SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING

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PHYSICAL AND CHEMICAL CHARACTERIZATION OF IRON ORE FROM EAST COAST OF PENINSULAR MALAYSIA

By

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled “The Characterization of Iron Ore from East-Coast of Peninsular Malaysia”. I also declare that it has not been previously submitted for the award of any degree nor diploma or other similar title of this for any other examining body or University.

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LIST OF ABBREVIATIONS

Al	Aluminium
Al_2O_3	Aluminium Oxide
Fe	Iron
Fe_2O_3	Hematite
Fe_3O_4	Magnetite
$FeTiO_3$	Ilmenite
$FeCO_3$	Siderite
K	Potassium
Mm	Milimeter
Qtz	Quartz
S	Sulphur
SEM	Scanning Electron Microscope
Si	Silicon
SiO_2	Silica
Ti	Titanium
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
µm	Micrometer

PENCIRIAN FIZIKAL DAN KIMIA BIJIH BESI DARI PANTAI TIMUR SEMENANJUNG MALAYSIA

ABSTRAK

Projek ini bertujuan untuk mencirikan sampel bijh besi dari Bukit Besi, Terengganu dan Yan,Kedah. Analisis taburan saiz partikel ditentukan dengan menggunakan getaran ayakan mekanikal. Pencirian mineralogi termasuk mikroskop optik dan mikroskop imbasan elektron (SEM) dengan tenaga serakan X-ray (EDX) telah digunakan untuk menkaji morfologi bijih dan mineral liberasi. Sinar-X berpendaflour (XRF) dan pembelauan Sinar-X (XRD) telah digunakan untuk mengenal pasti komposisi unsur kimia dan fasa mineral yang terdapat dalam sampel bijih besi ini. Analisis volumetrik digunakan untuk menentukan jumlah peratus bijih besi yang terdapat di dalam sampel. Taburan saiz partikel untuk kedua-dua sample berada di dalam julat tanah berbutir kasar iaitu pasir. Unsur utama yang hadir berdasarkan hasil daripada analisa XRF adalah elemen Fe dengan 67% dan tiga elemen kecil tertinggi adalah titanium, zink dan arsenik. Berdasarkan hasil corak analisa XRD, fasa mineral dominan yang terdapat didalam sampel ialah goethite, hematit dan kuarza. Hematit, kuarza dan sulfida adalah mineral yang diperhatikan di bawah mikroskop optik dan peratusan mineral liberasi untuk sampel A adalah 70% sementara sampel B adalah 68%. Peratusan jumlah Fe yang terdapat di dalam sampel A ialah 39.45% dan sampel B ialah 40.28%. Maklumat yang dikumpulkan daripada kajian pencirian ini akan menbanu dalam memilih proses yang sesuai untuk mengestrak bijih.

PHYSICAL AND CHEMICAL CHARACTERIZATION OF IRON ORE FROM EAST COAST OF PENINSULAR MALAYSIA

ABSTRACT

This study aims to physically and chemically characterize the iron ore from Bukit Besi, Terengganu and Yan, Kedah. The particle size distribution analysis is determined by using mechanical sieve shaker. Mineralogical characterization including optical microscope and scanning electron microscope (SEM) with an energy dispersive X-ray (EDX) studies were employed to determine the ore morphology and mineral liberation. X-ray fluorescence (XRF) and X-ray diffraction (XRD) were used to identify the elemental composition and mineral phases present respectively. The volumetric analysis is used to determine the percent of Fe content in the samples. The particle size distribution for both samples are in the range of coarse grained soil which is sand. The major element present based on the XRF result is Fe with 67% and the three highest minor element present are titanium, zinc and arsenic. Based on the XRD pattern, the dominant phases present are goethite, hematite and quartz. Hematite, quartz and sulphide are minerals that observed under optical microscope and the percentage of mineral liberation for sample A is 70% while for sample B is 68%. The percent of Fe content in the sample A is 39.45% and for sample B is 40.28%. The information gathered from this characterization study will aid in the selection of suitable ore extraction process.

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Iron is one of the most abundant rock forming element in the Earth. It represents about 5% of the Earth’s crust. Iron ores are rocks and minerals from which metallic iron can be economically extracted. It usually rich in iron oxides and differ in color from dark gray, bright yellow, deep purple and rusty red. Magnetite (Fe3O4), hematite (Fe2O3), goethite (FeO(OH)), limonite, siderite and pyrite are the types of iron bearing minerals that can be obtained (Kiptarus *et al.*, 2015).

