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MSc (50/50) RESEARCH REPORT

Topic

An investigation into the use of borates to improve coke CSR/CRI when blending metallurgical coal to make blast furnace coke

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Submitted to

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DECLARATION

I declare that this research report is my own unaided work. It is being submitted for the degree of Master of Science in Engineering to the University of the Witwatersrand, Johannesburg, South Africa. It has not been submitted before for any degree or examination to any other University.

……
(Signature of Candidate)

……..… day of ……..…… 2018
(Day) (Month) (Year)

ABSTRACT

The availability of prime metallurgical coking coals is limited in Africa. This lack of good coking coals results in steel manufacturers (ArcelorMittal in particular) incurring high costs when importing the necessary prime coking coals from abroad. Such products are then mixed with local partially-coking "blend" coals. The need therefore is to find alternative methods to obtain substitutes for imported costly coking coals or to blend local coals in a manner that would achieve the same technical properties of products comprising prime coking coals in a blend. If achievable, this would have the added benefit of reducing expensive imported prime coking coals (mainly from Australia) and would ensure a more cost-effective product for use in the vital iron and steelmaking process in South Africa. Most imported coals from Australia are three times more expensive compared to local blend coking coals.

ArcelorMittal has been using blends that contain prime coking coals from Mozambique, North America and Australia and have so far only been able to incorporate up to a maximum of 30% of the local South African "blend" coking coals, these arising primarily from Grootegeluk Colliery, Lephalale in the Waterberg region of the country. Such low proportions of local content with high proportions of imported products have led to ever-increasing high costs in recent years, prompting research into blend optimisation to enable the incorporation of higher percentages of local coals to produce cheaper and more cost-effective blends. The current research, in attempting to address this issue, focused on the evaluation of coke qualities from blends that contained increased proportions of Grootegeluk blend coking coal (GG), ranging from 31% and incrementally up to 40 %. In each case, samples of the resultant coke products were split into separate sub-samples with one sub-sample quenched using normal water while another sub-sample was quenched by water containing a solution of sodium tetraborate. A similar process was used successfully in China and Russia and this yielded very positive coke CSR/CRI results.

Results showed that, for each blend with an incrementally increased proportion of GG coal, the coke strength properties improved by 17% when the sub-sample was quenched by the borate solution. There was no change in coke strength properties in the sub-samples quenched by water. The ultimate cut-off point at which the blend coke quality properties would still be usable in a blast-furnace process was achieved when the proportion of GG

coal in the blend was 37% after quenching by the borate solution. The 7% increase of GG coal in the blend from 30% is likely to lead to considerable saving in blend coking production due to the reduction of imported coking coal in the mix.

ACKNOWLEDGEMENTS

I would like to extend my sincere gratitude to Professor Rosemary Falcon for not only guiding me and offering me advice on how to do this research but also for instilling in me, the excitement and passion towards coal research.

I would also want to express my sense of gratitude to the support offered by Dr Samson Bada in the final review of my research report.

Lastly, I would like to thank ArcelorMittal for the financial support and for availing laboratory equipment which allowed me to undertake this work.

GLOSSARY

BF	Blast Furnace
GG	Grootegeluck (Colliery or coal)
M10	Micum drum Index 10
CSR	Coke Strength after Reaction
M40	Micum drum Index 40
F-Factor	Factor derived from results obtained in the Gieseler Fluidity test
CRI	Coke Reactivity Index
SI	Strength Index
CBI	Composition Balance Index
G-Factor	Factor derived from results obtained in the Dilatation test
TRD	True relative density
ARD	Apparent relative density
IGP	Internal gas pressure
OWP	Oven wall pressure
Dbcoal	Dry coal bulk density
CR	Coking rate (mm/hr)
T,w	Wall temperature
VM	Volatile matter
MF	Maximum fluidity
Tsoft	Softening temperature
Tres	Resolidification temperature
Tmf	Temperature at maximum fluidity
TIC	Total inert content

DEFINITIONS

I. Mean coke size– This is the arithmetic mean size of the coke determined by manual sizing of the coke over a specified series of screens with different sized apertures. Minimum sizes of 40mm up to a top size of 80mm are normally preferred for blast furnace operation.

II. Coke reactivity index (CRI) – Originally developed in Japan at Nippon Steel, this is a measure of the reactivity of coke which seeks to predict the behaviour of coke when it descends the shaft of a blast furnace under the load/burden of iron ore and limestone during the reduction for iron ore to iron. The measurement of CRI is determined after a 200g sample of +19-22mm is heated in an atmosphere of CO/CO2 at 1100 degrees Celsius for 2hrs with the resultant cooled product being subjected to 600 revolutions in an I-type drum. Material screened below 10mm represents the CRI index of that coke. ArcelorMittal standards require a maximum of 23 for an acceptably reactive coke.

III. Coke strength after reaction (CSR) – The CSR measurement is quantified by the amount of coke above 10mm from the CRI test. ArcelorMittal standards require a minimum of 60 for a good CSR of the coke.

IV. Micum tests - This test is undertaken when 50kg of coke is loaded into a clean micum drum with particle sizes of above 30mm. The drum is set to rotate for 100 revolutions at a speed of 25revolutions per minute. The drum is set to stop after 100 revolutions, and the coke offloaded and screened through mats of 10mm, 20mm, 30mm and 40mm for the determinations of various micum indices; namely M10 (Abrasion Index which is quantified by percentage of material below 10mm) and M40 for material above 40mm.

V. IRSID test – To quantify this index, a further 400 revolutions is added to the micum test and the material then undergoes screen analysis conducted over 10mm, 30mm and 40mm mats.

VI. M40 – This is the percentage of coke remaining on the +40 mm screen after 100 revolutions.

VII. M10 - commonly referred as the abrasion index test, this is represented by the mass of coke material collected after passing the – 10 mm screen mats after 100 revolutions.

VIII. Wall pressure - This is the pressure emanating from the coal during the early stages of carbonisation when the coal mass, under temperature, fluidises and expands before it reaches its contraction phase. Good coking coals should not exceed 10Kpa of pressure.

IX. Maximum fluidity - This is a test which determines the plastic range of coals. Name. It is essentially a measure of the difference between the initial softening and re-solidification temperatures. The plastic range, reported as temperatures (^{O}C), and the maximum fluidity, as reported by dials per minute (DDPM), are key factors in determining which blends of coals will be optimal for coking.

X. Roga index – This index indicates the ability of coal to agglutinate or fuse and bind with "inert" material. It is also useful in determining the extent to which coke has undergone surface oxidation (figures above 70 are usually preferable, with values lower than this pointing to the effects of oxidation).

XI. Coke stabilisation - This is a process whereby coke is broken by dropping it onto a hard surface from a height of at least six meters. This enables the generation of coke particles which do not undergo further physical/mechanical changes.

XII. Fixed Carbon - This refers to the amount of carbon material left in coal or coke after moisture, volatile matter and ash contents have been removed from the original coal/coke sample. It is determined by difference from 100%.

XIII. Apparent porosity – This is a measure of the volume contained in pores within coal or coke, i.e. those that are in contact with the surface. It does not include the volume contained in sealed pores.

XIV. True porosity – This is a measure of the volume of all pores in coal or coke including the volume found in sealed pores.

Table of Contents

DECLARATION ..... **I**

ABSTRACT ..... **II**

ACKNOWLEDGEMENTS ..... **IV**

GLOSSARY ..... **V**

DEFINITIONS ..... **VI**

LIST OF FIGURES ..... **X**

LIST OF TABLES ..... **XI**

CHAPTER 1: INTRODUCTION **1**

1.1 BACKGROUND 1
1.2 MOTIVATION 2
1.3 OBJECTIVES 3
1.4 HYPOTHESIS 3
1.5 KEY QUESTIONS TO BE ADDRESSED 4

CHAPTER 2: LITERATURE REVIEW **5**

2.1 INDUSTRIAL PRODUCTION OF COKE 5
2.2 CRITICAL COKE CHARACTERISTICS FOR THE BLAST FURNACE 7
2.2.1 Summary of coke behaviour in a blast furnace *9*
2.2.2 CSR/CRI of coke *10*
2.2.3. Coal/coke wall pressure *13*
2.3 SULPHUR IN COAL/COKE 16
2.4 PHOSPHORUS CONTENT IN COKE 17
2.5 ASH IN COKE 17
2.6 THEORY BEHIND BORATE ADDITION ON COKE 18
2.7 SUMMARY OF THE LITERATURE REVIEW 21

CHAPTER 3: MATERIALS AND METHODS **22**

3.1 FEED COALS USED IN THE COKE BLENDS. 22
3.1.1 Breakdown of the test blends *22*
3.1.2 Tests done on the coke blends *23*
3.2 FACILITIES USED IN THE RESEARCH 23
3.3 SAMPLING METHODS 24
3.3.1 Sampling from coal trucks *24*
3.3.2 Sampling from a heap *24*
3.3.3 Sample preparation for Proximate Analyses and X-ray Analysis *25*
3.4 PILOT PLANT TESTS FOR THE BLENDED COALS 27
3.4.1 Sample preparation for the pilot plant oven tests *27*
3.4.2 Loading of coal into pilot plant oven *27*
3.4.3 Coking of the coal inside the pilot plant oven *29*
3.4.4 Pushing of coke out of the pilot plant oven *30*

3.4.5 Quenching of the coke. *31*
3.4.6 Stabilizing of the coke after quenching *33*
3.5 TESTING COKE FOR COLD STRENGTH (IRSID INDICES TESTS) 35

CHAPTER 4: RESULTS AND DISCUSSION 37

4.1. COAL COMPONENT ANALYSIS 37
4.2 EFFECT OF BORATE QUENCHING ON COKE CRI 38
4.3 EFFECT OF BORATE QUENCHING ON COKE CSR 39
4.4 EFFECT OF BORATE QUENCHING ON SULPHUR IN COKE 41
4.5 EFFECT OF BORATE QUENCHING ON COKE M_{10} 42
4.6 EFFECT OF BORATE QUENCHING ON DRY FIXED CARBON (DFC) 44
4.7 EFFECT OF BORATE ON COKE ASH CONTENT 45
4.8 EFFECT OF BORATE ON I40 46
4.9 EFFECT OF BORATE QUENCHING ON PHOSPHORUS CONTENT IN THE COKE 47
4.10 EFFECT OF INCREASE IN GG ON COKE OVEN WALL PRESSURE 48
4.11 POROSITY TESTS ON NORMAL AND BORATE TREATED COKE 49
4.12 SUMMARY OF TESTS RESULTS AND DISCUSSIONS 50
4.13 TECHNO-ECONOMIC EVALUATION 50
4.13.1 The coke making value chain (Industrial scale) *51*
4.14 LIMITATIONS OF THE RESEARCH 53

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS 54

5.1. CONCLUSIONS 54
5.2. RECOMMENDATIONS 55

REFERENCES 56

APPENDICES 59

APPENDIX 1 - SINGLE COMPONENT TESTS ON GROOTEGELUK COAL 59
APPENDIX 3. LINKS TO SODIUM TETRABORATE OCTAHYDRATE (MSDS) 64

LIST OF FIGURES

FIGURE 1: "COKE SIDE" OF A BY-PRODUCT COKE OVEN BATTERY [SOURCE: HTTPS://WWW.STEEL.ORG]. **(SEE REFERENCES)** 6
FIGURE 2: INCANDESCENT COKE IN THE OVEN WAITING TO BE "PUSHED". [ADAPTED FROM **HTTPS://WWW.STEEL.ORG/] (SEE REFERENCES)** 7
FIGURE 3: SCHEMATICS OF THE INSIDE OF A BLAST FURNACE [ADAPTED FROM ISPATGURU.COM] 9
FIGURE 4: MAIN FACTORS INFLUENCING THE COKE STRENGTH AFTER REACTION WITH CARBON DIOXIDE AT 1100 DEGREES CELCIUS (DIEZ ET AL., 2002). 12
FIGURE 5: THE MECHANISM OF COKING PRESSURE GENERATION (ROSHKOVA AND GUELTON, 2014) 14
FIGURE 6: MIXING DRUM FOR THE CRUSHED COAL 27
FIGURE 7: PUSHING OF COKE OUT OF THE PILOT OVEN 31
FIGURE 8: EQUIPMENT FOR THE PREPARATION OF THE BORATE SOLUTION FOR QUENCHING 32
FIGURE 9: EQUIPMENT FOR THE PREPARATION OF THE BORATE SOLUTION FOR QUENCHING 32
FIGURE 10: NORMAL QUENCHING OF THE COKE BY NORMAL QUENCH WATER 33
FIGURE 11: STABILISATION OF THE COKE BY USE OF THE STABILISING CHUTE 34
FIGURE 12: IRSID TEST DRUM 35
FIGURE 13: CRI UNITS VERSUS PERCENTAGE PROPORTION OF GG IN THE TABLE AND GRAPH FORMAT 39
FIGURE 14: CSR VERSUS PERCENTAGE GG IN THE BLEND (TABLE AND GRAPHIC FORM) 41
FIGURE 15: SULPHUR PERCENTAGE IN COKE VERSUS PERCENTAGE SULPHUR IN BLEND TABLE AND GRAPHICALLY PRESENTED. 42
FIGURE 16: SULPHUR PERCENTAGE IN COKE VERSUS PERCENTAGE SULPHUR IN BLEND TABLE AND GRAPHICALLY PRESENTED. 43
FIGURE 17: DFC VERSUS GG PERCENTAGE IN THE BLEND – TABLE ABOVE AND GRAPHICALLY PRESENTED 44
FIGURE 18: ASH CONTENT VERSUS GG PERCENTAGE IN THE BLEND (TABLE AND GRAPHICALLY PRESENTED) 45
FIGURE 19: I40 VALUES VERSUS PERCENTAGE GG IN THE BLEND (TABLE AND GRAPHICALLY PRESENTED) 47
FIGURE 20: PHOSPHORUS CONTENT VERSUS GG PERCENTAGE (TABLE AND GRAPHICALLY PRESENTED) 48
FIGURE 21: WALL PRESSURE VS PERCENTAGE GG IN BLEND (TABLE AND GRAPHICALLY PRESENTED) 49
FIGURE 22: SKETCH OF THE WATER CIRCULATION FOR INDUSTRIAL WET QUENCHING OF COKE 51

LIST OF TABLES

TABLE 1: BLEND PROPORTIONS OF THE DIFFERENT COAL COMPONENTS 22
TABLE 2: EQUIPMENT AND FACILITIES USED IN RESEARCH 23
TABLE 3: COAL COMPONENT ANALYSIS 37
TABLE 4: ARCELORMITTAL COAL CLASSIFICATION 38
TABLE 5: RESULTS FOR POROSITY TESTS ON BORATE AND NORMAL COKE 49
TABLE 6. TABLE 6: COST BENEFIT ANALYSIS CALCULATION 52
TABLE 7: BLEND PRICES WITH PERCENTAGE INCREASE IN GG 52

CHAPTER 1: INTRODUCTION

1.1 Background

Africa has reserves of coal which do not produce good quality metallurgical coke. Steel manufacturers in Africa in general (and South Africa in particular) have to rely on coals from Australia. To achieve the requisite qualities of the metallurgical coke, the blending of prime coking coals with lower value semi- or blend-coking coal takes place. To produce high grade metallurgical grade coke for blast furnace use, the current blends in South Africa utilize up to 70% of imported coal (mainly from Australia) while the weaker market coke blends utilize up to 20% imported coke, i.e. 80% local coals.

A reduction in imported coal usage in the metallurgical coal blend would not only incur financial savings spent on producing blast furnace coke but would also reduce the risk of process stoppages emanating from unforeseen supply constraints from overseas mines. A prime example of such delays was the case in 2010 and 2011 when many mines in Australia cut-back on supply due to widespread floods which affected most of the country, especially its mining operations (Bloomberg business, 2014) and the global prime coking coal market. ArcelorMittal continues to rely on imported coals for most of its blast furnace requirements. This scenario has left ArcelorMittal South Africa with a possible risk of supply as any break in metallurgical coal deliveries from overseas (mainly Australia and USA) has the potential of affecting its steel production. The production unit in Newcastle has the capacity of producing and supplying close to two million tonnes of steel every year while its Vanderbijlpark unit has the capacity to produce just over three million tonnes of steel every year. The requisite supply of coal to make coke in support of this annual steel production is about 900 000 tonnes of coal per year for the Newcastle plant and 1.4 million tonnes of coal per year for the Vanderbijlpark plant.

Mozambique supplies coking coal which can be incorporated up to a maximum of only 15% in the blend. Amounts above 15% results in high ‘wall pressure’ characteristics which renders it ‘dangerous’ to use in high proportions as this might result in oven damage. The main South African coal used in the metallurgical coke blend is produced from the Grootegeluk Colliery in the Waterberg. The product is known as a semi-soft coking coal (SSCC) and is called the Grootegeluk (GG) product. This can be incorporated in the

metallurgical blend to a maximum of 32%. However, for the sake of safety, GG coal has only been incorporated up to a maximum of 30%. An increase above 30% has proved less effective as it starts to generate coke of unacceptable coke strength properties. Efforts are currently being made to investigate methods whereby higher proportions of the GG product can be incorporated into the metallurgical blend without reducing coking properties. One such technology developed by an investigative team in Russia is a system whereby borates of potassium or sodium are added to the water quenching the coke on discharge from coke ovens (Filatov, 2012). Results indicated that this process produced a marked improvement in coke hot strength (CSR) and coke reactivity (CRI) by up to seventeen percent. The technology was based on the addition of solutions of borates for quenching. The theory behind this technology was based on the adsorption of the borate molecules which provided a 'skin' on the coke layer reducing the penetration of oxygen into the carbon matrix of the coke thereby increasing its hot coke strength (CSR).

Against this background, the object of this research, therefore, is to test the effect of borate applications on South African coke production, and more specifically to evaluate the effect on coke strength and reactivity. The research seeks to determine the impact on CSR/CRI from borate quenching of the coke. If successful, this would result in the usage of higher percentages of local coal in the metallurgical blend and a realization of significant cost savings on the overall metallurgical coal blend.

1.2 Motivation

The motivation to undertake this research is to reduce the risk related to the current dependence in ArcelorMittal South Africa on imported prime coking coals which, in turn, has the ability to significantly affect the production and cost of iron and steel in this country. Developing a new approach and mechanism whereby local blast furnaces could be supplied with metallurgical coke manufactured from high proportions of local coals would lead to a far more secure and cost-effective situation for South African iron and steel industries. This would reduce the risk of plant shutdowns in the event of logistical difficulties related to the importation of metallurgical coal.

One way of doing this was to improve the strength properties of coke without altering the coal blend to a more expensive one. The use of borates in quenching water has been proven

to increase hot strength properties and offers opportunity to incorporate higher percentages of cheaper coals. In this research, GG, which is nearly 70% cheaper than the prime coals was increased significantly without major losses on strength properties.

1.3 Objectives

I. **To determine the effect of borate on coke strength** when quenching coke composed of higher proportions of local coal (GG) and reducing quantities of imported prime coking coal components. To achieve this, the following will be undertaken.

II. **To determine the optimum percentage of local blend coking coal** i.e. to establish what proportion of local Grootegeluk (GG) semi soft coking coal from the Waterberg region could be blended with high value metallurgical coal to produce coke of acceptable quality (hot and cold strength properties and chemical composition) for the blast furnace.

III. **To determine the optimum percentage of local blend coking coal** i.e. to what extent could the Grootegeluk (GG) semi soft coking coal from the Waterberg region be blended with high value metallurgical coal to produce coke of acceptable quality for the blast furnace? i.e. acceptable in terms of the hot and cold strength properties and the chemical composition.

IV. **To evaluate the potential reduction in cost of the final coke** produced; i.e. to calculate the financial impact when establishing the optimum GG local blend and borate treatment while still maintaining the correct qualities of the final coke product for the metallurgical industry.

1.4 Hypothesis

Borate treatment can improve coke properties when using coke blends with higher proportions of local GG blend coking coal. Significant savings can be achieved by reducing the proportion of imported international prime coking coals

1.5 Key questions to be addressed

I. Does borate treatment have a beneficial effect on a given coke blend with a known quantity of local South African blend coking coal [Grootegeluk (GG)]?

II. What are the effects of borate treatment with incrementally increased local GG blend coking coal proportions?

III. What is the optimum proportion of GG in the coke blends, with borate addition and without borate addition?

IV. What are the inhibiting elements/factors when GG is present without borate treatment in higher proportions?

V. What will the cost benefits be for including higher percentages of GG in future proposed coal blends?

CHAPTER 2: LITERATURE REVIEW

This Chapter presents a summary of the key issues surrounding coke making and reviews work conducted in this regard by others in recent times. Special emphasis was placed on the effect of borates on coke strength and how this technology can be applied in the South African context.

2.1 Industrial production of coke

Coke is the special carbonised product of metallurgical grade coals which is used as a reductant, heat source and solid porous support in furnaces used in the making of iron, steel and ferroalloys. The coals used in this process include *prime coking coals* which are low ash, relatively reactive, mid-range bituminous coals and *blend coking coals* which may be found in relatively close ranges of rank and with lower proportions of reactive organic matter contents. Prime coking coals have the capacity to soften, swell, fuse and bind other materials into a porous, hard, thick-walled product which is used to enable good peripheral and internal flow of air up the burden in the shaft of the blast furnace. Such materials maintain their integrity within the burden in furnaces until they reach liquid metal phase at which they decompose and the carbon once again reduces the remaining ore materials in that phase. Blend coking coals are used to extend the amount of coke produced by being mixed in with prime coking coal which adds special characteristics such as rank, maximum fluidity, wall pressure and shrinkage properties.

