

**REDUCTION OF MALAYSIAN IRON ORE PELLET BY
USING COAL AS REDUCTANT**

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**REDUCTION OF MALAYSIAN IRON ORE PELLET BY USING COAL AS
REDUCTANT**

by

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- Appendix G : Microstructure of Unreduced Pellet
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LIST OF ABBREVIATION

XRF	X-ray Fluorescence
EDX	Energy Dispersive X-ray spectroscopy
SEM	Scanning Electron Microscopy
DRI	Direct Reduce Iron
XRD	X-ray Diffraction
ASTM	American Standard Testing Method
ICCD	International Center Diffraction Data
AAS	Atomic Absorption Spectroscopy

LIST OF SYMBOLS

μm	Micrometer
wt%	Weight percentage
K	Kelvin
g	Gram
ΔG	Gibbs free Energy
T	Temperature
R^2	R Square
K	Thermal Conductivity
κ	Thermal Diffusivity
ρ	Density
C_p	Specific Heat
ΔH	Delta H (Enthalpy)
ΔS	Delta S (Entropy)
kJ	Kilo Joule
X	Extent of Reduction
$^{\circ}\text{C}$	Degree Celsius
h	Hour
s	Standard deviation
ρ_t	True density or powder density

LIST OF PUBLICATIONS

Hanizam Shah Saidin, S. A. Rezan, H. Hussin, (2012). Characterization and Carbothermal Reduction of Malaysian Iron Ore, ISIJ International. (*In preparation*)

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PENURUNAN PELET BIJIH BESI MALAYSIA MENGGUNAKAN ARANG BATU SEBAGAI PENURUN

ABSTRAK

Sampel bijih besi bergred rendah yang diperolehi dari Kuala Lipis, Pahang, Malaysia telah digunakan dalam keseluruhan kajian ini. Pada masa ini, piawaian pada proses karbotermal yang digunakan oleh pengamal industri tempatan adalah berdasarkan piawaian antarabangsa oleh penyelidik luar yang menggunakan bijih besi bukan dari Malaysia. Oleh itu, pengoptimuman beberapa parameter penting dalam proses karbotermal bijih besi Malaysia perlu dikaji. Sehubungan dengan itu, antara pembolehubah yang penting termasuklah kesan suhu, nisbah molar karbon-besi oksida, masa pengurangan dan jumlah fluks dan pengikat terhadap pengurangan oksida besi di bawah keadaan bukan isotherma dan isoterma. Untuk tujuan pengoptimuman, reka bentuk eksperimen telah digunakan dengan mengaplikasikan reka bentuk faktorial dan kaedah gerak balas permukaan. Sampel bijih besi tersebut telah dicirikan dengan beberapa kaedah termasuk mikroskopi optik, XRF, SEM dan XRD. Analisis XRF menunjukkan bahawa sampel bijih besi mengandungi 81.2% berat Fe_2O_3 , 14.7% berat SiO_2 , dan 3.5% berat Al_2O_3 . Corak XRD mengesahkan hematit dan kuarza adalah fasa utama yang hadir dalam sampel. Kajian menunjukkan bahawa kekuatan mampatan pelet bijih besi terpengang menurun dengan peningkatan kebesan pelet. Pelet terpengang dengan fluks kalsium karbonat memberikan kekuatan mampatan tertinggi (7251 N/P), diikuti oleh barium karbonat (1410 N/P) dan magnesium karbonat (39 N/P). Selain itu, pada suhu bilik, nilai konduktiviti haba untuk pellet dengan fluks magnesium karbonat memberikan nilai tertinggi (2.77 WmK^{-1}) diikuti oleh pellet dengan kalsium karbonat (2.48 WmK^{-1}) dan barium karbonat (2.25 WmK^{-1}). Bawah keadaan bukan isoterma, fasa sampel bijih besi telah dianalisis. Mekanisme pengurangan adalah daripada $\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe}$. Bawah keadaan bukan isoterma, fasa sampel bijih besi telah dianalisis. Mekanisme pengurangan adalah daripada $\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe}$. Daripada analisis rekabentuk eksperimen, suhu merupakan parameter yang paling berpengaruh yang menunjukkan interaksi kuat dengan parameter operasi (penambahan fluks dan masa). Keputusan analisis tersebut menunjukkan bahawa takat penurunan mencapai sehingga 99.9% pada suhu 1373 K dengan fluks sebanyak 5.0% berat. Bawah keadaan isoterma, keputusan optimum bagi nisbah mol karbon-besi oksida ($\text{C}/\text{Fe}_2\text{O}_3$) adalah pada 1:4.5. Analisis penurunan menunjukkan bahawa sampel telah mengambil masa 60 minit untuk mencapai 92%. Selain itu, jumlah optimum fluks untuk mendapatkan penurunan setinggi 98.9% ialah 5.0% berat.

