COMBUSTION OF CARBONACEOUS SHALE: A CASE STUDY OF AN INCIDENT AT AN IRON ORE MINE

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DECLARATION

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ABSTRACT

Spontaneous combustion incidents are known to occur at coal mining operations, associated with the presence of coal, coal-shales, and pyrite. A premature detonation incident occurred at an iron ore mine in 2020 in a black carbonaceous shale unit, known to be associated with the presence of pyrite. The unit was never chemically classified or modelled separately, and the risk for spontaneous combustion or the potential for ground reactivity was poorly defined. This report investigates the incident with the aim of initially classifying the composition of the carbonaceous shale unit, and secondly determining the propensity for spontaneous combustion to occur in the future. The classification of the carbonaceous shale unit was determined through X-ray fluorescence (XRF), carbon and sulphur speciation (CCS), proximate and ultimate analysis, and X-ray diffraction (XRD). Numerous studies have been done on determining a method to evaluate the risk of a spontaneous combustion, the Wits-Ehac Index classification is one such method that was used in this study. The Wits-Ehac Index results were correlated with the XRF and proximate and ultimate analysis through linear regression. A comparison of the XRD results versus the Wits-Ehac results was also done. The iron ore mine carbonaceous shale was classified as a ‘poor’ carbonaceous shale, falling on the limit of being classified as a ‘rock’, due to low carbon content, low calorific value, and high ash content. The unit is characterised by a low Fe presence, except when siderite is present, and a low but variable sulphur content. Where sulphur values are relatively higher, it may be ascribed to the presence of pyrite that increases the risk of reactive ground. The Wits-Ehac results were between medium and high propensity for spontaneous combustion, and the results of the study were used to determine a spatial risk map to define risk areas within the mine. The linear regression plots showed very poor correlations between the Wits-Ehac Index results and the XRF and proximate and ultimate results. The strongest correlation existed with Nitrogen. The most valuable relationship determined in this study is between the presence of relatively high sulphur (greater than 3%) that is related to the presence of pyrite which may increase the risk for spontaneous combustion and ground reactivity with nitrate-bearing explosive emulsion.

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LIST OF ABBREVIATIONS

°C	Degrees Celsius
A	Ash
AEISG	Australian Explosives Industry and Safety Group Inc
Al	Aluminium
ANFO	Ammonium nitrate and diesel fuel oil
ASTM	American Society for Testing and Material Standards
C	Carbon
CFD	Computational fluid dynamics
CO	Carbon Monoxide
CO_2	Carbon Dioxide
COP	Code of practice
CSS	Carbon and sulphur speciation
CV	Calorific value
DSC	Differential scanning calorimetry
DSO	Direct shipping ore
DTA	Differential thermal analysis
ECE-UN	Economic commission for Europe-United Nations
E_S	Elemental sulphur
FC	Fixed Carbon
FCC	Feng, Chakravorty and Cochrane liability index
Fe	Iron
FT	Flammability temperature
G_C	Graphitic carbon
H	Hydrogen
HEM	Hematite
HPI	High potential incident
ISO	International organization for standardisation

K	Potassium
Kcal/kg	kilocalories per kilogram
LECO	Laboratory Equipment Corporation
M	Moisture
m	meter
MJ/kg	Megajoules per kilogram
mm	Millimetre
MT	Million tonnes
N	Nitrogen
O	Oxygen
ORG_C	Organic carbon
P	Phosphorus
PA	Proximate analysis
PFC	Pulse flow calorimetry
ROM	Run of mine
S	Sulphur
SH	Shale
SH-C	Carbonaceous shale
Si	Silica
SOP	Standard operating procedure
T	Tons
TC	Total Carbon
TG_{spc}	Thermogravimetric analysis index
TGA	Thermogravimetric analysis
TS	Total Sulphur
U	Ultimate analysis
VM	Volatile matter
WEI	Wits Ehac-Index

WOP	Wet oxidation potential
XPT	Crossing point temperatures
XRD	X-Ray diffraction
XRF	X-Ray fluorescence

1 INTRODUCTION

1.1 Research background

This research report is based on a mine referred herein as "Iron Ore Mine A", which had an unplanned detonation incident in 2020. Iron Ore Mine A is situated in the northern part of South Africa, in the Northern Cape Province as illustrated in Figure 1.1. The mine was designed to process 9 million tonnes (MT) of Run of Mine (ROM) iron ore. It is predominantly a direct shipping ore (DSO) operation from which ore is mined from the different pits and hauled to the primary crusher or designated stockpiles. The ore is crushed, dry-screened and transported via railway line to Saldanha Bay for export to the mine's clients. The current life of mine is 12 years for the Iron Ore Mine A operation, and in 2020 it produced 11.3 MT of ore and mined 59.4 MT of waste material (Otto & Lindeque, 2021). The operation has a 3.6:1 stripping ratio, which means that for every 1-unit weight of ore produced, 3.6 units of waste had to be removed.

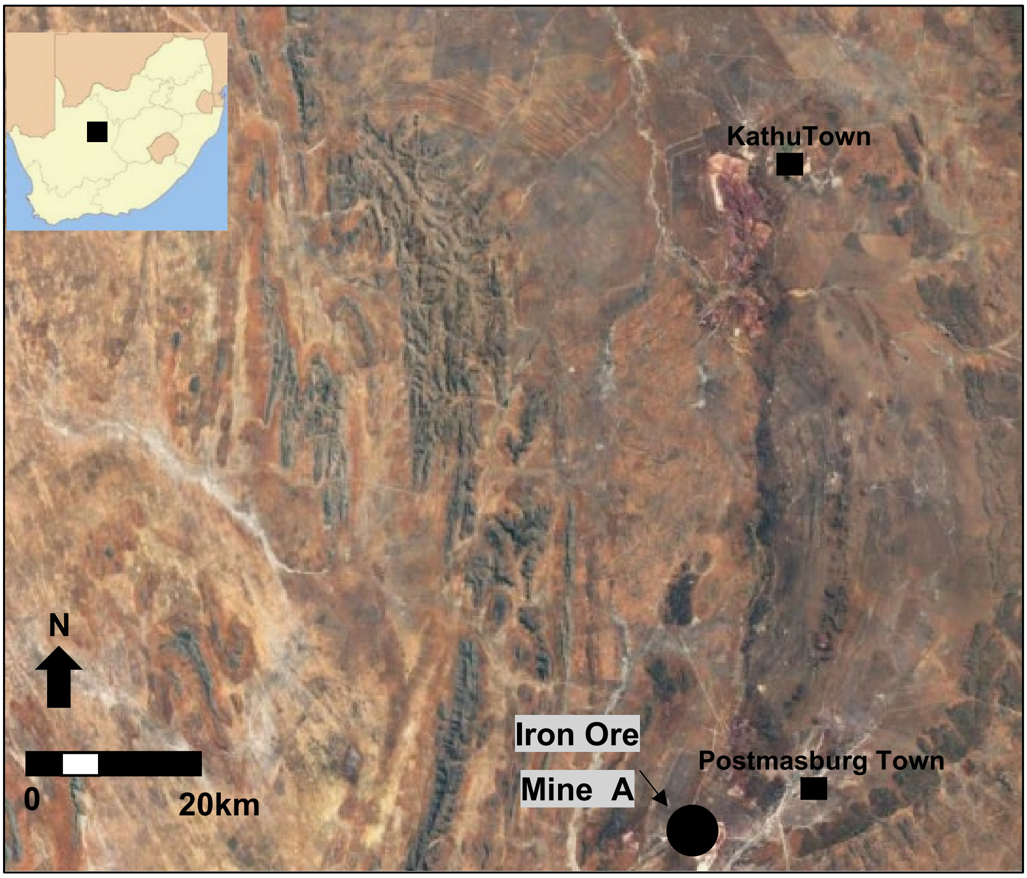

Figure 1.1: Iron Ore Mine A resource locality map.

An unplanned detonation incident occurred in April 2020 at Iron Ore Mine A, which was classified as a high potential incident (HPI) that could have caused a fatality or more than one fatality. The post-incident investigation confirmed the presence of carbonaceous shale near the detonation area in the mine. The carbonaceous shale had been known to be present in the mine, but the carbonaceous shale was neither separately modelled nor predicted to occur in the specific mining block (Scott & Gous, 2020). This was because carbonaceous shale was not identified as a major risk by the mine. The chemical properties of the carbonaceous shale were not studied extensively as the mine's drilling operations focused on intersecting the iron ore and were not concerned with the overlying waste domains. Apart from the lack of understanding of the chemical properties of the carbonaceous shale, the risk of spontaneous combustion and ground reactivity was also not defined in the mine procedures and risk assessments. The incident could have occurred due to self-heating of the carbonaceous shale lithology, leading to spontaneous combustion, or reactivity with the explosive emulsion.

1.1.1 Iron Ore Mine A mining method

The Iron Ore Mine A operation is a conventional truck and shovel open-pit mine, drilled and blasted in 10m benches as depicted in Figure 1.2. Ore material loaded from blasted benches is hauled to either the primary crusher or the correct ROM stockpile location. Waste is dumped on waste dumps or in-pit filling locations. Maintaining a high equipment efficiency on shovels aims to achieve bulk waste mining at a reduced mining cost. In addition, the ore mining is done selectively to ensure clean ore recovery (Otto & Lindeque, 2021).

Figure 1.2: Conventional truck and shovel open-pit mining schematic (Anglo American, 2021)

The mine operates with 75% owner-fleet and 25% contractor-fleet. The primary mining equipment deployed includes drill rigs drilling 251 millimetre (mm) diameter blast holes and hydraulic face shovels designated for either mining pre-strip waste or in-pit ore and waste. The primary hauling fleet is comprised of trucks with either 100 tonnes (T) or 190 T capacity (Otto & Lindeque, 2021).

The success of the mining operation relies on the interaction between the mining technical services and the mining execution activities as outlined in Figure 1.3.

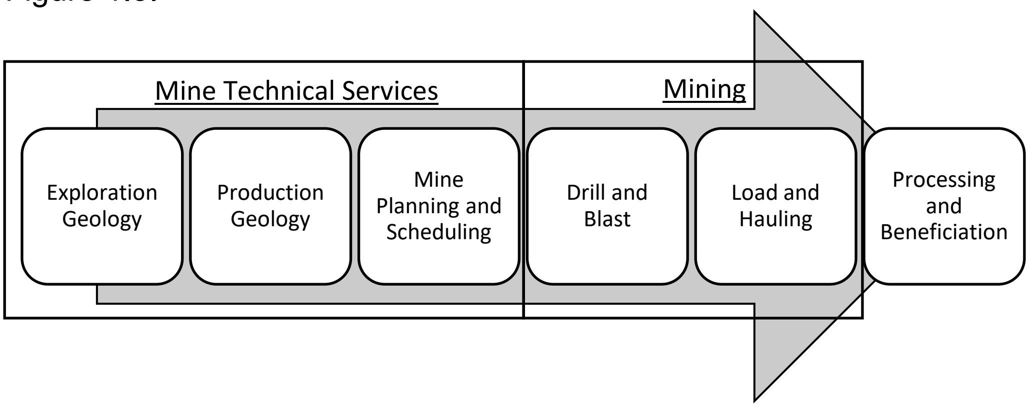

Figure 1.3: Mine value chain of the study area

The production geology section is involved in conducting in-fill drilling on a 15 meter (m) by 15 m grid to ascertain the properties of the iron ore mining blocks. The only waste lithologies included in the drilling are the hanging wall and footwall, with samples only taken 5m above and below the iron ore. Exploration geologists are responsible for drilling on a 50m-by-50m drill grid and building a resource model from the collected exploration data. The mine planning section determines a mining schedule based on set input parameters from the geology and mining teams. Compliance and execution of this schedule are the key performance indicators of the operation. Apart from the safety risk posed by potential spontaneous combustion incidents, the lack of understanding of the presence of the carbonaceous shale has a negative impact on the mine schedule compliance and productivity of the mining team.

1.2 Research motivation

The mining execution activities are drill and blast, thereafter, load and haul. The drill and blast team plans the holes drilled on all of the mining blocks, based on the material classification received from the production geology team. When the material classification given indicates the presence of material with carbonaceous content, such as the carbonaceous shale lithology present in Iron Ore Mine A, the Hothole and Reactive Ground Procedure is activated. A Hothole and Reactive Ground Procedure is a site-specific document that specifies the steps to mitigate the risk of drilling and blasting activities within ground conditions that might be conducive to spontaneous combustion or reactive ground. This minimises the risk of an uncontrolled detonation incident whether due to the rock chemistry or the temperature of the ground, or both.

In the Iron Ore Mine A procedure, a hot hole is defined as a hole with a temperature greater than 40 ^{o}C. This procedure is activated when the rock types present on the blasting block are classified as having the potential to contain sulphides or iron sulphides, in the case of Iron Ore Mine A, the sulphide is the mineral pyrite. This rock type containing pyrite might interact with the explosive product used, and this is the definition of ‘reactive ground’ on Iron Ore Mine A. The presence of carbonaceous shale implies that the hole temperatures need to be monitored by the blasting supervisor in charge, who is referred to as the Blaster. The monitoring of the hole temperatures in Iron Ore Mine A is done by measuring the temperatures, as well as limiting the standing time of the block between drilling and charging it with explosives. The holes will be charged specifically based on whether they measured above or below 40 °C.

The risk and incidents of spontaneous combustion in coal and coal-shales are commonly known and managed in the mining of coal deposits. Risk management relies on the establishment of a site-specific standard operating procedure (SOP). When dealing with the risk of spontaneous combustion or reactive ground, the following factors should be considered according to the Australian Explosives Industry and Safety Group Inc. (AEISG, 2017):

- Training of mine personnel;
- The time that explosives are left in the drilled holes;
- Monitoring temperatures in the drilled holes or on the surface; and
- The sequence in which the drilled holes are loaded with explosives.

Spontaneous combustion occurs due to the self-heating characteristic of coal and coal-shales (Onifade & Genc, 2018c). However, the risk of spontaneous combustion occurring in the iron ore environment is not well-known or widely documented.

The aim of this research is to define the carbonaceous shale occurrence in the Iron Ore Mine A and to highlight as well as quantify the risk of spontaneous combustion within the carbonaceous shale for drill and blast activities to be managed accordingly. Additionally, the research compares the results with that of the coal-shale risk available from the literature.

1.3 Problem statement

The geology team is responsible for the geological domaining and characterisation of the lithologies to an appropriate level to ensure the effective management of drill and blast activities through product selection and appropriate procedures that are fit-for-purpose (Scott & Gous, 2020). The presence of black carbonaceous shale will affect the drill and blast activities. At present, the carbonaceous shale at the Iron Ore Mine A is poorly understood in terms of its chemical characteristics and propensity for spontaneous combustion. Spontaneous combustion will result in lost working time which adversely impacts the mine performance, and the safety of the mine personnel (Phillips et al., 2011). These incidents occur commonly in underground coal mines as well as surface mines that are exposing underground material (Genc & Cook, 2015). The incidents also occur in sulphide mines where ground reactivity incidents were recorded as recent as April 2021 (Pieterse, 2021).

The incident in question in this study resulted in a delay of work in the drill and blast section where the equipment needed to be inspected and the SOP and risk assessments needed to be amended. The incident investigation itself took 14 weeks of personnel working on understanding the incident in addition to the normal workload of the mine operation.

1.4 Research objectives

This research focuses on the geochemical characterisation of the pyritic carbonaceous shale found at the Iron Ore Mine A with the following objectives:

- Identify the chemical composition of the carbonaceous shale;
- Determine the spontaneous combustion propensity of the iron ore samples based on the Wits-Ehac Index;
- Determine the correlation of the chemical properties with the Wits-Ehac Index; and
- Conduct a comparison of the results with the published results of the coal-shales from the Witbank coalfields.

These objectives aid in understanding the risk of spontaneous combustion that informs the drill and blast activities at the iron ore mine.

1.5 Structure of the report

This research report has five chapters. The first chapter is the introductory chapter that includes an overview of Iron Ore Mine A and the mining method, as well as the research motivation, problem statement and research objective. The second chapter is the literature review that outlines the spontaneous combustion and ground reactivity process and includes literature available on other coal and coal-shale investigations and occurrences, as well as a geological overview of the carbonaceous shale found in the Iron Ore Mine A. The third chapter describes the research method and chapter four discusses the results of the research, in two parts. The first part of chapter four characterises the carbonaceous shale unit as a whole and the second part is related to the spontaneous combustion results. The last chapter outlines the conclusion and recommendations following the investigation of the carbonaceous shale at Iron Ore Mine A.

2 LITERATURE REVIEW

2.1 Ground conditions with elevated temperature

The major risk of blasting in ground conditions that have elevated temperatures is the risk of unplanned premature detonation of blast holes that have the potential to cause loss of life (AEISG, 2017). This risk exists due to the potential of the ground to react with the explosives used on site (Orica Australia, n.d.; Pieterse, 2021).

Explosives used in drill and blast activities at Iron Ore Mine A at the time of the incident were a heavy ammonium nitrate and diesel fuel oil (ANFO) blend. ANFO refers to the explosive emulsion made up of ammonium nitrate and fuel oil. This mixture is soluble in water (Orica Australia, n.d.).

Ground conditions that have an elevated temperature may be caused by the inherent rock chemistry, such as the case with spontaneous combustion of coal seams, and when sulphides are present (AEISG, 2017). Other causes of elevated ground conditions are related to the geothermal gradient of the earth which is the rate of increase in temperature per unit depth into the earth due to heat flow from the core (Kanana & Matveyev, 1989). Solar radiation may also lead to elevated temperatures (AEISG, 2017).

Sulphide oxidation is a special case of elevated temperature ground conditions where the addition of nitrates causes the oxidation reaction that releases heat (AEISG, 2017), and may lead to the premature detonation of blast blocks (Orica Australia, n.d.). In this section of the report, the spontaneous combustion process of coal is described, as well as the ammonium nitrate and sulphide oxidation reaction process related to the reactive ground.

2.1.1 Spontaneous combustion

Spontaneous combustion is described by Kim & Chaiken (1990) as a function of the energy released and the rate at which it is released and transferred to the surrounding rock mass. The rate of combustion depends on the concentration of reactants (carbon and oxygen), the surface area and particle size, ambient/initial temperature, and the activation energy (Kim & Chaiken, 1990). During the oxidation reaction of coal or carbonaceous shale, CO_2, CO and heat are generated (Chaiken, 1977). With enough organic carbon as fuel, no external heat source may be required for spontaneous combustion to occur (Kim & Chaiken, 1990). In the case of carbonaceous shale, the presence of pyrite, and potentially water, increases the risk of spontaneous combustion, due to the exothermic oxidation reaction of pyrite (Kim & Chaiken, 1990).

This exothermic oxidation reaction of carbon-rich material (coal and related shales) with pyrite is a three-stage self-heating process that may lead to spontaneous combustion (Beamish & Theiler, 2016; Kim & Chaiken, 1990). This process can either occur on waste dumps or in in-situ material into which blasting drillholes have been drilled. Spontaneous combustion can also occur in places where there is airflow due to natural or unnatural fracturing (Potgieter, 2018; Restuccia et al., 2017). The result of this reaction leads to elevated ground temperatures of varying degrees of intensity (Beamish & Theiler, 2016).

Spontaneous combustion occurs in three reaction stages as explained in the following steps:

Stage I: Iron-sulphide undergoes an exothermic reaction with oxygen and water which results in gas and liquid by-products. The extent to which the host material heats up is dependent on the balance between the environment’s natural heat dissipation conditions and the exothermic reaction, as well as the restraining effect of the evaporation of moisture (Beamish & Theiler, 2016; Rosenblum et al., 2001).

