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THE USE OF IRON ORE FINES IN COLD-BONDED SELF-

REDUCING COMPOSITE PELLETS

Ву

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Abstract

The feasibility of producing Direct Reduced Iron from cold-bonded self-reducing composite pellets made from Anglo American Kumba Iron Ore Sishen upgraded slimes was considered on laboratory scale. Composite pellets were made by mixing Sishen upgraded slimes, coke and different binders using a pelletizing disc by addition of moisture. Binders investigated were dextrin, carboxymethylcellulose, calcium-lignosulphonate and combination of dextrin and bentonite. Dry strengths in excess of 300 N /pellet were attained by curing the pellets under ambient conditions. Dry strength of composite pellets increased with increase in binder quantity and decreased with increase in coke quantity. The composite pellets reduced within 20 minutes to degrees of metallisation in excess of 90 % at 1100 °C, with decrepitation indices significantly below 5 %. The degree of metallisation of composite pellets increased with an increase in reduction temperature (from 1000 to 1100 °C), reduction time (20 vs. 40 minutes) and coke quantity (15 vs. 20 wt. %). Carboxymethylcellulose was identified as the most economical and suitable binder for the Sishen upgraded slimes.

Key words: composite pellets, slimes, organic binder, cold-bonding, DRI

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1 Background

Anglo American Kumba Iron Ore is the world's fourth largest supplier of seaborne iron ore (1). In 2014 it exported 84% of its 48.2 Million tonnes production to customers in China, India, Japan, South Korea, Europe and Middle East (2).

Kumba Iron Ore's principal operating assets are Sishen and Kolomela Mines located in the Northern Cape Province. It also owns the Zandrivierspoort Project, in Limpopo Province, which is at feasibility stage (2).

At Sishen Mine, ore is extracted using open cast methods. The mining process involves removal of over-burden, drilling and blasting. Iron is then transported to beneficiation plants where it is crushed, washed and screened according to its grade (3).

However, during these beneficiation processes, fines are inadvertently produced. These fines are separated from the lump ore through the use of screens, classifiers and cyclones (3). Sishen mine produces approximately 3.5 Mt of slimes annually (4). Slimes from the Dense Medium Separation (DMS) plant were traditionally discarded into slimes dams as waste material (3). When compared to the 2012 production, this translates to 10.4 % lost from the mined iron ore. Due to the high iron oxide content of these slimes (53 % Fe) and the ever increasing demand for iron ore products, a study was initiated in 2005 to investigate means of concentrating the slimes (5). This would result in increased resource utilization, which in turn could increase the life of the mine.

Laboratory test work concluded that a 66.5 % Fe concentrate could be produced from Sishen DMS thickener underflow. This was achieved through a process which includes twostage SLon magnetic separation of thickener underflow. A recovery of 50% was achieved, which could however be increased by further beneficiation of SLon slimes (5).

The upgraded slimes however are unattractive in the market as a sellable product. This is due to their high moisture content and the difficulties associated with their handling and transportation because of their fineness (4). Consequently there is a need for investigating ways of adding value to these slimes for the company to realize more profits. One of the potential solutions is to produce Direct Reduced Iron (DRI) from the slimes. DRI is sold at a higher price when compared to iron ores fines.

Studies carried out by several researchers indicated that the reduction of iron ore pellets is accelerated if a limited amount of carbonaceous material is incorporated in the agglomerate (6, 7, 8, 9, 10 and 11). This is as a result of the presence of the reductant in-situ, resulting in shorter diffusion distances of the reducing gas and also an increased number of reaction sites (9 and 10). Based on this phenomenon, it was decided to examine the potential to produce cold-bonded self-reducing composite pellets from Sishen upgraded slimes, coke fines and an organic binder using a design of experiments (DOE) approach. The produced pellets were cured naturally at ambient conditions to attain strength adequate for handling. These pellets were then subjected to laboratory tests to evaluate their dry strengths, degree of metallisation and decrepitation characteristics.

1.0 Research objectives

The main objective of this study was to evaluate the feasibility of producing DRI from Sishen upgraded slimes, in the form of cold-bonded self-reducing, ore-coke composite pellets.

The specific objectives were:

- To identify the binder that imparts the highest dry strength and the highest degree of metallization on the iron ore pellets (dextrin, dextrin + bentonite, calcium-lignosulphonate versus carboxymethylcellulose),
- To determine the effects of the quantities of coke and binder on the strength of the dried pellets, and
- To determine the effects of the quantities of coke, the reduction temperature and the reduction time on the degree of metallization and the decrepitation index of the reduced composite pellets.

2 Literature survey

2.0 Iron and steel industry analysis

World production of crude steel reached an all-time high in 2014 at 1 670 Million tonnes, Mt, (12). This represents a 133 % increase in world crude steel production as compared to 1980 (12). The annual production of crude steel from 1980 to 2014 is shown in Figure 1.



Figure 1. Annual world production of crude steel 1980-2014 in (Mt) (12).

Growth has been gradual from 1980 up to 2000. However a drastic growth was witnessed since the turn of the century. This growth is being driven by the economic growth in Asian countries particularly in China.

Steel is produced in most countries of the world. However, the top 10 world crude steel producers accounted for over 83 % of the world crude steel production in 2014 (13). The crude steel contributions of countries or regions from 2007 to 2014 are summarised in Table 1.

 Table 1. Crude steel production by country or region 2007-2014 (Mt) (13).

Rank	Country/Region	2007	2008	2009	2010	2011	2012	2013	2014
1	China	494.90	500.30	573.60	626.70	683.30	724.70	779.00	822.70
2	Japan	120.20	118.70	87.50	109.60	107.60	107.20	110.60	110.67
3	United States	98.10	91.40	58.20	80.60	86.20	88.60	87.00	88.17
4	India	53.50	57.80	62.80	68.30	72.20	77.30	81.20	86.53
5	South Korea	51.50	53.60	48.60	58.50	68.50	69.30	66.00	71.54
6	Russia	72.40	68.50	60.00	66.90	68.70	70.60	69.40	71.46
7	Germany	48.60	45.80	32.70	43.80	44.30	42.70	42.60	42.94
8	Turkey	25.80	26.80	25.30	29.00	34.10	35.90	34.70	34.04
9	Brazil	33.80	33.70	26.50	32.80	35.20	34.70	34.20	33.91
10	Ukraine	42.80	37.30	29.90	33.60	35.30	32.90	32.80	27.17
11	Italy	31.60	30.60	19.70	25.80	28.70	27.20	24.10	23.74
12	Taiwan	20.90	19.90	15.70	19.60	22.70	20.70	22.30	23.12
13	Mexico	17.60	17.20	14.20	17.00	18.10	18.10	18.20	19.00
14	Iran	10.10	10.00	10.90	12.00	13.00	14.50	15.40	16.33
15	France	19.30	17.90	12.80	15.40	15.80	15.60	15.70	16.14
16	Spain	19.00	18.60	14.30	16.30	15.60	13.60	13.70	14.25
17	Canada	15.60	14.80	9.00	13.00	13.10	13.50	12.40	12.73
18	United Kingdom	14.30	13.50	10.10	9.70	9.50	9.60	11.90	12.12
19	Poland	10.60	9.70	7.20	8.00	8.80	8.40	8.00	8.54
20	Austria	7.60	7.60	5.70	7.20	7.50	7.40	7.90	7.86
21	Belgium	10.70	10.70	5.60	8.10	8.10	7.40	7.10	7.33
22	Netherlands	7.40	6.80	5.20	6.70	6.90	6.90	6.70	6.96
23	South Africa	9.10	8.30	7.50	8.50	6.70	7.10	7.20	6.55
24	Egypt	6.20	6.20	5.50	6.70	6.50	6.60	6.80	6.49
25	Saudi Arabia	4.60	4.70	4.70	5.00	5.30	5.20	5.40	6.29
26	Vietnam	2.30	2.70	2.70	2.70	4.90	5.30	5.60	5.85
27	Argentina	5.40	5.50	4.00	5.10	5.70	5.00	5.20	5.49
28	Czech Republic	7.10	6.40	4.60	5.20	5.60	5.10	5.20	5.36
29	Slovakia	5.10	4.50	3.70	4.60	4.20	4.40	4.50	4.71
30	Australia	7.90	7.60	5.20	7.30	6.40	4.90	4.70	4.61
31	Sweden	5.70	5.20	2.80	4.80	4.90	4.30	4.40	4.55
32	Indonesia	4.10	3.90	3.50	3.60	3.60	2.30	2.60	4.43
33	Malaysia	6.90	6.40	4.00	4.10	5.90	5.60	4.70	4.32
34	Thailand	5.60	5.20	3.60	3.70	4.20	3.30	3.60	4.10
35	Finland	4.40	4.40	3.10	4.00	4.00	3.80	3.50	3.81
36	Kazakhstan	4.80	4.30	4.10	4.30	4.70	3.90	3.30	3.68
37	Romania	6.30	5.00	2.70	3.90	3.80	3.30	3.00	3.16
38	Qatar	1.10	1.40	1.40	2.00	2.00	2.10	2.20	3.02
39	Belarus	2.40	2.60	2.40	2.50	2.60	2.70	2.20	2.51
40	UAE	0.09	0.09	0.20	0.50	2.00	2.40	2.90	2.39
41	Luxembourg	2.90	2.60	2.10	2.50	2.50	2.20	2.10	2.19
42	Venezuela	5.00	4.20	3.80	2.20	3.10	2.40	2.10	1.49
	Others	30.70	28.60	23.30	26.50	29.90	29.50	28.40	27.93
	World Total	1351.30	1326.50	1219.70	1413.60	1490.10	1552.90	1607.20	1 670.15

In 2014, Asia produced 1 132.3 Mt of crude steel, representing 68 % of the world crude steel production. This contribution was mainly due to China which produced 49 % of the world crude steel production (12).

In 2014 South Africa was ranked number twenty three in the world for producing 0.392 % of the world crude steel (13). In Africa, South Africa was rated the top crude steel producer followed by Egypt which occupied number twenty four in the world by producing 0.388 % of the world crude steel.

