AES/RE/11-10 An optimised iron ore grinding strategy

based on balling fundamentals

May 2011 F.G. van Schijndel

# **TATA STEEL**



Title : An optimised iron ore grinding strategy based on balling

fundamentals

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**PREFACE** 

This thesis is part of a larger research project that aims at increasing the production of the

pellet plant of Tata Steel IJmuiden. This Master thesis has been written in cooperation

between the Iron making (IRM) department of Tata Steel Research, Development and

Technology (RD&T) at IJmuiden and the Delft University of Technology (DUT) as a

completion of my master in Resource Engineering.

During the past 9 months I worked closely together with my supervisors Dr. Ir. T.P.R. de

Jong, Mrs. M. Oorsprong M.Sc. and Dr. E. Scheepers at Tata Steel IJmuiden and Dr. J.H.L.

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my examination committee.

Guus van Schijndel

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V

### **SUMMARY**

In order to meet the future demand of pellets for the blast furnace, the output of the grinding circuit of the Tata Steel IJmuiden pellet plant needs to be increased. As the power consumption of the grinding circuit is already at its maximum, the grinding itself and/or the grinding strategy needs to be optimised, leading to a higher circuit output while still achieving the required fineness.

Further processes within the pellet plant require the grinding product to be of a certain fineness which is currently determined by measuring the Blaine number of the grinding circuit output, which is a measurement of the available surface within the grind.

At the IJmuiden pellet plant, grinding is done in a closed grinding circuit, meaning that only particles smaller than a certain size are allowed to leave the circuit. Particles that are to coarse are screened out of the main flow of material and fed back to the grinding mills.

The pellet feeds of the IJmuiden pellet plant are blends of different ores. The compositions of these blends are based on several factors such as availability, price and iron content. Properties that affect the grinding of the ores, such as initial fineness and grindability, however, are not taken into consideration in the selection of ores.

Given this fact, a possibility to increase the grinding capacity might be found in treating the feed of the grinding circuit not as a single material, like is done at the moment, but as a collection of individual ores. This approach allows for a blending/grinding strategy to be designed based on overgrinding softer ores and leaving the harder ores coarser while still achieving the required overall fineness. As this would reduce the energy needed to achieve a certain fineness, this would increase the grinding capacity.

Based on knowledge gained through a literature study on the binding mechanisms in iron ore green pellets, the strength and plasticity of green pellets is believed to be affected by such variations in individual ore fineness within the ore feed.

To study the influence of individual ore fineness on green pellet strength and plasticity, an experimental study was designed that involved balling and testing of green pellets balled from

four different feeds that only varied in individual ore fineness. In addition, the influence of strain rate on strength and plasticity of these same pellets was tested.

It was found that variations in individual ore fineness had no significant influence on the strength and plasticity of green pellets. Pellet plasticity and strength were found to be strain rate dependent even for very low strain rates.

Based on these experimental results, recommendations for further work and possible improvements in pelletising process monitoring were put forward.

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### 1 INTRODUCTION

At IJmuiden efforts are made throughout the entire operation to optimise both production and efficiency of the steel making process to meet the growing global demand of steel. At the moment approximately 50% of the IJmuiden blast furnace ferrous burden consists of pellets which are produced in a pellet plant from raw iron ores that are shipped in from mines all over the world. To meet future demand of pellets, the total production as well as the quality of the IJmuiden pellet plant needs to be optimised.

The pelletising process at IJmuiden can be divided into three consecutive stages; the drying/grinding stage, the blending/balling stage and the induration stage. The process has a continuous character which means that the output of one stage is the immediate input for the next. Because of this, the total production as well as the quality of the final product of the process is directly affected by the performance of each individual stage.

To ensure a good performance of the pellet plant, a number of process parameters is closely monitored within or in between the different process stages. This monitoring is performed in two ways, namely quantitatively and qualitatively.

Quantitative monitoring includes online monitoring of process variables such as throughputs, final production and additive quantities, whereas quality monitoring consists of periodic sampling and testing of raw material inputs such as ore feed and water, intermediate products such as green pellets, and fired pellets at the end of the process. Sample points are carefully chosen in such a way that the output of these tests, combined with the online monitoring data, is assumed to provide sufficient information to run the pellet production process as efficiently as required.

One of the periodic qualitative tests performed in the pelletising process in IJmuiden is that of impact- and compressive strength of green pellets leaving the balling stage. Both of these are important green pellet properties in the further pellet production process as green pellets have to withstand a certain degree of impact stress during transportation after the balling stage as well as compressive stress during the drying and firing stages. Failure and/or deformations of

green pellets have a negative influence on both the total production and quality of fired pellets and should therefore be limited as much as possible.

In general pellet plants are located near mine sites and operate using a single ore. At IJmuiden, pellets are produced from a blend of ores of which the composition is based on several criteria such as ore price, availability and chemistry and therefore varies over time. The ores that make up these blends come from mines all over the world and between them there can be a considerable difference in characteristics such as mineralogy and grindability. This allows IJmuiden to use opportunities provided by the market in optimising their pellet feed in terms of chemistry and grindability. However, due to this constant variation in ore feed properties, the pelletising process at IJmuiden is more challenging, requiring additional knowledge and tighter process managing compared to regular single ore pelletising operations.

The capacity of the grinding stage is considered to be the bottleneck within the pellet plant in terms of production. Therefore, in order to optimise the entire process, increasing the capacity of the grinding stage is required, while maintaining an output which still satisfies the demands of the succeeding process stages in terms of quality.

In order to raise the output of the pellet plant grinding stage, an automation of the grinding circuit by on-line automatic particle size measurement is considered. Quality control of the ore feed leaving the grinding circuit is currently done by measuring the average Blaine number over a time interval of approximately eight hours.

By monitoring the circuit on a much smaller time scale and with a smaller measuring error, online particle size measuring is expected to make it possible to control the grinding circuit more accurate, resulting in a much more constant and more predictable grinding circuit output. This decrease in variation and increase in predictability will create additional room for optimisation.

A project was started of which the main goal was to identify improvement opportunities that would increase the pellet production at IJmuiden. This project was divided into three consecutive stages:

**Main Goal:** *Indentify improvement opportunities to increase pellet production* 

Stage 1: Quantify the effects of physical factors within the pelletising process

Stage 2: Indicate methods to increase the production of pellets of defined quality at the current grinding capacity possible at IJmuiden

Stage 3: Identify improvement opportunities in grinding and circuit control to reach this maximum

This report covers the whole of stage 1 and partially covers stage 2 by discussing one possible method to increase pellet production.

#### Stage 1

A thorough literature review was performed on the binding mechanisms responsible for green pellet strength and the main factors of influence on those. This literature review is discussed in chapter 2. Subsequently, process data from the last four years of IJmuiden's pellet plant were analysed to confirm the factors of influence on green pellet compressive strength as described in literature. This process data analysis is described in chapter 3. No strong correlation was found between green pellet strength and the assumed main factors of influence. Several possible explanations of this weak correlation have been assessed. The weak correlation found was attributed to measuring errors and unmeasured process variations.

#### Stage 2

The knowledge gained from the literature review performed in stage 1 was used to indicate possible opportunities to increase the pellet production at IJmuiden in. This report discusses one of these opportunities for improvement.

The grinding stage of the IJmuiden pellet plant is considered to be the bottleneck in terms of production. The feed of this grinding stage consists of a blend of ores which vary greatly in

grindability. Grinding is done in a closed grinding circuit, meaning that only particles smaller than a certain size are allowed to leave the circuit. Particles that are to coarse are screened out of the main flow of material and fed back to the grinding mills.

Given these facts, a possibility to increase the grinding capacity might be found in treating the feed of the grinding circuit not as a single material, like is done at the moment, but as a collection of individual ores. This approach allows for a blending/grinding strategy to be designed based on overgrinding softer ores and leaving the harder ores coarser while still achieving the required overall fineness. As this would reduce the energy needed to achieve a certain PSD, this would increase the grinding capacity.

The literature study showed that surface related ore characteristics such as wettability and both particle roughness and shape are believed to have an influence on the green pellet strength. A variation in individual ore fineness within a blend would result in an unevenly distribution of the ores over different size fractions. This means the relative available surface area for each ore no longer matches the initial blend composition, changing the overall surface related properties of the blend and potentially the green pellet strength.

Green pellets have to withstand a wide range of strain rates during the pelletising process. From the literature review, the influence of surface properties appeared not to be affected by the used strain rate during testing, where the other main believed binding force, the viscous forces of the binder, are believed to depend on strain rate above a certain strain rate. However, experimental work discussing the influence of strain rate on green pellet strength is limited.

In order to investigate the influence of both individual ore fineness and strain rate on green pellet strength the following hypothesis was formed:

"The <u>Compressive Strength</u> of green pellets balled with the use of a <u>Bentonite Binder from a</u> pellet feed with a <u>Fixed Particle Size Distribution</u> consisting of a <u>Blend</u> of several iron ores, is influenced by the <u>Individual Fineness of Ores</u> within- and the overall <u>Composition</u> of the blend. However, a <u>Critical Strain Rate</u> exists above which both variables loose this influence"

To test this hypothesis, an experimental design and procedure were developed which are discussed in chapter 4 and 5 respectively.

Three different ore blends, made from three different ores, were prepared with the same overall particle size distribution but varying individual ore finesses. The desirable individual ore fineness for each ore was obtained by blending samples of that ore which had been ground to different finenesses of known particle size distribution. The three blends were then balled with the use of a Bentonite binder into pellets of identical plasticity, porosity and with a equal level of saturation. After balling the wet compressive strength was determined at two different strain rates.

The experimental results are discussed in chapter 6. It was found that individual ore fineness had no significant influence on the green pellet strength. The strength of pellets was found to be strain rate dependent even for very low strain rates. Also, based on the experimental results recommendations for further work and possible improvements in monitoring of the pelletising process are discussed in chapter 7.

### 2 LITERATURE REVIEW

As described in chapter 1, the first stage of the project consisted of gain a good understanding of the binding mechanisms within iron ore green pellets and the main factors of influence on green pellet strength. The first step of this stage consisted of a literature review which is presented in this chapter.

#### 2.1 GREEN PELLET BINDING MECHANISMS

The research done in the field of green pellet strength until now can be divided in two different eras. This division is marked by the recognition of a new dominant binding force in green pellets resulting in a new and adjusted green pellet binding mechanism model. In this chapter, both models, and the differences between the two, are discussed.

#### 2.1.1 Classical model

The first efforts to explain green pellet strength were done by Firth in 1944. He concluded that the binding mechanism in balling was driven by compaction of single grains by the weight of the pellet itself during tumbling in a way similar to briquetting. In 1950, Tigerschiöld recognized the role of water surface tension- and capillary forces through experimental work and labelled these as the dominant binding forces in green pellets. These experiments formed the foundation of later work by Newitt and Conway-Jones (1958), Rumpf (1961) and Schubert (1977). The combination of their findings resulted into what can be considered the classical green pellet binding mechanism model.

In 1961, Rumpf was able to do good estimations on the compressive strength ( $\sigma$ ) of conglomerates based on porosity ( $\epsilon$ ), the size of the individual particles (x) and the binding force between them (F) using:

$$\sigma = \frac{(1 - \varepsilon)F}{\varepsilon x^2}$$
 (Equation 2.1)

The classical model states that this green pellet binding force predominantly correlates with the level of liquid saturation of the pellet. In determining green pellet strength, the attribution of other binding forces such as vanderWaals-, magnetic- and/or electrostatic forces was considered negligible. Three different saturation states can be identified in which different binding mechanisms are dominant; the Pendular (Fig. 2.1a), Funicular (Fig. 2.1b) and Capillary state (fig. 2.1c) (Newitt and Conway-Jones, 1958).

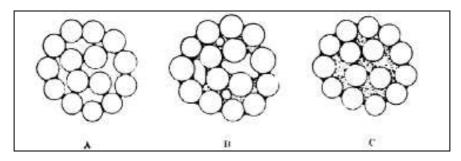


Figure 2.1 Different states in saturated green pellets (Newitt & Conway-Jones, 1958)

#### 2.1.1.1 Pendular state

At low saturation levels (S < 0.25) the green pellets are in what is described as the **Pendular state**. In this state, the dominant binding force is that of single water bridges forming between individual grains (Figure 2.1a). Figure 2.2 is a close-up of one of the grain-grain interfaces in Figure 2.1a in which all relevant parameters are labelled.

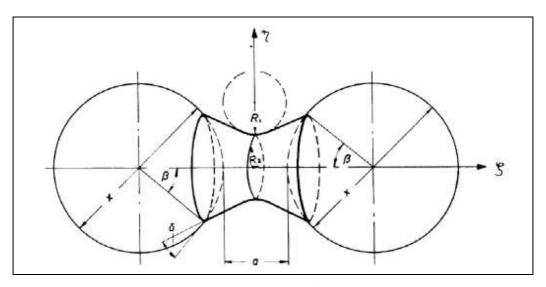


Figure 2.2 Water bridge between particles (Schubert, 1977)

Two mechanisms are held responsible for the total binding force in liquid bridges between wetting particles. The most important one is the capillary force resulting from the under pressure created in the bridge because of the difference in curvature of  $R_1$  and  $R_2$ . In addition, the contact force at the liquid – solid interface is directed in such a way that is adds to the total binding force. Based on this assumption it was stated that the binding force in the pendular state is a function of liquid surface tension, particle diameter (x), filling angle ( $\beta$ ), separation distance (a) and wettability op the solid (contact angle  $\delta$ ) in the form:

$$F_{binding} = f(\beta, \delta, \frac{a}{x})$$
 (Equation 2.2)

The binding force in liquid bridges can not be expressed in a closed formula and therefore needs to be calculated numerically (Schubert, 1977).

#### 2.1.1.2 Funicular state

As the saturation level is increased (0.25 < S < 0.8), more and more liquid bridges merge to form small capillary tubes. In this **Funicular state**, binding forces of both the capillary tubes and the remaining single water bridges are responsible for green pellet strength.