Stage II: Initially, Stage II is reliant on the presence of elemental sulphur that oxidises further via an exothermic reaction. When temperatures reach around 140 ^{o}C, organic carbon starts to oxidise and catalyses the exothermic reaction. Gaseous products are produced via this reaction (Beamish & Theiler, 2016; Rosenblum et al., 2001).

Stage III: The third stage is rarely reached without a catalyst being present and represents temperatures of material greater than 500°C (Rosenblum et al., 2001).

2.1.2 Reactive ground

In the above section, Stage I describes the natural oxidation reaction of sulphides (pyrite in the case of Iron Ore Mine A). The released iron compounds and acid may react with nitrates present in the explosives used in the drilled mining block. This reaction is an auto-catalytic exothermic reaction, which means that the reaction generates its own catalysts as the reaction continues (Figure 2.1).

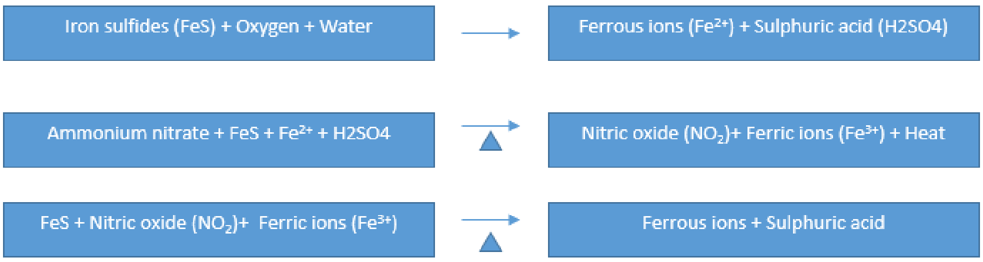

Figure 2.1: The sequence of auto-catalytic reaction process of iron sulphides and nitrates used in explosives (Orica Australia, n.d)

The reaction starts slowly, and gradually builds up to the intermediate stage where temperature spikes and the reddish-brown fumes of nitrogen dioxide are released. The reaction continues provided there is enough fuel (nitrates and more pyrite). The ignition stage is reached once the accumulation of catalysts reaches a critical level as illustrated in Figure 2.2 (Priyananda et al., 2015).

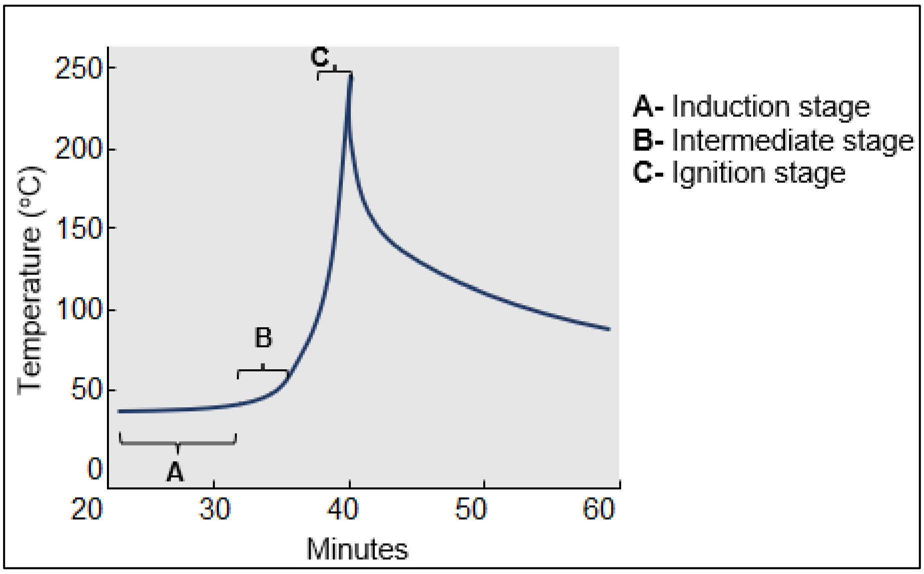

Figure 2.2: The temperature and time relationship of the reaction between iron sulfide (pyrite) and ammonium nitrate bearing explosives in an adiabatic calorimeter (adapted from Priyananda et al., 2015)

The progress of the induction stage is slow and usually occurs in areas of the mine that are exposed to oxygen such as the exposed high wall or free-face of the drill block. The presence of fractures, such as joints or faults, or fractures caused by previous drilling and blasting activities can increase the surface area for oxidation to take place (Potgieter, 2018.). The intermediate stage temperature spike of over 100 °C can easily occur in a matter of minutes. The ignition stage is the violent decomposition of the ammonium nitrate leading to the premature detonation of blast blocks (Orica Australia, n.d.; Priyananda et al., 2015). The presence of pyrite and an increase in the diameter of the drilled blast hole have been shown to lower the temperature at which the ammonium nitrate will detonate (Gunawan & Zhang, 2009).

The significance of having carbon present, as well as pyrite, in the host formation that is being drilled and blasted, is that it acts as a catalyst. The organic carbon content oxidises too (Stage II described in Section 2.1.1) and creates additional fuel in the form of heat to further catalyse the reaction between the iron sulphides and the ammonium nitrate. This leads to a snowball effect that culminates in the ignition stage.

It is important to understand and characterise the waste lithologies present at a mining operation because of the detrimental effect that the presence of iron sulphides and carbon has on safety, health and the environment. In this research report, the carbonaceous shale with known pyrite presence will be characterised for the first time at Iron Ore Mine A.

2.2 Coal and coal-shales

Coal is a sedimentary, heterogeneous material made up of organic and inorganic constituents, and is considered 'coal' when greater than 50% organic matter occurs (ISO 11760). Coal is classified based on grade (percentage of organic mineral matter), and rank (lignite, bituminous, anthracite). The higher the organic matter content, the higher grade a coal deposit is (Falcon & Ham, 1988). Rank is defined by volatile matter percentage, carbon content and vitrinite reflectance. Typically, the lower the rank of coal, the more prone it is to oxidation (Falcon & Ham, 1988). The rank of the coal depends on the maceral composition (Falcon & Snyman, 1986). Macerals are formed from the original plant material present during the early formation of the peat that accumulated through the process of biochemical degradation. Three types of macerals may be present, namely, liptinite, inertinite and vitrinite (Falcon & Snyman, 1986).

Coal-shale, or carbonaceous shale, is a sedimentary rock with less than 50% of organic material (Schopf, 1966), and according to the ECE-UN coal classification (1998), a rock with 80% - 50% ash content (Table 2.1). These shales are believed to be formed in depositional environments such as nondeltaic coastal salt-marsh environments from peats preserved in the stratigraphy. The non-carbonaceous and inorganic mineral matter content in carbonaceous shale is non-combustible and is represented by ash and sulphur forming compounds (Jones & Cameron, 1988). The organic constituents are lower in carbonaceous shales than in coal itself and are represented by microscopic macerals.

Table 2.1: Grade classification chart of coal and coal-shale, adapted and simplified from ECE-UN (1998)

	GRADE					
	SHALE		COAL			
	Rock	Carbonaceous Rock	Very low grade	Low grade	Medium grade	High grade
ASH%	*100-80*	*80-50*	*50-30*	*30-20*	*20-10*	*10-0*

The nomenclature surrounding coal and coal-related shales is enigmatic and varies between classification systems (Alpern & de Sousa, 2002). According to Alpern and de Sousa (2002), a further distinction might be made in carbonaceous shale when looking at whether the shale will produce more energy than it will consume. This limit of ‘autothermicity’ corresponds to a lower cut-off of 75% ash content as well as a calorific value of 1500 kcal/kg or 6.3 MJ/kg (Alpern & de Sousa, 2002). Therefore, a carbonaceous shale that has ash content above 75% and a poor calorific value may be considered a ‘poor’ coal-shale since there will be a negative energy balance when burned (Alpern & de Sousa, 2002). The SANS 11760 describes the classification of coal, based on coal rank. Where the ash content is between 30 and 50%, the coal is considered to be ‘High Ash’ with a low rank (ISO 11760). When more than 20% of a coal-sample is made up of inorganic (mineral) matter, and if there is less than 40% carbonaceous matter with no discernible coal bands, the lithological description of the unit is a carbonaceous shale (Wagner, 2021). A sediment with more than 20% mineral matter and less than 10% carbonaceous matter is referred to as a shale (Wagner, 2021).

The carbonaceous shale of Iron Ore Mine A is expected to fall within the carbonaceous rock grade classification. Characterising coal can be done via physical and chemical analyses (Mayes & Myers, 2014). The physical analyses include petrographic analyses to identify macerals, minerals, the degree of reflectance and macrolithotypes, and proximate analyses measure the moisture, volatile matter, ash and fixed carbon content (Mayes & Myers, 2014). The chemical analysis includes ultimate analysis (Mayes & Myers, 2014), which measures the chemical composition of the sample. In this research report, proximate and ultimate analyses were conducted on carbonaceous shale along with other chemical and mineralogy determination methods which are explained in more detail in the research methods section.

2.2.1 Witbank coalfield age and deposition

The Witbank Coalfield is a major coal mining area in South Africa, that has been the subject of numerous academic studies in the geological and mining engineering field, including case studies of reactive ground and spontaneous combustion, comparable to this research report. The coal in the Witbank Coalfields is mostly located in the stratigraphic Ecca group of the Karoo Supergroup. Five known coal seams are documented in the Witbank Coalfields, present within a clastic sedimentary rock sequence which is present above the Dwyka Formation tillite or pre-Karoo basement (Cairncross & Cadle, 1987). The deposition of the coal seams varies in their structural and sedimentary setting, their age, their genesis and the subsequent deformational or burial events. The coal seams are from the Permian to mid-Triassic age group and vary in rank from bituminous to anthracite (Falcon, 1986). The variation is seen with respect to the organic and inorganic matter, and the degree of maturity and rank, present in the different coal deposits and coal seams are related to differences in their genesis.

2.3 Iron ore carbonaceous and pyritic shales

Iron ore is a bulk commodity which implies that waste mining is a considerable contributor to the day-to-day operation of an iron ore mine (Twardowska & Szczepanska, 2004). This is also the case with Iron Ore Mine A, where the amount of waste removed in 2020 was five times the amount of iron ore produced (Otto & Lindeque, 2021). Waste dump management is important and therefore the characterisation of the overburden is necessary, particularly if this waste material could have the risk of spontaneous combustion (Restuccia et al., 2017). Pyritic carbonaceous shales are known to have this risk in specifically coal mines and occurrences of these shales in iron ore mines have been documented (Onifade & Genc, 2018c).

Pyritic carbonaceous shales are known to occur in the Mount Whaleback mining area in Australia (Han et al., 2005). The specific shale studied and characterised by Han et al. (2005) was the Mount McRae Shale which was split into three distinct units, namely the upper, nodule and lower zones. The three units were analysed in detail for their geochemistry, including pH, total sulphur, and sulphur forms (sulphate and sulphide), total carbon content and carbon forms (carbonate and graphitic carbon), soluble iron, and forms as well as metal element assay (Han et al., 2005). The results in terms of the sulphur and carbonate content for the Mount Whaleback Mount McRae Shale, which can be compared to the results of this study, are summarised below.

The Mount McRae shale varied with sulphur content between zones and the zones were classified based on the degree of weathering and pyrite habit and content. The shale had sulphur content ranging from <0.1% to >30% sulphur (Han et al., 2005). The highest zone (Nodule zone) had sulphur content between 2% and 30%, mostly above 5%, followed by the lower member zone with 2% to 5%, mostly around 2%, the upper member shale up to 4%, mostly around less than 1%, and the inert zone had less than 0.1% sulphur Han et al., 2005). Sulphide was the common sulphur-bearing substance for the samples between zero and 35% sulphur, as opposed to sulphate (Han, et al., 2005). The carbon content of the shales on average was around 3%, ranging between 0.5% and 6% with the bulk of the sample's carbon being the organic carbon form with negligible content of graphitic and carbonate carbon (Han, et al., 2005).

2.4 Gamagara Formation

The Iron Ore Mine A is located on the southern extent of the Maremane Dome in the Northern Cape province of South Africa, within the Palaeoproterozoic Transvaal Supergroup (Beukes, 1980). The local stratigraphic sequence is made up of basal Asbesheuwels dolomites, followed by Wolhaarkop Formation chert breccia (Beukes, 1980). The banded iron formations of the Asbesheuwel Subgroup follow next in the stratigraphy and host the hematite iron ore mineralisation. The Gamagara Formation of the Olifiantshoek Supergroup unconformably overlays the iron ore, followed by the Dwyka tillite unit and finally the sedimentary Kalahari sequence (Beukes, 1980; Alchin & Botha, 2006). The Gamagara Formation in which the carbonaceous shale of Iron Ore Mine A occurs is much older than the Witbank coalfield's Ecca group (Beukes,1980; Alchin & Botha, 2006; Cairncross & Cadle, 1987).

The Gamagara Formation is made up of different sedimentary members which consist of shale, quartzite and conglomerate interlayered and interbedded with varying thicknesses and varying grain sizes (Van Schalkwyk & Beukes, 1986). The black carbonaceous shale discussed in this research report is part of the Doornfontein Conglomerate Member, which forms the base of the Gamagara Formation (Van Schalkwyk & Beukes, 1986). A detailed stratigraphic, petrographic and geochemical study of the Gamagara Formation was done by Cousins (2016). The black carbonaceous shale was described as displaying lenticular laminae of quartz, chert, pyrophyllite and diaspore between zones of one to two centimetres of pyrite within a black carbonaceous clay matrix.

2.5 Prediction methods for spontaneous combustion

The prediction of spontaneous combustion is an important effort to create mitigating controls to prevent incidents from occurring in operating mines, stockpiles and abandoned underground workings. There is no universal standard method or model of testing for spontaneous combustion within the mining environment. This is mainly due to the variability associated with coal deposits across the world, and the limitations and assumptions made when developing a specific model (Onifade & Genc, 2020). Spontaneous combustion can also occur due to ground reactivity in sulphide-rich deposits (AEISG, 2017). The following section provides an overview of some of the existing methods recorded in the literature, followed by a description of the method currently accepted and used by many researchers of coal and carbonaceous-shale occurrences in South Africa, namely the Wits-Ehac Index.

2.5.1 Mathematical, experimental and statistical methods of spontaneous combustion prediction

Many methods exist to assess the propensity of coal to heat and spontaneous combustion and to analyse the factors influencing this event. These methods can be grouped into mathematical models, experimental models and statistical methods. Work done by Onifade & Genc (2020) provides a summary of global methods based on this grouping. The prediction techniques in their studies included methods in the coal industry whether it be stockpiles, underground workings or open-pit mines. The prediction techniques for spontaneous combustion from the studies can be listed as follows:

- crossing point temperatures (XPT),
- average heating rate,
- the Feng, Chakravorty and Cochrane (FCC) liability index,
- differential scanning calorimetry (DSC),
- differential thermal analysis (DTA),
- R70,
- flammability temperature (FT),
- wet oxidation potential (WOP),
- thermogravimetric analysis (TGA),
- Wits-Ehac tests,
- Wits-CT tests,
- the slope of the time-temperature curve on the XPT.

The prediction methods covered in this section will only include those based on experiments for open-pit operations since the incident that occurred at Iron Ore Mine A was not due to underground excavations or at the location of stockpiles or on waste dumps.

2.5.1.1 Mathematical models

Nelson & Chen (2007) used a single Arrhenius expression to calculate the rate of oxygen consumption in the low-temperature oxidation reaction of coal, and also described the Frank-Kamenetskii self-heating analysis. The effects of the particle size on the propensity for spontaneous combustion were studied by Arisoy & Akgün (1994), they defined an empirical exponent for the particle diameter that indicated the oxidation regime for a change from chemical to pore diffusion at successively higher temperatures. Bhat & Agarwal (1996) measured the effect of moisture interaction with coal. Firstly, they found that the moisture absorbs the oxygen from the oxidation reaction that prevents the heating of the coal. Secondly, the study found an opposing reaction in which the moisture caused condensation that led to the release of vapour which increased the rate of oxidation in dry parts of the sample that led to a heating effect of the coal (Bhat & Agarwal, 1996). The study proposed the first mathematical equation that correlated the potential of spontaneous combustion with moisture interaction. The work of Rosema et al. (2001) was used to evaluate the spontaneous combustion risk in the Chinese Rujigou Coal Basin. The numerical simulation model which was developed examined the oxidation reaction within a coal matrix (Rosema et al. 2001). Equations in this model illustrate the exchange of heat, radiation, oxygen and the differential equations of oxidation, heat and oxygen flow (Rosema et al., 2001). The results of this study showed a low risk of spontaneous combustion in the Rujigou basin and that thermal infrared scanning systems may be used to identify areas where self-heating is taking place (Rosema et al., 2001).

Arisoy & Beamish (2015) used data on the kinetics of coal oxidation reaction from adiabatic oven testing to evaluate the spontaneous combustion liability of coal. The influence of different starting temperatures on the rate of reaction in the initial stages was studied and a new rate model equation to determine the changes in the kinetic behaviour of low-temperature regions was established (Arisoy & Beamish, 2015). The study of Chen et al. (2015) provides an in-depth understanding of low-temperature oxidation in a blended mixture of coal with the use of a thermogravimetric simulation thermal analyser. It was found that the interaction of the oxidation reaction at low temperatures could be due to low moisture content and the presence of potential minerals which increase the inertness of the blended coal (Chen et al., 2015).

Yuan & Smith (2008) investigated mined out areas of a longwall mine with a computational fluid dynamic (CFD). The study modelled the effects of coal properties on the propensity for spontaneous combustion (Yuan & Smith, 2008). The CFD model used the porosity and permeability of the coal as inputs to model the low-temperature oxidation of coal (Yuan & Smith, 2008). This was used to indicate the risk of spontaneous combustion (Yuan & Smith, 2008). The model indicated that mine ventilation and the coal properties can affect the risk for spontaneous combustion (Yuan & Smith, 2008). The Frank-Kamenetskii theory explains the spontaneous combustion of reactants and provides an understanding of the opposing reaction of heat dissipating and being generated during oxidation (Yuan & Smith, 2008). The kinetic parameters are A that goes with the heat reaction, it is a pre-exponential factor, and E is the self-heating activation energy (Yuan & Smith, 2008). These parameters are used as inputs into a complex model for spontaneous combustion (Yuan & Smith, 2008). However, the use of the Frank-Kamenetskii theory does not consider some proven significant factors in the oxidation of coal at low temperatures (Onifade & Genc, 2020). According to Nelson & Chen (2007), some of the specific factors that also need to be taken into account are coal-pore structure, the moisture and oxygen content in the coal as well as surrounding air, the evolution and transport of heat by convection, the oxygen and moisture transport via convection and diffusion, the particle size as well as the exposed surface area of the coal, amongst others.

2.5.1.2 Experimental methods

Several studies at varying sites across the world have attempted to use experimental methods to determine the risk of spontaneous combustion. These investigations aim to classify the coal seams according to the risk for spontaneous combustion (Onifade & Genc, 2020).

Gouws et al. (1991) designed an adiabatic calorimeter to replace the ignition temperature apparatus and to simplify the test procedure. The designed method was correlated with the results from XPT and DTA tests (Gouws et al., 1991). Panigrahi et al. (1997) evaluated the Russian U-index by comparing the relationship between the Russian U-Index and the coals carbon, hydrogen, nitrogen and sulphur analysis, with the relationship that the XPT had with the same elemental analysis of the coal. The result was that the Russian U-index displayed a similar relationship to that of the XPT investigation (Panigrahi et al.,1997). The experiment was done on samples from the Indian Jharia Coalfield (Panigrahi et al.,1997).