Coke is divided into *metallurgical cokes* in which the highest level of coke particle strength and integrity is required in the iron and steel industry, which is maintained until reaching the liquid metal phase. *Market coke* has poorer strength properties and is more reactive in the upper reaches of furnaces where such material is used in the gaseous rather liquid metal phase.

The coke-making process involves the heating of the coal to high temperatures (up to 1300°C) in an oxygen deficient atmosphere. This is termed carbonization process (Diez, 2002). The coke produced at ArcelorMittal is produced in wet-charge, by-product-recovery coke oven batteries as shown below in (Figure 1). The overseas mines are blended (mixed), pulverized, and sometimes ‘oiled’ (mixed with heavy oil) in order to achieve adequate bulk

density control. The blended coal is then charged into the many slot-type ovens wherein each oven shares a common heating flue with the adjacent oven. The heating of the charged coal in ovens initially generates steam at temperatures up to 150 degrees. Copious amounts of volatiles comprising of tar naphthenic compounds and light gases are generated from temperatures around 200 degrees. The remaining material forms a solid matrix of semi-porous carbon containing a few inorganic compounds (ash) arising from the minerals and inorganic elements (e.g. sulphur, phosphorus, alumina and silica) which would have occurred in the original coal. This solid product is referred to as coke. The volatiles are extracted from the top part of the free space above the coal mass in the oven and conveyed to a gas treatment plant for the recovery and processing of mainly crude tar and benzoyl. entire coke-making operation is comprised of the following steps.



Figure 1: "Coke Side" of a By-Product Coke Oven Battery [Source: https://www.steel.org]. **(see references)**

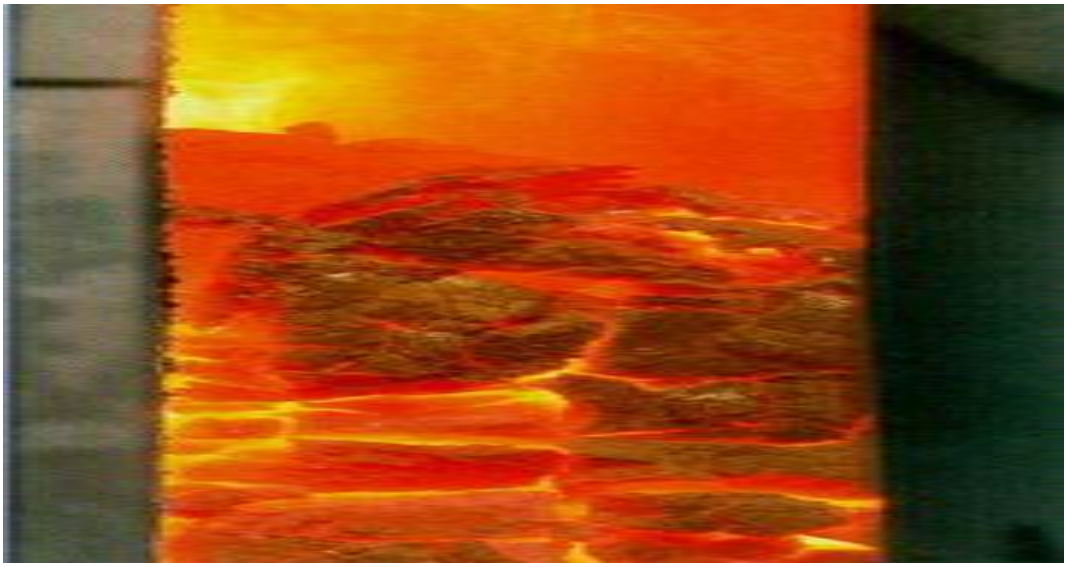


Figure 2: Incandescent coke in the oven waiting to be "pushed". [Adapted from ***https://www.steel.org/]*** **(see references)**

The coal-to-coke transformation phases are split generally into three; from 375°C to 475°C the water is vaporised and a plastic layer is generated which results in the expansion of the coal mass. From about 475°C to 600°C, heavy hydro-carbons and aromatic compounds are released and the re-solidification of the plastic mass occurs forming what is generally referred to as semi-coke. Between 600°C to 1100°C, there is contraction of the semi-coke resulting in the formation of the final structured solid carbon matrix known as coke. According to the American Iron And Steel Institute, 'the contraction of the coke allows the whole mass to 'pull' away from the oven walls as shown in figure 2 allowing the mass to be pushed more easily when the whole carbonisation process is completed'.

2.2 Critical coke characteristics for the blast furnace

Improvement of coke quality parameters in the blast-furnace operations results in significant reductions in coke consumption which allows for a cheaper production of hot-metal.

To improve parameters on hot strength such as CSR and CRI, one of the major challenges in steelmaking is to employ methods and techniques that would result in the reduction of energy inputs. In this regard, reduction of coke rate in the blast furnace and the substitution of the expensive coke with cheaper substitute such as pulverised coal and use of gas has gone long way in making the hot-metal production cost-effective.

Guoding (2010) observed that many steel mills in Europe, Asia, and America achieved reduced coke consumption of between 280 and 300 kg/t of pig iron by blowing up to 200 - 240 kg/t of pulverised fuel (PF) into a blast furnace. To achieve such a good range of coke rates, metallurgical coke plants need coke with CRI below 26.6 % and CSR in the range of 65 - 70%.

A high-quality metallurgical coke is required to be able to support the entire burden of ore and coke in a blast furnace. Good quality coke should allow smooth descent of that burden with as little degradation as possible. It must also provide the lowest amount of impurities, highest thermal energy, highest metal reduction and optimum permeability for flowage of gaseous and molten product through the burden. In order to test the capacity of cokes to provide such requirements, a coke hot strength testing device and method is necessary (Riley, 2013). A good metallurgical coke must fulfil the following requirements: the ability to apply and measure;

I. compressive force,
II. heating capability,
III. sample temperature,
IV. an inert atmosphere or vacuum to avoid gasification of coke during testing, and
V. sample size for statistical reliability.

Blast furnace dissections made in the course of early test work by Shiau *et al.* (2017) revealed a slight reduction in coke size in the shaft area, but a rapid degradation of both size and cold drum strength of the coke at around 3–5m above tuyere level. The estimated temperature around the area from tuyere level to 5m above was between 1400–1700°C. Such observations were valuable because cold coke strength is generally evaluated at room temperature and the effect of extremely high temperatures in the lower part of a blast furnace on coke strength is unknown in most industrial cases (Shiau *et al., 2017*).

In order to produce hot metal of the best quality, optimum blast furnace operations are required, run by the most experienced operators using the best operating parameters and the best materials. Coke is the most important raw material fed into the blast furnace in terms of its effect on blast furnace operation and hot metal quality. Valia (2017) describes a high-

quality coke as one that should be able to support a smooth descent of the blast furnace burden with as little degradation as possible while providing the lowest amount of impurities, highest thermal energy, highest metal reduction, and optimum permeability for the flow of gaseous and molten products as shown in figure 3. For this reason, the introduction of high quality coke to a blast furnace will result in lower coke consumption rate, higher productivity and lower hot metal cost. The definition of high grade metallurgical coke for the blast furnace (BF) is therefore a coke that is required to be resistant to abrasion, able to flow down the blast furnace shaft without breaking down, and, in this fashion, to be used economically.

2.2.1 Summary of coke behaviour in a blast furnace

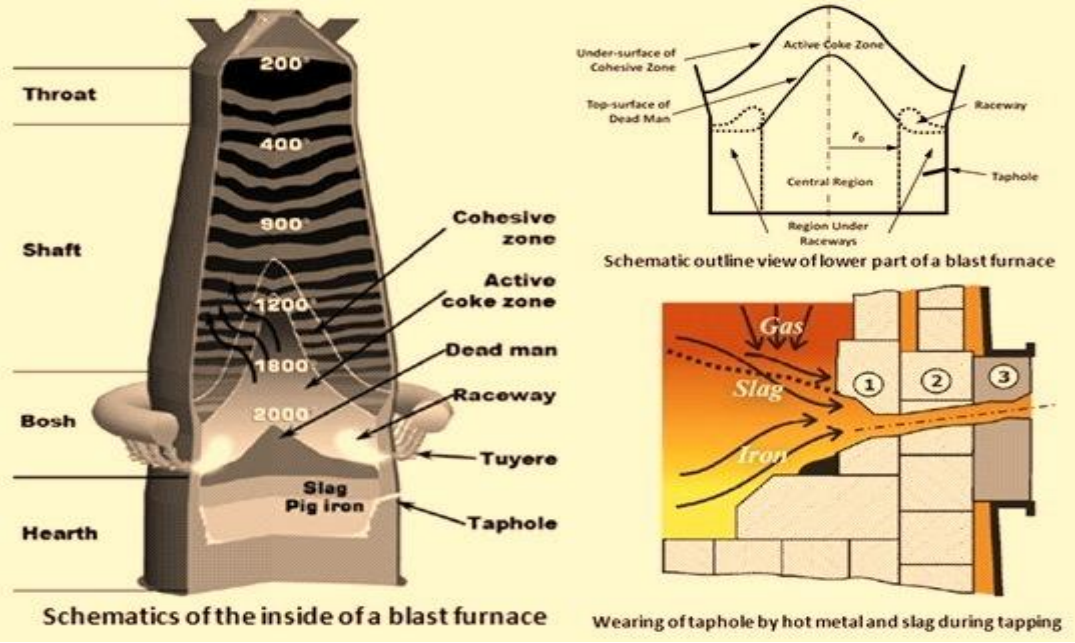


Figure 3: Schematics of the inside of a blast furnace [Adapted from ispatguru.com]

Several coke strength measurements are employed to determine and predict the behaviour of coke in a blast-furnace. Diez *et al.* (2002) stated that 'A realistic assessment of the likely performance of coke in the blast furnace operating with or without injection technology should include those properties of coke that reflect its resistance to degradation under the chemical and thermal environment of the blast furnace". This author lists guidelines for coke evaluation in which he includes determinations of lump size, particle shape, size uniformity, chemical composition, mechanical strength, and thermal and chemical

stabilities. Thus, coke for the blast furnace needs to be a successful compromise between structure and properties.

Hilding *et al.* (2005) added to the perceived requirements for coke evaluations stating that "coke reactivity is influenced by physical properties, including porosity as well as chemical characteristics including minerals in the coke and the carbon structure". These authors explain that reactions with oxidizing gases affect the porous carbon matrix during combustion/gasification. They explain that, as coke descends in a BF, its chemical structure is expected to change. The evolution of pore structure by growth and coalescence leads to increasing or decreasing available surface areas, changes in pore structure/distribution, gas diffusion and reactivity. However, the ultimate porous structure of coke is governed by the inherent properties of the coals comprising the coke. Hilding *et al.* (2005) state these properties are reflected particularly by the maximum fluidity and swelling number tests. To ensure good blast furnace performance, it is also claimed by Hilding *et al* (2005) that coke particles should be moderately large, with a narrow size range, and has a high mechanical strength in order to withstand the weakening reactions with carbon dioxide and alkali, abrasion, and thermal shock in the blast furnace.

Based on such recommendations and the many years of trial and error by many researchers, it is now possible to identify the most suitable properties for ideal metallurgical coke qualities (Hilding *et al.*, 2005). It is however, not possible to establish universal quality indices common to all blast furnaces due to the many unknown factors related to different designs and operations.

2.2.2 CSR/CRI of coke

A good measure of how the coke will behave in the blast furnace atmosphere is the hot strength measurement called *coke-strength-after-reaction* (CSR). This is a measure of coke strength after it has been heated in an atmosphere of carbon dioxide at 1100°C and then tumbled in an I-type drum at 20 revolutions per minute for 30 minutes. After this the product is passed over a -10mm screen. The weight loss after the reaction of the coke sample with carbon dioxide is quantified by a ratio called the *Coke Reactivity Index* (CRI) and the proportion retained on the 10mm screen is termed the *Coke Strength after Reaction* (CSR) index.

There have been many schools of thought pertaining to the usefulness of CSR/CRI as a measure for coke strength. Van Niekerk (1991) observed that arguments advanced in favour of the use of hot strength as a parameter to determine and predict coke performance relies on the knowledge that coke behaves quite differently at high temperatures from its norm at room temperature. Battacharyya (2014) describes the Coke Reactivity Index (CRI) and Coke Strength after Reaction (CSR) as internationally accepted testing standards for blast furnace cokes, as outlined in ISO 18894 (2006). Here, all coke samples (with and without added alkali) are tested under standardised conditions. For CRI, a test portion of the dried coke sample (200 ± 3 g) in a size ranging from 19,0 mm to 22,4 mm is heated in a reaction vessel to 1100°C in a nitrogen atmosphere. The coke sample is then subjected to an atmosphere of carbon dioxide for 2 hours. The coke (carbon) reacts with the carbon dioxide to form carbon monoxide. This is an endothermic reaction and known as Boudouard reaction, namely:

$C\ (solid) + CO_2\ (gas) = 2CO\ (gas)$

with ΔH_o rxn = +172,67 kJ/mol.

After this reaction period, the contents of the reaction materials are cooled down to 50°C in a nitrogen atmosphere. Following this reaction, coke material loss after the reaction in an atmosphere of carbon dioxide is quantified as CRI. After the coke is tumbled in a I-type drum for 600 revolutions minutes at 20 rpm the calculated mass having a particle size greater than 10 mm relative to the final reacted mass is defined as CSR (Battacharyya, 2014).

Most decisions regarding the rate at which coke is introduced into the blast furnace is dependent on its hot strength as governed and quantified by CSR/CRI values. Haapakangas (2013) notes that ‘coke with weak compressive hot strength also functioned poorly in a blast furnace causing strong kicking, rising pressure, and the plugging of blow pipes roughly 6–8 hours after charging. This indicated coke failure in the lower part of the blast furnace. Hot strength values correlated well with actual blast furnace operation whereas traditional tumbler strength tests, such as stability and hardness values, did not’.

For these reasons, the *hot strength of coke* is considered of major importance for successful blast furnace operation. However, research in hot strength tests have been conducted inhouse at the ArcelorMittal Newcastle plant where advanced facilities are available and where CSR/CRI tests have been undertaken routinely on a daily basis over many years (Kruger, 2017). On the basis of this work, the errors emanating from inexperience and unfamiliarity with the testing process were reduced to a bare minimum.

Strength is therefore one of the most important characteristics of coke. As the only solid material below the cohesive zone, coke is largely responsible for supporting the considerable mass of overhead material bed as well as maintaining good gas and liquid permeability in the blast furnace. Compression is therefore a further factor to be considered in assessing the suitability of a coke as this affects a coke's durability. Poor coke durability leads to breakage and the production of small coke lump sizes, which in turn impairs blast furnace operation and output.

In summary, Diez *et al.* (2002) listed the main factors affecting coke CSR, namely, coal rank, coal rheology, coal composition including inert organic and inert inorganic materials and coking conditions as shown below in figure 4 below.

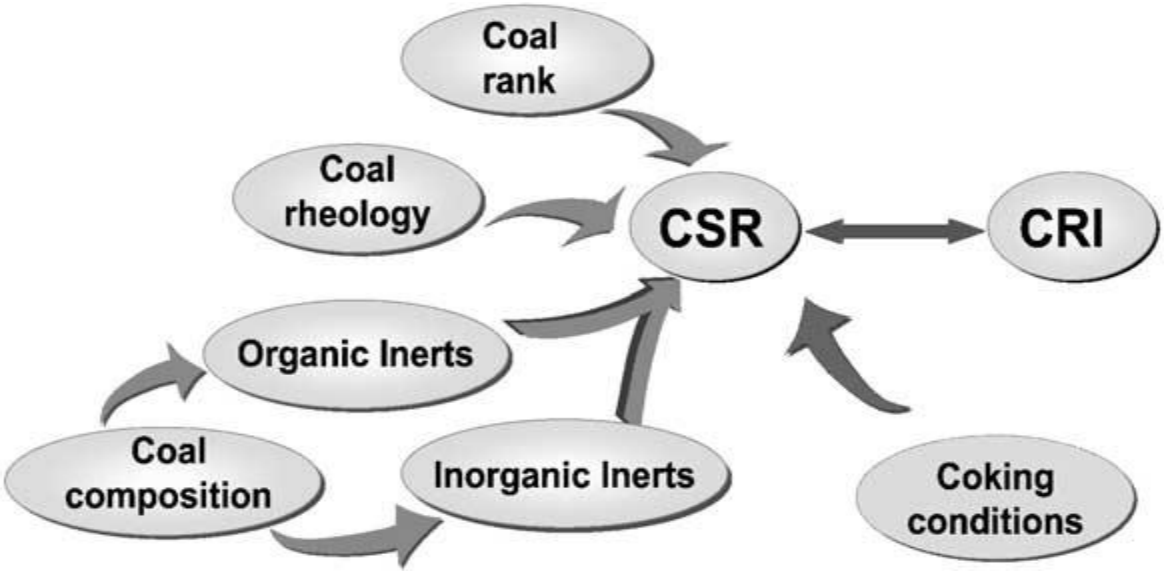


Figure 4: Main factors influencing the coke strength after reaction with carbon dioxide at 1100 degrees Celcius (Diez et al., 2002).

It should also be noted that there is inverse relationship between CSR and CRI. Diez et al, (2002) noted that 'from the almost linear correlation between CSR and CRI indices, it is deduced that the lower the reactivity the higher the coke strength. Catalytically accelerated coke reactivity lead to lower CSR values even if the coking coal blend has excellent rheological properties. It is from this understanding that the inhibition of coke reactivity in CO_2 led to technologies of borate usage as this would help improve coke CSR.

2.2.3. Coal/coke wall pressure

Duffy *et al.* (2010), in their research on blending for wall pressure reduction, observed that coal blending appears to offer the most beneficial means for reducing coking pressure, i.e. in most cases, the addition of 20–35% of a high volatile coal can significantly reduce the pressure generated by a dangerous highly swelling coal or coal blend without severely impacting coke quality. Similarly, and depending on their swelling characteristics, some low volatile coals can also be employed as pressure reducers, while coke breeze and semi-coke are often added for similar reasons.

2.2.3.1 Mechanism of oven wall pressure generation

Duffy *et al.* (2010) noted that 'Coal blending appears to offer the most beneficial means for reducing coking pressure, where in most cases the addition of 20–35% of a high volatile coal can significantly reduce the pressure generated by a dangerous coal or coal blend without severely impacting coke quality".

Evolution of volatile matter from the coal charge is a necessary function but on its own it is not sufficient to generate pressure in a conventional slot-type oven, since most of high volatile coals are usually low-pressure ones. Other causes and conditions for pressure to develop include the resistance to gas flow. It is now well established Roshkova (2014**)** that the restriction of volatiles released from two low permeable regions sandwiching the plastic layer can cause the volatiles to be trapped within the plastic coal. However, evolving gases must first escape the plastic layer itself before passing through the low permeable regions. Hence, there are three barriers to the movement of evolved gas, namely coal compaction after charging, plastic layer formation, and semi-coke formation. The mechanism of coking pressure generation is summarized in Figure 5 and the schematic cross-section of the charge in both cases of respectively high and low-pressure coals is shown.

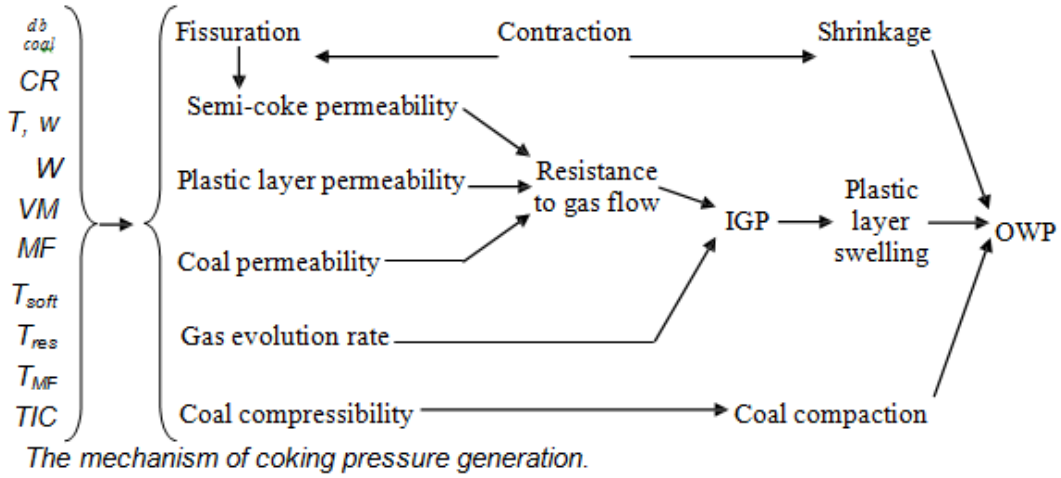


Figure 5: The mechanism of coking pressure generation (Roshkova and Guelton, 2014)

Roshkova (2014) noted that, upon continuous heating in the absence of oxygen, coking coal passes through a plastic state over a relatively narrow temperature range before transforming into a solid porous coke. This transient phase transformation, accompanied by a release of volatile matter (VM) is explained by two competitive reactions, respectively, i.e. decomposition and condensation. Coal softens at about *Tsoft*=400°C and then both fluidity and gas evolution increase until a maximum is reached at approximately 450°C. With increasing temperature, condensation takes over from decomposition and growth of large aromatic structures within the plastic mass develop leading to re-solidification near *Tres*=500°C. Gas evolution, which continues long after resolidification, causes the coke to contract. It is within this plastic temperature range that the fundamental coking processes, which convert granular coal into massive porous coke, take place. In a slot-type coke oven, plastic layers, delimited by the two isothermal planes *Tsoft* and *Tres*, are initiated at the two opposite walls of the oven then move inwards from the wall as a result of heat transfer and meet at the centre plane.