#### 2.1.1.3 Capillary State

As the saturation level is increased even further (S > 0.8), the strength of the green pellet is at its highest. All the pores of the pellet are filled with liquid creating one large capillary system. Liquid bridges are no longer present and only the capillary forces are responsible for the pellet strength. The binding forces active in this **Capillary state** are approximately three times higher than those in the Pendular state (Rumpf, 1961). The capillary (under) pressure which is responsible for the binding force can be expressed in the formula:

$$P_k = \frac{2\gamma \cdot \cos \theta}{R}$$
 (Equation 2.3)

With.

$$P_k$$
 = Capillary Pressure [Nm<sup>-2</sup>]  
 $\gamma$  = Surface tension [Nm<sup>-1</sup>]  
 $\theta$  = Contact angle [ $^0$ ]  
 $R$  = Radius of curvature [m]

In the case of a completely wetting solid ( $\theta = 0$ ), the radius of curvature (R) is equal to the pore radius (r) and equation 2.3 can than be simplified into:

$$P_k = \frac{2\gamma}{r}$$
 (Equation 2.4)

Equation 2.4 describes the capillary pressure in a single tube and is therefore not applicable to green pellets in which the void dimensions are not always tube-like and differ within the pellet. To account for this, the hydraulic pore radius was introduced (Tigerschiöld and Ilmoni, 1950). This theoretical radius is calculated using the porosity  $(\varepsilon)$ , solid density  $(\rho)$  and Specific surface  $(S_m)$ :

$$m = \frac{\varepsilon}{S_m \cdot \rho \cdot (1 - \varepsilon)}$$
 (Equation 2.5)

A combination of Equation 2.4 and Equation 2.5 gives:

$$P_{k} = \gamma \cdot S_{m} \cdot \rho \cdot \frac{1 - \varepsilon}{\varepsilon}$$
 (Equation 2.6)

From equation 2.6 it can be seen that, according to the classical green pellet binding mechanism model, the strength of green pellets leaving the balling circuit (S > 0.8) is mainly dependent on the fineness of the ore.

If even more liquid is added and the pellet becomes oversaturated, the pore openings at the surface of the pellet will overflow, turning the pellet in nothing more than a water drop with a suspended solid. The only binding force remaining is the water surface tension and the green pellet strength drops to nearly zero.

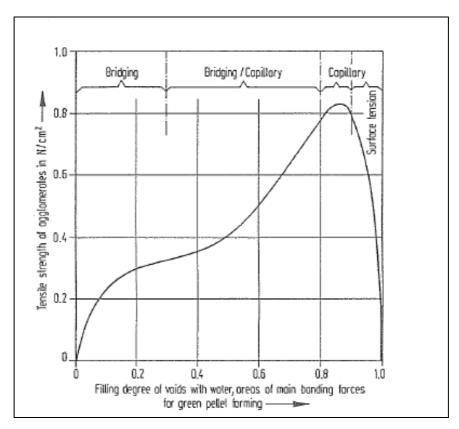


Figure 2.3 Classical relation between saturation and tensile strength of conglomerates, limestone ( $\epsilon$  = 0,41) (Tarjan, 1966)

#### 2.1.2 Modern green pellet binding model

The classical binding mechanism model, as described in chapter 2.1.1, provides a good overall understanding of the basic mechanisms involved in pelletising. Most classical work however, is limited to static experiments using spherical bodies of a narrow size distribution and low viscous Newtonian fluids alone, making the results of these experiments less applicable to real case processes. In the last two decennia, increasing efforts have been made towards understanding the mechanisms of real life granulation processes and being able to make quantitative predictions on these processes purely based on theory instead of laboratory and pilot scale tests (Iveson et al., 2001) In this research, several additional factors influencing green pellet strength and new insights to the pellet bonding mechanism have come to light which had previously been overlooked by the classical binding model. This chapter will discuss these new insights, resulting in the modern green pellet binding model.

#### 2.1.2.1 Bentonite Binder Properties & Viscosity

Bentonite is a mixture of clay that primarily consists of the smectite class mineral montmorillonite with the chemical formula:

$$(Na, Ca)_{0,33}(Al_{1,67}, Mg_{0,33})Si_4O_{10}(OH)_2$$
.  $nH_2O$ 

Bentonite particles, as is typical for clay minerals, are shaped like little sheets. These sheets are held together by cations in between different layers. A bentonite unit cell, consisting of two silica layers and one layer of cations is shown in Figure 2.4.

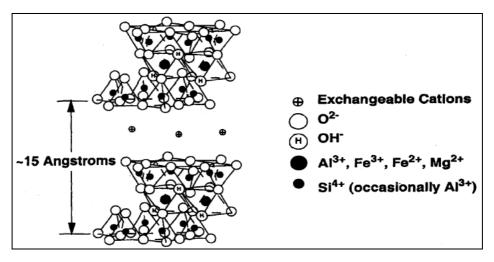


Figure 2.4 Bentonite unit cell. (Grim, 1968)

Through hydration of the cation layer, water can be absorbed by the mineral between the silica layers. This makes the bentonite expand upon wetting resulting in a thick viscous paste. This characteristic makes bentonite very suitable for the use of binder in pelletising processes. Due to isomorphic substitution of the Al<sup>3+</sup> ions, the overall charge balance of the crystal can be altered, which is compensated by the absorption of extra cations, usually Na<sup>+</sup> or Ca<sup>+</sup>. Since this increases the cation density and therefore the interlayer attraction, the crystal can not expand as much as usual. Because of this higher resistance to expand, the bentonite crystals become less capable of absorbing water (Kawatra and Ripke, 2003). Near the edges of the bentonite particles the particle charge is governed by the existence of aluminol and silanol groups which are dependent of the surroundings pH (Lagaly and Ziesmer, 2003)

Because of the sheet like structure, shear stresses after or during hydration can stretch bentonite crystals in a way comparable to a deck of cards creating long fibers that are believed to make bentonite even more effective as a binder (Kawatra and Ripke, 2002).

As mentioned before, mixing bentonite with water results in a viscous liquid making it suitable as a binder in granulation processes. In earlier work, water-bentonite mixtures were found to be non Newtonian, shear thinning, thixotropic fluid. This means that its viscosity not only decreases under increasing applied stress but also over time after being agitated (Kok et al., 2000; Kok, 2011).

#### 2.1.2.2 Viscous forces and inter-particle friction

One of the biggest differences between the experiments leading to the classical pellet binding mechanism model and real life pelletising plants is the addition of the binder Bentonite. Bentonite is widely used in pelletising operations because of its ability to, in some cases, absorb up to ten times its own weight in water, resulting in a viscous liquid. The mechanisms behind this are discussed in section 2.1.3.

Ennis et al. (1991) were one of the first to acknowledge and describe the influence of binder viscosity on granulation. They stated that, due to the dynamic nature of pelletising, the strength of liquid bridges also had to depend on liquid viscosity. Experimental work on single pendular liquid bridges (Ennis et al. 1990) showed that binder viscosity indeed had a mayor positive effect on liquid bridge strength. This work, however, only focused on pendular bridges which, as described in section 2.1.1.1, are only responsible for pellet strength at low saturation levels.

The influence of binder viscosity along the entire saturation range was reviewed by Forsmo et al. in 2006. This study also clearly showed an undeniable influence of binder viscosity. In saturation regions comparable to the ones present in actual pelletising processes, the compressive strength of green pellets even doubled after an addition of 0.5% Bentonite.

Also, in more dry pellets the frictional forces were believed to have a large contribution to overall strength.

It was concluded that pellet strength is controlled by the capillary forces of the liquid bridges, the viscous forces of the binder and the internal friction forces between particles (Iveson et al. 2001).

#### 2.1.2.3 100% saturation region and solid bridges

In the classical model, as can be seen in Figure 2.3, the maximum compressive strength is reached at a saturation level of about 90%. After this, the capillary tubes within the pellet overflow and the only remaining binding force is that of surface tension of the water. Because the classical model is based on smooth perfect spherical particles, all capillary tubes will overflow at roughly the same time, resulting in a sudden steep drop in compressive strength for increases of saturation above the optimal level. In real life however, the particle roughness and uneven nature of the surface will cause the pellet to overflow at different saturation levels

in different areas. In addition, the overflowed areas still possess a rather high bonding strength due to the viscosity of the liquid. This results in a much smoother drop in compressive strength as the saturation is raised above the 100% saturation level.

Also, an increase in saturation around the 100% saturation level is found to lead to a simultaneous increase in porosity rather than an overflow of the pellet pores. This is further explained in 2.2.3.1. Because of this behaviour, the operational balling area is found to be around 110% saturation of the original pores (Forsmo et al., 2006; Forsmo et al., 2008).

At low saturation levels (<50%), the bentonite particles come out of solution and start forming solid bridges. This leads to an exponential increase in compressive strength as the saturation level is lowered.

These new insights and additions, combined with the effects of bentonite, as summarized in Figure 2.5, alter the saturation/compressive strength relation considerably compared to the one presented previously by the classical binding mechanism model in Figure 2.3. Also, it should be mentioned that because of the concentration of bentonite under evaporation, Figure 2.5 represents the change in pellet strength under drying of the pellet, where the old model makes no distinction between drying and wettening.

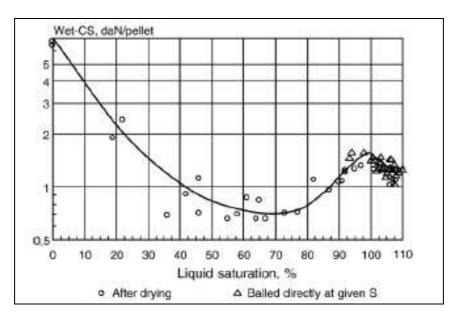


Figure 2.5 Modern relation between saturation and Compressive strength of conglomerates. (Forsmo et al., 2006)

#### 2.2 FACTORS OF INFLUENCE

The factors of influence on the pelletising of iron ore can be subdivided in three categories; the quality and characteristics of the ore feed and the quantity and chemistry of both added water and bentonite. This chapter will discuss in which way these different factors influence the iron ore pelletising process based on experimental work and the modern green pellet binding model.

#### 2.2.1 Ore Characteristics

#### 2.2.1.1 Particle Shape

The shape of a particle is proved to be of influence on the dynamic strength of liquid bonded granular materials by Iveson and Page in 2004. In this study it was found that powders consisting of spherical particles resulted in a pellet with a lower strength than pellet formed by irregular, even dendritic particles. This is in compliance with the classical green pellet binding theory since a more irregular shape implies a higher specific surface. According to equation 2.6 and equation 1.1, this automatically leads to higher green pellet strength.

Also, in conglomerates formed from irregular shaped iron ore powders, the compressive strength can be relative high due to the interlocking forces of the particles.

#### 2.2.1.2 Wettability / Absorption

Based on equation 2.3 it is expected that a higher wettability of particles is preferable for good pelletising. This expectation was confirmed by the work of Iwasaki et al. in 1967, who concluded that the liquid bridge forces between iron ore particles which had been treated with hydrophobic chemicals during flotation had decreased.

Iveson et al. (2001) however described that an increase in wettability also increased the amount of water being sucked into the particle pores resulting in the need of additional watering. This additional watering has a negative effect on the following drying process. Therefore, the existence of an optimum between these two phenomena was suggested. In a

later study, Iveson also showed that in iron ore consisting for at least 95% of hematite and goethite the contact angle increased as the hematite content increased (Iveson et al. 2004).

#### 2.2.2 Particle Size Distribution and Specific Surface

Particle size and distribution as well as specific surface are well known key factors of influence on green pellet strength. For instance, equation 2.1 directly correlates green pellet strength with particle size. The theory behind this is that, as average particle size decreases, the number of particles, and therefore the number of bindings, present at the plain of failure increases, resulting in a higher strength (Rumpf, 1961). A decrease in particle size also has its effect on the binding forces itself as smaller spheres have a lower volume to surface ratio; as particle size decreases, the specific surface increases. Equation 2.6 tells us this will lead to a further drop in capillary pressure, resulting in stronger bonds. This relation between particle size and green pellet compressive strength has been confirmed in several studies (Meyer 1980; Sportel 1995). The results of one of these studies are shown in Figure 2.6.

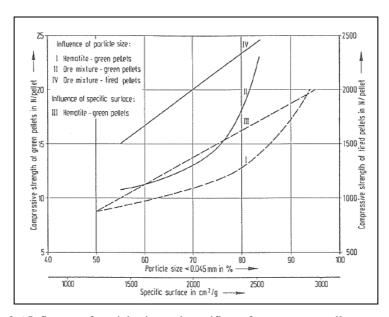


Figure 2.6 Influence of particle size and specific surface on green pellet compressive strength (Meyer, 1980)

Apart from average particle size, particle size distribution also plays an important part in green pellet strength. According to eq. (1), powders of narrow size distribution produce weaker pellets than expected judging on the average particle size alone. This is explained by

the fact that powders of wider size distributions result in a better packing, due to smaller fractions filling up the voids left between larger ones, which results in a decrease in pellet porosity. By decreasing the number of bindings present at the plane of failure as well as the available surface area, while simultaneously increasing the diameter of capillary tubes (see equation 2.1), an increase of porosity results in a lower compressive strength.

In order to optimise the packing of powders during agglomeration, research has been done on the ideal size distribution based on the filling of voids between larger fractions by smaller ones. It was found that in a binary particle system a minimum in porosity could be reached if the ratio between the two fractions was around 0.67. Also, In multiple component systems a minimum in porosity exists dependent of the particle size distribution (Ball et al., 1973).

In some cases a wide particle size distribution, often in combination with irregular particle shape, can result in relatively strong green pellets due to the increase in interlocking forces which counteract the compressive forces (Kristensen et al. 1985).

As described above, all three principal factors of influence on pellet strength as described in equation 2.1 are influenced by the particle size, distribution and/or specific surface. Therefore much experimental work has been done in this particular field which all confirm the relations mentioned above (Meyer, 1980; Sportel, 1995; Iveson et al. 2001).

#### 2.2.3 Water

The influence of water on the strength of green pellets has already been discussed in section 2.1. As an addition to this, in this chapter the influence of water content on the green plasticity as well as the influence of water chemistry on pellet strength is described.

#### 2.2.3.1 Water Content, Saturation and Plasticity

It should be noted that the influence of water on green pellet strength is based on the saturation of the particle and not the moisture content of the mixture to be balled. In fact, saturation is a function of moisture content, pellet porosity and the absorption of water by individual particles. This relation is easy to understand and widely accepted for saturation

levels encountered during drying (S<100), but the mechanisms at the saturation levels encountered during actual balling are less well understood.

Forsmo et al. concluded in 2006 that the saturation level at which pelletising operations operate are well above the theoretical 100%. At this point saturation becomes insensitive to moisture content. Any addition of water at this stage will result in an increase in porosity, leaving the saturation unchanged. This increase in moisture content does however have a big influence on the green pellets plasticity. As moisture content increases the green pellet becomes more and more plastic increasing its impact strength (drop number) and altering its breakage pattern under compression from brittle to more plastic. Compressive strength only decreases slightly, since the dominant binding force is still that of the viscous binder. This shift in breakage pattern, however, results in a huge increase in deformation during compression. Based on these findings, Forsmo concluded that plasticity should gain the status of a standard pellet quality test as it holds valuable information on whether a balling circuit is running at its most ideal moisture and bentonite level.