Uludag (2007) used a small-scale experimental setup done at the University of the Witwatersrand in South Africa called the Wits-Ehac Index which is described in more detail in this report in section 2.5.2. The test was conducted by comparing the coal properties with the determined DTA and XPT method, with the results of the Wits-Ehac Index (Uludag, 2007). The study showed that the moisture content of the samples increased the spontaneous combustion risk, while the volatile matter content accelerated the self-heating reaction when the moisture content decreased (Uludag, 2007). An increase in carbon content was found to lead to higher risk classification (Uludag, 2007). The opposite was true for the ash content, the higher the ash content, the lower the risk of spontaneous combustion (Uludag, 2007). The coal samples were from the Witbank coalfields.

Tognotti et al. (1988) conducted another simple experimental technique on South African coal samples to determine the ignition temperature of cylindrical coal beds. To interpret and extrapolate the results, thermal ignition models were applied with satisfactory results (Tognotti et al.,1988). Sahu et al. (2004, 2005) studied the spontaneous combustion liability using DSC, XPT and DTA with samples from the Indian coalfields. The results showed that an increase in temperature increases the spontaneous combustion risk (Sahu et al. 2004) which varies between coal seams (Sahu et al., 2005) and that the DSC method provides a more reliable temperature than the XPT.

Taraba & Pavalek (2014) used pulse flow calorimetry (PFC) to determine the risk for spontaneous combustion based on the value of oxidation heat and ranked accordingly. The difference in initial heat measurement and the oxidation heat using an adiabatic test were observed as well, and the results were similar to that of the PFC (Taraba & Pavalek, 2014). The samples used were from various countries across the world (Taraba & Pavalek, 2014). Gürdal et al. (2015) analysed the composition of Can Basin (Turkey) coal and the gas emitted when the coal was burnt, as well as investigated the mineralogical content of the samples, to determine the spontaneous combustion liability. In addition to the rank of the coal and the moisture content, pyrite was determined as a significant role player with the increase in propensity for spontaneous combustion (Gürdal et al., 2015).

Shales with organic carbon content are inherently porous which increases the potential for self-heating and reactivity of the unit (Restuccia et al., 2017), since the pores act as conduits for reactivity to take place and Restuccia et al., (2017) also found that the potential for spontaneous combustion is increased with a decrease in particle size (Restuccia et al., 2017). The experiment of Restuccia et. al. (2017) was based on the Frank-Kamenskii theory and was able to gather that with the decreased grain size, thinner shale beds had the potential to spontaneously combust, more so than their coarser grain size counterparts. The importance of ambient temperature was also highlighted in their experiment.

2.5.1.3 Statistical methods

Coal and coal-shales can be characterised in the laboratory by proximate, elemental and petrographic analyses (called intrinsic properties of coal, (Chakravorty & Kolada, 1988)). Numerous studies use these analyses and the results of spontaneous combustion liability indices to predict spontaneous combustion using statistical analyses (Onifade & Genc, 2020).

Through statistical analysis, Nimaje & Tripathy (2016) found that the results of the ultimate analyses of in situ coal samples showed a significant correlation to the Olpinski liability index when compared to other liability indices that use XPT, DTA, FT and WOP. Statistical analysis between the results of XPT and proximate analysis indicated that the mixture surface regression model produces superior results with respect to the residual values, compared to that of the polynomial and simple/multiple regression models (Nimaje & Tripathy, 2016). The model was deemed suitable for use in determining the propensity for spontaneous combustion (Nimaje & Tripathy, 2016). Kaymakçi & Didari (2000) established relationships between spontaneous combustion liability and coal properties through results of linear and multiple regression analysis. Ash content, volatile matter, total carbon, hydrogen, liptinite, and inertinite are parameters influencing spontaneous combustion as determined from the linear analysis (Kaymakçi & Didari, 2000). From the multiple regression analysis, the contents of volatile matter, carbon, hydrogen, nitrogen, oxygen, sulphur and inertinite were determined to affect the spontaneous combustion liability of coal (Kaymakçi & Didari, 2000). The statistical study of Singh & Demirbilek (1987) indicated that various intrinsic properties of coal affect the risk of spontaneous combustion. Their study involved statistical analysis that produced a set of practical models, ordered according to coal rank, to determine the spontaneous combustion risk (Singh & Demirbilek,1987). A study by Onifade & Genc (2018b) used statistical analysis of the results of the Wits Ehac and Wits-CT indices, and coal intrinsic factors. The results of these studies are covered in Section 2.5.2.

2.5.1.4 Thermogravimetric analysis

Thermogravimetric analysis (TGA) is an additional method to determine the propensity for spontaneous combustion in coal mines. This method has not been utilised a lot, it is an alternative that has the advantage of providing results with a much shorter turnaround time than other experimental methods (Onifade et al., 2020; Mthabela, 2016). Classification of samples according to the liability of spontaneous combustion is based on the TGA Index (TG_{spc}). The index classification is based on the change/rate of the derivative weight measured during oxidation of the samples as described by Equation 2.1:

$$TG_{spc} = \Delta\text{weight loss rate}/\Delta\text{Temperature} \qquad (2.1)$$

In a TGA study by Onifade et al. (2020), samples of coal and coal-shale from the Witbank coalfields were heated at different rates between the same starting and ending temperature ranges. Two variables existed in this experiment, namely; the derivative of weight versus time, and the slope of the derivative plot of the curve at low temperatures (Onifade et al., 2020). This data creates a profile of heating rates versus slope for each sample which forms the basis of the prediction of spontaneous combustion. The results of the Onifade et al. (2020) study included analyses using the Wits-Ehac method for comparison of results determined by TGA, as well as presented proximate, ultimate and petrographic analysis for interpretation of the spontaneous combustion prediction methods (TGA and Wits-Ehac). A summary of the findings by Onifade et al. (2020) are as follows:

- Moisture content as a property alone could not be related to higher spontaneous combustion risk;
- Variable ash content might be responsible for variable liability of spontaneous combustion, and in this experiment, samples with the highest ash content had the lowest Wits-Ehac Index, and sample samples with lower ash content had the higher Wits-Ehac Index;
- The presence of pyrite might be related to a higher Wits-Ehac Index;
- The presence of inertinite indicates a lower risk of spontaneous combustion
- Lower heating rates produce lower TG_{spc} results;
- Results of the liability for spontaneous combustion (TG_{spc}) vary between different coal seams; and
- Overall, the Wits-Ehac test agreed with the results of TGA, more so for the coal-shales than the coal samples.

The study suggested that more work should be considered to establish TGA as the preferred method for analysis of the risk for spontaneous combustion in coal and coal-shales (Onifade et al., 2020). The Wits-Ehac method used in this study has been used in numerous studies measuring the risk of spontaneous combustion (Gouws et al., 1991; Onifade & Genc, 2018a, 2018b, 2018c, 2020; Onifade et al., 2019, 2020; Uludag, 2007) in the South African coal environment. Detail on this method is discussed in the next section of the report.

2.5.2 Wits-Ehac Index

The spontaneous combustion process described in Section 2.1 alludes to the oxidation process that coal and coal-shales undergo. This self-heating property, and the temperature at which a sample might spontaneously combust, is investigated through laboratory techniques that analyse XPT and DTA.

The XPT test is done by constantly heating the coal sample and an inert sample in an oil-bath. The point at which the coal-shale sample equals the inert sample temperature is considered the XPT (Gouws & Wade, 1989a and, 1989b). The DTA test measures the temperature difference between the coal sample and the inert sample and plots the temperature against the inert sample temperature (Wade et al., 1987). The slope of the line has three stages. In Stage I the coal sample is cooler than the inert sample due to the cooling via evaporation. Stage II is reached once all the moisture is evaporated and the coal sample is heating up to reach the bath-medium temperature, as well as self-heating. Stage III is the volatile exothermic reaction surpassing the bath-medium temperature (Gouws & Wade, 1989a, 1989b; Wade et al.,1987). The point on the graph as illustrated in Figure 2.3b, at which the zero line is crossed is considered the XPT (Gouws & Wade, 1989a and, 1989b).

Measuring the propensity for coal and coal-shales to spontaneously combust is done at the School of Mining Engineering, University of the Witwatersrand via a small-scale experimental test, namely the Wits-Ehac test. The apparatus setup as depicted in Figure 2.3a includes an oil bath, six coal and inert material cell assemblies, an oil circular, a heater, a flowmeter that monitors airflow, a compressor for air supply and a microcomputer that records temperature every 20 seconds for the duration of a test, which spans four hours on average (Onifade & Genc, 2018c). The test uses a combination of the XPT and the DTA (Figure 2.3b) (Gouws & Wade, 1989a, 1989b).

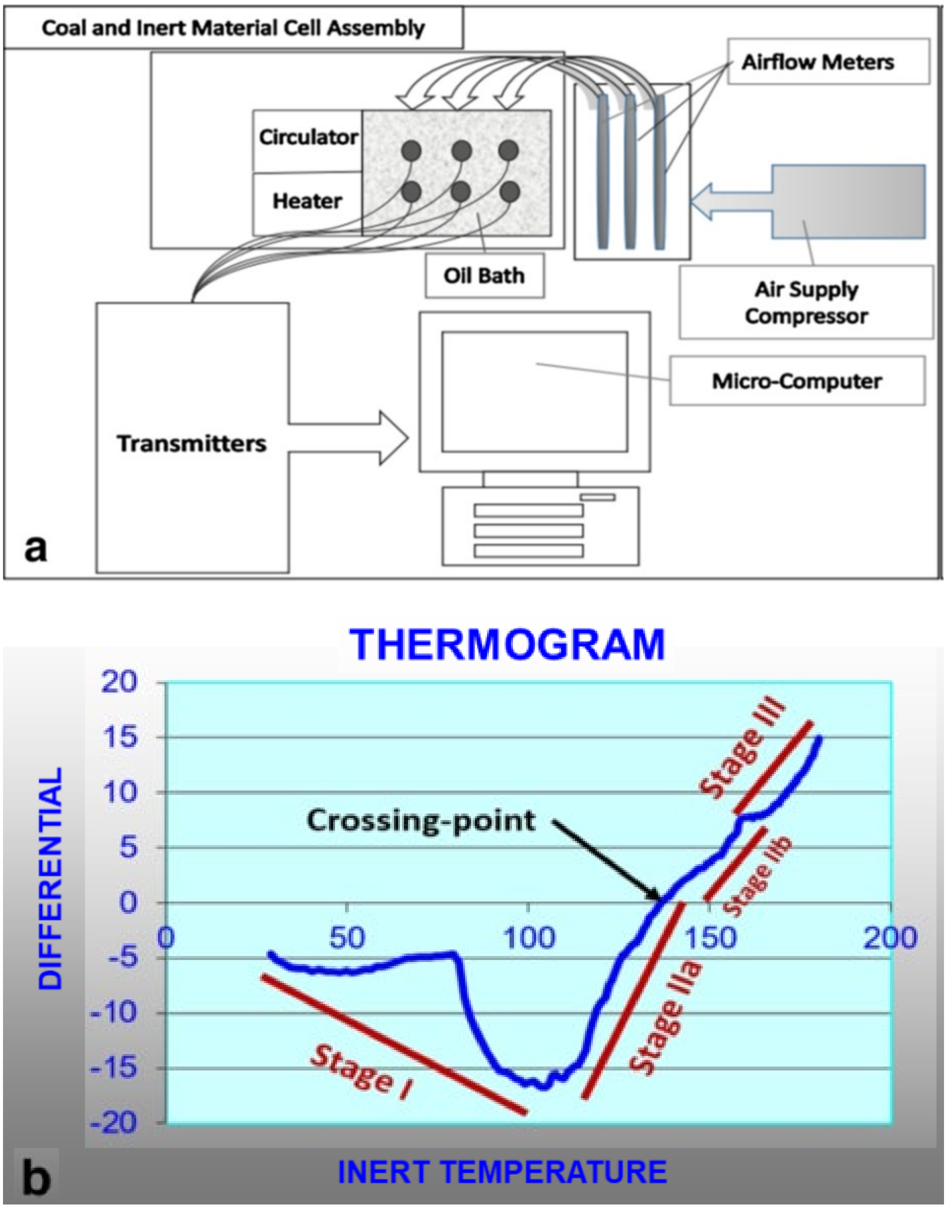

Figure 2.3: Wits-Ehac test setup (a) and typical differential analysis thermogram (b) (Wade et al., 1987)

The Wits-Ehac Index is calculated by the following equation (Equation 2.2):

*WITS-Ehac Index = (Stage II Slope / Crossing-point Temperature) * 500* (2.2)

The index classification in terms of spontaneous combustion liability is shown in Table 2.2.

Table 2. 2 Spontaneous combustion liability classification based on the Wits-Ehac Index (Gouws & Wade, 1989a; 1989b)

WITS-Ehac Index	Spontaneous Combustion Liability
0-3	Low
3-5	Medium
>5	High

2.5.3 Determination of the intrinsic properties of coal and coal-shales

Most of the studies investigating the liability of coal and coal-shales to spontaneously combust include a characterisation analysis of the physiochemical and organic composition of the samples (Chakravorty & Kolada, 1988). These include and are not limited to X-ray fluorescence (XRF) Analysis, Carbon and Sulphur speciation (CSS), Proximate and Ultimate Analysis and X-ray Diffraction (XRD) mineralogical analysis.

XRF analyses determine the chemical composition of a sample, and in the iron ore industry, the common chemical constituents analysed for in resource classification are iron (Fe), silica (Si), aluminium (Al), potassium (K), sulphur (S) and phosphorous (P) (Anglo American, 2021). Therefore, analysing the iron carbonaceous shale samples via XRF is important since a large database of similar analyses exists for the Iron Ore Mine A. If a strong correlation exists with either of these analytes, the existing sample database can be used as input into a model for spontaneous combustion propensity (Onifade & Genc, 2018b, 2018d; Uludag, 2007). Carbon and sulphur speciation (CSS) specified to which compound the carbon and sulphur chemical content belongs to (SGS, 2020). Here it is important since the carbon may be part of a carbonate mineral, or it might be organic carbon. For sulphur, the analyses can say whether the sulphur is present as pyrite, which is proven to be important in the determination of spontaneous combustion propensity (SGS, 2020). Intrinsic factors are the constituents that can be measured by proximate and ultimate analysis, namely moisture, ash, volatile matter, carbon, hydrogen, nitrogen, sulphur, pyrite, and oxygen (Chakravorty & Kolada, 1988). This suite of analyses is standard in the coal industry according to the American Society for Testing and Material Standards (ASTM) and equivalent international standards. XRD analyses provide a mineralogical breakdown of the coal and coal-shale samples. Now, not only the chemistry of the samples is known, but the mineralogical form in the element is present, is known.

The intrinsic properties that affect the spontaneous combustion propensity for coal have been studied in South Africa, in the coal fields (Onifade, M. and Genc, B. 2018c; Uludag, S., 2007; Onifade, et al., 2019; Kataka, et al., 2018), and elsewhere in the world (Kim, A.G., and Chaiken, R.F., 1990.) where spontaneous combustion occurs in coal mines. Literature for the analysis of spontaneous combustion in carbonaceous shales itself is limited, and even more so for carbonaceous shale in iron ore mines. The following table (Table 2.3) is a summary of the specific intrinsic property for analysis available on carbonaceous shales, and their effects on spontaneous combustion.

Table 2. 3 Summary of coal, and carbonaceous shale intrinsic properties and the effect it has on spontaneous combustion

INTRINSIC PROPERTY	EFFECT ON SPONTANEOUS COMBUSTION	SOURCE
Ash content	In coal-shales, relative lower ash content (51.5-68.4% ash) has the highest propensity for spontaneous combustion based on medium classification on Wits E-hac Index, the lower risk had ash content 74.0-79.1%, within the low Wits-Ehac Index range. Ash (60.0-80.0%) has the potential for self-heating based on the maximum temperature differential.	Onifade, M. and Genc, B. 2018c; Kim, A.G., and Chaiken, R.F., 1990.
Moisture content	A higher moisture content, 1.5-1.7% for coal-shales, increases spontaneous combustion risk.	Onifade, M. and Genc, B. 2018c;
Volatile matter	An increase in volatile matter content (>15.9%) increases spontaneous combustion risk in coal-shales.	Onifade, M. and Genc, B. 2018c
Pyrite presence	The presence of pyrite increases the spontaneous combustion of coal-shales, range of pyrite content 0.1% to 4.3% for medium propensity and <0.1% for low propensity based on Wits-Ehac Index.	Onifade, M. and Genc, B. 2018c
Maceral composition	Inertinite macerals have a strong correlation with an increase in the liability of spontaneous combustion.	Onifade, et al., 2019.

Table 2.3 continued:

INTRINSIC PROPERTY	EFFECT ON SPONTANEOUS COMBUSTION	SOURCE
Carbon (C), Hydrogen (H), Nitrogen (N), and Oxygen (O) content.	An increase in C, H and N content increases spontaneous combustion risk. The respective C, H, N content for low propensity samples versus medium is as follows: • C: 9.8-10.5% versus 11.0-33.7%, • H: 1.7-2.1% versus 1.3-2.9%, • N: 0.4% versus 0.3-1.0%. The Oxygen content does not seem to affect the liability for spontaneous combustion in coal-shales.	Onifade, M. and Genc, B. 2018c.

2.6 Witbank coal field results on Wits-Ehac Index

Occurrences of elevated ground conditions leading to spontaneous combustion in the coal environment are common and well documented by Genc & Cook (2015). The Witbank coalfields are one of these localities and work has been done in this area to evaluate the propensity for spontaneous combustion using an index developed in the late 1980s. Studies by authors such as Onifade et al. (2019) and, Onifade & Genc (2018a, 2018b, 2018c) aim to relate the intrinsic properties of coal-shales to the Wits-Ehac Index.

Analyses performed on samples from the Witbank coalfields by Onifade et al. (2019) included XRD and X-ray Fluorescence (XRF) analysis to geochemically characterise the shale, as well as proximate, ultimate, total sulphur, and forms of sulphur analysis. Petrography analysis was also done to identify maceral composition (Onifade et al., 2019). The relationship between the inherent properties and its risk of spontaneous combustion as determined via Wits-Ehac Index was investigated by Onifade & Genc (2018c) and summarised in Table 3.1. The Onifade, et al., (2019) study results determined that the carbonaceous shale of the Witbank coalfields is enriched with silica, aluminium and iron, and dominated by kaolinite and quartz minerals. The following relationship between geochemical property and the propensity for spontaneous combustion based on the Wits-Ehac Index was determined (Onifade, et al., 2019):

- Higher SiO_2 and Al_2O_3, have lower risk;
- Higher Fe_2O_3, P_2O_5 and K_2O, have higher risk;
- Higher quartz, kaolinite, and plagioclase (feldspar) have lower risk;
- Higher siderite, pyrite, and muscovite, have higher risk.

If a strong correlation exists between the intrinsic properties of coal-shales and the Wits-Ehac Index, the specific property or analyte may be used as a proxy for spontaneous combustion risk.

From a comparative study by Onifade & Genc (2018a), the strongest linear relationship exists between the Wits-Ehac Index and the proportion of maceral composition, and either a positive or a negative correlation exists between the intrinsic factors of coal, and coal-shales and the Wits-Ehac Index. In this study, the aim is to classify the carbonaceous shale found at the iron ore mine to relate it to the work done on the coal-shales of the Witbank coalfields.

Due to the heterogeneous nature of coal and coal-shales and all the physical and chemical interactions at play during oxidation, it is very difficult to identify one variable to explain the propensity of coal or coal-shales to self-heat (Carras & Young, 1994; Singh & Demirbilek, 1987). No single property should be aimed to be identified to predict spontaneous combustion (Carras & Young, 1994; Singh & Demirbilek, 1987).

