SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING

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MINERAL COMPOSITION ANALYSIS OF MALAYSIAN SULPHIDE GOLD ORE

By

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DECLARATION

I hereby declared that I have conducted completed the research work and written dissertation entitled “Mineral Composition and Phase Analysis of Malaysia Sulphide Gold ore”. I also declared that is has not been previously submitted for the award of any degree or diploma or other similar of this for any other examining body of University.

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Date : 22th June 2017

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ABSTRAK

Dalam penyelidikan ini, Buffalo Reef terletak di daerah Selinsing dimana terkenal dengan kejadian mendapam bijih emas yang berdekatan dengan Sasar Umum yang terdapat di Semanjung Malaysia. Mineralogi dan bijih paragenesis merupakan ppunca utama idea untuk mereka bentuk pemprosessan mineral dalam pemprosessan bijih emas. Objektif utama dalam penyelidikan ini untuk mengenalpasti tentang komposisi minera; dan fasa tentang sample bijih sulfide dari tempat yang berasingan di Buffalo Reef. Selain itu untuk mengenal pasti tentang mineral yang berkaitan tentang bijih sulfida dengan mengunakan microscope dan ujikaji SEM. Persampelan dengan rawak telah dilakukan di zon utara, dan zon selatan dan zon utara. Selepas persediaan bijih telah dibuat, analisis proses mineralogy telah dibuat. Komposisi mineral telah dikaji dengan mengguna kan XRF dan penentuan kadar taburan saiz untuk setiap sample. Penyelidikan morfologi telah dilakukan melalui sampel kilauan permukaan untuk SEM dan mikroskope. Pengiraan parameter yang optimal bagi pengilanganmelaui ujikaji eksperimen dengan menggunakan Rekabentuk Eksperimen tentang dua sampel yang berlainan zon. Pembebsan sulfide mineral telah ditentukan melalui dengn penggiraan nilai ambang di dalam Image J.

MINERAL COMPOSITION ANALYSIS OF MALAYSIAN SULPHIDE GOLD ORE

ABSTRACT

In this study, the Buffalo Reef situated at the Selinsing district is famous with the occurrence of gold ore near the Central Belt of Peninsular Malaysia. The mineralogy and paragenesis of ore deposit is main idea for establish better mineral processing for gold processing. The objective of this research is focus into determination of mineral composition and mineral phase of Sulphide ore samples from different location in Buffalo Reef. Besides that, the sampling is done at the North and South of Buffalo Reef by grab sampling method. Mineral phase analysis is done using the analytical method after the ore preparation using XRD. Mineral composition is study using XRF and determination of particle size distribution after the sieve for each of samples. The morphology study is done via the study of polish section for SEM and optical microscope. The optimum of milling parameter is investigated via the experimental work using DOE of MINITAB using two different type of sample The liberation of mineral sulphide is determine by using the image analyzer, Image J to calculate the threshold value.

Chapter 1:

INTRODUCTION

1.1 Significant Of Research Work.

Malaysia is one of the country which enrich with enormous and various mineral. The most common mineral was identified is , bauxite, clays, coal ,copper, gold ,ilmenite ,iron ore ,limestone, monazite, natural gas, petroleum, silica, silver ,tin and zircon according to 2013 Minerals Yearbook of Malaysia written by Pui-Kwan Tse. Gold is a precious metal which involve in many industries as a part of modification or advanced technologies mainly in electronic and spacecraft manufacture. In Malaysia, the gold mineralization in Central Belt is regarded as high grade quartz-carbonate-gold type related to a phyllic propylitic alteration of granite intrusion complex that intrusive weakly metamorphosed green schist facies sedimentary strata.{} Signs of mineralization such as extensive wall-rock alteration that give rise to carbonate and alkali metosomatism.

The Malaysian sulphide gold ore mostly occur at the state of Pahang as example at all the region of Selinsing. At Buffalo Reef, the gold mineralization is hosted by widespread occurrence of low regional grade metamorphism and subdivide into three section which is Buffalo Reef Centre, Buffalo Reef North and Buffalo Reef South. The various lithological and geological occurrence cause the alteration of diversity of mineral composition in rock or soil sample. The sulphide gold ore is a complexity ore which intend a specific observation and study to extract the ore from hard rock mining or alluvial mining (Joe Zhou, Bruce and Chris,2004)

Gold ores are commonly classified by the local metallurgist into two categories which is free milling and refractory ores. Typically, free-milling ore are defined as those where over 90% of gold can be recovered by conventional cyanide leaching and refractory ores are defined as those that give low gold recoveries or give acceptable gold recoveries only with the use of significantly more reagents ore complex pre-treatment processes (Joe Zhou, Bruce and Chris,2004). The gold is associates with the sulphide mineral mostly iron sulphides which include pyrite, arsenopyrite, and pyrrhotite and its floatation recovery is dependent on recovery of the associated minerals. In this case gold follows the associated sulphide mineral recovery (Celep, OktayAlp, İbrahimDeveci, Hacı,2011).

The mineralogy of Malaysia sulphide gold ore is done to investigate the potential unleachable of sulphide gold ore. The ore deposit and mineralogy that associate to occurrence of gold mineral in massive sulfides to placer and palaeoplacer deposit. Most of the vein is dominated by the gold and silver may or may not directly associated with intrusion.

The sulphide gold ore is a complexity ore which intend a specific observation and study to extract the ore from hard rock mining or alluvial mining. The further study of Malaysia sulphide gold ore can enhance the research of mineralogy and mineral processing which involve gold mineral in the form of sulphide mineral. Due to this, the production of gold by the mine company can be increase.

1.2 Problem Statement

Due to slightly different occurrence of geological structure between Buffalo Reef and Selinsing area, the mineralogy of sulphide gold ore is also different in soil or hard rock in present of gold mineralization. Although the deposit type is mesothermal gold ore deposit and mostly associated with pyrite, arsenopyrite and galena, the variation of

mineral composition is vary with different section at Buffalo Reef. The different location of sample collected can cause the variation of mineral composition. This characterization process covers the study of morphological features such as shape, grow patterns, size, the inclusions or heterogeneities within grains, as well as detailed in morphology analyses of previous research.

Most primary sulphide ore bodies have mineral assemblages that are unstable in near-surface conditions. At Buffalo Reef, the assemblage of argililite and limestone is a dominant structure with 200m wide range, north-south striking shear zone which parallel the tectonic Raub- Bentong suture to the west and within the gold mineralization occurrence which give the different level of oxidize zone within the ore body for North and South region (Naidu, 2005). The presence of numerous sulphide in the ores resulting in difficulty in extraction of gold due to its natural resistant to recovery by standard cyanidation and carbon adsorption. The process of metallurgy require pre-treatment process which is roasting, bio-oxidation, pressure oxidation and albion process.

The aim of the project to determine the mineral composition and the phase analysis of Malaysia sulphide gold ore using x-ray diffraction ,x-ray fluorescence with the advance analysis data using Expert High Score Plus and scanning electron microscope.

1.3 Objectives

Recent research on the mineralogy of sulphide gold ore in Malaysia is done by previous researcher and the reading of geological structure present at Buffalo Reef in state of Pahang shows some insufficient information. The objectives of this research are:

- To study mineral composition and mineral phase of Sulphide gold ore sample from different location in Buffalo Reef (Mineral phase identification and quantification using X-pert High Score plus Software).
- To determine the optimum milling parameter and its effect toward phase analysis.

- To study the mineral liberation of Sulphide Gold Ore (Mineral liberation studies using threshold value of J Image).

1.4 Scope Of Study

This scope of study mainly focus on the bulk characterization and different size fraction characterization follow by the optimum parameter of milling using planetary ball mill. The sample is a raw sample which directly taken from the site of the Buffalo Reef North and Buffalo Reef South. The techniques of sampling is a proper grab sampling method. Sulphide ore with different grades and types were undergo these characterization

This thesis concerning the particle-size distribution and the determination of optimum milling parameter. These bulk characterization analysis included sieve size analysis, elemental composition analysis (XRF), phase identification (XRD) and morphological study using optical microscope and SEM EDX of polish section on selected size fraction.

After doing sieve size analysis, the certain size selected to proceed with the polish section for morphology study of these samples for their properties that may exhibit by optical identification and mineral liberation observation under polarizing ore microscopy and scanning electronic microscopy (SEM). With the help of EDX, the semi-quantitative weight percentage of selected particle were detected

1.5 Thesis Organization

Thesis consist of five chapter which started with introduction on the research background with the aim of research follow by the literature review on the general related facts within the thesis title, the Malaysia sulphide gold ore which compile of information on the previous and current work regarding to the work project. Literature review is done by the two sources which is from the articles and journals of previous researcher and cited to protect the individual's copy right.

Chapter three is elaborate about the methodology been used in duration of research been done, starting from the proper sampling method, follow by the communition, pre-assessment of polish section, optimum grinding parameter, x-ray diffraction, x-ray fluorescence and scanning electron microscope. The result and discussion is display in the chapter four after all the data have been analyse. All method is given in flow chart for further any inquiry and information regarding to the project.

Finally, the conclusion is made regarding the results performance in this research. The recommendation for the future work also being explained in this chapter.

Chapter 2:

LITERATURE REVIEW

2.1 Mineral

A mineral is naturally occurring inorganic substance with ordered arrangement of atoms. Mineral can be classified into several mineral groups which is native elements, sulphides , oxides, halides, carbonates, phosphate and silicates. Thus, for further explanation in mineral and mining industrial which is important in world economic around the world, there is two type of mineral which can be extracted which is ore mineral and gangue mineral .The definition of ore mineral is a mineral that may be extracted profitably from an orebody and it classified into two group which is metallic mineral and non-metallic mineral.

2.1.1 Gold Mineral

Gold is a chemical element with the symbol Au and the atomic number 79. In its purest form, it is a bright, slightly reddish yellow, dense, soft, malleable and ductile metal. Chemically, gold is a transition metal and a group 11 element and period 6. Gold is one of the most popular and well-know minerals, for its value and special properties since the earliest of time. Gold has a density of 19.3 g/cm3 and form in solid state and the melting point and boiling point for pure gold is 1337.33 K and 3243 K respectively. Gold is one of the heaviest minerals and due to that, it can be panned because the gold sinks to the bottom. In addition, it can be easily separated from other substances due to the weight differences. Gold is also one of the most resistant metals. It won't tarnish, discolor, crumble, or be affected by most solvents. This adds on to the uniqueness and allure of this mineral.Gold is usually associated with Pyrite and other Sulfides and sometimes may not be noticed because of the association with these resembling minerals.

Gold is one of the most economically important metals produced. As of 1991, more than 83% of gold consumption was for jewelry, 6% was used for medals and official coins, 6% was used in electronic equipment, 2.2% was used for dental materials, and 2.8% was consumed in a variety of industrial applications. These markets support an annual gold production of about 2,200 tons worth almost $25 billion ([Kesler, 1994]). Globally, world gold production exceeded 2478 metric tons, or 79.667 million troy ounces in 2004 (Goldsheet Mining Directory - World Gold Production (more info)).Gold is precious metal which used as investment, for advanced technology and reserve asset management. The price for gold now days is about RM 182.03 per gram and its price intend to increase intensely due to the demand and supply of gold.

2.1.2 Geological Of Gold

Alluvial gold is one of the deformation of gold in physical state which high weathering properties and one of the placer deposit. Placer deposit is an accumulation of valuable minerals formed by gravity separation during sedimentary process. Placer materials must be both dense and resistant to weathering processes. To accumulate in placer mineral particles must be significantly denser than quartz (SiO2) and mainly form in river or stream sediments. Typical locations for alluvial gold placer deposit are on the inside bends of rivers and creeks. In natural hollows, at the tie break of slope on a stream, the base of an escarpment , water fall and other barrier (JMitchell,Ej Evans,& MT Styles,1997).

Alluvial placers are formed by redeposition of dense particles of a site where water velocity remains below that required to transport them further. To form a placer deposit, the particles desired must show a marked density contrast with the gangue material, which able to be transported away from the trap site. Most alluvium is geologically very young (quarternary in age) and is often referred to as “cover” because these sediments obscure

the underlying bedrock. Most sedimentary material that fills a basin that is not lithified in typically lumped together as alluvium (JMitchell,Ej Evans,& MT Styles,1997).

2.2 Sulphide Gold Ore In Malaysia

Malaysia had already established itself as one of the important producer ling before the development of the great gold-fields. The majority of the gold production apparently came from the states of Pahang and Kelantan within the Central Belt. Gold mineralization in the Central Gold Belt is generally categorized as a low mesothermal lode gold deposit due to its tectonic and geological setting[1]. In Raub, Selinsing and Buffalo Reef are among the old alluvial mining goldfields which are actively being revisited for the existent of low grade bulk-mineable gold deposits. The focus coverage area of study is Buffalo Reef. Small scale mining at Buffalo Reef dates back to the early 1900s[1]. The buffalo Reef deposit occurs approximately 1 km to the east of the Raub-Bentong Suture- a major geological and is dominated by eastern assemblage of conglomerate and sandstone of Denovian age. Low grade regional metamorphism up to Green schict facies occurs throughout the area . The sedimentary rocks have subsequently been intruded by granitic bodies of approximately Jurassic age. Outcropping rocks of these intrusive bodies occur to the east of Buffalo Reef and generally from the elevation highs (Gondwana Research,2012)

Gold mineralization at Buffalo Reef is structurally controlled and associated with Permian sediment within a 200 m wide shear zone that parallels the north- south trending Raub-Bentong Suture. Mineralization occurs overall total strike length of 2.6 km. Rocks, within the Buffalo Reef shear zone have typically undergone silica-sericite-pyrite alteration to varying degrees (Kamar Shah Ariffin,2006).

The occurs within moderately to steeply east-dipping veins and fracture zones, which range in thickness from 1 m up to 15 m in thickness, although local flexures in the veins can host mineralization up to 25 m in thickness. Veins which boudinaged in some areas, are generally composed of massive quartz with 1-5% sulphide minerals, namely pyrite and arsenopyrite along with varying amounts of stibnite, the stibnite generally occur in association with elevated gold grades; however the presences of gold does not necessarily indicate high stibnite levels (Vb, Vancouver B C Canada,2013).

2.3 Gold Recovery Methods

There are many ways to recover the gold form the ore either in physically or chemically extraction. The methods considered mainly involve the physical separation of gold from "gangue" mineral using gravity separation methods. Gold has a high specific gravity (19.3 g/cm^3) in relationship to most common gangue minerals and therefore it suitable for gravity processing. The high volume of throughput of alluvial gold ore is the suitable for this kind of mineral processing. The factor of certain gold grain characteristics influence the efficiency of gold recovery methods especially for the gravity separation. The influence of density upon the behaviour of a gold grain will lessen as the surface area to mass ratio increases. Gold is typically flakier with decreasing grain size and not spherical. The malleability of gold keep the shape of gold maintain rather than fracturing in response to loading and impact. This irregular shape leads to porosity; cavities and pores are often infilled with lower density material lowering the density of the composite particle. The flaky shape, porosity and hydrophobic surface properties can often cause the gold to float and it is a major problem for fine grained gold. The mineralogical character of the gold is often not considered when planning a processing plant if the gold is corresponding well to standard gravity and cyanidation processes. However, if the

recovery percentage of gold is poor (<80%) the ore is termed “refractory” and a detailed mineralogical investigation becomes necessary (JMitchell,Ej Evans,& MT Styles,1997).

2.3.1 Sluices box

The sluice box is most common methods for gravity separation of gold from alluvial gravels. They are generally cheap to make, easy to operate and require minimal technical knowledge to maintain (Hannock,1991). Essentially a sluice box consist of slopping open rectangular flume with regularly spaced transverse bars through which a dilute slurry of water and alluvial gravel flows. Gold and other heavy minerals are usually trapped in the upstream side of the bars. These are regularly removed by raking or cleaning-out the sluice box riffles.

2.3.2 Jigs

A jig is described as a ‘hindered settling device used to concentrate heavy minerals from feed material’. It consist of a shallow, flat tray with a perforated bottom plate through which water is pulsed up and down. This cause the heavy minerals to migrate downwards and the lighter minerals to remain at the top of the pulsing liquid. The heavy minerals are drawn through the base plate and the light minerals pass over the top as tailings. Jigs are effective in the processing of material in the size range 25mm to 75µm. Material is best pre-screened and processed as separate coarse and fine fractions.

2.3.3 Shaking Tables

The wet table is the common form of shaking table which is water as a medium of separation. It consist of a flat table or deck with parallel riffles to trap the heavy minerals. The deck is vibrated longitudinally and inclined laterally during operation. A perforated pipe feeds wash water from the upslope side. The slurried feed is introduced at the top upslope corner. The minerals in the feed segregate. The heavy minerals sink to

the deck, migrate along the riffles and are discharged over the end of the deck. The light mineral, entrained in the water, pass straight over the riffles and down to the bottom and so to the tailings. It is suitable and effective in the processing of material in the size range 3mm to 15µm.

2.3.4 Spiral

The spiral concentrator is described as a "low feed rate, low feed density" flowing film gravity separator. It consist of a helical conduit of modified semi-circular cross-section, usually with between 3 and 5 complete 'turns' (Wills,1992). Material is fed onto the top of the spiral as a slurry with typically 25% to 30% solids by weight. As the material flows spirally downwards the particles stratify due to factor such as centrifugal force, differential settling,hindered settling and reverse classification. There is usually a density gradation across the profile of the spiral with heavy minerals concentrating next to axis and minerals of lower density being swept to the outer edge. Concentrate,middling and tailing products are collected with use of adjustable splitter plates. The effective size of gold feed is range between 3mm and 75µm and most efficient.

2.3.5 Bowl Concentrators

Commonly used makes are the Knudsen and the Knelson bowl concentrators. A bowl concentrator consists of a rotating cylinder that segregates heavy minerals from light minerals by a combination of centrifugal force and wash water action. The knelson bowl concentrator is claimed to recover "gold particles ranging from 6mm to less than one micron in a single pass". Recovery is effective down to approximately 30µm.

2.3.6 Drum Concentrators

The Mozley Multi-gravity (MGS) consists of a tilted drum that tapers slightly to the downslope end. The drum simultaneously rotates and is shaken longitudinally.

Sample slurry is added to the upslope end and as it migrates down the drum it segregates into heavy and light minerals. The heavy minerals report to the inside wall of the drum, where they are pushed to the slope end by scarpers. The light mineral remain entrained in the wash water and report to the downslope end. The MGS has been likened to as shaking table wrapped round itself into a drum. The MGS can be used to process material in the size range 1mm until to $1\mu m$

2.3.7 Froth Flotation

Froth flotation is a widely-used processing technique which exploits different in the surface properties of minerals. Minerals with hydrophobic surfaces (either in natural or due to chemical treatment) attach to air bubbles passing through a suspension and float to the surface to form a froth. Gold grain surfaces are often coated with an hydrophobic organic layer or iron oxide coatings and some are leached free of impurities (such as silver) leaving a rim of pure gold, all of these render the surface hydrophobic.

2.4 Site Background Description

2.4.1 Selinsing Goldfield.

This is an active goldfield located in NW Pahang, Peninsular Malaysia. Mining at Selinsing commenced prior to 1888 and has operated intermittently through to 1966 (Johnston, 1998). Underground and open cut mining, together with tailings treatment, has produced an estimated 85,000 ounces of gold during this period. Lithology of the area consists of low-grade metamorphosed sedimentary and volcanic rocks of Gua Musang Formation of Late Permo-Triasic age. Wall rock alteration in Selinsing Gold mine shows a direct relation with hydrothermal solution, structures, formation of quartz veins and gold mineralization.The Selinsing deposit occurs along the north striking Raub Bentong Suture. The deposit is hosted by a series of auriferous quartz veins and stockworks of quartz veinlets in a package of sheared calcareous epiclastic sediments. The gold

mineralisation occurs in quartz veins that cut the host rocks and wall rocks with intensive alteration that are related to the N-S and NE-SW lateral faults and shear zones (Mohd Basril et al., 2009). Gold mineralization at Selinsing is associated with high grade quartz veining and accompanied by strong sericitization and silicification within a major shear zone. Formations of quartz veins are mainly related to the right lateral faults. Minerals often associated with gold mineralization are pyrite, arsenopyrite, chalcopyrite, tetrahedrite and sphalerite.(Hengl & Hoe 2006) Brecciation and cataclasite within shear zone are very prominent and show sensible relationship with mineralization. The host rocks of the deposit consist of a series of argillaceous and arenaceous of likely epiclastic origin, which have undergone low temperature regional metamorphism. This metamorphism has had little effect on their original mineralogy or texture. The ore samples show epigenetic gold and sulphide mineralisation in quartz veins with no direct relationship to the sediments hosting the quartz veins (Vb, Vancouver B C Canada,2013).