The plastic layer swells when gas evacuation proceeds slower than gas generation and, as a result of gas entrapment, *internal gas pressure* (IGP) builds-up in the plastic layer. Transmission of IGP to the walls by the surrounding semi-coke/coke gives rise to *oven wall pressure* (OWP) but *lateral shrinkage* (LS) may explain why OWP and IGP have been somewhat considered as separate phenomena. Indeed, a sticky charge does not necessarily

mean that the coal exerted a pressure on the walls and conversely, lateral shrinkage does not mean that, at a given time of coking, walls have never been subjected to the coking pressure. It nonetheless remains true that OWP implies IGP and a mechanical contact between the charge and the walls. The operational problems or dangerous situations that coke-makers can be faced with in the case of high OWP (hard pushes, stickers) are resulting damages to coke batter walls with significant economic implications. This explains why much attention has been paid to pressure generation mechanism. The problem is further exacerbated by reducing reserves of good coking coals with low-pressure characteristics and to adjustments in the production of coke to meet fluctuating demand as the latter requires frequent changes in blend and operating parameters. This, in turn, gives rise to uncertain conditions and a shortened life span of coke battery structures.

Low-volatile high-pressure coking coal is a normal component of a blend as it improves coke yield and maintains a pressure level promoting coal grain sintering and hence abrasion resistance. In the case of ArcelorMittal, coals from Mozambique, namely from Tete, Typical (a coal mining area which used to be called Chipanga) and Moatize, have been used for many years but their incorporation is carefully controlled and hardly ever exceeds 20% due to the high wall pressures they generate during coking (Kruger, 2017).

A maximum allowable pressure is thus required. The difficulty consists in finding a compromise between throughput, yield, quality, and preservation of the oven against possible damage arising from wall pressure. The maximum safe coking pressure (above which wall damage can be anticipated) depends on oven geometry. The smaller 4m tall walls in older ovens can withstand higher pressures better than the tall 6m walls in more modern batteries. Even if identification of a universally-accepted, single-value, safe coking pressure appears unlikely, it may reasonably be expected that the maximum allowable pressure is around 10 kPa. However, in the case of the ArcelorMittal Newcastle plant (which has 6.0m high batteries), the battery tends to suffer from production losses due to stickers when pressures above 7kPa are allowed (Kruger, 2017). Considerable effort has been expended in attempts to develop a reliable test, capable of determining the capacity of coals to generate excessive wall forces. Of these, the movable wall oven is considered the only universally recognized test tool for selecting coking coals (Falcon, 2017).

In the absence of physical tests, Roshkova and Guelton (2014) developed an empirical formulation of oven wall pressure (OWP) depicted in figure 5 expressed as a function of parameters deduced from a simple physical model of IGP. In this context, the model can be viewed as a preliminary assessment tool to guard against unsafe wall pressures: blends, investigate a replacement coal, identify coking pressure generating coals, monitoring the wall pressure, it can help to formulate low coking pressure coal blends, assess economic future blends, investigate a replacement coal, identify coking pressure generating coals and monitor the wall pressure. In the current research all the blends under investigation had their wall pressures determined as GG coal was increased from 31% to 40%.

2.3 Sulphur in Coal/Coke

Sulphur content in coke has debilitating resultant effects on the quality of steel in the downstream steel production process. Its content in coke should be carefully controlled to ensure savings which accrue by by-passing the sulphur removal process. In the study conducted by Rudyuk *et al.* (1974) on the inclusion of sulphur in steel, it was observed that increasing the sulphur concentration from 0.030 to 0.055% and the phosphorus concentration from 0.025 to 0.050% lowers the toughness of steel by (20–35%) and raises the ductile—brittle transition temperature by (20–30°C).

Sulphur is usually an undesirable impurity in steel rather than an alloying element (Rudyuk *et al.*, 1974). In amounts exceeding 0.05%, it tends to cause brittleness and reduce weldability. Alloying additions of sulphur in amounts from 0.10% to 0.30% will tend to improve the machinability of steel Rudyuk *et al.* (1974). Sulphur in coal also poses a serious environmental challenge. When coal burns, it produces Sulphur dioxide which, amongst other problems, reacts with rain water generating 'acid rain'. As an atmospheric pollutant, Sulphur dioxides causes respiratory diseases when inhaled.

Therefore, it is essential that the amounts of sulphur in coal be controlled to as low as possible levels. Sulphur content in coal ranges between 0.3% to 11% by mass but in general it rarely exceeds 3% by mass in metallurgical coals. Sulphur occurs as organic sulphur where its atoms are chemically bonded with the carbon structure of the, mainly aromatic components of the coal. It is also present in coal in inorganic form as iron sulphide (referred to as pyrites).

2.4 Phosphorus content in coke

Phosphorus is usually incorporated in steels at very low quantities and is treated as an impurity and a penalty element. The bulk of phosphorus found in steel can be traced back to coke. Amounts of up to 0.04% can be found in carbon steels. Satyendra (2014) noted that phosphorus is sometimes added intentionally to the steel to improve strength, machinability and atmospheric corrosion resistance. Thus, it tends to make the steel brittle, phosphorus can improve the hardness of steel. Ductility and toughness can however be impacted negatively in high carbon steels that are quench-hardened and tempered and this will have added costs in the steelmaking process.

Phosphorus content in most steels is usually not permitted to exceed 0.05%. Leonghuat (2017) stated that 'phosphorus tends to prevent the sticking of light-gauge sheets when it is used as an alloy in steel'. It strengthens low carbon steel to a degree, increases resistance to corrosion and improves machinability in free-cutting steels. In terms of welding, phosphorus content of over 0.04% makes weld brittle and increases the tendency to crack. The surface tension of the molten weld metal is lowered, making it difficult to control. The low levels of phosphorus required in steel require that the coke used in the blast furnace to produce hot metal must contain as low phosphorus as possible. In the current research it was critical to ensure that the blends employed do not result in high phosphorus. The maximum allowable amount in the produced metallurgical coke was 0.0045%.

2.5 Ash in coke

Mineral matter, which is largely constituted in the ash content in coal, is the non-combustible residue left after coal is burnt. It consists of the mineral matter after all the water and carbonaceous material has been burnt-off. An analysis of the ash composition and its performance on heating are key issues in ensuring certain critical parameters are kept in check. One such parameter is the ash fusion temperature. This parameter must be as high as possible (minimum 1450°C) to avoid fusion of the ash with the silica walls of the coke ovens. If these clinkers are allowed to form, they cause rapid deterioration of the coke oven heating walls and also present pushing problems due to the protrusions of the clinkers which block the passage and development of the coke (Kruger, 2017).

Coke ash also affects the slag volume and chemical composition. The presence of network formers (SiO_2 + Al_2O_3) will affect the basicity, which in turn affects the viscosity and liquidus temperatures.

Coke ash is a key parameter which needs close monitoring in metallurgical coke as it influences the rheological properties of slag. Blast furnace operators in ArcelorMittal Newcastle require coke ash not exceeding 13.5% Kruger (2017). Ash content has also been seen to influence the coke cold strength. Research undertaken in China by Jianliang *et al.* (2009) has shown that ash affects the M40 and M10 values of coke. Other effects noted in their research is that high ash results in high volumes of slag generation during the ironmaking process and leads to higher fuel rates which impacts negatively on the cost of production. Grootegeluk coal contributes the highest amount of ash in the coal blends employed by Arcelor Mittal. The higher percentage of GG in the blend tends to increase the amount of ash in the resultant coke. Such properties have to be carefully controlled when making the blend with imported coals.

2.6 Theory behind borate addition on coke

A novel development into the improvement of coke CSR/CRI has been established by the Russian researcher, Filatov (2012). This development is described in the Russian patent (number 2012102687 A1) published by (Filatov 2012) and Chinese patent (number CN 102071081 B) published by Guoqing (2010). The theory behind this technology is based on passivity of interstitial openings on the coke surface which renders it less reactive to oxygen thereby improving its coke strength after reaction (CSR). The method claims to produce high-quality coke by the application of borate on red-hot coke after discharge from coke furnaces at temperatures of 1050 ± 50°C. Quenching is conducted in water solutions with borates in concentrations of 3-10 g/dm^3. Guoqing (2010) observed that by spraying 2 - 20% aqueous solution of borates from the metals containing sodium, potassium and calcium onto coke lumps at temperatures of no lower than 20°C, an improvement of coke CSR/CRI of up to 15% can be easily registered. Further to this, Guoqing (2010) found that the cost benefit generated from the use of cheaper coals using this technology was found to be equivalent to savings which resulted in coke rates of between 200-220kg/tonne of hot metal produced in the blast furnace. Kovalev *et al.* (2008) noted one disadvantage in introducing borates to the

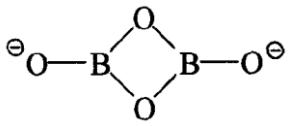
surface of coke. This is the formation of extra alkali which would adversely affects the ash basicity of the coke as shown in the following formula indicating the ash basicity index (Io):
$Io = (Fe_2O_3 + CaO + Na_2O + K_2O)/(SiO_2 + A1_2O_3)$

Kovalev *et al. (*2008) proposed that increasing the value of Io by 0.1 units reduces the quality parameters of coke by decreasing CSR by 9.5% and increasing CRI by 7.5%. A further possible reason for insufficient improvement of coke quality after borate treatment was postulated by those authors to have been due to a low content of boron in the tetraborate molecules, which, for the group of potassium, sodium and calcium tetraborates, is correspondingly low (18 - 22%). Such a situation would lead to limited reliable protective layer covering on the surface of the coke lumps and therefore would not protect them from extensive oxidation by O_2 and CO_2. Another research which was conducted by Li *et al.* (2014) indicated that the alkalis had a catalytic effect which would accelerate reactivity thereby reducing coke CSR. Thus a balance of the effect of alkalis versus the improvement of CSR by quenching with borates requires a further investigation.

From the investigation conducted by Filatov, (2012) on using borate solution to quench coke, it was observed that the positive effect of pentaborates in improving quality parameters (CSR) and (CRI) appears to be as a result of pentaborate solution covering the surface of coke lumps in such a manner that "the layer of pentaborate molecules are held on the surface by adhesion forces". In so doing, part of the pentaborate solution also penetrates well into the cracks and pores of the coke lumps forming a layer of pentaborate molecules which block access of oxidizing gases O_2 and CO_2 to the surface of coke. The author postulates that this situation is "able to deactivate coke with respect to the reactions with O_2 and CO_2, even at the temperatures below the pentaborate melting temperature." In addition, this author claims that, at melting temperatures, borates are subject to so-called borate molecular rearrangement, i.e. borate molecules form the corresponding metaborate and boron oxide as follows,

$2KB_5O_8 \rightarrow K_2B_2O_4 + 4B_2O_3$.
The metaborate is also ionized as follows:
$K_2B_2O_4 \rightarrow 2K+ + B_2O_4^{2-}$

In this manner, potassium cations are also believed to penetrate the interlayer space of coke crystallite and remain there (the so-called reaction of intercalation). Dimeric anion $B_2O_4^{2-}$ forms a cyclic structure as shown below;



Anion $B_2O_4^{2-}$, having adhered to the coke crystallite frame, will be kept thereon by intermolecular forces, determined by overlapping of P-orbitals of boron with corresponding P-orbitals of coke carbon. What assists is a strong donor-acceptor interaction between boron and carbon atoms. While on the surface of the coke crystallite, anions $B_20_4^{2-}$ are firmly held thereon, forming a protective film of anions $B_2O_4^{2-}$. This prevents the penetration and interaction of gaseous oxidizers with the carbon of the coke, both in the case of gasification of coke in blast furnaces and in the equipment for determination of CSR and CRI parameters of coke. Carbon-dioxide gas is transmitted through the pores in the coke lumps at the temperature of 1100°C.

However, it should be noted that the influence of the borate molecule in slowing down the CO_2 reaction with the coke is possibly countered by the catalytic effect of the alkali ion associated with the borate compound (in this case Na+). In their investigation into the catalytic effect of alkalis on coke reactivity, Li, *et al.* (2014) attributed this catalytic effect to the additional alkali metals which introduce extra electrons into the coke lattice matrix.

In the test work undertaken by Tamko *et al.* (2010), the most efficient borate for this application was found to be the decahydrate of sodium tetra borate. It is against this background that the current research is based. The investigation will use the same form of borate and in concentrations as determined by Tamko *et al. (*2010) in the hope that the benefits shown by those authors will be duplicated in the current research. The research focused on testing borate application on local South African blend coking coals (mainly Grootegeluk product, GG) to determine the cost-effectiveness of substitution of expensive import prime coal with this local coal. The predicted behaviour in the blast furnace was to be tested for CSR and CRI to determine the effect of the borates. Grootegeluk shall be the only local coal considered in these blends.

Consideration on the use of GG was done ahead of any other available coking coals in South Africa due to its availability. Further to this, an evaluation conducted by earlier scientists who published in the nineteen fifties and sixties such as Moodie (1956), indicate that there is very little if any good coking coal in South Africa and what there is 'yields a very inferior product when carbonised on their own, but acceptable coke can be produced by blending with a small percentage of certain Natal coking coals'. The Kwa-Zulu Natal coals are no longer available as coking coals and have since been abandoned from use. The other coal which was previous in use in South Africa was Tshikondeni which was later unavailable due to the closure of its mine (Cornish, 2012). This leaves Grootegeluk coal as the only product worth pursuing in terms blending with coals from overseas for metallurgical coking purposes.

2.7 Summary of the literature review

Based on the review undertaken, it has been established that coals making up coke are required to have carefully controlled qualities to avoid contaminating entities. More importantly, the addition of treatment after the coking making process has been proven to have considerable impact on the success of the coal. The quenching of coke by borates has been proven in Russia to have had beneficial effects by increasing the properties of the final coke. No work in this regard has been undertaken in South Africa and no work in the possibility of increasing the incorporation of less expensive local blend coking coals. It is these two aspects, the use of borate to improve cokability and the inclusion of higher local coals that will be researched in this Research Report.

CHAPTER 3: MATERIALS AND METHODS

This Chapter presents the number and nature of samples used in this research programme and the analyses and tests conducted on them.

3.1 Feed coals used in the coke blends.

The coals that were used for the testing of the coke blends included one South African coal, i.e. the semi soft blend coking coal (SSCC) from Grootegeluk Colliery (GG), and the following coking coals from abroad: namely, Shoal Creek (USA), Oaky North (Australia), Chipanga (Mozambique) and Goonyella (Australia). Sampling of each coal was conducted according to conventional methods (ASTM D 2234/D2234M-10), see also 3.4 below.

All feed coals were analysed prior to blending using proximate and ultimate analyses, the Free Swelling Index (ASTM 720-91), Ash Oxide composition by XRF (ASTM E 1361-02). Cokes made from the prepared blends were analysed for Hot and Cold Strength Properties (ASTM D 3402), Ultimate analysis (ASTM D 4239-04B) and Proximate analysis (ASTM 3172-89). Total Sulphur content was tested using ASTM D 4239-04A).

3.1.1 Breakdown of the test blends

The named feed coals were blended in 1% increasing proportions of the GG component, i.e. from 32% up to 40% where it was envisaged that the volatile and ash composition would fall out of specification for the produced coke. The current blend employed by ArcelorMittal with 30% GG composition was used as the control blend. Table 3 below presents the proportions that were used in the tests.

Table 1: Blend proportions of the different coal components

	Base blend	**Blend 1**	**Blend 1**	**Blend 2**	**Blend 3**	**Blend 4**	**Blend 5**	**Blend 6**	**Blend 8**	**Blend 9**	**Blend 10**
GG	30%	31%	32%	33%	34%	35%	36%	37%	38%	39%	40%
Oaky	27%	26%	25%	24%	23%	22%	21%	20%	19%	18%	17%
Goonyella	18%	18%	18%	18%	18%	18%	18%	18%	18%	18%	18%
Typical	8%	8%	8%	8%	8%	8%	8%	8%	8%	8%	8%
Shoal Creek	17%	17%	17%	17%	17%	17%	17%	17%	17%	17%	17%

The proportion of GG coal was varied by increasing one percentage each on subsequent tests while the import coal Oaky will be reduced by same amount. The proportions of all the other coal components were kept constant.

3.1.2 Tests done on the coke blends

The blends were loaded into the pilot plant coke testing oven with the capacity of 350kgs according to the ArcelorMittal pilot plant standard operating procedures based on ISO and ASTM standards as highlighted in section 3.1 above. The resulting coke samples derived from each test were pushed out of the pilot scale coke oven and quenched by normal un-treated water and separately by water treated with pre-determined constituents of borates. The coke samples were sent for preparation and testing. Major tests that were conducted on the resultant cokes included the following:

I. Mechanical strength tests (I40, M40, M10)
II. Hot strength tests (CRI/CSR)
III. Chemical analysis (Sulphur, Phosphorus,)
IV. Proximate analysis (Ash, volatile matter, fixed carbon and moisture)
V. Pushability tests (mainly wall pressure in Kpa)

3.2 Facilities used in the research

Preparation, test and analytical facilities as listed in the table below were available at ArcelorMittal and its network of laboratories and used for the testing and analysis.

Table 2: Equipment and facilities used in research

Item/Equipment/Facility	**Location**
Pilot oven and auxiliary equipment	ArcelorMittal Newcastle Laboratory
Sample preparation equipment	ArcelorMittal Newcastle Laboratory
Dilatometer	ArcelorMittal Newcastle Laboratory
Micum drums	ArcelorMittal Newcastle Laboratory
CSR/CRI Equipment	ArcelorMittal Newcastle Laboratory
X-Ray fluorescent machine	ArcelorMittal Newcastle Laboratory
Moisture determination crucibles	ArcelorMittal Newcastle Laboratory
Petrographic analysis with microphotographs of (i) GG coal (ii) the 'parent' blend coal and (iii) its resulting coke without the increased GG (iv) the various cokes with varying proportions of GG	ArcelorMittal Newcastle Laboratory approved partner for petrography tests (Bureau Veritas)

3.3 Sampling methods

3.3.1 Sampling from coal trucks

- Sampling of the coals was done from coal trucks and the procedure was as follows; three holes were dug, one-meter deep were dug diagonally and evenly spaced into the material in the truck.
- The inside surface of each hole was skimmed with a shovel, working from top to bottom.
- The material collected was placed from the three holes into a plastic bag.
- The bag was then sealed to prevent the loss of fine material or moisture.
- The material was clearly identified with each sample using two labels, one for outside and one for inside each bag, with date, material type and code as well as the sample number.
- Each truck was treated in the same way. A minimum of ten trucks were sampled this way to obtain a representative sample for a train load containing a coal component of the blend.

3.3.2 Sampling from a heap

- Five or more increments were taken, spaced evenly around the circumference of the heap to be sampled to realize a sample mass of 15kg's.
- The increments were taken, starting at chest height, by digging into the heap with a shovel.
- Holes were made to be 1 meter deep, where possible, depending on material type.
- The holes were made be at an angel of 30 - 40 degrees where moisture level/compactness of the material allowed.
- An increment was taken by scraping the sides of the hole with a shovel from top to bottom.
- Increments were deposited (composite) into one bag to form a sample.
- Sample was identified with sample number, material type, date and time taken as well as the word stockpile (heap) then transported to the Laboratory.

3.3.3 Sample preparation for Proximate Analyses and X-ray Analysis

- The following equipment was cleaned: Hammer mill, Raymond mill, hand roller, micro screens receiving pan and lid, 25mm riffler, V blender, hand grinder and quartering iron receiving pans.
- The contents of bags received were emptied onto a clean steel plate and mixed properly with spades. Quartering iron was placed on top of the cone and pressed into the sample to divide into four quarters.
- One quarter (A) was removed for moisture and screen. Opposite quarter D was removed to flush the system with and discarded afterwards.
- Remaining quarters (B&C) was combined and taken to the hammer mill and entire combined aliquot crushed.
- Contents on steel plate were emptied and mixed properly with spades and sample again divided into four quarters.
- One quarter was removed and dried out for 2 hours.
- Opposite quarter (G) was removed and the sample stored as a reference sample for possible repeated preparation. Opposite quarter (G) was removed and the sample stored as a reference sample for possible repeated preparation.
- Opposite quarter (G) was removed and the sample stored as a reference sample for possible repeated preparation.
- Opposite quarter (G) was removed and the sample stored as a reference sample for possible repeated preparation.
- After sample was been air dried, 25mm slot divider was used, the crushed aliquot repeatedly divided until there was ±150 grams in one receiving pan.
- Sample was marked for - 212-micron preparation. Above mentioned steps were repeated with remaining portion of the crushed and sample marked ± 150 grams for -1.400 mm preparation.
- Remainder of sample was stored for possible repeat preparation. Sample was taken for - 212 preparation in which the following/next preparation steps were taken.
- The total 150-gram sample was screened through a - 212-micron screen with a lid.
- The plus material was crushed once using the Raymond Mill.
- The crushed sample was screened again through the - 212-micron screen with lid on and the above-mentioned steps were repeated until the total mass of sample passed through the - 212-micron screen.