2.7 Risk management

Risk management in the mining industry is crucial to the success and sustainability of the operation. The risks may include economic and financial risks, as well as the risk to the safety of the personnel and the risk of preserving the actual resource for mining. Each mine's measures to quantify and manage risk are different but it all comes down to the following three questions (Simonsen & Perry, 1999):

- What can happen?
- How likely is it that it can happen?
- What are the consequences if it does happen?

Since the risk of spontaneous combustion can lead to fires that might destroy the resource, in the case of coal mines, and potentially lead to injury and death of mine personnel, the 'What can happen?' and 'How likely?' questions should be answered by defining the risk of such an occurrence happening. Then, mitigation controls can be implemented.

General controls for managing reactive ground conditions are defined in the code of practice (COP) from the AEISG (2017). The controls included are the establishment of site-specific SOPs, risk management through physical separation of the hot ground condition and the explosive and product selection. When control measures are already in place, it is important that monitoring of ground conditions is done intermittently to identify changes and to increase the understanding of critical areas (AEISG, 2017). Reactive ground indicators according to the AEISG (2017) are as follows;

1. Presence of sulphides (>1%);
2. Black sediments containing sulphides;
3. Mineralised (ore) rocks containing sulphides;
4. Oxidation indicators present such as white or yellow discolouration on rocks;
5. Acidic ground conditions;
6. Corrosion of metal equipment in the ground (rock bolts, safety mesh etc.);
7. Previous occurrences of spontaneous combustion;
8. The smell of ‘rotten eggs’ due to the oxidisation of sulphides and the release of sulfuric acid;
9. Elevated blast hole temperatures; and
10. Elevated ground surface temperatures.

Reactive ground indicators included in the AEISG COP have been tabled and used in a study as criteria to assess the risk for reactive ground on a Witbank Coalfield mine. The study by Mienie (2019), was done following an unplanned detonation incident at a Witbank Coalfield coal mine, to determine if the incident occurred due to reactive ground. The incident was not classified as having been caused by reactive ground, but since the mine scored 8 out of 10 on the reactive ground indicators, it was recommended that continuous monitoring and test work be conducted (Mienie, 2019).

2.8 Chapter summary

The literature review chapter contained an overview of the spontaneous combustion and reactive ground processes and how the two possibly relate to each other. Literature on the pyritic shale occurrence of the iron ore mine in Mount Whaleback (Australia) was included as well as a description of the Gamagara Formation, to which the Iron Ore Mine A carbonaceous shale belongs. Methods for determining the propensity for coal and coal-shales to spontaneously combust were discussed, followed by a detailed review of the Wits-Ehac Index, and results from some of the studies that had been conducted in the Witbank coalfield using the Wits-Ehac Index. Finally, the general management of risk at mines was discussed with the brief inclusion of a case study conducted using the AEISG (2017) COP.

3 RESEARCH METHODS

3.1 Mapping and domaining of the extent of the carbonaceous shale unit in Iron Ore Mine A

The carbonaceous shale unit was modelled using implicit modelling techniques on the geological modelling software Leapfrog, with drill hole information used as a first action to delineate the problem area of the Iron Ore Mine A. Implicit modelling is an automated wireframing method where surface chronology rules and structural guides are used to automatically build a wireframe from drill hole data (Seequent, 2021). This is opposed to the traditional method of building a wireframe manually by digitising points to connect drill hole information (Seequent, 2021). Since the carbonaceous shale forms part of the greater Gamagara shale unit, the first action was to compare the modelled boundary to the exploration borehole logs to identify carbonaceous shale, which followed these steps:

- Step 1: View the Gamagara shale (SH-wireframe) modelled boundary versus the drill hole contacts;
- Step 2: Read through drill hole comments and interrogate the logged 'Colour' field in the database to define intervals to include in the new carbonaceous shale (SH-C) wireframe; and
- Step 3: Refine the original SH-wireframe so that the SH-C wireframe can be modelled on its own by the software.

Visual inspection of the pit area was also conducted post the detonation incident. The presence of carbonaceous shale was confirmed through visual identification based on the black colour in the pit. The location of the black lithology is indicated by the labelled SH-C in Figure 3.1.

Figure 3.1: In-pit expression of carbonaceous shale (SH-C) at the Iron Ore Mine A

Post the incident, a carbonaceous shale wireframe was built from the exploration borehole data which combined the colour information and lithological code used to log the Gamagara shale, as described in the steps above (Section 3.1). In combination with this wireframe information, six existing boreholes were identified that had the potential to be twin drilled. Twin drilling is the process of drilling a duplicate drill hole within close vicinity of the original drillhole to recover duplicate information. In this case, the drilling was to target the intervals from the exploration drillholes that mentioned sulphide and, or carbon content in the existing drill hole lithological logs so that samples could be taken from the twin-drilled hole for laboratory testing.

Figure 3.2 depicts the black carbonaceous shale wireframe (SH-C) that was refined from the full Gamagara shale wireframe (brown). The SH-C wireframe can then be exported in a number of formats for use in other applications to assist in the communication of risk areas to the blasting section at Iron Ore Mine A.

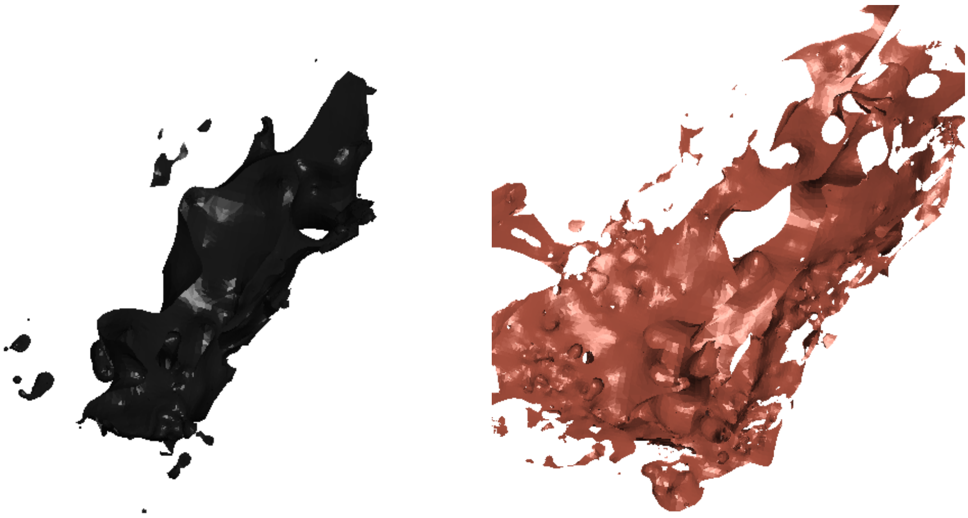

Figure 3.2: Refined SH-C-wireframe (left) created from borehole data from within the full SH-wireframe (right)

Figure 3.3 and Figure 3.4 display the different domains of shale modelled post the incident. The Plan View and Section A-A depict the extent of the surface expression of the carbonaceous shale layer and the vertical extent.

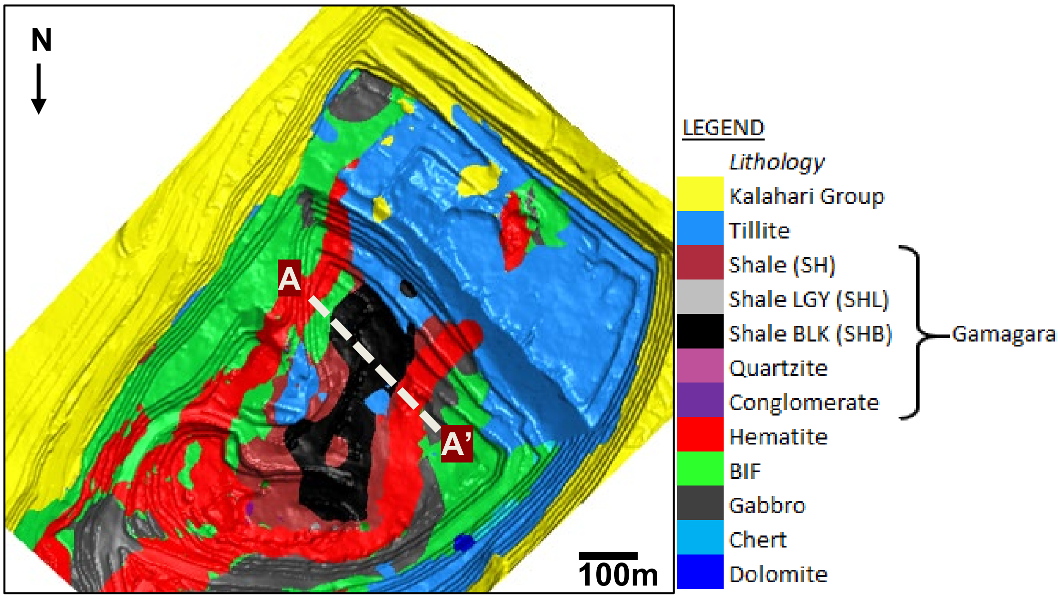

Figure 3.3: Plan view of Iron Ore Mine A pit with surface expression of lithologies on face positions from August 2020. The different Gamagara domains are indicated, with emphasis on the black carbonaceous shale layer (SH-C)

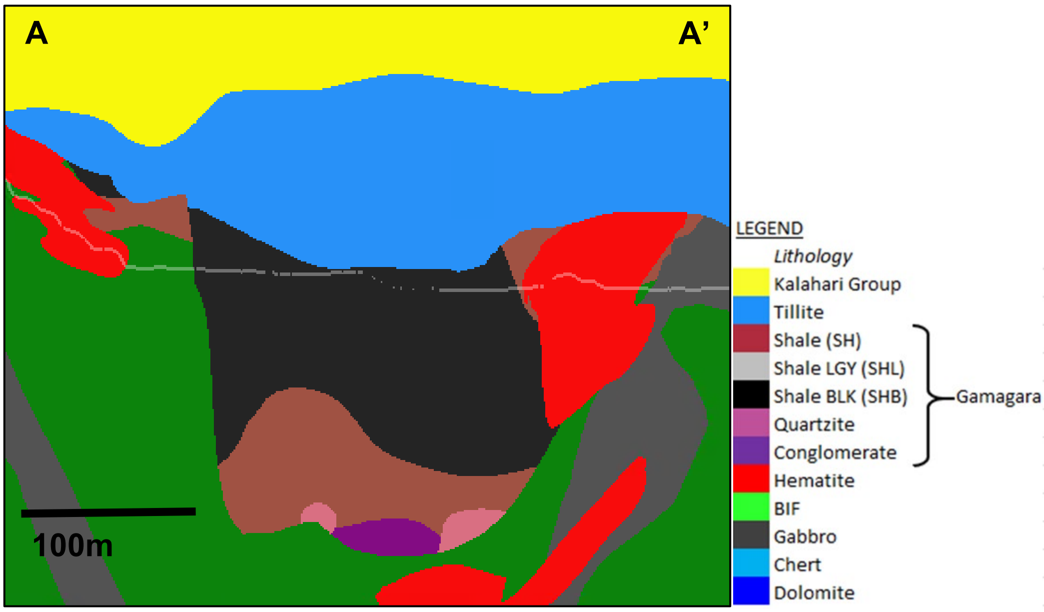

Figure 3.4: Section A-A' through the geological model (see Figure 3.2 for a plan view of the section location). The different Shale domains are indicated, with emphasis on the black carbonaceous shale layer (SH-C)

From the plan view as illustrated in Figure 3.3, the black SH-C wireframe surface expression can be seen as modelled onto the face positions of the open-pit mine close to the time that the incident occurred. In the horizontal plane, the risk area (black) is extensive (300-400m). When looking at a sectional view along the section A-A' per Figure 3.4, the carbonaceous shale is also vertically extensive for an additional 100-120m. The carbonaceous shale is also within the vicinity of the iron ore mineralization (red) which means that drill and blast activities will need to take place in the shale to liberate the iron ore.

3.2 Targeted drilling for geological characterisation and domaining of risk areas within the carbonaceous shale.

Six drillholes were identified to twin-drill and planned to intersect the extensive carbonaceous shale, depicted in Figure 3.5, using the modelled boundary and by evaluating existing exploration drill hole logs based on comments on the presence of sulphides and carbonaceous material.

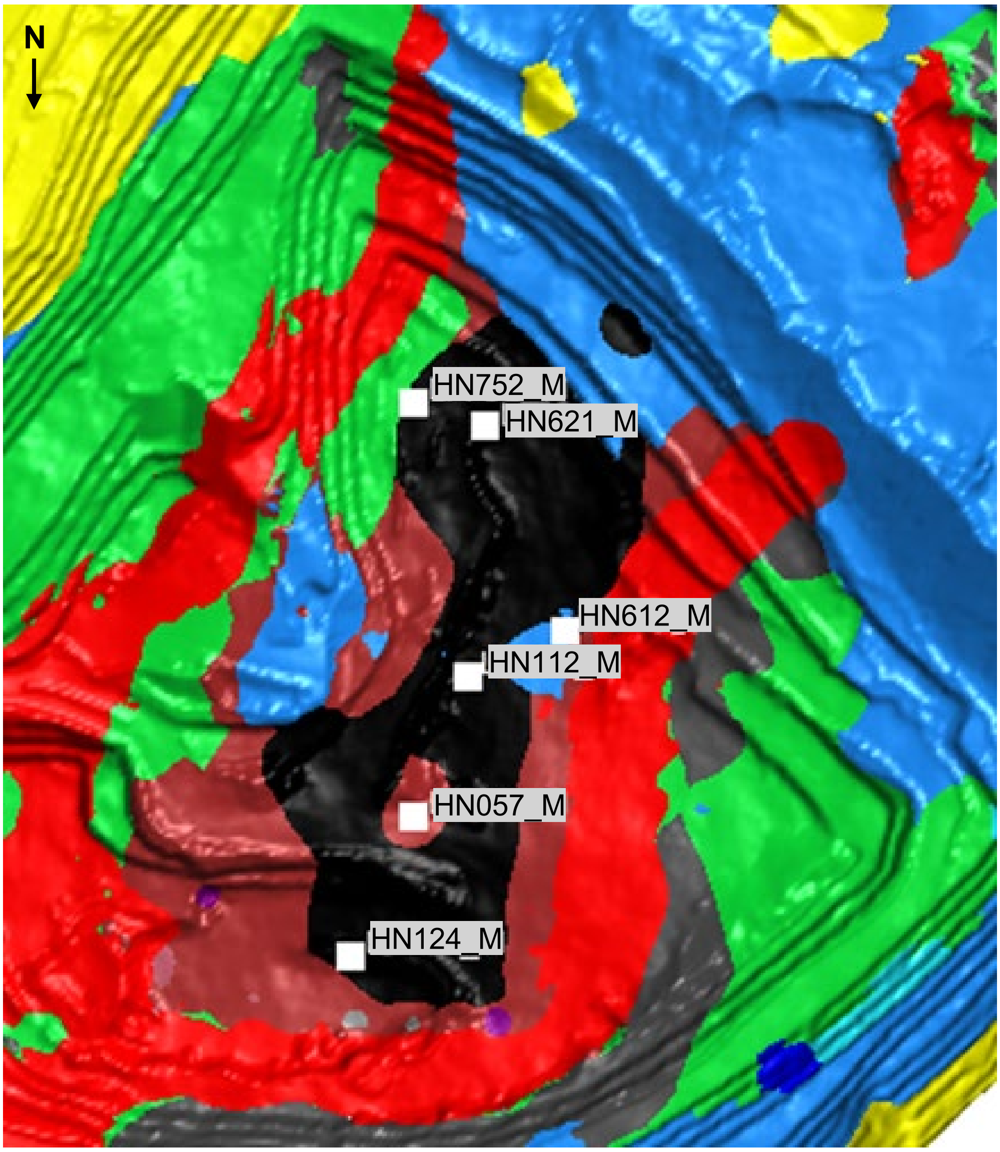

Figure 3.5: Planned boreholes to target carbonaceous shale

The planned drillholes (Figure 3.5) shows the planned collar positions to cover the extents of the modelled carbonaceous shale domain. The drillholes were chosen to cover the expanse of the carbonaceous shale, to better delineate the risk area, as well as to confirm the modelled contacts. Due to the drillholes extending into the deposit, coverage of the carbonaceous shale was targeted at depths as well. The drillholes were planned 100 m - 200 m from each other. The purpose of the extensive testing was to describe risk levels for reactivity and spontaneous combustion and to identify potential proxies between geological features, such as chemistry and mineralogical content, and the reactivity or spontaneous combustion risk. This would aid in future risk identification and delineation per mining bench.

Of the six planned holes, four were successfully completed. One of the drillholes was abandoned due to the proximity to permanent infrastructure, and one intersected no carbonaceous shale and was abandoned. Three holes (HN112_M, HN124_M, HN621_M) were drilled to a depth of 35 m and one (HN612_M) to 20.5m. The latter (HN612_M) was stopped at 20.5 m due to the area being scheduled for drilling and blasting activities that could not be postponed. The hole could not be drilled further. The four successfully drilled holes were logged in detail, paying specific attention to relative sulphide and carbon content.

3.3 Sampling and Analyses

3.3.1 Sample collection and preparation

From the targeted four drillholes, 28 core drilled samples were identified using the worst-case scenario approach as outlined in the AEISG COP (AEISG, 2017). The samples were collected from each drill hole as soon as it was drilled and stored in an air-tight plastic bag, clearly labelled, and recorded, to prevent oxidation and to retain sample integrity. The samples were dispatched to the laboratory for analyses, where they were separately milled and split into representative samples for the following tests:

- XRF Analysis;
- Carbon and Sulphur speciation;
- Proximate and Ultimate Analysis;
- Propensity for spontaneous combustion (Wits-Ehac); and
- XRD mineralogical analysis.

The sampling of the Iron Ore carbonaceous shale was planned to have two streams, Stream A with 16 samples and Stream B with 10 samples, with tests as outlined in Figure 3.6. The samples were chosen to be representative within each drill hole. Stream A sample results are used to classify the Iron Ore Mine A carbonaceous shale unit as a whole with respect to geochemical content. The stream B samples were chosen to investigate the propensity of the Iron Ore Mine A unit to spontaneously combust. Therefore, not all samples were sent for each test.

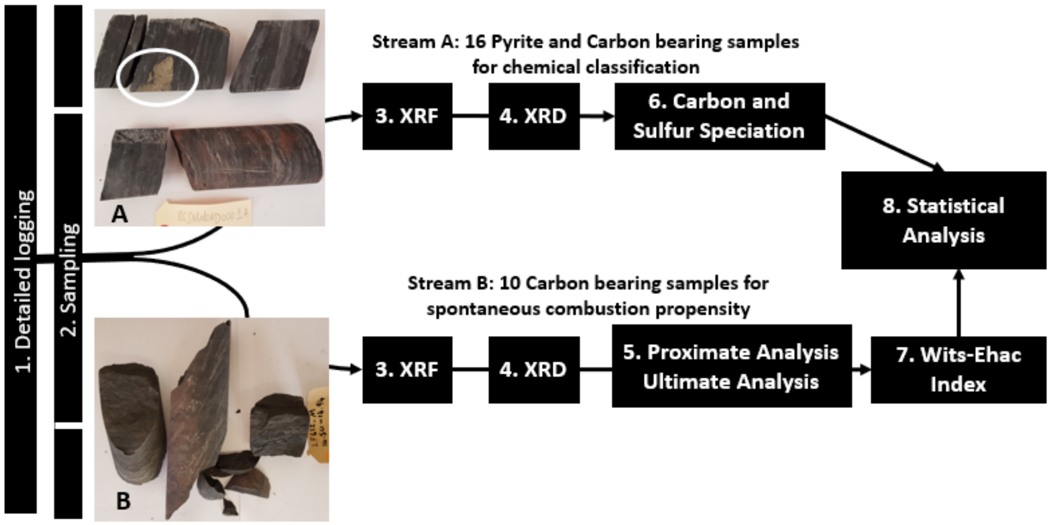

Figure 3. 6: Diagrammatic flow of planned sampling and analysis of Iron Ore Mine A carbonaceous shale. A – Image of SAMPLE0001B with visible pyrite encircled, as an example of 'worst-case' sample sent for chemical classification. B – Image of SAMPLE0016 as an example of 'worst-case-sample sent for spontaneous combustion propensity.