2.4.2 Buffalo Reef

Small scale mining at Buffalo Reef dates back to the early 1900s with 1,000 meters of underground workings developed in 1934, including adits, drifts, crosscuts, winzes and shafts. Production details from that time are not available and assumed to be relatively small. Antimony in the form of stibnite occurs in the gold-bearing veins at Buffalo Creek. Stibnite was mined in the 1970s and that amount of material is also assumed to be small. A historical review at the Buffalo Reef deposit (October 2006) estimated of 2.5 Mt grading at 2.26 g/t Au,for a total of 185,300 oz of contained gold. However, this historical estimate is not considered a Mineral Resource or Mineral Reserve as defined under sections 1.2 and 1.3 of NI 43-101. The Buffalo Reef gold deposit was acquired by Monument in July 2007 through its acquisition of Damar Consolidated Exploration Sdn Bhd (“Damar”), a wholly owned subsidiary of Avocet

(Cavey & Gunning,2007). Damar (and Avocet) owned the project from 1993 through to 2007, when initial exploration.commenced. Buffalo Reef mineralization is

their proximity to a regional crustal suture – an unconformity between Devonian and Permian age equences named the Raub-Bentong Suture. Buffalo Reef deposits occur along the major sature zone of Raub-Bentong which lies by the block that divided into two main regional blocks. The Buffalo Reef deposit occurs approximately 1 km to the east of the Raub-Bentong Suture – a major geological feature previously described in the Regional Geology section above.

The Buffalo Reef area is dominated by an eastern assemblage of argillite and limestone of Permian age in faulted contact with awestern assemblage of conglomerates and sandstones of Devonian age. Low-grade regional metamorphism up to Greenschist facies (locally up to Amphibolite facies) occurs throughout the area (Naidu, 2005). The sedimentary rocks have subsequently been intruded by granitic bodies of approximately Jurassic age. Outcropping rocks of these intrusive bodies occur to the east of Buffalo Reef and generally from elevation highs.The dominant structural feature present is a 200 m wide, north-south striking shear zone, with an apparent sinistral sense of displacement, which parallels the tectonic Raub-Bentong Suture to the west. The host rocks within the shear zone are composed of graphitic shale with minor interbedded fine-grained sandstone and tuffaceous rock (Naidu, 2005). Bedding within the sedimentary rocks typically dips 6575degrees to the east and strike towards a bearing of 333 to 360 degrees(Flindelletal.,2003).The dominant rock types within the Buffalo Reef area are Permian age argillites and limestones, which are cross-cut by later granitic to intermediate intrusive rocks. Gold mineralization is structurally controlled within a 200 m wide shear zone that trends sub-parallel to the regional Raub-Bentong Suture to the west. Gold occurs within veins and is typically associated with pyrite, arsenopyrite and stibnite. At

the current state, the Buffalo Reef is divided into 3 section area, which is North, Central and South (figure below)

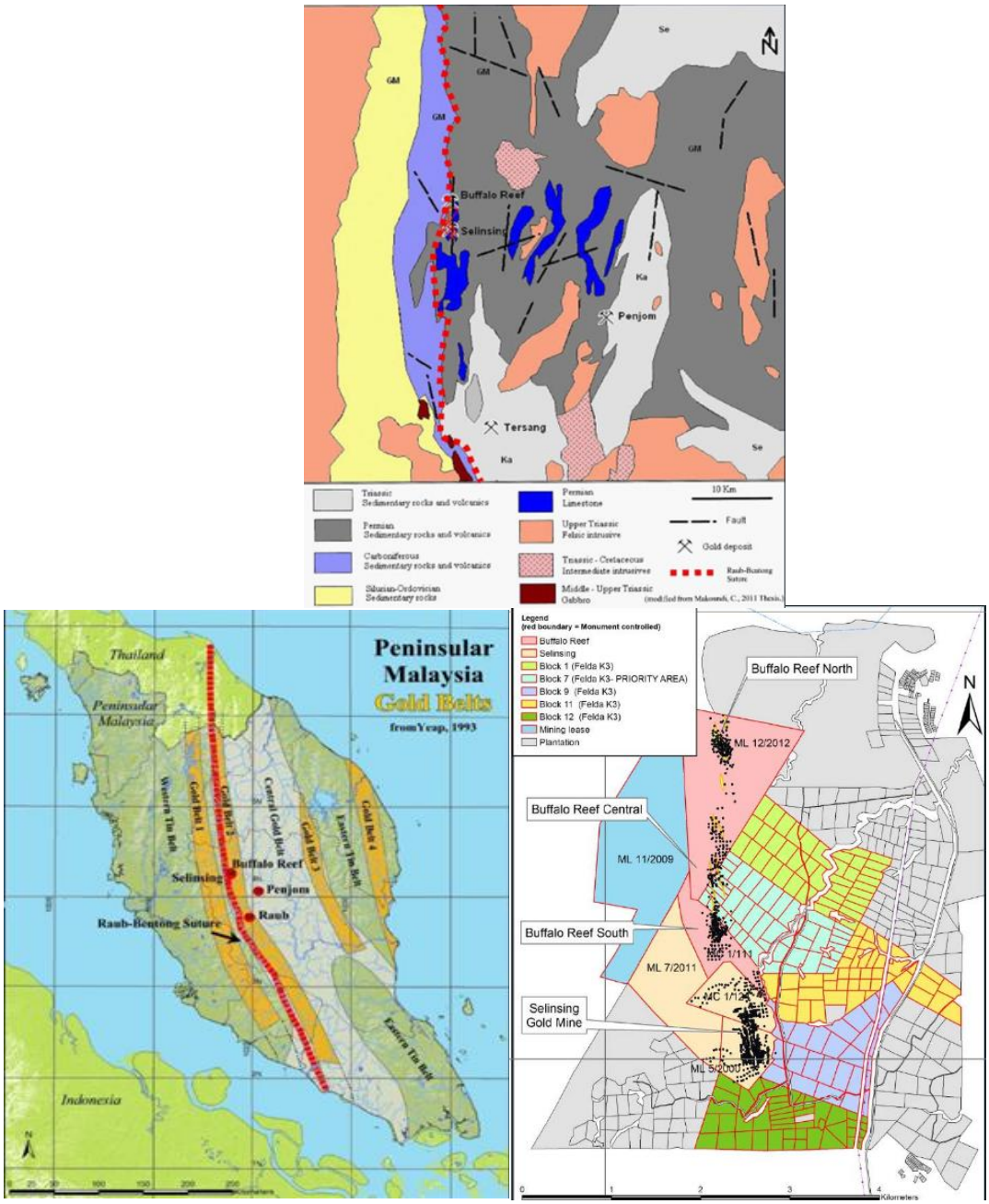


Figure 2.4.2.1 TOP; The formation of Gold along the Central belt, BOTTOM ; (left) The Gold Central Belt (right) The location of buffalo reef

2.5 Introduction On Applied Mineralogy.

Applied mineralogy related to gold involves determining mineral characteristics that have a bearing on exploration, mineral processing and hydrometallurgy. Applied mineralogy with respect to exploration involves using ore textures and specific minerals as tracers to gold deposits, and using specific assemblages of silicate and/or ore minerals as indicators of favorable environments for deposition of gold. Applied mineralogy in connection with mineral processing and hydrometallurgy plays a major role when gold is recovered by leaching techniques and floatation. The adequate data from ore characteristic which is the type of mineral associates with the gold ore can determine the mineral processing and the used of reagents. Knowledge of ore characteristics that affect gold recoveries can help in designing a flow sheet and can indicate whether maximum recoveries have been obtained.

2.5.1 Particle Size Analysis

Size analysis is the initial step and fundamental part of laboratory testing procedure. It is of great importance in determining the quality of grinding and establishing the degree of liberation of the values from the gangue at various particle sizes. To reduce any losses for occurring during the mineral processing, the size range have to be determine during the separation stage which to determine the optimum size of the feed to the proc

Figure 2.4.2.2 a) right: The location of Buffalo Reef b) the Gold Central Belt.

must be accurate but less precise but reliable, as important for any changes in plant operation may based on the result of laboratory test. The objective of precision particle size analysis is to obtain quantitative data about the size and size distribution of particles in the material,(Bernhardt,1994;Allen 1997).The irregular shape cannot be determine easily due it shape, sometimes it is desirable to quaote the size of a particle in terms of

singular quantitative and expression most often used is “equivalent diameter”. For the size of a spherical particle is uniquely defined by its diameter. For a cube, the length along one edge is characteristic. There are many methods can be used for particle size analysis which is test sieving , laser diffraction optical microscopy, electron microscopy and many more.(Hawke et al. 2015)

Sieve analysis is one of the oldest and commonest methods of size analysis and is accomplished by passing a known weight of sample material suceessively through the finer size fraction. The graph can be determined and due to establish of graph, the table for the cumulative distribution graph can determine the passing 80%(P80). Partition curve of the graph Cummulative percentage of passing versus the size fraction. Nowdays, there are a new technologies which can lead to more advance in particle size analysis. Malvern instrument have invented Particle Size Analyzer and provided a scientific instrumentation that are used to measure rheology, particle size, particle shape, particle concentration, protein aggregation, zeta potential and more. Malvern supplies and supports both industry and academia, in sectors from pharmaceuticals and biopharmaceuticals to bulk chemicals, cement, plastics and polymers. Each characterisation technique will measure a different property of a particle.

2.5.2 Mineral Composition

Mineral is made up from arrangement of atoms from inorganic substance and it compile with the abundance of other mineral existing within the mineral. Ore deposit can occur either in rock or soil material and it depend on the process of its undergoes. A rock can be defined as a solid substance that occurs naturally because of the effects of three basic geological processes: magma solidification; sedimentation of weathered rock

debris; and metamorphism. While the soil is the result of the weathered rock which undergoes chemical and biological weathered process. A mineral deposit is an abnormal concentration of minerals within the earth's crust. Mineralogist are really need to identify the mineral proportions of the minerals contain in order to control the economic mineral value. For example, in a deposit that contains 0.5% copper, all this copper may occur as coarse grains of the mineral chalcopyrite (CuFeS2) such a deposit may also contain 0.5% copper, but if all this copper occurs as a minor constituent in pyroxene (i.e. if the copper forms part of the structure of the pyroxene) then that copper is economically unrecoverable and the deposit is of no commercial value. (Merig P.Jones, 1987).

There are a few techniques which can implies for mineral composition study, which is X-ray Fluorscence and Scanning Electron Microscope(SEM-EDX). In gold process mineralogical analysis, its can be classified into 2 catergories: conventional and advanced instrumental techniques. For SEM, it classified as the advanced instrumental and gives high quality images of particle textures, such as surface morphology, pore structure, permeability and coatingswith an energy dispersive X-ray spectrometer (EDX) , in addition to its capability of being used for gold scanning.

For XRF,method for the production of a series of certified gold reference materials is presented. These reference materials are intended for use in the analysis of the elemental composition of gold alloys using a nondestructive X-Ray Florescence (XRF) spectrometry method. The chemical composition of the reference materials covers the complete range of conventional coloured and white carat gold jewellery alloys. The XRF method based on this series of reference materials produces analytical results which are comparable with those obtained when using traditional chemical methods of analysis. In X-ray fluorescence spectroscopy, the process begins by exposing the sample to a source of x-rays. As these high energy photons strike the sample, they tend to knock electrons

out of their orbits around the nuclei of the atoms that make up the sample. When this occurs, an electron from an outer orbit, or "shell", of the atom will fall into the shell of the missing electron. Since outer shell electrons are more energetic than inner shell electrons, the relocated electron has an excess of energy that is expended as an x-ray fluorescence photon. This fluorescence is unique to the composition of the sample. The detector collects this spectrum and converts them to electrical impulses that are proportional to the energies of the various x-rays in the sample's spectrum.

2.4.2 Phase Analysis

Phase analysis of mineral is crucial procedure in determine the analysis present by the particular mineral. Analysis of minerals is quite different from analysis of rocks. Analysis of mineral is more complicated because the individual mineral are much smaller than rocks and it is therefore difficult to obtain enough sample of mineral to perform the analysis procedure. Individual minerals may be chemically zoned. That is there may be differences in the chemical composition of the mineral from its center to its outermost shell. Mostly the phase analysis is carry out using laboratory equipment such as x-ray diffraction, Induced-plasma membrane, electron microscope study and many more. The principle of x-ray diffraction is standard guide to determine the phase analysis. When a monochromatic x-ray beam with wavelength is incident on the lattice planes in a crystal planes in a crystal at an angle, diffraction occurs only when the distance traveled by the rays reflected from successive planes differs by a complete number n of wavelengths. By varying the angle, the Bragg's Law conditions are satisfied by different d-spacing in polycrystalline materials. Plotting the angular positions and intensities of the resultant diffraction peaks produces a pattern which is characterised of the sample. Where a mixture of different phases is present, the diffractogram is formed by addition of the individual patterns.

The uses of x-ray diffraction (XRD) can determine the grade control in order to optimize concentrator feed and operational effiency. The mineralogy of ore sample is often not considered or at best inferred from visual geological logging of sampled material because not all the mineral contaminants cannot be identified visually or by chemical analysis which not recommended. The information provided by XRD will lead to creation of maps of spatial distribution of minerals. Common practice in grade control include the chemical analysis of ore and waste for key elements. The result will be analyse and conclude as cluster analysis for the raw scans which include phase identification and quantification. It can be used to simplify data processing significantly by automatically sorting closely related scans into clusters. It can reduce the amount of data that has to process. After completion of the cluster analysis of all the characteristic ore mineral, the result can be sorted and filtered all following of similar sample with varying mineralogy before quantitative analysis is applied.

The most characteristics scans of each cluster are used for phase identification. Phase identification was performed using Highscore'plus software.

2.5 Mineral Liberation

Minerals are form during various geological occurrence which can be associated with many type of minerals. Mineral liberation can be define either in research study or in industrial scale, in industrial scale, mineral liberation is define as the release of the valuable minerals from their waste gangue mineral and in research study, the mineral liberation is focus of determine the size fraction which the focused mineral available at. Mineral liberation can be conducted using the communition process and followed by sieving for size fraction analysis. The determination of size fraction which the mineral available at can determine the proper flow sheet of mineral communition process.

When the rock is break during either blasting, communition or during the passage way of the process, particles are produced with a continuous size distribution from the coarsest particle in the product to the finest (which is theoretically a particle of zero size). The continuity of this particle size distribution may be broken when the fragmented material is subjected an imposed process such as screening or hydraulic classification - in which case any products of the process will have their own unique size distribution that will differ from the that of the original material.

The textural relationships between minerals within an ore, and their relation to process selection requires the introduction of the concept of liberation size. This is the size to which an ore must be crushed or ground to produce separate particles of either value mineral or gangue that can be removed from the ore (as concentrate or tailings) with an acceptable efficiency by a commercial unit process. Liberation size does not imply pure mineral species, but rather an economic trade-off of grade and recovery. Liberation size is a function of the relevant physical or chemical process and may differ greatly between processes (Mills, 2006). Since ARD generation is a sulphide (pyrite) leaching process and base metal concentration is usually by flotation from gangue sulphide (pyrite) these differences may have significance in ARD prediction.

The liberation size of pyrite for both flotation and leaching (and gravity concentration if it is included in the mill flowsheet) is therefore a most important parameter. This information is extremely difficult to determine without quantitative mineralogical studies. Size reduction for liberation naturally produces a size range of particles. This range is dependent upon the type of size reduction method used, and the hardness, texture, friability and degree of weathering of the rock and its constituent minerals.

Chapter 3:

METHODOLOGY

3.1 Introduction

Methodology of the project is carried out base in the instrumental lab , pilot laboratory and at the project site. The methodology consist of four major categorizes which is preparation step, process step, analysis step and final results. The uses of chemical reagent is limited and the uses of epoxy resin is the only chemical substance used in this final project.

For preparation step, there are three major work must be proceed which is sampling, crushing and distribution sample. Sampling is done by the grab sampling method and a proper sampling methods is considerable crucial methods which will determine the representatives of gold ore sample. Next, the crushing is one of the major procedure in liberation of ore. Done by machinery tools such as Jaws Crusher and Cone Crusher. Distribution sample is done by two ways which is by cone and quartering and by the sieving for size distribution analysis.

Process steps consist of experimental work for determination of milling parameter with representative sample for each site beside the LOI of each sample. After that, analysis steps using analytical instruments and machine for data analysis consist of XRD, XRF, SEMEDX and Wet analysis. Results is an outcome of the experimental and technical procedures which consist of ANOVA, XRD, XRF and Ore microscopic study. .

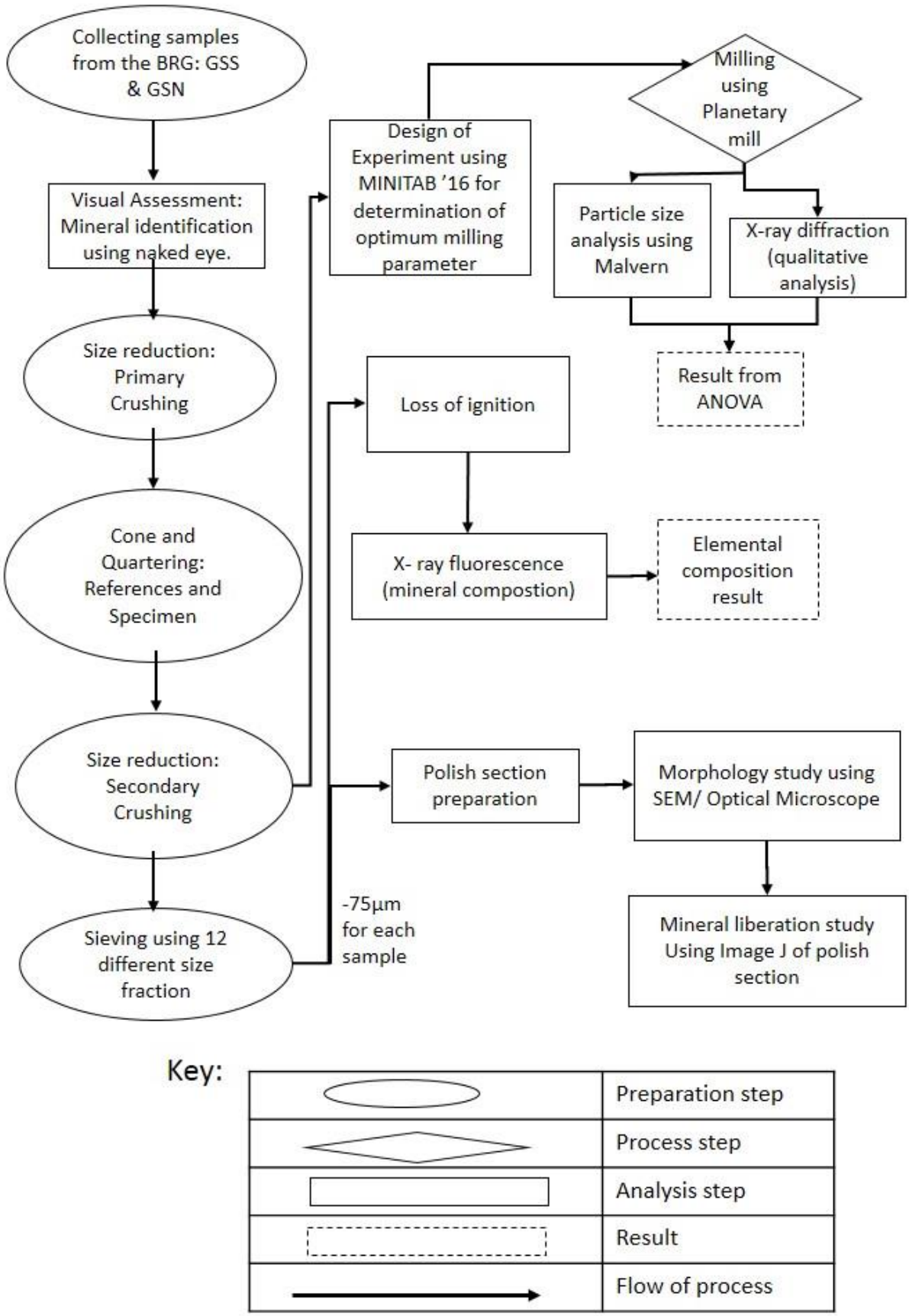


Figure 3.1 Flowchart depicting the stages involved

3.2 Preparation of Ore.

The sample of Malaysian Sulphide Gold ore is taken directly from the project site which situated at the Pahang state, Selinsing Gold Mine. All the samples is labeled base on the collection site which is North and South of Buffalo Reef and at the ore body and transaction zones which is GSN1, GSN2 ,GSS1 and GSS2. The bedrock samples were firstly visually examined with naked eye in order to describe the rock types and to decide which samples were best suited for the further study

3.3 Sampling

The sampling is taken at the ore body which situated at the Buffalo Reef. Each of section, the sampling is taken from the ore body itself and the transaction zone. Sampling is employed in connection with a number of different operations and with several different purposes in view. Sampling purpose can be vary with the location and procedure, sampling at site in Buffalo Reef is done by grab sampling and sampling using cone and quartering is done at the pilot laboratory and using Jones Riffles for splitting the selected sample for the ball mill's parameter determination.