- The total 212-micron material was placed in the V Blender and turned 30 times in clockwise direction.
- The contents of the V Blender were emptied and transferred it into properly identified packets and the prepared sample handed over to Laboratory Assistant.
- The sample mentioned for - 452 preparations were taken and the following preparation screen the total 150-gram sample through a - 425-micron screen with a lid on was done.
- The (+425) material in a hand grinder was placed and turned until everything passed through the grinder.
- The above-mentioned steps were repeated until the total mass of sample passed through the 425-micron screen.
- The sample from the pan was removed and transferred into a properly sealed and identified plastic bag and sample handed over to the Laboratory Assistant.
- The portion mentioned for – 1.4mm was taken for petrography preparation and the following done:
- The total 150-gram sample was screened through a 1.4mm screen with lid on and the + 1.4mm material placed on a steel plate and rolled over the material three times with a hand roller. The hand crushed material was re-screened through the 1.4 screen with a lid on.
- The +1.4 materials were placed on the steel plate and the above steps repeated until everything passed through the 1.4mm screen.
- The sample form the receiving pan was removed and transferred it to a properly sealed, marked plastic bag and the prepared sample handed over to the Laboratory assistant to be sent to the petrography section

3.4 Pilot plant tests for the blended coals

3.4.1 Sample preparation for the pilot plant oven tests

- An air-dried sample was weighed by physically placing a clean empty bin on scale and then pressing the **"Tare"** key;
- The bin was then filled with an air-dried sample using a shovel.
- When the bin was full the bin was placed on the scale and the mass was recorded.
- The contents were then emptied into the loading bin and these steps repeated until the required mass was obtained.

NB* Mass was obtained from a calculation, that is, 350kg including addition of water

3.4.2 Loading of coal into pilot plant oven

- The air-dried sample was then transported to mixing drum (figure 6 below) by physically pressing the **"On"** button on the mixing drum and then by physically coupling the hook of the overhead crane to the hook of the loaded bin.



Figure 6: Mixing drum for the crushed coal

- The loaded bin was then lifted by use of the crane`s remote control and slotted on top of the running mixing drum. Once the loading bin and mixing drum was aligned

properly a rubber mallet was taken and used to hit the sides of loading bin with lots of force ensuring that all contents were emptied into mixing drum. The stand was pushed with loading bin underneath the mixing drum and while sample was being mixed for 2 minutes the required quantity of water was measured out and poured into mixing drum (quantity of water was obtained from calculation).

- Liters of diesel was then measured out and poured into mixing drum. (Diesel helps make the flow process through the chute much easier).
- Allowance was made for the sample to be mixed for another 13 minutes. (In total the mixing must be done for 15 minutes).
- Once all the contents were out of the loading bin, the loading bin was moved carefully and lowered into its stand and uncoupled.
- After the material had been mixed for 15 minutes the lever was then physically pulled on the side of the mixing drum into an open position.
- The contents will then flow into the loading bin that is positioned underneath the mixing drum via a chute. Once all the contents had flown into loading bin physically, lever was pulled into a closed position and switched off.
- Opening of the six element flaps was then done by physically taking hold of the handle of each element flap which is situated on either side of the oven and turning them into an open position.
- Opening of trap door lid was done by physically taking hold of the handle of the top cover and lifting it up.
- Cover was then placed to one side and then hook was used to lift trap door and place to one side.
- Inserting of loading chute into oven was achieved by physically turning the **E-Stop** button on and then switching the "**On/Off"** button to an "**On"** position.
- Thermocouples were inserted into oven (± 20cm) by physically inserting the 3 thermocouples into appropriate slot i.e. the top thermocouple is inserted into the right-hand side slot which is the **"Wall Temperature"**. The middle thermocouple is inserted into the center-slot which is the **"Top Temperature"** and lastly the bottom thermocouple is inserted into the left-hand side slot which is the **"Middle Temperature".**
- Transporting mixed sample to the top of oven was then done by physically coupling the hook of the overhead crane to the hook of the loaded bin and carefully lifting the

loaded bin with the use of the crane`s remote control and then slotting it on top of the loading chute.

- Once loading bin was properly aligned with the loading chute take a rubber mallet and hit the sides of the loading bin with lots of force ensuring that all the contents is emptied / charged into pre-heated oven.
- Once all the contents were out of the loading bin the loading bin was carefully moved and lowered it into its stand and uncoupled. The loading chute was then physically removed from oven by holding the handles on either side of loading chute and lifting it out of oven.

3.4.3 Coking of the coal inside the pilot plant oven

- The trap door was then closed by physically grabbing hold of the trap door lid with the hook provided and then closing the oven by ensuring that the trap door aligns properly with the framework. The top cover was replaced by taking hold of the handle of the top cover and placing it back ensuring that it is aligned properly. Inserting the thermocouples fully into oven was done by physically pushing each thermocouple into the oven until the collar of each thermocouple contacted the outside of oven door.
- Coal inside pilot oven was then levelled by physically untightening the bolts of the leveling door and removing leveling door from main door, ensuring the leveling door is placed to one side and then physically taking leveling bar and levelling out coal inside the oven. NB# Note that excess coal is dropped into a pan which is placed at the bottom of oven door which is weighed and used for the calculation of the bulk density.
- The leveling door was replaced once the coal was properly leveled out.
- Lowering of charge level indicator was then done by physically unhooking the charge level indicator cable and then lowering the charge level indicator on top of the coal.
- Extraction was then switched off by physically switching the "**On/Off**" button to an "**Off**" position.
- Chimney was then cleaned by physically removing the chimney lid and then thrusting chimney cleaner in and out of vertical chimney until chimney was clean.

- Chimney cleaner was moved in and out of horizontal chimney until chimney was Clean.
- Recording of coking process was started by physically clicking on the "start recording" icon on the process computer of the pilot plant (After 3 minutes three graphs would appear).
- Each graph was highlighted separately and each graph smoothened separately by clicking on the "Hourglass" icon until a straight line appeared.
- Ramping display was then achieved by physically pressing the "Run/Hold" button on the master controller which was marked B2.
- Closing of the six element flaps after first hour of coking process has elapsed was done by physically taking hold of the handle of each element flap which is situated on either side of the oven and turning them into a closed position.
- Chimney was then cleaned after first hour of coking cycle had elapsed and thereafter for the next five hours by physically removing the chimney lid and then thrusting chimney cleaner in and out of vertical chimney until chimney is clean.
- Recording of coking process was then stopped by physically clicking on the "stop recording" icon on the process computer only if the B2 master controller was displaying "E" which indicated that the coking process had ended.

3.4.4 Pushing of coke out of the pilot plant oven

- Firstly, all three thermocouples were physically removed from the oven by pulling each one out very carefully and placing them on their stand.
- The charge level indicator was then physically lifted by pulling onto attached cable and hooking it onto a slot.
- The back-oven door was opened next and the quenching trolley positioned properly.
- The front oven door was then open and Pusher Machine positioned in front of open oven to ensure that pusher is correctly aligned.
- Stoppers must be lifted to ensure that Pusher will not move or roll. Once the Pusher was properly aligned a check was made to see that the Pusher Controller is on "System Watt Hours" and the top and bottom reading noted down physically.
- Once the readings were noted, the 'Push' button was activated so that the coke could now be pushed.
- Once pusher arm reached the end it would automatically stop.

- All the coke was physically scraped into quenching trolley and once again noted down the top and bottom readings.
- The pusher arm was retracted by activating the 'Push' button to the 'Return' position so as the pusher ram beam to return to its original position. (Top and bottom readings had to be noted down once again).
- Oven roof, wall and floor were thoroughly scraped out before both doors could be shut. Machine was then pushed back to its original position.
- Coke was levelled out in the quenching trolley by use of a shovel to ensure a uniform spreading of the load.



Figure 7: Pushing of coke out of the pilot oven

3.4.5 Quenching of the coke.

- The loads were split (each with about 175 kg each). One load was sent straight to the quenching tower for normal quenching and the other pushed just outside the building for a physical quench by use of a jerry-can.
- A solution for 10g/dm^3 was made simply by weighing 200g of Sodium tetra borate into a bucket with 20 litres of water (using simple equipment in figure 8 below).



Figure 8: Equipment for the preparation of the borate solution for quenching

- The borate solution was then sprayed onto the hot coke, precautions being made not inhale the fumes. The spraying was done uniformly across the trolley.
- Quenching was done by use of at least three buckets full of the borate solution (about 60 litres) until no red coke was visible (as in figure 9 below).



Figure 9: Equipment for the preparation of the borate solution for quenching

- The other coke sample from the other half was sent through to the quench tower for normal quenching by physically pushing the quenching trolley to the quenching tower and ensuring that trolley is properly positioned.



Figure 10: Normal quenching of the coke by normal quench water

- The pre-set quenching cycle (set at 3 min) was physically activated to begin the onset of quenching.

3.4.6 Stabilizing of the coke after quenching

- The quenching trolley was physically removed from the quenching tower and the trolley pushed close to the stabilization chute.
- A second empty quenching trolley was then placed underneath the stabilization chute ensuring that trolley was in a centralized position.
- The trolley containing the coke was coupled to hook of the overhead crane and the trolley lifted to the maximum height.
- Once trolley had reached its maximum height, the trolley was positioned in the right direction over the stabilization pan.



Figure 11: Stabilisation of the coke by use of the stabilising chute

- The trap door at the back of the trolley was now opened allowing the coke to fall into the stabilizing pan which passes through the stabilizing chute which ultimately fell into the empty trolley (see figure 11 above).
- The rap door was then closed and trolley lowered to the ground.
- Trolley with coke from underneath the stabilizing chute was removed and the empty trolley underneath the stabilizing chute pushed ensuring that the trolley was properly centralized.
- Once again couple the trolley containing the coke to hook of the overhead crane and lift trolley to the maximum height.
- Once trolley had reached its maximum height, the trolley was positioned in the right direction over the stabilization pan.
- The trap door was opened at the back of the trolley allowing the coke to fall into the stabilizing pan which passes through the stabilizing chute which ultimately falls into the empty trolley.
- The trap door was then closed and trolley lowered to the ground.

Trolley was removed with the coke from under stabilizing chute and pushed and coupled to the hook of the overhead crane.

Seven coke drums were then placed next to one another on a steel plate.

- Once again lift the trolley containing the coke one meter above the coke bins ensuring it were directly above the bins. Once the trolley was in correct position the trap door of the quenching trolley was opened so as to fill the coke bins with the coke.
- Once the trolley was empty it was lowered to the ground and trap door closed. Spillages were scooped up and put into coke bins.

3.5 Testing coke for cold strength (Irsid Indices Tests)



Figure 12: Irsid test drum

- The coke micum drum was cleaned using a hand broom to prevent contamination.
- Appropriate safety equipment was used always (PPE) Hard hat, Safety glasses, respirator, gloves and safety boots. (This is because this task can cause injury through nip / pinch points).
- Precautions were taken to ensure the coke micum drum was isolated with a padlock at all times at the lockout isolator box and the keys to the padlock kept in the position of the personnel that are working on the coke micum drum. (This was to prevent anyone starting the coke micum drum while personnel are still busy).
- 50 Kg coke sample was charged into a thoroughly cleaned micum drum carefully to avoid breakage.

- The door which was hanging on top of the drum was placed on a chain in position and locked with wing nuts.
- The two receiving pans were placed in position underneath the micum drum.
- The counter was set at 400 revolutions (25 RPM) then de-isolated and the micum drum started (the drum would automatically stop at the end of 16 minutes or 400 revolutions cycle).
- The sample had now been drummed for a total of 500 revolutions i.e. 100 for the micum and additional 400 for the irsid test.
- After this, the drum was stopped to make sure it is isolated with a padlock and then the door cover removed.
- The cover on the chain on top of the micum drum was then hanged.
- The sample from the drum was removed by means of a scraper and a hand broom into a receiving pan after turning the drum with its opening towards the bottom and securing it with a stopper that prevents the drum from any movement or rotation with all precautions being taken to avoid stepping on the material.
- The coke was then screened over the big coke shaker with the following aperture sizes: 40, 30, 20 and 10 mm.
- Accurate determination and recording of the cumulative masses of the + 40mm, +30 mm, + 10mm and - 10mm fractions was then made.
- The difference between the sum of the masses of all the fractions and the mass of the coke charged was added to the - 10 mm fraction.
- Process above was repeated above mentioned test for the sample B.
- Using the [Lab Calc program no.19] on the computer the weights were entered to calculate the irsid indices and report analysis by calculating the amount below 10mm (I10) and coke

CHAPTER 4: RESULTS AND DISCUSSION

This Chapter presents the results of the research undertaken with a summary of the impacts of the results. SABS/ISO standards were used in all relevant cases. ArcelorMittal testing procedures were all according to standard SABS/ISO methods for coke testing.

4.1. Coal component analysis

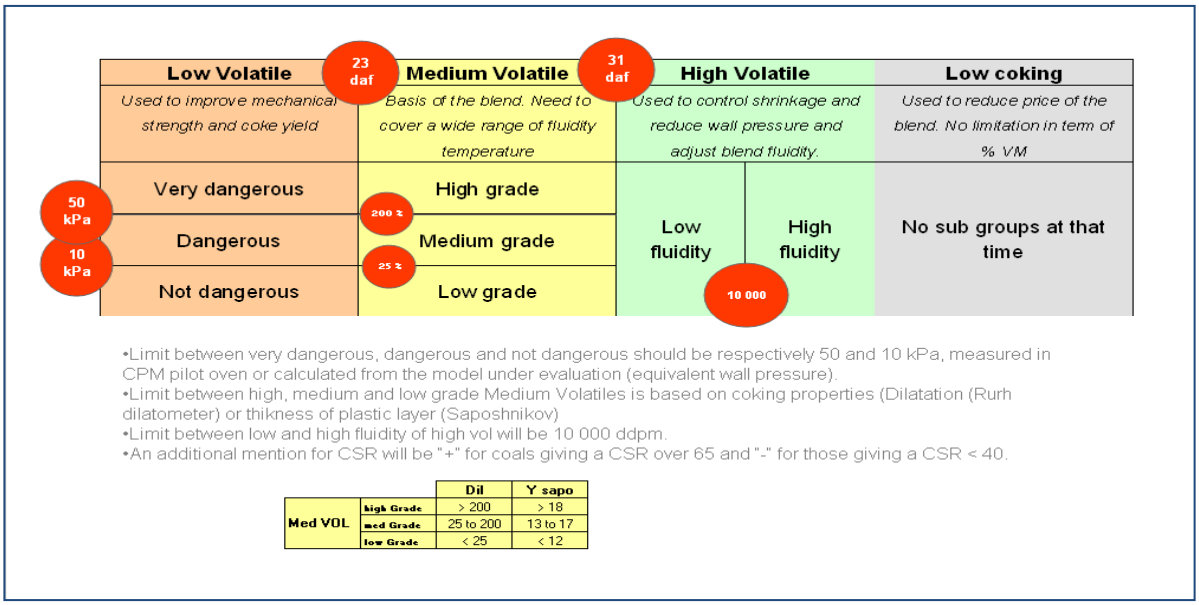
Each coal component for the blends was individually tested for chemical and proximate analysis and the results were shown on the table 3 below.

Table 3: Coal component analysis

Coal	Grootegeluk	Oaky-North	Goonyella	Typical	Shoal-creek
Ash	10.1	9.2	8.9	10.3	9.2
Sulphur	1.14	0.61	0.64	0.93	0.71
Volatile matter	36.4	22.2	23.2	22.8	26.8
Roga	72	82	77	78	82
Max Fluidity	26	488	609	617	21876
Maximum Dilatation	21.7	124.5	101.2	146.5	278.9
Al_2O_3	2.1	2.6	2.5	2.5	2.6
SiO_2	6.2	4.9	5.2	5.6	5.2
P	0.006	0.065	0.029	0.062	0.044
K_2O	0.11	0.12	0.08	0.18	0.23
CaO	0.06	0.21	0.08	0.24	0.21
TiO_2	0.28	0.24	0.21	0.36	0.17
Mn	0.007	0.005	0.004	0.005	0.003
Fe	0.4	0.4	0.05	0.5	0.5

The component coals were all medium volatile coals except for GG whose volatile matter content was just over 36% according to the ArcelorMittal classification on Table 4 below.

Table 4: ArcelorMittal Coal Classification



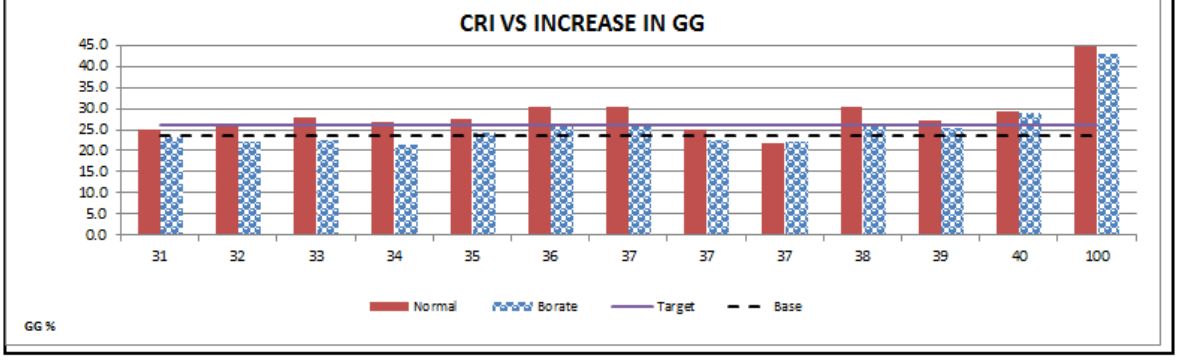
Ash composition was all within moderate levels of 9 to 10%. Sulphur for all components was below the expected limit of 1% except GG whose sulphur content was 1,14%. The fluidity of all coal components was moderate (below 300 ddpm) with very high fluidity exhibited by Shoal-Creek, an American coal which is added into the blends to enhance good fluidity. Dilatation and Roga indices which indicate the level of oxidation of each coal, were positive for all coal components (i.e. thereby indicating a non-oxidised state). Chemical elements of metals elements were all at trace levels in all coal components.

4.2 Effect of borate quenching on coke CRI

The primary test to establish the effect of borates on coke properties is the reactivity test (CRI). This is an index ranging for normal coke from 20 to 28. The reactivity index acceptable for blast furnace operations at ArcelorMittal Newcastle should be less than 26. This minimum is the acceptable minimum for the global group of steel companies in ArcelorMittal. 'The Methodology and Specifications Guide' is a marketing guide used in the global marketing of coke properties. This also uses CRI of less than 26. The results of the tests showed a gradual deterioration of this parameter with each percentage increase in GG percentage. A normal coke quenched in normal water without the borate solution was hardly within limit for blends with GG above 32% GG while the coke treated with the borate water was able to retain its strength (CRI) within the 26% maximum limit with the inclusion of GG in the blend of up to 39% in the blend. Of all the tests done to compare the

coke treated with normal water and the equivalent coke treated with borate solution, there was a very clear and visible impact on the coke in all the blends when borate was added to the quenching water. i.e., the coke quenched with borate water showed superior CRI compared with coke quenched with normal water. Cokes with normal water were erratic in their CRI results and predominantly above the target level of 26%. Figure 13 below presents the bar graph and supporting table showing the effect of borate water quenching compared to the coke quenched with normal water on coke CRI.

CRI (limit-26)													
GG %	31	32	33	34	35	36	37	37	37	38	39	40	100
Normal	24.9	25.6	27.9	26.7	27.5	30.3	30.5	24.9	21.7	30.3	27.1	29.4	44.7
Borate	23.1	22.2	22.3	21.4	24.1	26.4	26.5	22.3	22.1	25.9	25.4	29.0	42.8
Base	23.5	23.5	23.5	23.5	23.5	23.5	23.5	23.5	23.5	23.5	23.5	23.5	23.5
Target	26	26	26	26	26	26	26	26	26	26	26	26	26



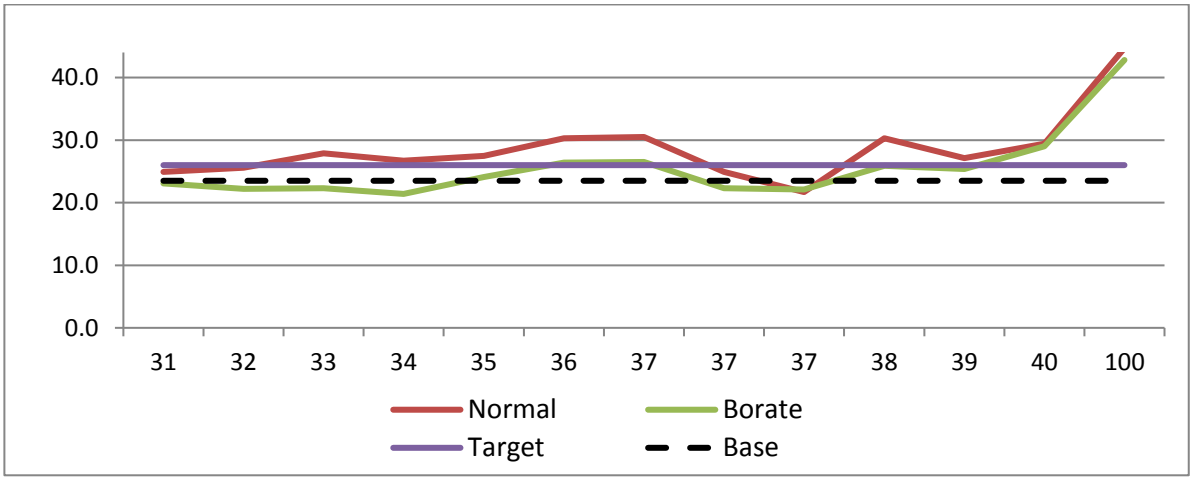


Figure 13: CRI units Versus percentage proportion of GG in the table and graph format

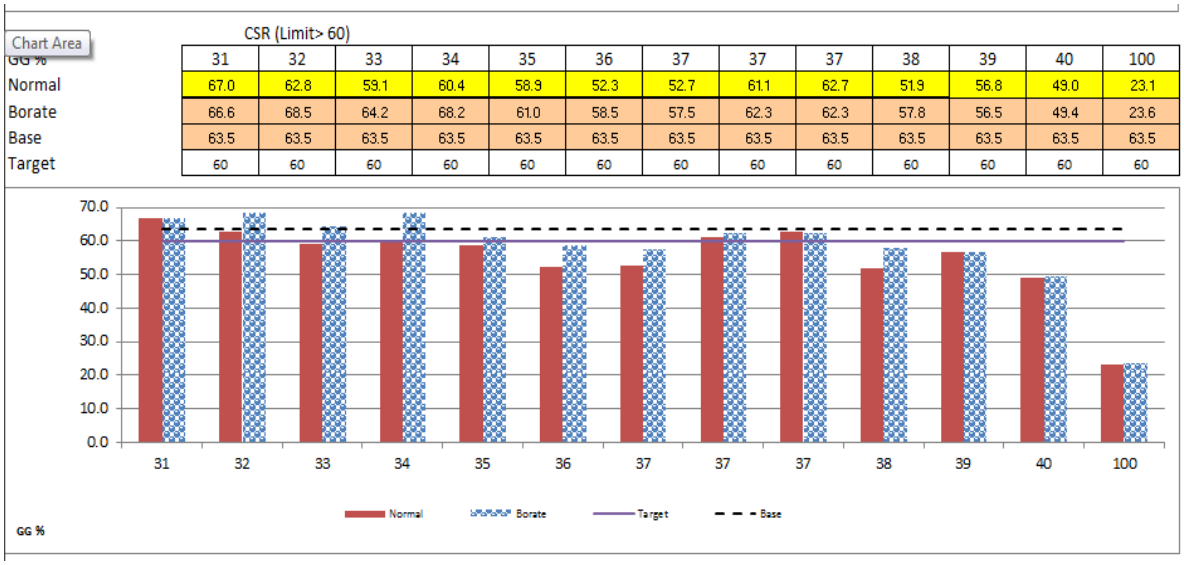
4.3 Effect of borate quenching on coke CSR

The second primary test to establish the effect of borates on coke properties is the Coke Strength after Reaction tests (CSR). This is an index ranging from 58 to 70. The CSR index acceptable for blast furnace operations at ArcelorMittal Newcastle should be ***more than 60***.

