsing, Tui, Penjom, Tersang, Raub, Pason, Kanan Kerbau and Batu Bersawah (Yeap, 1993 and Ariffin & Hewson, 2007). Selinsing Gold Manager and Penjom Gold Mine in Pahang are the major gold producers which contribute a lot in Malaysia’s economy due to the high price and popularity of gold (Pui-Kwan, 2013). According to Yeap (1993), the Peninsular Malaysia Central Belt was recognized as the Golden Belt since the belt has large gold deposits which were revealed to be Permo-Triassic and having low grade metasediments as formed in a shallow marine depositional environment.

In Malaysia, the ore genesis studied by Ariffin & Hewson (2007) describes the ductile deformation of foliation developed in carbonaceous shale and led to the mineralization of pyrite and arsenopyrite that later having extremely fractured and brecciated due to the hydrothermal activities which contributed to the intrusion of silica-rich fluid and caused the formation of quartz-carbonate vein. This brittle deformation basically resulted to the deposition of sphalerite, tetrahedrite and chalcopyrite. This dilatant zone also been accumulated which causing the precipitation of several minerals like electrum, molybdenum and others. The gold mineralization as well filled up in that zone soon after the deposition of sulphides except galena which occur at the end of gold occurrences.

Gold mineralization in Central gold belt occurred as a low grade meta sedimentary volcanic terrain, and this belt can be classified into two types: type I (gold belt 2) and type II (gold belt 3). The type I belt mineralization is identified as the gold geochemical zone which provides two majors goldfields, the Buffalo Reef (Kanan Kerbau) and further south, the Selinsing Gold Manager and the Tersang alluvial goldfield (Yeap, 1993). However, the type II mostly occurred as marine classic sediments, intermediate to acid volcaniclastics, and subordinate rhyolitic lava sequences, like Penjom gold mine. The type II mineralization belt is gold bearing with galena-tetraherite-tellurides ore, gold bearing with arsenopyrite and pyrite, and gold bearing pyrite (Ariffin, 2012). The Selinsing Gold Manager deposit is hosted by meta-sedimentary units (Yeap, 1993; and Makoundi et al., 2014).

Pui-Kwan (2013) stated that there were more than 10 operating gold mines in Malaysia, which were located in the States of Kelantan, Pahang, and Terengganu, and more than 90% of gold in Malaysia was mined was from many areas in Pahang, such as Penjom gold mine in Penjom, the Selinsing Gold Manager in Sungai Koyan, and Raub gold mine in Raub, and they were the main role to lead the country's economy.

Gold mineralization in the Central Belt associated mainly with hydrothermal quartz vein system including skarn and volcanogenic massive sulfides which are commonly arsenopyrite, pyrite, galena and sphalerite, and the origin of gold was come from volcanic exhalative during volcanic activity in the Permian to Triassic (Ariffin, 2012; and Li et al., 2015). Anyway, the mineralization in those areas are still uncertain in the researcher's understanding, and gold resources cannot be used directly from mine sites due to the impurities, especially sulfide gold ore (Adams, 2016). The relationship between the gold mineralization of sulfide ore and silicification is so complicated, thus the mineralogical characterization of gold ore needs to be well carried out before starting with the recovery processes.

The gold mineralization in Ajmal mine, Pahang Malaysia was found that quartz veins as parallel veins in shear zone. Native gold is fine grain, and it is found as gold bearing veins as sulfide-poor associated with tertrahedrite and some galena, chalcopyrite and sphalerite and petzite. The wall rock alteration is visible and associated with silicification. The mineral deposit occurred at low temperature and was far from the sources (Hassan, 2008).

Wan Fuad et al. (2008), stated that the lithology in Selinsing gold mine area which seemed to be broadly related to low grade metasediments as it consists of three main units; calcareous unit where shale-limestone becomes the host layer, sediments unit that made of shales, claystone as well as siltstone and lastly tuffaceous rocks unit which composed of shale and siltstone tuffaceous as host layer and also lithic tuff-sand. Prior to the geological setting and the tectonic, the gold belonged to the low mesothermal lode gold type (Ariffin, 2012) where the hydrothermal fluid was act as a conduit that carries gold and other metallic minerals from the hydrothermal activities. The precipitation of the fluid causing the deposition of the metallic minerals in fractures and forming a vein, such as quartz vein.

The gold formation, for instance, in Selinsing occurs at 1-5 km depth at 200 to 300 ℃ with moderate pressure and this gold mineralization in quartz vein became the primary deposit with fine grain sized of gold particles and often found interlock in sulfide minerals like pyrite, arsenopyrite and chalcopyrite (Ariffin, 2012), where they are particularly found stable in the anaerobic dry environment. The secondary ore deposit is the deposit that had undergone weathering process either through supergene or oxidation process. The oxidation process usually take place at the outcropped of the surface, for example, from the weathering process, several common minerals are formed such as hematite, goethite and limonite and these hydrated oxidized iron minerals were resulted from the oxidation of the metallic sulphides.

2.5 Characterization techniques for mineralogical study

The mineralogical study of an ore for characterization involve X-ray Fluorescence (XRF) and X-ray Diffraction (XRD) technique to determine elemental composition and mineral phases which are present within the ore respectively. In addition, Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray (EDX) technique can be used to study the ore morphology and elements present in the ore.

Accurate characterization of an ore is essential to identify the mode and occurrence of gold and gold bearing minerals and hence, to evaluate the refractory behaviour of gold in the traditional cyanidation process. Mineral liberation size of the ore, mineral associations and modal of mineralogical analysis are a pre-requisite for plant design and mineral processing operations (Celep and Serbest, 2015). With this information, it is then possible to determine the amenability of an ore to different processing options.