This behaviour of oversaturated green pellets as described above was observed earlier by Maidorn in 1962 as can be seen in Figure 2.7. He however had no explanations for this behaviour.

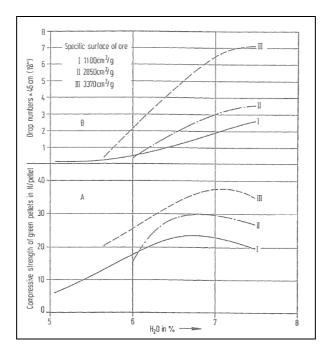


Figure 2.7 Effect of moisture content on compressive (A) and Impact (B) strength for green pellets of different specific surface, based on Maidorn. (Meyer, 1980)

### 2.2.3.2 Water Chemistry

The influence of water chemistry on green pellet properties described in literature particularly focuses on its influence on bentonite. Several studies have shown that the effectiveness of bentonite as a binder depends on the type of cations in the layer between bentonite platelets, which are described in chapter 2.1.3. The most common cations present in water are Na<sup>+</sup> and Ca<sup>2+</sup>. If the cation layer mostly consists of sodium cations, the silica platelets become capable of moving more freely, resulting in a more effective binding. In this state the bentonite is considered to be hyperactivated and less bentonite is needed to meet the same green pellet requirements (Kawatra and Ripke, 2003).

#### 2.2.4 Bentonite

As described in section 2.1.2.2 the influence of bentonite on green pellet strength is fully acknowledged in modern literature. This chapter discusses the influence of bentonite dosage and chemistry on the strength of green pellets.

### 2.2.4.1 Bentonite Dosage

In recent studies, the addition of bentonite to ore feed in pelletising processes has proved to increase both the impact- and compressive strength of green pellets as can be seen in Figure 2.8.

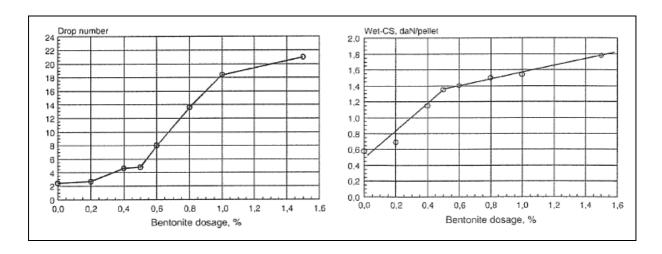


Figure 2.8 Influence of bentonite dosage on green pellet impact- (left) and compressive (right) strength. Moisture content between 8.7% and 9.3%. (Forsmo et al., 2006)

Especially the positive effect on impact strength (drop number) has been confirmed in several studies (Sportel, 1995; Forsmo, 2006) and is contributed to the increase in viscosity of the binder. This higher viscosity enables the binder to absorb a lot of the impact energy and increases the plasticity of the green pellets. As can be seen in Figure 2.8, this binding mechanism starts being dominant for bentonite values higher than 0.5%. Also, after this point up, a distinct switch in the green pellet breaking pattern was observed from brittle to plastic (Forsmo et al., 2006).

The influence of bentonite on compressive strength as described in Figure 2.8 is not fully accepted and several studies have concluded the influence of bentonite addition on green pellet compressive strength to be minimal. (Meyer, 1980).

This contradiction in outcomes can possibly be explained by the difference in strain rate used in the different experiments and suggests the existence of a critical strain rate. For strain rates lower than this critical strain rate, the viscosity of the binder no longer resists the deformation and therefore has no influence on the green pellet strength leaving the capillary force to be dominant.

# 2.2.4.2 Bentonite Chemistry / Viscosity

As described in section 2.1.2.1, bentonite is a mixture of different minerals, primarily consisting of montmorillonite. As most clay minerals, its chemical formula allows the existence of various types of montmorillonite varying in the type of ions present in the crystal roster. The amount of montmorillonite in a certain bentonite and the type of this montmorillonite determines how suitable it is as a binder. The most important property of bentonite with regards to pelletising is its swelling capacity. This capacity mainly depends on the ability of the cation layer, to expand and absorb water molecules.

This ability to expand is at its highest when the charge between the two silica layers is at its lowest. For this reason, the ratio between single charged Na<sup>+</sup> cations and double charged Ca<sup>2+</sup> cations in the cation layer is a good indicator of the swelling capacity of a certain montmorillonite and therefore its quality as a binder (Kawatra and Ripke, 2002).

The change from calcium-bentonite to sodium-bentonite is referred to as "hyperactivation" of bentonite and can also be attained artificially by the addition of NaOH (Saidi et al., 2001).

Not only bentonite dosage and type, but also the way in which it is applied is believed to be of influence on the strength of green pellets.

Experiments using different types of mixing have shown that mixing types in which shear forces are involved resulted in green pellets of higher compressive strength. This was attributed to the formation of long fibers during mixing as the bentonite layers slide along each other due to these shear stresses (Kawatra and Ripke, 2001).

#### 2.2.5 Strain Rate

Unlike the capillary forces and internal friction forces, viscous forces are proven to be strain rate dependent. In tests at different strain rates and using binder of varying viscosities, it was shown that a critical strain rate exists below which the viscous forces are negligible and the pellet strength is mainly dominated by the capillary forces as can be seen in Figure 2.9 (Iveson at al., 2002)

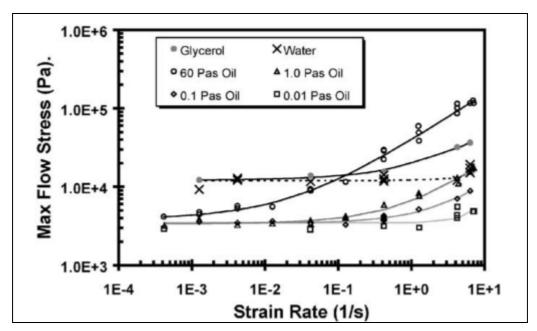


Figure 2.9 Influence of strain rate on granule max stress for binders of different viscosity. Saturation 0.7. (Iveson et al. 2002)

The critical strain rate was found to be dependent of the viscosity of the binder. The more viscous the fluid, the lower this critical strain rate was found to be. These studies were however conducted using narrow size distribution of relatively large glass ballotini. These results are therefore not directly applicable to describe the influence of strain rate on iron ore green pellets.

### 2.3 SUMMARY

Through the literature review described in this chapter, a thorough understanding of the binding mechanisms responsible for green pellet strength was acquired.

The total binding force responsible for green pellet strength is believed to be the sum of three different forces; capillary forces, frictional forces and viscous forces. Both viscous and capillary forces are believed to increase for increasing saturation levels lower than 100%. At over saturation (saturation > 100%), pellet porosity increases resulting in a weaker and more plastic pellet.

A grinding strategy involving over grinding softer ores and leaving harder ores coarse, as presented in section 1, will affect the overall surface related properties of the blend.

As frictional and capillary forces are dependent of surface related properties such as roughness and wettability, such a grinding strategy is believed to influence green pellet strength. The influence of these properties on pellet strength however have never been studied experimentally.

The contribution of capillary forces to the overall pellet strength are also believed to be dependent of strain rate. As strain rate increases, viscous forces within the bentonite binder increase, reducing the influence of frictional and capillary forces.

Viscous forces were found to be strain rate dependent for strain rates above a certain critical value. Experiments that confirmed this however, were performed using relatively large and often unisized particles as well as Newtonian fluids making their results not directly applicable to iron ore green pellets.

In order to make a valid statement on the influence of the proposed grinding strategy on green pellet strength, the influence of strain rate and variation in individual ore fineness on green pellet strength need to be further assessed. Therefore, a hypothesis has been formulated and tested. This experimental work is discussed in section 4, 5 and 6.

The next step of stage 1 of the project was to confirm the findings from the literature review using process data from the IJmuiden pellet plant.

# 3 PROCESS DATA ANALYSIS

### 3.1 INTRODUCTION

During the literature study a thorough understanding was acquired of the factors of influence on the strength of green pellets produced in processes similar to that of the IJmuiden pellet plant. To confirm the assumption that the strength of IJmuiden pellets was governed by the same mechanisms as described in the literature review, the relationships between IJmuiden green pellet strength and these main factors of influence were analysed using 4 years of process data.

# 3.2 DATA COLLECTION, INSPECTION AND FILTERING

The process input variables used in the data analysis were derived from the literature study. All available process variables that were assumed to have a considerable influence on green pellet strength were taken into account. The data used was obtained from the RADAR<sup>™</sup> database, which holds all measured process data, for the period of January 2006 till September 2010.

Each of the process variables is measured in a different way and/or on a different time scale. Most measurements result in a single value, but some test result in a collection of values as is the case for the measurement of ore fineness by determining the particle size distribution. Before the particle size distribution of the ground ore feed, consisting of 32 entries, could be used as an input parameter, a representative way needed to be found to describe the distribution in far less variables.

#### 3.2.1 Green Pellet Strength

The strength of green pellets is measured every 8 hours. A sample is taken from the product pellets leaving one of seven balling circuits of which the 10mm - 12,5mm fraction is screened off. The compressive strength of 25 pellets is measured by applying an increasing load on the pellets up to the point of fracturing. The load at the point of fracturing is visually judged by the operator and rounded off  $\pm 50$  grams. The average of a 25 pellets is reported as the Green Pellet Compressive Strength in grams.

#### 3.2.1 Water and Bentonite content

The pellet plant has three blending circuits in which water and bentonite are added to the pellet feed to create a mixture suitable for balling. The weight percentages of added bentonite and water are measured continuously and an hourly average is stored for both values. These values are reported as a percentage of added Bentonite and water.

#### 3.2.2 Blaine Number

The output of all three grinding circuits of the IJmuiden pellet plant grinding circuit is sampled and collected every 10 minutes. Every shift, corresponding to approximately every 8 hours, the Blaine number of this collection sample is determined. This measurement is done for all three grinding circuits and is reported as the Blaine number in cm<sup>2</sup>/gram.

#### 3.2.3 Particle Size Distribution

The same collection sample used to determine the Blaine number is also used to determine the particle size distribution. The particle size distribution (PSD) of the ground pellet feed is measured using laser diffraction (provided by Sympatec<sup>®</sup>) and consists of a cumulative series with 32 entries. In order to be able to analyse the correlation between particle size distribution and other process variables, the information contained in a PSD needed to be compressed to a

much smaller amount of variables which would still be representative enough for correlation analysis. There are many methods available to describe a PSD in one or more variables for all kinds of different purposes (Schubert, 1989; King, 2002). The method that would best represent the PSD characteristics that are of influence on green pellet strength was not explored. In this data analysis, the PSD has been described using a range of methods.

#### 3.2.3.1 Size Fractions

From the literature review it was found that especially the amount of fine particles in the pellet feed are of influence on green pellet strength. This is due to the fact that small particles have a relatively large impact on the specific surface of the pellet feed and the eventual porosity of the green pellet. Because of this, the fractions  $<10\mu m$  and  $<25\mu m$  were selected as input parameters.

At IJmuiden, historically, the  $45\mu m$  and  $90\mu m$  fraction are used to describe the particle size distribution. Therefore, these fractions were also chosen as input parameter for the process data analysis.

#### 3.2.3.2 Percentiles

Cumulative particle size distributions of ground material such as pellet feed are known to resemble distributions which can be described using one or more percentiles. Two of these distributions are commonly used to describe grinding products;

#### **Rosin-Rammler Distribution**

The Rosin Rammler Distribution is based on the 63.2 percentile  $(d_{63.2})$  and is defined as:

$$P(d) = 1 - \exp[-\frac{d}{d_{63.2}})^{\alpha}]$$

#### **Gates-Gaudin-Schuhmann Distribution**

The Gates-Gaudin-Schuhmann Distribution is a distribution based on the 5, 50 and 95 percentiles  $(d_5, d_{50}, d_{95})$  and is defined as:

$$P(d) = (\frac{d}{d_0})^k$$

with,

$$d_0 = d_{50} \frac{d_5 d_{50} - 2 d_5 d_{95} + d_{50} d_{95}}{d_{50}^2 - d_5 d_{95}}$$

In both distributions, P(d) represents the probability of any value within a distribution to be smaller than a certain value d. Constants  $\alpha$  and k need to be determined empirically.

To study whether the particle size distribution of pellet feeds could be described with the use of certain percentiles, the average PSD of the output of all three grinding circuits for a period of 2 months was compared to both Rosin-Rammler distributions and Gates-Gaudin-Schuhmann distributions for different values of  $\alpha$  and k. All percentiles used were estimated using lineair interpolation of the original PSD entries. Because the first entry of the studied PSD's exceeded 5%, the 5% percentile (D<sub>5</sub>) was estimated at 1µm for all PSD's.

It was found that the  $<24\mu m$  portion of the curve could be described as a Gates Gaudin Schuhmann distribution with a k-value of 0.6 and the  $>24\mu m$  portion as a Rosin Rammler distribution with a  $\alpha$ -value of 1.2 with an error of <5% for all size fractions, as can be seen in Figure 3.1.

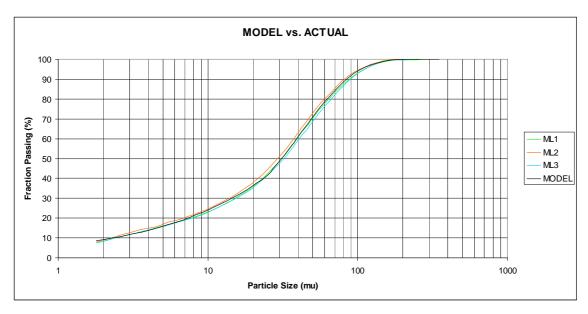


Figure 3.1 Description of the average PSD of output from grinding circuits 1, 2 and 3 using Rosin-Rammler distribution for d > 24 $\mu$ m and Gates-Guadin-Schuhmann distribution for d < 24 $\mu$ m.

Because of this resemblance between the studied PSD's and both the Rosin-Rammler and the Gates-Gaudin-Schuhmann distribution, the  $D_{63.2}$ ,  $D_{95}$  and the  $D_{50}$  were used as input parameters for the data analysis.

#### 3.2.3.3 Percentile Ratios

The ratio between certain percentiles is also used to determine the steepness of a PSD. The steepness of a PSD provides information about the width of the distribution. The width of the PSD of a powder is believed to be of influence on the packing of the powder which, in turn, has an influence on porosity and therefore green pellet strength. To account for this in the data analysis of the PSD's, the ratio of the 75 and 25 percentile,  $D_{75}/D_{25}$ , was used as an input parameter.