'The Methodology and Specifications Guide' is a marketing guide used in the global marketing of coke properties also recommends CSR of above 62.The Newcastle Blast Furnace has lee ways to accommodate CSR as low as 58 but this usually requires that other fuel additives (mainly pulverized coal injection) to be used to avoid 'choking' and slipping in the blast furnace.

Figure 14 below presents the bar graph showing the effect of borate water quenching compared to the coke quenched with normal water on coke CSR. The results of the tests show a gradual reduction of CSR with increase in GG percentage in the coke.
For all blends, the superiority of borate water quenching over normal water use was observed to consistently contribute to better hot strength results. Three duplicating tests were conducted using 37% GG which marked the point where this parameter started to become weaker (lower than 60 for the borate treated coke).

While the borate treated coke could produce hot strength results above 60 for blends with GG of up to 37%, the normal treated coke was hardly within specification for blends with GG above 32% indicating that the borate had an impact which resulted in inclusion of GG with up to a further 5% compared to the normal treated coke sample.



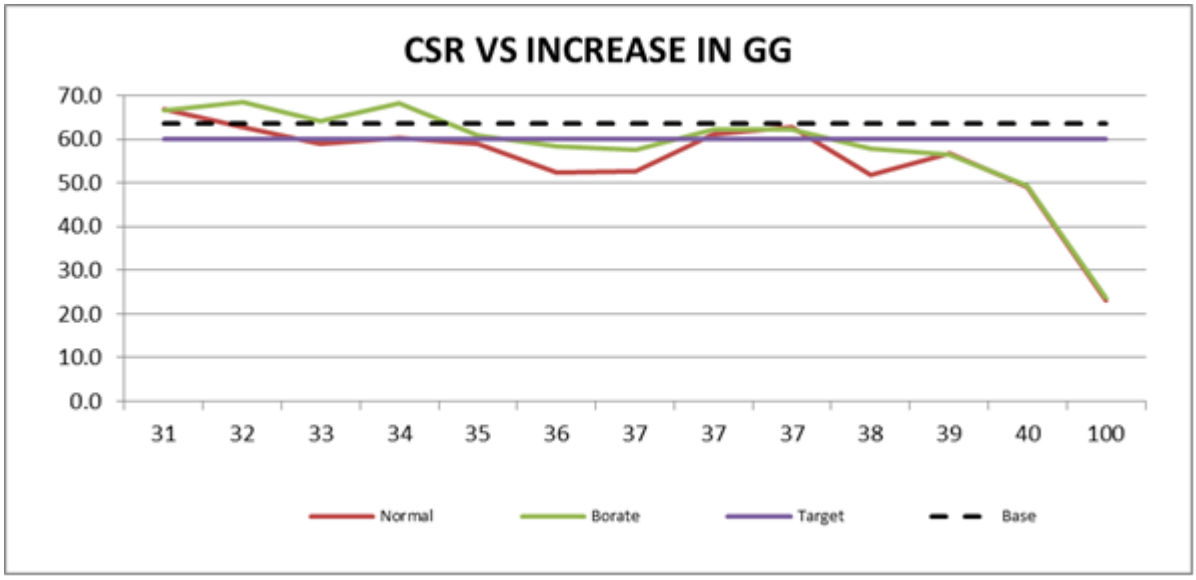
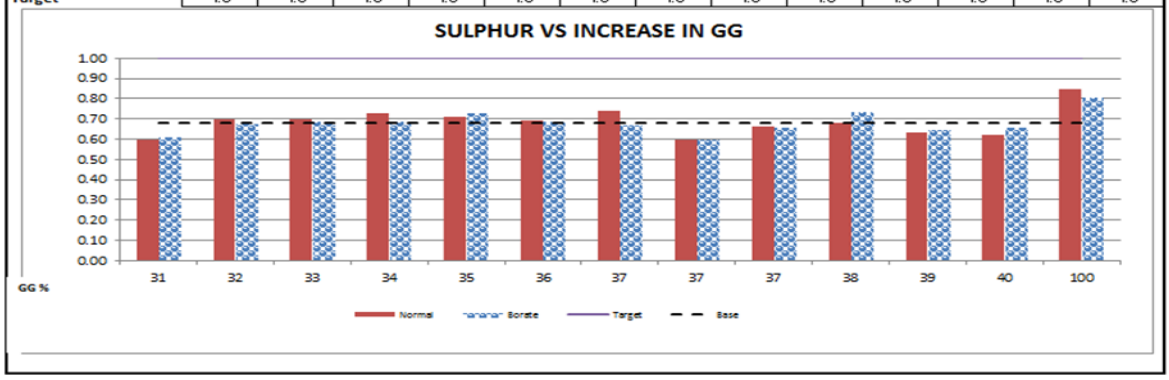


Figure 14: CSR Versus percentage GG in the blend (table and graphic form)

4.4 Effect of borate quenching on Sulphur in coke

The maximum limit of total sulphur in cokes is 1%. Analyses of all the cokes with increasing GG proportions showed that sulphur was under the maximum requirement of 1.0 across all the GG percentages and there were no major deviations on both the normally quenched and borate solution quenched cokes. From all the tests with percentages from 31 to 40% of GG the Sulphur content stayed well within the limits of under 1%. This trend was to be expected as most blend components have very low Sulphur contents with the 100% GG single component product exhibiting the highest sulphur content of 0.85%. As in the case of analysing Ash, DFC and Phosphorus, this test was undertaken to establish whether any chemical alteration had arisen from borate addition versus normal quenching. Thus, although GG has the highest contribution compared to other coal components in the blends, all coke blends presented low total sulphur results. Figure 15 presents the impact the increase in GG in the blends had on the sulphur content of the resultant coke.

Sulphur (Limit 1.0 Max)													
GG %	31	32	33	34	35	36	37	37	37	38	39	40	100
Normal	0.60	0.70	0.70	0.73	0.71	0.69	0.74	0.60	0.66	0.68	0.63	0.62	0.85
Borate	0.61	0.68	0.69	0.69	0.73	0.69	0.67	0.60	0.66	0.74	0.65	0.66	0.81
Base	0.68	0.68	0.68	0.68	0.68	0.68	0.68	0.68	0.68	0.68	0.68	0.68	0.68
Target	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0



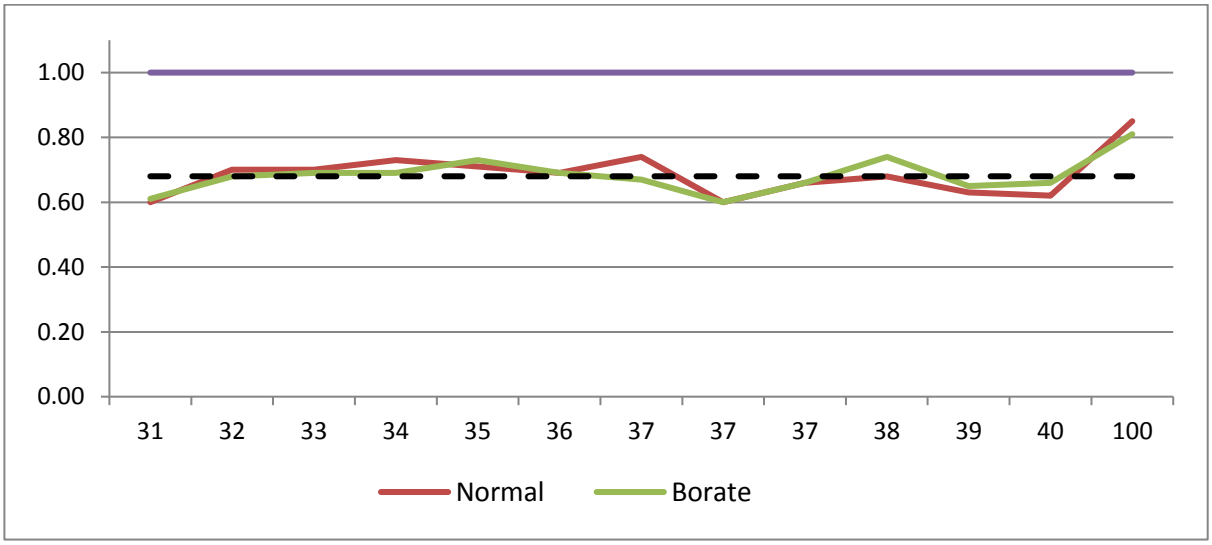


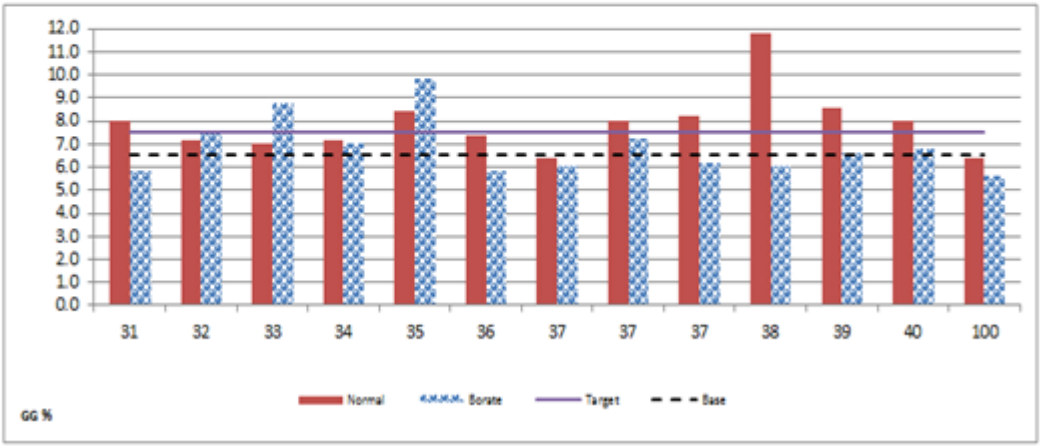
Figure 15: Sulphur percentage in coke versus percentage Sulphur in blend table and graphically presented.

4.5 Effect of borate quenching on coke M_{10}

Abrasion resistance (as is determined by the M10 test) of **below 7.5** (ArcelorMittal standard) is detrimental to furnace operations since it results in the generation of fines which has the tendency of "choking" the furnace. The results of the experiments conducted were clear on the impact of borate on coke abrasion strength. There was a general trend on all blends to have better abrasion resistance on the sample treated with borate compared to the ones quenched with normal water. i.e. Good M10 values for the borate-treated coke were registered for all blends up to the blend with 40% GG.

On the other hand, the samples treated with normal water were all out of specification, namely, above the limit even with the lowest proportion of GG level (31%) in the blend, i.e. Coke treated with normal water had M10 values which were well beyond the target and base levels for all the blends from 31 to 40%. The 100% GG sample had levels of M10 as predicted by the fact that the single component of the GG (being a semi-soft coal) has poorer coking ability and hence poorer coke strength properties compared to hard coking coals. Figure 16 below presents the tabulated and graphic results below.

	M10 (7.5 Max)												
GG %	31	32	33	34	35	36	37	37	37	38	39	40	100
Normal	8.0	7.2	7.0	7.2	8.4	7.4	6.4	8.0	8.2	11.8	8.6	8.0	6.4
Borate	5.8	7.6	8.8	7.0	9.8	5.8	6.0	7.2	6.2	6.0	6.6	6.8	5.6
Base	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5
Target	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5



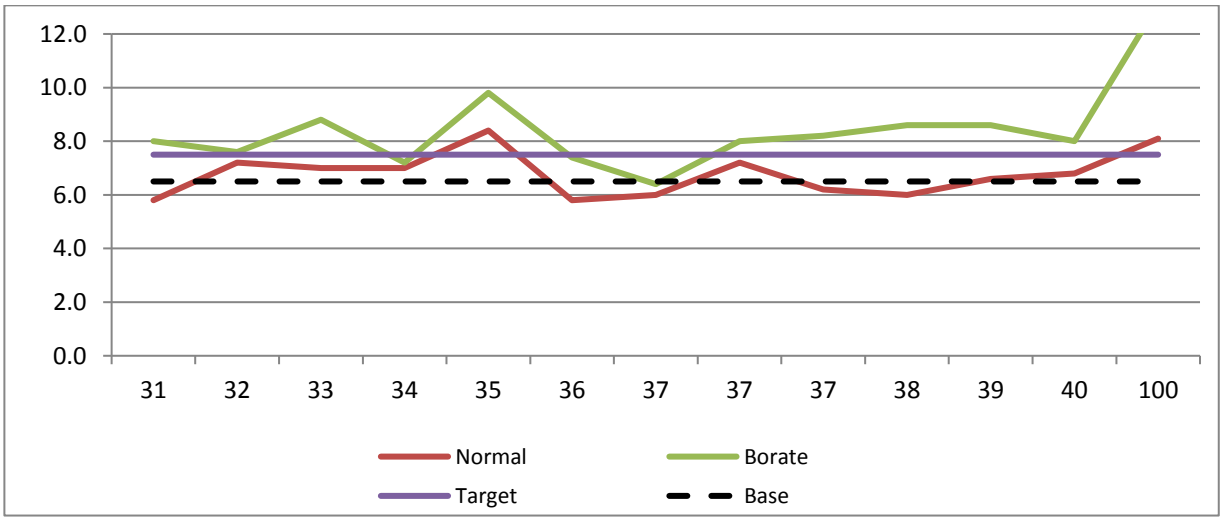
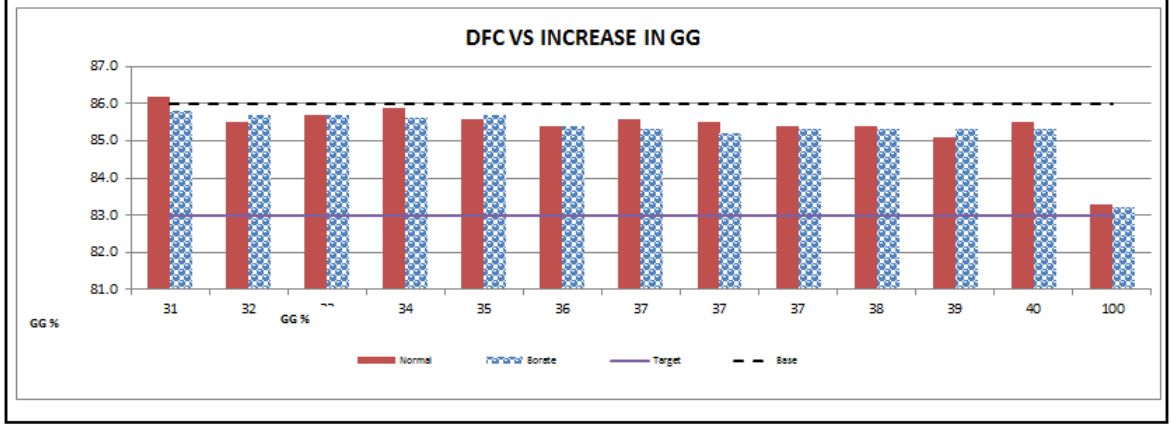


Figure 16: Sulphur percentage in coke versus percentage Sulphur in blend table and graphically presented.

4.6 Effect of borate quenching on Dry Fixed Carbon (DFC)

Dry Fixed Carbon (DFC) in coke should represent at least 83% by mass or more (ArcelorMittal standard). The results how that there is a steady decline in fixed carbon content with increased proportions of GG coal in the blends, but all values are higher than the minimum acceptable value of 83% for all blends including up to 40% GG. Thus there is no clear gain in DFC when one compares the results of the normally quenched coke with the borate solution quenched coke. The results also showed that the single GG component had carbon content above 83% for both the borate and the normal treated coke. Thus, quenching had no effect on the carbon content of the cokes produced, whether using borate-treated or untreated water, as indicated in Figure 17 below.

DFC (>83)													
GG %	31	32	33	34	35	36	37	37	37	38	39	40	100
Normal	86.2	85.5	85.7	85.9	85.6	85.4	85.6	85.5	85.4	85.4	85.1	85.5	83.3
Borate	85.8	85.7	85.7	85.6	85.7	85.4	85.3	85.2	85.3	85.3	85.3	85.3	83.2
Base	86.0	86.0	86.0	86.0	86.0	86.0	86.0	86.0	86.0	86.0	86.0	86.0	86.0
Target	83	83	83	83	83	83	83	83	83	83	83	83	83



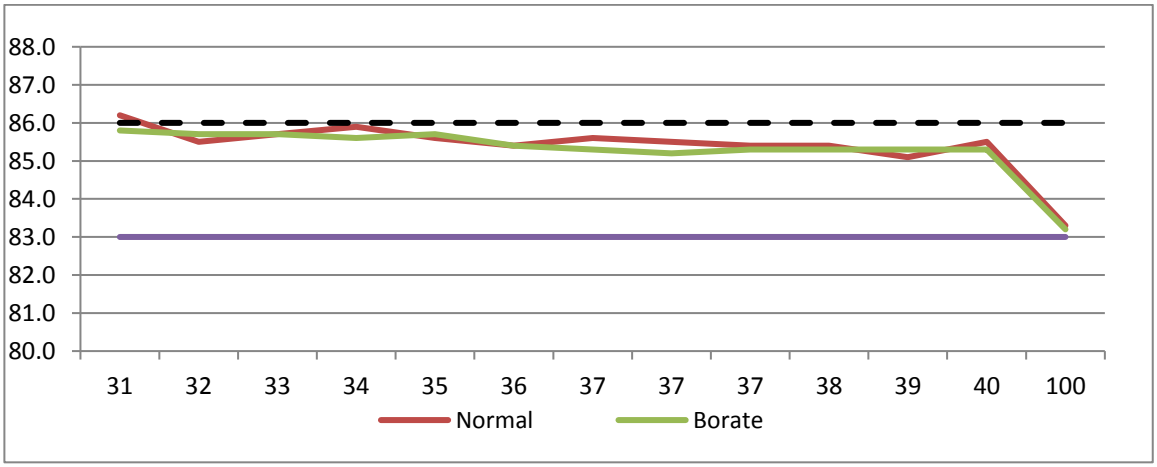


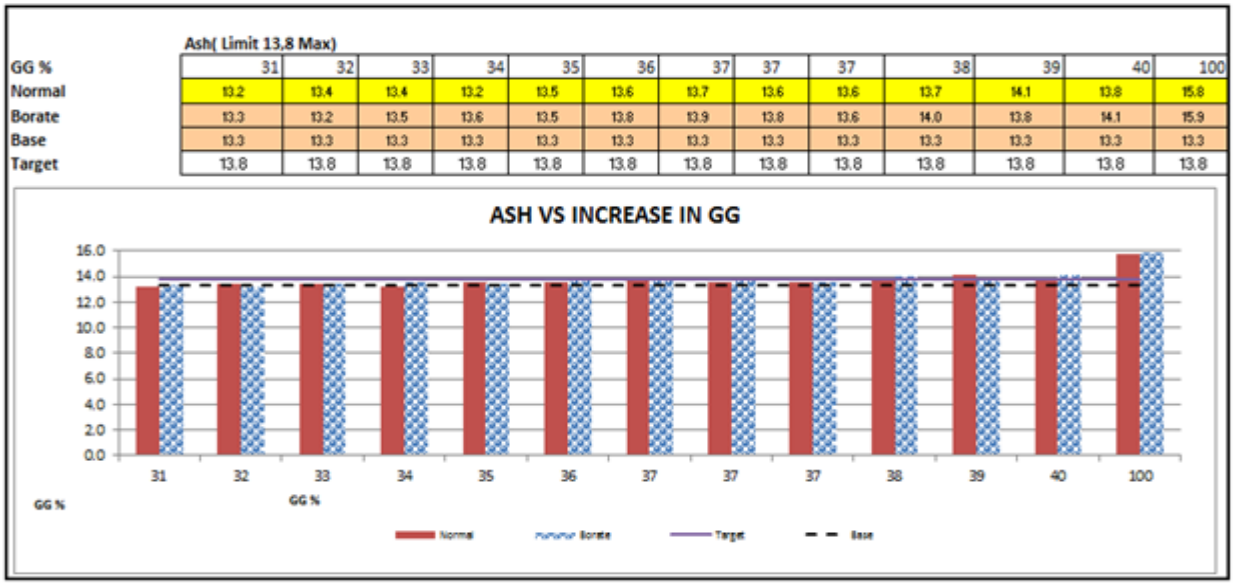
Figure 17: DFC Versus GG percentage in the blend – table above and graphically presented

4.7 Effect of borate on coke ash content

The upper limit on ash content for blend coals used in Arcellor Mittal coke making is 13.8%. The analysis of ash contents in all cokes produced was stable for most of the blends up to 39% GG content. Comparison between cokes treated with borate-containing water and those treated with normal water showed that borate had no effect on the ash content of the resulting cokes in all cases. The single component coal, GG (100%), showed a high ash content of up to 15, 9%, well above the acceptable limit of 13, 8% required for blast furnace operation. Figure 18 presents all the relevant results in tabulated and graphic form.