All drilled holes were logged in detail, followed by sampling. All samples were sent for XRF and XRD analyses. Based on visible pyrite (gold) and carbon (black) enrichment, only specific samples were deemed as 'worst-case' following the AEISG (2017) protocol. These samples were all earmarked for CSS. In reality, 11 of the 16 samples of Stream A went for CSS, and 4 for Proximate and Ultimate analysis. For Stream B, which was the 10 samples earmarked for spontaneous combustion liability testing with Wits-Ehac, 9 were sent for Proximate and Ultimate analysis, and 4 of the 10 were sent for CSS. All 10 were sent for Wits Ehac-Index. The 10 samples chosen for the Wits-Ehac Index classification to determine spontaneous combustion risk, were chosen to be representative spatially of the 'worst-case' carbon-bearing lithology. Linear regression analyses of the Wits-Ehac Index results with that of the XRF and Proximate and Ultimate analysis results were conducted.

Due to the different laboratory requirements, not all samples could be sent for all tests at the same time. It was also imperative that samples not undergo oxidation prior to each test spontaneous combustion test (AEISG, 2017). Even though results were not received for each test for all samples, the results achieved are representative of the Iron Ore Mine A carbonaceous shale. And the 10 samples with WEI are indicative of the propensity for the unit to spontaneously combust. Chapter 4 describes the detail of each sample and covers which samples were sent for the different tests.

3.3.2 Method of sample analyses

All samples were analysed at accredited laboratories according to international standards with quality controls in place to deliver accurate and precise results. The samples were crushed to 3mm and then split into fractions for XRF, XRD and CSS. These analytical splits were milled to 75 µm. The samples identified for spontaneous combustion liability via Wits-Ehac Index were crushed to 3mm and sent for analyses at the University of Witwatersrand laboratory. For the XRF analyses, the sample loss of ignition is determined by a thermogravimetric analyser (ISO 9516-1 and ISO 11536), thereafter a fused glass-bead is prepared from the sample for analyses via Axios and Axios advanced X-Ray Spectrometers following methods validated and accredited by ISO /IEC 17025:2017. The percentage (%) of total sulphur is measured using the ELTRA Sulphur analyser according to ASTM4239, and the relative density is determined via the volumetric-pressure relationship in the Micrometrics Accupyc II pycnometer.

CSS was done by Laboratory equipment corporation (LECO) carbon sulphur analyser based on the international organisation of standardisation (ISO) standards (ISO/IEC 17025). The carbon analyses include total carbon (TC), carbonate carbon (CO_3), organic carbon (ORG_C) and graphitic carbon (G_C), done according to LECO methods CSA01V, CSA02V, CSA03V and CSA05V respectively. The sulphur content is measured as total sulphur (S%), sulphate (SO_4%), sulphide (S_2%) and elemental sulphur (ELEM_S%) according to LECO method code CSA06V, CSA11V, CSA08V, and CSA09V respectively. The results are achieved by repeating LECO analyses following selected sample treatments. The proximate and ultimate analyses were done according to accredited and validated methods under ISO/IEC 17025 laboratory with practices outlined in ISO 17246 and ASTM 3176, for the proximate and ultimate analysis respectively. Proximate analyses were done as per ISO 1171 for the ash (A), ISO 562 for volatile matter (VM) andISO 11722 for moisture (M). Total sulphur (TS) and calorific value (CV) were determined according to ASTM D4239 and ISO 1928 respectively. Ultimate analyses were done according to ASTM D3176-15 for determining the elemental composition of the samples, namely carbon (C), hydrogen (H), nitrogen (N), sulphur (S) and oxygen (O). The XRD analyses were done on the Bruker X-ray diffractometer and identification of minerals was conducted using the Bruker AXS Topas software and is based on the best matched peaks according to accredited and validated methods under ISO/IEC 17025. Lastly, the mineral phases were identified and quantified via the Rietveld refinement method.

The propensity for spontaneous combustion analyses was done via the Wits-Ehac test described in Section 2.5.2 of this report, at the University of the Witwatersrand. The samples were received as a split of the original sample that was chemically assayed as described above, in a -3mm size fraction, in vacuum sealed bags. The test apparatus is used to test coals under predefined conditions and a combustibility index is obtained. The apparatus consists of an oil bath, six coal and inert material cell assemblies, an oil circular, a heater, a flowmeter, an air supply compressor, and a computer. The experiment flow is diagrammatically presented in Figure 3.7.

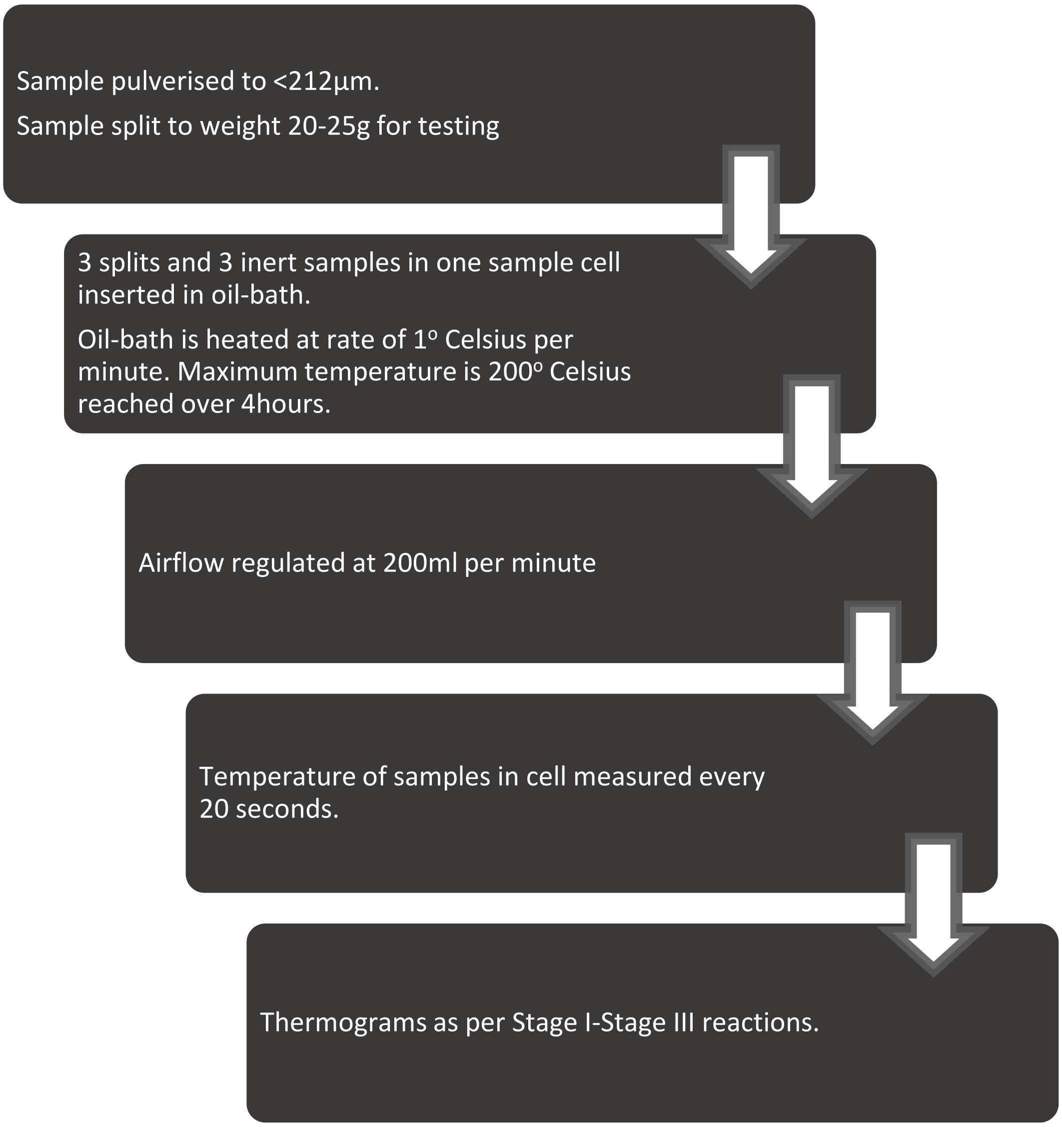

Figure 3. 7: Diagrammatic flow describing the Wits-Ehac test done on Iron Ore Mine A carbonaceous shale samples.

The thermograms produced from the Wits-Ehac Index test is generated by plotting the difference in the temperature of the sample under testing against the temperature of the inert material. During the DTA, three stages are obtainable. The initial temperature of the inert material is greater than the carbonaceous shale sample temperature at Stage I, due to the cooling impact of moisture evaporating from the carbonaceous shale sample. Secondly at Stage II, the carbonaceous shale sample starts to heat up at a greater rate than the inert material during the evaporation of the moisture content and this is based on the propensity of a carbonaceous shale sample to self-heat and attempt to meet the temperature of the oil bath (surrounding temperature). At a point where the line crosses the zero baseline, the high exothermicity is reached and referred to as the XPT. The highest point of exothermicity is achieved at the point where the curve crosses the zero baselines known as the XPT. Stage III follows where the carbonaceous shale sample burns beyond its kick-point. Appendix II contains the thermograms of the 10 samples analysed in this study.

The results of all the chemical and physical tests to ascertain the risk of spontaneous combustion will be compiled into a ‘risk-map’ to delineate areas of lower and higher risk that can be used in the Iron Ore Mine A risk assessment process.

3.3.3 Statistical analyses

A statistical summary was provided per test to characterise the carbonaceous shale unit. The summary included the minimum, maximum, mean and median. The results of all the samples per test are presented in part one of the results section. These results can be used as an average or default for the Iron Ore Mine A carbonaceous shale unit in future studies or comparisons with results from other iron ore mines carbonaceous shale.

The statistical analyses were done using JMP software as well as linear regression analysis. The linear relationship of the chemical properties of the iron ore mine carbonaceous shale with the Wits-Ehac index was determined graphically and the correlation was done following the same categories (Table 4.9) as Onifade & Genc (2018a). The second part of the results section focussed only on the samples on which Wits-Ehac tests were conducted. In this section, the samples’ intrinsic properties were correlated with the Wits-Ehac Index results.

3.4 Chapter summary

The research method chapter described the approach of the research on the Iron Ore Mine A carbonaceous shale unit. The method involves evaluating the existing information available to delineate and model the extent of the shale, after which drillholes are identified to drill, from which to collect samples for analysis. The chemical analysis done on the samples was listed, and described, followed by a description of the statistical analyses to characterise the shale unit as a whole and to analyse and compare the results with that of the Wits-Ehac Index results.

4 RESULTS AND DISCUSSIONS

The results of analyses on the samples from the Iron Ore Mine A carbonaceous shale are presented in this chapter. In Section 4.1 of this report, all samples were used for the data analyses done per analysis type. Section 4.2 covers only the data analyses of the samples sent for WEI. A summary of the analyses conducted per sample as part of this study is shown in Table 4.1.

Table 4.1: Summary of analysis available per sample of Iron Ore Mine A carbonaceous shale

SAMPLE DETAIL				ANALYSIS TYPE					
Hole ID	**From (m)**	**To (m)**	**Sample ID**	**XRF**	**XRD**	**CSS**	**PA**	**U**	**WEI**
HN112_M	14.99	15.37	SAMPLE0001B	Y	Y	Y	N	N	N
HN112_M	17.10	17.50	SAMPLE0002B	Y	Y	Y	N	N	N
HN112_M	26.60	27.10	SAMPLE0003B	Y	Y	Y	N	N	N
HN124_M	4.00	4.34	SAMPLE0006B	Y	Y	N	Y	Y	N
HN124_M	6.49	6.85	SAMPLE0007B	Y	Y	Y	N	N	N
HN124_M	12.90	13.32	SAMPLE0008B	Y	Y	Y	N	N	N
HN124_M	16.38	16.72	SAMPLE0009B	Y	Y	Y	N	N	N
HN124_M	18.48	18.83	SAMPLE0010B	Y	Y	N	Y	Y	N
HN124_M	6.85	7.25	SAMPLE0011	Y	Y	N	Y	Y	Y
HN124_M	26.59	26.94	SAMPLE0013B	Y	Y	Y	N	N	N
HN124_M	31.35	31.72	SAMPLE0014B	Y	Y	N	Y	Y	N
HN124_M	1.50	2.12	SAMPLE0027	Y	Y	N	Y	Y	Y
HN124_M	16.72	17.31	SAMPLE0028	Y	Y	Y	Y	Y	Y
HN612_M	6.00	6.40	SAMPLE0004B	Y	Y	Y	N	N	N
HN612_M	9.28	9.65	SAMPLE0005B	Y	Y	Y	N	N	N
HN612_M	14.50	14.94	SAMPLE0016	Y	Y	Y	N	N	Y
HN612_M	4.00	4.54	SAMPLE0025	Y	Y	N	Y	Y	Y
HN612_M	19.00	19.55	SAMPLE0026	Y	Y	Y	Y	Y	Y
HN621_M	0.55	1.05	SAMPLE0017	Y	Y	N	Y	Y	Y
HN621_M	3.63	4.00	SAMPLE0018B	Y	Y	Y	N	N	N
HN621_M	4.00	4.30	SAMPLE0019	Y	Y	N	Y	Y	Y
HN621_M	7.41	7.77	SAMPLE0020B	Y	Y	N	Y	Y	N
HN621_M	19.35	19.72	SAMPLE0021B	Y	Y	N	N	N	N
HN621_M	19.72	20.17	SAMPLE0022	Y	Y	Y	Y	Y	Y
HN621_M	29.17	29.60	SAMPLE0023B	Y	Y	Y	N	N	N
HN621_M	31.84	32.19	SAMPLE0024	Y	Y	N	Y	Y	Y

Carbon & Sulphur Speciation (CSS), Proximate Analysis (PA), Ultimate Analysis (U), Wits-Ehac Index (WEI)

Table 4.1 gives sample details, the hole number and sample intervals per sample that may aid in correlating analysis between intervals that are within close proximity or even adjacent to each other. This section of the report is presented in two parts; Section 4.1 presents the characterisation results of all samples from the Iron Ore Mine A carbonaceous shale. In section 4.2 only samples that have Wits-Ehac Index results are presented, along with the linear regression analyses to compare it with the results from the XRF, and proximate and ultimate analyses. The XRD results done on the samples with Wits-Ehac results are also discussed in Section 4.2.

4.1 Chemical characterisation of Iron Ore Mine A carbonaceous shale

4.1.1 XRF analysis

XRF analyses were done on 26 samples to determine the chemical constituents and percentage thereof that occur in the carbonaceous shale. The results are presented in Table 4.2.

Table 4.2: XRF Results per sample

SAMPLE ID	Fe%	SiO_2%	Al_2O_3%	K_2O%	P%	S%
SAMPLE0001B	1.959	75.388	14.046	1.023	0.068	1.039
SAMPLE0002B	1.739	71.490	16.963	1.278	0.012	0.789
SAMPLE0003B	1.180	66.937	20.974	1.608	0.013	0.130
SAMPLE0004B	0.925	69.030	19.655	1.462	0.011	0.206
SAMPLE0005B	1.002	66.483	21.146	1.541	0.012	0.223
SAMPLE0006B	1.748	65.865	20.066	1.610	0.012	0.698
SAMPLE0007B	1.840	66.986	19.063	1.652	0.012	0.110
SAMPLE0008B	3.210	83.714	5.835	0.541	0.003	0.363
SAMPLE0009B	13.243	50.937	14.590	1.283	0.009	4.192
SAMPLE0010B	28.328	27.444	8.896	0.755	0.006	3.295
SAMPLE0011	1.340	63.512	21.890	1.945	0.014	0.214
SAMPLE0013B	5.960	57.635	19.932	1.782	0.012	0.722
SAMPLE0014B	39.465	13.725	3.124	0.063	0.005	0.629
SAMPLE0016	0.708	65.786	20.608	1.590	0.011	0.210
SAMPLE0017	1.202	64.356	21.953	1.771	0.012	0.584
SAMPLE0018B	1.364	66.100	20.909	1.688	0.012	0.519
SAMPLE0019	1.226	66.457	20.307	1.646	0.012	0.490
SAMPLE0020B	32.378	22.684	7.770	0.254	0.007	0.787
SAMPLE0021B	1.459	61.356	23.153	2.130	0.014	0.117
SAMPLE0022	1.670	60.472	24.203	2.247	0.014	0.038
SAMPLE0023B	22.656	32.366	12.804	1.043	0.007	1.924
SAMPLE0024	1.187	62.414	22.880	1.982	0.013	0.205
SAMPLE0025	0.697	73.891	15.904	1.161	0.010	0.264
SAMPLE0026	0.957	64.534	21.593	1.662	0.012	0.539
SAMPLE0027	1.624	64.119	20.925	2.024	0.013	0.110
SAMPLE0028	3.433	63.781	18.240	1.779	0.011	0.424

Four samples (SAMPLE0010B, SAMPLE0009B, SAMPLE0001B, SAMPLE0023B) are above 1.000% Sulphur, with two out of the four being 3.295% and 4.192% respectively. Four samples have an Fe% greater than 20.000% (SAMPLE0010B, SAMPLE0020B, SAMPLE0014B, SAMPLE0023B). These samples were not visibly ferruginised since the colour of the sample was not reddish-brown, as it is when hematite is present (Rossman, 1996). XRD analysis confirmed the mineral to be siderite and not hematite. Siderite is an iron carbonate mineral, and hematite is an iron oxide mineral that is being mined at Iron Ore Mine A. Two samples (SAMPLE0010B and SAMPLE0023B), that have elevated sulphur and iron (Fe) in common, relative to the other samples, might be indicative of high pyrite content. The minerals present in these samples are pyrite and siderite according to the XRD analysis. The presence of pyrite can increase the risk of reactive ground occurrence (Kim & Cheiken, 1990, Gunawan & Zhang 2009).

The following is a summary of the geochemical composition of the Witbank coal-field carbonaceous shale studied in Onifade, et al. (2019) that had low to medium propensity for spontaneous combustion:

- SiO_2: 59.6-60.1% - Medium liability; 59.7-67.9% - Low liability
- Al_2O_3: 23.5-24.9% - Medium liability; 24.9-30.9% Low liability
- Fe_2O_3: 9.0-9.3% - Medium liability; 1.1-3.4% - Low liability
- P_2O_5: 0.1-0.2% -Medium liability; 0.1-0.2% - Low liability
- K_2O: 2.7-2.9% - Medium liability; 1.9-2.8%-Low liability

Based on these results, the student notes that with an increase in silica, and aluminium the liability for spontaneous combustion in the carbonaceous shale decreases. An increase in iron, phosphor, and potassium content has the opposite effect, the liability increases. The risk for SAMPLE0010B, SAMPLE0014B and SAMPLE0020B to spontaneously combust is relatively higher than the rest of the samples, based on the relatively elevated Fe, in combination with a lower Al_2O_3% composition.