The world production of iron ore stood at 3 242 Mt in 2014 as shown in Table 2.

Country	Production	% of the		
	(Mt)	World		
China	1500	46.3		
Australia	660	20.4		
Brazil	320	9.9		
India	150	4.6		
Russia	105	3.2		
Ukraine	82	2.5		
South Africa	78	2.4		
United States	58	1.8		
Iran	45	1.4		
Canada	41	1.3		
Kazakhstan	26	0.8		
Sweden	26	0.8		
Venezuela	20	0.6		
Others	131	4.0		
World total	3242			

 Table 2. 2014 World iron ore production (Mt) (14).

China, again, led the world in 2014 at 1 500 Mt of iron ore. South Africa ranked number seven produced 78 Mt of iron ore translating to 2.4 % of the world total.

There are three process routes for producing steel. The blast furnace-basic oxygen furnace (BF-BOF) iron and steel making route is the principal route (15, 16). It currently accounts for 60 % of the world steel production. The electric arc furnace (EAF) steel making process is placed second, accounting for 35 % of the world steel production. The EAF process uses

scrap metal and DRI as feed stocks. Open hearth furnace (OHF) is the third steel making method responsible for 5 % of world steel production. This route is being phased out due to the environmental, operational and cost factors (16).

A schematic of an integrated steelmaking plant employing the two dominant steel making routes is shown in Figure 2.

Figure 2. The BF-BOF and EAF steel making routes (17).

2.1 Evolution of alternative iron making processes

Despite the blast furnace route being the principal method for primary iron making, the late 1960s witnessed the birth of alternative iron making processes. These processes were innovated to cater for the shortcomings of the blast furnace. Zervas et al (15) pinpointed these shortcomings:

For a blast furnace to be economic, it must produce more than 3 Mt of pig iron per year. This means high capital costs and long lead times. This makes the blast furnace inflexible to respond rapidly to market changes. The blast furnace relies on high quality metallurgical coke as fuel and reductant.

Direct reduction and direct smelting processes evolved to overcome these shortcomings in the blast furnace iron making route by accommodating cheap raw materials at relatively lower scales of operation.

Direct reduction processes are concerned with the removal of oxygen associated with iron in the ore without melting, using hydrocarbons and/or carbonaceous materials (18). The product is referred to as DRI, which is a highly metallised porous solid. Pietsch (19) highlighted that DRI comprise of the same, but porous, structure of the iron bearing oxide feed material that was reduced. Since there is no melting, the removal of oxygen that is chemically bonded with iron leaves the gangue materials trapped in the product. Direct reduction process which are currently operational include vertical shaft furnace processes (Midrex and HYL), rotary kiln processes (SL/RN, Jindal,DRC,CODIR,TISCO,SIIL, OSIL, DAV and BGRIMM) and rotary hearth furnace process (Iron Dynamics Process) (20). In vertical shaft furnaces, the iron bearing feedstocks (Pellets, lump ore, or their mixture) fall under gravity, counter-currently to rising natural gas (15 and 18). Proper bed permeability is therefore key to process efficiency. Generation of fines from the feed material must be avoided. Consequently the iron bearing feed stock needs to have high load-bearing strength (18).

In rotary kiln furnaces, iron bearing feedstocks (pellets, lump ore, and self-reducing green balls) and coal gravitates down a slightly inclined rotatin cylindrical shell (18). The iron bearing charge is thus required to be very strong. Rotary hearth furnace processes are coal-based. Pellets, lump ore and self-reducing green balls are used as iron bearing feedstocks (18). The charge to rotary hearth furnaces is stationary and thus the iron bearing feedstock is not required to have a high load-bearing strength.

2.2 World DRI production

Direct Reduced Iron production has firmly increased since 1970s reaching an all-time high of 74.55 Mt in 2014 (20).

The annual production of DRI from 1970 to 2014 is depicted in Figure 3 while the top DRI producing countries of 2014 are listed in Table 3.

Figure 3. World annual DRI production 1970-2014 (20)

Table 3. Top 14 DRI producing countries in 2014 (20).

Country	DRI production (million tonnes)			
India	17.31			
Iran	14.55			
Saudi Arabia	6.46			
Mexico	5.98			
Russia	5.35			
Trinidad and Tobago	3.24			
Egypt	2.88			
Qatar	2.64			
United Arab Emirates	2.41			
Venezuela	1.68			
Argentina	1.67			
South Africa	1.55			
Canada	1.55			
Oman	1.45			

India was the number one producer of DRI in the world in 2014, closely followed by Iran. Egypt produced 2.88 Mt of DRI, contending at number seven in the world but holding the top position in Africa. South Africa was at number twelve in the world, but placed second in Africa for producing 1.55 Mt of DRI in 2014.

Kopfle and Hunter (21) outlined the reasons why DRI production is increasing. DRI is traditionally charged with scrap metal into the EAF. High grade scrap is now diminishing, making DRI the main feed material to the EAF. EAF steelmaking is also growing due to its lower capital costs and less environmental pollution (21). These two factors have resulted in an increased demand for DRI.

2.3 Chemical reactions in direct reduction processes

Chatterjee 1994 (22) and Zervas et al 1996 (15) discussed the chemical reactions which occur in direct reduction processes.

Figure 4. Chemical reactions participating in direct reduction processes (15, 22).

Solid carbon reduces the iron oxide according to reactions equations 5, 6 and 11. These reactions using carbon are referred to as direct reduction processes. These reactions are crucial for direct reduction processes that produce direct reduced iron from solid carbonaceous materials with no gasification of their fixed carbon. Carbon monoxide and hydrogen participate in reaction equations 1, 2, 3, 4, 7, 8, 9, 10, 12 and 13. These reactions are termed indirect reduction processes.

Below 1000 ° C reactions 2, 3, 7, 8, 12 and 13 are dominant, resulting in porous DRI with similar size and shape of the original iron bearing oxide.

Reactions 14 – 16, are responsible for carbon absorption by metallic iron and these reactions are called carburization reactions.

Reaction 18, the Boudouard (solution loss) reaction and reaction 19, the water-gas reaction are dominant above 1000 $^{\circ}$ C. Reactions 18 and 19 are essential for increasing the reduction potential of the reducing gas by furnishing CO and H₂ respectively.

Since there is no melting or partial fusion in direct reduction processes, the rate of reduction reaction is essentially controlled by the reducibility of the iron oxide particles (22). In this regard, the most important characteristic of the iron bearing oxide becomes the porosity of the ore. With hard and dense ores, the partially reduced particles show a topo-chemical type of magnetite at the core, preceded immediately by wustite and an outer shell of metallic iron. This is typical for lump ore. For very porous iron ores, a more diffused type of reduction is observed with no distinct interface but a gradual transformation from iron at the periphery to hematite at the core (22).

2.4 Pelletizing of fine iron ores

Most of iron ores must be milled to allow liberation and concentration of iron oxide particles. The concentrate, however, has to be clustered back into big particles that are usable in iron reduction units (23). Many agglomeration techniques are used in different industries. In the iron ore agglomeration industry, pelletizing is widely used owing to the good qualities of the agglomerates produced. Pellets can be transported for long distances without deteriorating in physical properties (23). Pellets also have excellent reducibility and good bed permeability which result in high reduction kinetics (23).

The pelletizing process involves formation of green balls from a mixture of fine ore concentrate and binders by addition of water. The fine ore concentrate that is to be agglomerated must have optimum fine particle size distribution. Binders are added to render plasticity to the concentrate and thus helping it to form seeds that will grow into pellets. It also helps to maintain strength in the green pellets as well as during drying and subsequent handling (23).

Traditionally pellets are made using bentonite as a binder (23). Bentonite is a clay mineral which predominantly consists of montmorillonite (23). Bentonite adds silica to the concentrate thereby reducing its grade. Dried pellets are then indurated at temperatures ranging from 1200-1350 °C in grate, kiln or shaft furnaces (24). The induration process imparts excellent physical and metallurgical properties which are required for subsequent downstream processes. However, large amounts of energy are consumed during this process of hardening the pellets. Recent induration furnaces equipped with heat recovery systems consume 317 - 475 MJ per tonne of magnetite pellets and 791 MJ of energy per tonne of hematite pellets (25). For economic and environmental considerations, it is therefore desirable to find alternatives to the traditional way of producing pellets. One of the possible solutions is the cold-bonding of fine iron ore concentrate.

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2.5 Cold-bonding of fine iron ores

Cold-bonding involves the pelletizing of fine iron ore concentrates at ambient or low temperatures. During the process, fine iron ore concentrate is mixed with a fine carbonaceous material and a suitable binder to produce composite or cold-bonded pellets by addition of water. A composite pellet is an agglomerate consisting of iron oxide and carbon-bearing fines. Dry strength is attained by curing under ambient conditions. Iron oxide fines that can be used include concentrate, slimes, steelmaking sludge, rolling mill scale or blast furnace dust, while carbon bearing fines can be in the form of coal, coke or char fines (6, 7, 8 and 9).

2.6 Advantages of composite pellets

It is more advantageous to feed cold-bonded composite pellets to the iron making process as compared to cured pellets (6, 7, 8, 9, 10 and 11). The most important technical advantage of these pellets is the high rate of reduction. This is due to smaller particle size, which results in increased surface area for the reaction, and good contact among constituent particles, internal gas generation and smaller gas diffusion distances (6, 7, 8, 9, 10 and 11). This leads to lower retention times in furnaces resulting in higher productivity. Composite pellets are not fired so as to retain the internal carbonaceous material for subsequent reduction. This translates to a huge saving in energy. Environmentally toxic gases are not involved in the cold-bonding route since there is no heat hardening. Composite pellets can be produced from relatively cheap and readily available raw materials like iron ore slimes, mill scale, blast furnace dust, steel sludge, coal fines, coke breeze and char (9 and 26). Since the cost of production of these raw materials has already been incurred, cold-bonding techniques therefore have the benefit of increasing resource utilisation and environmental pollution control. Cold-bonding techniques also help in environmental pollution control by consuming these potential pollutants in a beneficial way.