3.3.1 Grab Sampling

The method to be used at the site varies primarily with the type of mineralization, mode of occurrence and other geological features of the deposit, the end sought should be obtain samples which is properly mixed and representative within the area sampled. The grab sampling in generally consist of taking samples of broken or intact rock from the site. The grab sampling is done at the South and North of Buffalo Reef with the help of Selinsing's geologist and the tools which is hammer and plastic bags is provide by the Selinsing Gold Mine administration. It is commonly done unsystematically as indicated

by ‘grab’ and by taking random handfuls from the scattered points on the free face of ore body.

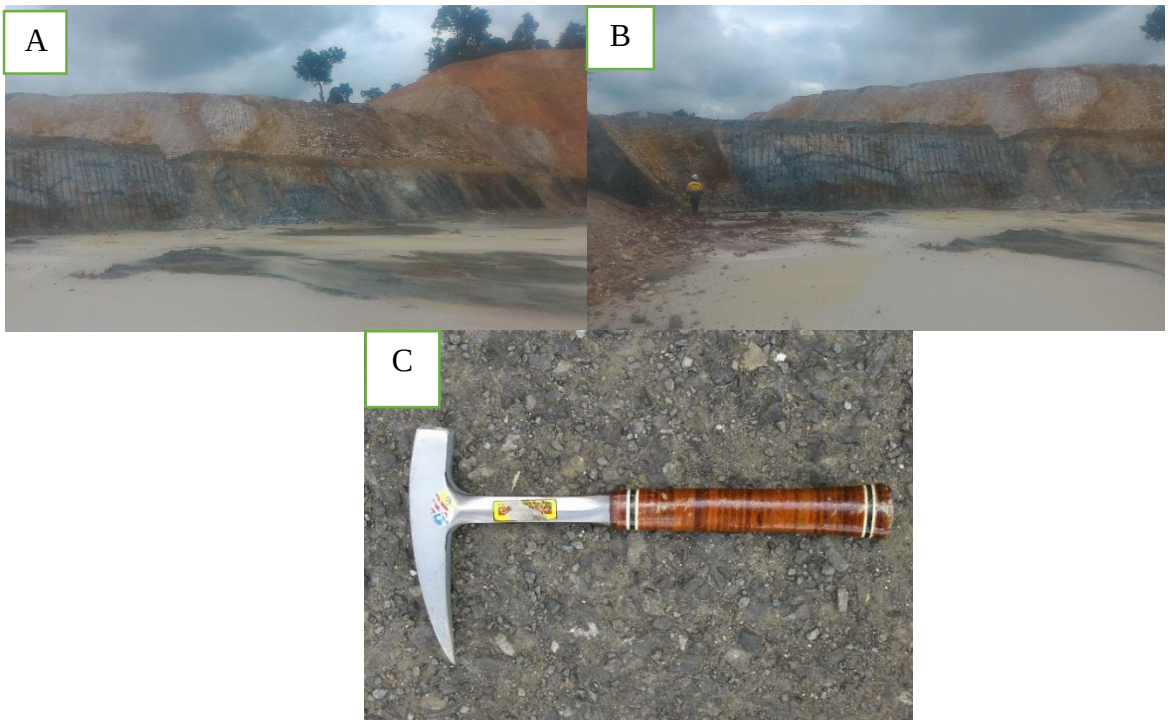


Figure 3.3.1 TOP; the location of South Zone , Right; the location of North Zone, at Buffalo Reef. Below, geological hammer for sampling tools

3.4.2 Cone and Quartering.

At the pilot laboratory, the sample of GSN1, GSS2,GSS1 and GSS2 is split after the jaw crusher using the cone and quartering method. All the output from the cone crusher were put on the wide plastic blanket provided by technician in order to get the homogeneous portion when doing sampling. It consists of pouring the material into a conical heap and relying on its radial symmetry to give four identical samples when the heap is flattened and divided by cross-shaped metal cutter. Two opposite corners are taken as sample, the other two corners being discarded as a revision sample.

3.4 Crushing

Crushing is the mechanical stage in the process and can be conduct in open or close circuit. The objective of crushing is to reduce the particles size and further study to liberate valuable minerals from gangue minerals .These samples are undergo crushing steps using the primary crusher (jawcrusher) and secondary jaw crusher (cone crusher).

3.5 Jaw Crusher.

Jaw crusher is the primary crusher in metalliferous operation features which imply crushing principal involve 2 plate which open and shut like animal jaws. Materials fed into the jaws was alternatively nipped and released to fall into the crushing chamber or discharge aperture if apply in industry. A fixed jaw, mounted in a ‘V” alignment is stationary breaking surface, while the movable jaws exerts force on the rock by forcing it against the stationary plate. The space at the bottom of the "V" aligned jaw plates is the

crusher product size gap, or the size of the crushed product from the jaw crusher. The rock remains in the jaws until it is small enough to pass through the gap at the bottom of the jaws. .The model of Jaw crusher is SVEDALA which is single toggle crusher and its reduction ratio is 5:1.

The procedure of crushing process using jaws crusher is initially, blow with air gun of the mechanical device to avoid any contamination of sample. The big size of sample is fed into the jaw crusher follow up by the smallest size. After all the sample is totally crush and pass through the gap, blow up the machine with air gun before the second sample been crushed.

Figure 3.5.1 Jaw Crusher

3.6 Cone and Quartering

All the samples after the crushing operation done by Jaws Crusher will undergo Cone and Quartering method for divide into references and hand specimens. The method is using the cone and quartering instrument and the method is shown at appendix.

3.7 Cone Crusher

The model of cone crusher situated in the lab is Marcy GV- roll crusher SVEDALA which features of reduction ratio 3:1. The cone crusher is a modified gyratory crusher and it is a secondary crushing process. Cone crusher have shorter spindle and supported in a curved bearing. The cone crusher have high capacity due to non-requirement of a large gape and the crushing shell flares outward and the flare of bowl allows a much greater head angle than gyratory crusher. Before that, the sampling of the feeds is accomplished using the Cone and Quartering method to reduce the excessive feed into the cone crusher and the remaining will be stored as a revision. The product from the previous jaw crusher will become as a feed for the cone crusher located near the jaw crusher. The size of material is about 0.5-5.0 cm is feed into the

feed hopper. Certain feed is have to push using rod bar due to large size which cannot impact directly to the surface of crushing shell. The minimum discharge of material is escaped through the opening set on the cone crusher as the material passing via the parallel zone received more than one impact between the materials.

Figure 3.7.1. Cone Crusher Machine

3.8 Riffle Splitting

This riffle splitting used in ball mill's parameter determination. The selected sample which is GSN002A and GSS001A is split until obtain the 7 sub-samples of each samples and weighed about 50g respectively. Johns Riffle is an open V-shaped metal box in which a series of chutes is mounted at the right angles to long axis to give a series of rectangular slots of equal area of opening. The feed is alternatively divide into two trays placed on either side of the trough. The samples is poured into the chute and split into equal portion and the repeated cycle is needed until the small amount of feed samples in achieved. One half becoming the sample project and one half was kept as reference. The biased product sampling could be reduced by using this method as kept pouring the sample at the center of the riffle splitting.

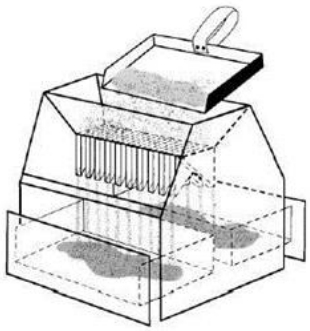


Figure 3.8.1 Jones Riffles
for sampling

3.9 Sieving

Sieving is carried out with dry materials. The sieve size fraction is determined using the Ratio of aperture width of adjacent sieves is the square root of $2(\sqrt{2})$ The advantage of such a scale was that the aperture areas double at each sieve, facilitating graphical presentation of results.. The total number of sieve fraction pans is 12 units which it start with +4.75 mm, +3.35mm, +2.36mm, +1.18mm,+0.60mm,+0.425mm, +0.300mm ,+0.212mm,+0.150mm, +0.090mm,+0.075mm and -0.075mm.

For the sieving to run, the sieve shaker only limited for 6 pieces of size fractions. Each run will consume about 10 minutes and each of the sample to complete sieve need to run 2 times. The total time taken for each sample is about 40 minutes runs. The earplug and face mask must be wear all time during the process is conducting due to avoid the noise that cause ear damaged and avoid small dust particles from sieve enter the mouth.

The mass of sample in each size fraction is taken to construct the graph of Particle Size distribution. The particle size were analyzed from this sieving result. The precision of particle size analysis were determine the quantitative data about the size and to do the polish section for study of mineral liberation and SEM analysis.

Figure 3.9.1 Sieve Shaker

3.10 Ball Mill Parameter Determination.

The parameter of milling is very crucial in determine the most optimum milling rate which can save consumed energy and time taken. The parameters which been investigated is time taken (minutes) and the speed of rotation (rpm). Using the MINITAB software, the DOE concept is used to construct the running of experiment which using 2 factorial.

Before runs the experiment, the preparation of sample is needed, The GSN2 and GSS1 is selected for represent each of the site which is North and South of Buffalo Reef respectively. The Planetary mill is used rather than standard ball mill due to the control of investigated parameter. The mechanism of planetary mill and ball mill is still same only the size and the direction rotation is not exactly the same. The planetary mill is rotate

vertically rather than horizontally. The type of ball mill is steel ball because the previous data show that the presence of quartz and iron are high in the Malaysian sulphide gold ore.

The procedure of the planetary ball mill must be taken cautiously. Place the sample inside the container mill and pour the steel ball as grind medium about 10 pieces with 20mm width each. Weight the current sample with the container and the lid. Adjust the weight bearing and tight the closure of the lid. Pull in the safety torque on the lid several times and tight the closure simultaneously and properly. Setup the detailed parameter on the machine. After done, let the machine cold down about the same exact time of milling. Open the safety torques and take out the container and poured the feed into the sieve of 2.80mm and clean the ball. Ensure the feed is press with spatula so that the feed is liberated and not conglomerate. Repeat the steps for next detailed parameter.

3.10.1 Design of Experiment.

In the research work , DOE is adopted for experimentation of optimum parameter milling for sulphide gold ore for two type of sample which is GSS1 and GSN2. In DOE, there are two manipulated parameters in the experimental work are known as the factors, which are the speed rotation of planetary mill and the time taken. X1 is the code notation used for speed rotation of planetary mill, while X2 is the code notation for time taken. The full factorial design utilized in this DOE is (22+3), where there are two factors at two levels, and adding three centre points as the stability process measurement and to check the curvature. Levels are the term used to indicate the degree of level. It consist of three level in this DOE, which are ‘-1’, ‘0’ and ‘+1’ stands for low level, ‘0’ stands for the medium level and ‘+1’ stands for high level.

3.10.1 X-ray Diffraction (Qualitative Analysis)

All the samples from the planetary mill will be proceed into the phase analysis using X-ray Diffraction. The samples was been analyzed to define it phases after undergo different milling parameter.

3.10.2 Particle Size Analysis using Malvern.

The samples is divided into two for further assessment which is XRD and Particle Size Analysis. For PSA, the sample is weighed about 10mg which is the acceptable weight

3.11 Loss On Ignition.

Loss on ignition (LOI) analysis has been widely used as a method to determine the organic matter and volatile matter in the sample. The procedure started with heat the empty porcelain crucible in a carbolite furnace for an hour. The temperature used is 105^0C. The smallest size fraction (75 µm) of each samples GSN1, GSN2, GSS1A and GSS2 is taken and weighed and place inside the porcelain crucible.

The purpose of preheating process is to remove vapour or some volatile substances in the porcelain crucible. The sample was run in 1000^0C. Three crucible were filled with sample. The crucible were labeled of sample A, sample B, sample C and sample D. The heating process was conducted with increasing of temperature of 10^0C/min until the ignition temperature of 1000^0C .

The porcelain crucibles uncovered with lid in order to allow the volatile substances to evaporate and escape from the samples. The samples were left at room

temperature with 10^0C/min after undergo heating. The LOI results are used for the XRF analysis. The loss on ignition of the dry mass of a solid sample expressed in percentage shall be calculated from equation 1 :

$$WV = \frac{Wb - Wa}{Wb - Wc} x100\% \ldots Equation\ 3.11$$

WV is the loss on ignition of the dry mass of a solid sample, in percentages;

Wa is the mass of the empty crucible, in grams;

Wb is the mass of the crucible containing the dry mass, in grams;

Wc is the mass of the crucible containing the ignited dry mass, in grams;

The results shall be rounded to the nearest 0.01% and listed in appendix A

3.12 X- ray Fluorescence (XRF)

After the ignition is done, all the sample was sent to the XRF lab for mineral composition identification. Before that, all the sample must be sure that the size required is 75µm and the weight was more than 25mg. The XRF analysis was done by skill full technician which using mould and press to form a pellet.

3.13 Preparation Of Polish Section For Scanning Electron Microscopic (SEM) and Optical Microscope Study.

The selected size fraction of sieve is taken for each samples, which is +2.36mm, +0.60mm, +0.30mm,+0.15mm, +0.075mm and -0.0075mm as a polish section preparation. This polishing samples then studied under the polarizing microscopy in order

to identify the mineralization characterization and classify regarding the result from microscopy study. Each of polishing method required 4 stages of preparation of polish section:

3.13.1 Specimen selection and preparation

The selected size fraction of each samples is taken using the small spatula. The number of moulds needed and the type of mould used is a pieces of rubber cover table which cost only 0.50 cent per piece. A stacks of cup needed to mix the resin with epoxy resin before poured into the mould. After that, a wooden sticks is used for to stir the mixer of the resins. Suitable type of resin would strengthened the impregnation of specimens for samples GSN1 ,GSN2,GSS1, and GSS2A under the room temperature.

3.13.2 Specimen Mounting

Specimens were mounted in standard-size mould with 1 inch width of epoxy resin. This done for ease of handling and to standardize the finished size so that all the specimens fit in the mould for polishing stages. The mixing process between hardener and epoxy resin is done in the cup with initially weighed to ensure the ratio between hardener and epoxy resin is 1:1. Using spatula, take a one spatula of sample and put into another new cup and pour about 30% of mix of hardener and epoxy resin . Stirred about 2 minute until the previous air bubble is all gone. Pour into the mould following the remaining of mix substances. Then, stirred with sample and the mixer together. After that, discharge it to the mould holder. Let the specimen dry at room temperature for a day.

3.13.2 Grinding a flat surface

After hardening, the specimens were removed from the moulds. The appropriate face were then ground on sand paper. Sizes of 180, 240, 360, 420, 600, 800, 1000 and 2000 sand papers used in grinding in order to get the smooth and flat surface. Water was used as a lubricant. Concentration must be made to produce a flat surface. The flat surface reduces the need for constant re-focusing of microscope during subsequent examinations.

3.13.3 Polishing that flat surface

The alumina oil was used polishing procedure. The auto-polishing machine would undergo only four specimens in one time. One hour was needed to ensure the flat specimen surface are clean and clear for the mineral identification.

3.14 Mineral Identification Using Polarizing Microscope

All the polished section were analysed under the polarizing microscope. The optical properties of a wide range of transparent minerals and of opaque minerals were determined. The various crystallographic features of the individual minerals also were observed. Polished section samples was analysed under an Olympus BX41 polarizing light microscope with stabilized 100W halogen illumination. Magnification used for the sample involved 5X, 10X, 20X, 50X and 100X magnification. Digital camera was used to capture the images of sample. Software of Isolation DT was used to scale the image of grain samples. The scaled used in 10 micron sizes. Each picture was scaled according to its magnification size.

3.15 Scanning Electron Microscopy (SEM)

The polished specimen were studied for ore and rock- forming mineral by SEM-EDX brand Gemini. The morphology and mineral qualitative analyses were performed using SEM-EDX. The samples were coated with sputter coating with gold to cover a specimen with a thin layer of conducting material. The coating process took 1 hour. The sample were observed and imaged at a different working distance and accelerating voltage to produce Backscattered Images (BSI) for each samples. Then, the location for the image in ore microscope was searched under SEM. The possible minerals are spotted with EDX to determine the composition of the sample.

3.16 Percentage of Mineral Present using Image J Software (Imaging Processing)

ImageJ software are used in particle analysis and liberation study in this experiment. ImageJ is a public domain, Java-based image processing program and was designed with an open architecture that provides extensibility via Java plugins and recordable macros.. Area and statistics value selections were calculated.. The particles in the raw image were separated from the background by a process known as “threshold”. The purpose of this process is to measure the area of mineral present in each sample based on the size in percentage.

CHAPTER 4:

RESULT AND DISCUSSION

4.1 Introduction

The study of mineral composition and phase analysis are the fundamental concept in determine the mineralogy of ore bodies located in south and north section within the Buffalo Reef region. Process Mineralogy can be considered as the practical application of mineralogical knowledge to aid mineral exploration, and to predict and optimise how an ore can best be mined and processed. The geological study which related to process mineralogy is essential for setup the mineral processing and it is a specialization within the field of applied mineralogy. Process mineralogy is dominantly used in areas such as geometallurgy, ore characterisation, process design and optimisation, within increasingly complex ore bodies and the increasing intention in reducing operational cost. In environmental field, the environmentalist often request the further understanding mineralogy of the minerals and their textures in order to reduce risk. The aim of process mineralogy to provide useful information on the process selection, flowsheet development, recovery improvement , reagent consumption optimization, and to understand either the benefits of these that can be harnessed or the limitation that need to be considered for.

Intensive study in mineralogy especially in the texture of an ore can dictates how the ore can be mined and processed efficiently without ignoring the potential environmental ramifications in doing so. Process mineralogy is utilised in all stage of mining cycle from the exploration until the waste tailing management form the mineral processing plant. It is closely linked to geometallurgy, being fed directly in to a geometallurgical predictive model, which spans the whole process.

Process mineralogy is often used to predict the response of gold ire to various candidate processes. In addition, during the processing of a gold ore, periodic mineralogical analyses of ore feed and mill products are needed to determine the nature of the problem if the gold recovery is lower than expected. Therefore, a routine process mineralogical study includes the objective of quantify the fractions of liberated gold, gold associated with sulfides, gold associated with oxides and gold associated with carbonaceous material. Besides , determine any other valuable metal in terms of species, phase, distributions and liberation characteristics.

In this chapter, the result and discussion of the whole methodology execute the process mineralogy is carrying out from the communition until the data analysing procedure. The element composition in weight percent and mineral phase identification of samples after the milling process with different parameter set can be obtained from the results of Xrf and Xrd analysis respectively. Most important, the mineral morphology by polarizing microscope and scanning electron microscope are also being discussed in this chapter.

4.2 Sieve Size Analysis

The samples of Buffalo Reef which are GSN001, GSN002, GSS001 and GSS002 undergoes further crushing process via cone crusher which reduction ratio is 5:1. After completing the communition and sizing by the cone crusher, the next procedure is sieving using sieve shaker with different and selected size fraction from the smallest to the largest size aperture and afterwards the determination of weight percentage of each sie fraction is done. The choice of sieve size is by the formula of square root of two 2($\sqrt{2}$= 1.414).

Size reduction is the most energy intensive and thus costly part of mineral processing. Thus, the determination of the percentage passing of ore during assessment process mineralogy can provide information on the median diameter of the particle size distribution in sieve size analysis. It is the value of the particle diameter at 50% in cumulative distribution. This method explain clearly about central size of sample particle and it is one of the important parameter characterizing particle size.

Sieve size analysis is the number of particles that pass through size fraction with different size range of aperture given as a percentage passing form the total number of all sizes in the sample. D_{90} show particle size distribution at D_{90} the amount of cumulative weight percentage which represent the undersize particle diameter. D_{90} is crucial in determine the efficiency of crushing machine and the size fraction which have highest value of total passing percentage

The following table represent the cumulative weight percentages (%) for each of the samples

Table 4.2.1 Percentage passing of D_{10}, D_{50}, and D_{50} of the GSS1,GSS2,GSN1 and GSN2

Type of Samples	**Percentage Passing(%)**		
	D_{10} (mm)	**D_{50}(mm)**	**D_{90}(mm)**
GSS1	0.38	2.31	4.20
GSS2	0.45	1.75	3.40
GSN1	0.27	1.82	3.33
GSN2	0.40	2.14	3.25

The size of feed after the crushing by cone crusher is reduce into the smallest size. The weight of each size fraction is weighed for each of sample and plot a table for each of sample in cumulative frequency to identify the size of sample which high in passing percentage and retain percentage.