In the optical microscopy study, samples were mounted with sufficient mixture of epoxy resin and hardener as the matrix, were subsequently cut, and followed by mechanical polishing. Initial polishing was achieved with a rotary grinder (Metaserv), using 180, 320, 600, 1000 silicon carbide grits, followed by diamond lap polishing with oil-based lubricant. The specimens then were washed with ethanol, prior to visualization of optical properties and surface morphology (Ismail et al., 2016).

A mineralogical examination normally suggests reasons for the refractoriness of the ore. Craig and Vaughan (1994) identified specific mineralogical techniques, which can be used for specifying ore type and for distribution of the gold in the ore. These techniques are listed in Table 2.2.

Table 2.2 : Mineralogical techniques for locating gold in the ores (Craig and Vaughan, 1994)

Technique	**Comment**
Optical Microscopy	Suitable for coarse gold (> 0.5 μm)
X-ray Diffraction	Identification of free mineral grains
Scanning Electron Microscopy	Elemental analysis
Electron Microprobe	Lower detection limits for gold in solid solution (10 – 20 ppm)
Ion Microprobe	More sensitive for solid solution gold, claimed to 1 ppm

In this research work, the mineralogical techniques that have been used to characterize the gold ore received were optical microscopy, X-ray Diffraction and Scanning Electron Microscopy.

Deportment of gold refers to the distribution of gold in an ore, based on specification, grain size and mode of occurrence (liberation, exposure and mineral association). This study is important in locating and describing the gold-containing particles as well as to generally characterize the mineralogical composition of the ore. With the information obtained from gold deportment study, problematic or refractory ore can be treated by means of pre-treatment which can be designed based on the diagnostic leaching experiment (Coetzee et al., 2011b).

2.6 Chemical and instrumental analysis technique for gold determination

The nature of many gold deposits (low grade-high tonnage) and the increasing the sophistication of deposit assessment procedure and exploration geochemistry demand better quality analytical data, particularly for the lower range of the gold values near and below cut-off grade. Fire assay still remain as the best assaying method for gold. Before any chemical and instrumental analysis, accurate method of obtaining representative sample is important from the large bulk mass sample to produce a smaller mass sample for analysis since the small mass should be representative of the material submitted for analysis (Fulton, 1907).

2.6.1 Cyanide bottle roll test

A cyanide bottle roll test is a test method conducted in determining the free gold after dissolution using sodium cyanide as the lixiviant. The results from the test offer information on estimated recovery rates, reagent consumption and accurately predict the results obtainable from pilot and industrial plants. The recovery results are measured maximums because attrition grinding creates fines and liberates recoverable values that would not be liberated from the material through a static leach. In standard procedure, the cyanide bottle roll tests are conducted on ores as coarse as 50 mm (Oraby and Eksteen, 2015).

The cost to conduct the cyanide bottle roll test is cheaper compared to column percolation heap leach amenability test. The most vital thing to conduct the experiment is to ensure the pH of the solution ranging from 10.5 to 11 before the experimental run. The cyanide bottle roll test involved placing the cyanide solution in bottle containing ore for a 24 hours run with intermittent sampling and finally the solution is filtered to obtain the pregnant solution containing the aurocomplex for free gold determination (Inspectorate et al., 2013).

There are several information and data obtaining from the preliminary cyanide bottle roll test including the amenability to heap cyanidation processing, presence of coarse (visible) gold particles and gold bearing sulphides minerals, clay content which indicate agglomeration before heap leaching, 'preg-robbing' character of ore and the degree of to which values are liberated from the various particles size. In this research work, the cyanide bottle roll test was conducted at the initial stage to determine the free gold before diagnostic leaching experiment.

2.6.2 Fire assay technique for total gold content

The principal advantages of fire assay technique include the long history of success of the method, easy adaptation to a mine setting, moderate cost and rapidity with which results can be obtained. Certain matrices, for example, sulphide-bearing rocks or rock with higher levels base metal, must be pretreated and rock of different composition must be properly fluxed to achieve good recovery of precious metals.

To determine the total gold content, fire assay is an effective way for the concentration of 0.001 to more than 50 g/t. Fire assay is a primary component in many parts of the distinctive study as it offers bulk gold data, to tribute the mineralogical data achieve from the earlier characterization methods (Zhou et al., 2004).

Table 2.3 shows the flux composition used in fire assay technique (Marsden and House, 1992).

Table 2.3 : Flux composition for fire assay (Marsden and House, 1992)

Chemical compound	**Weight (g)**
Sodium Borate ($Na_2B_4O_7$)	30
Sodium Carbonate (Na_2CO_3)	40
Calcium Fluoride (CaF_2)	10
Lead Monoxide (PbO)	40
Silica (SiO_2)	30-40
Flour	5.5

In reserve estimation, it is common practice to reduce the sample size to one assays tonne for analysis, although large and small amounts are used in certain cases. The basic procedure for fire assay involves mixing an aliquot of powdered sample (10 g, 15 g, 30 g, or 50 g are the common size used) with soda ash (calcium carbonate), borax (sodium borax), litharge (PbO), flour (baking flour used to add carbon as reductant), silica and possible nitrate (potassium nitrate). To this mixture, Ag or Pd as a collector can be added in solution (Fulton, 1907).

The well mixed material is fired at temperatures ranging from 1000 ºC to 1200 ºC. As the Pb and Ag in the melt settle to the bottom of the crucible, it scavenges the Au from the melt. The hot molten mixture is poured into a mold when cool; slag