2.7 Criteria for binder selection

The process of selecting a suitable binder and its dosage-level is quite difficult in the coldbonding of iron ores. Qiu et al (27) summarized the characteristics that are desired in suitable binders. A suitable binder must impart sufficient dry strength to cold-bonded pellets. Minimum dry compression strength of 300 N/pellet has been reported to be sufficient for pellet handling in rotary hearth furnace (10 and 26).

The chemical composition of a binder affects its suitability in cold-bonding of iron ores. A good binder must not add metallurgically harmful elements (like phosphorus, sulphur and arsenic) to the pellet. It should not markedly reduce the pellet grade by introducing gangue oxides like silica and alumina.

Good metallurgical performance of the product pellets should also be assured by a good binder. An appropriate binder should impart high reducibility and low swelling of the pellets. Finally a good binder should be readily available at an acceptable cost. Its addition, its mixing and dispersion should not be complicated.

2.8 Classification of binding systems

Holley (28) categorised binders into five groups according to the type of binding action used by the binder:

2.8.0 Inactive film binding systems

The binders which fall under this category require mixing with the concentrate prior to moisture addition. The surface tension of the moist part pulls the particles together while the solid portion acts as both lubricant and glue. The glued portions in the system transform into solid bridges after drying thereby imparting strength to the pellet. This class of binding system is used with agitative and extrusion types of agglomeration. The binder dosage level is related to the particle size distribution and surface condition of the particles, and also to the efficiency of the mixing process. Under this group, the commonly used binders in cold bonding are calcium and sodium lignosulphonates, molasses, starch, glucose and dextrin.

2.8.1 Chemical film binding systems

Under this class are binders which form a film around the particles to be agglomerated. The film then sets by a chemical reaction, the rate of which dictates the duration of curing period. The common binder used is sodium silicate.

2.8.2 Inactive matrix binding systems

Binders in this class form a continuous matrix which embeds the particles to be agglomerated without the addition of moisture. Typical binders include tars, pitches, starches and lignosulphonates. Tars and pitches are heated before they are added to the raw pellet mix to decrease the viscosity. Starches and lignosulphonates are used in powder form since their high surface energies enable them to adhere to surrounding particles (28).

2.8.3 Chemical matrix binding systems

Chemical reaction between components of the binding system forms a matrix which bonds the agglomerate together. Under this grouping we have cement materials and lime. The major drawbacks of cement as a binder include longer curing times and tendency of green pellets to stick together.

2.8.4 Chemical binding systems

Strong bonds are formed during chemical reactions between components of the binder and components to be agglomerated. Binders in this class are yet to be developed for iron ore pelletizing.

2.9 Types of cold-bonding processes

Several binders have been studied in the cold-bonding of iron ores (7, 8, 10, 26, 29, 30, 31, 32, 33, 34 and 35). Cold-bonding types of binders include carbonate bonding, cement bonding, hydrothermal bonding and organic bonding.

2.9.0 Carbonate bonding

The carbonate bond forms as a result of the reaction between hydrated or slaked lime and carbon dioxide (29). The carbonate bond is formed according to the equation:

$$Ca(OH)_2 + CO_2 = CaCO_3 + H_2O$$
 (21)

The product $CaCO_3$ acts as a cement which binds the agglomerate together. In cold-bonding, iron ore concentrate, hydrated lime and a carbonaceous material are blended together in a mixer. Pellets are produced by addition of water to the blend. The pellets are then hardened with carbon dioxide in a carbonator (29).

Typically 1-10 % lime is added with 10 % water to produce pellets with dry compression strength in the range 300-400 N per pellet. Sah and Dutta reported that carbonation is completed by applying a stream of carbon dioxide at 1500 cm³/minute for 8 minutes (26).

2.9.1 Cement bonding

Cement bonding is as a result of the reactions between hydraulic minerals with water. These complicated reactions depend on the cement chemistry. The most important reactions were presented by Takano et al (7):

$$2C_3S + 6H = C_3S_2H_3 + 3CH$$
 (22)

$$2C_2S + 4H = C_3S_2H_3 + CH$$
 (23)

During pelletizing iron oxide bearing material is mixed with carbonaceous material and cement (6-10 %) and pellets are formed by addition of water (7 and 30). Dry strength is imparted by curing under ambient conditions. Although cement bonding is capable of producing pellets of high strength, it suffers in that it reduces the pellet grade by introducing silica. Cement bonding also suffers in that pellets take 30 days to attain maximum strength thereby requiring large curing yards translating to large capital costs.

2.9.2 Hydrothermal bonding

Hydrothermal bonding refers to processes in which a combination of lime and silica rich materials is partially dissolved under hydrothermal conditions in autoclaves (23, 26). Reaction between lime and silica forms calcium hydro silicates which bind the particles together. Pellets produced from a mixture of iron oxide bearing material, burnt lime, fine silica and a carbonaceous material are cured in an autoclave at 21 atmospheres and 220 °C for 1 hour. Disadvantages inherent to the process include pellet grade lowering by acidic gangue and high capital and operational costs associated with the use of autoclaves.

2.9.3 Organic bonding processes

All the discussed cold bonding techniques, except carbonate bonding, suffer a major disadvantage of product contamination due to addition of silica and alumina as the binding components. Addition of acidic gangue oxides results in increased energy demand (30 kWh with addition of 1 % silica) and flux cost (\$2.50 per tonne of hot metal with addition of 1 % silica) in electric arc furnaces (31, 36). This has resulted in interest in organic binders which brings no gangue materials into the pellet. Organic binders are also capable of imparting sufficient dry strengths to pellets by curing at low temperatures (10, 11, and 26). Huge energy savings can therefore be realised since, 317 to 791 MJ of energy is consumed in indurating a tonne of traditional hematite pellets (25). Organic binders, however, have the disadvantage of burning away before the pellet sinters, resulting in loss in strength and decrepitation of the pellets at temperatures below 500 °C (23, 36).

Several organic binders have been evaluated for their suitability in cold-bonding. These include carboxymethylcellulose (31, 32, and 37), molasses (26 and 33), funa (34), lignosulphonates (31 and 38), dextrin (8, 10, and 11), starch (31) and peridur (39). Gosh et al (8) and Agrawal et al (10 and 11) concluded that cold-bonded composite pellets could be produced from fine iron ore by adding 3-5 % of dextrin. Carboxymethylcellulose was successfully used by adding 1-2 % of the binder. In the case of dextrin, pellets with dry compression strengths greater than 300 N per pellet were produced by curing at ambient conditions for five days.

Organic bonding seems to be the most suitable cold-bonding technique since it brings no acidic gangue into the pellets. Curing of pellets made from organic binders can be achieved at ambient conditions in relatively short times. This will result in less capital and operational costs since no sophisticated equipment is required for curing of pellets. In terms of pellet quality and curing costs, organic bonding is the best choice for the agglomeration of iron fines.

2.10 Mechanisms of pellets growth and formation

Natural and applied forces play a vital role in the formation of green pellets from fine materials. Sastry and Fuerstenau (40) and Qiu et al (27) identified the sources of these natural forces in composite pellets as:

- a) cohesive force of the binder,
- b) cohesive force of the ore particles,
- c) cohesive force of the carbonaceous particles
- d) adhesion force at the binder-ore particles interface,
- e) adhesion force at the binder-carbonaceous material particles interface,
- f) adhesion force at the ore-carbonaceous material particles interface,
- g) forces of interaction between carbonaceous material particles
- h) forces of interaction between ore particles, and
- i) capillary forces in the presence of moisture.

The strength of green pellets is a function of the magnitude of these forces. Particle size distribution, surface charges, crystal structures and amount of additives determine the magnitudes of these forces (27 and 40). Applied forces are responsible for bringing individual moist particles, in the raw pellet mix, into contact thereby allowing natural forces to impart growth. Applied forces are a result of movements of moist raw pellet mix by means of rolling and agitating in pelletizing drums and discs (40). The growth mechanism of green pellets involves nucleation, coalescence, breakage, abrasion transfer and layering (40).

2.10.0 Nucleation

Nucleation refers to the formation of new well-formed species from the moist raw pellet feed mix. The growth potential of the nucleus is due to the surface energy associated with a thin film of water surrounding the particles (41). Collusion of such particles results in the destruction of a certain amount of water-air interface which is related to the neck of the formed joints. The strength of the neck of joints formed is linked to the particle size distribution, shape, packing and thickness of the water layer (41).

2.10.1 Coalescence

Coalescence refers to the formation of bigger species as a result of joining of colliding granules. This happens when two or more balls are able to stick together in a favourable configuration relative to the rest of the charge resulting in them rolling and deforming into a larger sphere (41).

Nucleation is the process where some fines particles start to agglomerate into clusters. These then form the 'seeds' for further growth, through the layering of particles on these nuclear particles. The growing clusters will also collide with each other and when this happens coalescence takes place. Most coalesced clusters are weak, do not have favourable configurations and break-up during the rolling and cascading process. However, some survive and are deformed and incorporated to form larger spherical units.

2.10.2 Breakage

Breakage refers to the formation of well-formed species from the breaking up of coalesced pellets. Breaking results from conglomerate being unable to roll due to unfavourable shape (41). The resulting smaller species then re-distribute on existing pellets.

2.10.3 Abrasion transfer

Abrasion transfer occurs when material is transferred from one species to another species as a result of interaction and abrasion of agglomerates. The net effect is the growing of one species at the expense of another (40).

2.10.4 Layering

Layering is when new moist pellet feed mix collects on existing species in the agglomeration system. This results in pellets growing due to continuous addition of layers of new pellet feed mix (40).

The foregoing mechanisms are depicted in Figure 5.

Figure 5. Green pellet growth mechanism. Pi represents a green pellet of mass mi and P1 represents fresh nuclei of mass h. (a) coalescence stage, (b) breakage stage, (c) abrasion transfer stage, (d) layering and nucleation stage (40).