The economically processed of iron ore will produce a metallic element known as iron. Iron is used widely in this world as it has many significant usages such as manufacturing components of automobiles, machineries, tools and building structure. It main use is in the steel industry. Steel is the most useful metal known being used compare than any other metals.

Before any processing takes place, the characterization of a mineral ore is a fundamentally crucial step in analyzing the grade, shape and characteristics of the ore. All the characteristics of the mineral need to be confirmed beforehand in order to apply an appropriate economic parameters to support production planning (Kiptarus *et al.*, 2015). The information obtained play an important role in choosing the satisfactory flow sheet to recover the constituent metals (Olubambi *et al.*, 2008).

Understanding the ore deposit in terms of their mineralogical and geological occurrence will help in deciding the successful mining methods, mineral processing requirement and the most favorable route for the metal extraction process. Details of the ore mineralogy and chemical composition are important in order to choose the most efficient processing method in the metal extraction process.

1.2 Project Summary

Numerous analysis such as ore visual assessment and morphology study, particle size analysis, x-ray diffraction (XRD) technique, x-ray fluorescence (XRF) study, mineral liberation study using SEM with EDX and volumetric analysis have been accomplished in this project. The data gathered will be useful in deciding the favorable extraction method of the iron ore.

For the primary step for characterization, the samples were taken for the ore visual assessment. The sample is observed carefully using both naked eyes and hands. The details such as color, surface of the ore and hardness are obtained through this observation. Then, the ore morphology studies were done via optical images analysis and mineral liberation studies were performed using scanning electron microscope with energy dispersive x-ray analyzer (SEM with EDX).

The mineral phase identification and elemental composition analysis were accomplished using x-ray diffraction (XRD) and x-ray fluorescence (XRF) method respectively. Information of mineralogical/chemical composition, size, morphology and association with other minerals will provide facts on the characteristics, type, nature and amount of minerals and elements present within the ore which will aid in the determination of the optimal processing route for its constituent minerals/metals (Olubambi *et al.*, 2008).

Particle size analysis is carried out by using mechanical sieve shaker to determine the range of the particle size distribution. The various size fractions are sampled and used in the study of mineral liberation and optical microscopy. In addition, the total iron content in the sample is determined by the volumetric analysis.

Finally, the physical and chemical characterization study can help to decide the successful performance of metal extraction process. The result from this study will provide a relevant mineralogical information and the data will be useful for extracting of iron ore according to the industry demand.

1.3 Problem Statement

Every minerals have their own unique features and difficulties. The complete knowledge of chemical and mineralogical compositions are required in the selection of an optimum technical processing of minerals (Olubambi *et al.*, 2006). This study is performed by the fact that it is quite impossible to distinguish the elements contain in the ore just by looking at the hand specimens. Other than that, the major distribution constituents in the ore cannot be confirmed just by depending on the hand specimens only. Therefore, a proper characterization study is carried out to solve of these problems so that the ore beneficiation process can be optimized in actual plant.

1.4 Objectives

This study aims to characterize the iron ore samples from two different locations, Kedah and Terengganu. The result of this characterization will provide a relevant mineralogical information which can help in determining the suitable route of processing the iron ore. The complete knowledge of chemical and mineralogical composition is required in the selection of optimum mineral processing.

The first objective in this characterization study is to determine the particle size distribution of the samples. This is done by using mechanical sieve shaker. The next objective is to determine the elemental composition and mineral phases present within the ore by using XRF and XRD method respectively. The third objective is to study the mineral liberation and ore morphology by using SEM and optical microscope. The last objective is to determine the percentage of iron content in the samples by using volumetric analysis.

All the data gathered in this characterization study will be useful in analyzing the grade, shape and characteristics of the ore. Other than that it will help in the selection of favorable method for metal extraction.