### 3.2.3.4 Sum of Size Fraction

Within the cement industry, another commonly used parameter that describes a PSD is the sum of all size fractions within a specific range of the cumulative PSD curve. This sum increases as the particles within the PSD become finer and/or the curve becomes less steep.

As small fractions are believed to have a relatively high influence on green pellet strength, in this study the coarser end of the PSD (>  $25\mu m$ ) should be excluded when using this method. The values of fractions are too high in relation to the smaller fractions, which would leave variation in the smaller fractions unnoticeable.

#### 3.2.4 Outliers

Through consultation of a pellet process expert, for each of the parameters a certain range was defined within which a value could be considered valid (Scheepers, 2010). All values outside this range were considered outliers and removed from the data set. The ranges used for each parameter as well as their units are listed in Table 3.1.

Table 3.1 Variables and ranges used during Data Analysis

Variable	Unit	Used Range
Green Pellet Compressive Strength	gram	500 - 1500
Water content	%	8 - 12
Bentonite content	%	0.2 - 0.8
Blaine	cm <sup>2</sup> gram <sup>-1</sup>	1000 - 3000
Particle Size Distribution		
$<10\mu$	%vol.	
$<45\mu$	%vol.	
<90μ	%vol.	
D50	μ	
D60.3	μ	
D95	μ	
$D_{\theta}$		
D75/D25		
D13/D23		
Cum. <25	%	

### 3.3 DATA GRIDDING AND STRUCTURING

Based on the literature review, the compressive strength of green iron ore pellets is suspected to strongly correlate with the fineness of the ore as well as the moisture and bentonite content of that particular pellet. These parameters are all taken into account during this data analysis. The measurements of these parameters however are done at different points in the process and/or at different time intervals.

Because of this, the exact ore fineness and moisture and bentonite content for a certain batch of tested green pellets is unknown and needs to be estimated. An estimation of the properties of all tested green pellets was done by gridding and averaging each of the data sets of the parameters.

#### 3.3.1 Discrete vs. continuous data

The average quantity of bentonite and water added to the feed for the balling circuits is stored continuously and an average value is generated every hour. The measurements of green pellet compressive strength and both Blaine number and particle size distribution of the grinding circuit output however, are measured approximately every 8 hours.

In order to be able to study the correlation between these parameters, the discrete datasets of compressive strength, Blaine number and Particle size distribution needed to be converted to a continuous set. To do so, an hourly value was generated through gridding between existing data points by using linear interpolation as described in Figure 3.2a and b. Gridding by linear interpolation was chosen over other gridding methods such as zero-order hold because changes in the grinding circuit output were believed to occur gradually instead of abruptly. Once hourly values had been generated for all discrete data sets, these were averaged over 8 hours (one shift), as described in Figure 3.2c and d.

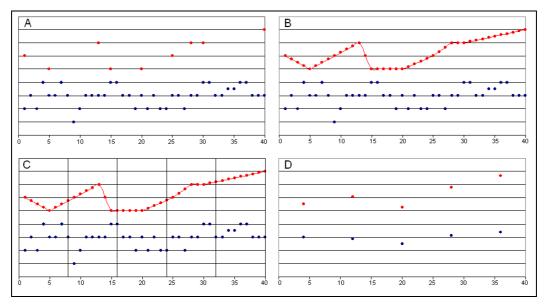


Figure 3.2 Conversion of discrete (red) to continuous data set using linear interpolation of existing points (A and B), and aligning with other continuous data set (blue) by averaging values within a certain time frame (C and D)

### 3.3.2 *Time lag*

Another difficulty faced when trying to estimate the Blaine number and PSD of a particular batch of tested green pellets is that the green pellet strength and the fineness of the feed are measured at different stages within the pelletising process. To ensure production during grinding circuit downtime, ground pellet feed leaving the grinding circuit is not directly mixed with bentonite and water but stored in silos. The silo retention time was estimated at roughly 8 hours (Scheepers, 2010). To account for this delay in the process, the collected data set of both Blaine number and PSD needed to be offset for +8 hours.

# 3.4 MODEL SELECTION & RESULTS

After the data had been filtered, gridded and structured, three different modelling techniques were used to test how well the green pellet strength of the individual grinding stages could be expressed in the variables ore fineness, water content and bentonite content.

In order to study which representation of feed fineness would correlate better, the correlation between green pellet strength and the feed fineness represented by Blaine and PSD were performed separately.

### 3.4.1 Multiple Linear Regression

The correlation between green pellet strength and ore fineness, water content and bentonite content was first tested by Multiple Linear Regression (MLR) using Microsoft Excel Software. The outcome of this correlation analysis is listed in Table 3.2.

Table 3.2 Correlation between Pellet strength, ore fineness, moisture content and bentonite content based on IJmuiden pellet plant process data using MLR.

		$R^2$
Grinding Circuit 1	Blaine	0.21
	PSD	0.27
Grinding Circuit 2	Blaine	0.21
	PSD	0.24
Grinding Circuit 3	Blaine	0.15
	PSD	0.22

As can be seen in Table 3.2, none of the values for R<sup>2</sup> are higher than 0.3. This means that, using MLR, only 30% of the variation within the values of green pellet strength could be explained by variations in the values of feed fineness, moisture content and Bentonite content. Based on this it can be stated that there is only a very weak correlation between the four variables.

### 3.4.2 Partial Least Squares & Neural Networks

In order to see whether other modelling techniques would be able to find a stronger correlation, green pellet strength was described as a function of moisture and bentonite content and feed fineness from grinding circuit 1 using the Partial Least Squares method (PLS) and using Neural Networks. During these analyses, the fineness of the feed was expressed in both Blaine number and PSD. The results of these analyses are listed in Table 3.3.

Table 3.3.Correlation between Pellet strength, ore fineness, moisture content using PLS and Neural Networks

	$R^2$
Partial Least Squares	0.15
Neural Networks	0.19

As can be seen in Table 3.3, the other two modelling techniques also found a weak correlation between the four variables.

### 3.5 DISCUSSION

As shown in section 1.4, data analysis of the process data from the IJmuiden pellet plant was unable to confirm that any of the factors of influence described in the literature where indeed of influence on green pellet strength. Several reasons for the weak correlation between the measured pellet strength and estimated ore fineness, moisture content and bentonite content were suggested and, where possible, verified.

### 3.5.1 Measuring errors

One explanation for the weak correlation found during the data analysis could be that of erroneous measurements. The error of the data points within the initial data sets used in a correlation study, such as the one described in this chapter, will automatically introduce an error in the outcome of this analysis.

Especially the measurement of Blaine number and that of green pellet strength were believed to contain relatively high measuring errors caused by the used equipment and the fact that these measurements are relatively operator dependent.

#### 3.5.2 Unrepresentative data

Another factor that could have affected the accuracy of the data analysis is how well certain measurement reflects the actual value. How representative a measurement is, is affected by the measuring system itself, but also by the place of measurements in the process and the frequency of measurement.

#### 3.5.2.1 Point of measurement

The points of measurement within a process are especially important when studying the correlation between quantities that are measured at different stages of the process. When

studying the correlation between a value A, measured at point 1 and a value B, measured further down the process in point 2, any changes that occur to value A after it has been measured will have a negative influence on the correlation. In other words, the measured value of variable A in point 1 is than no longer representative for the value of A in point 2 because of unmeasured changes in the value of A that have occurred after point 1.

The values for Blaine and Particle Size distribution used in this data analysis were measured at the end of the grinding stage. These values for feed fineness were then correlated to green pellet strength which is measured further down the pelletising process. This means, that any changes to the fineness of the feed that occur after leaving the grinding circuit are left unmeasured.

At IJmuiden, the ground ore feed leaving the grinding circuits is stored in silos. From experience it was known that under emptying, the fineness of the ore feed would increase drastically. This phenomenon was studied and confirmed (Scheepers, 2010). They both contributed this phenomenon to the segregation of the small and large particles within the feed, resulting in areas of relatively high and low fineness.

These variations in ore feed fineness has introduced an additional error to the data analysis.

### 3.5.2.2 Measurement Frequency

Another factor which has an effect on how representative a certain measurement is, is the frequency at which this measurement is done.

Processes such as grinding and balling circuits are known to have a self regulating nature, resulting in a periodic variation called "surging" (Scheepers, 2010). In order to do a representative measurement of the output of such processes, the sampling frequency needs to be chosen at least a number of times smaller than the frequency of this natural variation in order to obtain a good average value of the measured quantity. Also, the larger the amplitude of this surging is, the larger the error in the measured average value.

In order to study the variation of the fineness of the grinding circuit, the Blaine number was determined of samples taken from one of the grinding circuit outputs every 20 minutes for approximately 6 hours. During this 6 hour period, no changes to the grinding circuit's settings were made. The results can be seen in Figure 3.3.

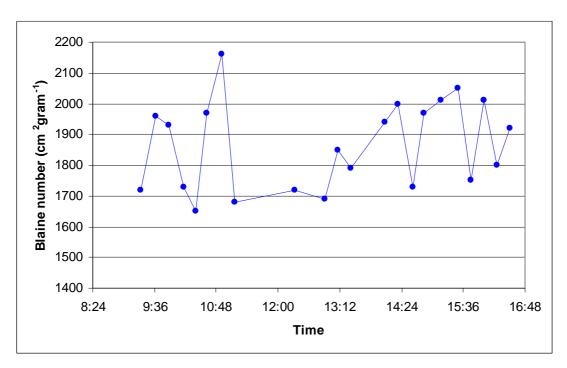


Figure 3.3. Variations in Blaine of Grinding Circuit Output on small time scale. (Grinding circuit 3; October 13, 2010)

In Figure 3.3, it can be seen that a large variation exists in the fineness of the grinding circuit output. As described, the Blaine number of the grinding circuit was sampled every 10 minutes and collected. Roughly every 8 hours this collection sample is analysed. This method should give a good value for the average fineness of the grinding circuit output. However, due to the large variation shown in Figure 3.3, this average value is believed to have a considerable standard error.

### 3.5.3 Invalid Assumptions & missing variables

During this correlation study, certain techniques were used and assumptions were made in order to estimate the properties of a tested batch of green pellets as accurate as possible. However, by using these techniques and assumption however an additional error was introduced.

Especially the generation of hourly values from the discrete data sets through linear interpolation and the estimation of the retention time of the ground feed in the silo's prior to blending are believed to have introduced a considerable error.

In addition, it should be mentioned that only the variables that were believed to have the largest effect on green pellet strength and that are actually measured were taken into account in this data analysis. Minor factors of influence such as ore type, water chemistry and bentonite chemistry have been left out.

### 3.6 CONCLUSION

No strong correlation was found between green pellet strength and feed fineness, water content and bentonite content after data analysis of IJmuiden pellet plant process data. Three different mathematical methods all failed to find any strong correlation. The weak correlation found is believed to be the result of a combination of errors:

- 1) Because the exact Blaine value and particle size distribution of the feed of a certain batch of tested pellets was unknown, these properties needed to be estimated from measurements of these values at different places in the process.
- 2) The data set manipulation which had to be performed and the assumptions that had to be made in order to be able to do these estimations are believed to have introduced a considerable error in the resulting data set.
- 3) Additional errors were introduced by the measuring systems used and unmeasured process variations.

# 4 EXPERIMENTAL DESIGN

### 4.1 BACKGROUND & SCOPE

After completing a literature review on green pellet strength as well as a process data analysis of the IJmuiden pellet plant, discussed in respectively chapters 2 and 3, a thorough understanding was formed on the mechanisms responsible for iron ore green pellet strength. Thereby, Stage 1 of the project had been completed.

The knowledge gained during Stage 1 could now be used in Stage 2, of which the goal was defined as:

"Indicating methods to increase the production of pellets of defined quality at the current grinding capacity possible at IJmuiden."

As mentioned before in Chapter 1, the main bottleneck in terms of total production of the IJmuiden pellet plant is the grinding stage. Therefore, any increase in grinding circuit output, that does not compromise pellet quality, is believed to proportionally increase overall pellet production.

The fineness of the grinding circuit output is currently tested by measuring the Blaine number which is an indication of the available surface within the grind. Through experience, ground pellet feed with Blaine values higher than 2000 cm<sup>2</sup>/gram are considered suitable for balling. Because the IJmuiden grinding circuit already operates at its maximum power capacity, the only possibility to increase the grinding circuit output is through optimisation of the grinding strategy and/or the grinding itself. This study will only focus on increasing the grinding circuit output by optimisation of the grinding strategy.

The pellet feeds of the IJmuiden pellet plant are blends of different ores which are grinded in a closed grinding circuit. The compositions of these blends are based on several factors such as availability, price and iron content. Properties that affect the grinding of the ores, such as

initial fineness and grindability, however, are not taken into consideration in the selection of ores.

Given these facts a possible method to increase production was to reduce the required energy to grind a blend of ores to a desirable fineness by over grinding the ores with a high grindability and leaving the harder ores coarser.

Such a grinding strategy would however influence the individual fineness of the different ores within a blend. These differences in individual ore fineness have no effect on the overall composition of the blend in terms of weight ratio and volume ratio; but will have a large impact on the ratio of available surface between the different ores in the blend.

Literature study showed that not only the particle size distribution of the feed, but also certain surface related properties such as roughness and wettability are believed to have an influence on the capillary forces within a green pellet. As these forces are partially responsible for the green pellet strength, changes in individual ore fineness, and therefore overall roughness and wettability, could have an effect on green pellet compressive strength.

From the literature review it was also found that the contribution of capillary forces to overall binding force of pellets balled with a bentonite binder appeared to be strain rate dependent. As the strain rate increases, the viscous forces of the binder become more dominant and the relative contribution of capillary forces to the pellet strength decreases.

Based on the literature review, a theory was formed on the way individual ore fineness could influence the compressive strength of green pellets. This theory is summarized in the following hypothesis:

"The <u>Compressive Strength</u> of green pellets balled with the use of a <u>Bentonite Binder</u> from a pellet feed with a <u>Fixed Particle Size Distribution</u> consisting of a <u>Blend</u> of several iron ores, is influenced by the <u>Individual Fineness of Ores</u> within- and the overall <u>Composition</u> of the blend. However, a Critical Strain Rate exists above which both variables loose this influence"

In order to test this hypothesis an experimental method was designed.

### 4.2 EXPERIMENTAL METHOD

In order to test the hypothesis an experimental program was designed which can be divided into two separate experiments.