REDUCTION OF MALAYSIAN IRON ORE PELLET BY USING COAL AS REDUCTANT

ABSTRACT

Low grade iron ore sample obtained from Kuala Lipis, Pahang, Malaysia have been used throughout this study. Currently, the standard on carbothermal process that has been used by practitioners of the local industry is based on the international standard by external researchers using iron ore not from Malaysia. Therefore, the optimization of some significant parameters of Malaysian iron ore carbothermal process needs to be studied. With this regards, among the important variables including effects of temperature, carbon-iron oxide molar ratio, reduction time and amount of flux and binder on the reduction of iron oxide under nonisothermal and isothermal condition. For optimizations purposes, design of experiments (DOE) was utilized by applying the factorial design and response surface methodology. The iron ore sample was characterized by several methods including optical microscopy, XRF, SEM and XRD. XRF analysis indicated that the iron ore sample contained 81.2 wt. % of Fe_2O_3 , 14.7 wt. % SiO_2 and 3.5 wt. % Al_2O_3 . XRD pattern confirmed that hematite and quartz were main phases present in the sample. The investigation showed that the compressive strength of roasted iron ore pellets decreased with the increasing of pellet basicity. Roasted pellets with calcium carbonate flux gave the highest compressive strength (7251 N/P), followed by barium carbonate (1410 N/P) and magnesium carbonate (39 N/P). Besides that, at room temperature, the thermal conductivity value for the pellet with magnesium carbonate flux gave the highest value (2.77 WmK^{-1}) followed by pellet with calcium carbonate (2.48 WmK^{-1}) and barium carbonate (2.25 WmK^{-1}). Under nonisothermal conditions, the phase development of direct reduced iron ore samples has been analyzed. The mechanism of reduction was from $\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe}$. From DOE analysis, temperature was the most influential parameter that showed strong interaction with the operating parameters (flux addition and time). The results of the analysis showed that the extent of reduction reached up to 99.9% at 1373 K with 5.0 wt.% flux. Under isothermal conditions, optimum results for carbon-iron oxide molar ratio ($\text{C}/\text{Fe}_2\text{O}_3$) were at 1:4.5. The analysis of reduction time showed that the sample took 60 minutes to achieve 92% reduction. Besides that, the optimum amount of flux to obtain a high reduction of 98.9% was 5.0 wt.%.

CHAPTER 1 INTRODUCTION

1.1 Introduction

Malaysia has significant reserves of iron ore deposits that are still economical for mining and processing. Malaysian iron ores are produced as a small-scale mines located in several areas around peninsular Malaysia. Among the well-known is in Bukit Ibam and Kuala Lipis in Pahang, Pagoh and Kota Tinggi in Johor, Ulu Paka and Merchang in Terengganu, Bukit Besi and Gua Musang in Kelantan and Sungai Siput in Perak. Most mining and mineral processing businesses incorporated in Malaysia are privately owned or a joint venture with State-owned Company (Tse, 2009). Malaysian iron ore output in 2007 increased by 20 per cent to 802,030 tonnes from 667,082 tonnes in 2006. The ores were consumed mainly by the local cement, and iron and steel plants (Anon, 2009). The low-grade iron ores are consumed by the pipe-coating industry that supplies the oil, gas and cement plants. Malaysia imports high-grade iron ore in descending order of tonnage from Brazil, Chile, Mexico, (Tse, 2009) and Bahrain (Taib, 2010).

1.2 Geological Aspects of Iron Ore

Iron is the most abundant element in the earth with a concentration of 37 weight percent (wt.%). In the Earth crust, iron is one of the top four elements with 4.6 wt.% concentration, after oxygen, silicon and aluminum. It can be found in the in meteoritic masses and generally associated with nickel and cobalt. Iron deposits can also be found in

the remains of eruptive rocks that formed from a volcanic eruption. Most of the iron in the crust is found combined with oxygen as iron oxide minerals. These minerals include hematite (Fe_2O_3) and magnetite (Fe_3O_4), hydrated oxide such as goethite ($\text{FeO}(\text{OH})$) and limonite ($\text{FeO}(\text{OH}) \cdot n\text{H}_2\text{O}$). Besides that, it can also occur as a carbonate such as siderite (FeCO_3), sulphide (FeS_2) and as a component of a wide range of complex minerals. Iron atoms gives the red coloring in nature and it is found in the remains of most plants and animals (Hussain, 1985; Marsden, 1990).

Iron ores are found in many geological systems, associated with most of the main geological ages. In Scandinavia, rich ore deposits are associated with the Pre-Cambrian rocks. The Silurian rocks of America contain bedded iron stone of the Clinton series, which are of great economic value. These iron ore deposits are found in the middle Silurian Clinton Formation deposits and equivalents that extend along the eastern side of the Appalachian Basin from New York to Alabama. This carboniferous system provided black-band and argillaceous carbonates (Ball *et al.*, 1973).

1.3 Iron Ore in a Global Perspective

Essentially, all of the iron ore produced globally is used in the manufacturing of steel. Based on statistics compiled by Jorgensen (2010), global iron ore production was expected at 2,400 million tonnes, with China leading the industry as both the world's largest producer and consumer. The Chinese iron ore industry has grown at an annual rate in excess of 20 % over the last several years versus the global average (excluding China)

of 7 %. This in turn is driven by the country's burgeoning steel industry and rapid industrialization.