1.5 Scope of Work

To attain the objectives of this project, several steps of analysis need to be accomplished. Below are the list of the step and analysis that need to follow:

1) Visual assessment: Observe the samples visually and the information can be drawn by naked eyes, hand and smell.
2) Crushing: Primary crushing (single toggle jaw crusher) and secondary crushing (cone crusher).
3) Sampling: Use riffle box for homogenous sampling.
4) Screening: The product discharge from the cone crusher will be screen using vibrating sieve to get a various size fractions.
5) Mineral characterization analysis: X-ray diffraction (XRD), x-ray fluorescence (XRF) and scanning electron microscope with energy dispersive x-ray analyzer (SEM with EDX)

6) Polish section: Samples preparation for optical images by mounting the samples with epoxy resin.
7) Optical microscope: Observe the images from polish section in reflected polarized images.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Almost 95% of worldwide metal production used iron and it is the mostly widely used of all metals in daily life. Its strength make it required in engineering applications, for example automobiles, structural component for buildings and construction of machinery and machine tools. However, iron need to be mixed with alloying element to produce steel as the pure iron characteristics is quite soft. So, steel cannot be produced by using iron only(Kumar, 2011).

Pure iron has insufficiency of strength and resistance to rust. However, this characteristics can be modified through alloying, thermal and mechanical processing(Kumar, 2011). Nickel and chromium are the type of alloy that are familiar with steel. Alloy is a mixture that consist two or more metallic element or metallic with non-metallic element. The mixture normally fused together through the process of molten iron.

The oxygen need to be separated from the ore so that the iron can be extracted from the ore. This reduction reaction can be done by using a suitable reactant. Carbon is the reactant that normally used in the iron making industry. It will easily bonded with the oxygen of the ore and acts as reducing agent.

The properties of the mineral is fundamentally crucial in the mineral processing. Specific gravity, conductivity and magnetism are the properties that normally take into account to achieve an efficient processing in plant. The equipment that normally used in the processing of iron are shaking table, jig, spiral, magnetic separator and froth floatation(Wills and Napier-Munn, 2005).

2.1.1 Iron ore

Iron ore deposits usually are found in the sedimentary rocks. It is one of the most abundant rock element in the Earth's crust. It ranked fourth of the most abundant element after oxygen, silicon and aluminium. Iron ores are rocks where metallic iron can be economically extracted. The rocks found usually in the form of hematite or magnetite.

Iron ores are normally rich in iron oxides and they are differ in colour such as dark grey, bright yellow, deep purple and rusty red. Normally, iron is attached with oxygen, water, sulphur or carbonate in a variation of minerals(Maranga *et al.*, 2014) . Magnetite is black in colour and consist of 72.4% Fe while hematite is red in colour and consist of 70% Fe. Limonite and carbonate siderite consist of 59.9% Fe and 48.3% Fe respectively. Both are brown in colour(Maranga *et al.*, 2014).

Iron ores develop in variety of geological environments in deposits. It occur with different sizes, shapes, origins and significantly different ore characteristics. The various nature of the geology of iron ores must be identified in exploration, estimation and in the investigate methods used in their study in order to ensure the best possible discovery and development system is followed. However, there is a common similarity of geology, problems and existence of iron ores within each ore type which entitle a rational basis for exploration, estimation and development planning.

Pure iron normally bluish grey in colour and contain a granular texture. It is hard, ductile and malleable, and also is considered as the most tenacious metals next to gold.

Similar as cobalt and nickel, it is magnetic and so readily is polarity received by iron, that a bar remaining a long time in a vertical position, or even approaching it becomes magnetic. The lower extremity usually is the northern pole. The specific gravity of iron is about 7.

Iron is a constituent of mica, which enters largely into composition of some of the oldest and most abundant primitive rock. It found in all soils and almost in every rock, thus it is considered to be the most generally diffused substances in nature. It has been met with in the nearly pure metallic state in considerable masses, said to have fallen from the atmosphere, but these masses are generally alloyed by nickel.

The ores of iron, which are a dark brown or black in colour, and in which the iron is considered to be merged with small amount of oxygen, as example the magnetic and brown iron ores belong primarily to primitive country though not exclusively. They often occur as an integral part of primitive rocks.

The red iron ores mainly belong to secondary and alluvial countries. They periodically met with in the veins of primitive mountains but are not found entering into formation of primitive rocks. This type of iron ores are normally found in the region of coal, so necessary to their reduction to the metallic state. It is either resting on the coal or filling up in the fissures in it.

Magnetic iron normally appear in iron black colour with a slight metallic lustre. It is found massive and crystallized on some variety of the octahedron which is considered to be its primitive crystal. It is suffiently magnetic to take up iron fillings and own polarity. It also nearly a pure oxide of iron. The specific gravity is about 4.5 and it is most abundantly found in primitive countries. Normally it is found commonly in beds and large masses and it accompanied by hornblende, granular limestone and garnet.