The strength and plasticity of batches of pellets that were balled from an actual IJmuiden pellet feed with a fixed bentonite content but with different moisture content were measured to study the correlation between moisture content, plasticity and pellet strength. Of one of these batches, the compressive strength was determined at five different load speeds to study the influence of strain rate on green pellet strength and plasticity.

The experimental method used to investigate the influence of variations in individual ore fineness within a fixed blend involved the balling and testing of green pellets from different feeds. Three different feeds were prepared by blending three different types of ore. These feeds were prepared in such a way that the overall particle size distribution as well as the overall blend composition was kept the same for all three blends. However, the individual ore finenesses of the different ores within the feed were varied between the three.

After preparation, each feed was balled into pellets using a lab scale pelletising disc under the addition of a fixed amount of water and bentonite. After balling, the compressive strength and plasticity of 25 of the pellets were determined at different load speeds. In addition, the periodic property and saturation level of the pellets was measured after each test. This procedure was repeated three times for each of the feeds.

During balling, all variables that were believed to have an influence on the compressive strength had to be kept as constant as possible. For this reason the balling procedure had been standardized.

In addition, an actual pellet feed used in the pellet plant with the same particle size distribution but a different composition from the prepared feeds was balled and tested following the same procedures.

During the experiments only the individual fineness of the three ores were varied between the feeds, while all other variables of influence on green pellet strength were kept constant. Therefore, any differences in strength and/or plasticity in green pellets balled from different feeds and tested at the same strain rate can directly be attributed to differences in surface related ore properties such as roughness, shape and wettability.

The influence of strain rate was studied by measuring the compressive strength and plasticity of each batch of green pellets using two different load speeds.

# 5 EXPERIMENTAL PROCEDURES

### 5.1 INTRODUCTION

As described in section 4.2, the correlation between moisture content, plasticity and pellet strength was studied using test data from other experimental work which was done to investigate the ballability of a certain pellet feed called MH2630. Also, pellets that were balled in the light of this other project were used during this work to study the influence of strain rate on pellet strength and plasticity. The methodology and equipment used during these tests are discussed in section 5.5.

The experimental work performed to study the influence of individual ore fineness within a blend on green pellet strength and plasticity involved the preparation of different feeds, followed by the balling and testing of pellets from these different feeds. The materials, equipment and procedures used in all steps of this experimental work are discussed in sections 5.2 to 5.5.

### 5.2 MATERIALS USED

The influence of variations in individual ore fineness within a blend was studied using three ores of a different nature that are commonly used in IJmuiden pellet feeds. An actual pellet feed from the IJmuiden pellet plant was used as a reference.

### 5.2.1 Ores in A, B and C-feeds

As described in section 4.2, three different feeds were prepared of three different ores that had a fixed overall blend composition and particle size distribution, but were different in terms of fineness of the individual ores within the blend. The ores used to make up this blend had to have very different properties. Therefore three different ore types were used for the feed preparation.

The ores used were a magnetite ore and two different hematite ores. The two hematite ores were different in the fact that one was build up from coarse crystalline primary Hematite with specular particles while the other hematite was a so called banded ore, consisting of bands of silicates, hematite, goethite and limonite. The exact names and origin of the ores are confidential and are named by ore type; Magnetite, S-Hematite and R-Hematite. The differences in ore properties are further discussed.

#### 5.2.1.1 Initial Fineness

The fineness at which the ores were delivered at IJmuiden was very different. The initial finenesses of the three ores was measured using screening for the fraction larger than  $250\mu$  and laser diffraction (Sympatec<sup>TM</sup>) for the smaller sizes. Because screening and laser diffraction measure respectively weight and volume percentages, the two methods would not form a representative PSD curve when combined. Therefore, the values in the region between  $174\mu$  and  $250\mu$  are left out. For each ore the PSD was determined three times. The cumulative particle size distributions of the raw ores used can be seen in Figure 5.1.

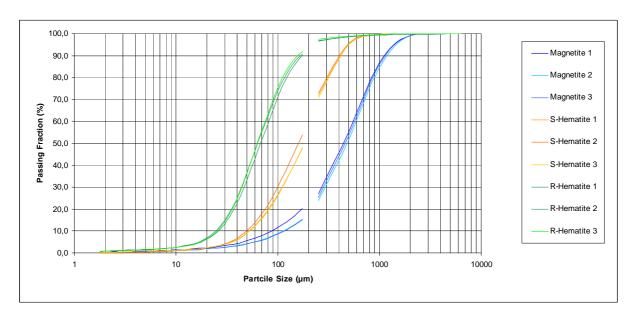


Figure 5.1 Raw ore finenesses of the Magnetie, S-Hemate and R-Hematite ores.

In Figure 5.1 it can be seen that the raw fineness of the three ores is somewhat different, with the magnetite being the most coarse.

### 5.2.1.2 Chemical composition and wettability

The main difference between the three ores was their mineralogy. In previous work, the mineral proportions of the Magnetite and S-Hematite ore had been determined. In the same study a hematite ore that is believed to be very similar to the R-Hematite ore was also analysed. The results of this study are shown in Table 5.1.

Table 5.1 Mineral composition of ores used in feed preparation

		Magnetite	S-Hematite	R-Hematite
Hematite	Fe <sub>2</sub> O <sub>3</sub>	0.0	86.4	90.6
Magnetite	$Fe_3O_4$	96.4	5.0	0.7
Goethite	FeO(OH)	0.0	0	5.6

In this study the contact angle of the used ores have not been measured directly. However, from literature, it is known that the contact angle of magnetite, hematite and goethite are very different (Iveson et al., 2004; ) The contact angles of pure magnetite, hematite and goethite derived from earlier work are listed in Table 5.2.

Table 5.2 Contact angles of pure minerals derived from literature (Iveson et al. 2004; Potapova et al., unpublished)

	Contact angle	
	(°)	
Hematite	53 (± 10)	
Magnetite	23 (± 15)	
Goethite	8 (± 10)	

It should however be mentioned that the contact angles of especially magnetite ores is known to vary due to chemical treatment during concentration. Contact angles as high as 50<sup>0</sup> have been measured for magnetite concentrates (Potapova et al., unpublished). The measured value of contact angle is also know to vary significantly between different measuring methods.

### 5.2.1.3 Particle Shape and Roughness

Differences in particle shape and roughness between the three ores after grinding was studied using Electron Microscopy. In Figure 5.2 the three different ores ground to similar finesses using Secondary Electron Imaging (SEI) are shown in Figure 5.2.

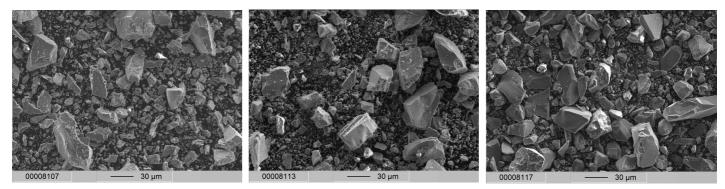


Figure 5.2 EMP SEI images of ores used in ABC feeds preparation ground to similar fineness.

Magnetite (left), S-Hematite (center) and R-Hematite (right)

It can be seen that there is not much difference in particle shape between the three ores when ground. In the R-Hematite ore however much more rough and porous particles were detected.

### 5.2.2 MH2547

The MH2547 ore feed was used as a reference during the experimental work on the influence of individual ore fineness. All feeds prepared were constructed in such a way that they would have the same particle size distribution as the MH2547 ore feed. The particle size distribution of the MH2547 ore feed is shown in Figure 5.2.

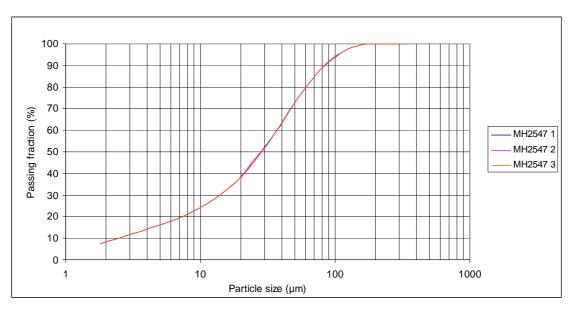


Figure 5.3 PSD of MH2547, 3 measurements

The MH2547 is a blend of seven different ores. All three ores used to build up the A, B, and C feeds are present in MH2547. Of the other four ores, three are very similar to one of the ores used in the A,B and C feeds. The seventh ore however is a more weathered ore, consisting mostly of porous goethite. This ore has a high particle roughness and inner particle porosity as can be seen in Figure 5.4.

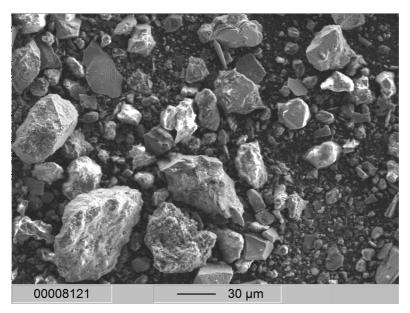


Figure 5.4 EMP SEI Image of weathered ore in MH2547 feed

Most ores used at IJmuiden are similar to one the three ores used in the A,B and C feeds or the weathered ore which is present in MH2547. The blend composition of MH2547 expressed in these four ores can be seen in Table 5.3.

Table 5.3 Blend composition MH2547

Ore type	Fraction	
	(%)	
Magnetite	20.1	
S-Hematite	5.2	
R-Hematite	66.8	
Goethite	8.4	

### 5.3 FEED PREPARATION

As described in section 4.2, in order to test the hypothesis, three different feeds with exactly the same blending composition and particle size distribution, but with different individual ore fineness needed to be prepared. In this work, the MH2547 pellet feed was chosen as a reference. Therefore, the particle size distribution of all prepared feeds was kept the same as that of MH2547, which is shown in section 5.2.

The blend composition was chosen at 33.3% Magnetite, 33.3% S-Hematite and 33.3% R-Hematite based on weight for each of the three feeds. Because of their different densities this translates to 33.1 vol.% Magnetite, 33.3 vol.% S-Hematite and 33.6 vol.% R-Hematite.

While keeping the PSD and blend composition fixed at these values for all three feeds, the fineness of the individual ores was varied between the three different feeds.

In order to achieve this, a method to construct a given PSD needed to be designed that would allow manipulation of the ratio between the three different ores within specific size fractions of that particle size distribution.

For particle size distributions made up by relatively large particles, this could easily be done by simply mixing specific screening fraction of all three ores at different ratios. Practical screening however is limited by a minimal screen size of approximately 40µm. Given the particle size distribution of MH2547, screening and mixing was therefore not an option and another method needed to be developed.

The method that was eventually used involved grinding all three different ores to a range of finesses which were then mixed in different ratios for each feed. Hereby the individual ore finesses could be varied between the feeds while having the same overall particle size distribution and blend composition. These two stages of the feed preparation are discussed in detail.

#### 5.3.1 Grinding

The first step of the feed preparation was to grind all three ores to a range of five different finenesses. Grinding was done using a lab scale ball mill known as a Bond mill which is discussed in section 5.3.3.

This type of mill is commonly used to determine the grindability of individual ores and ore mixtures (Karra, 1981). In these types of studies, the difference in fineness between two grinds is based on the difference in the 80 percentile, the  $D_{80}$ . Therefore, during the feed preparation, the  $D_{80}$  was used as indicate the fineness of the grinds.

The number of balls of a defined weight in the mill was fixed during all the grinding. Therefore, for a fixed weight and fineness of the input, the finesses of a grind was assumed to be only effected by the number of revolutions.

The range of the finesses of the grinds of each ore had to be chosen in such a way that some grinds were finer than the MH2547 reference and some were coarser. The MH2547 pellet feed was found to have a  $D_{80}$  of around  $60\mu m$ .

For each of the ores, the number of revolutions needed to reach certain finenesses for a given input weight were determined empirically by grinding each ore at a range of revolutions.

The particle size distribution of all grinds was determined using laser diffraction (Sympatec<sup>TM</sup>) which has a measuring range up to  $350\mu m$ . Therefore the range of finesses that could be used was limited by the fact that all particles needed to be smaller than  $350\mu m$  in order to be still measurable.

For the finer grinds, the range of finenesses was limited by the fact that in very fine grinds particles tended to stick to the grinding balls, causing a cushioning effect. This cushioning effect would affect the grinding, making the fineness of very fine grinds less predictable. Given these limitations, it was determined that the different ores were to be ground to grinds

Given these limitations, it was determined that the different ores were to be ground to grinds with  $D_{80}$ 's between 40µm and 120µm.

In Figure 5.5, the PSD's and the  $D_{80}$ 's of the used grinds for all three ores that were eventually used during feed preparation are listed.

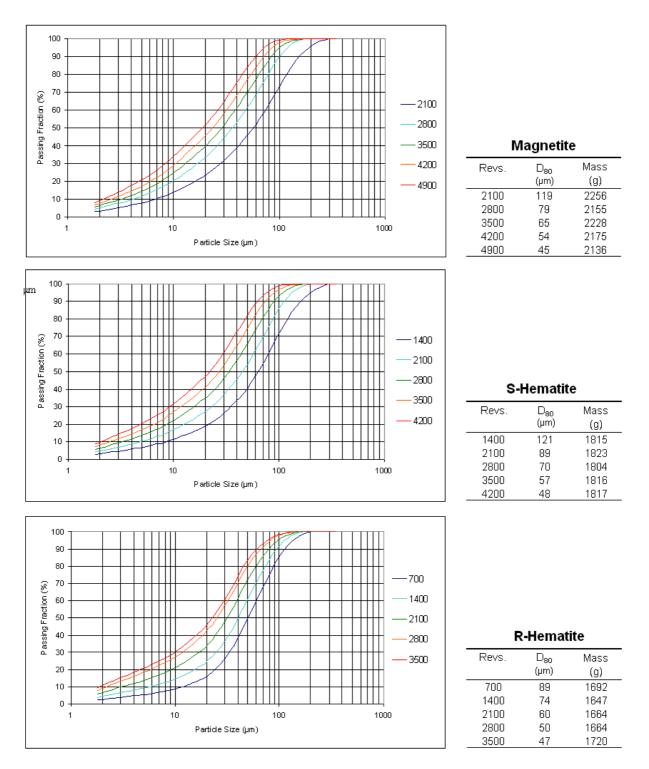


Figure 5.5 PSD's and D<sub>80</sub>'s of grinds used during feed preparation

As can be seen in Figure 5.5, the weight of the input material was not constant between the grinds. The large differences in input weight between the different ores can be attributed to

the fact that the input volume was kept constant between the ores at approximately 700 cc. Due to variations in initial ore fineness and specific gravity, this led to variations in input weight.