As the world's largest producer of crude steel, China, with its limited domestic supply of iron ore, is also the world's largest importer. According to the U.S. Geological Survey (USGS), China produced 880 million tonnes of iron ore in 2009. According to USGS (2010), it also imported another 628 million tonnes to meet its domestic steel manufacturing demand. The next largest importer is the European Union at 154 million tonnes, followed by Japan with 140 million tonnes and South Korea with 50 million tonnes (Jorgenson, 2009).

1.4 Steel Industries in Malaysia

The homogeneity of the product and the lack of brand name make the demand for local steel very elastic. The largest buyer of steel was lead by the construction sector. Because the steel industry was viewed as an important element for national development, the government had imposed export control tariffs and price ceilings to ensure a stable supply of steel for the construction sector. The price of steel increased by 55 % to about RM 4,000 per ton after the government lifted the ceiling price in May 2008. This was in line with world prices that exceeded USD 1,000 per ton in mid-2008 (Poon and Yap, 2010).

The steel industries are comprised of companies that manufactured upstream products such as direct reduced iron (DRI) and billets. The midstream products made are

bars and rods. It was dominated by a few public-listed companies such as Ann Joo Steel Berhad, Kinsteel Berhad, Malaysia Steel Works (KL) Berhad (MASTEEL), Perwaja Holdings Berhad, Southern Steel Berhad and Lion Group. Among the companies listed, Lion Group owns three major steelmaking companies locally which are Amsteel Mills Sdn. Bhd., Antara Steel Mills Sdn. Bhd. and Megasteel Sdn. Bhd. Some industry observers believed that the steel producers should integrate upstream and downstream activities like manufacturing pipes, wires and so forth to be more competitive. In 2008, manufacturers of basic iron and steel products such as iron and steel bars, rods, wires, pipes and drums generated sales of about RM28 billion (Poon and Yap, 2010).

Moreover, the support of State government also has boosted the development of iron ore activities in the country. For example, in 2008, Vale S.A. of Brazil planned to establish a distribution hub for iron ore fines and a pelletizing plant at Teluk Rubiah, Lumut, in the State of Perak. The Vale S.A is the leading iron ore supplier in the world. Anon (2010b) estimates the \$900 million project from Vale S.A includes the construction of a terminal with an initial capacity to stockpile 30 Mt/yr of iron ore. It could be expanded to 90 Mt/yr in the future. The company aims to sell the iron ore to markets in Asia and the Middle East with the closest competitor being Rio Tinto PLC and BHP Billiton Ltd. The terminal was scheduled to be completed by the first half of 2013.

Apart from that, Ann Joo Steel Bhd. (a subsidiary of Ann Joo Group) renovated its electric arc furnace to increase its steelmaking output capacity, thus has made the country's shipment at 900,000 tonnes in 2008 (Anon, 2010a). In 2007, Ann Joo had

awarded a contract to China's Tangshan Iron and Steel Design and Research Institute to design and build a 450 cubic meter blast furnace and a 75 square-meter sinter plant. The blast furnace will have a pig iron output capacity of 500,000 t/yr, was scheduled to be put into operation by the end of 2010. Ann Joo also planned to expand the pig iron output capacity to 1.1 Mt/yr through the addition of another blast furnace (Tse, 2009).

1.5 Problems Statement

Iron ores are found in various geological systems associated with most of the main geological ages period (Ball *et al.*, 1973). Therefore, there are some disparities in their mineral composition respective to the origin that become as crucial factors for grade determination and mineral processing. Thus, this matter has drawn the interest of many researchers around the world. Although, there are numerous research papers published about iron ore processing and reduction process, but the information about parameter related to carbothermal reduction process on Malaysian iron ore such as optimum temperature, carbon-iron oxide mole ratio and reduction time is still not fully understand.

Tse (2009) stated that, the standard on carbothermal process that has been used by practitioners of the local industry is based on the international standard by external researchers using iron ore not from Malaysia. Therefore, the optimization parameters of Malaysian iron ore carbothermal process need to be studied. With this regards, the study on the effect of variables such as temperature, carbon-iron oxide mole ratio, reduction time and amount of flux and binder on the reduction of iron oxide under nonisothermal

and isothermal condition can provide hindsight to conversion of Malaysian iron ore to metallic iron.

1.6 Objectives of Studies

The main objectives of this work are:

- i. To characterize iron ore sample from Kuala Lipis and to study the properties its roasted iron ore pellet with fluxes.
- ii. To apply the design of experiments (DOE) for optimization of experimental factors.
- iii. To optimize the experimental factors such as temperature, carbon-iron oxide molar ratio, progress time and amount of flux and binder to the extent of reduction.