Iron ore is the main raw material from which metallic iron is separated to produce steel. China is the largest iron ore producing country in the world of iron ore production followed by Australia and Brazil(Holmes and Lu, 2015). Nowadays, steel is used widely in many industries such as construction, automotive and other manufacturing sectors(Gonzales and Kaminski, 2011).

The common method to extract the iron is by using magnetic separation. Iron is known for their magnetic properties. The magnetic separator is used to separate natural magnetic iron ore such as magnetite from less magnetite or non-magnetite materials. The magnetic separator can be classified into high intensity and low intensity separator. High intensity separator is used to separate weak magnetite material such as hematite from non-magnetite materials. Meanwhile, low intensity separator is used for strong magnetic materials(Maranga *et al.*, 2014).

Materials are divided into two classes which are diamagnetic and paramagnetic. Diamagnetic is unable to be concentrated magnetically due to the small forces involved. Diamagnetic materials tend to repel along the lines of magnetic force to appoint where the field intensity smaller while paramagnetic tend to attract along the lines of magnetic force to points of greater field intensity(Maranga *et al.*, 2014).

Steel is manufactured by process of melting the iron ore and reduced their carbon content to the desired amount. Primary steelmaking and secondary steelmaking are two basic production methods for most of world steel production. A blast furnaces (BF) with a basic oxygen furnaces (BOF), a blast furnace with an open hearth furnace (OHF), a direct reduction unit (DR) with an electric arc furnace (EAF) and a smelting reduction unit (SR) with a basic oxygen furnaces are the types of primary method to produce steel. The secondary method is by recycled the used steel and ferrous scrap in electric arc furnaces(Gonzales and Kaminski, 2011).

2.2 The Geological Occurrence of Iron Ore Deposits

Iron ores are related with most of the main geological ages are found in many geological structures. Rich ore deposit are bounded with the Pre-Cambrian rocks in Scandivania while the rich ore deposit in Canada and United States occurred around the shores of Lake Superior. The Silurian rocks of American consist bedded iron stone of the Clinton series which have a significance trade value. The argillaceous carbonates and blackband are produced by Carboniferous system. The Devonian or Old Red Sandstone strata give local beds of significance economic value(Belhaj, 2008).

Maranga *et al.*, (2014) discussed that iron ore deposits are developed by three geological processes. Firstly, direct sedimentation forming bedded sedimentary deposit which will result into banded iron formation and ironstones. Next is iron ore deposit of igneous origin. It is occurred due to the magmatic segregation of iron bearing minerals which developed as veins and tabular substitution bodies of magnetite and hematite. Thirdly is iron ore deposit that developed by surface or near surface enrichment where less resistant minerals were detached.

In addition, iron ores tend to happen in a variation of geological environments. As for the example it occurs in igneous, metamorphic or sedimentary rocks and also can appear as weathering products of various primary iron bearing materials. It can be classified into types of similar composition, geology occurrence and structure.

Heavy iron bearing minerals normally tend to separate because of gravity or to more involved geology process. Hence the final rock mass comprises areas that are richer in iron than others. Iron bearing minerals might be settled or gangue minerals might be leached out when surface water descends or when underground water ascends through rock strata. Iron bearing minerals also can be accumulated by precipitation from lakes, rivers and seas. This is mainly because of the chemical or bacteriology activity(Belhaj, 2008).

Weathering is a mechanical process that involved in the process of deposition of iron. It breaks down the rocks into small particles. Then the particles were transported away by the water and settled at other places. The iron bearing minerals that contain higher specific gravity will settled shortly compare than other lighter minerals(Belhaj, 2008).

2.2.1 Igneous ores

This type iron ore deposits are developed by crystallization from liquid rock materials. It may occur as layered type deposits because of the crystals of heavy iron bearing minerals settling as they crystallize to form iron rich concentrations. Other than that, the iron ore also may occur in the form of deposits which show obtrusive relationship with their wall rocks. These ore deposits are constitute of magnetite with varying amounts of hematite. Their shape can be either tabular or irregular. Igneous ores are normally excessive in iron content however, it also high in phosphorus and titanium content.

2.2.2 Contact ores

Magnetite and hematite with the existence of carbonates and pyrite are the types of iron deposits that occurred at or near the contact between igneous and sedimentary rocks. Their shapes are irregular or tabular replacement bodies. Normally, the deposits appeared in the sedimentary rock.