The table 4.2.1 shows that size particle of sample GSS1A at D90 was 4.20mm, while D50 was 2.31mm and at D10 was 0.38mm. On this sample, 50% of the particles were larger than 2.31mm. For the sample GSS2, the particle size at D90 was 3.40 mm, at D50 was 1.75 mm and at D10 was 0.45 mm. Apart from that, the sample GSN1 shows at D90, the value of sieve size was 3.33mm, for D50 was 1.82 mm and for the D10 the value was 0.27 mm. In other words, 90% of the particles in the tested sample were smaller than 3.40 millimetre. For the sample GSN2 the sieve analysis at the D90 gave a value of 5.000mm similar in sample GSN1, for D50 the value was 2.14mm and for the D10 the value was 0.27mm. It show that D90 or the percentage of particles smaller than 3.33 mm is 90%. D90 is a typical point in particle size distribution analysis.

4.3 Loss on Ignition

The size fraction less than 0.075mm of each samples is collected and weighed for the further process which is loss on ignition. The samples are heated on 1000°C in the carbolite furnace for the 6 hours of retention time to remove organic matter. Samples must be cool so that convection currents do not affect the balance, and kept in the furnace so that they do not absorb atmospheric water. The table is constructed consist of the mass of the empty crucible in grams,W_a , the mass of the crucible containing the dry mass in grams ,W_b ,and the mass of the crucible containing the ignited dry mass in grams, W_c.

Table 4.3.1 Result of LOI of Samples from Buffalo Reef

Samples	GSS1	GSS2	GSN1	GSN2
W_a	50.02g	35.79g	36.35g	35.73g
W_b	87.16g	70.95g	62.28g	63.24g
W_c	86.41g	69.41g	60.89g	61.04g
Result	2.0%	4.4%	5.4%	8 %

LOI was dependent on the several factors which is sample size , different in handling between standard and laboratory method, expose time and position of samples in the furnace. Larger sample needed more time to combust and the size of recommended samples used is 0.075mm. The position of samples in the middle have high combustion rate due to warner temperature and quicker heating of smaller samples are needed to be the most likely explanation for different weight loss. Intercomparison of laboratory methods and standard method yield a different in LOI results measured. The value of LOI% for GSN2 is higher due to the higher content of organic and carboneous matter.

The colour of each sample is change after the heating in the furnace due to loss of inorganic matter and carbon. GSN2 has the highest LOI% due to carbon content and moisture content is higher. The location of North region is near the surface which laterally all of the ore body undergoes weathering and merges with the soil.

4.4 Mineral Composition.

The following data presence is accomplished by the x-ray diffraction of samples GSN1,GSN2, GSS1 and GSS2. The result related to XRF analysis of the Malaysian sulphide gold ore from the Buffalo Reef region are given in the Table (). The common and significant oxides identified through XRF analysis are MgO, Al_2O_3, SiO_2, SO_3, K_2O, CaO, TiO_2, MnO, Fe_2O_3, As_2O_3, Sb_2O_3 and Au_2O. The quantity of each oxides is vary with the samples.

SiO_2 was observed to be the most abundant oxide with its content varying between 65.17%, 57.18%, 56.7% and 39.90% for GSS1, GSS2, GSN1 and GSN2 respectively. The second abundant element in GSS1A is Mg which is about 8.882% and for the GSS2, GSN1 and GSN2 is Al about 2.608%, 5.567%, and 8.244% respectively. Au was noted to be the most less abundant oxide in each of samples. According to geochemistry, the presence of As which arsenic is used as pathfinder of gold in mineral exploration scheme. The abundant of SiO because the formation of soil is composed of silica sand commonly. The weight percentage of Au in sample GSS1, GSS2, GSN1 and GSN2 are 3.7ppm, 4.9ppm, 3.4ppm and 4.0ppm.

The mineralogical and chemical composition of each samples taken directly from Buffalo Reef are known to be in oxide ore which is literally been weathered ,cause the alteration in mineralogy, mechanisms of transportation, depositional environment and post depositional processes.

Table 4.4.1 Result XRF for Element Composition Of Samples

	Samples			
Element Composition In %	**GSS1**	**GSS2**	**GSN1**	**GSN2**
Mg	**8.88200**	**0.17700**	**0.22300**	**0.13600**
Al	**-**	**2.60800**	**5.56700**	**8.24400**
Si	**65.17000**	**57.18400**	**56.70000**	**39.90800**
S	**0.61000**	**0.12000**	**-**	**-**
K	**0.08500**	**0.68400**	**1.25200**	**1.38100**
Ca	**0.23400**	**1.41500**	**0.14200**	**0.11000**
Ti	**-**	**0.19990**	**0.43500**	**0.65400**
Fe	**0.67200**	**0.69500**	**1.73400**	**0.77900**
As	**0.25000**	**0.79800**	**0.40400**	**0.15360**
Sb	**0.17800**	**0.59600**	**0.03090**	**0.00680**
Au	**0.00037**	**0.00049**	**0.00034**	**0.00040**

4.5 Morphology study

4.4.1 Polarizing Microscope

Optical microscope study of polish section for each samples at given size fraction is one of the methods used for morphology study of Buffalo Reef region. This polarizing microscope is one of the most useful instrument used in mineralogical laboratory to identify the optical properties of a wide range of transparent minerals and of opaque minerals. For mineral identification purpose, the determination of certain mineral characteristics is done by using the polarized light.

Observation of sample GSS1, GSS2, GSN1 and GSN2 with ore microscope are made by using plane polarized light with polarizes and analyser inserted. The color, bireflectance, anisotropy, internal reflectance, shape, luster, cleavage, fracture and transparency are observed under the polarized microscope. All the properties are summarized in the table 4.

Gold is an opaque mineral which have a few optical properties that can lead. The colour, colour intensity, shape, cleavage, and hardness are the present features which can be determine under plane-polarized light. Colour is identified by part surrounding of target area and colour intensity is influenced by reflectant.

Colour should be made with a low-power objective when observed under-polarized light and a very small number of ore minerals are strongly and distinctly colored but most are weak colored within the range of white and dark colour. A problem is that the apparent color of a mineral depends on its surroundings (eg, the mineral chalcopyrite appear distinctly yellow against a white or gray phase, but a greenish-yellow when seen next to native gold) (David J Vaughan,1981). Colored property description must take caution as fact that colors are also dependent on the illumination employed. However, color are best described in comparison with their associated common minerals

Images that analysed by the optical microscope of sample GSS1, sample GSS2 sample GSN1 and sample GSN2 for the sizes of (-2.360+0.600)mm, (-0.600+0.300)mm, (-0.300+0.150)mm, (-0.150+0.075)mm, (-0.075+0.075))mm and -0.075mm as in Figure 4.4.1, Figure 4.4.2, Figure 4.4.3 and Figure 4.4.4.

From figure 9. below ore paragenesis and paragenetic in size sequence of GSN1 ore. At (A) a conchoidal and pale brass-yellow of pyrite after Fe- spinel polarized reflected light (P.R.L.). Irregular fracture of silver white of arsenopyrite existance,P.R.L .At image (B) stibnite occur as granular aggregates mineral along the growth zoning, P.R.L (C) Chalcopyrite that common alters along cracks and grain boundaries associated with pyrite and the intergrowth of Hematite as subparallel or radiating aggregates, PRL. (D) weakly anisotropic, yellowish of pyrite growth associate with crystal of chacolpyrite (Apy), P.R.L. (E) a poor cleavage of Chalcopyrite hosted at the edge of interganular quartz., P.P.L. (F) The scattered of minerals such as stibnite , pyrite and chacolpyrite all over the matrix conclude that at size fraction 2.36mm the occurrence were abundant.

Furthermore, for Fig. 10. Ore paragenesis and paragenetic sequence in GSN2 fom Buffalo Reef Region include the transition of ore body near to surface. (A) color yellow to brassy yellow of chalcopyrite associated with a metallic pyrite, P.R.L. (B)euhedralcubes of pyrite at the growth zoning along with the crystalline growth of arsenopyrite, P.R.L. (C) a massive aggregrate of galena that associated with the arsenopyrite of P.R.L. (D) weakly anisotropic, yellowish of pyrite growth associate with crystal of chacolpyrite (Apy) , P.R.L. (E) Hematite occur intergrowth with prismatic stibnite , PRL. (F) metallic stibnite is well separated or well liberated at size 2.36mm.

In addition, Fig.11. Ore paragenesis and paragenetic sequence in GSS1. (A) the ore brittle sulfides such as pyrite and arsenopyrite deform fracturing in shateered crystals and Idiomorphic pyrite cube (Py) with coarse and fine silicate inclusions (Sil) that are zonal toward rim, P.R.L. (B) the presence of individual of stibnite in presence of fluid inclusion .(C) light pink of metallic galena, P.R.L. (D) Interstitial spaces filled by chalcopyrite and white shiny stibnite, P.R.L. (E)inclusion of intergrowth stibnite in massive form associated with galena in quartz , P.R.L. (F) Idiomorphic pyrite cube (Py) with coarse and fine silicate inclusions (Sil), P.R.L.

Lastly, Figure. 12 Ore paragenesis and paragenetic sequence in GSS2 ant transition zone in Soth region. (A) stibnite occur as granular aggregates that exhibit deformation texture associated with pyrite and arsenopyrite P.R.L. (B) subhedral crystals shape of chalcopyrite, P.R.L (C) the presence of cubical and anisotropic galena. (D) pyrite growth zoning with metallic greenish sphaleritte associate with chalcopyrite and stibnite , P.R.L (E) perfect cleavage of white dull stibnite and pyrite during fluid inclusion at the deposition zone, P.P.L (F) subconchoidal crystals with angular shape of galena,P.R.L.

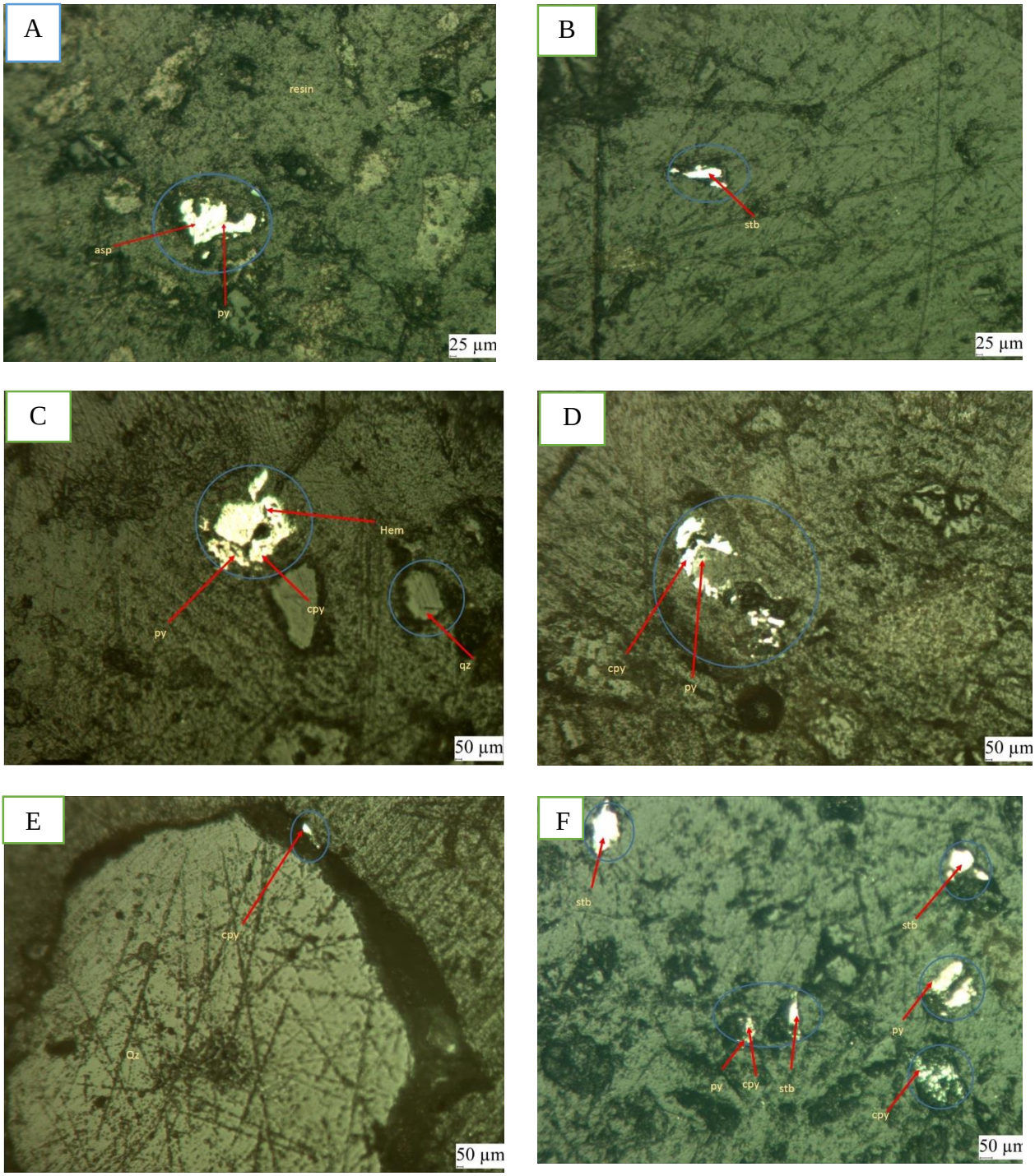


Figure 4.4.1 Optical Microscope of GSN1 (A) -0.075mm of size fraction (B) +0.0075mm of size fraction (C)0.15mm of size fraction (D) 0.3mm of size fraction (E) 0.60mm of size fraction.

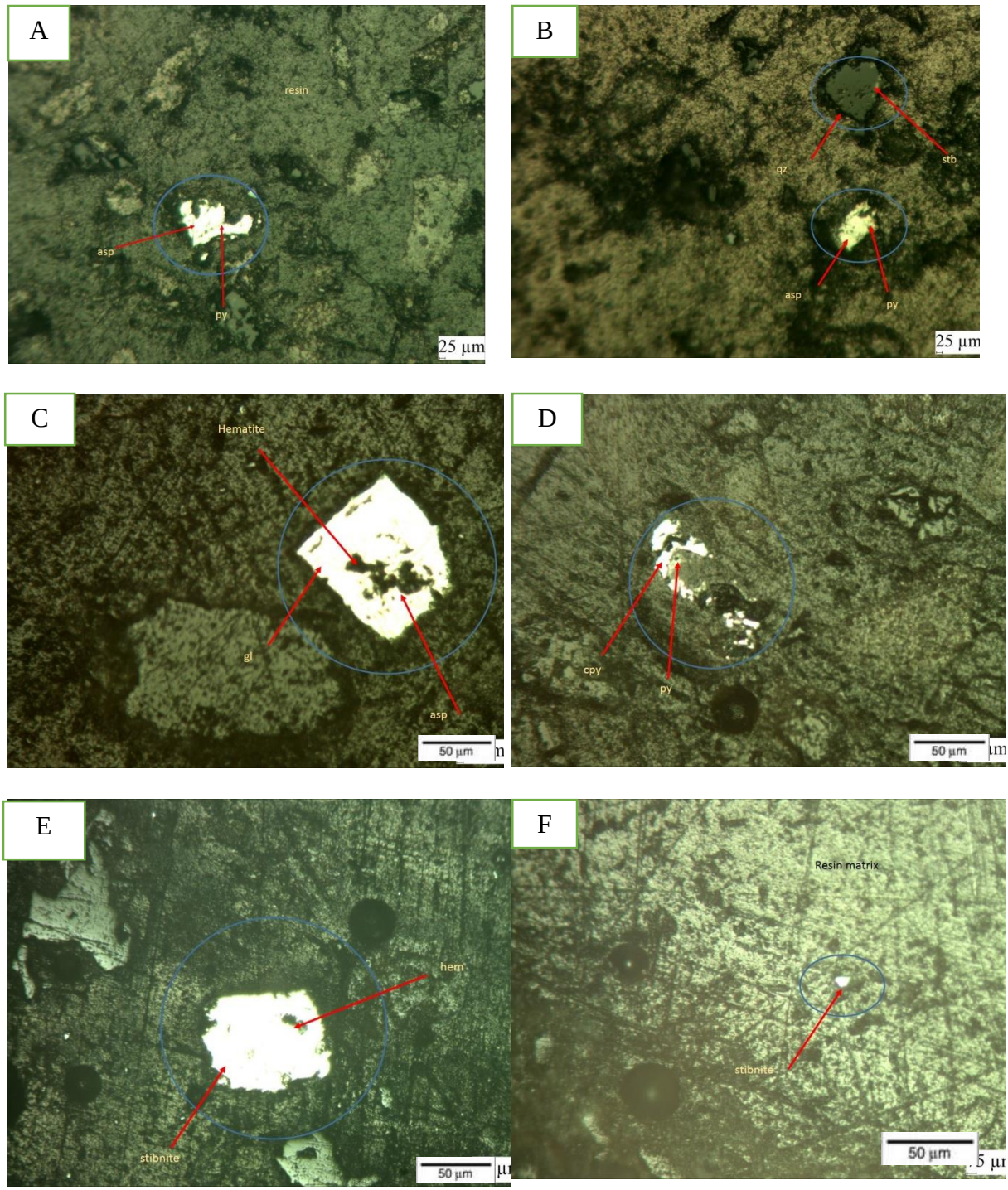


Figure 4.4.2 Optical Microscope of GSN2 A) -0.075mm of size fraction (B) +0.0075mm of size fraction (C)0.15mm of size fraction (D) 0.3mm of size fraction (E) 0.60mm of size fraction.

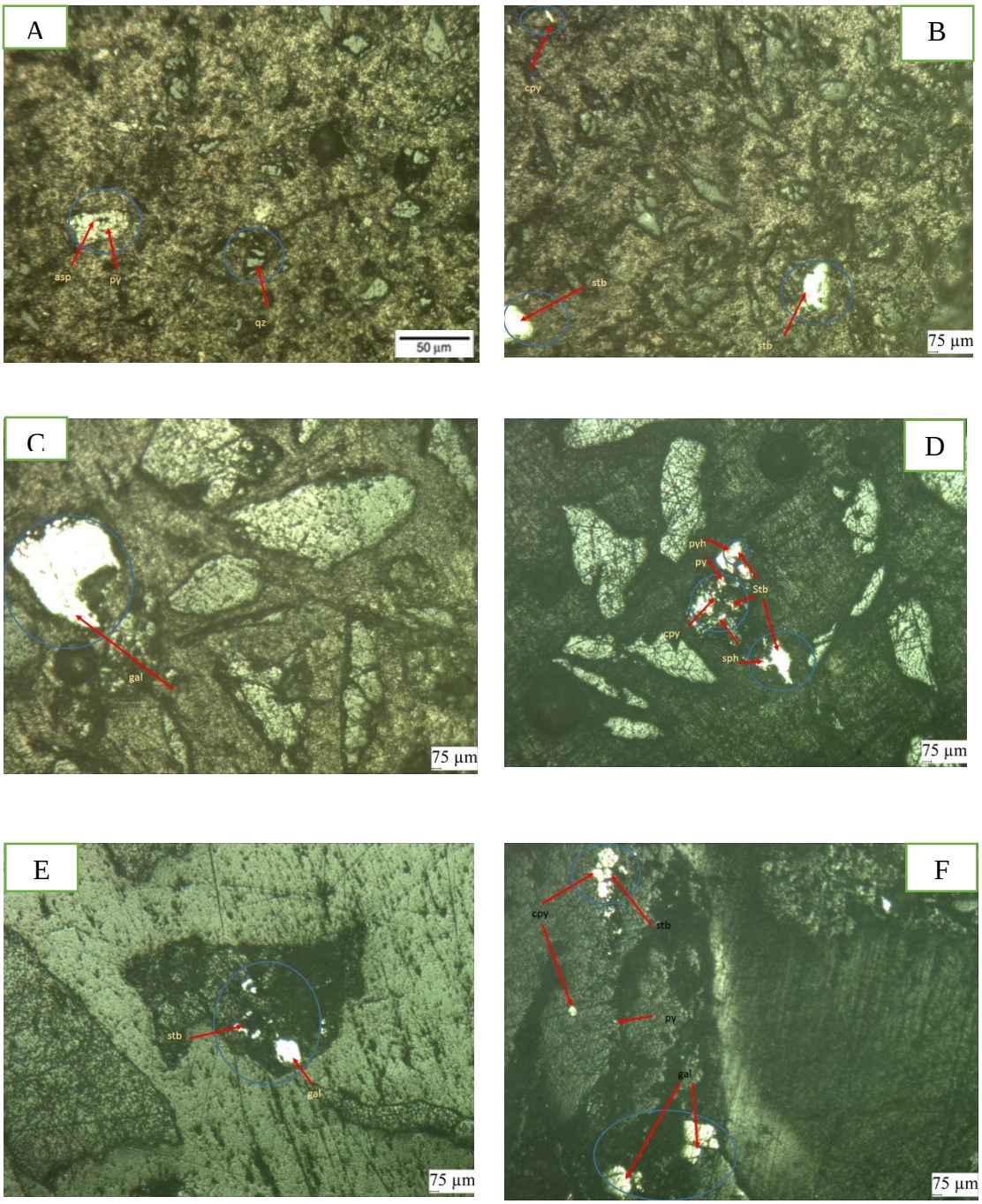


Figure 4.4.3 Optical Microscope of GSS1 A) -0.075mm of size fraction (B) +0.0075mm of size fraction (C)0.15mm of size fraction (D) 0.3mm of size fraction (E) 0.60mm of size fraction.