2.11 Mechanism and kinetics of composite pellets reduction

Many researchers have investigated the behaviour of composite pellets under different reducing conditions. Srinivasan and Lahiri (42) looked into composite pellets consisting of hematite and graphite in nitrogen atmosphere in the temperature range 925-1060 °C. They concluded that, in this temperature range, there is a small temperature difference between the pellet centre and its surroundings. They also indicated that temperature has a marked effect on reduction rate. Reduction rate increased with an increase in temperature for the same C/Fe₂O₃ ratio and pellet diameter as shown in Figure 6.

Figure 6. Effect of temperature on reduction for the same C/Fe_2O_3 and pellet diameter (42).
Reduction rate was also found to increase with an increase in C/Fe_2O_3 ratio as shown in Figure 7.



Figure 7. Effect of C/Fe₂O₃ ratio on reduction rate for a fixed temperature of 1295 K (42).

Wang et al (43) looked at the fast heating of composite iron ore-coal pellets in the temperature range 1050-1300 °C. They concluded that reduction by coal volatiles was negligible at high temperatures owing to their early release during heat-up. They also agreed with Srinivasan and Lahiri (42) that reduction rate increases with increase in reduction temperature. They further indicated that the reduction is governed by chemical reaction in the early stages of heating and by heat transfer in the later stage. Rao (44), agreed with Srinivasan and Lahiri (42) and Wang et al (43) that reduction increased with an increase in temperature and C/Fe₂O₃ ratio. He concluded that reduction of composite pellets was governed largely by the availability of carbon monoxide. The rate determining

step was therefore the Boudouard reaction occurring between carbon and carbon dioxide. He further argued that the particle size of the carbon-bearing material has a marked effect on the reduction kinetics.



Figure 8. Effect of particle size of the carbon-bearing material on reduction at fixed C/Fe₂O₃ and temperature of 1110 K (44).

A decrease in the particle size of carbonaceous material improves reduction kinetics. Sun and Lu (45) observed a large temperature difference exists between the pellet centre and its surroundings. They concluded that the dominant rate determining step was therefore heat transfer across the product layer and within the mixture of solid reactants. They attributed this to low thermal conductivity of the porous media and highly endothermic Boudouard reaction. They further argued that the most dominant mass transfer mechanism was convection due to gas flowing through the porous media. The type of carbonaceous material used as an internal reductant is of great significance. Fruehan (46), Donskoi et al (47) and Sun and Lu (45) agreed that soft coal is a better reductant than coke, and that coals with higher volatiles showed higher reduction rates. Dutta and Ghosh (6) however did not find a significant difference in the degree of reduction between ore-coal and ore-char pellets. If reduction is carried out at temperatures higher than 1000 °C, coal volatiles will not participate in the reduction owing to their early release during heat up. In such scenarios any carbonaceous material of sufficient fixed carbon content can be used. The overall rate limiting step is the availability of carbon monoxide and hydrogen as a result of the Boudouard and water-gas reactions (6, 9, 15, 42, 46 and 47).

3 Experimental

3.0 Raw materials

Raw materials used in this study included an iron bearing oxide, carbonaceous reductant and a range of organic binders as well as bentonite.

The iron bearing oxide was in the form of upgraded iron ore slimes received from Sishen mine, Anglo American Kumba Iron Ore. ArcelorMittal South Africa supplied the coke which was used as the carbonaceous material. Dextrin, calcium-lignosulphonate, carboxymethylcellulose (CMC) and bentonite were used as binders. Crest Chemicals supplied the dextrin, while Lignotech supplied the calcium-lignosulphonate. Bentonite was procured from Keramicalia and CMC was provided by ChemQuest Africa.

3.1 Sample characterisation

The received upgraded iron ore slimes sample was split in a giant rotary sample divider. A 3 kg sample obtained from the giant sample divider was further split in a smaller rotary sample divider. The final 30 g sample was subsequently characterised in terms of chemical composition using XRF analysis and particle size distribution using a Malvern Mastersizer Hydro2000MU. The coke sample was milled in a laboratory scale ball mill to 100 % passing 75 µm. Coke samples were then collected using a rotary sample divider and sent for proximate and chemical analysis. The binders were semi-quantitatively analysed using XRF and used as received.

The chemical composition of Sishen upgraded slimes is given in Table 4 and Appendix 1 while its particle size distribution is shown in Figure 9 and Appendix 2. The total iron of upgraded Sishen slimes is within specifications for a typical iron ore pellet feed which must be above 50 % by weight percentage (22). The silica content was also of typical pellet feed of below 5 % by weight (22). The particle size ranged from 0.32-955 µm with 77 % passing 75 µm and 63 % passing 45 µm (Figure 9). Typical iron ore pellet feed have 80 % passing 75

 μ m and 64 % passing 45 μ m (4 and 49). Results from the chemical and particle size distribution analyses suggest that as-received Sishen slimes could be used to produce pellets.

The proximate and chemical analyses of the coke used in this study are summarised in Table 4. The fixed carbon content of the coke used was 80.5 % and the ash content 15.96 %, of which the total acidic gangue content (wt % SiO_2 + wt % Al_2O_3) was 85.65 %. Introduction of this coke together with the beneficiated Sishen slimes would bring a combined acidic gangue content of 5.5 % to the pellet at high levels of coke and low levels of binder.

	Sishen slimes	Coke	Bentonite
Fe _{total}	64.93		
Fe ₂ O ₃	92.30	1.41	5.65
SiO ₂	3.65	10.59	49.7
Al ₂ O ₃	2.79	3.08	16.7
K ₂ O	0.26	0.18	0.52
Na ₂ O	0.01 0.1		2.11
TiO ₂	0.13	0.28	
P ₂ O ₅	0.13	0.09	0.08
CaO	0.12	0.17	1.84
MnO	0.06	0.01	
MgO	0.03	0.08	3.65
С		80.50	
Н		0.35	
N		0.91	
Moisture		1.95	
LOI	0.52		19.7
Total	100.00	99.72	99.95
Ash		15.96	

Table 4. Chemical composition of iron ore concentrate, coke and bentonite (mass %)



Figure 9. Particle size distribution of upgraded Sishen slimes.

Semi-quantitative XRF analysis indicated that the undesirable elements in DRI that are associated with dextrin and CMC are Al, Cl, P, Si and S, whilst calcium-lignosulphonate is associated with Ca, Cl,P, S and Si (Table 5). Bentonite is an alumina-silicate clay (Table 4) which consequently increases the acidic gangue content of the pellet. Considering the binder dosage levels (Table 6), addition of these binders will not cause a significant reduction in pellet grade.

Element	Dextrin	Calcium-	Carboxymethyl-
		lignosulphonate	cellulose (CMC)
AI	0.11		<0.01
Si	0.09	0.06	0.18
Р	0.10	0.11	0.13
S	0.06	1.26	0.09
Са		1.24	
Cl	0.03	0.029	5.44

 Table 5. Main impurity components of the organic binders (mass %)

3.2 Experimental design

A design of experiments (DOE) approach was adopted in this study. DOE is a technique used for exploring new processes and gaining increased knowledge on existing processes (48). Design of experiments involves planning, designing and analysing an experiment in order to draw valid and objective conclusions.

The approach involves the identification of process parameters (factors) which affect the performance of the process. One must decide on the range of settings of these factors which are delineated by levels of the factor. The level of a factor is the specified value of the factor under investigation (48). Having identified these factors and their level of settings, one has to simultaneously vary them, at their different levels, to determine the effect of the factors on the required response. The term 'effect of a factor', refers to how the average response changes with a change in factor level. An investigator is therefore afforded an opportunity to analyse interaction between factors. Interaction occurs when the effect of one factor depends on the level of the other factor.

A number of analytical tools are used in DOE using the MINITAB software. One can draw main effects plots, interactions plots, cube plots, Pareto plot of factor effects and Normal Probability Plots (NPP) of factor effects.

3.2.0 Main effects plot

A main effects plot reflects the average response values at all the levels of a factor. This allows one to deduce the difference in strength of the effects of a number of factors. The strength of the effect is determined by the gradient of the line on the graph.

3.2.1 Interactions plot

An interactions plot reflects the average response of two factors for all the existing interactions. Non-parallel lines show the presence of an interaction, while parallel lines depict the absence of interaction.

3.2.2 Cube plot

Cube plots give the mean response values of the existing interactions. Cube plots are used to visualise the maximum and minimum limits of each response for different combinations of levels of factors. The mean responses are shown at the corners of the cube. The best and worst combinations of factors can therefore be easily identified.

3.2.3 Pareto plot

A Pareto plot of factor effects is used to determine the most important factors and factor interactions by displaying the real values of these effects against a reference line. Important factors are those that extend past the reference line. The findings are then checked with findings from the Normal Probability Plot of the estimates of these effects. The position of the reference line depends on whether an error term exists. If no error term exists, Minitab uses Lenth's pseudo-standard error which is based on the concept of sparse effects that assumes the variation in the smallest effects to be due to random error. Otherwise if an error term exists, the reference line is drawn at the $(1 - \alpha/2)$ quantile of a t-distribution with degrees of freedom equal to the degrees of freedom for the error term.

3.2.4 Normal Probability Plot

A Normal Probability Plot plots the main effects and the interaction effects of factors against their cumulative probability per cent. Insignificant effects will lie on a straight line while the important ones are scattered on both sides of the straight line. Active effects are judged to be statistically significant at 5 per cent significance level.

The most widely used DOE approach in manufacturing processes is a factorial design at two levels (48). Full factorial designs allow the researcher to study all the possible combinations of levels of all factors.

The number of experimental runs, *n*, for evaluating the effects of *k* factors at two levels is given by:

$$n = 2^k$$
 (24)

A two-stage approach was used in this study. The first stage was concerned with pelletizing of the upgraded slimes to produce composite pellets. The second stage was concerned with the reduction of these composite pellets.