2.2.3 Hydrothermal ores

These type of deposits occurred by hot solutions which conveyed iron and replaced rocks of favorable chemical composition with iron minerals, thus developing an irregular ore bodies. Siderite or oxides are the types of iron that formed in this type of deposits.

2.2.4 Sedimentary ores

Sedimentary ores deposit can be include as bedded ores, siderite ores, placer ores or bog iron ores. Bedded iron ores are consisted of oolites of hematite, siderite, iron silicate or calcite. These ores attached with sedimentary rocks and contain fairly high phosphorus. Siderite ores composed of beds of siderite or siderite nodules attached with shales. These ores normally would contain impurities such as phosphorus and sulphides. Iron oxides will withstand to weathering and erosion when it is close packed. It may developed as placer deposit when under suitable conditions. Bog iron ores normally form in swampy areas. They appear as dark brown, cellular masses or granular or fine particles of limonite.

2.2.5 Metamorphosed iron formation

Metamorphosed bedded ferruginous rocks consist of alternate thin layers of fine grained quartz and ferric oxides. The iron will occur in the form of hematite and magnetite with the present of small amounts of iron silicates and iron carbonates. The metamorphosed types also involve the original form of the ores that unclear by immeasurable crystallization.

2.2.6 Residual ores

Residual ores are the products of the surficial weathering of rocks. However, it also may be the ore that developed by hydrothermal oxidation and leaching. These types of ores were occurred immeasurably in Precambrian iron formations which is by leaching of silica. Iron carbonates, silicate minerals and magnetite changed to hematite or limonite by oxidation process.

2.3 Classification Type of Iron

Iron ore is found in the form of chemical compound with other elements. It occurs in the form of oxides, carbonates, sulphides and silicates. Commercially, the most important iron bearing minerals in industry are hematite (Fe_2O_3) and magnetite (Fe_3O_4).

Pure hematite with a chemical formula of Fe_2O_3 contain 70% iron. It is one of the most important iron bearing minerals. The name is obtained from the Greek word for “blood” because of its appearance red in colour. Hematite normally happens in a soft, fine grained and earthy form called ochre or ruddle.

Much hematite mined is high grade with 64-68% iron content and contain only small impurities which are mainly silica and alumina(Belhaj, 2008). There are also low grade hematite which contain 20-40% iron with high amount impurities is being mined. Silica is removed through mineral processing and the product acquired contain about 60-69% iron(Potnuru, 2012).

Other common iron oxide that is found in igneous, metamorphic and sedimentary rocks is magnetite. With a chemical formula Fe_3O_4, it is a mineral with the highest iron content which is 72.4%. The percentage of iron content in high grade ore usually is above 60% with a small impurities present such as silica, alumina, phosphorus and sulphur(Belhaj, 2008).

Limonite and goethite are hydrated iron oxides. They carry about 60-63% of iron and can appear as a primary minerals. Limonite and goethite tend to happen near to the surface as the outcome of weathering of exposed ore(Potnuru, 2012).

Pure siderite comprise only 48.3% iron. It can easily deteriorated to hematite because of heat. Siderite form only small proportion of the total world iron reserves(Potnuru, 2012). However, siderite is still one of the significant source of ore in some countries.

Chamosite mineral is known for their low grade quality. Normally, chamosite happens together with limonite and siderite. This ore also contain some other impurities such as sulphur, phosphorus, quartz and calcite(Belhaj, 2008).

Normally, all types of ore will occur together with some impurities. Same goes happen to the iron ore. The type of impurities that usually present together with iron ore are silica, alumina, sulphur and phosphorus. In addition, the complex mineral chamosite can be understood as the mixture of iron oxide (FeO) and gangue mineral which are silica (SiO_2) and alumina (Al_2O_3)(Belhaj, 2008).

Table 2.1: Common iron bearing minerals and its properties.