CHAPTER 2 LITERATURE REVIEW

2.1 Introduction

There are a lot of research and investigation conducted on iron ore in the literature (Prakash, 1996; Kumar and Patel, 2009). The interest today is their characterization and properties in relation to the reduction process. This is carried out due to the uniqueness of the iron ore from different mineral deposits throughout the world.

Generally, Malaysian iron ores have been characterized as having high iron content with relatively high amount of alumina and silica. The alumina can be as high as 4-5 wt. % and silica about 14-15 wt.% respectively (Hatem, 2009). Ghosh *et al.* (2010) noted that the alumina to silica ratios that are greater than one ($\text{Al}_2\text{O}_3/\text{SiO}_2 > 1$) in these ores could cause serious operational problems during sintering and subsequent reduction in the blast furnace. For example, the high alumina to silica ratio resulted in the slag production with high viscosity and low sulphide capacity. Earlier efforts made to reduce alumina in the ore primarily focused on flocculation techniques that met with limited success (Gujraj *et al.*, 1983; Mahiuddin *et al.*, 1989). In addition, Pradip (1994) has concluded that the multi-gravity separation is the most promising technique for treating the iron ore slime and it is particularly effective for reducing alumina and silica.

The behavior of iron ores fines in downstream beneficiation operations or in subsequent processing operations needs to be determined. Among the properties, which

are important, are agglomeration and sintering which can reliably predict the processes mentioned above (Clout, 1998; Donskoi *et al.*, 2007). Generally, the information incorporated consists of mineralogical study, physical and chemical analysis. Based on Panigraphy *et al.* (1990) reviews, he stated that various factors commonly affect the pellet's quality such as nature of ore, gangue minerals, type and amount of fluxes added. These factors will play an important role in successive steps of treatment to produce the pellets. The above factors will have direct impact in variation of physicochemical properties of the phases and their distribution in the pellet making process.

In the pelletizing industry, the use of additives containing fluxes is gradually becoming a subject of interest due to cost reduction of producing DRI. Li *et al.* (2009) in their works claimed that increasing the weight percentage of flux content of the pellets is regarded as a method to improve pellet quality. Another method is by roasting the iron ore with the flux to make a roasted pellet. Roasting is a simple process that has a significant role in ironmaking industry. The roasting mechanism of individual hematite pellets were characterized by the growth and recrystallization of Fe_2O_3 grain at high temperature (Li *et al.*, 2009). Apart from that, Fan *et al.* (2010b) have suggested that the routes to making efficient fluxes addition rely on how roasting parameters vary with fluxes added and the subsequent quality of pellets produced. The quality of the pellet here is generally defined as to have contribution for high extent of reduction, porosity and strength. In addition, experimental data in the literature have established that limestone, dolomite and magnesia could be used to make a basic fluxed pellet that has a superior metallurgical properties relative to conventional acid pellets (Friel and Erickson, 1980).

Thus, it is important to study the influence of fluxes on the roasting process with different basicity value. The change in basicity of the samples with respect to mechanical and thermal properties of the pellets are not well established in the literature. This problem is compounded by the use of iron ore minerals with impurities that vary from one deposit to another.

The thermal properties of the iron ore will determine the amount of energy required for reduction and sintering. For the reduction part, it will influence the reaction kinetics in the reduction process (Takegoshi *et al.*, 1984). Furthermore, McCann *et al.* (2004) agreed that investigating these thermal properties is thus extremely important with respect to controlling the iron making processes. In addition, Akiyama *et al.* (1992) have suggested that accurate thermal conductivity values of iron ore at elevated temperatures are strongly related to heat transfer. Thermal conductivity of iron ore is known to be dependent upon temperature, pore, combined water and gangue minerals.

The reduction reactions of iron ore to iron have been a subject of research and discussion for many years, but it are still beset with uncertainties, with inconsistent experimental data and with conflicting theories. Although a number of investigations have been carried out on the several of gaseous and solid reduction of iron ores, uncertainties arise because of the complex nature of these oxides with impurities. Prakash (1996) in his review has stated that the complexities in the case of iron ores come from uncertainties in their chemical composition due to impurities, flux additions, other additives and binders. During reduction process, the existent of impurities or additives

such as binder, fluxes and catalyst can modify the crystalline structure, lattice diffusivity, porosity, surface morphology and even the reduction sequence of the iron oxides (Srb and Ruzickova, 1988). Besides the temperature, the chemical composition and purity of the reducing gases and the type of reductant are important in determining the carbothermal reduction parameters.