Ash(Limit 13,8 Max)

GG %	31	32	33	34	35	36	37	37	37	38	39	40	100
Normal	13.2	13.4	13.4	13.2	13.5	13.6	13.7	13.6	13.6	13.7	14.1	13.8	15.8
Borate	13.3	13.2	13.5	13.6	13.5	13.8	13.9	13.8	13.6	14.0	13.8	14.1	15.9
Base	13.3	13.3	13.3	13.3	13.3	13.3	13.3	13.3	13.3	13.3	13.3	13.3	13.3
Target	13.8	13.8	13.8	13.8	13.8	13.8	13.8	13.8	13.8	13.8	13.8	13.8	13.8



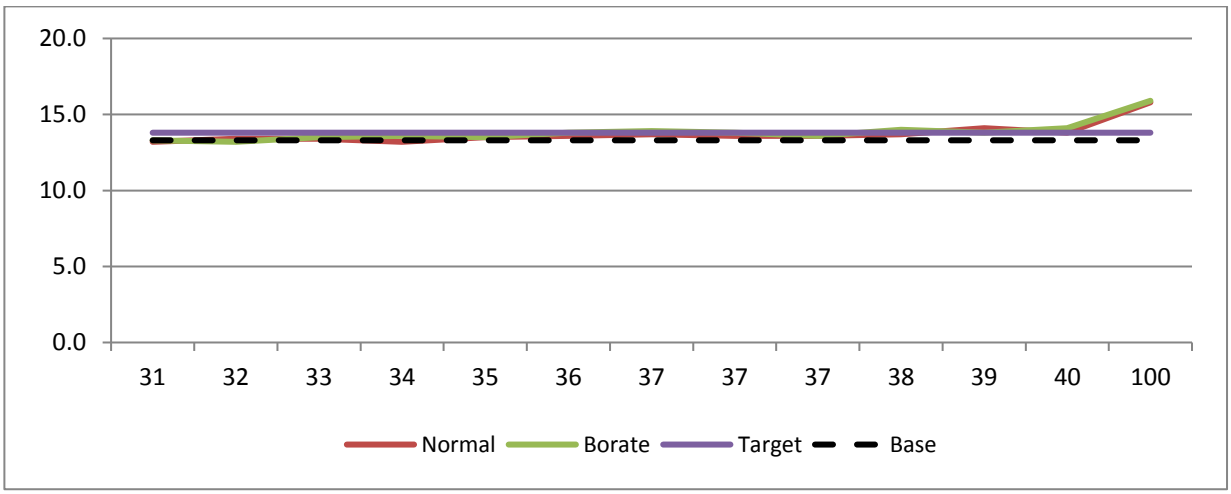
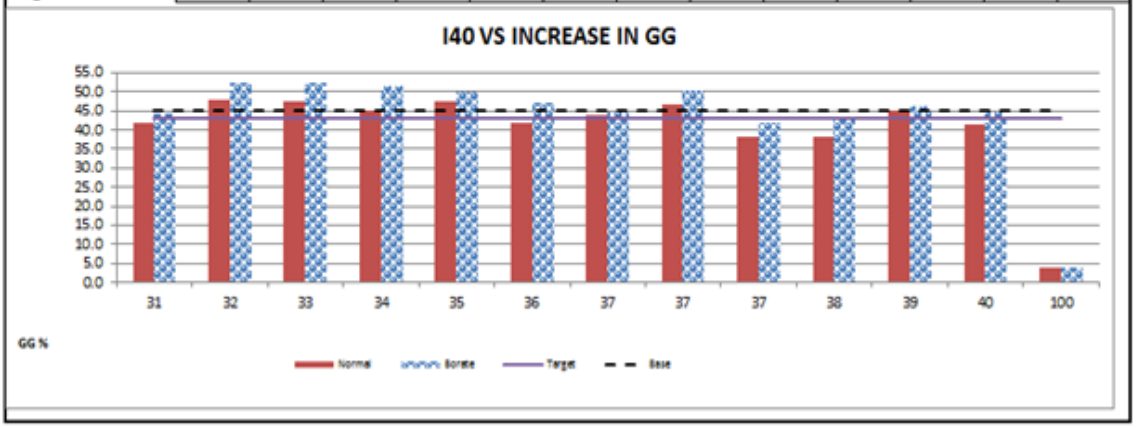


Figure 18: Ash content Versus GG percentage in the blend (table and graphically presented)

4.8 Effect of borate on I40

The minimum limit on I40 index for blend coals used in Arcellor Mittal coke making is 43. I40 of the coke also showed a clear and positive trend whereby borate-treated cokes exhibited stronger I40 values compared to the normal water treated coke components. i.e. borate-treated cokes retained their strength and larger size distribution after screening following borate treatment as evidenced by values above base level. This trend was evident for all the blends including the sample with pure 100% GG coal. Again, this confirms the strong and positive impact on the mechanical strength of the cokes after being subjected to borate-treatment. Another trend related to the effect of increasing GG proportions in the blends, showed that, while the normal water treated cokes presented poorer I40 results on blends with 32% GG and above, the blends treated with borate water with the same GG% proportions had good I40 values, up to 39% GG. Figure 19 presents the tabulated and graphic results of both these trends.

I40(limit 43 minimum)													
GG %	31	32	33	34	35	36	37	37	37	38	39	40	100
Normal	41.8	48.1	47.4	45.2	47.6	42.0	44.0	46.8	38.4	38.2	45.2	41.6	4.0
Borate	44.4	52.6	52.4	51.6	50.2	47.2	45.0	50.6	42.0	43.2	46.4	44.8	4.2
Base	44.9	44.9	44.9	44.9	44.9	44.9	44.9	44.9	44.9	44.9	44.9	44.9	44.9
Target	43	43	43	43	43	43	43	43	43	43	43	43	43



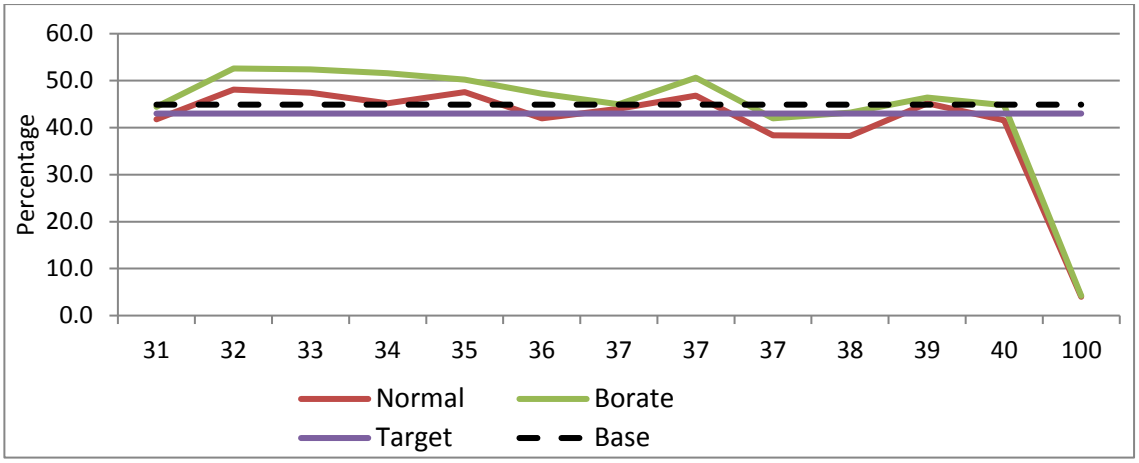
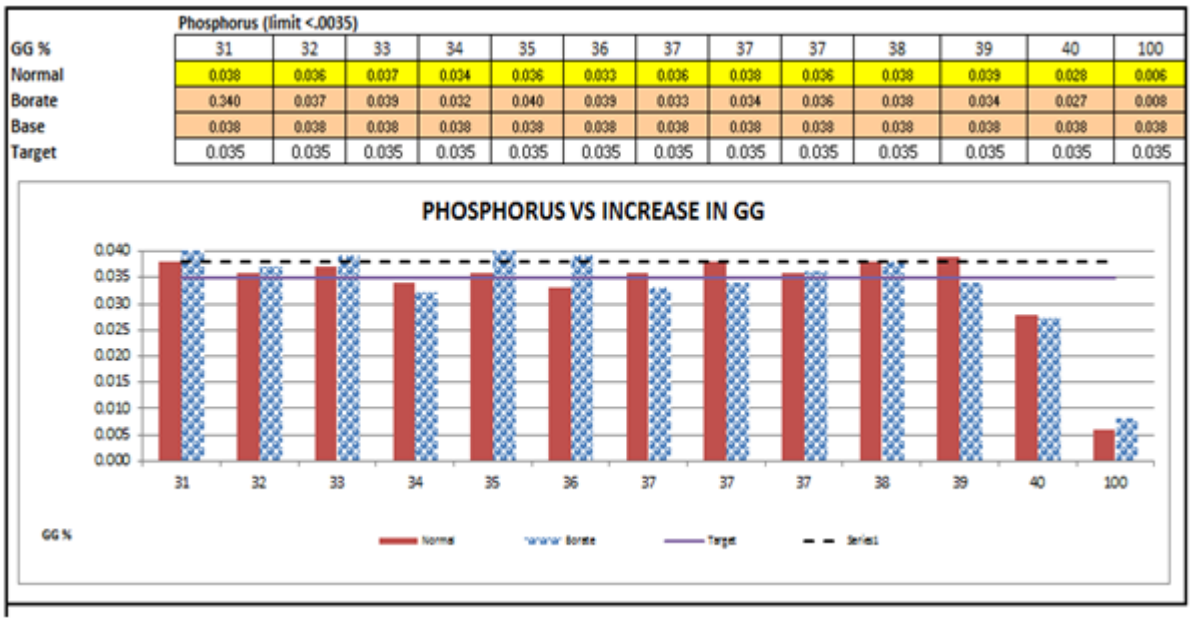


Figure 19: I40 values Versus percentage GG in the blend (table and graphically presented)

4.9 Effect of borate quenching on phosphorus content in the coke

As observed with all chemical compositions such as suphur ,dry fixed carbon and ash content, the borate treatment of the coke during quenching did not alter the chemical balance of the coke. All blends stayed well below the maximum limit of 0.043 required by the blast furnace. Thus the range up to 40% GG was well within upper limits compared to both target and the base (which is the phosphorus content at 30%). The different blends from 31 to 40 percentage GG did not have any impact on the upper limit of the phosphorus.



Phosphorus (limit <.0035)

GG %	31	32	33	34	35	36	37	37	37	38	39	40	100
Normal	0.038	0.036	0.037	0.034	0.036	0.033	0.036	0.038	0.036	0.038	0.039	0.028	0.006
Borate	0.340	0.037	0.039	0.032	0.040	0.039	0.033	0.034	0.036	0.038	0.034	0.027	0.008
Base	0.038	0.038	0.038	0.038	0.038	0.038	0.038	0.038	0.038	0.038	0.038	0.038	0.038
Target	0.035	0.035	0.035	0.035	0.035	0.035	0.035	0.035	0.035	0.035	0.035	0.035	0.035

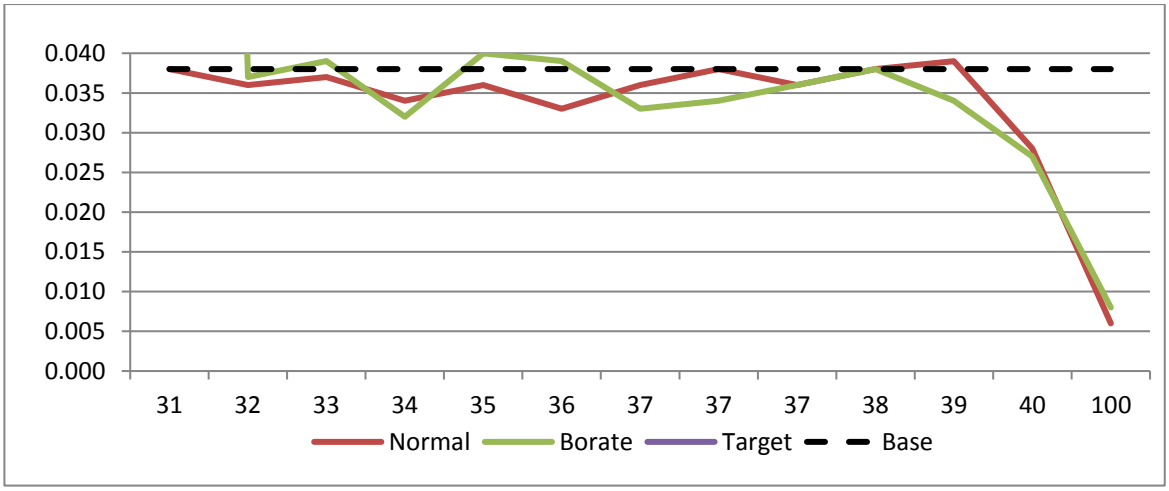


Figure 20: Phosphorus content Versus GG percentage (table and graphically presented)

4.10 Effect of increase in GG on coke oven wall pressure

Wall pressures above 10Kpa are not acceptable for any coking coal blends in a coke plant as this causes difficulties in pushing the coke out of the coke ovens. When tested alone, GG coke exhibited a low propensity to create dangerous wall pressure in the coke oven and for this reason GG coal requires to be a significant part of any future blend. Grootegeluk (GG) is a high volatile coal and is generally expected to not generate excessive wall pressures but its incorporation into the blend when increasing its percentage must be watched closely to see if any increase (or decrease) in oven wall pressure is observed as this will have a bearing on the ease of pushing of the coke. The results of the tests undertaken showed that all the coke blends under investigation, ranging from blends with 31% GG up to 40% GG were well below the acceptable upper limit.

This infers that at no proportion of GG coal, and with borate treatment quenching as well as normal water treatment, there was no impact on wall pressure for any of the blends. Figure 21 presents these results in tabulated and graphic form.

Wall Pressure	32.00	32.00	33.00	33.00	34.00	34.00	35.00	35.00	36.00	36.00	37.00	37.00	38.00	38.00
Kpa	2.85	2.85	3.38	3.38	3.57	3.57	3.45	3.45	4.27	4.27	4.36	4.36	2.72	2.72
Target	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
Base	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00

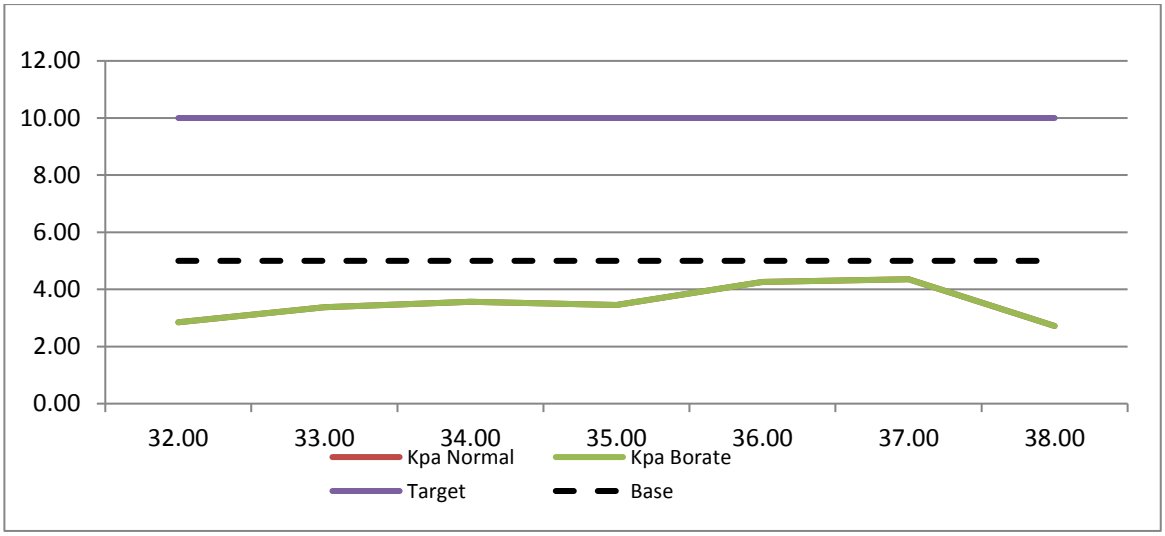


Figure 21: Wall pressure Vs percentage GG in blend (table and graphically presented)

4.11 Porosity tests on normal and borate treated coke

Table 5: Results for porosity tests on borate and normal coke

Sample Identification	COKE SAMPLE BORATE	COKE SAMPLE NORMAL
Total Moisture *(%)*	2.3	3.3
ARD	0.96	0.89
TRD	1.38	1.49
Apparent Porosity *(%)*	16	17
Sealed Porosity *(%)*	15	23
True Porosity *(%)*	31	40

Tests on porosity were conducted on both normal quenched coke and the coke treated with borate water indicated that the borate water quenched coke had lower porosity. The tests were not done by the researcher in the pilot plant. Two samples were sent to an independent laboratory where porosity tests were done as per ASTM D167, ISO 13909-4; 2001 and AS 1038.21.1.1-2008. This result is assumed to infer that the borate molecules, once attached or absorbed onto the available surfaces of the coke matrix, block access to the normal pores which would be available when only water is used to quench the cokes. The reduction in moisture content supports this apparent blockage of pores but the presence of some total

moisture indicates retained moisture content internally, within the coke particles, possibly where porosity has not been reached by borate molecules. CRI results earlier showed an increased reactivity on coke quenched with normal water. The results obtained by Longbottom (2016) showed an increased reactivity on coke with less porosity and has a direct relationship with the results of this research.

4.12 Summary of tests results and discussions

In summary, and based on the results and observations above, it is postulated that the intermolecular bonding of borate molecules with those on the surface of coke particles provides a film or "skin" covering the surface of the coke particles and block the surface pores. This situation would provide lower surface porosity for the coke particles quenched with the borate treated water and, in terms of overall effect, the apparent film or skin is likely to give rise to greater strength to the surface of the particle leading to higher resistance to breakage. This would explain the improvement of the CSR/ CRI results giving an overall benefit from the opportunity presented from the good CSR at as high as 37%GG local coal inclusion in the blend. Research done by Longbottom (2016) which showed an increased reactivity on porous coke agrees with findings of this research. Inhibition of coke reactivity by the borate molecules adsorbed onto the coke is the main driving force behind the improved hot strength observed in this research.

Work done by Tamko *et al.* (2010) and Filatov (2012) where improvements of CSR by 17% has shown that it can be replicated in the South African blends to achieve significant gains in CSR improvement by the quenching of the use of borates for quenching coke.

Chemical and proximate composition of the quenched coke does not change at all because the act of quenching does not alter the chemical constitution of the coke. This is because the act of quenching happens after the coking process.

4.13 Techno-economic Evaluation

This chapter provides a techno-economic impact of the results of this research, i.e. the potential economic benefit of improving the proportional content of local coals in the coke blends being produced in South Africa versus importing expensive international coals, and the impact borate quenching may have on this situation. The technological evaluation of the above steps in making the coal blends was supplemented by an economic evaluation of the

cokes currently being produced, both with and without borate treatment, and with and without increased GG coal inputs.

4.13.1 The coke making value chain (Industrial scale)

In the coke making value chain, the quenching step appears to be highly significant. The following figure (Figure 22) the methods of quenching. The impact of quenching using borate chemicals needs to be considered versus the increase in GG coals along with the reduction of imported coals. The quenching cycle is a simple one which starts with the pumping of quenching water from the pond into the quenching tanks. The hot load of coke inside a quench car is then quenched by use of about 33 000 litres of water releasing a third of that as quenching steam. It is then assumed in the calculation, that the make-up rate after each quench would then be approximately 13 000 litres.