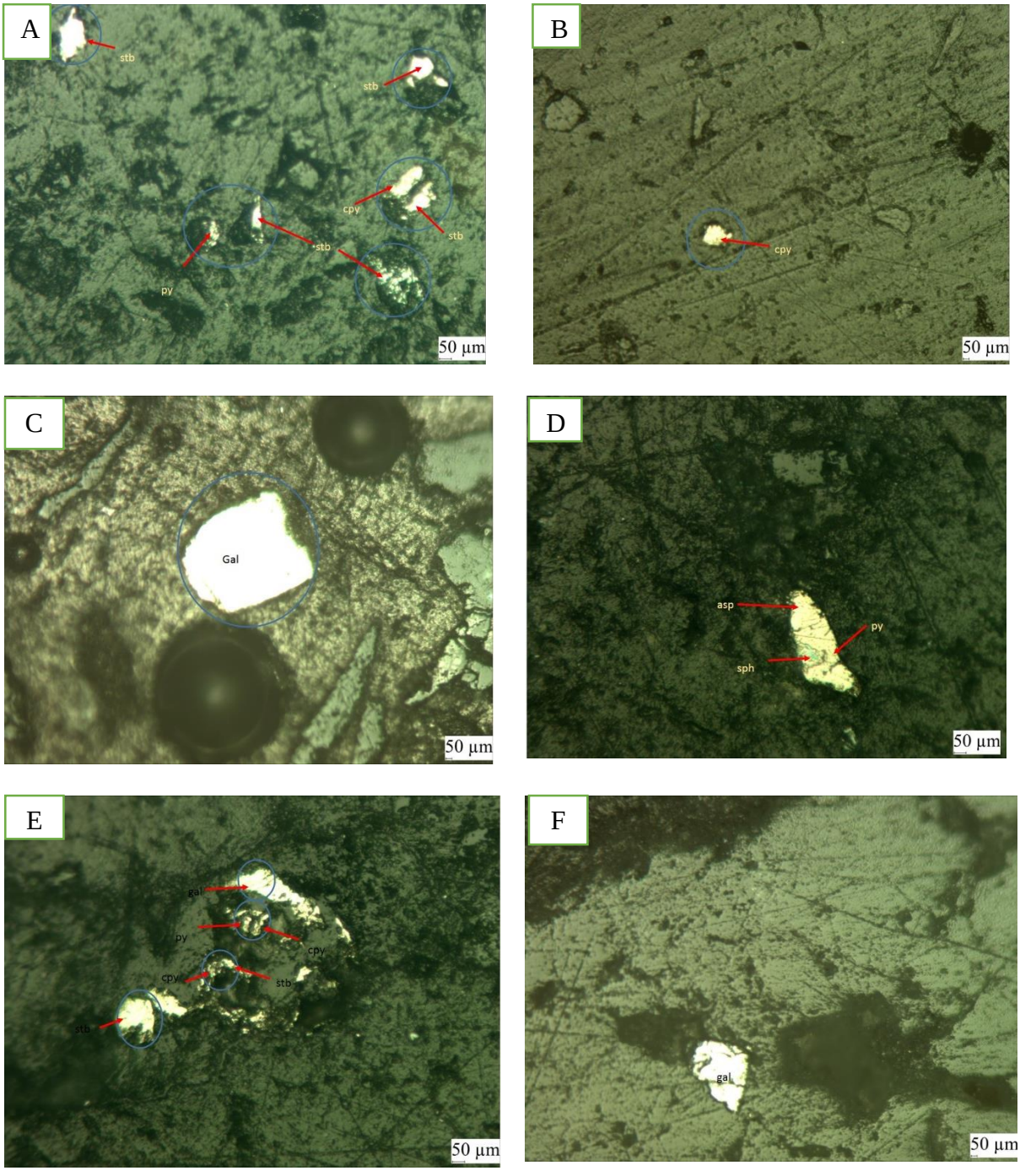


Figure 4.4.4 of Optical Microscope of GSS2 A) -0.075mm of size fraction (B) +0.0075mm of size fraction (C)0.15mm of size fraction (D) 0.3mm of size fraction (E) 0.60mm of size fraction.

Table 0-1.4.1 Mineral Properties of Morphology Study

MINERALS	PROPERTIES									
	CHEMICAL FORMULA	COLOUR	BIREFLECTANCE	ANISOTROPY	INTERNAL REFLECTIONS	SHAPE/ CRYSTAL SYSTEM	LUSTRE	CLEAVAGE	FRACTURE	TRANSPARENCY
CHALCOPYRITE	CuFeS2	Brassy yellow	Weak Light grey	USUALLY WEAK BUT DISTINCT: GREY-BLUE AND GREENISH YELLOW	Not present	Tetragonal/tetragonal	metallic	Poor/Indistinct	Irregular/uneven	Opaque
GALENA	PbS	Bright white, sometimes with a pinkish tint	Not present light grey	Isotropic; a weak anomalous anisotropic is uncommon: medium grey to grey black	Not present	Cubic/ Isometric	Metallic, Dull	Perfect	SubConchoidal	Opaque

GOLD (not present)	Au	Not present Bright or ‘golden’ yellow	Not present	Not present Isotropic,many scratches suggest otherwise	Not present	Not present Cubic/ Isometric	Not present Metallic	None	Not present Hackly	Not present Opaque transparent in thinnest layers
ARSENOPYRITE	FeAsS	White with a faint creamy or pinkish tint	Weak, but noticeable	Strong Blue,green,reddish brownyellow	Not present	Triangular / Monoclinic	Metallic, SubMetallic	Distinct/Good	Irregular/ Uneven	Opaque
PYRITE	FeS	Pale brassyellow Yellowish white	Not present	Often weakly to distinctly anisotropic; blue green to orange red. This may due to polishing method, distortion caused by impurities (Stanton,1957, Klemm,1962b,	Not present	Cubes, pyritohedron,octahedron / Isometric	Metallic	None	Conchoidal to uneven	Opaque

				Gibbons,196 7, Saager and Mihalik,1967, Bayliss,1969)						
PYRRHOTITE	Fe7S8	Bronze brown	Strong Pinkish brownish - brownish yellow	Strong Dark brown to bluish grey		Pseudohexagonal / Monoclinic	Metallic	None Observed	SubConchoidal to uneven	Opaque
SPHALERITE	ZnS	Grey(in air) in oil very dark grey.sometimes with abrownish tint	Not present	Isotropic	Always visible,esp in oil and in badly polished spec.; for Fe-rich spec reddish brown; for Fe-poor spec. yellowish brown or yellowish white	Cubic/ Isometric	Adamantine, Resinous: non-metallic	Prefect	Conchoidal	Transparent to opaque

STIBNITE	Sb2S3	Metallic silvergray	Strong	Very strong	Not present	Prismatic / Orthorhombic	Metallic	None	Uneven/ subconchoidal	Opaque
QUARTZ	SiO2	Colourless, white, smokey, variously colored	Not present	Anisotropy	Milky	Hexagonal	Vitrous/ waxy/ glassy	Seldom exhibit parting	Conchoidal	Transparent to opaque
HEMATITE	Fe2O3	Gray white; pyrite ,bluish gray,	weak	Distinct,gray,blue , gray yellow	Deep red common	subparallel	metallic	perfect	Conchoidal	Transparent to opaque

4.6 Phases Analysis using XRD and SEM

To identify the phases of mineral in the hand specimen perform by the XRD and SEM. Both are use in mineral characterization but basically XRD attempts to characterize the material by analysing the crystal structure, and comparing it against a database of known structures. While, EDS is an elemental analysis technique by stimulating the sample by electrons or high-energy photons, and detect the spectrum of outgoing photons.

To determine the optimum milling parameter towards the sulphide ore presence in Malaysia, the analytical experiment is conduct using the MINITAB'16 as describe in next sub-topic. To determine whether the occurrence of phase changes in samples depend on the speed rate and milling time. the two samples of Buffalo Reef region which GSN2 and GSS1 as representative for North and South region were sent to XRD after the milling using planetary mill with different time and speed rate As shown in Figure () , the XRD pattern obtain revealed that the Buffalo Reef mainly contain quartz. Rietveld refinement (separation of overlapping peaks, structure determination, phase conformation and quantitative phase analysis) was performed after phase identification using PANAlytical X'Pert Highscore Plus software.

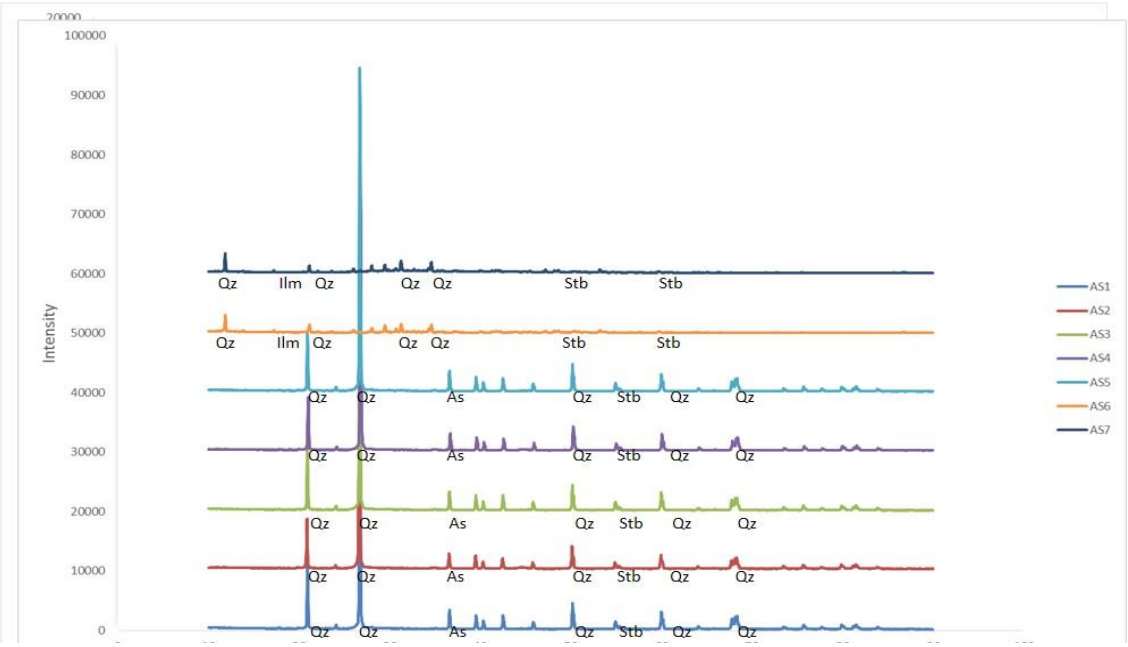


Figure 4.6.1 Results XRD of GSN2

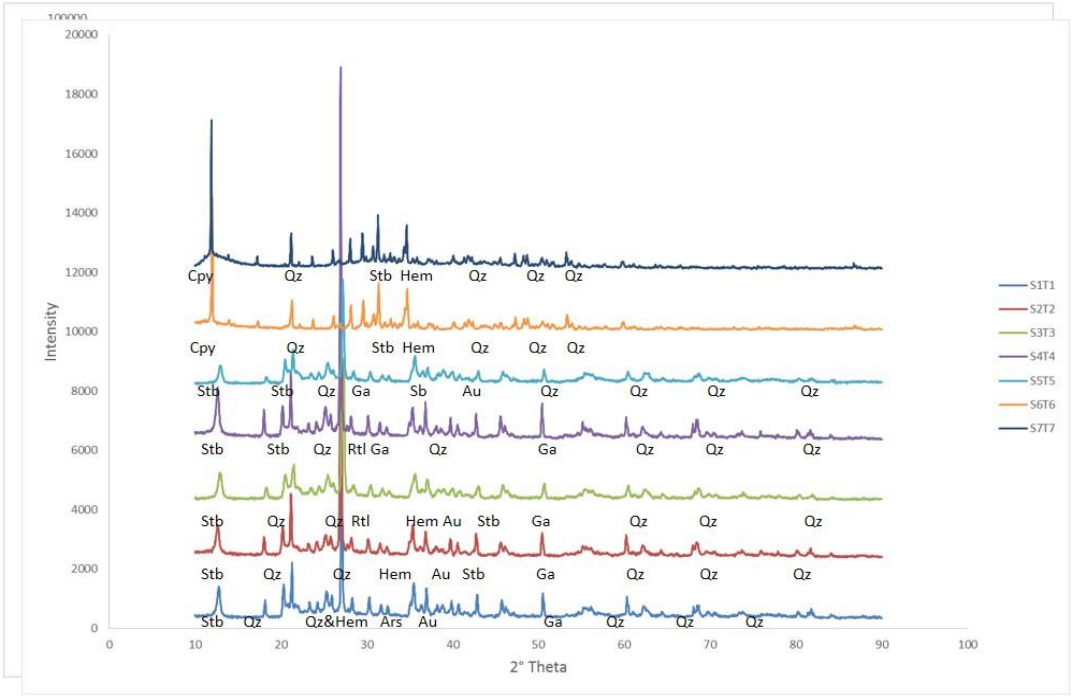


Figure 4.6.2 Result XRD of GSS1

Based on Figure13 GSN2, the seven times of experiment runs were preform with detailed parameter. The interpretation data from XRD show the most significance phase for each run is quartz, were major existence at the rock sample. The AS1,AS2, AS3,AS4 andAS5 show that the significant phase were quartz which is 98.7%, 98.7%, 99.6%,97.7% and 98.6% respectively. Due the presence of large particle, the major phases for AS6 and AS7 is quartz and stibnite with value of 53.8% and 33.8% for the quartz and 20.0% and 25.8% for stibnite quantity.

Even though only two phases can be get from this interpretation, the goodness of fit of retrieve for AS1, AS2, AS3, AS4, AS5, AS6 and AS7 is 19.61, 11.707, 9.65, 6.907, 9.08 28.81 and 32.93 respectively. Base on the figure above, mostly the runs of 7^{th} and 6^{th} of the samples, there is a shifted crystallinity toward the different peaks .

Based on Figure14 GSS1 , the seven times of experiment runs were preform with detailed parameter. The interpretation data from XRD show the most significance phase for each run is quartz, were major existence at the rock sample. The S1T1,S2T2, S3T3,S4T4 and S6T6 show that the significant phase were quartz which is 41.9 %, 46.3%, 69.2%,51.1% and 28.4% respectively. The second major of phase which is hematite of each samples runs is different, for S1T1, S2T2, S3T3 and S6T6 is 26.8%,14.5%12.1%and 24.4% respectively. The second highest phase present for S5T5 and S4T4 is rutile which is 32.3% and 58.2% respectively. The highest phase present for S7T7 is arsenopyrite within 28.6%.

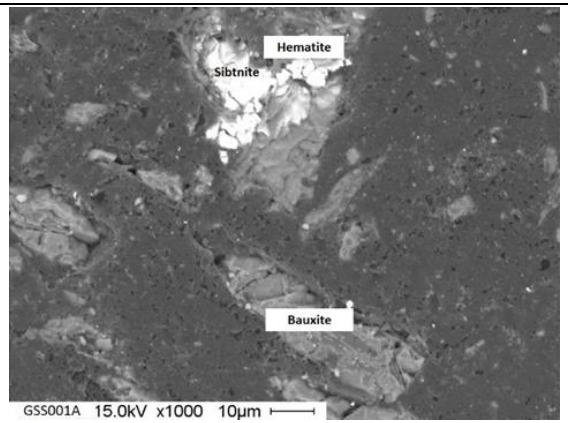
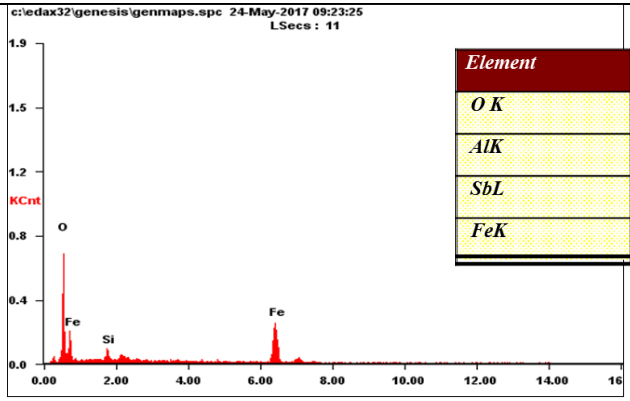
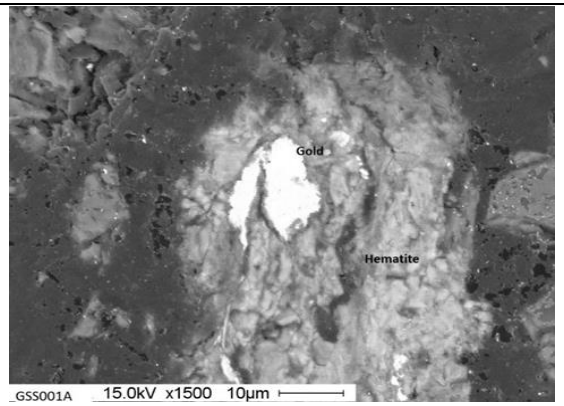
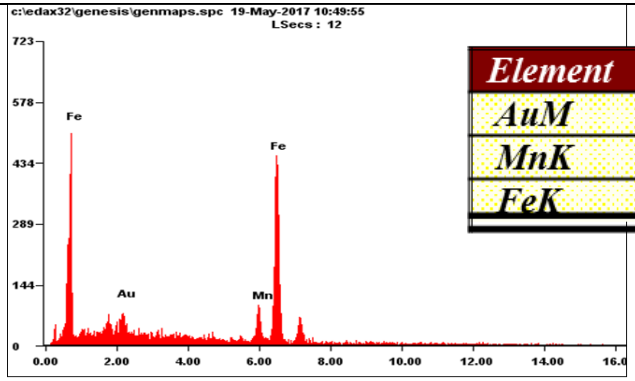
Even though only two phases can be get from this interpretation, the goodness of fit of retrieve for S1T1, S2T2, S3T3, S4T4, S5T4, S6T6 and S7T7 is 25.29, 26.94, 19.49, 22.39, 17.60, 33.21 and 34.49 respectively. Due to the different milling parameter, the phases occur are also different for each runs.

There are still more phases actually can be gain from this Xrd data. However it could not be interpret as the peak value of diffraction are same with the others. The others value are overlapping each other and not all are be tabulated in the data.

4.7 SEM and EDX Investigations

The scanning electron microscope (SEM) was used to examine the contamination at a higher magnification to determine whether the contamination was imbedding on the surface of the mineral (surface contamination), embedded in the surface (contamination from the pressing operation) or within the surface (contamination in the polished section). Through SEM and EDX analysis, contamination has been identified in most of sulphide ore. Sulfide minerals have a restricted composition range and solid solutions are rare. Most sulfides (except sphalerite) are opaque semiconductors with metallic luster which need to be examined carefully.

The samples of GSS1, GSS2, GSN1 and GSN2 in the size of +0.075mm and -0.075mm are investigated by the SEM and EDX. The details of the investigation as in the Figure 15 Figure 16, Figure 17 and Figure 18.

Samples	Size Fraction (mm)	Image	Graph
GSS1	-0.075		
	+0.075		

Element	Wt%	At%
O K	25.27	64.51
AlK	04.10	06.20
SbL	56.52	18.96
FeK	14.11	10.32

Element	Wt%	At%
AuM	06.19	01.83
MnK	12.07	12.81
FeK	81.74	85.35

Figure 4.7.1 SEM and EDX Investigations in Sample GSS1

From the figure above of sample GSS1, at size fraction of -0.075mm the element that exist in the specimen were oxide, aluminum, iron and antimony. The weight percentage of each element is well calculated which is 25.27%,, 0.41%, 56.52% and 14.215 respectively. The iron show the highest value of weight percent at 56.52% compared with 25.27% oxide,0.41 % aluminum, and 25.27% antimony. This indicate that sample GSS1 contain more Fe and O, which same as hematite chemical formula, Ferric(11) Oxide (Fe2O3). When undergo smaller in size, the iron element and antimony can be seen. At size -0.075mm, the content of sulphur and iron in the sample GSS1 is abundant. Possible mineral that might be there were arsenopyrite ,pyrrhotite., Stibnite and alumina minerals.