In order to characterise the chemistry of the Iron Ore Mine A carbonaceous shale, Table 4.3 presents a summary of statistics on the XRF analysis done. The mean and median data for Sulfur is below the risk limit of 1%, as described in the literature (Mienie, 2019; AEISG, 2017). The risk of elevated Sulfur is known to contribute to the risk of spontaneous combustion (AEISG, 2017, Kim & Chaiken, 1990) and therefore the Iron Ore Mine carbonaceous shale is at lower risk due to the low sulphur composition. The samples have a typical Fe of 1.647%, SiO_2 of 64.445%, Al_2O_3 of 19.999%, K_2O of 1.609%, P of 0.012%, and S of 0.457%.

Table 4.3: Statistics on Carbonaceous Shale XRF Results

	Fe	SiO_2	Al_2O_3	K_2O	P	S
	%	%	%	%	%	%
Min	0.697	13.725	3.124	0.063	0.003	0.038
Max	39.465	83.714	24.203	2.247	0.068	4.192
Mean	6.635	59.518	17.593	1.443	0.013	0.724
Median	1.647	64.445	19.999	1.609	0.012	0.457
Typical Gamagara shale*	17.091	52.701	16.324	1.443	0.032	0.310

*From Scott & Gous, (2020)

This chemical composition for the Iron Ore Mine A carbonaceous shale can be used as a preliminary 'default chemistry' for the unit, and it can be used to inform the blasting operational team. Before this study, there was no chemistry available except the chemistry of the typical Gamagara shale unit (Scott & Gous, 2020) before it was refined to include the carbonaceous shale unit (Figure 3.2). The median chemistry for SiO_2, Fe, and K_2O for the Iron Ore Mine A carbonaceous unit, falls within the same ranges as the Witbank coal-shale reported in Onifade, et al., (2019) which had a low risk for spontaneous combustion based on the Wits-Ehac Index. The median results for the carbonaceous shale of Iron Ore Mine A are similar to the low-reactive upper member, and the inert zone of the Mount McRae shale discussed in the literature review. The Iron Ore Mine A carbonaceous shale has a low chance for spontaneous combustion based on these results.

In Figure 4.1, the mean composition of the Iron Ore Mine A carbonaceous shale is plotted against the default chemistry used for Gamagara shale unit as a whole in Iron Ore Mine A. The biggest difference is in the Fe% and SiO_2%, with a minor difference in the Al_2O_3%, K_2O%, P% and S%.

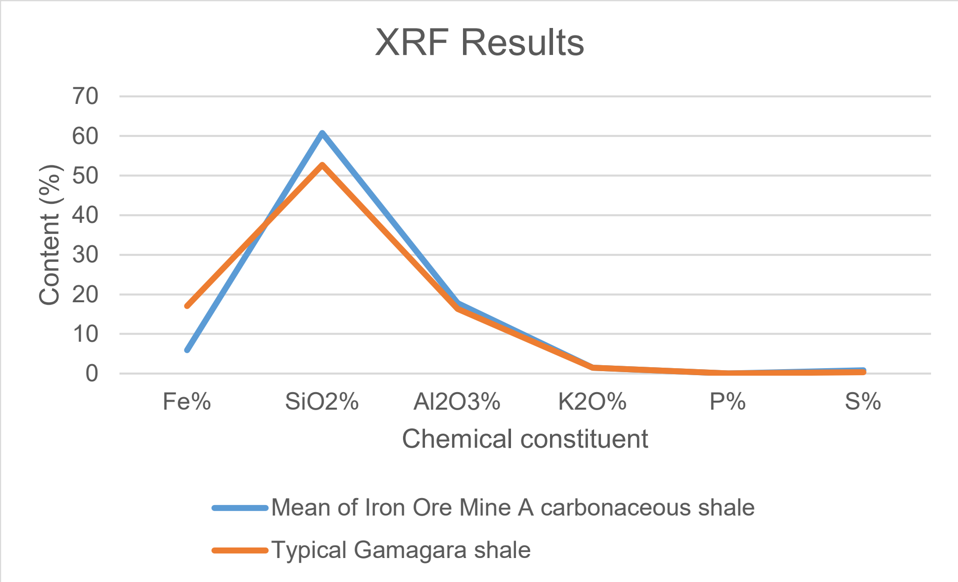

Figure 4.1: Mean composition of the Iron Ore Mine A carbonaceous shale versus default chemistry used for Gamagara shale unit as a whole in Iron Ore Mine A

4.1.2 Carbon and sulphur speciation (CSS)

Organic carbon content increases the risk of ground conditions with elevated temperatures due to its low ignition point (Han et al., 2005). When pyrite occurs the risk of spontaneous combustion is aggravated due to oxidation and has the potential to react with ammonium nitrate in the explosives used during blasting (Han et al., 2005). The presence of carbonate compounds acts as a buffer, absorbing energy during spontaneous combustion (Descotes et al., 2002).

Fifteen samples were sent for carbon and sulphur speciation to further refine the carbonaceous shale unit. The results per sample are presented in Table 4.4.

Table 4.4: Carbon and Sulphur speciation results per sample

	TS	S_2	E_S	SO_4	TC	CO_3	ORG_C	G_C
SAMPLE ID	%	%	%	%	%	%	%	%
SAMPLE0001B	0.84	0.72	<0.30	<0.40	0.33	<0.05	0.30	<0.05
SAMPLE0002B	0.89	0.88	<0.30	<0.40	0.37	<0.05	0.35	<0.05
SAMPLE0003B	0.18	0.18	<0.30	<0.40	0.43	<0.05	0.54	<0.05
SAMPLE0004B	0.33	0.21	<0.30	<0.40	0.51	<0.05	0.49	<0.05
SAMPLE0005B	0.35	0.33	<0.30	<0.40	0.56	<0.05	0.49	<0.05
SAMPLE0007B	0.14	0.12	<0.30	<0.40	0.57	0.83	0.35	<0.05
SAMPLE0008B	0.45	0.40	<0.30	<0.40	0.61	0.32	0.49	<0.05
SAMPLE0009B	3.83	3.73	<0.30	<0.40	2.26	9.33	0.38	<0.05
SAMPLE0013B	0.58	0.55	<0.30	<0.40	1.25	3.27	0.56	<0.05
SAMPLE0016	0.25	0.21	<0.30	<0.40	0.53	0.20	0.51	<0.05
SAMPLE0018B	0.48	0.42	<0.30	<0.40	0.56	0.10	0.50	<0.05
SAMPLE0022	0.04	0.04	<0.30	<0.40	1.03	0.27	0.79	0.20
SAMPLE0023B	1.94	1.84	<0.30	<0.40	4.66	20.70	0.47	<0.05
SAMPLE0026	0.45	0.38	<0.30	<0.40	0.55	0.31	0.53	<0.05
SAMPLE0028	0.41	0.38	<0.30	<0.40	0.76	1.35	0.46	<0.05

TS-Total Sulphur; S_2-Disulphur; E_S-Elemental sulphur; SO_4-sulphate; TC-Total Carbon; CO_3-Carbonate; ORG_C-Organic carbon; G_C-Graphitic carbon

The bulk of the total sulphur content shown in Table 4.4 occurs as sulphide S. The concentrations for sulphate (SO_4) or elemental sulphur (Elem_S) fall below the detection limit, which is 0.40% and 0.30% respectively. The presence of pyrite (FeS_2) is identified from visual logging and is confirmed by XRD analysis. Except for two samples, SAMPLE0009B and SAMPLE0023B, all samples fall below 1% TS. The low total sulphur compares with sulphur values of the inert to low propensity for spontaneous combustion of the Mount McRae unit in an iron ore mine, as well as carbonaceous shale of the Witbank coal area.

The total carbon ranges fall below 1% for most samples, except for SAMPLE0009B, SAMPLE0013B, SAMPLE0022 and SAMPLE0023B. This carbon content is still very low, since coal-shales with low spontaneous combustion risk, average around 10% C, as published by Onifade & Genc (2018a, 2018b, 2018c) and Onifade et al. (2019). All the samples analysed have similar organic carbon content. For samples SAMPLE0001-SAMPLE0005, the total carbon is present as organic carbon, with no measurable carbonate content. For the rest of the samples, the total C content is higher due to increased carbonate (CO_3) content. The CO_3-bearing mineral present is siderite.

According to Beamish & Theiler (2016), reactive black shale has a TS of 14.8% with a total organic carbon of 2.58%. The only sample with high enough organic carbon within this range is SAMPLE0023B. Non-reactive samples that did display in initial heating have 2.64% TS, and 0.51% total organic carbon, but the reactive sulphide is insufficient to sustain the exothermic reaction (Beamish & Theiler, 2016). Based on these criteria the samples of the Iron Ore Mine A have the potential to self-heat, but not enough for combustion to take place.

Table 4.5 displays summarised statistics of the CSS results of the Iron Ore Mine A carbonaceous shale. The sulphur presence is confirmed to be pyrite and still poses a risk for spontaneous combustion and ground reactivity. According to the AEISG (2017), acidic ground conditions contribute to the risk for ground reactivity, and the presence of carbon as CO_3 acts as a buffer, neutralising the acid produced during pyrite oxidation (Descotes et al., 2002). Therefore, it is significant information to supply to the drill and blasting team on Iron Ore Mine A that very little organic carbon is present in the Iron Ore Mine carbonaceous shale.

Table 4.5: Statistics on Carbonaceous Shale Carbon/Sulphur Speciation Results

	TS	**S_2**	**E_S**	**SO_4**	**TC**	**CO_3**	**ORG_ C**	**G_C**
	%	**%**	**%**	**%**	**%**	**%**	**%**	**%**
Min	0.04	0.04	-	-	0.33	0.10	0.30	0.20
Max	3.83	3.73	-	-	4.66	20.70	0.79	0.20
Mean	0.78	0.73	-	-	1.04	3.67	0.48	0.20
Median	0.45	0.39	-	-	0.57	0.58	0.49	0.20

In the literature review, the presence of a carbonaceous shale unit in an iron ore mine was discussed based on a study by Han et al. (2005) on the Mount

McRae shale unit. The Mount McRae shale had sulphur content ranging from <0.1% to >30% sulphur (Han et al., 2005) between the three different zones in the shale. The Iron Ore Mine A shale falls within this same range, but the maximum of 3.83% is significantly lower than the 35% maximum of the Mount McRae shale (Han, et al., 2005). Similar to the Mount McRae shale, the common sulphur-bearing substance for Iron Ore Mine A is Sulphide. The carbon content of the Iron Ore Mine A shale compares better with the Mount McRae shales than the sulphur content does. Mount McRae's total carbon, ranges between 0.5% and 6%, and Iron Ore Mine A between 0.33 and 4.66%. Iron Ore Mine A carbonaceous shale has an average of 1.04% total sulphur versus an average of 3% for Mount McRae. In contrast with the Mount McRae shale bulk carbonate form being organic carbon (Han, et al., 2005), the bulk of the Iron Ore Mine A's shale contains carbonate carbon and not organic carbon. According to the CSS results, in comparison to the Mount McRae shale unit, the Iron Ore Mine A samples can be considered a low organic carbon, low sulphur bearing shale.

4.1.3 Proximate and ultimate analysis

Coal and coal-shales are known to undergo spontaneous combustion due to the interaction of the organic carbon with atmospheric conditions (Onifade & Genc, 2018a, 2018b, 2018c). The carbonaceous shale present in Iron Ore Mine A is not related to any coal deposit, and the risk for spontaneous combustion in situ is highly unlikely when considering spontaneous combustion incidents typically occur in the coal environment (Kim & Chaiken, 1990). However, the risk for elevated ground temperatures does exist within the carbonaceous shale, especially when areas are fractured due to blasting activities (Potgieter, 2018; Restuccia et al., 2017) as what was the condition in the area in which the high potential incident (HPI) occurred.

To relate the carbonaceous shale of the Iron Ore Mine A to the coal-shales of the Witbank area, the Proximate and Ultimate analyses were conducted. The results from this analysis are shown in Table 4.6.

Table 4.6: Proximate and Ultimate analyses result per sample

SAMPLE ID	Proximate Analyses				Ultimate Analyses				
	M (%)	A (%)	CV (MJ/kg)	VM (%)	TS (%)	C (%)	H (%)	N (%)	O (%)
SAMPLE0006B	0.72	92.5	0.19	6.7	0.73	0.01	0.91	0.09	5.76
SAMPLE0010B	0.50	79.5	0.59	9.7	2.94	3.58	0.41	0.06	13.51
SAMPLE0011	0.91	91.8	0.21	7.3	0.32	0.03	0.89	0.10	6.88
SAMPLE0014B	0.55	74.5	1.05	12.5	0.67	5.74	0.17	0.05	18.87
SAMPLE0017	0.75	91.9	0.21	7.4	0.58	0.01	0.88	0.09	6.54
SAMPLE0019	0.74	92.5	0.16	6.7	0.58	0.01	0.81	0.09	6.01
SAMPLE0020B	0.36	80.7	0.53	9.8	0.87	3.87	0.42	0.06	14.1
SAMPLE0022	0.94	91.9	-	7.2	0.40	0.06	0.96	0.07	6.61
SAMPLE0024	0.78	91.8	0.23	7.4	0.31	0.01	0.9	0.08	6.91
SAMPLE0025	0.43	94.1	0.11	5.3	0.39	0.01	0.66	0.06	4.78
SAMPLE0026	0.91	92.0	-	7.0	0.45	0.00	0.79	0.05	6.71
SAMPLE0027	0.71	92.5	0.14	6.6	0.12	0.01	0.82	0.05	6.50
SAMPLE0028	0.91	92.4	-	6.6	0.41	0.00	0.79	0.05	6.35

Air dried results for; M-Inherent Moisture, A-Ash content, CV-Calorific Value, VM-Volatile Matter, H-Hydrogen (including H in moisture), N-Nitrogen, O-Oxygen (including O in moisture).

The samples of carbonaceous shale with the following properties have a higher spontaneous combustion liability index (Onifade & Genc, 2018c):

- Lower ash content (Lower spontaneous combustion risk with Ash% between 51.5% and 68.4%, and above);
- Higher moisture content (Lower risk with moisture content less than 1.5%);
- High volatile matter (Lower risk with VM less than 15.9%); and
- High Sulphide presence (Low risk with pyrite less than 0.1%, medium with 0.1% to 4%).

According to these criteria, all the samples for the Iron Ore Mine A carbonaceous shale has low to zero potential for spontaneous combustion when compared to that of the Witbank coal-shale (Onifade & Genc 2018c). However, when looking at the relative liability for spontaneous combustion between the samples of the Iron Ore Mine A shale itself, the following samples have a higher risk of spontaneous combustion, based on its proximate analysis: SAMPLE0010B, SAMPLE0011, SAMPLE0014B, SAMPLE0017, SAMPLE0020B and SAMPLE0024. From these, SAMPLE00010B has the highest risk due to its 2.94% S content, although it will fall into the medium category for spontaneous combustion risk when comparing it with the results of Onifade & Genc (2018c).

A summary of the results of proximate analysis from the 13 carbonaceous shale samples are presented in Table 4.7.

Table 4.7: Statistics on Carbonaceous Shale Proximate Analysis Results

	M	**A**	**CV**	**VM**
	%	**%**	**MJ/kg**	**%**
Max	0.94	94.1	1.05	12.4
Min	0.36	74.5	0.11	0.0
Mean	0.71	89.1	0.34	2.5
Median	0.74	91.9	0.21	0.1

The average Ash% content for the Iron Ore carbonaceous shale samples is 89.1%, placing it in the ‘Rock’ classification as opposed to ‘carbonaceous shale’ class (ECE-UN, 1998). However, the minimum result of 74.5% ash does place the lithology into the ‘carbonaceous’ category. When considering the classification of coal-shales of Alpern and de Sousa (2002), the Iron Ore Mine A shale is poor coal shale due to the lowest Ash% sitting on the limit with the calorific value of only 0.3MJ/kg. High ash content is related to high mineral matter content (Snyman, 1989) The Witbank coal-shale described in Onifade and Genc (2018c) has a lower ash content to that of the Iron Ore Mine A carbonaceous shale, making the Iron Ore Mine A carbonaceous shale a lower risk with respect to spontaneous combustion.

The low calorific values are expected since it is a measurement of importance in coal analyses as it determines the grade, which is not applicable to a carbonaceous shale. However, the low calorific value may be an indication that the material may be more inert since more energy will be consumed during heating/combustion than released (Alpern & de Sousa, 2002). The volatile matter content is also relatively low, with only one sample (SAMPLE0014B) above 10%. The higher the volatile matter content, the higher the proneness for spontaneous combustion (Onifade & Genc, 2018c; Banerjee, 2000), and since the Iron Ore Mine A volatile matter content is much lower than that of the Witbank coal-shales, it is less at risk for spontaneous combustion.

The ultimate analysis provides the elemental organic composition of the coal-shale. The carbon and sulphur content are very low, as supported by the CSS test result. Thirteen samples were sent for ultimate analysis with a statistical summary of the results presented in Table 4.8.

Table 4.8: Statistics on Carbonaceous Shale Ultimate Analysis Results

	S	C	H	N	O
	%	%	%	%	%
Max	2.94	5.74	0.96	0.10	18.87
Min	0.12	0.00	0.17	0.05	4.78
Mean	0.67	1.03	0.72	0.07	8.43
Median	0.45	0.01	0.81	0.06	6.61

The average C, H, and N composition of the Iron Ore Mine A carbonaceous shale is significantly lower than that of the Witbank coal-shales reported on by Onifade and Genc (2018c), resulting in it having a relatively lower risk for spontaneous combustion compared to the Witbank coal-shale.

The sulphur composition is significant to drill and blast activities due to the potential reactive ground incident. The average sulphur is low, 0.67%, well below the limit of risk of 1% as supplied in the AEISG (2017) COP. The carbon and sulphur are present mostly in the minerals siderite (carbonate) and pyrite (sulphide), as discussed in the CSS section of the report (section 4.1.2).

4.1.4 XRD (X-Ray Diffraction) analysis

The mineralogical breakdown of 26 Iron Ore Mine A carbonaceous shale samples is given in Figure 4.2, analysed as described in Chapter 3.