The input weight was limited by of the dimensions of the bond mill to a maximum of approximately 2.5 kg.

For the feed preparation, of all grinds, much more material was needed than was produced in one grinding run. Therefore the grinds needed to be reproduced. The reproducibility of the grinding runs was tested by comparing the grinding product of four separate grinding runs of the same weight of the Magnetite ore at 4900 revs. The results are shown in Figure 5.6.



Figure 5.6. Reproducibility of the Magnetite 4900 revs. grind.

As can be seen in Figure 5.6, the grinding runs proved to be very reproducible. All other grinds were assumed to have the same reproducibility.

### **5.3.2** *Mixing*

After the different ores had been ground to the desired finesses, three different feeds were prepared by blending these grinds. For all feeds the overall blend composition was kept constant at 33.1 vol.% Magnetite, 33.3 vol.% S-Hematite and 33.6 vol.% R-Hematite.

Through iteration using Microsoft<sup>®</sup> Excel, the ratios between different grinds for each ore could be varied in such a way that the weighted sum would best fit the particle size distribution of MH2547 pellet feed. The parameters used to describe the particle size distribution during this iteration were the  $<10\mu m$  and  $<25\mu m$  fractions and the D<sub>80</sub>. During iteration the maximum error between the generated feeds and the MH2547 PSD was 4%.

In each of the three feeds another ore had to make up a relatively large percentage of the surface area within the feed. To accomplish this, for each of the feeds, only the finest grind of another ore was used in the iteration. By doing so, in each feed, another ore would make up most of the fines. The feeds that were eventually prepared are shown in Figure 5.7.

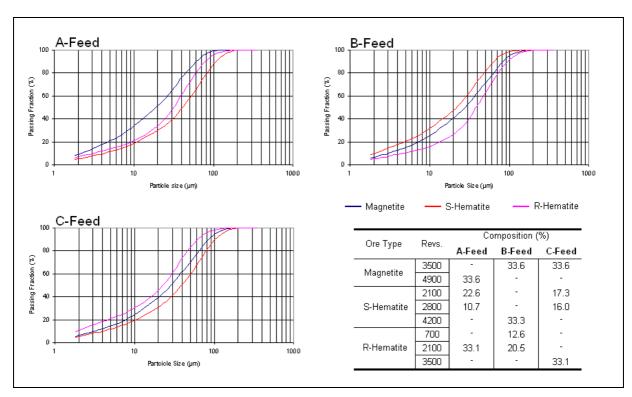


Figure 5.7. Blend composition and individual ore finesses for all three blend used in experiments.

After the blending ratio between the different grinds was determined for each of the three feeds, the feeds were composed. In order to homogenise the feeds after composition, the feeds were treated in a Turbula<sup>TM</sup> mixer for five minutes. The maximum input of this mixer was 750g. Therefore, the feeds were prepared in batches of 750g. For each balling experiment, 4.5kg of feed was needed. One balling feed was therefore made up from 6 of these small batches. As an additional check, the particle size distribution of each of the prepared feeds was determined using laser diffraction. The particle size distributions of the used feeds are shown in Figure 5.8.

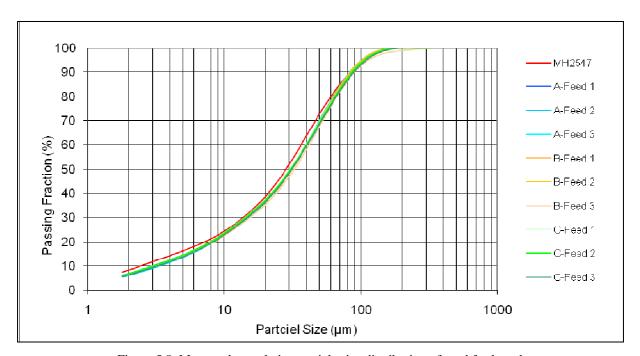


Figure 5.8. Measured cumulative particle size distribution of used feeds and MH2547.

As can be seen in Figure 5.8, the particle size distributions of all feeds were very similar. The particle size distribution of MH2547 seems to be slightly finer, but the differences are minimal.

# 5.3.3 Equipment used

The main equipment used during the grinding stage of the feed preparation were the Bond mill and the Turbula<sup>TM</sup> mixer.

The Bond mill that was used to prepare the different grinds is shown in Figure 5.9. The mill has a diameter and length of both 12" and a fixed revolution speed of 70 rpm. The amount of steel balls with a specific diameter was kept constant for all grinding runs and is shown in Figure 5.9. After each grind the ball mill was emptied and cleaned.



Ball Diameter (")	Number of Balls
1.45	41
1.17	65
1	10
0.75	65
0.61	70

Figure 5.9 Bond ball mill used in feed preparation

The Turbula<sup>™</sup> powder mixer used is shown in Figure 5.10. The user manual



Figure 5.10 Turbula© powder mixer used during feed preparation.

The user manuals of both the bond ball mill and the Turbula<sup>TM</sup> powder mixer have been documented earlier (Tata Steel, 2010 (1); Tata Steel, 2010 (3)).

# 5.4 BALLING

Each of the different feeds was balled into pellets using a lab scale pelletising disc under the addition of water and Bentonite. The balling procedure was standardized and kept the same in all balling experiments. The balling procedure was based on procedures described in earlier work (Sportel, 1995) and consisted of three different stages; the addition of bentonite and water to the feed, seed pellet production and the production of pellets.

#### 5.4.1 Bentonite and Water Addition

For one balling run, 4.5kg of dry feed was needed. Before balling, water and Bentonite were added to the feed. During all experiments, the percentage of added Bentonite and water were kept constant at respectively 0.5% and 7.0 % based on the weight of the dry feed. The feed was first mixed with the Bentonite using a small Lödige<sup>™</sup> Mixer. After three minutes the water was gradually added during another 3 minutes of mixing.

After mixing the mixer was emptied and the moisture content of the mix was determined by measuring the difference in weight before and after drying.

Not all material was retrieved after mixing. Roughly 20% of the material stayed behind in the mixer. The material that was retrieved from the mixer was weighted and 25% was used to produce so called "seed pellets".

### 5.4.2 Seed Pellet Production

The material available for seed pellet production was approximately 1 kg. After the pelletising disc was slightly wetted using a regular water spray, about 400 g of material was brought onto the disc. This material was balled under occasional addition of water using a water spray.

After 3 minutes of balling, the disc was emptied and the fractions 5mm-6.85mm and 3.65mm-5mm were screened off. The 5mm-6.85mm fraction was kept aside and the 3.65mm-5mm fraction was returned onto the disc. This fraction was balled further under alternating addition of water and feed. This was repeated every 3 minutes until no more feed was left.

After all feed was used, the entire 5mm-6.85mm fraction was returned onto the disc and balled for two minutes without any addition of water and/or feed in order to compact. After this the 5mm-6.85mm fraction was again screened off. This fraction was used as "seed pellets" during further pellet production.

#### 5.4.3 Pellet Production

After seed pellet production the pelletising disc was cleaned. After this, 250g of seed pellets was brought onto the disc. Under the alternate addition of water and feed, seeds were formed into pellets.

During balling on a pelletising disc, pellets tend to grow at a different rate, resulting in a certain distribution in size. In order to keep this distribution as narrow as possible after 6 minutes of balling the fraction <6.85mm was screened off; the oversize was returned onto the disc. The same procedure was performed after 15 minutes at a screening size of 10mm.

After 20 minutes of balling the pellets were balled for another 2 minutes without any addition of water and/or feed. After this the 12mm-12.5mm fraction was screened off. This fraction was used for testing.

# 5.4.4 Equipment used

During the balling stage, the main equipment used was the lab scale Lödige™ mixer and the balling disc.

# 5.4.4.1 Balling disc

The settings of the balling disc used during all balling experiments were fixed in order to keep the balling process as constant as possible. The disc and its properties can be seen in Figure 5.11.



Disc diameter	(mm)	400
Disc depth	(mm)	110
Disc angle	(°)	47
Rotation speed	(rpm)	15

Figure 5.11 Lab scale balling disc used during balling

# 5.4.4.2 Lab scale Lödige™ mixer

The lab scale Lödige<sup>TM</sup> mixer used during this study is shown in. The mixer was operated following work instruction which had been formulated earlier (Tata Steel, 2010 (3)).





Figure 5.12 Lab Sclae Lödige $^{\text{TM}}$  mixer used during experiments ouside (left) inside (right).

# 5.5 PELLET TESTING

After balling a range of properties of the 12mm-12.5mm fraction of the produced green pellets was tested using different types of equipment. All tests were performed within 30 minutes after balling to prevent the drying of the pellets to have an influence on pellet strength.

# 5.5.1 Green Pellet Compressive Strength and Plasticity

The green pellet strength and plasticity were measured using a common USC test. The instrument and the pellet strength and plasticity calculations are further described in section 5.5.3. This instrument is capable of testing the compressive strength of pellets at both constant load rates (N/s = constant) as constant load speeds (m/s = constant).

The tests on the MH2630 pellets balled with different moisture and bentonite content were performed at a constant load rate of 2.7 N/s, which is the ISO standard.

As mentioned in section 5.1, loading tests was performed on green pellets from other MH2630 pellets to further investigate the influence of strain rate on pellet strength and plasticity. These tests were performed at load speeds of 0.01, 0.05, 0.1, 0.25 and 0.5 mm/s.

During the experiments to study the effect of strain rate on green pellet compressive strength and plasticity, the pellets were tested at two different load speeds. The ISO standard load speed used to test green pellet strength is 0.25 mm/s. Based on this ISO value and the practical limitations of the UCS instrument, the two selected load speeds for the test were 0.5 mm/s and 0.05 mm/s. At each load speed 25 green pellets of each formed batch were tested.

# 5.5.2 Additional Testing

The experiments were designed in such a way that all variables that would have an effect on green pellet strength and plasticity other than the individual ore fineness were kept constant for all formed pellets. As any variations in these variables could lead to incorrect conclusions, the moisture content, saturation and porosity of a sample of 25 pellets from each batch of

formed pellets was tested. The methodology used has been documented before (Tata Steel, 2011). In addition, the microstructure of formed pellets was studied using EMP/SEM and Micro CT-scan.

#### 5.5.3 UCS Test Instrument

During the tests performed on the green pellets, several instruments and methods were used. In the case of green pellet strength and plasticity, the raw test data needed to be processed in order to be able to draw valid conclusions from it. The instruments and steps of this data processing are discussed below.

The Universal Compression Tests Instrument that was used to determine the strength and plasticity of green pellets is a Lloyd LR-10k. This semi- automatic instrument is capable of lowering a piston from a fixed height onto a pellet at a constant load speed or increasing force. The exerted force on the piston as a function of the travelling distance is recorded and stored as a graph. In addition, key values in the graph—such as the Max Force and the extension of the piston at first contact, at a force of 1N and at breakage were automatically collected and reported separately. A typical output from the UCS instrument with these four collected values highlighted is shown in Figure 5.13.

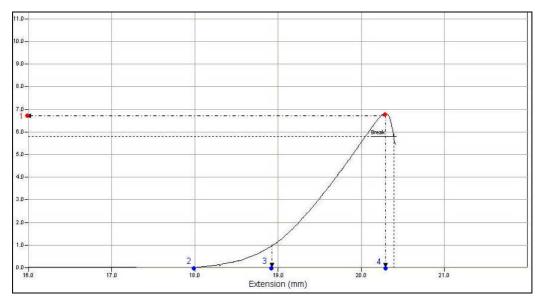


Figure 5.13 Typical UCS test Force-extension output curve.

From these four values and the properties of the instrument, the compressive strength and plasticity were calculated as described in the following sections.

# 5.5.3.1 Green Pellet Compressive Strength

Traditionally, at IJmuiden, the green pellet strength is reported as the force (N) at which a pellet breaks (point 1 in Figure 5.13). As green pellets do not all have the exact same diameter, the stress in a green pellet at a given load is not constant.

To compensate for these differences in diameter, in this work, the compressive strength of green pellets was described by the maximum yield stress a pellet can withstand before breaking. The diameter was calculated by subtracting the extension at first contact (Point 2 in Figure 5.13) from the max extension of 300mm. The max yield stress is calculated from the Max Force using Equation 5.1.

$$\sigma_{\text{max}} = \frac{4F_{peak}}{\pi D^2_{pellet}}$$
 (Equation 5.1)

With,

 $\sigma_{max} = Max Yield Stress [Nm^{-2}]$   $F_{max} = Max Force [N]$   $D_{nellet} = Pellet Diameter [m]$ 

### 5.5.3.2 Green Pellet Plasticity

At IJmuiden, the deformation of a green pellet at a load of 1 N is reported as the green pellet plasticity, known as the  $d_{100}$  (point 3 in Figure 5.13 ). This value only describes the non linear part of the force-deformation curve.

Another description of the green pellet plasticity is given by the steepness of the linear part of the force-deformation curve. This description of plasticity is commonly used in present literature on green pellet properties (Forsmo et al., 2007).

In the force-deformation ratio however, pellet diameter still has an influence on the measurement. This can be corrected by transforming the Force-deformation curve into a

stress-strain curve, because the relationship between stress and strain is independent of dimension. The transformation form Force into stress was shown before in Equation 5.1. The transformation from deformation u, to strain  $\varepsilon$ , is shown in Equation 5.2.

$$\mathcal{E} = \frac{u}{D_0} \tag{Equation 5.2}$$
 With, 
$$\epsilon = \text{Strain} \qquad \text{[-]}$$
 
$$u = \text{Deformation} \qquad \text{[m]}$$
 
$$D_0 = \text{Initial pellet Diameter} \qquad \text{[m]}$$

The steepness of the stress-strain curve is known as the Young's Modulus and is a material constant. In this study the Young modulus was used to describe the plasticity of green pellets. Given Equation 5.1 and Equation 5.2, this Young's Modulus E was calculated using the  $d_{100}$ , the pellet diameter, the deformation at breakage ( $u_b$ ) (Point 4 in Figure 5.13) and the max yield stress ( $\sigma_{max}$ ) by:

$$E = \frac{\Delta\sigma}{\Delta\varepsilon} = \frac{\left[\frac{(F_{peak} - 1)}{\frac{1}{4}\pi D^2}\right]}{\left[\frac{u_b - d_{100}}{D}\right]} = \frac{4(F_{peak} - 1)}{\pi D(u_b - d_{100})}$$
 (Equation 5.3)

With,

E	=	Young Modulus	$[Nm^{-2}]$
σ	=	yield stress	$[Nm^{-2}]$
3	=	strain	[-]
$F_{\text{max}}$	=	Max Force	[N]
$D_0$	=	initial pellet diameter	[m]
$u_b$	=	deformation at breakage	[m]
$d_{100}$	=	deformation at load of 1N	[m]

### 5.5.3.3 Strain Rate

The influence of strain rate on green pellet strength was studied by performing UCS tests at different load speeds. From the USC output data, the total loading time ( $t_{load}$ ) between initial start of compression and breakage can be calculated. As a check, the load speed was calculated by dividing the deformation at breakage by the loading time. The load speed showed small variations. These variations are believed to be caused by the fact that the UCS instrument has to calculate the force needed for a certain load speed based on the plasticity of the sample. For the non-linear part of the force-deformation curve as seen in Figure 5.13, this causes the instrument to miscalculate the needed force for a certain load speed.