3.3 Pelletizing tests

Factors chosen in the DOE were the amount of binder and the amount of coke to be added to the raw pellet mix. The levels of binder amount and coke amount added to the upgraded slimes are shown in Table 6. The low level of dextrin was 3 % and the high level was 4 %. For CMC and calcium lignosulphonate 1 % and 2 % was added as low and high levels respectively. These values were chosen from literature (10 and 31). Either 15 % or 20 % coke was added to the raw pellet mix. These values are respectively 0.75 and 1.0 times the stoichiometric amount of carbon required for the reduction of hematite. The same pellet design matrix was used for different binders and binder combinations used (Table 7).

Process parameter	Low level (-1)	High Level (1)
Dextrin	3	4
СМС	1	2
Ca-Lignosulphonate	1	2
Coke	15	20

 Table 6. Low and high level settings of binder and coke amounts (mass %)

 Table 7. Pelletizing tests design mix.

Pellet type	Binder quantity	Coke quantity
P1	-1	-1
P2	1	-1
Р3	1	1
P4	-1	1

The same pelletizing procedure was adopted for pellet production using different binders and binder combinations. Pellets were produced using dextrin, CMC, calciumlignosulphonate and a combination of dextrin and bentonite. For pellets produced using a combination of binders, 0.075 % of bentonite as well as the usual amount of dextrin was added to the pellet raw mix according to Table 6.

3.3.0 Pelletizing procedure

Required masses of upgraded slimes, fine coke and binder were weighed and stored separately according to their predetermined proportions (Tables 6 and 7). Upgraded slimes and fine coke were then mixed in their determined proportions together with the required amount of binder. This raw pellet mix was then mixed thoroughly for thirty seconds in a high intensity mixer. A small amount of the raw pellet mix was then added to a one meter diameter pelletizing disc, rotating at sixty revolutions per minute at an angle of seventy degrees. Seed pellets were produced by spraying water to the rolling raw pellet mix feed. The seed pellets grew into large pellets by consuming freshly added moist feed. The pellets were periodically removed from the disc and screened. Approximately 7 kg of pellets with diameters in the range 10-12.5 mm were produced from 10 kg of raw pellet mix. A 500 g sample of the pellets was dried in an oven at 105 °C for 2 hours. Moisture content of the pellets, as weight percentage, was calculated by comparing the mass lost by the pellets to the original mass. These pellets were collected into drying pans and left to dry under ambient conditions for five days. Dried pellets were then evaluated for compression strength using an Instron 3367 compression tester. For a given pellet mixture design, the compression strength was the average of compression strength of twenty pellets of the same pellet design. A minimum strength of 300 N/pellet was required (10, 26). The objective was therefore to identify a binder and pellet design mixture that produced composite pellets with a minimum dry strength of 300 N/pellet.

To minimize the number of experimental runs in the reduction stage of the study, two pellet types (P2 and P3), those with highest cold strengths, were chosen for each binder type.

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3.4 Reduction tests

Factors chosen for the reduction stage were reduction temperature and reduction time (Table 8). The low level of temperature was chosen to be 1000 °C since it is the minimum temperature for the Boudouard and the water-gas reactions to participate in the reduction reactions (15). The reduction furnace could be operated safely at a maximum temperature of 1100 °C which was then selected to be the high level temperature. The levels of time were as reported in the literature, namely 20 and 40 minutes (8, 43 and 45). The same pellet reduction matrix was used for the different pellet types reduced as depicted in Table 9.

Table 8. Low and high level settings of the reduction temperature and reduction time.

Process parameter	Low level (-1)	High Level (1)
Reduction Temp (^o C)	1000	1100
Reduction time (min)	20	40

 Table 9. Reduction test design matrix.

Run	Reduction Temp ([°] C)	Reduction time (min)
1	-1	1
2	1	1
3	1	-1
4	-1	-1

3.4.0 Reduction procedure

Reduction tests were carried out in an electrically heated laboratory scale furnace, Figure 10. A 500 g sample of the air dried composite pellets was placed in a reduction tube made from an Inconel alloy. The pellets formed a bed on a perforated plate located on a bed of porcelain pellets. After closing the top, the reduction tube was carefully inserted into the furnace and suspended centrally between Kanthal elements. A thermocouple was inserted into a thermocouple holder, the tip of which was in the middle of the test sample. A 13 l/min flow of nitrogen was allowed into the reduction tube and heating of the test sample commenced. The sample was heated to the required reduction temperature in the flow of nitrogen at a heating rate of 10 °C/min. After reaching the reduction temperature, the sample was allowed to reduce in the nitrogen environment for the required time according to Tables 8 and 9. The reduction tube, with reduced sample, was then removed from the furnace and allowed to cool to room temperature. Nitrogen was purged through the reduction tube.



Figure 10. The schematic representation of the furnace used for reduction test.

3.4.1 Tumbling procedure

The reduced sample was placed in a tumbling drum (13 cm internal diameter, 20 cm in length, equipped with lifters) and tumbled for 10 minutes at 30 rev/min. The tumbled sample was carefully removed and sieved in a sieve shaker for 3 minutes. The decrepitation index was calculated as the percentage of the -0.5 mm mass fraction of the reduced sample. The tumbled sample was sent to UIS analytical services for chemical analysis to determine the degree of metallisation. The degree of metallisation, as a percentage, was calculated from the ratio of the metallic iron content to the total iron content of the sample.

4 Results and discussion

4.0 Moisture content of composite pellets

The moisture contents of the composite pellets ranged between 7.2 and 9.8 % (Table 10). Moisture content increased with an increase in binder and coke amounts for all composite pellets. Pellets constituted of CMC binder absorbed the highest amount of moisture, followed by respectively, combination of dextrin and bentonite, dextrin and calciumlignosulphonate, similar to Haas et al (31).

Pindor typo		% Moisture				
Binder type	P1	P2	P3	P4		
Dextrin	7.6	8.8	9.3	7.8		
Dextrin + Bentonite	8.0	8.9	9.4	8.2		
СМС	8.3	9.2	9.8	8.7		
Ca-Lignosulphonate	7.2	7.8	8.1	7.4		

Table 10. Moisture contents of the green pellets

4.1 Dry strength of composite pellets

The dry strengths of the composite pellets, constituted from different binders, are given in Table 11 and Appendix 3.

Table 11.	Dry strengths of the composite pellets according to the binder used.	

Pellet type	Dry strength and the standard deviation of composite pellets (N/pellet)							
	Dextrin σ^* Dextrin + Bentonite σ CMC σ Ca-Lignosulphonate							
1	370	26	578	25	384	27	56	17
2	677	38	874	46	531	34	78	18
3	540	25	803	44	519	38	61	10
4	338	27	578	24	378	31	53	13

*: Standard deviation

Composite pellets made using dextrin, a combination of dextrin and bentonite as well as CMC as binders had dry strengths greater than the required 300 N/pellet at all levels of binder and coke additions. Pellets made using a binder combination of dextrin and bentonite had the highest strength, followed by dextrin and CMC. Dry strengths of pellets made using dextrin, CMC and a combination of dextrin and bentonite are comparable with those reported in the literature (10, 26, 37, 50 and 51). Calcium-lignosulphonate as binder produced pellets with by far the lowest dry strengths. These dry strengths were lower than those reported by Chellan et al (38).

4.2 Effect of binder and coke quantities on dry strength of composite pellets

The main effect plots of binder and coke quantities on the dry strength of composite pellets are shown in Figures 11-14. The dry strength of the composite pellets increased with an increase in binder quantity, but decreased with an increase in coke quantity from 15-20 % of the raw pellet mix. The decrease in dry strength with an increase in carbonaceous materials was also observed by Agrawal et al (10). This decrease in the dry strength of the pellets with an increase in coke quantity could be attributed to the increase in hydrophobic material (carbon) in the raw pellet mix. The coke quantity, within the examined limits, did not have a significant effect on the dry strength for composite pellets made using CMC binder (Figure 13). However, it had a pronounced effect on the composite pellets made using calcium-lignosulphonate (Figure 14). This can be concluded from the gradient of the slopes which is proportional to the effect of the factor on the dry strength. In the case of the CMC binder, it meant that one could increase the quantity of coke (within the examined limits) without a marked decrease in the dry strength of the composite pellets.



Figure 11. Main effects plot of binder and coke quantities on dry strength for dextrinbonded composite pellets.



Figure 12. Main effects plot of binder and coke quantities on the dry strength for dextrin + bentonite-bonded composite pellets.



Figure 13. Main effects plot of binder and coke quantities on the dry strength of CMCbonded composite pellets.



Figure 14. Main effects plot of binder and coke quantities on the dry strength for calciumlignosulphonate-bonded composite pellets.

The interaction plots between binder and coke quantities on the dry strength of composite pellets containing different binders are shown in Figures 15-18. Interaction effects with parallel lines do no contribute to response, while those with converging lines have weak contributions and those with intersecting lines have strong contribution to the response (48). There was no significant interaction between binder and coke quantities on the dry strength of composite pellets made using a combination of dextrin and bentonite and CMC (Figures 16 and 17). However, interaction between these factors on the dry strength of the composite pellets was very pronounced in pellets respectively constituted of dextrin (Figure 15) and calcium-lignosulphonate (Figure 18) binders.



Figure 15. Interaction plot of binder and coke quantities on the dry strength for dextrinbonded composite pellets.



Figure 16. Interaction plot of binder and coke quantities on the dry strength for dextrin + bentonite-bonded composite pellets.



Figure 17. Interaction plot of binder and coke quantities on the dry strength for CMCbonded composite pellets.



Figure 18. Interaction plot of binder and coke quantities on the dry strength for caliciumlignosulphonate-bonded composite pellets.

For all of the binders studied, dry strength was highest at the higher level of the binder and it decreased as coke quantity increased. However this decrease was marginal in pellets made using CMC binder.

In consideration of the dry strengths of the composite pellets, a combination of dextrin and bentonite was the best binder, followed by dextrin, CMC and calcium-lignosulphonate respectively. All the binders evaluated produced composite pellets with dry strength greater than the minimum required dry strength of 300 N/pellet except for calciumlignosulphonate. CMC was therefore the best binder considering its dosage level of 1-2 % compared to 3-4 % of the raw pellet mix for dextrin. However, the best binder should also be chosen with due consideration given to the reducing characteristics of the composite pellets produced.