2.2 Nature of Iron Ores

Iron ores are rocks and minerals from which metallic iron can be extracted after suitable hydrometallurgical or pyrometallurgical operations are applied. It is found in varying amount in ore deposits where it occurs chiefly as the oxides, Fe_2O_3 (hematite) and Fe_3O_4 (magnetite), the FeS_2 (iron sulfides), FeCO_3 (iron carbonates), silicates and as many other minerals of lesser significance (Hussain, 1985; Marsden, 1990). Ball *et al.* (1973) have concluded that an iron-bearing mineral can only be considered as an iron ore if the total cost of extracting iron from it is comparable with the cost of extracting iron from other ores. This will be governed by several factors of which the iron content of the mineral, the nature of the impurities and the location of the deposit area of particular importance which can make the overall operation is worth and economic. Apart from that, Kumar and Patel (2009) have stated that iron ore is typically classified as high grade if contains more than 65% iron, medium grade within 62 – 65% iron and low grade if less than 62% iron. The most important and economic iron ores are hematite, magnetite, siderite (FeCO_3) and hydrated iron oxides such as limonite ($\text{FeO}(\text{OH}) \cdot n\text{H}_2\text{O}$) and goethite ($\text{Fe}^{3+}\text{O}(\text{OH})$) (Ball *et al.*, 1973).

Hematite is widely distributed and is the most important source of iron. The chemical formula of hematite is Fe_2O_3 . When pure, it contains 70 weight (wt.) % of iron. Much of the hematite mined is high grade, with 64-68 wt.% of iron. The major impurities that occur in small quantities are silica and alumina. Sulphur and phosphorus contents if any are normally very low. There are also very large deposits of low grade hematite containing only 20-40 wt.% iron with high silica contents. Much of the silica is removed by mineral beneficiation and the product obtained can contain 60-69 wt.% of iron (Ball *et al.*, 1973; Hussain, 1985; Podolosky and Keller, 1994).

El Habaak (2004) has stated in his reviews that deposits of high grade magnetite occur in a number of places in the world. Pure magnetite contains 72.4 wt.% iron, whereas the high grade ore normally contains more than 60 wt.% iron with some impurities such as silica, phosphorus and apatite as the common impurities. Low grade magnetite deposits are also mined in many places, and a product is obtained after mineral processing which has an iron content in excess of 60 wt.% (Ball *et al.*, 1973; Hussain, 1985; Podolosky and Keller, 1994).

2.3 Iron Ore Processing

Generally, iron ore loaded at the mine site need special characteristics for ironmaking. Among them are the ore should be in the form of lumps that have a size up to 1.0-1.25 m. This rule of thumb must also be applied to high grade ores and it is necessary for it to be reduced in size and graded before ironmaking. At the mine site, the

ore is crushed to a maximum size in the region 100-200 mm. After crushing and screening (comminution processes) to an appropriate size, the ore is transported out by an export terminal. On receipt at the import terminal, or at the ironmaking plant, the fine ores may be treated to another shape such as a pellet or sphere. The product shipped to the ironmaking plant should have a uniform composition as high as possible. In order to minimize variation in composition, the ores may be blended with different grades. With low grade iron ores, the content is graded by gangue removal. This removal process requires grinding to a specific liberation size so that the iron-bearing mineral and the gangue are separated.

Before the invention of the modern blast furnace, most of the world's iron ore was simply mined, crushed, and then shipped. Today's blast furnaces now demand ores with specific size and chemical specification that make crushing and screening essential steps. Many blast furnaces specify ores with high purity with fixed size. Most blast furnaces specify the maximum size of the ores at about 38.1 mm with the minimum of 9.5 mm (de Beer *et al.*, 1998). These fines may be pelletized at the mine, or shipped to the furnace for sintering. A low grade ore needs to be crushed to the liberation size which makes it difficult to separate the gangue minerals. Olubambi and Potgieter (2005) have suggested that among the methods used to separate iron ores from gangue minerals are gravity method, magnetic separator, reduction roasting followed by magnetic separation, floatation and electrostatic separation. In addition to these methods, some degree of concentration can be achieved by washing (Devaney, 1985) as a pretreatment stage.

Besides that, the concentration of valuable minerals from the gangue involves comminution and separation based on density (Olubambi and Potgieter, 2005).

2.4 Characterization of Iron Ore

In general, iron ore has high silica content that typically generates more fines (-10 mm size) during comminution process. These fines being relatively of lower grade cannot be used directly in a blast furnace. It has been reported in the literature that even 100 mesh fraction in the sinter feed can be accepted up to 40 wt.%. This is achieved by micro-balling the sinter mix prior to sintering (Srivastava *et al.*, 2001). For this reason, it has become increasingly attractive to upgrade the fine ores using appropriate beneficiation techniques (Upadhyay *et al.*, 2009).

The behaviour of fine iron ores in downstream beneficiation or in subsequent processing operations such as granulation and sintering are very important. Clout (1998) and Donskoi *et al.* (2007) claimed that these properties allow reliable prediction about the ore fines behavior during sintering and ironmaking. Among the properties needed are particle mineralogy, porosity, mineral associations, texture, hardness, size distribution, mineral liberation, class densities and mineral composition. Combinations of different techniques such as sizing, pycnometry density, chemical analysis, X-ray Diffraction (XRD), microscopy and image analysis can provide these informations.

The mineralogy of iron ore particles can be understood by several methods such as indirect or direct measurements. Donskoi *et al.* (2007) stated that among the direct techniques are quantitative XRD, optical image analysis and automated image analysis using scanning electron microscopy (SEM) techniques. The advantage of using an electron microscope is the chemistry of the minerals can be simultaneously analyzed with information about mineral liberation by the availability of EDX technology. Mineral liberation can be defined as the textural relationships between minerals within an ore, and their relation to process selection requires the introduction of the concept of liberation size.