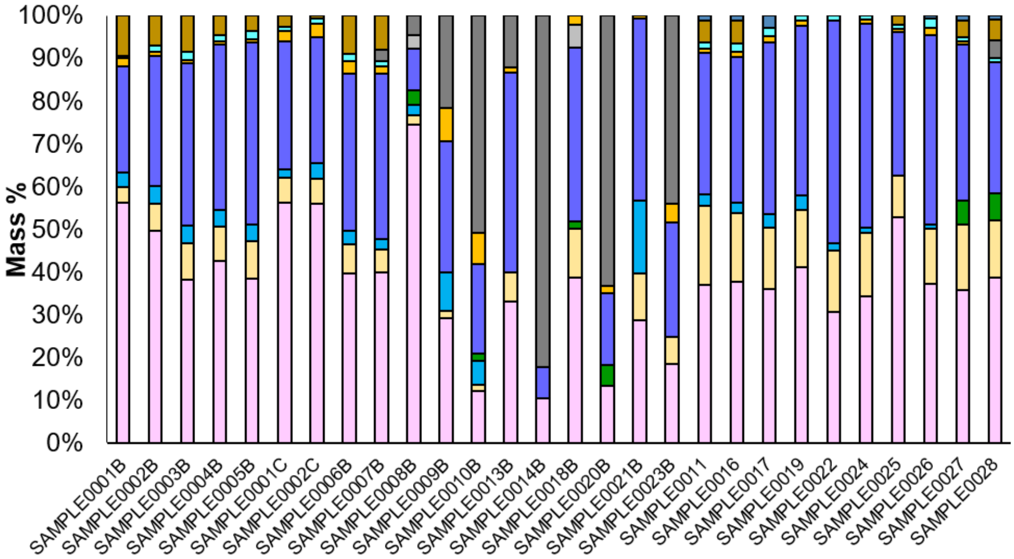

Figure 4.2: Mineralogical composition of carbonaceous shale samples from XRD results

The mineralogical composition of the Iron Ore Mine A carbonaceous shale is typical of that occurring in the coal environment, namely clays, pyrite and carbonates (Wagner, 2021). The dominant minerals in the Iron Ore Mine A samples are kaolinite, quartz and siderite, whereas the Witbank coal-shale reported on in Onifade, et al., (2019) was dominated only by kaolinite and quartz. Kaolinite is a common clay mineral, and quartz is a common silicate mineral. As discussed previously, the significance of the siderite presence is that the carbonate reacts as a buffer to the exothermic oxidation reaction that may lead to spontaneous combustion (Descotes et al., 2002). However, the 10 samples sent for Wits-Ehac Index and its' related XRD analysis are discussed in Section 4.2 in further detail. Pyrite content logged during visual inspection of the samples was confirmed with the XRD results. The average pyrite content is 2% and varies in its abundance. Three samples have a pyrite percentage greater than 3%, SAMPLE0009B, SAMPLE0010B and SAMPLE0023B with 8%, 7% and 4% respectively (see Appendix I for the table of mineralogical composition percentages). The pyrite presence can have a catalytic effect on the oxidation reaction that may lead to an increased spontaneous combustion propensity (Kim & Chaiken, 1990). The Witbank coal-shale results studied in Onifade, et al. (2019) inferred that the relative higher pyrite and muscovite has a higher risk for spontaneous combustion, and higher kaolinite, plagioclase (feldspar mineral) and siderite has a lower proneness for spontaneous combustion. The significance of siderite is discussed in Section 4.2.3.

4.2 Propensity for spontaneous combustion (Wits-Ehac Test)

This section evaluates the Wits-Ehac Index results of 10 Iron Ore Mine A carbonaceous shale samples.

Table 4. 9: Sample detail with respect to WEI results.

SAMPLE ID	HOLEID	FROM (m)	TO (m)	WEI	XPT (°C)	SC Liability
SAMPLE0027	HN124_M	1.50	2.12	3.89	132.4	Medium
SAMPLE0011	HN124_M	6.85	7.25	5.58	95.1	High
SAMPLE0028	HN124_M	16.72	17.31	4.75	113.2	Medium
SAMPLE0025	HN612_M	4.00	4.54	5.31	96.1	High
SAMPLE0016	HN612_M	14.5	14.94	6.25	95.7	High
SAMPLE0026	HN612_M	19.00	19.55	4.34	121.5	Medium
SAMPLE0017	HN621_M	0.55	1.05	5.59	91.7	High
SAMPLE0019	HN621_M	4.00	4.30	6.05	86.7	High
SAMPLE0022	HN621_M	19.72	20.17	4.95	105.8	Medium
SAMPLE0024	HN621_M	31.84	32.19	5.33	104.3	High

XPT-Crossing Point temperature, SC Liability – Spontaneous combustion liability based on WEI.

The 10 samples (Table 4.9) sent for WEI were chosen from the carbonaceous shale unit intersected by three successfully completed drillholes within Iron Ore Mine A. The samples were taken at different intervals within each hole to be representative of the vertical extent with a minimum of 3 samples per hole. The liability of all samples is between medium and high, with variation between holes, as well as variation with depth in each hole. These results correspond with the Genc & Cook (2015) results for Witbank coal-field samples. The crossing-point temperatures (XPT) are on average lower per liability category for the Iron Ore Mine A carbonaceous shale compared to that of the Witbank coal-field (Genc & Cook, 2015).

The Wits-Ehac Index results for the Iron Ore Mine A carbonaceous shale were further analysed by correlating them with the intrinsic factors and chemical composition to determine the linear relationship with the spontaneous combustion propensity. This correlation can then be compared with the correlations determined from the results of studies of the Witbank coalfields. The relationship of the Wits-Ehac with the properties of the carbonaceous shale is presented graphically. The first relationship presented is with the XRF results. A strong correlation of Wits-Ehac with either of these properties of carbonaceous shale could aid in identifying risk with future analyses done at Iron Ore Mine A.

The correlation was done by grouping the results according to the categories defined by Onifade & Genc (2018b) as presented in Table 4.10. The categories group properties according to the R-squared value to evaluate the strength of the linear relationship. The closer to 1 the R-squared value is, the more perfect the correlation is between the two variables plotted (Onifade & Genc, 2018b).

Table 4.10: Table of categories used to define a relationship between carbonaceous shale properties and the Wits-Ehac index (Onifade & Genc, 2018a)

Category	R^2 value range	Relationship
1	0.95 to 1 *or* -0.95 to -1	Very strong to perfect positive or negative relationship
2	0.51 to 0.94 *or* -0.51 to -0.94	A strong positive or negative linear relationship
3	0.25 to 0.50 *or* -0.25 to -0.50	A moderate positive or negative linear relationship
4	0.1 to 0.24 *or* -0.1 to -0.24	A weak positive or negative linear relationship
5	<0.1, not 0	Very weak positive or negative relationship
6	0	No linear relationship

Table 4.10 displays the categories in which results of statistical analysis can be grouped based on the determined R^2 values. The categories are numbered 1-6, which corresponds to ranges of R^2 intervals between 1 and zero. The interval ranges in turn correspond to a 'very strong to perfect relationship' to 'no linear relationship' at zero.

The Wits-Ehac liability index is the dependent variable, and the XRF, proximate analysis and ultimate analysis are the independent variables in the analysis.

4.2.1 Statistical analyses of results: Linear regression with XRF data

The XRF results for the carbonaceous shale unit as a whole were discussed in Section 4.1, in some detail. In Table 4.11, the XRF data is presented in comparison with the measured Wits-Ehac Index.

Table 4.11: XRF results of samples corresponding with the Wits-Ehac Index (WEI)

SAMPLE ID	Fe%	SiO_2%	Al_2O_3%	K_2O%	P%	S%	WEI
SAMPLE0011	1.34	63.51	21.89	1.95	0.014	0.214	5.58
SAMPLE0016	0.71	65.79	20.61	1.59	0.011	0.210	6.25
SAMPLE0017	1.20	64.36	21.95	1.77	0.012	0.584	5.59
SAMPLE0019	1.23	66.46	20.31	1.65	0.012	0.490	6.05
SAMPLE0022	1.67	60.47	24.20	2.25	0.014	0.038	4.95
SAMPLE0024	1.19	62.41	22.88	1.98	0.013	0.205	5.33
SAMPLE0025	0.70	73.89	15.90	1.16	0.010	0.264	5.31
SAMPLE0026	0.96	64.53	21.59	1.66	0.012	0.539	4.34
SAMPLE0027	1.62	64.12	20.93	2.02	0.013	0.110	3.89
SAMPLE0028	3.43	63.78	18.24	1.78	0.011	0.424	4.75

At Iron Ore Mine A, the Fe%, Si%, Al_2O_3%, K_2O%, P% and S% are the common analytes used to classify the quality of the iron ore deposit. Therefore, this suite of analyses is commonly understood by mine personnel and was used to investigate a link with the Wits-Ehac Index. If a proxy could be picked up between any of these analytes with the potential for spontaneous combustion, it would be useful to manage the risk going forward.

The Fe% values fall within 0-3%, SiO_2 above 60%, Al_2O_3% above 15%, K_2O% below 2.5%, P% ranging from 0.010 to 0.014% and S% values are low, all below 1% limit defined as a risk in the AEISG (2017) COP. The analysis falls within the same ranges as reported for the carbonaceous shale unit as a whole in Table 4.3, just with an even lower Fe% and S%. The highest S% values are for samples SAMPLE0017 and SAMPLE0026, and these samples fall within the high and medium range of liability respectively, based on their Wits-Ehac Index result. When looking at Fe%, the highest sample is SAMPLE0028 which has an outlying Fe% greater than 3%, compared to the rest of the spread of Fe% which is lower than 1.7%. The Wits-Ehac Index for this outlying sample is 4.75, falling within the medium risk range for spontaneous combustion.

To aid in this correlation determination between the chemical analytes and Wits-Ehac Index, linear regression was conducted for XRF with the Wits-Ehac Index, depicted in Figure 4.3.

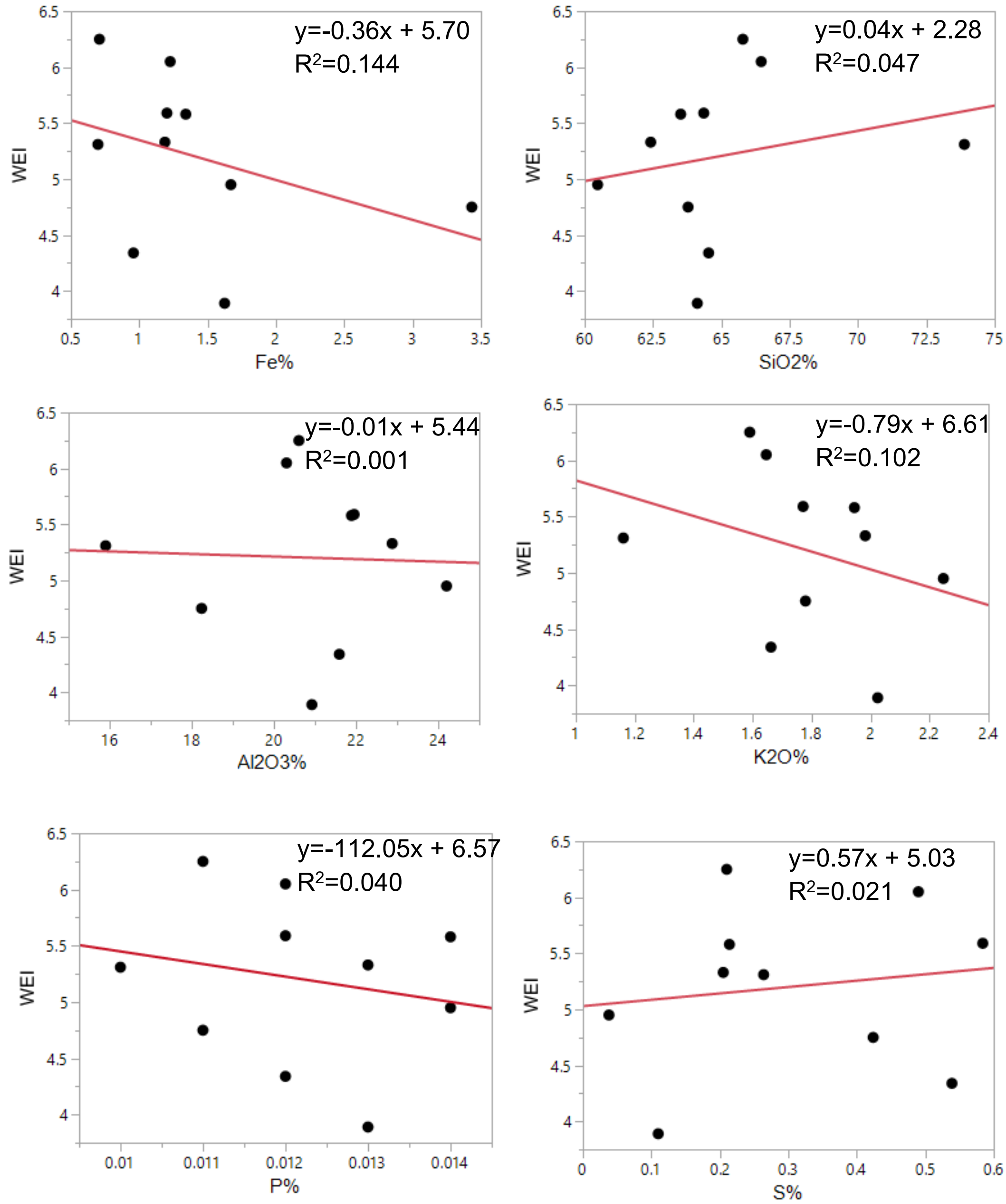

Figure 4.3: XRF analyses correlated with Wits-Ehac Index

Based on Figure 4.3, the Fe%, K_2O%, and P% show a weak negative relationship and the S% show a weak positive relationship when using the categories set out in Table 4.10 according to R^2 values. The SiO_2% and

Al_2O_3% have a very weak positive and negative relationship respectively. Even though the correlation is weak, the strongest correlation is with the Fe and K_2O since their R^2 values are the highest. The correlation between Fe and the Wits Ehac Index might make sense when the Fe is from pyrite which is known to increase spontaneous combustion risk.

4.2.2 Statistical analyses of results: Linear regression with Proximate and Ultimate results

The proximate and ultimate analyses determine the intrinsic and chemical properties of the Iron Ore Mine A shale and are usually done on coal and coal-related rocks. The analyses were done on 9 out of the 10 samples sent for Wits-Ehac Index classification. Sample SAMPLE0016 was sent for CSS and not proximate and ultimate analyses. Therefore, the sample is excluded from the linear regression analyses, but for reference, the CSS values for C and S are included in Table 4.12.

Table 4.12: Ultimate, proximate and spontaneous combustion test results for samples of carbonaceous shale from the iron ore mine

	M	**VM**	**A**	**C**	**H**	**N**	**TS**	**O**	**WEI**
SAMPLE ID	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	
SAMPLE0011	0.91	7.3	91.8	0.03	0.89	0.10	0.32	6.88	5.58
SAMPLE0016	na	na	na	0.53*	na	na	0.25*	na	6.25
SAMPLE0017	0.75	7.4	91.9	0.01	0.88	0.09	0.58	6.54	5.59
SAMPLE0019	0.74	6.7	92.5	0.01	0.81	0.09	0.58	6.01	6.05
SAMPLE0022	0.94	7.2	91.9	0.06	0.96	0.07	0.40	6.61	4.95
SAMPLE0024	0.78	7.4	91.8	0.01	0.9	0.08	0.31	6.91	5.33
SAMPLE0025	0.43	5.3	94.1	0.01	0.66	0.06	0.39	4.78	5.31
SAMPLE0026	0.91	7.0	92	0.00	0.79	0.05	0.45	6.71	4.34
SAMPLE0027	0.71	6.6	92.5	0.01	0.82	0.05	0.12	6.5	3.89
SAMPLE0028	0.91	6.6	92.4	0.00	0.79	0.05	0.41	6.35	4.75

* values are from CSS analysis, since no proximate and ultimate analysis result existed for the sample, this sample was excluded from regression analysis.

The volatile matter is below 10% for the carbonaceous shale samples listed in Table 4.12., whilst the ash content is greater than 90%. The high ash content indicates that the Iron Ore Mine A carbonaceous shale has high mineral (inorganic) matter, and low organic matter (Falcon & Ham, 1988).

The volatile matter content is indicative of the organic matter that determines the rank of coal. At a low rank, as is the case for Iron Ore Mine A carbonaceous shale, the volatile matter content is indicative of the maceral (organic) composition (Snyman, 1989). The total carbon results are very low, none above 0.5%. The Wits-Ehac Index results for the Iron Ore Mine A carbonaceous shale are that the unit has a medium to high propensity for spontaneous combustion. The sample SAMPLE0016 has the highest Wits-Ehac Index, no proximate or ultimate analysis is available, but when comparing the C content, it is significantly higher at 0.5% compared to the 0.01-0.06% C range for the rest of the samples. The samples have medium to high spontaneous combustion propensity, with higher ash content, and lower moisture, volatile matter and sulphide content. This is in line with results for Witbank mines' coal-shales where higher ash correlates to higher Wits-Ehac Index results (Onifade & Genc, 2018c).

The relationship between ultimate and proximate results is investigated further by linear regression analysis to correlate the results with the Wits-Ehac Index results shown in Figure 4.4. The correlation is compared to results previously reported for the Witbank coal-shales (Onifade & Genc, 2018a).

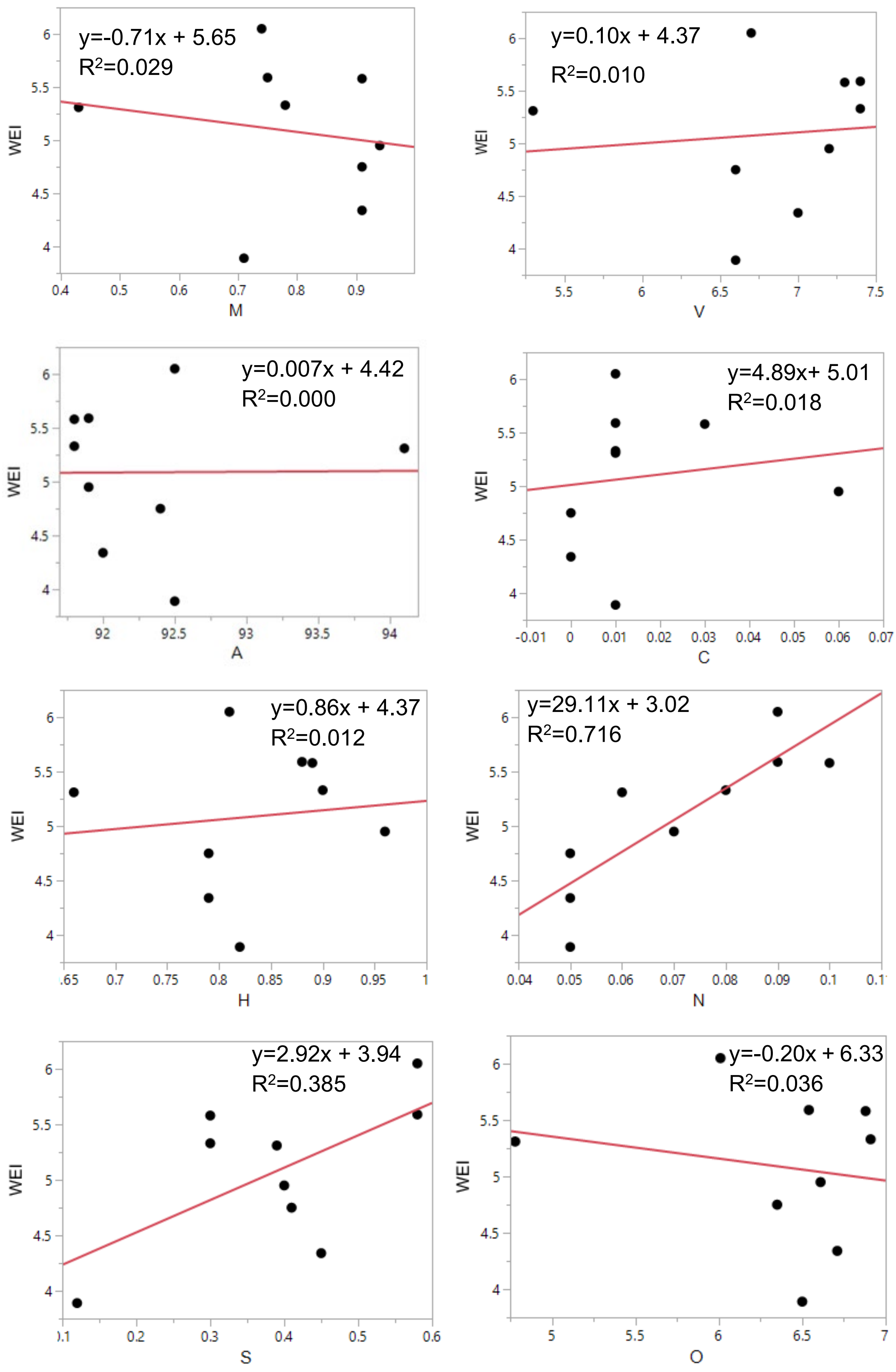

Figure 4.4: Proximate and Ultimate analyses correlated with Wits-Ehac Index

Based on the R-squared values in Figure 4.4, the strongest linear relationship is between Nitrogen and Wits-Ehac Index which indicates a strong positive linear relationship. Total sulphur has a moderate positive relationship, whilst the rest of the analysis (volatile matter, hydrogen, oxygen, moisture, total carbon and ash) have very weak relationships with Wits-Ehac Index. Nitrogen occurs in the organic carbonaceous material itself (Kim & Cheiken, 1990), none of the minerals in the XRD analysis contains nitrogen. This may prove a relationship exists between the carbonaceous material content (albeit low carbonaceous content) and the propensity for spontaneous combustion.