4.3 Metallisation of reduced composite pellets

The degree of metallisation of the reduced composite pellets is summarised in Table 12 and Appendix 4. Most composite pellets reduced rapidly to attain between 88 and 96 % metallisation within 20 to 40 minutes at 1100 °C, similar to previous studies (45 and 46). These composite pellets can therefore increase DRI productivity as it comparably takes 90 minutes to achieve the same degree of metallisation when using an external reductant (33).

Pellet	Temperature	Time (min)	Degree of metallisation and standard deviation (%)*						
туре	(C)	(11111)	Dextrin	$\sigma^{\#}$	Dextrin + Bentonite	σ	СМС	σ	
P2	1000	20	67.49	0.52	65.39	0.37	83.65	0.37	
P2	1000	40	89.41	0.62	86.58	0.52	91.06	0.27	
P2	1100	20	92.45	0.29	88.34	0.86	93.44	0.98	
P2	1100	40	94.46	0.59	94.67	0.67	95.34	0.61	
Р3	1000	20	70.75	0.29	69.84	0.9	85.49	0.92	
Р3	1000	40	90.22	0.21	88.91	0.55	92.85	0.45	
Р3	1100	20	94.81	0.25	90.56	0.53	95.52	0.59	
P3	1100	40	94.83	0.1	93.86	0.17	95.61	0.65	

 Table 12. Metallisation of the reduced composite pellets (%).

*: Average of two samples

[#]: Standard deviation

Composite pellets type P2 failed to reduce to minimum required metallisation degrees of 90 % at 1000 °C at both levels of reduction time for pellets made using dextrin and a combination of dextrin and bentonite as binders. However pellets made using CMC binder reduced to 91 % metallisation at a higher level of time (40 minutes) at 1000 °C. Composite pellets of Type P2 exceeded 90 % metallisation at 1100 °C at both levels of time (20 and 40 minutes) except for pellets containing a combination of dextrin and bentonite that were reduced for 20 minutes. Composite pellets of type P3 met the required minimum percentage metallization at the high level of temperature (1100 °C) and both levels of time (20 and 40 minutes).

Composite pellets comprising of CMC reduced to the highest degree of metallisation for the same reducing conditions. This could be attributed to pellets made from CMC having highest moisture content (and therefore highest porosity in dried pellets) leading to easy diffusion of reducing gas (CO) and therefore higher reduction rates.

4.4 Effect of coke quantity, temperature and time on the degree of metallisation

The effect of coke quantity, temperature and time on the degree of metallisation is shown in Figures 19-21. The degree of metallisation was found to increase with an increase in coke quantity, reduction temperature and reduction time, similar to what was in the literature (10, 42, 43, 44, 46 and 47). Comparison of the slopes of the main effects (Figures 19-21) shows that the effects of reduction temperature and reduction time, on metallisation, were more pronounced as compared to the effect of coke quantity. This shows that rates of heat and mass transfer play critical roles in the reduction rate of composite pellets as reported in previous studies (9, 43, 45 and 52). Under same reducing conditions, composite pellets containing CMC as binder had the highest degree of metallisation followed respectively by pellets made from dextrin and the combination of dextrin and bentonite at all factor settings (Table 12 and Appendix 4). At the high level of coke content (20 wt. %) and the high level of reduction temperature (1100 °C), reduction time did not significantly affect the degree of metallisation of composite pellets made using CMC (95.52 % and 95.61 %). The low level setting of reduction time (20 minutes) is therefore the better choice to increase productivity and save energy.



Figure 19. Main effects plot of coke quantity, reduction temperature and reduction time on the degree of metallisation for dextrin-bonded composite pellets.



Figure 20. Main effects plot of coke quantity, reduction temperature and reduction time on the degree of metallisation for dextrin + bentonite-bonded composite pellets.



Figure 21. Main effects plot of coke quantity, reduction temperature and reduction time on the degree of metallisation for CMC-bonded composite pellets.

The interactions plots of coke quantity, reduction temperature and reduction time are shown in Figures 22-24. There was very little interaction between coke quantity and reduction temperature and between coke quantity and reduction time on the degree of metallisation for all pellet types, as evidenced by the close to parallel lines. However, there was strong interaction between reduction temperature and reduction time on the degree of metallisation for all pellet types as seen from the converging lines. This corroborates that rates of heat and mass transfer play critical roles in the reduction rate of composite pellets as reported in previous studies (9, 43, 45 and 52). Higher degree of metallisation was observed at higher level of reduction temperature (1100 °C), increasing from low to high level of time (20 to 40 minutes).



Figure 22. Interaction plot of coke quantity, reduction temperature and reduction time on the degree of metallisation for dextrin-bonded composite pellets.



Figure 23. Interaction plot of coke quantity, reduction temperature and reduction time on the degree of metallisation for dextrin + bentonite-bonded composite pellets.



Figure 24. Interaction plot of coke quantity, reduction temperature and reduction time on the degree of metallisation for CMC-bonded composite pellets.

The Pareto plots of the standardised effects of the factors and their interactions on the degree of metallisation are shown in Figures 25-27. The reference line depicts where the alpha risk is 5 %. Factors that extend past the reference line are statistically significant to the degree of metallisation at 95 % confidence. Factors to the left of the reference line are statistically insignificant at 95 % confidence limit. Reduction temperature (B), reduction time (C) and the interaction between reduction temperature and reduction time (BC) were found to be the important factors on the degree metallisation of pellets made using dextrin and a combination of dextrin and bentonite. This observation was in agreement with the findings of the main effects and interaction plots.

For composite pellets containing CMC binder, the most important factor on the degree of metallisation was found to be the reduction temperature (B). Coke quantity (A), the interaction between coke quantity and reduction time (AC), the interaction between coke quantity and reduction temperature (AB), and interaction between coke quantity, reduction temperature and reduction time (ABC) were found to have a statistically insignificant effect on the degree of metallisation for all pellet types.



Figure 25. Pareto plot of coke quantity, reduction temperature and reduction time and their interactions on degree of metallisation for dextrin-bonded composite pellets.



Figure 26. Pareto plot of coke quantity, temperature and reduction time and interactions on degree of metallisation for dextrin and bentonite-bonded composite pellets.



Figure 27. Pareto plot of coke quantity, reduction temperature and reduction time and their interactions on degree of metallisation for CMC-bonded composite pellets.

These observations are further confirmed by the Normal Probability Plot of factor effects, Figures 28-30. Insignificant main and interaction effects lie on a straight line while the significant ones are scattered on both sides of the line (48). Reduction time (C), reduction temperature (B) and the interaction between reduction time and reduction temperature (BC) were found to be statistically significant at five per cent significance level for composite pellets constituted of, respectively, dextrin and a combination of dextrin and bentonite. At five per cent significance level, reduction temperature was the only significant factor for composite pellets with CMC. This was in agreement with the observations made on the main effect and interaction plots.



Figure 28. Normal Probability Plot of factor effects on degree of metallisation for dextrinbonded composite pellets.



Figure 29. Normal Probability Plot of factor effects on degree of metallisation for dextrin + bentonite composite pellets.



Figure 30. Normal Probability Plot of factor effects on degree of metallisation for CMCbonded composite pellets.

Cube plots for degree of metallisation of composite plots are shown in Figures 31-33. Higher values of the degree of metallisation were observed at high level settings of coke quantity, reduction temperature and reduction time. However the degree of metallisation did not significantly increase as reduction time increased from the low to high value. Consequently, one could choose the low level of reduction time to increase productivity.



Figure 31. Cube Plot of factor effects on degree of metallisation for dextrin-bonded composite pellets.



Figure 32. Cube Plot of factor effects on degree of metallisation for dextrin + bentonitebonded composite pellets.



Figure 33. Cube Plot of factor effects on degree of metallisation for CMC-bonded composite pellets.

The degree of metallisation of the composite pellets increased with an increase in coke quantity, reduction time and reduction temperature. Composite pellets constituted of CMC gave the highest degree of metallisation followed, respectively by pellets made from dextrin and the combination of dextrin and bentonite at all factors settings. At the high level of coke quantity (20 %) and the high level of reduction temperature (1100 °C), reduction time did not significantly affect the degree of metallisation of composite pellets made using CMC (95.52 % and 95.61 %). Low level setting of reduction time (20 minutes) is the better choice to increase productivity.
4.5 Decrepitation index of reduced composite pellets

The decrepitation indices of the reduced composite pellets according to the reduction temperature, the reduction time and the binder used during pelletizing are shown in Table 13 and Appendix 5. The decrepitation indices of all reduced composite pellets met the required specification of less than 5 % of particles below 0.5 mm (22). Decrepitation index values of pellet type P3 were slightly lower than those of pellet type P2. This could be attributed to the higher degree of metallisation as a result of increase in the coke quantity from 15 % to20 %, in respectively P2 and P3. Generally, composite pellets comprising of CMC had the lowest decrepitation indices, followed by those constituted from a combination of dextrin and bentonite, and dextrin respectively. This is agreement with CMC having the highest degree of metallisation for the same reducing conditions.

It is known that at temperatures below 500 °C organic binders already start to decompose and burn off (23 and 36). A general concern therefore exists that the use of organic binders creates operational problems due to loss in strength and decrepitation of the pellets in this low temperature range. Cold-bonded self-reducing pellets are therefore not expected to be fed to rotary kiln and shaft kiln furnaces where the iron bearing feed stocks need to have high load-bearing strength. In this study however, the pellets were reduced in a stagnant bed. On removal of the pellets from the bed, after reduction at either 1000 or 1100 °C, no signs of significant decrepitation were observed. Clear evidence of decrepitation of the pellets at temperatures below 500 °C could therefore not be found.