Challenges in processing the iron ores primarily arise from the compositional characteristic of the ore. The composition of the Malaysian iron ores is typified by high iron content with relatively higher amounts of alumina and silica as high as 4-5 wt.% and 14-15 wt.% respectively. The alumina to silica ratio that is typically greater than one in these ores can cause serious operational problems during sintering and subsequent smelting in blast furnace. Gujraj *et al.* (1983) and Mahiuddin *et al.* (1989) stated that an earlier effort has been made to reduce alumina in the ore primarily focused on flocculation techniques that met with limited success. The multi-gravity separation is the most promising technique for treating the iron ore slime and it is particularly effective for reducing silica and alumina (Pradip, 1994). Hatem (2009) found that low grade iron ore can be concentrated by gravity method to produce a concentrate assaying 68.6 wt.% Fe and 1.20 wt.% Al_2O_3 .

2.5 Agglomeration of Iron Ore

For some operations in ironmaking processes, the iron ore fines concentrate are not suitable as a feed and the fine particles must be agglomerated. One such method for agglomeration is briquetting or mechanically compressing the powder into small briquettes. Another method that is widely used is pelletizing in which the finely divided material is rolled in a drum or on an inclined disk. By doing this, the particles cling together and rolls up into small spherical pellets with constant radius. Material discharged from the pelletizer is screened and the pellets that are too small are re-circulated back for recycling. For either pelletizing or briquetting, some binder normally added and mixed with the fines and the appropriate moisture content must be controlled. This is to ensure the pellets or briquettes will have enough green strength to be handled. Usually the green body is given a subsequent heating about 378 K, which dries them out (Forsmo, 2007). As a result of this, the iron ore develops much greater bonding forces within the pellet or briquette.

Interest in iron ore-coal composite pellets has grown from the 1980s and was said to have a lot of advantages (Dutta and Ghosh, 1994; Agrawal *et al.*, 2000; Sah and Dutta, 2011). A principal advantage of the composite pellet route is its ability to utilize fines directly. This utilization can be carried out without high temperature burden preparations such as sintering, hardening, and coke making. The carbonaceous material is non-coking coal that acts as a reducing agent. As stated by Ghosh (1999), by pelletizing, the reduction kinetics can be improved as the carbonaceous reductant is in intimate contact

with iron ore fines. The close contact allows shorter path for the reducing gas and reactions to occur and consequently improves productivity.

2.6 Binders

Binders are used in agglomeration operations such as pelletization to ensure that the agglomerate possesses desired mechanical properties. These properties are required for subsequent handling, transportation and processing. These properties also depend on the requirements of a particular operation and installation. Binders are added prior or during agglomeration to increase the strength of the agglomerated product.

The binder accomplishes two very essential tasks in iron ore pelletization. Firstly, it makes the moist ore behave plastically, so that it will nucleate iron ore seeds that grow at a controlled rate into well-formed pellets (forces due to binding medium). Secondly, during drying, the binder holds the particles in the agglomeration together while the water is removed. As the water is removed, the binder holds them together until the pellet is heated sufficiently to sinter into the grain together (forces resulting from pelletizing process) (Eisele and Kawatra, 2003).

In the development of the iron ore pelletization process, hundreds of binders have been investigated. Pietsch (2002) classified the binders into two groups, organic and inorganic components. A wide variety of binder choices are available including inorganic, organic, and polymer binders. The binder must be inexpensive and useful at low dosages to be economical.

2.7 Flux

In the 1990s, almost all pellets produced in the world had fluxes addition such as limestone (calcite) and dolomite. It was concluded that limestone, dolomite, magnesia and so on could be used to make a fluxed pellet that had superior metallurgical properties relative to conventional acid pellet (Friel and Erickson, 1980; Takeshi *et al.*, 1983). Beside the fluxes has been used for reducing the reduction temperature, it also are used in EAF steelmaking for refining, refractory protection and aiding formability of the slag (Fruehan *et al.*, 2003). At a small number of mineral deposits, „self-fluxing“ of the magnetite concentrate occurs due to the presence of dolomite and calcite in the ore body, however this is rare (Firth and Garden, 2008). Carbonate fluxes generate CO₂ as they calcine and decompose, around 1073 K. The calcination reaction is thermally activated, so it occurs simultaneously across the pellet.