The strain rate was calculated from the pellet diameter and calculated load speed using:

$$\dot{\mathcal{E}} = \frac{\mathcal{E}_{breakage}}{t_{...}} \tag{Equation 5.4}$$

With,

 $\dot{\mathcal{E}}$  = strain rate [s<sup>-1</sup>]  $\varepsilon_{breakage}$  = strain at breakage [-]  $t_{load}$  = total loading time [s]

# 6 EXPERIMENTAL RESULTS & DISCUSSIONS

In this chapter the results of the experimental work as described in chapter 4 and 5 are presented and discussed.

The results from tests performed on green pellets balled from the MH2630 blend were used to study the influence of moisture and strain rate on green pellet strength and plasticity.

The insights that were acquired during this work helped analysing the results of the tests performed on the pellets formed from the MH2547 blend and the three different prepared feeds in order to study the influence of individual ore fineness on green pellet strength.

In addition, the microstructure of green pellets balled during the experiments is analysed.

# 6.1 WET COMPRESSIVE STRENGTH AND PLASTICITY

During ballability tests on MH2630, the blend was balled into pellets under the addition of varying amounts of bentonite and water. The max stress of each batch of pellets was tested at a constant load rate of 2.7 N/s using the same equipment as described in section 5.4.3. In addition, the average porosity and saturation of approximately 100 pellets of each batch were measured.

In Figure 6.1, the measured max stress and porosity are shown as a function of the measured moisture content of three batches of green pellets that were balled under addition of the same amount of bentonite (0.5%) but with different moisture contents.

Although all variables within a batch were presumed constant, there is still a lot of variation between the values for max stress within one batch. These variations within a batch are attributed to minor differences in saturation, porosity and bentonite content from the fixed predetermined value between individual pellets.

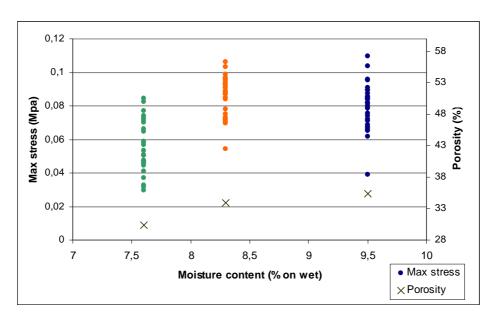


Figure 6.1 Pellet strength and porosity of MH2630 pellets, 0.5% bentonite.

For a fixed fineness of the feed green pellet plasticity is believed to be affected by pellet porosity and saturation (Forsmo, 2007). Therefore, in order to account for these variations between individual pellets, pellet max stress can be described as a function of plasticity. The max stress as a function of pellet plasticity is shown in Figure 6.2.

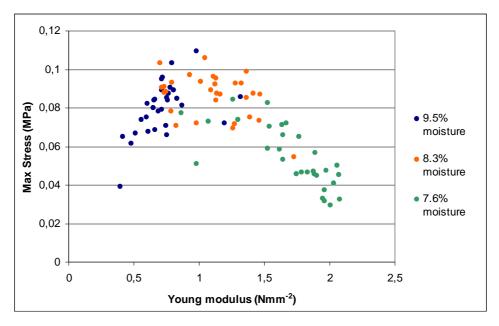


Figure 6.2 Pellet strength as a function of plasticity for MH2630 pellets, 0.5% bentonite.

In Figure 6.2, two regions can be seen in which the relationship between max stress and placticity of the pellets is very different. For relatively wet pellets, the max stress seems to

decrease for more plastic pellets while for relatively dry pellets the max stress increases with plasticity. The pellets formed with a moisture level of 8.3%, seemed to be at the transition point between the two. The relationship between plasticity and max stress for pellets at different saturation levels has been been observed and explained in earlier work (Forsmo et al., 2006).

# **Saturation > 100%**

Pellets are believed to be at their max strength as they are just at the 100% saturation level. If the saturation level is raised above the theoretical 100% level, as is the case for the pellet of 9.5% moisture in Figure 6.1, the pellets porosity will increase in order to adjust back to 100% saturation. This increase in porosity will increase the pellet plasticity and simultaniously decrease pellet strength.

This difference in change in plasticity and pellet strength is also witnessed in the type of breakage. Wet pellets (S>100%) are found to break more ductile where relatively dry pellets tend to act brittle as can be seen in Figure 6.3

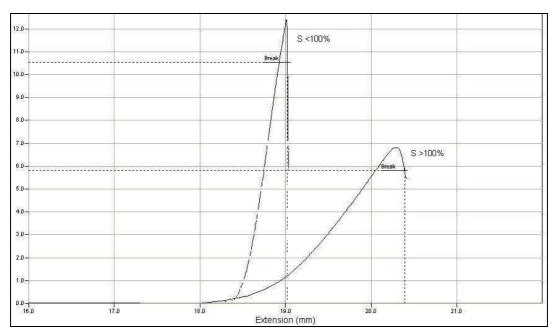


Figure 6.3 Different pellet breakage patterns. Brittle for S<100% (left) and plastic for S>100% (right)

### **Saturation < 100%**

For pellets with moisture contents of S<100% however, Forsmo et al. found that the plasticity remained practically constant. This contradicts the results of this study. Possible explanations for these different findings are discussed in sections 6.2.3 and 6.4.

At IJmuiden, the average strength of 25 pellets is reported as green pellet strength. As mentioned before, there is much variation in pellet strength within single batches. As these variations are caused by differences in pellet properties such as saturation, porosity and bentonite content, the distribution of pellet strength within a testes batch hold valuable information on the consistency of the pelletising process. By only reporting this average value, all additional information on process consistency is lost.

Therefore, during the experiments of this study, tested pellets were treated as single data points.

# 6.2 INFLUENCE OF STRAIN RATE

# 6.2.1 Green Pellet Plasticity

In order to study the influence of strain rate on green pellet strength, the maximum stress and plasticity of pellets balled from the MH2630 feed with an average bentonite content of 0.7%, moisture content of 7.7% and porosity of 31.7% were tested at load speeds of 0.01, 0.05, 0.1, 0.25 and 0.5 mm/s.

The Young Modulus of all tested pellets as a function of the strain rates (calculated from the load speed as described in section 5.5.3.3) at which each pellet was tested is shown in Figure 6.4.

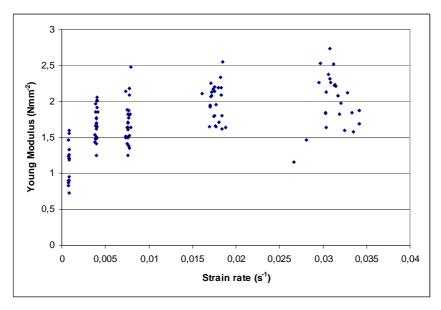


Figure 6.4 Plasticity of MH2630 pellets at different strain rates.

0.7% bentonite, 7.7% moisture.

The plasticity of the tested green pellets seems to be strain rate dependent for the lower four strain rates which means green pellets behave viscoplastic. The variation in the measured values of both strain rate and plasticity in the batch tested at 0.5m/s (strain rates around 0.03 s<sup>-1</sup> in Figure 6.4) make it hard to draw any valid conclusions on the influence of strain rate in that range.

This increase in plasticity at lower strain rates could be explained by the viscosity of the bentonite binder. As strain rate decreases, so does the binder's resistance to flow, resulting in a more plastic pellet. This theory is further backed up by the fact that the plasticity-strain rate curve is steeper for lower strain rates. This correlates very well with the typical shear thinning properties of the bentonite binder.

These findings help further understand the witnessed difference in breakage patterns between over saturated and under saturated green pellets described in section 6.3.1. Wet pellets (S>100%) have a higher porosity making it easier for the bentonite binder to flow when a pellet is compressed, resulting in a ductile breakage with a low max strength. For more dry pellets, the porosity is low and the high resistance to flow of the binder makes the pellet less plastic.

# 6.2.2 Green Pellet Strength

After plotting the max stress of all tested pellets against their plasticity, a clear correlation between these two pellet properties was observed as can be seen in Figure 6.5.

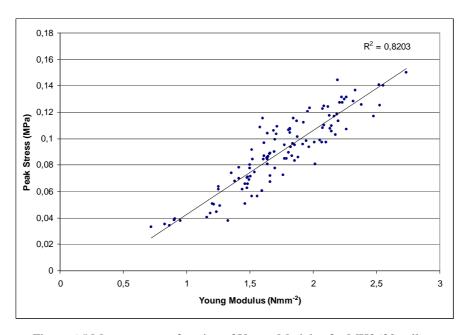


Figure 6.5 Max stress as a function of Young Modulus for MH2630 pellets.

0.7% bentonite, 7.7% moisture.

Based on these findings it was concluded that the dominant binding mechanism within the tested green pellets at all tested strain rates is strain rate dependent. Therefore the bentonite binder was believed to be of influence on pellet strength for all strain rates.

In addition, the correlation between pellet plasticity and pellet strength was studied with plasticity described by the  $d_{100}$  instead of the Young Modulus. When correlating pellet strength with the  $d_{100}$ , the correlation was found to be much weaker ( $R^2 = 0.20$ ).

These result show that there is no region in which strain rate has no effect on pellet strength as was found in earlier work (Iveson et al., 2002) within the range of applied strain rates. Iveson's studies however were performed on pellets formed from relatively large unisized spherical ballotini particles.

Because of this, the permeability of pellets used in Iveson's work is believed to have been much higher. Therefore, the binder had much more freedom of flow, decreasing the viscous binding forces.

# 6.2.3 Pellet strength testing

When studying the relationship between Young Modulus and max stress in this study, it was found that this was very different from the one found during earlier tests, performed at constant load rate. In Figure 6.6, the max stress of the pellets used during this study is shown when tested at a constant load rate of 2.7N/s and at a constant load speed of 0.25 mm/s.

It can be seen in Figure 6.6 that for pellets tested at constant load rate the max stress is higher for more plastic pellets. The exact opposite was witnessed when testing the same pellets at constant load speed. This can be explained by the viscoplasticity of the pellets demonstrated in Figure 6.4 and the difference between the two test methods.

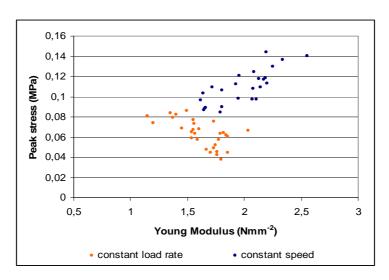


Figure 6.6 Max stress as a function of Young Modulus for the same MH2630 pellets tested at constant load rate (2.7N/s) and constant speed (0.25mm/s), 0.7% bentonite, 7.7% moisture.

When testing green pellets at a constant load rate, the applied load speed during the tests depends on the plasticity of a pellet. Plastic pellets are compressed at a higher strain rate than more stiff ones in order to maintain a constant load rate. As seen in Figure 6.4, a higher strain rate results in higher max strength due to the viscosity of the binder.

The max stress of the same MH2630 pellets as a function of the strain rates applied during tests at a load rate of 2.7 N/s and constant load speed of 0.25mm/s is shown in Figure 6.7.

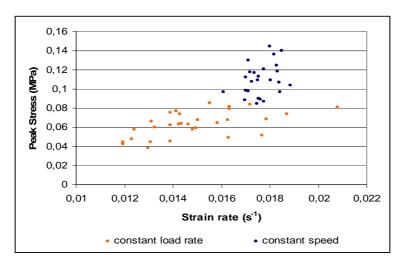


Figure 6.7 Max Stress as a function of strain rate for the same MH2630 pellets tested at constant load rate (2.7N/s) and constant speed (0.25mm/s), 0.7% bentonite, 7.7% moisture.

It is seen that for tests conducted at constant load rate, the difference in strain rate has a large influence on the measured max strength of the pellets.

Based on these findings it was concluded that, due to their viscoplastic nature, green pellets should be tested at a constant rate of strain instead of a constant rate of stress.

# 6.3 INFLUENCE OF INDIVIDUAL ORE FINENESS

As described in section 4.2, for each of the four feeds (MH2547 and A, B, and C-feed) three batches of pellets were balled under the addition of a constant amount of moisture and bentonite. The plasticity and max stress of the pellets of each batch were tested at load speeds of 0.05 and 0.5 mm/s.

# 6.3.1 Moisture, saturation and porosity

In order to be able to draw any valid conclusions from the conducted experiments, all factors other than individual ore properties that were believed to have an influence on green pellet strength and/or plasticity such as saturation, porosity and bentonite content had to be kept constant between all batches.

The measured average moisture content, saturation and porosity of all batches are listed in Table 6.1. As can be seen the porosity and saturation levels of the A, B, and C-feed are very similar. The porosity of the MH2547 feed however was found to be somewhat higher. Also, the moisture content of the pellets formed from the MH2547 feed were higher.

Table 6.1 Measured moisture content, saturation and porosity of all batched of MH2547 and A, B, C-feed

		Moisture (% on wet)	Saturation (%)	Porosity (%)
MH2547	1	7.4	87.6	30.5
	2	7.3	86.2	30.5
	3	7.4	85.3	30.8
A feed	1	6.3	85.8	28.5
	2	6.4	87.1	28.2
	3	6.6	86.6	29.3
B feed	1	6.3	85.1	28.1
	2	6.3	85.4	28.8
	3	6.4	87.6	27.9
C feed	1	6.4	85.4	28.0
	2	6.3	86.2	28.3
	3	6.4	85.9	28.6

In previous work it was concluded that porous particles of hydrophilic ores such as goethite can absorb moisture under wetting (Iveson et al., 2004). As decribed in section 5.2.2, the

MH2547 contains around 9.5% of goethite which has a relatively higher wettability and higher innner particle porosity than magnetite and hematite.