From the figure above of sample GSS1, at size fraction of +0.075mm the element that exist in the specimen were gold, manganese and iron. The weight percentage of each element is well calculated which is 6.19%, 12.07% and 81.74% respectively. The iron show the highest value of weight percent at 81.74.% compared with 6.19% gold, and 12.07% manganese. This indicate that sample GSS1 contain liberated gold associated with pyrite due the presence of high contain of iron. The gold mineralize in fluid inclusion due to the hydrothermal vein deposits. Based on the table 3, the element weight percentage of Au is only 0.00037% of total weight percent. The smaller the size of liberation, the percentage of discovering the formation of gold is increases. The gold grain intergrowth within the formation of pyrite and chacolpyrite.

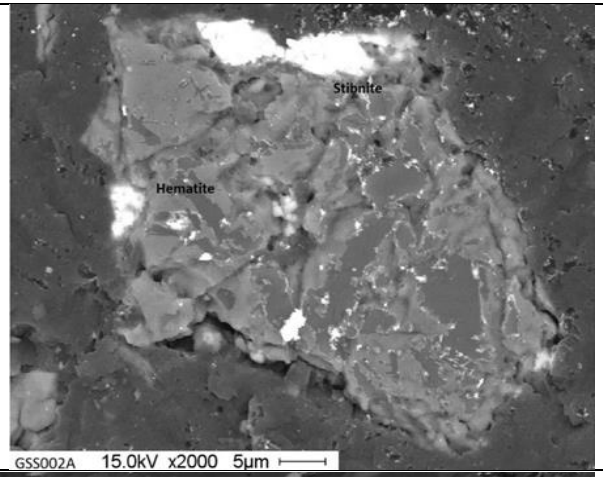
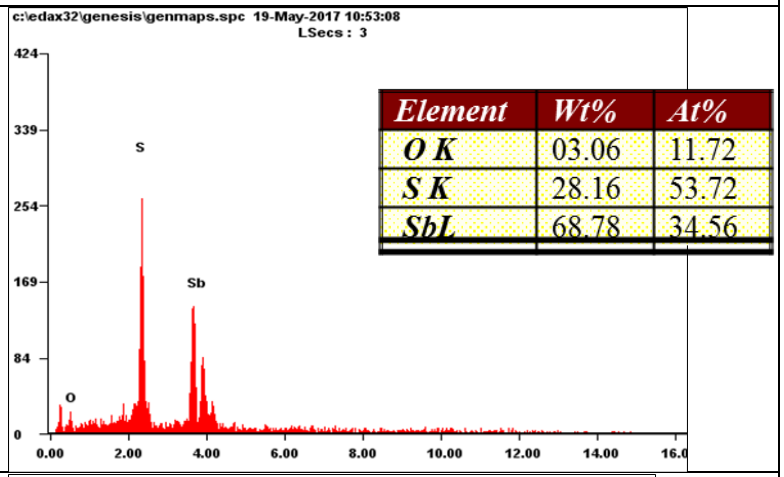
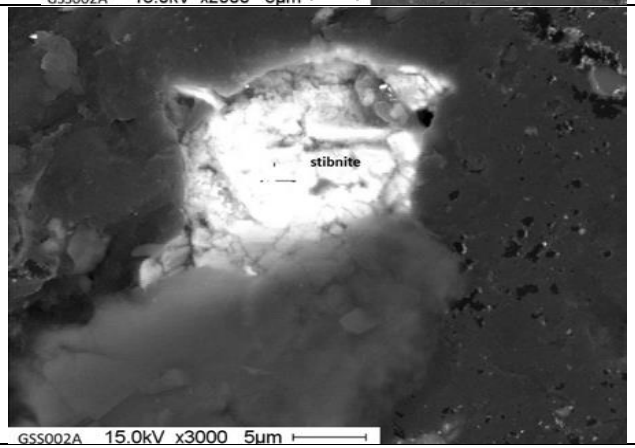
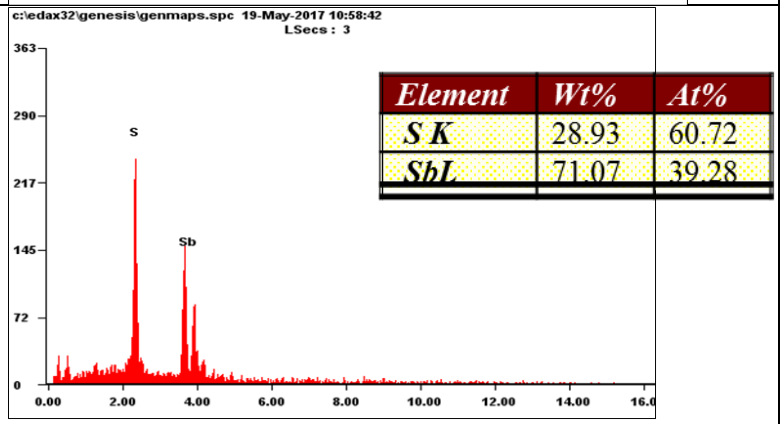
Samples	Size Farction (mm)	Image	Graph
GSS2	-0.075		
	+0.075		

Figure 4.7.2 SEM and EDX Investigation of GSS2 sample at -0.075mm and +0.075mm

From the figure 4.7.2 of sample GSS2, at size fraction of -0.075mm the element that exist in the specimen were oxide, sulphur and antimony. The weight percentage of each element is well calculated which is 3.06%,, 28.16% and 68.78% respectively. The antimony show the highest value of weight percent at 68.78% compared with 28.16% sulphur and 3.06%. This indicate the presence of massive stibnite in the sample GSS1, and the stibnite chemical formula, S3Sb2. When undergo smaller in size, the iron element and antimony can be seen. At size -0.075mm, the content of sulphur and iron in the sample GSS1 is abundant. Possible mineral that might be there were pyrite base on the dark gray along the angular white-gray (Possible stibnite).

From the figure 4.7.2 of sample GSS1, at size fraction of +0.075mm the element detected are sulphur and antimony. The major mineral formed at the size fraction +0.075mm of GSS2 sample is stibnite. From the image EDX, the color of white dull region indicated the formation of prismatic and orthombic shape of stibnite. The content of Sulphur is higher because the sulfides mineral associate enormously in the GSS2 sample.

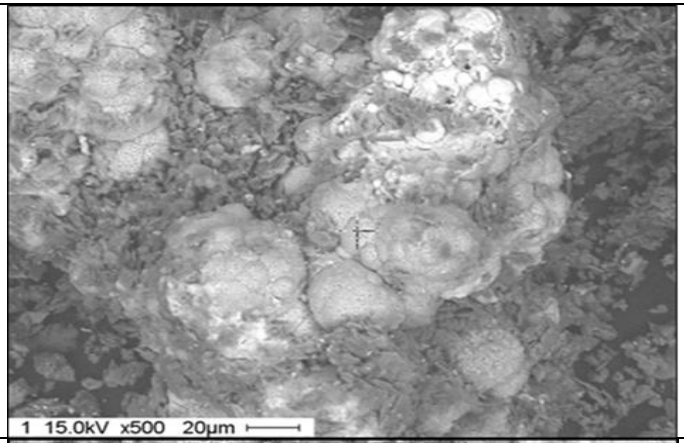
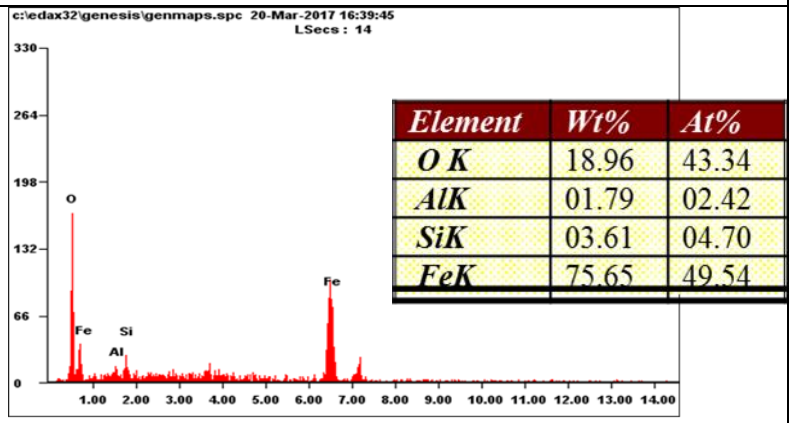
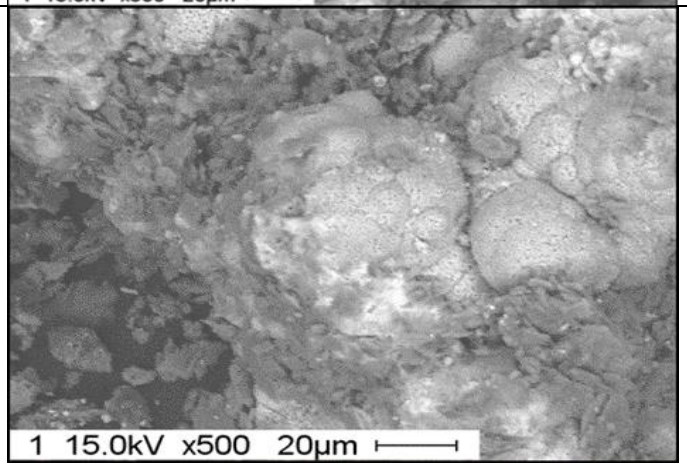
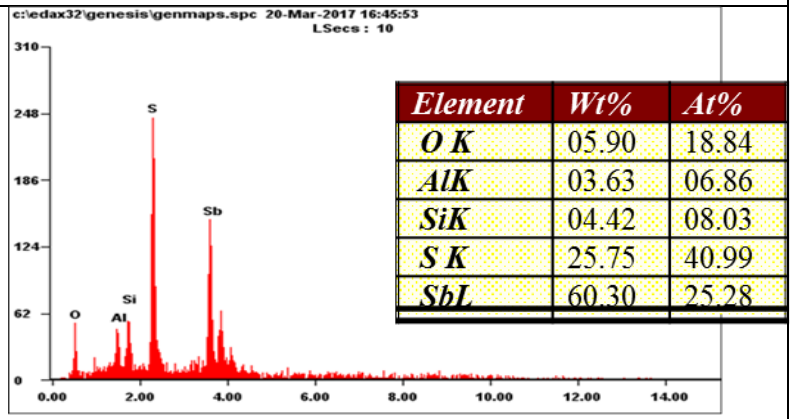
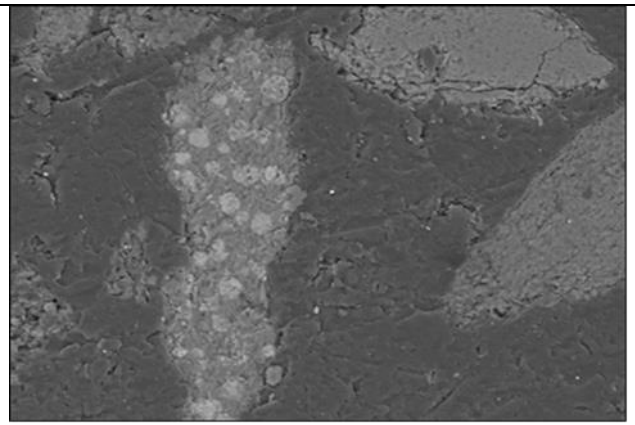
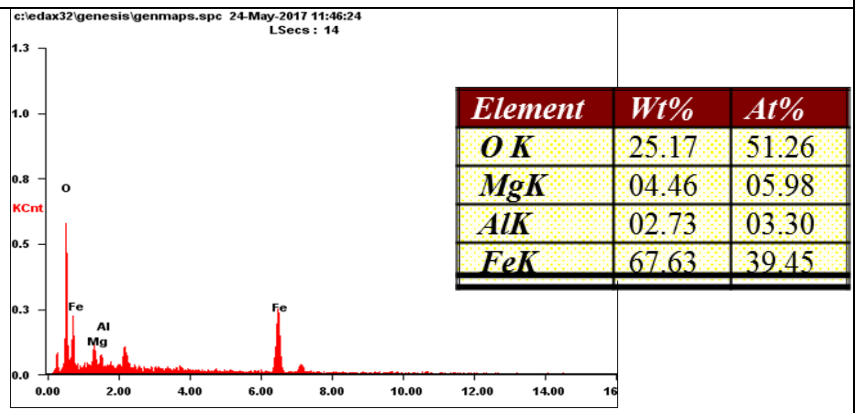
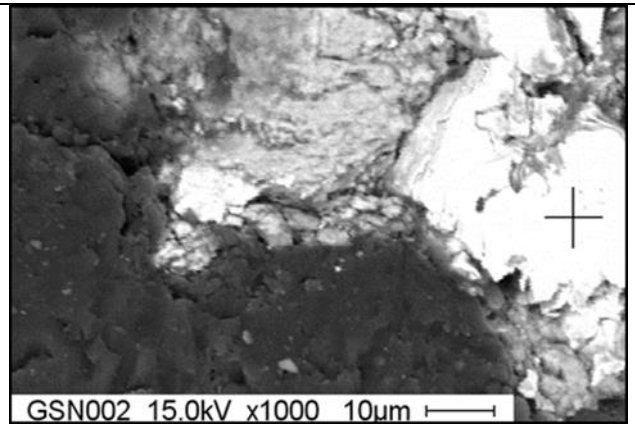
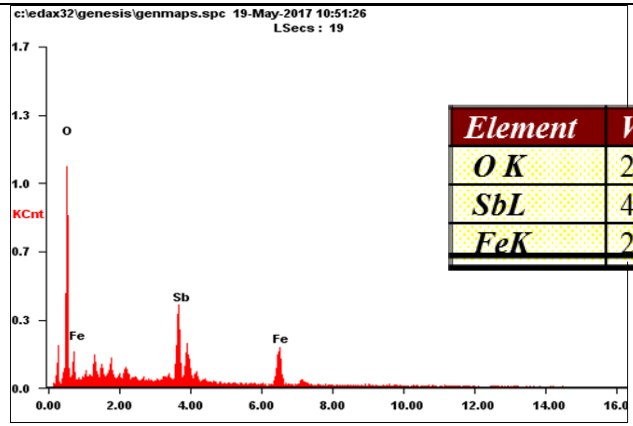
Sample	Size Fraction (mm)	Image	Graph
GSN1	-0.075		
	+0.075		

Figure 0-1 SEM and EDX investigation of GSN1 sample at -0.075mm and +0.075mm.

From the figure 17, the hand specimen is investigate as the bulk specimen when undergo the characterization process using SEM and EDX due to technical problem during the experiment session. In the sample GSN1, at size fraction of -0.075mm the element that exist in the specimen were oxide, aluminium, silicon, and iron . The weight percentage of each element is well calculated which is 18.96.%,, 1.79%, 3.61% and 75.65% respectively. The iron show the highest value of weight percent at 75.65% compared with 18.96% oxide and 3.61% silicon .This indicate the presence of massive sulfides mineral mainly pyrite in the sample GSN1. From the image of EDX the crystallography of pyrite reveal the crystal system and the colour of iron

From the figure 6 of sample GSN1, at size fraction of +0.075mm the element detected are oxide, aluminium, silicon, sulphur and antimony. the antinomy show the highest weight percentage, 60,30% compare to the 5.90% oxide, 3.63% aluminium , 4.42% silicon and 25.75% of sulphur. The major mineral formed at the size fraction +0.075mm of GSN1 sample is stibnite. From the image EDX, the color of white dull region indicated the formation of prismatic and orthombic shape of stibnite. The content of Sulphur is higher because the sulfides mineral associate enormously in the GSN2 sample mostly light-dark gray.

Samples	Size Fraction (mm)	Image	Graph
GSN2	-0.075		
	+0.075		

Element	Wt%	At%
O K	25.17	51.26
MgK	04.46	05.98
AlK	02.73	03.30
FeK	67.63	39.45

Element	Wt%	At%
O K	23.01	60.94
SbL	47.11	16.39
FeK	29.88	22.67

Figure 4.7.4 SEM and EDX Investigation in sample GSN2 at -0.075mm and +0.075mm.

From the figure 4.7.4, the hand specimen is investigate as polish section which is non-conductive and undergo coating with gold coat and then proceed to characterization process using SEM and EDX . In the sample GSN2, at size fraction of -0.075mm the element that exist in the specimen were oxide, magnesium, aluminium , and iron . The weight percentage of each element is well calculated which is 25.17.%,, 4.46%, 2.73% and 67.63% respectively. The iron show the highest value of weight percent at 67.63% compared with 25.17% oxide and 4.46% magnesium . This indicate the presence of massive sulfides mineral mainly pyrite in the sample GSN2. From the image of EDX , the occurrence of two distinct colour in mineral between the dark-grey and the light grey show the iron contain with the association during the deposition of mineral.

From the figure 4.7.4 of sample GSN2, at size fraction of +0.075mm the element detected are oxide, antimony, and iron. The antimony show the highest weight percentage, 29.80% compare to the 23.01% oxide, 29.88% iron. The major mineral formed at the size fraction +0.075mm of GSN2 sample is stibnite and possible pyrite. From the image EDX, the color of white dull region indicated the formation of prismatic and orthombic shape of stibnite. The content of Sulphur is higher because the sulfides mineral associate enormously in the GSN2 sample mostly light-dark gray and the present of galena will indicate the white shine colour and cubic in crystal system.

4.8 Full Factorial Design

Full factorial design in Minitab software is a statistical method that being applied in the experiment to determine the effects of the manipulated parameters and their interaction. By using this full factorial design analysis, it can analyze the all possible combination of factors in the experiment.

Table 4.8.1 described the code and the design of factorial. The value of high level and low level for each parameter is determined. This code is used during the model fitting to describe the coded regression model. The relationship between X_1 and X_2 give interaction effects towards the percent particle size distribution.

Table 4.8.1: The two parameter with their coded and level values

Parameter	Code	Low Level	Medium Level	High level
Speed rotation (rpm)	X_1	100	250	400
Time Taken (minute)	X_2	12	21	30

Table above indicate the response of experiment can be obtained directly from the PSA result of Malvern Analysis. The results of particle Size analysis is obtained from the three corresponding results. Different degrees of parameters give differently results on PSA.

StdOrder	RunOrder	CenterPt	Blocks	Speed Rate(rpm)	Time (minute)	VMD	D50	s
3	1	1	1	100	30	6.84	4.96	2.76
2	2	1	1	400	12	4.67	2	3.31
1	3	1	1	100	12	8.17	4.81	2.5
5	4	0	1	250	21	8.69	4.36	2.77
6	5	0	1	250	21	7.94	4.61	2.44
7	6	0	1	250	21	10.6	4.89	2.22
4	7	1	1	400	30	6.59	1.98	3.47

Table 4.8.2 Experimental design matrix and its corresponding response for GSS1

Table 4.81.and 4.8.2 shows the analysis of variance, ANOVA for the particle size analysis in a coded unit for GSS1 and GSN2 respectively. ANOVA is applied to evaluate the significant of ay two main effects towards the response. The interaction between the parameter cannot be taken due to the unreliable regression values. The main focus on this experimental to determine the lower value of differences between R^2 and $R^{2.}$

Table 4.8.3 Analysis of Variance (ANOVA) for Particle Size Analysis of GSS1

Source	DF	Seq SS	Adj SS	Adj Ms	F	p
Main Effects	2	8.1620	8.16205	4.08102	5.22	0.077
Speed of rotation	1	6.7340	6.73402	6.73402	8.62	0.043
Time	1	1.4280	1.42803	0.79107		
Residual Error	4	3.1243	2.19220	2.19220	7.06	0.077
Curvauture	1	2.1922	0.91203	0.91203091203	90.90	0.011
Lack of fit	1	0.9120	0.02007	0.01000		
Pure Error	2	0.0201				
Total	6	11.2863				

Where ,DF= degree of freedom; Seq SS= sequential sum of square Adj SS= Adjusted sum of square;F= fisher's test data ; P= hypothesis.

In this full factorial design analysis, it is looked at the P value, T value and the F value in order to determine the significances of the parameters and their interactions. The analysis of the experiment is about 95% confidence level with 5% the alpha, α value. The results are significant, if the P value is less than 0.05 ($P<0.05$), else it will be not significant. This significant and not significant data can describe how much the main effect and their interactions affect the model fitted and what are the major parameters that influence the model the most.