During the processing of pellets in ironmaking process, the physical, metallurgical and microstructural properties of the flux have a major influence on the reduction behavior. In general, the main types of pellets produce for iron making process are acidic, basic and fluxed pellets. The term of basicity is referred to the ratio of the percent content of the alkaline oxides and the alkaline-earth oxides to the other nonferrous oxides. Chatterjee (2010) suggested that to produce a good quality pellets, additives are important. In pelletization process, limestone is employed to modify the pellet basicity. It can be simplified in the formula of basicity, B₁ or B₂ as showed in Eq. 2.1 and 2.2.

$$B_1 = \frac{\% \text{ CaO}}{\% \text{ SiO}_2} \quad (\text{Eq. 2.1})$$

$$B_2 = \frac{\% \text{ CaO} + \% \text{ MgO}}{\% \text{ SiO}_2 + \text{Al}_2\text{O}_3} \quad (\text{Eq. 2.2})$$

In recent years blast furnace iron making system prefers fluxed pellets as iron bearing feed because, fluxed pellets gives precise quality requirements (Umadewi *et al.*, 2011). The quality of the pellet is affected by the ore chemical composition, material mix proportion, gangue content, and flux proportion. In addition, the properties of the pellets are largely influenced by the type and degree of bonding achieved between ore particles and fluxes. These factors results in variation in the physicochemical properties, structure, homogeneity of the phases (Panigraphy *et al.*, 1990). Apart from that, lime makes the pellet self-fluxing and aids as a slag former. Added to this, limestone is cheap and readily available (Sah and Dutta, 2010).

2.8 Properties of Pellets in Direct Reduction Process

Some characteristic of the pellets for direct reduction (DR) processes are the use of indurate pellets which is made from high grade concentrates. Pellets for DR are subjected to different conditions compared with blast furnace pellets. Both types of pellets must have sufficient physical strength. However, the pellets for direct reduction must also have a low gangue content, high reducibility, and minimum tendency to disintegrate or stick together during reduction. Low gangue content is essential, especially for the silica content, since high silica content adversely affects the cost of

melting the reduced pellets in electric arc furnaces during steelmaking. For example, the power consumption will go up as silica content increases.

High reducibility is important because the reduction process takes place via gas-solid reactions and at lower temperature than in a blast furnace. Similarly, disintegration of the pellets caused by thermal expansion that takes place when hematite is reduced to magnetite and wuestite has to be minimized. This is to ensure the stack permeability in gas-based and coal-based DR processes. In the wuestite phase of reduction, pellets have very little strength and the impurities and additives are important for holding the pellets together (Forsmo, 2007). In general, the main types of pellets for iron making units are acid, basic and fluxed pellets. Acid pellets are made from ore concentrates in which silica, an acidic oxide, is the major impurity. However, few iron ore deposits lend themselves to low-silica pellets. The more common type of pellets is lime-bonded/dolomite-bonded pellets where lime/dolomite is added to the concentrate for fluxing the silica prior to pelletizing.

2.9 Coal

Coal is a carbonaceous material that was formed through the physical and chemical alteration of peat by processes involving bacterial decay, compaction, heat and time. It is an agglomeration of many different complex hydrocarbon compounds (Francis, 1961). Naturally, coal contains a wide variety of organic and mineral phases in a complex and porous structure. Variation in contents from one coal deposit to another

and from one location to another within the same seam is common (Schroeder *et al.*, 2004). Based on Francis (1961), coal is classified into three major types namely anthracite, bituminous, and lignite. Coal is also further classified as semi-anthracite, semi-bituminous, and sub-bituminous since it is difficult to differentiate among the previous 3 classifications. Anthracite is the oldest coal from geological perspective. It is a hard coal composed mainly of carbon with little volatile content and practically no moisture. Lignite is the youngest coal from geological perspective. It is a soft coal composed mainly of volatile matter and moisture content with low fixed carbon. Fixed carbon refers to carbon in its free state, not combined with other elements. For example, carbon can combine with titanium to form titanium carbide. Volatile matter refers to those combustible constituents of coal that vaporize when coal is heated (Speight, 2005).

Coal is an extremely complex material and exhibits a wide range of physical and chemical properties. Francis (1961) in his report has stated that by analyzing the chemical elements of coal samples, information on the quality of the carbon in the coal can be established. Speight (2005) has stated that there are some of the test methods applied to coal analysis that are empirical in nature, and strict adherence to the procedural guidelines is required to obtain repeatable and reproducible results. In general, the types of analysis requested by the coal industry are classified as proximate analysis and ultimate analysis.

2.9.1 Analysis of Coal

There are two methods to analyze coal that are called as ultimate and proximate analysis. The ultimate analysis is also a chemical properties analysis that determines all component elements, solid or gaseous, meanwhile the proximate analysis determines physical properties that include the fixed carbon, volatile matter, moisture and ash percentages.

The ultimate analysis is determined by utilizing the equipment such as Carbon-Hydrogen-Nitrogen-Sulfur (CHNS) and AAS analyzer. Proximate analysis indicates the percentage by weight of the fixed carbon, volatiles, ash, and moisture content in coal. The amounts of fixed carbon and volatile combustible matter directly contribute to the calorific value of the coal. In other words, fixed carbon is carbon content of the coal which is not easily decomposed or combusted at a lower temperatures meanwhile, total carbon content of coal is volatile carbon present in form of other hydrocarbons forms like volatile organic compounds which easily combusted at a low temperatures. Fixed carbon acts as a main heat generator during burning process meanwhile high volatile matter content indicates the easy ignition of fuel.