The measuring method used to determine the pellet porosity (Tata Steel, 2011) does not make a distinction between saturation of inter- or inner particle pores. Because the value for saturation is similar to the ones of the pellets from the other feeds, the interparticle porosity of the pellets balled from the MH2547 feed is believed to be the same as that of the pellets balled from the other feeds.

# 6.3.2 Pellet plasticity

The plasticity of the tested pellets from all batches is shown in Figure 6.8. It can be seen that there is no significant difference in plasticity between pellets of the four feeds when tested at the same load speed. However, for the lower strain rate, plasticity was higher for all four feeds.

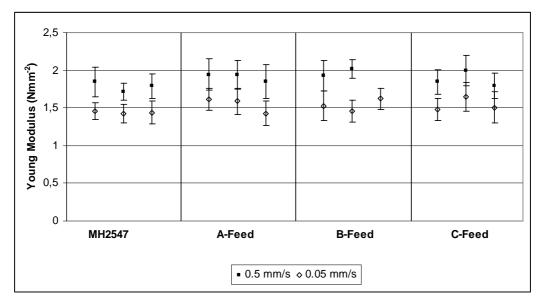
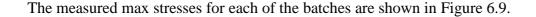


Figure 6.8 Average measured Young Modulus for pellets balled from MH2547 and the A, B, C-feeds tested at different strain rates. error bars mark  $\pm \sigma$ . (No data on 3<sup>rd</sup> B-Feed at 0.5mm/s due to insufficient amount of produced pellets)

This drop in plasticity for lower strain rates shows that the pellets from all batches are viscoplastic.

### 6.3.3 Pellet strength



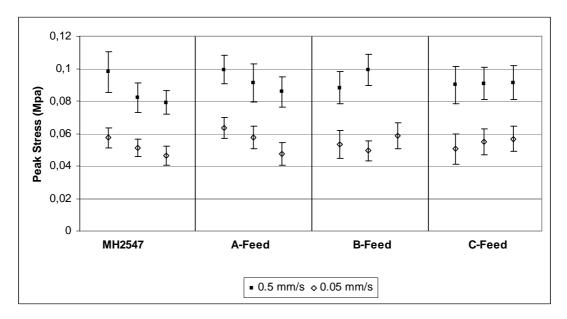


Figure 6.9 Average measured Max Stress for pellets balled from MH2547 and the A, B, C-feeds tested at different strain rates. error bars mark  $\pm \sigma$ . (No data on 3<sup>rd</sup> B-Feed at 0.5mm/s due to insufficient amount of produced pellets)

As can be seen in Figure 6.9, there is no significant differences in the measured max stresses between the different feeds when measured at similar strain rate for both load speeds. However, the max stress was higher for all batches when measured at 0.5mm/s than when measured at 0.05 mm/s. This decrease in strength is believed to be caused by the decrease in viscous forces.

In previous work it was found that the binding force within green pellets is the sum of the capillary forces, viscous forces and frictional forces (Forsmo et al., 2004). Out of these three, only the viscous force of the binder is strain rate dependent. The contribution of the viscous force to total strength decreases with decreasing strain rate. In it can be seen that the difference in strength between the pellets loaded at 0.5 and 0.05 mm/s is similar for all tested batches.

Based on these findings it can be stated that the difference in contribution of both capillary and frictional forces to the total strength between the four batches is insignificant. Therefore it can be concluded that differences in individual ore fineness and overall blend composition were found to have no significant effect on pellet strength.

### 6.3.4 Relative Grinding Energy

Both the input weight and number of revolutions of every individual grind is known. Based on these numbers each grind could be given a value for the energy needed to create a certain amount of that grind given by:

$$E_g = \frac{revs.}{mass_{input}}$$
 (Equation 6.1)

With,

 $E_g$  = Energy consumption [gram<sup>-1</sup>]

R = Number of Revolutions [-]

 $mass_{input}$  = Input mass for grind [gram]

Using these  $E_g$ -values for individual grinds, the maximal difference in energy needed to prepare a particular blend from ores of different individual fineness but a constant overall PSD could be investigated.

The maximum and minimum energy needed to prepare a feed of the same blend composition as the A, B, and C-feeds and the same overall PSD as MH2547 were generated. Through the same iteration as described in section 5.3.2, the weighted sum of all available grinds of the three different ores was fitted against the PSD of MH2547. This time however, the target was not to find the best fit, but the minimum and maximum of the calculated  $E_g$ -value. To assure similar overall PSD, the values of  $10\mu$  and  $25\mu$  fractions as well as the  $D_{80}$  of the created PSD had to be within 5% of the ones of the MH2547 PSD.

The PSD's of the individual ores and overall PSD of both the blend of minimum and maximum overall  $E_g$ -value can be seen in Figure 6.10.

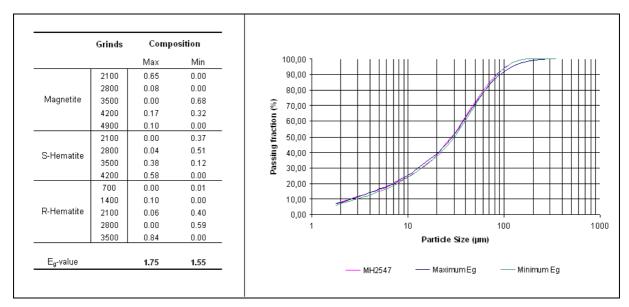


Figure 6.10 Ratio between different grinds and resulting PSD's for generated mixtures of the maximum and minimum  $E_{\rm g}$ -value.

It was found that the maximum E<sub>g</sub>-value was 12% higher than the minimum value.

These results add to the assumption that a grinding strategy based on over grinding softer ores and leaving harder ores coarse can definitely positively affect the energy consumption of the grinding circuit.

# **6.4 PELLET MICROSTRUCTURE**

During the experiments, a large variation in plasticity and pellet strength was measured within the batches. As mentioned before, this variation was attributed to variations in saturation, porosity and binder viscosity. Another explanation of these variations could however be found in differences in the distribution of these properties within a green pellet. In Figure 6.11, a Backscatter Electron Image (BEI) of a green pellet from one of the C-batches is shown. It can be clearly seen that within the pellets there are various regions of different porosity. The origin of these regions are believed to be different and are further discussed.

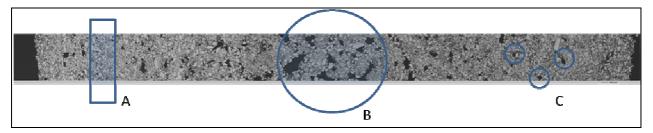
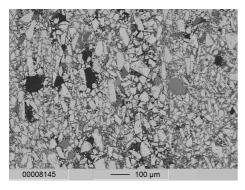
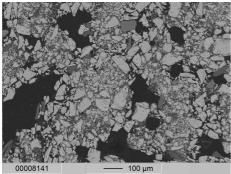


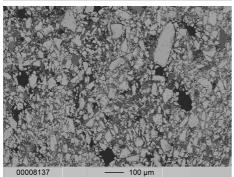
Figure 6.11 Cross section ( $\pm 12$ mm) of dried green pellet using BEI showing variation in porosity between different regions in the pellet. Pores are shown as black spots.



A. Due to inconstant feed and/or moisture addition some areas are developed at a different growth rate. Due to the difference in compaction, variation occurs in porosity within different regions of the green pellet.



**B.** The core of the pellets have a relatively high porosity due to the fact that during seed production the seeds were not compacted as much due to their low mass.



**C.** During balling, air bubbles are trapped inside the pellet, resulting in areas of relatively high porosity.

In addition to the EMP images a green pellet from the same C-batch was analysed using a Micro CT-scan. A cross sectional image of a pellet from the C-batch is shown in Figure 6.12.

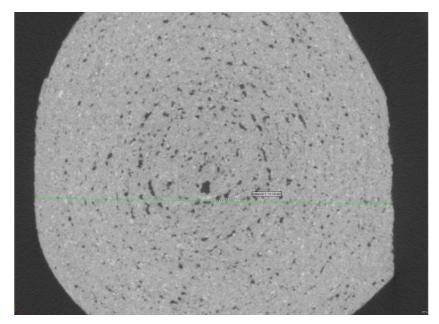


Figure 6.12 Micro CT-scan Image of a dried green pellet from the C-Batch. Pores can be seen as black spots. Cross section is  $\pm$  12mm.

Once more, a non homogenous distribution of pores is seen. Porosity in the core appears to be much higher than in the outer layers, probably due to the fact that the mass of the pellets during seed production is to small to create the level of compaction during balling required to drive all trapped air out.

This inhomogeneous porosity distribution within the tested green pellets is believed to have an effect on the pellet plasticity and strength. From section 6.1, it was found that pellet plasticity and strength are closely related to the freedom of the bentonite binder to flow. This is affected by the porosity of the pellet, but maybe even more by its permeability.

Pellets with the same porosity might have a very different distribution of pores resulting in different permeability. Also, the permeability is different between different regions within the pellets. This will create zones of different plasticity and strength within a pellet. These variations within a pellet are believed to be partially responsible for the measured variation in pellet plasticity and strength within a batch.

As can be seen in Figure 6.12, growth of pellets balled in a lab scale pellet disc or drum is done by layering. In full scale balling circuits, the higher pellet plasticity and higher impact velocities give way to other growth mechanisms such as the coalescence of smaller pellets. Such growth mechanisms are believed lead to pellets with an even more inhomogeneous distribution of porosity making these pellets weaker. The type of growth is known to be dependent of the deformability of the pellet (Iveson et al., 2001; Forsmo, 2006). Therefore, measuring pellet plasticity could provide valuable information in terms of balling process control.

# 7 CONCLUSIONS & RECOMMENDATIONS

# 7.1 GRINDING STRATEGY

Based on the experimental results , the hypothesis that had been formulated in Chapter 1 was tested. The hypothesis was:

"The <u>Compressive Strength</u> of green pellets balled with the use of a <u>Bentonite Binder from a</u> pellet feed with a <u>Fixed Particle Size Distribution</u> consisting of a <u>Blend</u> of several iron ores, is influenced by the <u>Individual Fineness of Ores</u> within- and the overall <u>Composition</u> of the blend. However, a <u>Critical Strain Rate</u> exists above which both variables loose this influence"

From the experiments using the A, B, and C-feed, it was found that the individual fineness of the ore had no influence on the green pellet strength when tested at both 0.05 and 0.5 mm/s.

Also, no significant difference in both plasticity and strength was found between pellets balled from these feeds and those formed from the MH2547 feed, which had a very different composition. This also suggests that blend composition has no influence on green pellet strength and plasticity.

During tests performed on the MH2630 pellets, it was found that even for strain rates as low as 0.01 mm/s the pellet strength remained strain rate dependent. The existence of a critical strain rate at which viscous forces had no further influence on pellet strength as was seen in earlier studies (Iveson et al., 2002) could not be verified.

Therefore, for the range of strain rates used in this study, the hypothesis fails. Individual ore fineness and/or blend composition was found to have no significant influence on pellet strength over the entire range of applied strain rates. For strain rates higher than the ones applied during this study the dominance of the viscous forces is believed to be even higher, as the resistance to flow of the binder will increase.

The results of the experiments however do not rule out the existence of such a critical strain rate at strain rates even lower than the ones applied during this study.

It should however be mentioned that, because no difference was found in pellet strength between the batches in tests at both strain rates, the contribution of the viscous force to the total binding force was the same in all tested pellets. From this and the fact that the total binding force is the sum of frictional, capillary and viscous force, it can be concluded that pellet strength at strain rates lower than the ones used during this study are not influenced by individual ore fineness and/or blend composition.

A simplified calculation of the grinding energy needed to construct an ore feed with the same blend composition as the A, B, and C feed and the same PSD of MH2547 from the raw ores used in these experiments, showed that there was 12% difference between the minimum and maximum needed grinding energy. This difference between maximum and minimum grinding energy is believed to be even higher for actual blends, as ores in these blend demonstrate much larger variations in grindability than the three ores used in these experiments.

These results allow for further exploration of a grinding strategy involving over grinding soft ores and leaving harder ores coarser.

# 7.2 PROCESS MONITORING

Pellet strength within a batch of green pellets was found to show much variation. This variation is believed to be caused by small variation in saturation, bentonite content and other pellet properties. Approaching green pellet strength not as a single average value but as a distribution of data points will provide additional information on the consistency of the process.

A strong correlation was found between MH2630 pellet plasticity, described by the pellet Young Modulus, and pellet strength ( $R^2$ =0.82). The current measure of plasticity used at IJmuiden, the  $d_{100}$ , had a much weaker correlation ( $R^2$ =0.201).

The strong correlation between plasticity and strength is explained by the fact that both quantities are functions of saturation, porosity and viscosity of the binder. This correlation was earlier described by Forsmo et al. in 2006, which led to the conclusion that pellet plasticity should gain the status of a standard pellet quality test as it holds valuable information on whether a balling circuit is running at its most ideal moisture and bentonite level. Based on findings of Forsmo's work and the results of this study, the author of this report shares this view.

In other work (Forsmo et all. 2007), it was found that the ideal moisture level for balling best correlated with the fineness of the feed when expressed by the steepness of the PSD instead of the Blaine number. This, in combination with the errors in the measurement of pellet feed fineness that are currently encountered as described in chapter 3, suggest that online measurement of the PSD of ore feed leaving the grinding circuit could help optimising the grinding circuit performance and therefore the entire pelletising process.

Based on the result that green pellet strength is strain rate dependent it was concluded that comparing green pellet strength measured at constant load rate (N/s), as is currently done at IJmuiden, can lead to false conclusions. It is concluded and recommended that green pellet strength should be tested at constant load speed (m/s).

# 7.3 FUTURE WORK

As described in section 1, this the work discussed in this report is part of a larger project which consists of three stages. Through a literature review and process data analysis, the first stage of this project has been completed. Stage two is still ongoing as additional methods to increase the pellet production can still be indicated. The third stage, which is not included in this work, is to study the feasibility of the possible improvement which are identified in stage 2. In the case of a blend grinding strategy such as described in this work, this would include a redesign of the grinding circuit.

During this study the influence of variations of individual ore fineness within the pellet feed on green pellet strength have been studied. The effect of the proposed grinding strategy on the firing of green pellets should be further studied.

Also it was shown in this work that pellet strength strongly correlated to the freedom of flow of the viscous binder. Through studying the microstructure of green pellets is was found that, within a pellet, regions of different porosity and permeability exist. The mechanism responsible for the pellet growth is believed to have a large influence on this. Therefore, the relations between green pellet growth mechanism, microstructure and strength should be studied. The Micro CT scan has proven to be a fast and useful instruments for such measurement as it is much faster and requires much less sample preparation than EMP imaging.

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