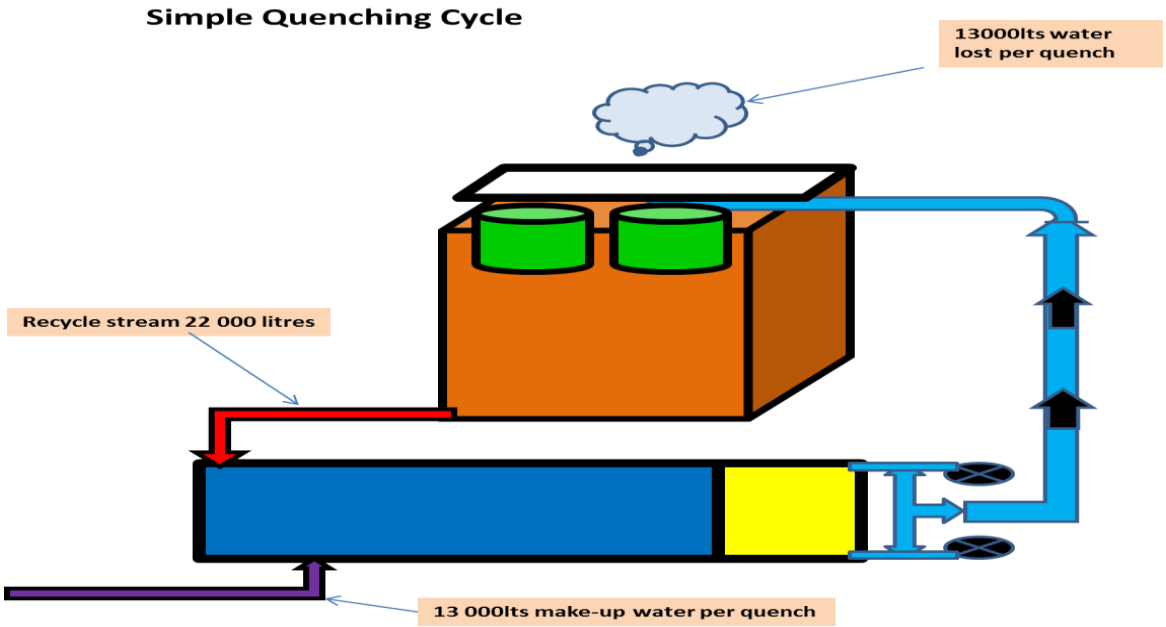


Figure 22: Sketch of the water circulation for industrial wet quenching of coke

Below is a simple calculation showing the impact of borate on the overall cost of the coke for the production of a tonne of hot-metal at the blast furnace.

Table 6. Table 6: Cost benefit analysis calculation

Description	Figure/ Calculation
Quench water amount used per load	35 000 litres
Water vaporing off the quench	13 000 litres
The amount of borate used per quench	13000 x 10grams = 0.13tonnes
The price per tonne of the borate	$300 (general international price for industrial borate)
cost per quench per 18 tonnes (one oven worth of coke)	0.13x300/18 = $2.1666x14.6 = R 31
Saving per tonne (see Table 5)	1586-1528 = 58/ tonne (difference between cost of control blend at 30% GG and the cost at 37%) ….see table 5
Grand saving	R27/tonne
Saving per day	27 x 2000 = R54000/ day
Saving per year	R19, 710,000 per year

Table 7: Blend prices with percentage increase in GG

Comments	Control blend	Blend 1	Blend 2	Blend 3	Blend 4	Blend 5	Blend 6	Blend 7
Blend Composition %								
GrooteGeluk	30	31	32	33	34	35	36	37
Oakey North	27	26	25	24	23	22	21	20
Typical (Ex - Mozambique)	8	8	8	8	8	8	8	8
Shoal Creek	17	17	17	17	17	17	17	17
Hail Creek (Ex-Australia)	18	18	18	18	18	18	18	18
Total	100	100	100	100	100	100	100	100
Blend Price	R 1 586	R 1 577	R 1 568	R 1 561	R 1 552	R 1 544	R 1 536	R 1 528

In summary, the economic data above presents the significant cost benefit of including higher quantities of GG coal in the coke blends but only if borate treatment can be used to enhance the cokability properties of the resultant cokes. It is strongly recommended that further work be done to expand this text work to higher levels of commercial trials in order to verify the benefits derived during the course of this research.

4.14 Limitations of the research

I. The wet quenching methods introduce non-uniformity in the constitution of the resultant quenched areas in the coke. Zolatev *et al.* (2010) noted that the production conditions in chamber furnaces yield polydispersity in coke, which differs in properties in different lumps of coke and in granulometric composition. Therefore, even with precise specification of the cooling water supply and other factors (such as the level, composition, and temperature of the water), the moisture content of the coke may fluctuate.

II. An increased number of experiments to obtain statistically verifiable results was not possible. This was because each experiment was extremely expensive to conduct. One single test for one blend cost in excess of R30 000 to complete. The current research included a total of thirteen tests.

III. The industrial use of the borates generates boric acid which needs to be handled with caution. The costs related to this step were not explored in this research as information on methods used to handle in borate liquids is lacking.

IV. There is no readily available data and information on the cost of industrial installation of the borate quenching facility. The pay-back period needs to be considered in the savings calculation. This information is missing in the techno-economic-evaluation

V. The calculations on the techno-evaluation could only be made by assuming that all the salt is absorbed per quench. In real terms, this is not possible. The tools to get a better evaluation would have to be pursued.

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusions

Based on the research undertaken in this investigation, the following conclusions may be drawn:

I. It is possible to improve the key properties of blend coking coals, and more specifically CSR/CRI, by quenching the coke with a solution of borate as it is exits the coke ovens.

II. The success rates and proven results in terms of increased cokability specifications when borate chemicals are used to quench cokes agree exactly with those results reported by authors in Russia and China

III. Based on the success achieved using borate quenching, it has been possible to incorporate higher proportions of local blend coking coals with lower proportions of imported prime coking coals

IV. Incorporating higher proportions of local coals (GG) in the cokes produced in South Africa has incurred a considerable reduction in the cost of the specialised metallurgical cokes required by the iron and steel industries.

V. The proportions of GG in the blend of coals increased from 30% to 37%, a gain of seven percentage points which represents a saving of over R19 million per year in the manufacture of metallurgical coke.

VI. In practical terms, the pushability of the coke made with increasing amounts of GG was not affected, and neither was the quality of the resulting cokes as determined by chemical composition, ash content, sulphur and phosphorus contents.

In summary, if implemented, it is believed that the results of this research are likely to lead to significant techno-economic improvements in the production of South African metallurgical coke through the use of a higher proportion of local coals whilst reducing the necessity for the importing of expensive global coking coals.

5.2. Recommendations

It is recommended that further work be conducted in the following areas:

I. Increase the number of experiments to obtain statistically verifiable results and to include testing additional variables such as different particle sizes in the coke blend mix.

II. Establish how to handle boric acid arising from borate as when it reacts with water as it has the effect of causing contaminations and toxic effects in the biological treatment of wastewater.

III. Evaluate changes or additions in emissions that may arise from the use of borate matter in coke blends

IV. Evaluate other South African coals, apart from the GG coals, that could be incorporated into imported prime coking coals and still achieved the same acceptable coke specifications. This is important as it would address the need for substitution, should GG coal become unavailable in future.

V. The effect of reduction of import coal with increase in GG is not articulated in this research and calls for further research

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APPENDICES

Appendix 1 - Single component tests on Grootegeluk coal

Details						
Date	23-Jan-17	26-Jan-17	16-Feb-17	08-Mar-17	06-Apr-17	28-06-2017
Identity	NCO1932	NCO1935	NCO1946	NCO1952	NCO1965	NCO1990
Origin / Batch	Lab	Lab	Lab	Lab	Lab	Lab
Comments	100 % GG	100 % GG	100 % GG	100 % GG	100 % GG	100 % GG
Blend Composition						
Grootegeluk	100	100	100	100	100	100
Total	**100**	**100**	**100**	**100**	**100**	**100**
Coal Proximate Analysis						
Ash	10.0	10.1	9.9	10.0	10.2	10.4
S	1.15	1.10	1.07	1.13	1.10	1.14
Vol	36.0	36.5	36.3	36.2	36.2	36.7
Roga	73	74	71	70	69	78
Coal Chemical						
Na_2O	0.01	0.00	0.00	0.01	0.00	0.00
MgO	0.01	0.01	0.00	0.00	0.00	0.00
Al_2O_3	2.5	1.9	1.7	1.5	2.4	2.3
SiO_2	5.7	7.1	6.3	7.0	6.7	6.3
P	0.006	0.005	0.005	0.005	0.005	0.005
K_2O	0.10	0.11	0.10	0.09	0.09	0.10
CaO	0.10	0.11	0.05	0.31	0.08	0.07
TiO_2	0.28	0.26	0.31	0.01	0.27	0.26
Mn	0.008	0.009	0.01	0.01	0.006	0.008
Fe	0.5	0.5	0.6	0.5	0.4	0.5
Plasticity						
SIT	423	394	396	386	401	403
TMF	468	430	430	421	429	428
MF (ddpm)	44	29	64	65	8	38
RT	503	454	454	447	452	459
PR	80	60	58	61	51	56
Dilatation & Koppers						
T1	387	393	387	385	411	405
T2	444	420	408	441	447	438
T3	465	483	441	471	489	465
Max C%	18.6	19.9	26.2	24.9	15.5	21.8
Max D%	12.5	21.7	26.7	5.4	26.8	13
Amplitude	31.1	41.6	52.8	30.3	42.3	34.8
Koppers	-25.5	-31.4	-31.6	-33.0	-30.3	-26.5
Coal Screen Analysis						
+25mm	0.0	0.0	0.0	0.0	0.0	0.0
+20mm	0.0	0.0	0.0	0.0	0.0	0.0
+16mm	0.0	0.0	0.0	0.0	0.0	0.0
+10mm	0.0	0.0	0.0	0.0	0.0	0.0
+8mm	3.5	2.6	2.2	3.4	2.6	0.3
+6.3mm	10.4	7.4	7.4	9.8	8.4	8.5
+5mm	18.4	13.3	14.1	17.4	15.8	15.6

+4mm	26.4	20.1	21.9	25.2	23.8	23.2
+3.35mm	29.4	22.3	24.9	27.4	27.5	25.6
+2mm	52.6	44.2	48.7	51.1	51.7	48.2
+1mm	74.2	67.7	73.8	72.3	75.6	70.0
+0.5mm	88.8	84.1	89.3	87.0	90.0	86.6
-0.5mm	11.2	15.9	10.7	13.0	10.0	13.4
+.212mm	93.9	94.2	96.1	94.5	95.8	92.6
+0.150mm	94.7	95.8	97.2	96.3	96.9	94.3
+0.106mm	95.4	96.9	98.0	97.5	97.6	95.6
-0.106mm	4.6	3.1	2.0	2.5	2.4	4.4
Sm	2.7	2.4	2.5	2.7	2.6	2.5
Total -3.35	70.6	77.7	75.1	72.6	72.5	74.4
Coal Density						
-	839	832	825	839	835	810
Oven (Wet)	966	966	966	966	966	854
ASTM (Wet))	864	868	854	879	860	901
Oven (Dry)	903	903	903	903	903	798
ASTM (Dry))	808	812	798	822	804	842
Coke Details						
Coke Identity	PLCS	PLCS	PLCS	PLCS	PLCS	PLCB
Origin	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven
Coke Proximate Analysis						
Ash	15.7	15.8	15.7	15.5	15.5	15.6
S	0.89	0.95	0.99	0.95	0.97	0.95
Vol	1.0	1.1	1.0	0.9	1.0	1.00
Fixed Carbon	83.3	83.1	83.3	83.6	83.5	83.4
Coke Chemical Analysis						
Na_2O	0.00	0.00	0.0	0.0	0.0	0.0
MgO	0.1	0.1	0.1	0.1	0.1	0.1
Al_2O_3	2.9	2.9	3.0	2.9	2.9	3.0
SiO_2	10.0	9.7	10.6	9.6	9.5	8.6
P	0.007	0.009	0.009	0.008	0.008	0.007
K_2O	0.17	0.16	0.2	0.2	0.2	0.2
CaO	0.20	0.30	0.1	0.2	0.2	0.3
TiO_2	0.30	0.29	0.3	0.3	0.3	0.3
Mn	0.000	0.010	0.0	0.0	0.0	0.0
Fe	0.9	1.0	0.8	0.8	0.7	0.8
Other Coke Properties						
H_2O	8.4	8.8	8.0	7.4	6.8	8.0
Theoritical Yield %	64.6	64.1	64.3	64.4	64.4	63.9
Coke Actual Yield %	76.6	76.5	74.8	76.0	82.2	77.7
Stability						
Hardness						
Hot Strength						
CRI	48.1	45.5	44.6	47.1	47.4	45.2
CSR	28.0	28.6	27.2	25.7	27.4	25.3
Corrected CRI (- 5 units)	45.1	42.5	41.6	44.1	44.4	42.2
Corrected CSR (+ 5 units)	33.0	33.6	32.2	30.7	32.4	30.3
Micum & Irsid						
M40	28.9	27.8	29.8	29.4	30.6	26.0

M30	57.1	57.4	56.7	56.5	61.4	54.6
M10	8.3	7.7	9.1	8.3	7.7	6.4
I40	7.8	7.2	6.7	7.4	7.4	8.6
I30	26.3	26.6	22.7	22.5	28.5	25.4
I20	64.4	64.6	61.7	61.4	67.1	72.0
I10	20.4	20.0	21.3	19.3	19.0	7.6
Coke Screen Analysis						
%-125+100mm	0.0	1.1	0.4	1.3	0.0	0.8
%-100+ 80mm	5.4	5.7	5.5	5.5	2.1	3.1
%-80+ 63mm	10.7	15.1	12.4	11.4	7.0	9.1
%-63+ 40mm	55.2	51.3	50.5	50.1	52.9	52.9
%-40+ 35mm	6.1	6.3	6.4	6.9	9.8	11.3
%-35+ 30mm	8.2	8.3	7.6	8.0	9.5	4.4
%-30+ 20mm	8.0	7.6	8.4	9.5	9.5	11.3
%-20 +10mm	3.3	2.7	4.1	4.0	4.6	4.1
%-10mm	3.0	2.0	4.7	3.2	4.6	3.0
Sm	48.6	51.0	48.1	48.7	44.2	46.7
Tot +80mm	5.4	6.8	5.9	6.8	2.1	3.9
Tot+35mm	77.5	79.5	75.1	75.2	71.7	77.3
Tot+30mm	85.7	87.7	82.7	83.2	81.2	81.7
Tot -30mm	14.3	12.3	17.3	16.8	18.8	18.3
Depth Meter						
Pp.(Inkrimp)	0.299	0.493	meter	0.11	0.287	0.034
TE (Uitsetting)	0.518	0.675	broken	0.51	0.433	0.212
Wall Pressure						
kN	3.70	2.45	2.67	2.70	2.13	4.03
kPa	4.77	3.16	3.44	3.48	2.74	5.19
mb	47.68	31.57	34.41	34.79	27.45	51.93
PSI	0.69	0.46	0.50	0.50	0.40	0.75
Coke End Temp						
tfin.°C	1050	1051	1051	1050	1031	1051
Coke Rate						
Plastic Zone (474°C) /Hours	13.55	14.70	13.75	13.95	11.90	15.05
Plastic Zone target to 474°C	14.2	14.2	14.2	14.2	14.2	14.2
Coke rate to 900°C/Hours	16.20	16.55	16.85	17.20	15.20	17.20
Coke Rate target to 900°C	16.3	16.3	16.3	16.3	16.3	16.3
Total Coking Cycle / Hours	18.05	18.65	18.65	19.20	17.25	18.65
k (g/cmh)	32.8	32.1	31.5	30.9	34.9	27.3
P Force						
Energie Kw/h	43	43	48	39	48	40
Oven Temperatures Settings						
Start Temperature	900°C	900°C	900°C	900°C	900°C	900°C
End Temperature	1200°C	1200°C	1200°C	1230°C	1230°C	1230°C