Pellet Type	Temperature (°C)	Time (min)	Decrepitation indices and standard deviations (%) *					
			Dextrin	σ#	Dextrin + Betonite	σ	CMC	σ
	1000	20	0.76	0.05	1 22	0.03	0.3/	0.05
F Z	1000	20	0.70	0.05	1.22	0.05	0.54	0.05
P2	1000	40	0.46	0.03	0.09	0.01	0.18	0.03
P2	1100	20	0.08	0.00	0.00	0.02	0.08	0.10
P2	1100	40	0.08	0.02	0.08	0.00	0.06	0.00
Р3	100	20	0.70	0.00	1.03	0.04	0.29	0.02
Р3	1000	40	0.40	0.01	0.08	0.02	0.12	0.02
Р3	1100	20	0.08	0.01	0.08	0.00	0.06	0.00
Р3	1100	40	0.08	0.00	0.08	0.00	0.06	0.00

 Table 13. Decrepitation indices of the reduced composite pellets (%).

*: Average of two tests *: Standard deviation

4.6 Effects of coke, reduction temperature and reduction time on decrepitation index of reduced composite pellets

The main effect plots of coke quantity, reduction time and reduction temperature on the decrepitation index are shown in Figures 34-36. The decrepitation index was found to decrease from a low to a high level of coke quantity, reduction temperature and reduction time. Again the effects of reduction time and reduction temperature on decrepitation were pronounced while coke quantity had little effect. The lowest decrepitation index was associated with pellets constituted of CMC binder. This is in agreement with these pellets having the highest degrees of metallisation.





the decrepitation index for dextrin-bonded composite pellets.



Figure 35. Main effects plot of coke quantity, reduction temperature and reduction time on the decrepitation index for dextrin + bentonite-bonded composite pellets.



Figure 36. Main effects plot of coke quantity, reduction temperature and reduction time on the decrepitation index for CMC-bonded composite pellets.

Interaction plots for the factors are shown in Figures 37-39. There was insignificant interaction between coke quantity and reduction temperature on the decrepitation index of all composite pellets. Insignificant interaction was observed between coke quantity and reduction time for composite pellets made using dextrin and a combination of dextrin and bentonite. However there was marked interaction between reduction temperature and reduction time on decrepitation for all pellets. Lower decrepitation index values were observed at 1100 °C (higher level of temperature), decreasing with an increase in reduction time for CMC.



Figure 37. Interaction plot of coke quantity, reduction temperature and reduction time on the decrepitation index for dextrin-bonded composite pellets.



Figure 38. Interaction plot of coke quantity, reduction temperature and reduction time on the decrepitation index for dextrin + bentonite-bonded composite pellets.



Figure 39. Interaction plot of coke quantity, reduction temperature and reduction time on the decrepitation index for CMC-bonded composite pellets.

The Pareto plot of effects and their interactions on the decrepitation index are shown in Figures 40-42. The reference line again depicts where the alpha risk is 5 %. Factors that extend past the reference line are statistically significant to the degree of metallisation at 95 % confidence. Factors to the left of the reference line are statistically insignificant at 95 % confidence limit. For pellets made using dextrin and a combination of dextrin and bentonite, reduction temperature and reduction time and their interaction proved to be the most important factors. This is in agreement with observations made on main effect and interaction plots for these pellets. Reduction temperature was again confirmed as the most important factor for pellets made using CMC as binder.



Figure 40. Pareto plot of coke quantity, reduction temperature and reduction time and their interactions on decrepitation index for dextrin-bonded composite pellets.



Figure 41. Pareto plot of coke quantity, reduction temperature and reduction time and their interactions on decrepitation index for dextrin + bentonite-bonded composite pellets.





The findings from the Pareto plot are compared with the observations from the Normal Probability Plot of factor effects in Figures 43-45. Normal Probability Plot of factor effects on decrepitation index confirmed the observations made from the Pareto plot of factors. Reduction time and reduction temperature and their interaction were found to be the statistically significant factors at five per cent significance level for composite pellets made using dextrin and a combination of dextrin and bentonite as binder. Reduction temperature was also found to be the only significant factor for pellets made from CMC as binder.



Figure 43. Normal Probability Plot of factor effects on decrepitation index for dextrin bonded composite pellets.



Figure 44. Normal Probability Plot of factor effects on decrepitation index for dextrin + bentonite-bonded composite pellets.



Figure 45. Normal Probability Plot of factor effects on decrepitation index for CMC-bonded composite pellets.

In terms of dry strength of composite pellets, a combination of dextrin and bentonite was the best binder, followed by dextrin and CMC respectively. CMC-bonded composite pellets reduced to the highest degree of metallization, followed by dextrin and a combination of dextrin and bentonite.

Dextrin costs R18.24/Kg (G Ndou, personal communication, November 17, 2015) and CMC costs R23.40/kg (T Mutongoreya, personal communication, November 18, 2015). CMC is regarded to be the best binder in terms of strength, metallisation and cost, considering its dosage level of 1-2 % compared to 3-4 % of the raw pellet mixture for dextrin.

5 Conclusions

The study was set out to examine the feasibility of producing cold-bonded self-reducing composite pellets from Sishen upgrade slimes, coke fines and several binders. The study has also sought to identify the binder that economically imparts the highest dry strength and the highest degree of metallization on the composite pellets. The effects of coke and binder quantities on the dry strength of the composite pellets was also investigated together with the effects of coke quantity, reduction temperature and reduction time on the degree of metallization index of the reduced pellets.

This study confirmed that cold-bonded self-reducing composite pellets of sufficient dry strength can be produced by mixing upgraded Sishen slimes and coke using various binders. These pellets reduced rapidly in an electrically heated furnace to give DRI of high degree of metallization and excellent decrepitation indices.

A combination of dextrin and bentonite, dextrin and CMC produced composite pellets that met the required minimum dry strength of 300 N/pellet. Calcium-Lignosulphonate, however, could not produce composite pellets meeting the required dry strength. Of the binders studied, the combination of dextrin and bentonite produced composite pellets with highest dry strength, followed by dextrin, CMC and calcium-lignosulphonate respectively.

The composite pellets reduced rapidly to attain between 65 and 96 % metallization within 20 and 40 minutes at 1000 and 1100 °C. Composite pellets type P2 failed to reduce to minimum required metallisation degrees of 90 % at 1000 °C at both levels of reduction time for pellets made using dextrin and a combination of dextrin and bentonite as binders. However pellets made using CMC binder reduced to 91 % metallisation at a higher level of time (40 minutes) at 1000 °C.

Composite pellets of Type P2 exceeded 90 % metallisation at 1100 °C at both levels of time (20 and 40 minutes) except for pellets containing a combination of dextrin and bentonite that were reduced for 20 minutes.

Composite pellets of type P3 met the required minimum percentage metallization at the high level of temperature (1100 °C) and both levels of time (20 and 40 minutes). The decrepitation indices of all reduced composite pellets met the required specification of less than 5 % of particles below 0.5 mm. Decrepitation index values of pellet type P3 were

slightly lower than those of pellet type P2. This could be attributed to the higher degree of metallisation as a result of increase in the coke quantity from 15 to 20 %, in respectively P2 and P3. Composite pellets comprising of CMC had the lowest decrepitation indices, followed by those constituted from a combination of dextrin and bentonite, and dextrin respectively. This is agreement with CMC having the highest degree of metallisation for the same reducing conditions.

The dry strength of the composite pellets increased with an increase in binder quantity, but decreased with an increase in coke quantity from 15-20 % of the raw pellet mix. The decrease in the dry strength of the pellets with an increase in coke quantity could be attributed to the increase in hydrophobic material (carbon) in the raw pellet mix. The degree of metallisation was found to increase with an increase in coke quantity, reduction temperature and reduction time. The effects of reduction temperature and reduction time. The effects of reduction temperature and reduction time. The effects of reduction temperature and reduction time, on metallisation, were more pronounced as compared to the effect of coke quantity. This shows that rates of heat and mass transfer play critical roles in the reduction rate of composite pellets.

The decrepitation index was found to decrease from a low to a high level of coke quantity, reduction temperature and reduction time. The effects of reduction time and reduction temperature on decrepitation were more pronounced while coke quantity had little effect. The lowest decrepitation index was associated with pellets constituted of CMC binder, which is in agreement with these pellets having the highest degrees of metallisation. CMC was found to be the most suitable binder as dry strengths in excess of 300 N/pellet, metallisation up to 95.5 % after 20 minutes at 1100 °C and decrepitation indices below 0.1 % were obtained. Only low concentrations of CMC are required in these pellets, which mean that the iron content of the pellet is not notably diluted and the sulphur concentration in the pellet is low. Of the examined binders CMC is therefore also the most economical. This study therefore contributes to the body of knowledge in the field of direct reduction of iron ores. Significant savings can be realised by producing DRI from cold-bonded selfreducing pellets as compared to the traditional way of using indurated pellets. Higher productivity can also be achieved by charging cold-bonded self-reducing pellets which reduce rapidly to attain high degree of metallization. The use of organic binders also eliminates the issue of dilution of pellets with silica and alumina which is prevalent with the use of bentonite.

6 Recommendations for future work

Having discovered that it is feasible to produce DRI of good degree of metallisation from Sishen upgraded slimes, it is important to investigate this finding on plant trial scale. If confirmed, further laboratory work is needed to investigate if composite pellets of sufficient strength can be produced at lower binder levels since these promising binders imparted dry strengths much greater than the targeted value. This could result in saving on binder costs resulting in Anglo American Kumba Iron Ore realising more profits.