Mineral	Composition	Iron content (% Fe)	Specific gravity	Colour
Hematite	Fe_2O_3	70	4.9-5.3	Steel grey to red
Turgite	$2Fe_2O_3.H_2O$	66.1	4.2-4.6	Brown to red
Goethite	$Fe_2O_3.H_2O$	62.9	3.4-4.2	Brown to red Brown to yellow
Limonite	$2Fe_2O_3.H_2O$	60	3.4-4.2	
Lepidocrosite	$Fe_2O_3.H_2O$	62	4.09	Brown to reddish brown
Ilmenite	$FeTiO_2$	36.8	4.5	Black to brownish black
Maghemite	Fe_2O_3	69.9	4.88	Brown
Magnetite	Fe_3O_4	72.4	5.17	Black, blue or brown black
Siderite	$FeCO_3$	48.2	3.7-3.9	Ash grey to brown
Pyrite	FeS_2	46.6	4.8-5.1	Brass yellow
Marcasite	FeS_2	46.6	4.9	Light brass yellow
Pyrrhotite	FeS_2	61.6	4.4-4.65	Bronze yellow
Chamosite	$(Mg.Fe.Al)_6(Si.Al)_4Ol_4(OH)_8$	33.42	3-3.5	Green to light yellow

2.4 Mineral Characterization

It is essential to prepare a comprehensive data on all minerals exist and their relative proportion in the ore in order to exploit the mineral deposit. A comprehensive analysis should not be carried only on economic minerals but also focus on harmful minerals as their existence may contribute to negative impact to the exploitation and mineral processing(Cook, 2000).

Moreover, the chemical composition of minerals, grain size and mineral morphology are the elements that influencing the appealing of a deposit or product produce from it. All those properties also can affect the profitability and selection of mineral processing route to be used. Reliable mineral characterization information can guide exploitation of deposit for maximum profit and optimization process for ore recovery(Cook, 2000).

2.4.1 Particle Size Analysis

Particle size is crucial as it can give impact to many other properties of particulate materials. It also acts as a good indicator of quality and performance of a process. The size and shape of the particles can affect the flow and compaction properties. As the more larger and spherical the materials is, the more easily it will flow compare than a smaller or high aspect ratio particles. In addition, the dissolve rate of small particles is more faster compare than larger particles. Thus, it will lead to higher suspension viscosity(Horiba, 2013).

Shape element will creates dissimilarity when particles are evaluated with different particle size analyzer. This is because, each of the measurement method identify the size through the use of its own physical principle. A sieve will tend to highlight the second smallest dimension due to the way of particles must align themselves to pass through the mesh opening. A sedimentometer determine the rate of fall of the particle through viscous medium. Hence, to increase drag forces while sedimenting and shifting the smaller particle size direction, the flaky and plate-like particles will place. However, the only method that can relate particle size using multiple values are microscopy or automated image analysis(Horiba, 2013).

Particle size analysis is used to characterize the range of particle size distribution in the sample. It is one of the great importance in the design of optimal process for mineral processing and metal extraction(Olubambi *et al.*, 2008). The particle size can be measured by many different methods such as optical microscopy electron microscopy, sieving, laser diffraction, elutriation and sedimentation(Wills and Napier-Munn, 2005)

The optimum size of feed to the process for maximum efficiency in the actual plant can be determined by particle size analysis. Thus, the size ranges at which any losses are occurring in the plant can be minimized(Wills and Napier-Munn, 2005). It also provides relevant information on measuring the extent of the liberation of valuable minerals from the gangue minerals at various size range(Olubambi *et al.*, 2006).

The methods of size analysis must be precise and dependable because an essential changes in plant operation may be made due to the outcome of laboratory test. It is necessary that the sample taken is a representative of the bulk material since only small amount is used in the sizing test(Wills and Napier-Munn, 2005).

One of the oldest method of size analysis is sieve analysis. A known of weight sample material is passing through finer sieves. Hence the sample retained in the each of the sieve is weighted. The data recorded is used to calculate the percentage weight in each of size fraction. Sieving can be accomplished with both wet and dry materials. The sieves are normally agitated to make sure all particles are exposed to the openings(Napier-Munn, 2005).

The success of sieving test rely on the amount of material put on the sieve and also the type of movement use on the sieve during the test. When too much charge of material use, the chances of the material to meet an aperture in most favorable position for sieving will be decreased. Therefore, the charge is restricted for the maximum amount of material retained at the end of sieving suitable to the aperture size(Wills and Napier-Munn, 2005).

Therefore, particle size analysis normally will be carried out before and after the sample being processed. As for the example in the separation step process like screening, cyclone and filtering, the particle size is determined before and after the process(Horiba, 2013). The purpose is to determine the effectiveness of the equipment by comparing the result of the sample size before and after it went through the equipment. The size analysis is crucial in the mineral processing as it helps in determining the efficiency to process of the mineral.