Table 4.8.4: Estimated effects and coefficients for percent crystallinity in coded units for GSS1

Term	Effects	Coef	SE Coef	T	p
constant		6.067	0. 3340	18.16	0.00
Speed of rotatiom	-2.595	-1.297	0.4419	-2.94	0.043
Time	1.195	0.598	0.4419	1.35	0.243

Based on the table above, it shows the estimated value for effect, coefficient,SE coefficient, T and P value for every term. The p-va;ue for speed rotation (X_1) as highlight above in the table 7 is seen to be less than 0.043 and it show there is one significant effect. The time taken is not significant effect since the p-value is higher than 0.05 This indicate the time taken is not a significant toward the response which is particle size analysis for GSS1.

For GSN2 the procedure and analysis using ANOVA is similar to GSS1. The following table indicated for the ANOVA of GSS1.

Table 4.8.5 Analysis of Variance (ANOVA) for Particle Size Analysis of GSN2

Source	DF	Seq SS	Adj SS	Adj Ms	F	p
Main Effects	2	8.3852	8.38525	4.19262	6.59	0.054
Speed of ofrotation	1	8.3810	8.3810	8.38102	13.17	0.022
Time	1	0.042	0.00423	0.00423	0.01	0.939
Residual Error	4	2.5449	2.54492	0.63623		
Curvauture	1	2.3971	2.39710	2.39710	48.65	0.006
Lack of fit	1	0.0072	0.00722	0.00722	0.10	0.779
Pure Error	2	0.1406	0.14060	0.07030		
Total	6	10.9302				

Where ,DF= degree of freedom; Seq SS= sequential sum of square Adj SS= Adjusted sum of square;F= fisher's test data ; P= hypothesis.

Table 4.8.6 Estimaed Effects and Coefficient for GSN2

Term	Effects	Coef	SE Coef	T	p
constant		3.944	0.30105	13.08	0.000
Speed of rotation	-2.895	-1.227	0.3015	-3.63	0.022
Time	0.065	0.033	0.3988	0.08	0.939

Based on the table above, it shows the estimated value for effect, coefficient, SE coefficient, T and P value for every term. The p-value for speed rotation (X_1) as highlight above in the table 9 is seen to be less than 0.022 and it show there is one significant effect. The time taken is not significant effect since the p-value is higher than 0.05. This indicate the time taken is not a significant toward the response which is particle size analysis for GSN2.

Table 4.8.7 The different of Regression value

	GSS1	GSN2
R^2	72.32	76.72
R^2 adjst	58.48	65.07
Different	13.84	11.65

The different between R^2 and R^2 adjusted as shown in table 10. The closeness of R^2 to R^2 Adjusted provides insight into how good the model is. It is also implies a better statistical model is obtained. The normal probability plot (Appendix B) is the points of the percent particle size analysis.D50 against residual. It is used to determine either the points are normally distributed or not. The points are scattered around the straight line, indicates that the plot is normal distribution. However there are some points are potential to be outliers. These potential outliers need to check and cannot be rejected straight away. Since the plot is normal distribution, then it is a good model for this experiment.

Table 4.8.8 Estimated Coefficients for D59 for GSS1 GSN1 (uncoded)

Sample	**GSS1**	**GSN2**
Terms	**Coef**	**Coef**
Constant	6.83548	6.28095
Speed of Rotation	-0.00865	-0.00965
Time Taken	0.0663889	0.0036111

Based on the table 11, the regression model equation can be interpret as follow:

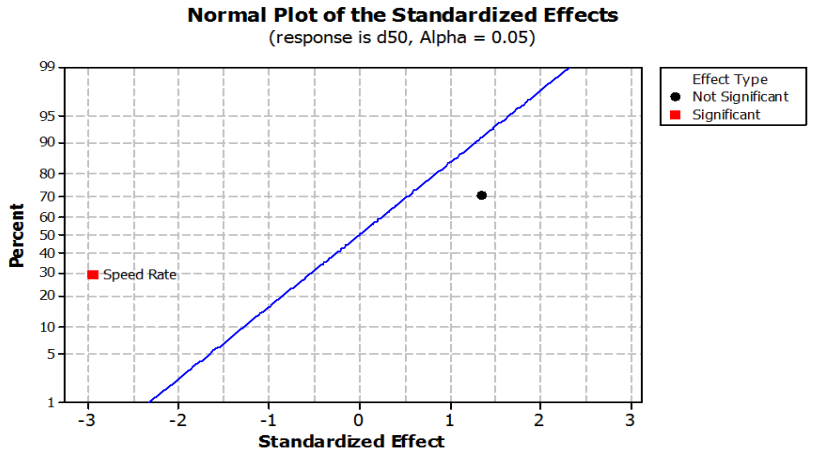
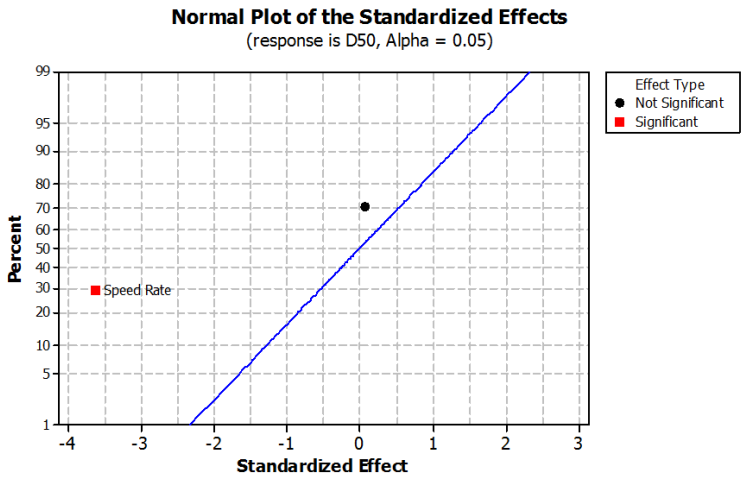
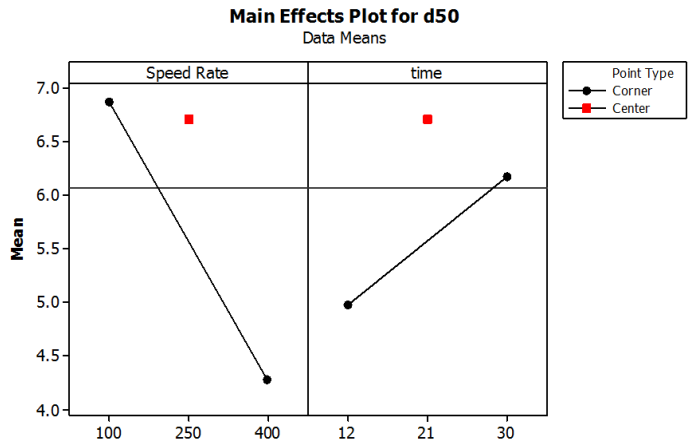
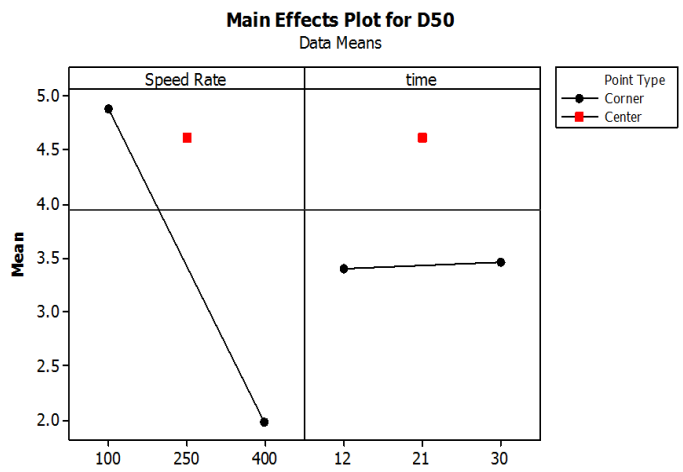
Coded:

GSS1: $\mathbf{Y_{d50} = 6.83548 + 0.0663889X_2 - 0.00865X_1}$ ..... **(4.1)**

GSN2: $\mathbf{Y_{d50} = 6.28095 + 0.0036111X_2 - 0.00965X_1}$ .....**(4.2)**

When X_1 is the speed of rotation coefficient (rpm) and X_2 time taken coefficient. Based on the regression model equation (4.2) and (4.1), it is explained on the main effects to the response. Speed of rotation, X_1 seems to be the major influences parameter to the particle size analysis, D50. Time taken, X2 also influence the response, but its are not as strong as the influences of speed of rotation. For future research, the best possible parameter which can give high accuracy and precise, the uses of equation GSS1 for south region and equation GSN2 for the north region.

Table 4.8.9 Graph of Main Effect and Normal Plot for GSS1 and GSS2

GSS1	GSN2
	
	

4.9 Percentage of Mineral Present in the Polish Section

The particles in the raw image were separated from the background by a process known as “threshold”. The purpose of this process is to measure the area of liberated samples images from each sample based on the size. The sample of GSS1, GSS2, GSN1 and GSN2 from the image optical microscope of size 0.15mm were used in the Image J software. The techniques used involve the threshold adjusted image in order to clarify the sub area that want to be calculate.

The area of liberated mineral is calculate in threshold value and it only can be estimation. The area is in pixel.

GSS1

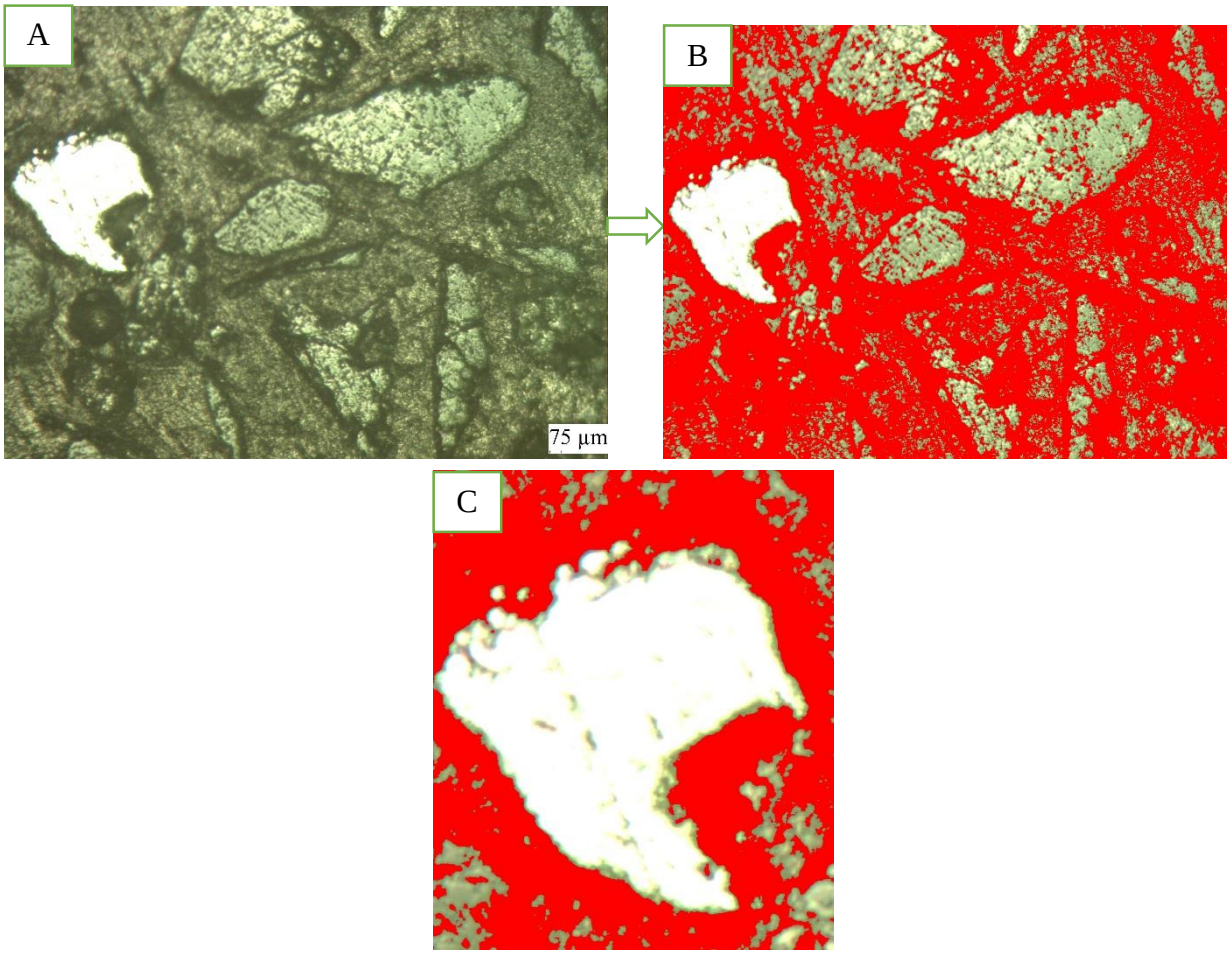


Figure 4.9.1 Mineral Liberation of GSS1 usig image-J

Table 0-2 Analysis Result of Image J

Label	Area	Mean	Min	Max	Circ.	%Area	AR	Round	Solidity
B	8372880	112.408	0	255	0.759	0	1.446	0.691	1
C	922950	153.25	85	255	0.779	0	1.195	0.837	1

The percentage of mineral liberation:

$\frac{922950}{8372880}$X 100%= 11.02% , the mineral liberation for GSS1 at size 0.15mm is only

11.02%.

GSS2

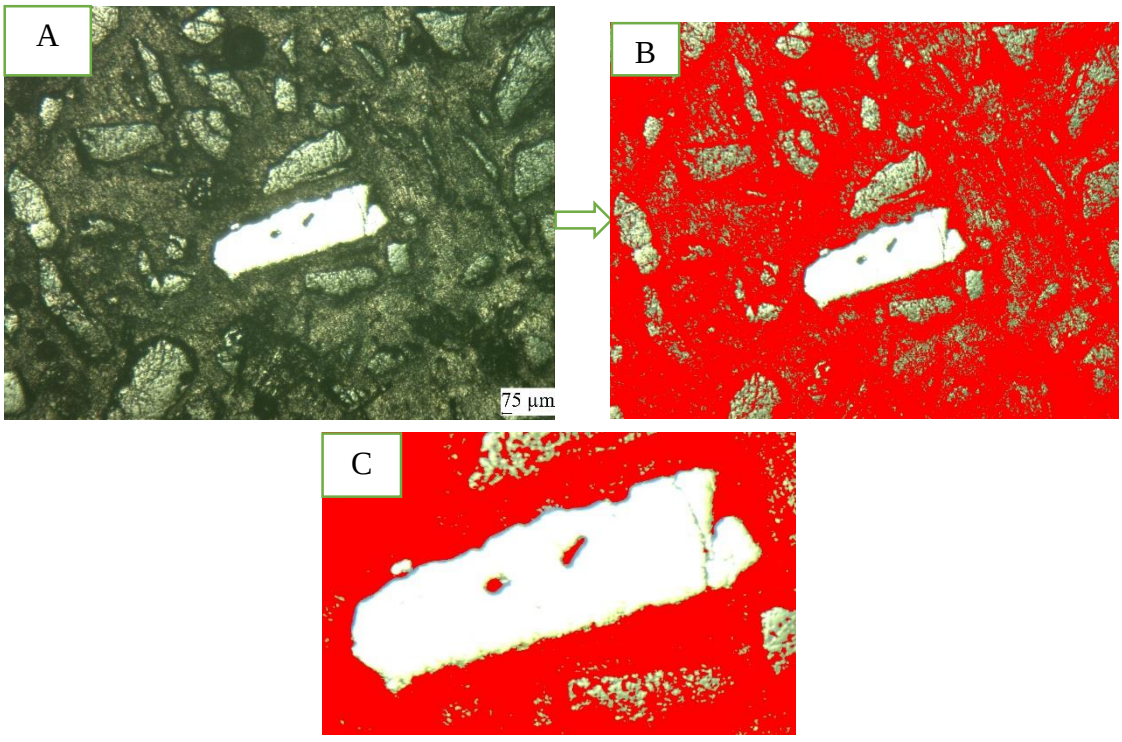


Figure 4.9.2 Mineral Liberation of GSS2 using imageJ

Table 0-3Analysis Result of Image J

Label	Area	Mean	Min	Max	Circ.	%Area	AR	Round	Solidity
B	8268480	102.548	85	255	0.757	0	1.465	0.683	1
C	1034488	139.859	85	255	0.747	0	1.569	0.637	1

The percentage of mineral liberation:

$\frac{1034488}{8268480}$ X100%= 12.51%

GSN1

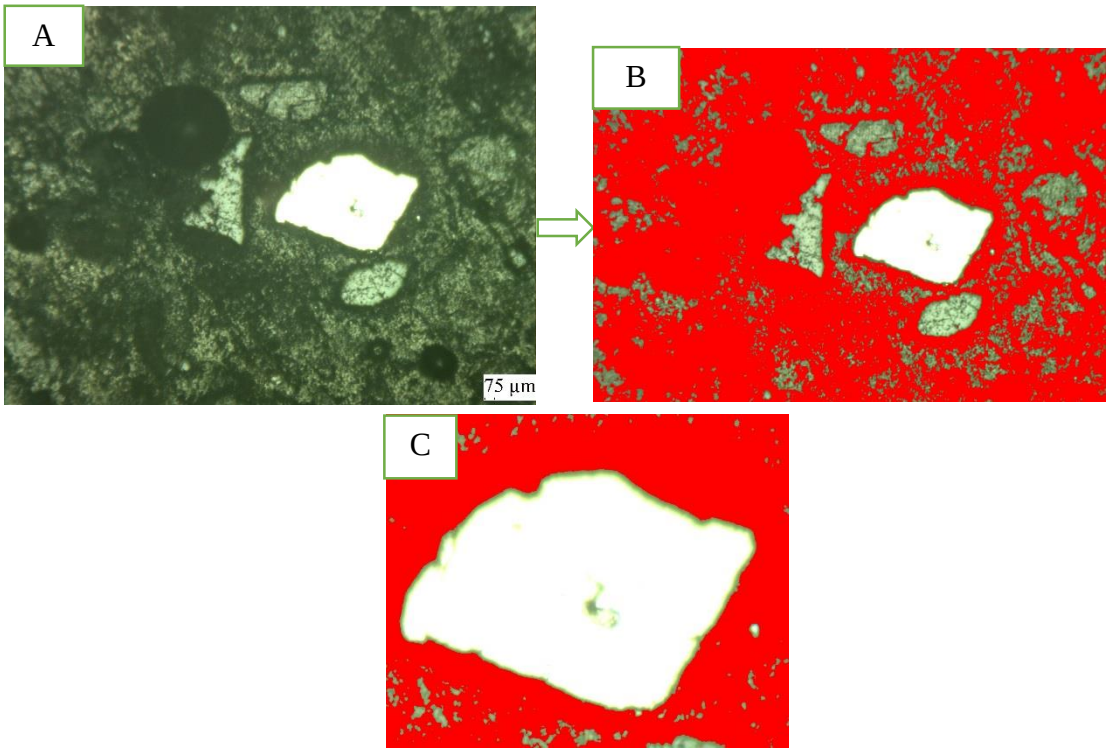


Figure4.9.3. Mineral liberation of GSN1 using Image J

Table 0-4 Analysis result of Image J

Label	Area	Mean	Min	Max	Circ.	%Area	AR	Round	Solidity
B	8352000	99.147	73	255	0.759	0	1.45	0.69	1

C	951936	153.955	85	255	0.778	0	1.207	0.828	1

The percentage of mineral liberation:

$\frac{951936}{8352000}$x100%=11.39%

GSN2

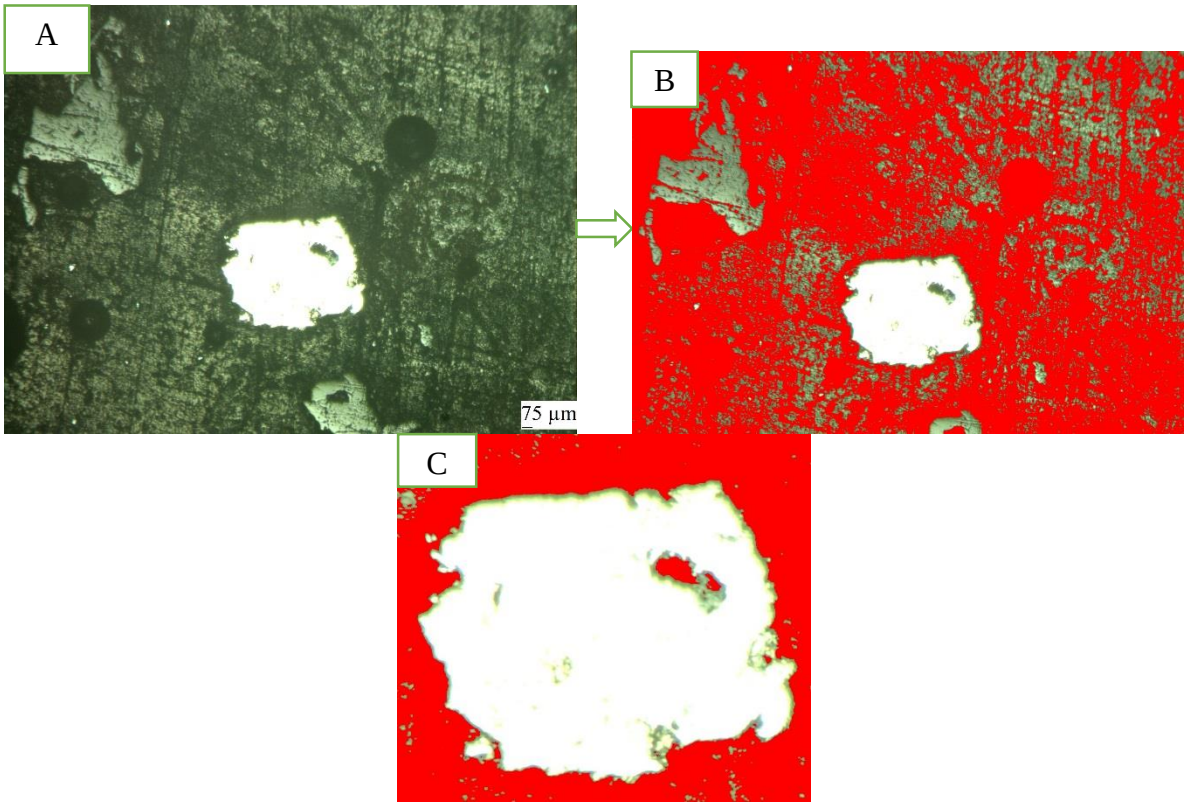


Figure 4.9.4 Mineral Liberation of GSN2 using Image J

Table 0-5 Analysis Result of Image J

Label	Area	Mean	Min	Max	Circ.	%Area	AR	Round	Solidity
	8308800	101.28	80	255	0.76	0	1.442	0.693	1
	817920	176.555	85	255	0.783	0	1.127	0.888	1

The percentage of mineral liberation:

$\frac{817920}{8308800}$x100%= 9.84%

CHAPTER 5:

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From the result and discussion on previous chapter, it can conclude that the d_{90} of the sample GSS2, GSN1 and GSN2 are below 4.0mm while the GSS1 is exceeded 4.0mm. The ore body at North zone of Buffalo Reef are more oxidize due to the closer the distance from the surface which the ore body is nearly full weathered, causes the LOI for GSN1 and GSN2 is higher. The major element present in the most of the samples of Buffalo Reef is silica because the orebody is near to the surface. GSS and GSN ore are mostly made up of the minerals; arsenopyrite, pyrrhotite ,pyrite galena , and stibnite. All the minerals are observed under polarizing light microscopy and SEM with the aid from the results of XRF and XRD minerals data consisting. From the XRD result, the different sample with different milling parameter yield the different phases of crystallite The numerous of polish section been investigate using SEM and EDX, to study the phases and mineral composition, show that the stibnite and arsenopyrite is abundant at the Buffalo Reef sample. From the various graph and table from ANOVA, the rotation speed of planetary mill play a significance towards the respond of particle size analysis,$d_{50.}$ Liberation of mineral using threshold value by image J conclude the liberation of mineral start at the size fraction 0.15mm(Gao et al. 2015)

5.2 Recommendation

For comprehension of geological survey, the proper sampling is need to access the geological data.It is really important to do the chemical process in order to identify the possible minerals and further the application on the industry about the precious minerals. The quality of the image taken either from optical microscope or SEM is very important for further liberation study.

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APPENDICES

APPENDIX A

Particle Size Distribution after the sieve.

GSN1

Sieve Size Range	Size Fraction Weight (g)	Weight Percentage (%)	Nominal Aperture Size (mm)	Cumulative Percentage Undersize (%)	Cumulative Percentage Oversize (%)
+ 4.75	46.51	1.38%	4.75	98.62%	1.38%
-4.75 + 3.35	275.116	8.19%	3.35	90.42%	9.58%
-3.35 + 2.36	924.58	27.53%	2.36	62.90%	37.10%
-2.36 + 1.18	952.17	28.35%	1.18	34.55%	65.45%
-1.18 + 0.60	408.01	12.15%	0.6	22.40%	77.60%
-0.60 + 0.425	133.14	3.96%	0.425	18.44%	81.56%
-0.425 + 0.30	161.7	4.81%	0.3	13.62%	86.38%
-0.30 + 0.212	317.52	9.45%	0.212	4.17%	95.83%
-0.212 + 0.15	17.69	0.53%	0.15	3.64%	96.36%
-0.15 + 0.090	55.01	1.64%	0.09	2.01%	97.99%
-0.090 + 0.075	20.32	0.60%	0.075	1.40%	98.60%
-0.075	47.09	1.40%	0.07	0.00%	100.00%

GSN2

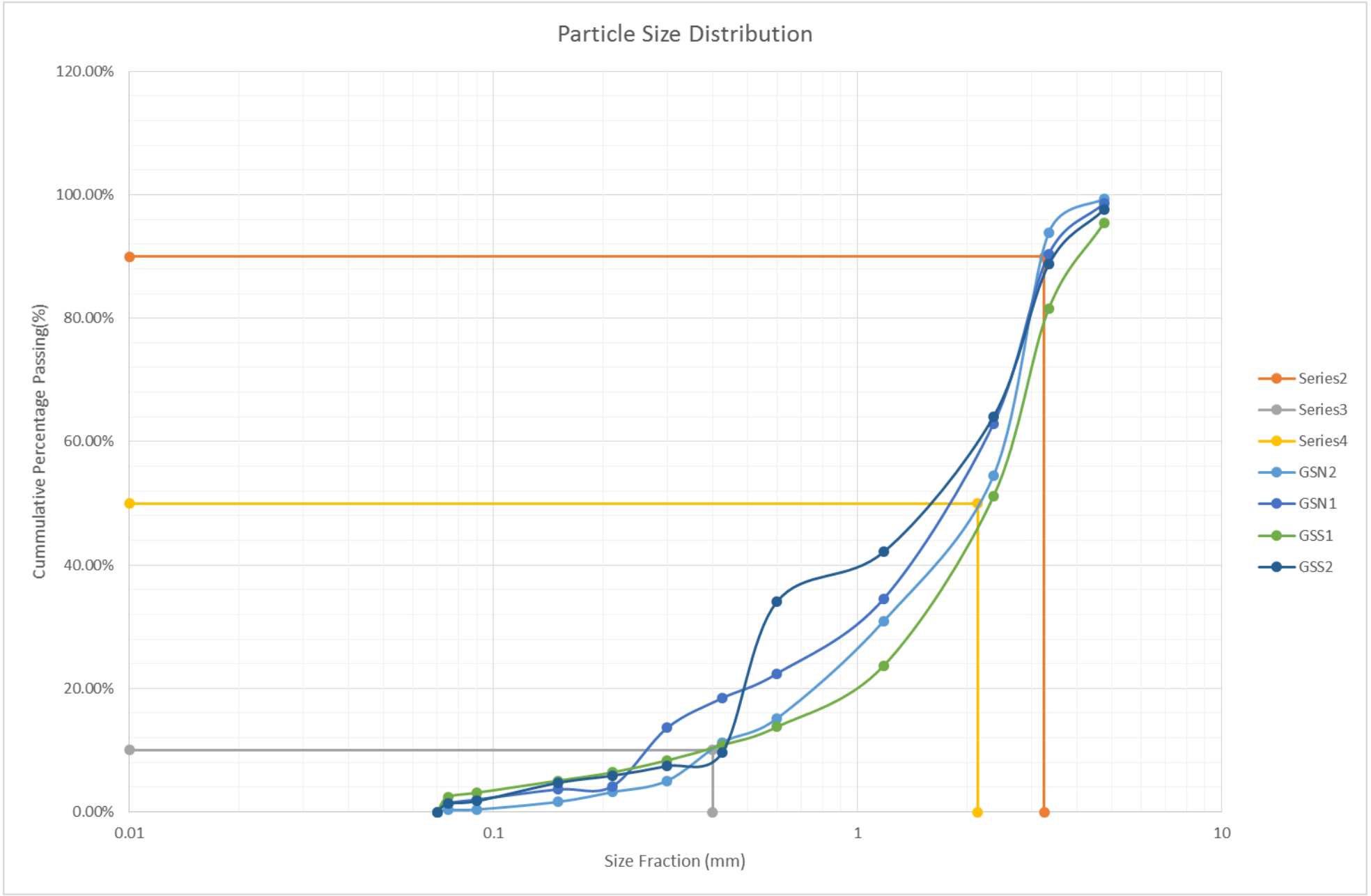
Sieve Size Range	Size Fraction Weight (g)	Weight Percentage (%)	Nominal Aperture Size (mm)	Cumulative Percentage Undersize (%)	Cumulative Percentage Oversize (%)
+ 4.75	16.94	0.63%	4.75	99.37%	0.63%
-4.75 + 3.35	146.92	5.48%	3.35	93.89%	6.11%
-3.35 + 2.36	1057.4	39.44%	2.36	54.45%	45.55%
-2.36 + 1.18	630.23	23.51%	1.18	30.94%	69.06%
-1.18 + 0.60	423.84	15.81%	0.6	15.14%	84.86%
-0.60 + 0.425	103.67	3.87%	0.425	11.27%	88.73%
-0.425 + 0.30	166.59	6.21%	0.3	5.06%	94.94%
-0.30 + 0.212	49.35	1.84%	0.212	3.22%	96.78%
-0.212 + 0.15	42.45	1.58%	0.15	1.63%	98.37%
-0.15 + 0.090	33.15	1.24%	0.09	0.40%	99.60%
-0.090 + 0.075	1.66	0.06%	0.075	0.33%	99.67%
-0.075	8.94	0.33%	0.07	0.00%	100.00%

GSS1

Sieve Size Range	Size Fraction Weight (g)	Weight Percentage (%)	Nominal Aperture Size (mm)	Cumulative Percentage Undersize (%)	Cumulative Percentage Oversize (%)
+ 4.75	161.197	4.53%	4.75	95.47%	4.53%
-4.75 + 3.35	494.62	13.89%	3.35	81.58%	18.42%
-3.35 + 2.36	1084.49	30.46%	2.36	51.11%	48.89%
-2.36 + 1.18	975.56	27.40%	1.18	23.71%	76.29%
-1.18 + 0.60	353.82	9.94%	0.6	13.77%	86.23%
-0.60 + 0.425	104.59	2.94%	0.425	10.83%	89.17%
-0.425 + 0.30	88.18	2.48%	0.3	8.35%	91.65%
-0.30 + 0.212	69.47	1.95%	0.212	6.40%	93.60%
-0.212 + 0.15	47.9	1.35%	0.15	5.06%	94.94%
-0.15 + 0.090	69.25	1.95%	0.09	3.11%	96.89%
-0.090 + 0.075	23.99	0.67%	0.075	2.44%	97.56%
-0.075	86.74	2.44%	0.07	0.00%	100.00%

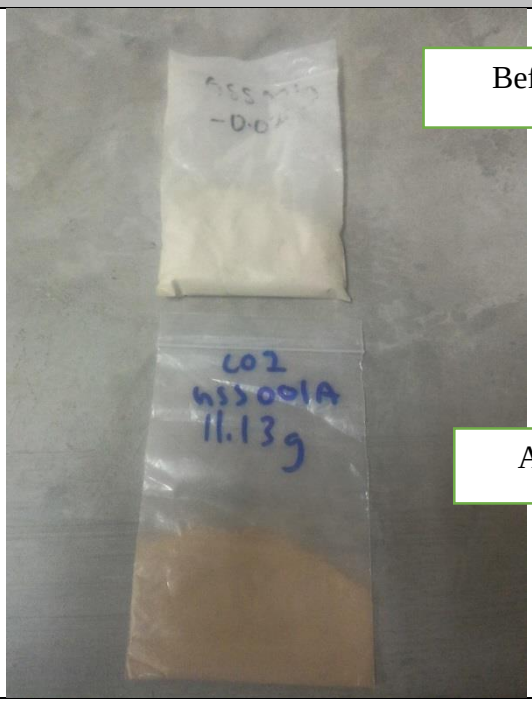
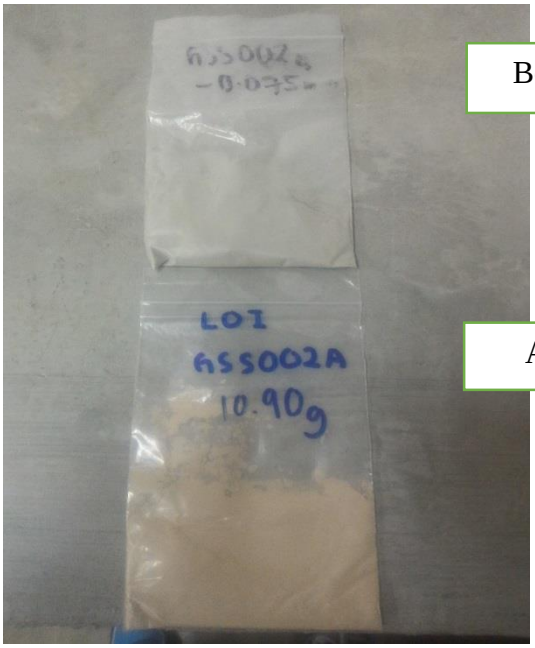
GSS2

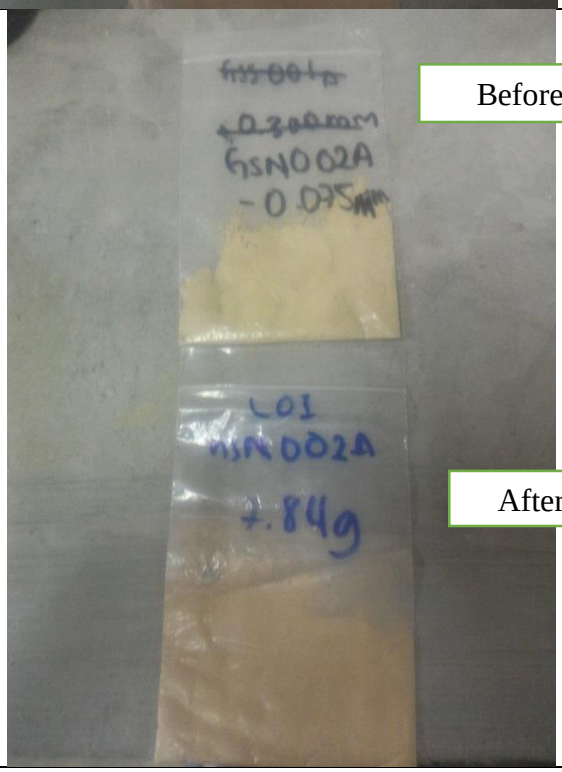
Sieve Size Range	Size Fraction Weight (g)	Weight Percentage (%)	Nominal Aperture Size (mm)	Cumulative Percentage Undersize (%)	Cumulative Percentage Oversize (%)
+ 4.75	100.67	2.37%	4.75	97.63%	2.37%
-4.75 + 3.35	377.69	8.89%	3.35	88.74%	11.26%
-3.35 + 2.36	1050.67	24.72%	2.36	64.02%	35.98%
-2.36 + 1.18	929.92	21.88%	1.18	42.14%	57.86%
-1.18 + 0.60	343.98	8.09%	0.6	34.04%	65.96%
-0.60 + 0.425	1037.79	24.42%	0.425	9.62%	90.38%
-0.425 + 0.30	92.71	2.18%	0.3	7.44%	92.56%
-0.30 + 0.212	66.42	1.56%	0.212	5.88%	94.12%
-0.212 + 0.15	50.17	1.18%	0.15	4.70%	95.30%
-0.15 + 0.090	125	2.94%	0.09	1.76%	98.24%
-0.090 + 0.075	17.53	0.41%	0.075	1.34%	98.66%
-0.075	57.09	1.34%	0.07	0.00%	100.00%


Particle Size Distribution
120.00%
100.00%
80.00%
60.00%
40.00%
20.00%
0.00%
Cummulative Percentage Passing(%)
0.01
0.1
1
10
Size Fraction (mm)
Series2
Series3
Series4
GSN2
GSN1
GSS1
GSS2

APPENDIX B

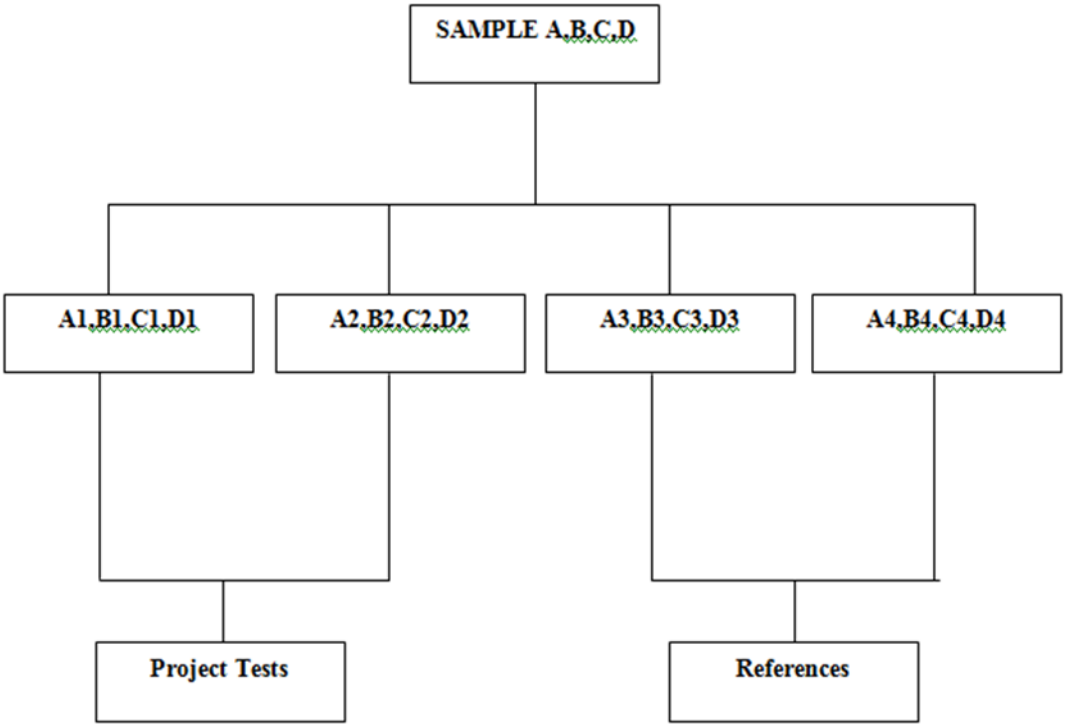
The color changes after and before Loss on Ignition

Sample	Colour Changes	Explanation
GSS001A		Before: White-Gray After: Slightly brown
GSS002A		Before: Gray After: milky brown.

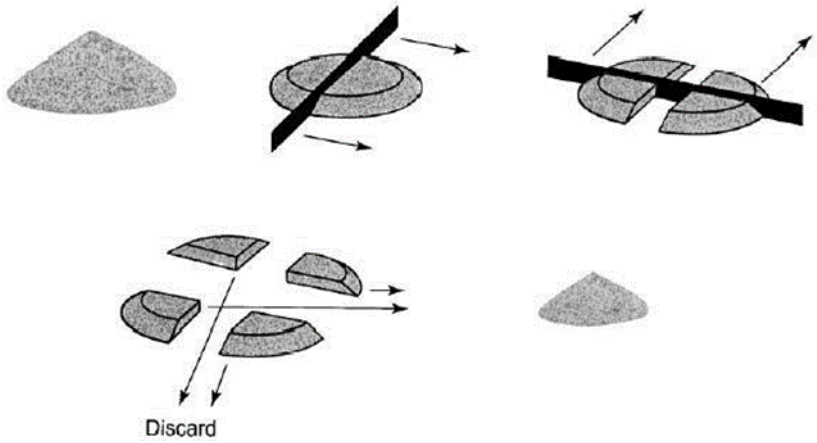
GSN001A		Before: slightly Brown After : slightly dark-brown
GSN002A		Before: Slightly-Brown After: Milky Brown.

APPENDIX C

The proper sampling:;



GSS1	GSS2	GSN1	GSN2
A	B	C	D



APPENDIX D

Visual Assessment:

The raw sample of North region
The sample is dry under the sun.

The raw sample North region.
The sample is dry under the sun

The raw sample from the south region. The sample is white and gray.

The raw sample from the South region. The presence of dark colour due to iron content

The sample size is analyze

The sample size is analyze

	
The formation of iron bearing can be seen visually	The sample size is analyze.