Appendix 2. Chemical and physical analyses on coal blends

Details																							repeat	repeat	repeat 2	repeat 2
Date	13-May-16	13-May-16	09-Apr-16	09-Apr-16	10-Apr-16	10-Apr-16	12-Apr-16	12-Apr-16	15-Apr-16	15-Apr-16	16-Apr-16	16-Apr-16	17-Apr-16	17-Apr-16	18-Apr-16	18-Apr-16	11-May-16	11-May-16	12-May-16	12-May-16	01-Jun-16	01-Jun-16	16-Jun-16	16-Jun-16	22-Jun-16	22-Jun-16
Identity	NCO1809	NCO1809	NCO1795	NCO1795	NCO1796	NCO1796	NCO1797	NCO1797	NCO1800	NCO1800	NCO1801	NCO1801	NCO1802	NCO1802	NCO1803	NCO1803	NCO1807	NCO1807	NCO1808	NCO1808	NCO1828	NCO1828	NCO1829	NCO1829	NCO1830	NCO1830
Origin	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
Comments	Blend 1 (normal)	Blend 1 (borate)	Blend 2 (normal)	Blend 2 (borate)	Blend 3 (normal)	Blend 3 (borate)	Blend 4 (normal)	Blend 4 (borate)	Blend 5 (normal)	Blend 5 (borate)	Blend 6 (normal)	Blend 6 (borate)	Blend 7 (normal)	Blend 7 (borate)	Blend 8 (normal)	Blend 8 (borate)	Blend 9 (normal)	Blend 9 (borate)	Blend 10 (normal)	Blend 10 (borate)	100% GG (normal)	100% GG (borate)	Blend 7 (normal)	Blend 7 (borate)	Blend 7 (normal)	Blend 7 (borate)
Blend Composition %																										
GrooteGeluk	31	31	32	32	33	33	34	34	35	35	36	36	37	37	38	38	39	39	40	40	100	100	37	37	37	37
Oakey North	26	26	25	25	24	24	23	23	22	22	21	21	20	20	19	19	18	18	17	17			20	20	20	20
Typical (Ex - Mozambique)	8	8	8	8	8	8	8	8	8	8	8	8	8	8	8	8	8	8	8	8			8	8	8	8
Shoal Creek	17	17	17	17	17	17	17	17	17	17	17	17	17	17	17	17	17	17	17	17			17	17	17	17
Hail Creek (Ex-Australia)	18	18	18	18	18	18	18	18	18	18	18	18	18	18	18	18	18	18	18	18			18	18	18	18
Total	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**
Coal Proximate Analysis																										
Ash	9.5	9.5	9.6	9.6	9.8	9.8	9.7	9.7	9.9	9.9	9.7	9.7	9.9	9.9	9.8	9.8	9.6	9.6	9.6	9.6	10.0	10.0	9.7	9.7	9.9	9.9
S	0.80	0.80	0.74	0.74	0.77	0.77	0.80	0.80	0.78	0.78	0.78	0.78	0.74	0.74	0.78	0.78	0.81	0.81	0.82	0.82	1.12	1.12	0.82	0.82	0.82	0.82
Vol	27.4	27.4	27.4	27.4	28.2	28.2	27.6	27.6	27.6	27.6	28.5	28.5	28.2	28.2	28.4	28.4	28.7	28.7	28.8	28.8	36.2	36.2	28.9	28.9	28.3	28.3
Roga	70	70	77	77	73	73	74	74	69	69	67	67	65	65	64	64	84	84	77	77	70	70	70	70	76	76
Coal Chemical																										
Na_2O	0.03	0.03	0.04	0.04	0.04	0.04	0.04	0.04	0.05	0.05	0.04	0.04	0.04	0.04	0.03	0.03	0.03	0.03	0.05	0.05	0.00	0.00	0.03	0.03	0.10	0.10
MgO	0.06	0.06	0.04	0.04	0.04	0.04	0.07	0.07	0.06	0.06	0.07	0.07	0.06	0.06	0.06	0.06	0.06	0.06	0.07	0.07	0.01	0.01	0.05	0.05	0.09	0.09
Al_2O_3	2.8	2.8	2.2	2.2	2.1	2.1	2.4	2.4	2.7	2.7	2.8	2.8	2.8	2.8	2.5	2.5	2.4	2.4	2.5	2.5	2.1	2.1	2.5	2.5	2.6	2.6
SiO_2	5.5	5.5	4.0	4.0	5.0	5.0	5.1	5.1	5.2	5.2	5.7	5.7	5.7	5.7	5.7	5.7	4.8	4.8	4.8	4.8	6.3	6.3	5.7	5.7	4.8	4.8
P	0.043	0.043	0.035	0.035	0.034	0.034	0.036	0.036	0.039	0.039	0.039	0.039	0.038	0.038	0.037	0.037	0.037	0.037	0.034	0.034	0.004	0.004	0.041	0.041	0.041	0.041
K_2O	0.16	0.16	0.14	0.14	0.13	0.13	0.17	0.17	0.15	0.15	0.15	0.15	0.16	0.16	0.15	0.15	0.15	0.15	0.16	0.16	0.10	0.10	0.16	0.16	0.17	0.17
CaO	0.21	0.21	0.17	0.17	0.16	0.16	0.13	0.13	0.20	0.20	0.21	0.21	0.22	0.22	0.17	0.17	0.18	0.18	0.32	0.32	0.06	0.06	0.17	0.17	0.29	0.29
TiO_2	0.24	0.24	0.26	0.26	0.27	0.27	0.22	0.22	0.24	0.24	0.24	0.24	0.24	0.24	0.23	0.23	0.24	0.24	0.27	0.27	0.20	0.20	0.25	0.25	0.27	0.27
Mn	0.005	0.005	0.004	0.004	0.004	0.004	0.003	0.003	0.005	0.005	0.005	0.005	0.006	0.006	0.004	0.004	0.005	0.005	0.009	0.009	0.007	0.007	0.005	0.005	0.010	0.010
Fe	0.6	0.6	0.5	0.5	0.6	0.6	0.4	0.4	0.5	0.5	0.5	0.5	0.6	0.6	0.5	0.5	0.5	0.5	0.8	0.8	0.4	0.4	0.5	0.5	0.9	0.9
Plasticity																										
SIT																					397	397	409	409	408	408
TMF	out	out	out	out	out	out	out	out	out	out	out	out	out	out	out	out	out	out	out	out	436	436	452	452	458	458
MF (ddpm)	of	of	of	of	of	of	of	of	of	of	of	of	of	of	of	of	of	of	of	of	58	58	213	213	346	346
RT	order	order	order	order	order	order	order	order	order	order	order	order	order	order	order	order	order	order	order	order	457	457	489	489	494	494
PR																					63	63	80	80	86	86
Dilatation & Koppers																										
T1	399	399	390	390	396	396	375	375	393	393	393	393	384	384	390	390	390	390	390	390	378	378	387	387	381	381
T2	420	420	426	426	426	426	426	426	426	426	423	423	423	423	432	432	429	429	408	408	411	411	420	420	414	414
T3	483	483	471	471	480	480	474	474	474	474	483	483	477	477	486	486	465	465	483	483	471	471	477	477	480	480
Max C%	31.7	31.7	25.4	25.4	30.1	30.1	34.4	34.4	24.7	24.7	33.6	33.6	22.5	22.5	24.9	24.9	23.7	23.7	26.5	26.5	25.2	25.2	24.2	24.2	26.8	26.8
Max D%	54.4	54.4	38.9	38.9	26.5	26.5	26.4	26.4	47.3	47.3	43.3	43.3	37.6	37.6	24.8	24.8	22.0	22.0	61.3	61.3	8.9	8.9	62.3	62.3	69.9	69.9
Amplitude	86.1	86.1	64.3	64.3	56.6	56.6	60.8	60.8	72.0	72.0	76.9	76.9	60.1	60.1	49.6	49.6	45.7	45.7	87.8	87.8	34.1	34.1	86.5	86.5	96.8	96.8
Koppers	-25.6	-25.6	-22.9	-22.9	-28.4	-28.4	-30.0	-30.0	-29.5	-29.5	-32.9	-32.9	-34.3	-34.3	-26.5	-26.5	-26.2	-26.2	-26.6	-26.6	-29.3	-29.3	-31.9	-31.9	-33.5	-33.5
Coal Screen Analysis																										
+25mm	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
+20mm	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
+16mm	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
+10mm	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
+8mm	1.7	1.7	2.4	2.4	1.4	1.4	1.7	1.7	1.4	1.4	1.8	1.8	1.8	1.8	1.4	1.4	2.6	2.6	2.0	2.0	3.6	3.6	2.2	2.2	2.0	2.0
+6.3mm	5.2	5.2	7.2	7.2	4.8	4.8	5.8	5.8	4.8	4.8	5.7	5.7	6.2	6.2	4.7	4.7	7.3	7.3	6.2	6.2	10.2	10.2	6.8	6.8	6.0	6.0
+5mm	10.3	10.3	12.8	12.8	10.0	10.0	11.2	11.2	10.0	10.0	11.3	11.3	12.3	12.3	10.0	10.0	13.3	13.3	11.4	11.4	17.8	17.8	12.0	12.0	10.9	10.9
+4mm	16.2	16.2	18.2	18.2	15.5	15.5	16.9	16.9	15.6	15.6	17.4	17.4	18.9	18.9	15.9	15.9	19.2	19.2	17.5	17.5	26.0	26.0	18.0	18.0	16.7	16.7
+3.35mm	18.3	18.3	20.8	20.8	17.5	17.5	19.5	19.5	17.6	17.6	20.4	20.4	21.3	21.3	18.3	18.3	21.7	21.7	20.0	20.0	29.3	29.3	19.8	19.8	18.7	18.7
+2mm	35.3	35.3	36.9	36.9	34.1	34.1	36.8	36.8	35.3	35.3	37.4	37.4	39.6	39.6	34.6	34.6	39.3	39.3	37.5	37.5	50.4	50.4	37.3	37.3	36.4	36.4
+1mm	54.7	54.7	56.9	56.9	55.0	55.0	57.9	57.9	56.3	56.3	57.7	57.7	60.2	60.2	56.0	56.0	57.7	57.7	56.3	56.3	72.0	72.0	56.7	56.7	56.3	56.3
+0.5mm	71.7	71.7	72.7	72.7	71.7	71.7	73.4	73.4	72.5	72.5	73.7	73.7	75.1	75.1	72.5	72.5	73.5	73.5	73.3	73.3	86.9	86.9	72.9	72.9	72.4	72.4
-0.5mm	28.3	28.3	27.3	27.3	28.3	28.3	26.6	26.6	27.5	27.5	26.3	26.3	24.9	24.9	27.5	27.5	26.5	26.5	26.7	26.7	13.1	13.1	27.1	27.1	27.6	27.6
+.212mm	82.0	82.0	85.9	85.9	84.8	84.8	86.4	86.4	85.3	85.3	86.2	86.2	84.9	84.9	85.3	85.3	86.4	86.4	86.6	86.6	93.4	93.4	86.8	86.8	85.8	85.8
+0.150mm	85.9	85.9	89.2	89.2	88.5	88.5	89.7	89.7	88.9	88.9	89.3	89.3	87.9	87.9	88.9	88.9	89.7	89.7	89.7	89.7	95.0	95.0	89.5	89.5	89.1	89.1
+0.106mm	89.1	89.1	90.4	90.4	91.4	91.4	92.8	92.8	91.6	91.6	91.7	91.7	90.2	90.2	91.6	91.6	92.7	92.7	92.6	92.6	96.6	96.6	91.4	91.4	92.1	92.1
-0.106mm	10.9	10.9	9.6	9.6	8.6	8.6	7.2	7.2	8.4	8.4	83.0	83.0	9.8	9.8	8.4	8.4	7.3	7.3	7.4	7.4	3.4	3.4	8.6	8.6	7.9	7.9
Sm	2.0	2.0	2.1	2.1	1.9	1.9	2.1	2.1	2.0	2.0	2.1	2.1	2.2	2.2	2.0	2.0	2.2	2.2	2.1	2.1	2.7	2.7	2.1	2.1	2.0	2.0
Total -3.35	81.7	81.7	79.2	79.2	82.5	82.5	80.5	80.5	82.4	82.4	79.6	79.6	78.7	78.7	81.7	81.7	78.3	78.3	80.0	80.0	70.7	70.7	80.2	80.2	81.3	81.3
Coal Density																										
Routine Density (no diesel)	759	759	814	814	792	792	803	803	788	788	792	792	795	795	799	799	788	788	777	777	872	872	792	792	814	814
Oven (Wet)	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966	966
ASTM (Wet))	879	879	875	875	937	937	886	886	879	879	883	883	886	886	894	894	875	875	868	868	883	883	897	897	890	890
Oven (Dry)	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903	903
ASTM (Dry))	822	822	818	818	876	876	828	828	822	822	826	826	828	828	836	836	818	818	812	812	826	826	839	839	832	832

Coke Details																											
Coke Identity	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCB	PLCS	PLCS	PLCB	PLCB	PLCB	PLCB
Origin	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven	Pilot Oven
Coke Proximate Analysis																											
Ash	13.2	13.3	13.4	13.2	13.4	13.5	13.2	13.6	13.5	13.5	13.6	13.8	13.7	13.9	13.7	14.0	14.1	13.8	13.8	14.1	15.8	15.9	13.6	13.6	13.6	13.8
S	0.60	0.61	0.70	0.68	0.70	0.69	0.73	0.69	0.71	0.73	0.69	0.69	0.74	0.67	0.68	0.74	0.63	0.65	0.62	0.66	0.99	1.01	0.66	0.66	0.60	0.60
Vol	0.6	0.9	1.1	1.1	0.9	0.8	0.9	0.8	0.9	0.8	1.0	0.8	0.7	0.8	0.9	0.7	0.8	0.9	0.7	0.6	0.9	0.9	1.0	1.1	0.9	1.0
DFC	86.2	85.8	85.5	85.7	85.7	85.7	85.9	85.6	85.6	85.7	85.4	85.4	85.6	85.3	85.4	85.3	85.1	85.3	85.5	85.3	83.3	83.2	85.4	85.3	85.5	85.2
Coke Chemical Analysis																											
Na_2O	0.03	0.08	0.03	0.11	0.02	0.10	0.01	0.06	0.00	0.00	0.01	0.06	0.03	0.04	0.04	0.17	0.03	0.03	0.01	0.07	0.02	0.05	0.02	0.06	0.02	0.06
MgO	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.2	0.1	0.2	0.2	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Al_2O_3	3.3	3.2	3.6	3.4	3.1	3.3	3.1	3.2	3.4	3.4	3.2	3.2	3.4	3.1	3.1	3.0	3.4	2.5	2.3	2.1	3.3	3.6	3.4	3.5	3.5	3.5
SiO_2	7.3	7.8	7.2	7.2	6.9	7.2	7.1	7.1	7.3	7.2	7.3	7.2	7.5	7.4	7.4	7.5	7.6	7.3	5.6	5.1	9.8	9.5	7.7	7.6	7.4	7.4
P	0.038	0.034	0.036	0.037	0.037	0.039	0.034	0.032	0.036	0.040	0.033	0.039	0.036	0.033	0.038	0.038	0.039	0.034	0.028	0.027	0.006	0.008	0.036	0.036	0.038	0.034
K_2O	0.21	0.19	0.20	0.20	0.19	0.20	0.18	0.18	0.19	0.19	0.18	0.21	0.21	0.19	0.20	0.21	0.21	0.18	0.18	0.17	0.14	0.14	0.19	0.19	0.19	0.19
CaO	0.37	0.31	0.33	0.36	0.32	0.33	0.33	0.34	0.31	0.36	0.30	0.32	0.39	0.35	0.38	0.36	0.37	0.44	0.34	0.32	0.21	0.26	0.30	0.31	0.30	0.29
TiO_2	0.27	0.25	0.26	0.26	0.26	0.26	0.24	0.25	0.24	0.25	0.25	0.25	0.26	0.26	0.29	0.30	0.26	0.24	0.25	0.25	0.26	0.26	0.26	0.27	0.26	0.26
Mn	0.002	0.002	0.002	0.004	0.003	0.004	0.003	0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.001	0.001	0.002	0.019	0.002	0.002	0.005	0.004	0.002	0.002	0.003	0.003
Fe	0.7	0.7	0.9	1.0	0.9	0.9	0.7	0.7	0.5	0.6	0.6	0.8	0.8	0.6	0.8	0.9	0.9	0.8	0.7	0.9	0.8	1.0	0.6	0.7	0.8	0.9
Other Coke Properties																											
H_2O	7.6	6.8	8.4	3.2	8.2	3.4	8.6	3.8	8.6	3.6	8.0	3.2	9.2	5.0	9.6	5.4	7.6	5.2	9.2	8.0	6.4	8.2	6.8	9.2	3.2	5.6
Theoritical Yield %	73.3	73.3	73.3	73.3	72.5	72.5	73.1	73.1	73.1	73.1	72.2	72.2	72.5	72.5	72.3	72.3	72.0	72.0	71.9	71.9	64.4	64.4	71.8	71.8	72.4	72.4
Hot Strength																											
CRI	27.9	26.1	28.6	25.2	30.9	25.3	29.7	24.4	30.5	27.1	33.3	29.4	33.5	29.5	33.3	28.9	30.1	28.4	32.4	32.0	45.8	47.7	24.7	25.1	27.9	25.3
CSR	62.0	61.6	57.8	63.5	54.1	59.2	55.4	63.2	53.9	56.0	47.3	53.5	47.7	52.5	46.9	52.8	51.8	51.5	44.0	44.4	18.1	18.6	57.7	57.3	56.1	57.3
Corrected CRI (- 3UNITS)	24.9	23.1	25.6	22.2	27.9	22.3	26.7	21.4	27.5	24.1	30.3	26.4	30.5	26.5	30.3	25.9	27.1	25.4	29.4	29.0	42.8	44.7	21.7	22.1	24.9	22.3
Corrected CSR (+ 5 UNITS)	67.0	66.6	62.8	68.5	59.1	64.2	60.4	68.2	58.9	61.0	52.3	58.5	52.7	57.5	51.9	57.8	56.8	56.5	49.0	49.4	23.1	23.6	62.7	62.3	61.1	62.3
Micum & Irsid																											
M40	66.6	71.2	70.1	75.2	69.6	75.6	68.8	74.0	68.0	72.4	68.2	72.8	69.0	70.8	63.6	65.4	66.0	69.8	65.6	70.4	23.8	25.2	64.2	69.2	68.6	74.0
M30	87.6	86.0	86.1	87.8	83.6	87.2	86.6	87.2	83.6	85.2	87.0	87.0	86.8	86.4	86.0	83.2	87.4	85.8	86.2	86.4	53.6	63.8	87.2	85.4	83.0	86.4
M10	5.8	8.0	7.6	7.2	8.8	7.0	7.0	7.2	9.8	8.4	5.8	7.4	6.0	6.4	6.0	11.8	6.6	8.6	6.8	8.0	5.6	6.4	6.2	8.2	7.2	8.0
I40	41.8	44.4	48.1	52.6	47.4	52.4	45.2	51.6	47.6	50.2	42.0	47.2	44.0	45.0	38.2	43.2	45.2	46.4	41.6	44.8	4.0	4.2	38.4	42.0	46.8	50.6
I30	73.4	69.2	70.4	71.8	68.4	71.6	70.8	71.6	71.6	69.0	70.4	69.8	71.4	69.2	69.8	68.8	69.8	69.8	68.6	69.0	20.4	26.4	70.2	68.6	65.4	69.6
I20	83.4	76.0	75.7	78.0	75.4	78.0	78.2	78.0	79.6	75.6	80.4	77.2	86.1	77.6	81.0	74.4	78.4	76.4	78.0	76.2	62.6	61.6	79.6	76.2	75.6	77.0
I10	14.4	21.0	21.6	20.6	20.0	19.8	19.2	20.2	17.8	19.4	17.0	20.8	15.6	18.0	16.6	23.6	19.0	19.6	18.4	21.8	14.4	20.6	18.2	21.4	21.2	20.8
Coke Screen Analysis																											
%-125+100mm	0.9	2.4	4.3	6.9	6.3	4.7	3.3	2.9	4.7	4.3	0.9	3.9	1.3	2.6	1.6	3.9	1.0	4.5	0.5	2.3	0.3	1.7	1.1	6.0	2.6	0.5
%-100+ 80mm	9.9	10.8	12.8	13.3	13.4	18.4	12.6	11.6	18.2	24.0	13.5	14.3	8.1	9.1	7.6	12.4	10.9	9.0	8.2	11.2	1.3	5.9	12.2	13.9	15.0	16.3
%-80+ 63mm	37.0	37.4	36.2	38.9	28.8	31.3	31.4	35.3	31.7	28.2	36.7	37.5	33.9	32.7	30.8	33.0	31.3	39.6	32.9	33.0	14.3	17.2	24.6	29.4	22.6	32.9
%-63+ 40mm	40.9	35.0	32.8	28.5	36.0	32.4	38.1	37.7	38.5	29.3	37.2	31.2	43.8	43.2	46.5	38.3	44.7	35.6	42.8	37.7	40.9	38.8	46.6	34.0	47.1	39.8
%-40+ 35mm	5.2	4.3	3.2	3.5	3.6	3.8	4.0	3.7	2.0	3.2	3.8	3.9	5.0	4.4	4.6	4.3	4.8	3.6	5.7	5.3	15.8	11.5	6.8	4.8	2.5	1.9
%-35+ 30mm	1.3	1.6	2.0	1.3	2.3	2.6	2.1	1.6	0.9	2.0	2.0	1.8	2.3	2.3	2.8	1.8	1.4	1.2	2.7	2.5	9.2	6.5	2.2	2.1	3.0	2.0
%-30+ 20mm	1.7	3.1	2.6	2.2	3.0	2.3	2.4	1.8	1.1	2.4	1.9	2.4	2.3	1.7	2.4	1.9	2.0	1.8	2.4	2.9	11.5	10.1	2.1	3.5	3.0	2.2
%-20 +10mm	0.8	2.2	2.3	2.1	2.8	1.7	2.9	2.2	1.2	3.2	1.4	2.5	1.5	1.6	1.6	1.6	1.6	1.6	1.9	2.0	3.7	4.0	2.2	3.2	1.8	1.6
%-10mm	2.3	3.3	3.8	3.3	3.8	2.8	3.2	3.2	1.7	3.3	2.4	2.6	1.8	2.3	2.2	2.9	2.3	3.1	2.9	3.2	2.9	4.3	2.3	3.2	2.4	2.9
Sm	60.4	60.5	62.1	65.0	61.7	64.1	60.6	61.2	65.7	64.9	61.5	63.2	59.1	60.0	58.1	61.9	59.4	62.4	57.5	59.6	45.3	49.3	58.0	61.7	59.9	61.5
Tot +80mm	10.8	13.2	17.1	20.2	19.7	23.1	15.9	14.5	22.9	28.3	14.4	18.2	9.4	11.7	9.2	16.3	11.9	13.5	8.7	13.5	1.6	7.6	13.3	19.9	17.6	16.8
Tot+35mm	93.8	89.8	89.3	91.1	88.1	90.5	89.5	91.2	95.0	89.1	92.2	90.8	92.1	92.1	91.0	91.9	92.7	92.3	90.2	89.5	72.6	75.1	91.3	88.0	89.8	91.4
Tot+30mm	95.2	91.4	91.3	92.4	90.4	93.1	91.6	92.8	95.9	91.1	94.2	92.6	94.5	94.3	93.8	93.7	94.1	93.5	92.8	91.9	81.8	81.6	93.4	90.1	92.8	93.4
Tot -30mm	4.8	8.6	8.7	7.6	9.6	6.9	8.4	7.2	4.1	8.9	5.8	7.4	5.5	5.7	6.2	6.3	5.9	6.5	7.2	8.1	18.2	18.4	6.6	9.9	7.2	6.6
Depth Meter																											
Pp.(Inkrimp)	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter	meter
TE (Uitsetting)	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken	broken
Wall Pressure																											
kN	2.02	2.02	2.21	2.21	2.62	2.62	2.77	2.77	2.68	2.68	3.31	3.31	3.38	3.38	2.11	2.11	1.95	1.95	1.87	1.87	1.49	1.49	2.62	2.62	2.09	2.09
Kpa	2.60	2.60	2.85	2.85	3.38	3.38	3.57	3.57	3.45	3.45	4.27	4.27	4.36	4.36	2.72	2.72	2.51	2.51	2.41	2.41	1.92	1.92	3.38	3.38	2.69	2.69
mb	26.0	26.0	28.5	28.5	33.8	33.8	35.7	35.7	34.5	34.5	42.7	42.7	43.6	43.6	27.2	27.2	25.1	25.1	24.1	24.1	19.2	19.2	33.8	33.8	26.9	26.9
PSI	0.38	0.38	0.41	0.41	0.49	0.49	0.52	0.52	0.50	0.50	0.62	0.62	0.63	0.63	0.39	0.39	0.36	0.36	0.35	0.35	0.28	0.28	0.49	0.49	0.39	0.39
Coke End Temp																											
tfin.°C	1053	1053	1036	1036	1025	1025	1051	1051	1040	1040	1037	1037	1050	1050	1035	1035	1050	1050	1050	1050	1050	1050	1039	1039	1050	1050
Coke Rate																											
Plastic Zone (474°C) /Hours	13.40	13.40	14.50	14.50	15.15	15.15	15.75	15.75	14.75	14.75	15.25	15.25	13.65	13.65	13.05	13.05	14.90	14.90	14.40	14.40	14.25	14.25	15.20	15.20	13.40	13.40
Plastic Zone target to 474°C	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2	14.2
Coke Rate to 900°C/Hours	16.60	16.60	17.45	17.45	17.60	17.60	17.55	17.55	17.55	17.55	16.95	16.95	16.10	16.10	16.70	16.70	17.00	17.00	16.70	16.70	17.05	17.05	17.40	17.40	16.95	16.95
Coke Rate target to 900°C	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3
Total Coking Cycle / Hours	18.05	18.05	20.25	20.25	20.15	20.15	20.00	20.00	19.80	19.80	20.00	20.00	18.00	18.00	19.90	19.90	18.80	18.80	18.45	18.45	18.05	18.05	19.95	19.95	19.55	19.55
k (g/cmh)	32.0	32.0	30.4	30.4	30.2	30.2	30.3	30.3	30.3	30.3	31.3	31.3	33.0	33.0	31.8	31.8	31.2	31.2	31.8	31.8	31.2	31.2	2.5	2.5	2.6	2.6
P Force																											
Energie Kw/h																											
Oven Temperatures Settings																											
Start Temperature	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C	900°C
End Temperature	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C	1200°C

Appendix 3. Links to Sodium Tetraborate Octahydrate (MSDS)



MSDS for Sodium Tetraborate.PDF

i. http://www.esciencelabs.com/sites/default/files/msds_files/Sodium%20Borate%20Powder.pdf

ii. https://static1.squarespace.com/static/54f0b505e4b017cb0d7a6f26/t/556c79c4e4b07e55e36e21a4/1433172420751/Searles+Valley+Sodium+Tetraborate+Pentahydrate+%28V-BOR%29+SDS+1-20-2015.pdf

iii. https://www.mtixtl.com/msds/Sodium%20Borate%20Hydrate%201303-96-4%20101116.pdf