2.4.2 X-Ray Diffraction (XRD)

X-ray powder diffraction (XRD) is an analytical technique used to identify phase of a crystalline material. This analysis allow semi to full quantitative analysis assessment of the minerals available in the sample and in what relative proportions they happen(Cook, 2000). It also can provide information on unit cell dimension. The material analyzed is finely ground and homogenized.

This technique is acceptable for the study of material containing significant small scale variation in mineralogy such as carbonates. Other than that it also suitable for study of material present in mineral chemistry of component minerals or coarse grained sample which hinder the dependable of image analysis studies of thin sections(Cook, 2000).

XRD analysis is produced based on constructive interference of monochromatic X-rays and a crystalline sample. A cathode ray tube produced x-ray and then it is filtered to yield monochromatic radiation. Hence it is collimated to concentrate before aimed toward the sample.

The interaction of the incident rays with the sample generates constructive interference and a peak in intensity when the conditions fulfill the Bragg's Law equation ($n\lambda = 2d\sin\theta$). This law relates the wavelength of electromagnetic radiation (λ) to the diffraction angle (θ) and the lattice spacing in crystalline sample (d) and n is an integer(Scrivener *et al.*, 2004).

All the diffracted X-rays are then detected, processed and counted. The sample is scanned through a range of 2θ angles hence all possible diffraction directions of the lattice should be obtained due to the random orientation of the powdered sample. Each of crystalline material have a distinctive of XRD pattern hence enable the identification of mineral due to the transformation of the diffraction peaks to d-spacing.

Based on the Bragg's Law, the positions of the peaks are determined by the spacing of crystallographic planes. The intensity of the peaks is discovered by the position and types of atom in the crystal lattice based on the structure factor(Scrivener *et al.*, 2004).

Basically, the identification of phases is achieved by the comparison of the spacing of crystallographic planes with standard reference.

The data obtained through XRD analysis were analysed using the software of Pan Analytical Expert Highscore Plus and Pan Analytical ICSD database for the iedentification of minerals. Each mineral phase were present in unit weight percent after Rietveld refinement. Rietvield method is a full profile diffractogram fitting method that provides calculated diffractograms and strive to match to these experimentally derived ones(Macchiarola *et al.*, 2007).

The Rietveld function by computing a theoretical diffractogram for a given phase. Hence, it advances through a series of least square iterations such as background, two theta displacement, unit cell parameters and site occupancy factors. The parameters are differed until a defined convergence value is acquired(Macchiarola *et al.*, 2007).

XRD is a norm method and one of the crucial basic characterization in investigation of industrial mineral deposit and product. It is an appropriate starting point in the mineral characterization process. It also relevant to be used as addition to microscopic study of thin sections and followed up by other methods that can provide needed information(Cook, 2000).

However, there are some precaution that need to take into account before XRD analysis can be carried out. For example, before the analysis is run the X-ray source should be confirmed is monochromatic. Other than that, the broadening of the XRD machine need to be removed. Lastly, the integral width need to be applied instead of full width of the most intensive peak at the half maximum (FWHM)(Soliman, 2012).

2.4.3 X-Ray Fluorescence (XRF)

X-ray fluorescence (XRF) spectrometer is a tool that used for quantitative and qualitative elemental analyses of rocks, minerals, sediments and fluids. It is nondestructive method. This method required only minimal sample preparation and it provides rapid and explicit result. XRF analysis is used widely in many industries such as pharmaceuticals, telecommunications, cosmetics, rubber, textiles and agriculture.

Different energy is equivalent to a different colour. The energies of radiation that emitted from the sample can determine the element present within the sample. Meanwhile, how many each element present can be deduced by computing the intensities of the emitted energies(Brouwer, 2010).

XRF works based on the fact that when the atoms irradiated with x-rays, it will radiate a secondary x-rays which is known as the fluorescence radiation(Gupta, 2014). The chemistry of the sample is discovered by measuring the fluorescent x-ray released from the sample when it is excited by a primary x-ray source. The energy and wavelength of the fluorescence radiation is specific for each element while the intensity of the fluorescence radiation represent the concentration of each element(Gupta, 2014).

The precision of XRF analysis is very high. The result acquired is very reliable when good standard specimens are obtainable but the precision of the result also applied in applications where no specific standards can be found(Brouwer, 2010).