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Appendices

Appendix 1

	Sample A	Sample B	Average	σ
SiO ₂	3.82	3.55	3.68	0.19
TiO ₂	0.13	0.13	0.13	0.00
Al ₂ O ₃	2.7	2.91	2.81	0.15
Fe ₂ O ₃	92.73	92.76	92.74	0.02
MnO	0.06	0.06	0.06	0.00
CaO	0.12	0.12	0.12	0.00
K₂O	0.24	0.27	0.26	0.02
P ₂ O ₅	0.12	0.14	0.13	0.01
Cr ₂ O ₃	0.02	0.01	0.01	0.01
V_2O_5	0.02	0.01	0.02	0.01
SO₃	0.01	0.02	0.01	0.01
Cl	0.02	0.01	0.01	0.01
Co ₃ O ₄	0.01	0.01	0.01	0.00
SrO	0.02	0.01	0.01	0.01

XRF of KIO Sishen Slimes (wt %)

Appendix 2

Size,	Cumulative % undersize								
μm	Run 1	Run 2	Run 3	Average	σ				
1905	100	100	100	100	0.00				
1660	100	100	100	100	0.00				
1455	100	100	100	100	0.00				
1259	100	100	100	100	0.00				
1096	100	100	100	100	0.00				
955	99.9	100	100	100	0.06				
832	99.8	99.9	100	99.9	0.10				
724	99.6	99.7	100	99.8	0.21				
631	99.4	99.4	99.9	99.6	0.29				
550	99.1	99	99.8	99.3	0.44				
479	98.6	98.5	99.5	98.9	0.55				
417	98	97.8	99.1	98.3	0.70				
363	97.1	97.1	98.5	97.6	0.81				
316	96.1	96.4	97.8	96.8	0.91				
275	95	95.6	97	95.9	1.03				
240	93.8	94.7	96	94.8	1.11				
209	92.4	93.6	94.8	93.6	1.20				
182	90.8	92.4	93.4	92.2	1.31				
158	89.1	90.9	91.8	90.6	1.37				
138	87.1	89.1	90	88.7	1.48				
120	84.8	87	87.8	86.5	1.55				
105	82.3	84.5	85.2	84	1.51				
91	79.5	81.6	82.4	81.2	1.50				
79	76.5	78.5	79.2	78.1	1.40				
69	73.1	75.1	75.8	74.7	1.40				
60	69.6	71.4	72.1	71	1.29				
52	65.8	67.5	68.2	67.2	1.23				
46	62	63.4	64.1	63.2	1.07				
40	57.9	59.1	59.8	58.9	0.96				
35	53.7	54.7	55.3	54.6	0.81				
30	49.4	50.2	50.7	50.1	0.66				
26	45	45.6	46	45.5	0.50				
23	40.6	41	41.3	41	0.35				
20	36.1	36.4	36.7	36.4	0.30				

Size distribution of KIO Sishen Slimes

17	31.8	32	32.2	32	0.20
15	27.6	27.7	28	27.8	0.21
13	23.7	23.8	24	23.8	0.15
11	20.1	20.3	20.5	20.3	0.20
10	17	17.1	17.4	17.2	0.21
8.7	14.3	14.4	14.7	14.5	0.21
7.6	12	12.1	12.4	12.2	0.21
6.6	10.1	10.3	10.5	10.3	0.20
5.8	8.6	8.8	9	8.8	0.20
5	7.4	7.6	7.8	7.6	0.20
4.4	6.5	6.6	6.8	6.6	0.15
3.8	6	5.8	6	5.9	0.12
3.3	5	5.1	5.3	5.1	0.15
2.9	4.5	4.6	4.7	4.6	0.10
2.5	4	4	4.1	4	0.06
2.2	3.5	3.6	3.6	3.6	0.06
2	3	3.1	3.2	3.1	0.10
1.7	2.6	2.7	2.7	2.7	0.06
1.4	2.2	2.3	2.3	2.3	0.06
1.3	2	2	2	2	0.00
1.1	1.6	1.6	1.6	1.6	0.00
1	1.3	1.3	1.3	1.3	0.00
0.8	1	1.1	1.1	1.1	0.06
0.7	0.8	0.8	0.9	0.8	0.06
0.63	0.6	0.6	0.6	0.6	0.00
0.55	0.4	0.4	0.5	0.4	0.06
0.48	0.3	0.3	0.3	0.3	0.00
0.42	0.2	0.2	0.2	0.2	0.00
0.36	0.1	0.1	0.1	0.1	0.00
0.32	0	0	0	0	0.00

Appendix 3

	Pellet 1	Pellet 2	Pellet 3	Pellet 4
1	320	624	537	366
2	361	677	514	354
3	355	666	579	321
4	371	690	512	331
5	345	654	553	340
6	372	634	525	365
7	411	633	583	345
8	319	633	570	311
9	380	649	537	305
10	399	710	511	339
11	398	676	519	345
12	380	752	562	341
13	365	715	540	422
14	346	691	511	308
15	411	662	530	351
16	338	647	517	310
17	399	742	570	355
18	373	682	508	321
19	376	742	551	303
20	384	661	567	328
Average	370	677	540	338
σ	27.09	38.64	25.36	27.97

Dry strength of composite pellets (N/pellet)

Dextrin

	Pellet 1	Pellet 2	Pellet 3	Pellet 4
1	617	951	943	548
2	638	851	800	478
3	592	867	756	463
4	609	829	783	502
5	581	866	802	480
6	570	912	808	500
7	569	848	842	491
8	578	807	798	463
9	596	842	773	499
10	549	961	822	501
11	536	838	833	473
12	560	887	787	464
13	552	952	836	477
14	534	914	830	475
15	579	911	789	456
16	580	881	787	438
17	583	865	747	473
18	567	817	760	450
19	596	869	736	504
20	582	812	836	456
Average	578	874	803	480
σ	25.94	46.77	45.33	24.91

Dextrin and Bentonite

C	ΝЛ	C
L	IVI	L

	Pellet	Pellet	Pellet	Pellet
	1	2	3	4
1	376	574	527	420
2	381	512	497	414
3	367	518	575	353
4	411	494	580	385
5	349	507	491	358
6	415	473	525	409
7	357	574	486	365
8	357	526	531	415
9	388	583	496	382
10	377	549	475	385
11	381	534	474	439
12	426	566	548	322
13	378	584	564	384
14	390	503	474	397
15	354	564	483	386
16	391	554	598	358
17	462	502	553	364
18	357	506	506	326
19	382	506	501	339
20	371	494	489	362
Average	384	531	519	378
σ	27.70	34.41	39.02	31.70

	Pellet	Pellet	Pellet	Pellet
	1	2	3	4
1	57	67	57	47
2	41	107	53	59
3	110	78	62	58
4	49	109	42	57
5	71	74	87	50
6	90	93	61	54
7	55	84	63	56
8	53	58	57	88
9	63	57	61	75
10	49	80	49	35
11	38	45	76	64
12	64	77	59	41
13	47	72	75	40
14	41	112	48	56
15	48	66	64	54
16	52	75	59	41
17	58	89	58	57
18	47	77	55	55
19	45	53	65	42
20	36	83	72	34
Average	56	78	61	53
σ	17.86	18.14	10.44	13.09

Calcium-lignosulphonate

Appendix 4

Pellet	Temperature	Time	Sample	Sample	Average	σ
type	(°C)	(min)	1	2		
P2	1000	20	68.01	66.97	67.49	0.74
P2	1000	40	88.79	90.03	89.41	0.88
P2	1100	20	92.16	92.74	92.45	0.41
P2	1100	40	95.05	93.87	94.46	0.83
P3	1000	20	70.46	71.04	70.75	0.41
P3	1000	40	90.01	90.43	90.22	0.30
P3	1100	20	94.56	95.06	94.81	0.35
P3	1100	40	94.93	94.73	94.83	0.14

Degrees of metallisation of composite pellets (%) Dextrin

Dextrin and Bentonite

Pellet	Temperature	Time	Sample	Sample	Average	σ
type	(°C)	(min)	1	2		
P2	1000	20	65.76	65.02	65.39	0.52
P2	1000	40	86.06	87.1	86.58	0.74
P2	1100	20	87.93	88.75	88.34	0.58
P2	1100	40	95.114	94.23	94.67	0.63
P3	1000	20	69.43	70.25	69.84	0.58
P3	1000	40	89.46	88.36	88.91	0.78
P3	1100	20	90.03	91.09	90.56	0.75
P3	1100	40	94.03	93.69	93.86	0.24

CMC

Pellet	Temperature	Time	Sample	Sample	Average	σ
type	(°C)	(min)	1	2		
P2	1000	20	83.39	83.91	83.65	0.37
P2	1000	40	91.25	90.87	91.06	0.27
P2	1100	20	92.75	94.13	93.44	0.98
P2	1100	40	95.77	94.91	95.34	0.61
P3	1000	20	84.84	86.14	85.49	0.92
P3	1000	40	92.53	93.17	92.85	0.45
P3	1100	20	95.94	95.1	95.52	0.59
P3	1100	40	96.07	95.15	95.61	0.65

Appendix 5

Pellet	Temperature	Time	Sample	Sample	Average	σ
type	(°C)	(min)	1	2		
P2	1000	20	0.81	0.71	0.76	0.07
P2	1000	40	0.43	0.49	0.46	0.04
P2	1100	20	0.08	0.08	0.08	0.00
P2	1100	40	0.06	0.1	0.08	0.03
P3	1000	20	0.77	0.63	0.7	0.10
P3	1000	40	0.41	0.43	0.42	0.01
P3	1100	20	0.09	0.07	0.08	0.01
P3	1100	40	0.08	0.08	0.08	0.00

Decrepitation indices of composite pellets (%) Dextrin

Dextrin and Bentonite

Pellet	Temperature	Time	Sample	Sample	Average	σ
type	(°C)	(min)	1	2		
P2	1000	20	1.19	1.25	1.22	0.04
P2	1000	40	0.1	0.08	0.09	0.01
P2	1100	20	0.1	0.06	0.08	0.03
P2	1100	40	0.09	0.07	0.08	0.01
P3	1000	20	0.99	1.07	1.03	0.06
P3	1000	40	0.1	0.06	0.08	0.03
P3	1100	20	0.08	0.08	0.08	0.00
P3	1100	40	0.08	0.08	0.08	0.00

CMC

Pellet	Temperature	Time	Sample	Sample	Average	σ
type	(°C)	(min)	1	2		
P2	1000	20	0.29	0.39	0.34	0.07
P2	1000	40	0.21	0.15	0.18	0.04
P2	1100	20	0.07	0.09	0.08	0.01
P2	1100	40	0.06	0.06	0.06	0.00
P3	1000	20	0.31	0.27	0.29	0.03
P3	1000	40	0.14	0.1	0.12	0.03
P3	1100	20	0.06	0.06	0.06	0.00
P3	1100	40	0.06	0.06	0.06	0.00

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