Here is a briefly description on the fixed carbon, volatiles, ash, and moisture content in coal. Fixed carbon is the solid carbon left in the crucible after volatile matter burnt. It consists mostly of carbon but also contains some hydrogen, oxygen, sulphur and nitrogen not driven off with the volatile gases. In fact, the fixed carbon gives a rough estimate of the calorific value of the coal. Meanwhile, in general, volatile matters are the

methane, hydrocarbons, hydrogen and carbon monoxide and incombustible gases like carbon dioxide and nitrogen found in coal. Thus, the volatile matter is an index of the gaseous fuels present. Typical range of volatile matter is 20 to 35 wt.%. Volatile matter proportionately increases the flame length, and helps in ignition.

Ash is left over impurity minerals that not burnt. Typical range of ash is 5 to 40 wt.%. Ash in the coal will reduces handling and burning capacity and causes clinkering and slagging. Furthermore, ash can affect combustion efficiency and boiler efficiency hence will increases the handling costs (Speight, 2005). Lastly, moisture in coal must be controlled during transportation, handling and storage. Since it replaces combustible matter, it decreases the heat content per kg of coal. Moisture will increases heat loss due to evaporation and super heating of vapor.

2.9.2 Coal Pyrolysis

Coal pyrolysis is an initial step in the coal decomposition process and plays an important role in coal-based direct reduction process. In the initial stage of reduction process, the gases that evolved are acts as a reductant. When volatile gases reach the outside of a sample, it will reduce the reoxidation potential of the surrounding atmosphere and provide thermal heat. During pyrolysis, the structure of the coal changes and becomes more porous. This increases the reductant concentration through the Boudouard and water gas reactions while decreasing thermal conductivity. The final extent of reduction increases with increasing volatile matter content (Sharma, 1993).

The presence of volatile matter increases metallization rate and raises the melting temperature of the sponge iron (Bryk and Lu, 1986). The presence of iron oxides affects the coal thermal transformation and, hence, coal pyrolysis. Besides that, investigation by Cypres and Soudan-Moinet (1980) showed that the presence of iron oxides reduces the primary devolatilization rate of coal between 573-873 K. This in turn reduces the yield of tar and hydrocarbon. In the secondary devolatilization zone, the evolution of CO, H₂O, and CH₄ increases with the presence of iron oxides, while the hydrogen yield decreases.

2.10 Iron Ore Roasting

Primarily the purpose of roasting is to convert metallic sulphides into other forms (oxides, sulphates) amenable to further chemical treatment. Roasting was applied for the removal of impurities by the volatilization of certain oxides such as SO₂, SO₃, As₂O₃, Sb₂O₃, TeO₂, and SeO₂. This metal oxide volatiles has a low sublimation temperature range. In addition, Newton (1959) in his review stated that other purpose of roasting is in the reduction of certain metallic oxides by heating in contact with coal or other reducing agent (reducing roast) in air. The common metallic oxides that can be reduced are hematite. Roasting is often employed to convert nonmagnetic compounds into magnetic compounds as an initial stage to magnetic separation. For example nonmagnetic Fe₂O₃ may be converted into magnetic Fe₃O₄. The production of solid metallic (sponge) iron from iron oxides would be an example of a reduction roasting and the gas used would be hydrogen. Many metals are produced as metal powders by the direct reduction of solid oxides by a process which is essentially reduction roasting.

Jiang *et al.* (2008) claimed that hematite pellets have poor roasting properties and do not achieve adequate strength until the roasting temperature is above 1573 K. This creates strict demands for materials used in the roasting equipment and causes production costs to increase. Generally, the main factors affecting compression strength of roasted pellets are preheating temperature, roasting temperature and time. The induration mechanism of individual hematite pellets are characterized by the growth and recrystallization of Fe_2O_3 grain at high temperature. It is shown that hematite particles may keep their original shape and remain separate below 1473 K and only reduced when the roasting temperature reaches 1573 K. In the roasting process, when temperature is lower than 1523 K, the strength provided by the growth of Fe_2O_3 crystalline grains from secondary hematite grains. However, Li *et al.* (2009) found that the pellet strength decreases over 1623 K because of decomposition. Thus, a higher roasting temperature and narrower firing range is required for hematite pellets that give rise to difficulties. Furthermore, the roasting behavior of hematite pellets is inferior to that of others iron oxide pellets such as magnetite.

The influence of fluxes additives on the oxidized iron ore were extensively investigated by Fan *et al.* (2010a). The fluxes studied were CaO and MgO. For roasted pellet with additives of calcium oxide, the compressive strength increases initially and then drops with an increase in basicity. The maximum compression value is obtained at the basicity of 0.4–0.6. The explanation given was that the reaction of CaO with Fe_2O_3 and SiO_2 at the roasting temperature of 1173 K produces calcium-ferrite. At this temperature, the calcium ferrite is in a liquid phase that will improve the recrystallization