The XRF analysis can be operated to take quantification on samples that are prepared by drying and grinding. The sample normally composes of a few grams of soil held in a special cup purposed for XRF analysis. Other than that, this analysis also can be applied to take quantification on bagged samples which have experience very little preparation. XRF also can be done for in situ measurements of exposed surface in field(Web, Module and Concepts, 2008).

The XRF analysis is generally restricted to elements that have an atomic number that is greater than 16. However, it is not compulsory to detect elements with an atomic number greater than 16 at concentrations. Other than that, the XRF analysis is not effective to identify elements such as lithium, beryllium, sodium, magnesium, aluminium, silicon and phosphorus. This is because those elements are judged as light element. Still in practice, the interference effects among elements can make some elements hidden to the detector or difficult to accurately measure(Web, Module and Concepts, 2008).

XRF can be calibrated either by fundamental parameters calibration or empirical calibration. The fundamental parameters calibration is a calibration build on known detector response properties. It is called “standardless” calibration and it is the most typical to be done. The empirical calibration is a calibration computed using regression analysis and known standard. The known standard are either site specific media with known concentration or prepared spike standards(Web, Module and Concepts, 2008).

2.4.4 Mineral Liberation Studies

Most valuable minerals are associated with the gangue minerals initially. Hence, before separation process can be managed, they must be unlocked or liberated so that they are partially or fully exposed(Wills and Napier-Munn, 2005). The particle size is reduced progressively either by crushing or grinding. This process is called comminution. The liberation of valuable minerals is very crucial in the mineral processing in order to achieve high recoveries from downstream separation process such as gravity separation and froth flotation.

Liberation analysis is very useful tool in determining the mineralogy of the rock. The detail provides cannot be acquired by other analytical techniques such as XRF and XRD(Ismail *et al.*, 2016). Mineral liberation studies is assessed with the help of Scanning Electron Microscopy with Energy Dispersive X-rays (SEM/EDX). High resolution images of the sample can be provided by SEM. It occurs when rastering a focused electron beam across and detecting backscattered electron signal. Meanwhile, EDX is used to provide elemental identification and quantitative compositional information.

SEM provides information of identification individual minerals. The information gained through investigation of sample of a polished thin section prepared from a rock sample or a sample mount prepared from concentrate or other processing product. Other than that, SEM also serves an optical image that can be processed and treated by image analysis method. The information obtained are characterization of size, morphology, habit and association(Cook, 2000).

Back scattered electron imaging increase the chances to search for minor or trace phases of interest. Other than that it also can be used to disclose morphological characteristics and mineral fabrics in more detail compare than conventional optical microscopy. It is an essential tool for the study of many types of mineral deposits(Cook, 2000).

The mineral liberation features are divided into several different categories. Firstly is the mineral locking. It is based on how much weight % of a given phase occur with other phases in binary or tertiary constitute particles. Next is the mineral liberation by particle composition. Basically it indicates how many weight% of a given phase occurs in a given liberation class. For example, there are particles of liberation class 95-100% which are composed of 95% weight of copper sulfide ore and 5% weight of gangue minerals.

The third is the mineral liberation by free surface list and graph. It shows how many particles with free surface of a given phase occur in a given liberation class. As for example, the copper sulfide ore occurs in particles with a completely free surface so the liberation class is 100. Meanwhile, a liberation class 95-100 means a surface locked 5% by a gangue phase and liberation class 0 indicates the surface is completely locked. The share in weight % of the particles belonging to this liberation class is plotted in cumulative way(Gutzmer and Germany, 2013).

The cost to process the minerals that have small grain size is much higher and consume more energy. The fine particle size will increase the chances of material losses while processing. Hence the result from grinding the ore is a settlement between high grade concentrates, operating cost and losses of minerals(Napier-Munn, 2005).

2.4.5 Ore Visual Assessment and Morphology Study

Identification of the mineralogy of iron ores particles can be performed by several techniques such as indirect or direct measurements. The indirect technique normally conclude that the minerals in the analysed ore are stoichiometric and that not always the case. Other than that, diagnostic leach experiments and calculation of mineralogy from assays also can be included in this indirect methods(Donskoi *et al.*, 2007).

Next, the direct methods are included the quantitative X-Ray Diffraction (XRD), optical image analysis and automated image analysis via scanning electron microscopy. Optical image analysis are able to determine the porosity in variety of minerals and