Table 4.13 contains the categorical results of the Witbank coal-shales as reported by Onifade & Genc (2018a), along with the Iron Ore Mine A categorical results from this study.

Table 4.13: Categorical comparison between the iron ore mine carbonaceous shale and the reported Witbank Mines coal-shale

Category	R2 value range	Relationship	Iron Ore Mine A Shale Ultimate and Proximate Result	Witbank Mines' Coal-shale Ultimate and Proximate Result
1	0.95 to 1 or -0.95 to -1	Very strong to perfect positive or negative relationship	-	-
2	0.51 to 0.94 or -0.51 to -0.94	A strong positive or negative linear relationship	N	-
3	0.25 to 0.50 or -0.25 to -0.50	A moderate positive or negative linear relationship	S	V, A, C, H, N
4	0.1 to 0.24 or -0.1 to -0.24	A weak positive or negative linear relationship	-	-
5	<0.1, not 0	Very weak positive or negative relationship	M, V, C, H, O	M, O, S
6	0	No linear relationship	A	-

The categorical result of the iron ore mine carbonaceous shale is different to that of the Witbank mines' coal-shales. No proximate or ultimate result reports to Category 1 or Category 4 for either the Witbank coalfields or the Iron Ore Mine A. The only analytes of both mines reporting to the same category (Category 5) are moisture and oxygen. Sulphur and nitrogen are the chemical constituents with the strongest relationship to the Wits-Ehac results of Iron Ore Mine A, where the volatile matter, ash, carbon, hydrogen and nitrogen analytes have the strongest relationship for the Witbank shale. Therefore, nitrogen is the only common analyte with a relatively strong relationship with the Wits-Ehac Index for both the Witbank coalfields' shale and the Iron Ore Mine A shale. Sulphur does not have a common relationship with Wits-Ehac Index between the two sites. Contrasting the Witbank shale results, the ash content of Iron Ore Mine A has no relationship with the Wits-Ehac Index.

4.2.3 XRD results

The XRD results for the Wits-Ehac samples are presented in Figure 4.5.

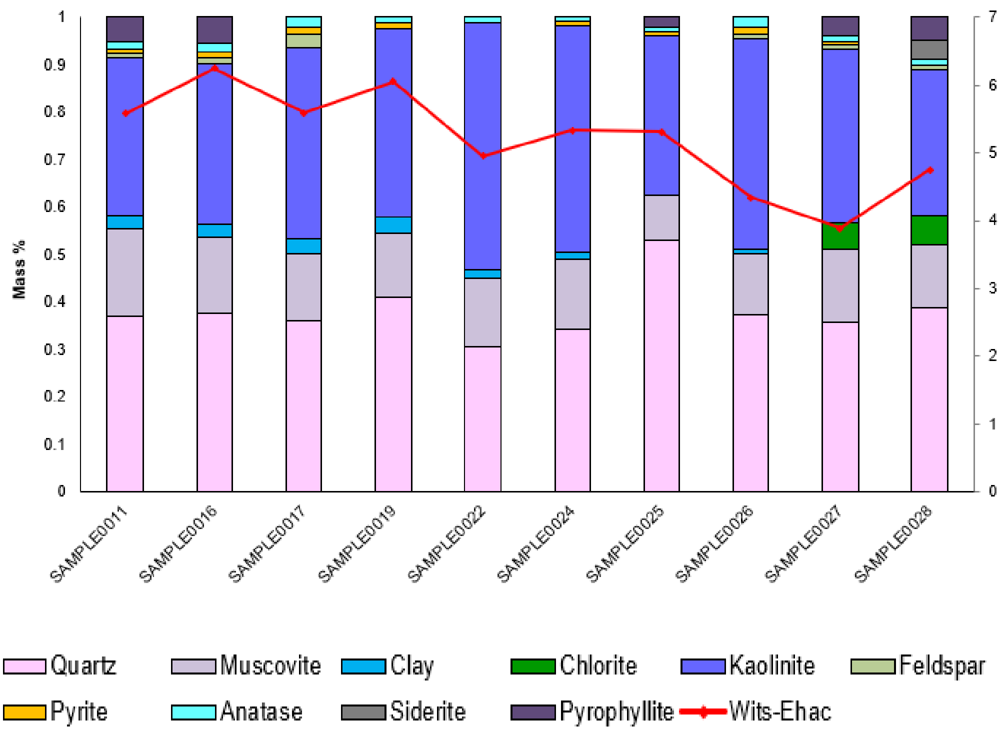

Figure 4.5: XRD results for Iron Ore Mine A carbonaceous shale samples presented with the Wits-Ehac Index result per sample

The major minerals present are quartz and kaolinite, in line with the XRD results for the whole of the carbonaceous shale as classified in Section 4.1. The siderite (iron carbonate) is present in lesser amounts, and the relative abundance of it in SAMPLE0028 might contribute to the samples' relative lower Wits-Ehac Index, due to carbonate's inhibiting (buffering) capacity (Descotes, et al., 2002). The pyrite content is the highest in SAMPLE0026, but this sample has the second lowest Wits-Ehac classification. Pyrite is present in almost all of the samples, ranging from the highest to the lowest Wits-Ehac Index, therefore no direct relationship is apparent to link the spontaneous combustion risk with the presence of pyrite. The mineral composition percentages are tabled in Appendix I. The Witbank coal-shale results studied in Onifade, et al. (2019) suggest that relative higher pyrite and muscovite has a higher risk for spontaneous combustion, and higher kaolinite, quartz, plagioclase (feldspar mineral) and siderite has a lower proneness for spontaneous combustion. Apart from the presence of pyrite and siderite, the mineralogical composition of the Iron Ore Mine A carbonaceous shale does not exhibit the same trend. The high Wits-Ehac Index samples (SAMPLE0011, SAMPLE0016, SAMPLE0019, SAMPLE0024, SAMPLE0025) does not have any significant correlation with a specific mineral, the medium liability samples (WEI<5 and >3) has a higher siderite content, and high liability samples (WEI>5) has a higher pyrite content.

4.3 Spatial spontaneous combustion risk

The samples submitted for Wits-Ehac Index are spatially distributed along the different boreholes that were successfully drilled. The intersections sampled are considered indicative of the height of the 10 m mining bench, as well as any mining block intersecting the borehole. Samples with above-average pyrite content from sample results in this study and samples classified according to the Wits-Ehac Index are summarised in Table 4.14.

Table 4.14: Wits-Ehac Index and pyrite mineralogy per interval and bench.

SAMPLE ID	HOLEID	FROM	TO	BENCH	WEI	Pyrite
SAMPLE0027	HN124_M	1.50	2.12	B	Medium	1%
SAMPLE0011	HN124_M	6.85	7.25	B	High	1%
SAMPLE0009B	HN124_M	16.38	16.72	C	na	7%
SAMPLE0028	HN124_M	16.72	17.31	C	Medium	Tr
SAMPLE0010B	HN124_M	18.48	18.83	C	na	8%
SAMPLE0025	HN612_M	4.00	4.54	A	High	1%
SAMPLE0016	HN612_M	14.50	14.94	B	High	1%
SAMPLE0026	HN612_M	19.00	19.55	B	Medium	2%
SAMPLE0017	HN621_M	0.55	1.05	A	High	1%
SAMPLE0019	HN621_M	4.00	4.30	A	High	1%
SAMPLE0022	HN621_M	19.72	20.17	B	Medium	1%
SAMPLE0023B	HN621_M	29.17	29.60	C	na	4%
SAMPLE0024	HN621_M	31.84	32.19	C	High	1%

Note 1: (na- no analyses available, Tr-trace amount reported)

Table 4.14 presents the Wits-Ehac Index liability for spontaneous combustion risk per sample interval. The sample intervals are then related to the applicable bench in Iron Ore Mine A, depending on the hole's starting elevation. In Figure 4.6, spatial classification per bench is given by colourcoding according to the liability index determined by Wits-Ehac, as well as considering if there was pyrite (from XRD results) present in the carbonaceous shale at Iron Ore Mine A. Where sample results are unavailable, or the area has been mined out the classification is considered undefined. When more than one sample result is present per bench the worst-case classification is plotted in Figure 4.6.

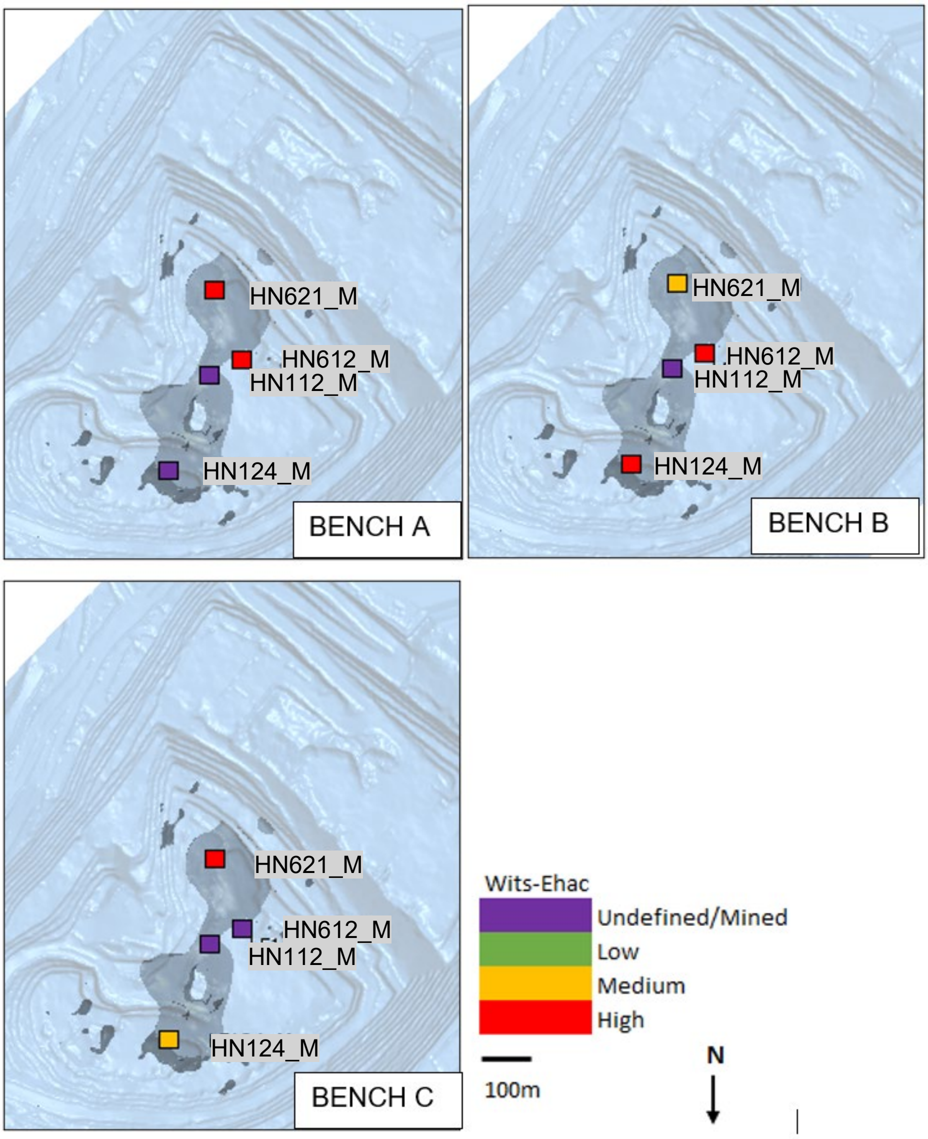

Figure 4.6: Spatial distribution of spontaneous combustion liability from data presented in Table 4.14.

From Figure 4.6, there is no spatial trend of the Wits-Ehac Index noticeable within the pit. The potential for constraining the risk area further within the carbonaceous wireframe itself is apparent and highlights the need for additional drilling and sampling to constrain the liable area further, not only in the horizontal orientation but at depth too. Due to sparse and variable data between the different drillholes' extrapolation of the Wits-Ehac Index was not possible.

4.4 Chapter summary

Chapter 4 contains the results of the XRF, CSS, XRD, proximate, ultimate and Wits-Ehac analyses on the Iron Ore Mine A carbonaceous shale samples. In Section 4.1 the results aim to characterise the shale unit as a whole through the summary statistics provided. In Section 4.2 the results presented are limited to the samples for which Wits-Ehac Index results are available. The results of the Wits-Ehac Index are statistically correlated with XRF and proximate and ultimate analysis to see if there is a strong correlation possible to aid in future determination of spontaneous combustion propensity. The proximate and ultimate linear regression results of the Iron Ore Mine A samples are compared to that of the Witbank Mines' coal-shale results in table format. The mineralogical composition from XRD analysis is presented with the Wits-Ehac Index results in this chapter to determine any apparent trends. A spatial interpretation of the risk for spontaneous combustion is derived from the Wits-Ehac Index results to be used in defining risk levels per mining level and area in the mine to aid drill and blast activities' risk management.

5 CONCLUSIONS, RECOMMENDATIONS AND FUTURE RESEARCH

Understanding the properties of the carbonaceous shale at Iron Ore Mine A is of importance to the propensity for spontaneous combustion, and the potential for ground reactivity. The risk of spontaneous combustion and ground reactivity influences drill and blast activities. The high potential incident HPI that occurred at Iron Ore Mine A led to the study to define the presence of the carbonaceous shale, as well as to characterise the unit according to its chemical and intrinsic factors. This was done by test work on samples from the carbonaceous shale unit.

5.1 Conclusions

The chemical composition of the carbonaceous shale unit itself at Iron Ore Mine A has not been specifically determined in any literature, and this has now been concluded in this research. The average chemical composition of the Iron Ore Mine A compares well with the general composition of inert to low-reactive shales of the Mount McRae unit, as well as low liability Witbank coal-shale results. In general, the Iron Ore Mine A carbonaceous shale has never been analysed by proximate and ultimate analyses and the mean results in Table 4.7 and Table 4.8 can be used in future investigations should questions arise on the drill and blast risk assessment process for reactive ground at Iron Ore Mine A.

Based on the proximate and ultimate analyses, the carbonaceous shale in question falls on the limit between being classified as a 'rock' as opposed to a carbonaceous shale. The implication of this result brings to question the probability of a spontaneous combustion reaction taking place at Iron Ore Mine A. The results revealed that the carbonaceous shale of the Iron Ore Mine A is characterised by a low Fe presence, except when siderite is present. Carbon content in all the samples was low, typically around 1% or less. Sulphur content is variable and where it is elevated, it is ascribed to the presence of pyrite. Pyrite content is localised and not present consistently throughout the shale, therefore visual identification and chemical analysis throughout the carbonaceous shale are of importance to highlight areas of risk within the carbonaceous shale unit. In terms of the intrinsic properties of the Iron Ore Mine A carbonaceous shale, the shale is classified as a 'poor' carbonaceous shale, at best, and at the least, it can be classified as a 'rock', due to low carbon content, low calorific value and high ash content. According to the classification of Wagner (2021), the Iron Ore Mine A carbonaceous shale can be referred to as 'shale' and not 'carbonaceous shale' due it having less than 10% carbonaceous matter. The potential for self-heating of the Iron Ore Mine A carbonaceous shale exists based on its carbon content, but the sulphide content is insufficient for spontaneous combustion (Beamish & Theiler, 2016) to take place without an external catalyst.

Upon investigating the intrinsic properties of the carbonaceous shale with the propensity for spontaneous combustion, it is seen that with respect to the XRF results, all analytes have a weak to poor correlation with the strongest relationship being with Fe. Concerning the proximate and ultimate analysis results, the strongest relationship is with nitrogen, and the second strongest relationship is with sulphur. Sulphur has a moderate linear relationship with the Wits-Ehac Index and may be indicative of a risk for spontaneous combustion in areas where the sulphur content in the carbonaceous shale is elevated. The risk for spontaneous combustion according to the Wits-Ehac Index is on medium to high risk with no spatial trends. No correlation between the Wits-Ehac Index and the presence of pyrite could be made. However, a negative correlation is observed between the presence of siderite and the Wits-Ehac Index analysis results.

In comparison with the Witbank coal-shales, the Iron Ore Mine A carbonaceous shale has little to no common trends with chemistry or spontaneous combustion propensity. The only common relative high correlation with the Wits-Ehac Index exists with nitrogen. The validity of the Wits-Ehac test as a measure of spontaneous combustion risk in Iron Ore Mine A carbonaceous shale may be low due to the inconsistency between the response variables in this study compared to the Witbank coal-shales (as tabled in Table 4.13). The reason for the inconsistency may be the inherent differences in composition and genesis of the lithologies between the two localities. It might also indicate that the incident occurred due to the oxidation reaction of pyrite with the ammonium nitrate (reactive ground) and not due to the spontaneous combustion of carbonaceous shale. The presence of siderite in the Iron Ore carbonaceous shale may have a buffering reaction to self-heating propensity, and the shale should not be liable to spontaneously combust without external catalysts.

The objective of the study was to highlight the risk level of spontaneous combustion according to the properties of the carbonaceous shale found in the iron ore mine. From this study, the sulphur presence can be considered a proxy for an increased spontaneous combustion risk, as well as highlighting prospective reactive ground. The presence of elevated Fe in the carbonaceous shale, may indicate a lower risk for spontaneous combustion, until further test work can either prove or contradict this relationship.

5.2 Recommendations

Routine sampling of waste is not part of the normal procedure at Iron Ore Mine A, and it is recommended that intermittent sampling campaigns are undertaken to better classify the carbonaceous shale regarding its sulphur and carbonaceous content. The increased sampling to determine the carbonaceous content will determine if the lithology as a whole can be classified as a shale as opposed to a carbonaceous shale. XRF analysis is standard for all samples taken at the iron ore mine and it is recommended that samples within the carbonaceous shales are flagged as higher risk when sulphur is greater than 3%.

The application of the Wits-Ehac classification index has been successful for samples from the Witbank mines' coal-shales. However, future comparative studies are recommended to test the suitability of it to determine the risk of spontaneous combustion in the Iron Ore Mine A environment.

The awareness regarding carbonaceous shale presence in mining blocks has become standard procedure with regard to monitoring borehole temperatures due to the risk of spontaneous combustion. The focus should however also be placed on the reactive ground risk from the presence of pyrite oxidising, and hot ground conditions that may act as a catalyst for reactivity of ammonium nitrate in drilled holes charged with explosives. It is recommended that the mine's drill and blast risk assessment is reviewed intermittently along with the results of carbonaceous shale sampling campaigns, focussing especially on the reactive ground risk management.

5.3 Future research work

Future research work can include the intermittent sampling of the carbonaceous shale for spontaneous combustion risk via the Wits-Ehac test to further test its applicability in the iron ore environment. Other methods of determining spontaneous combustion risk such as TGA can be investigated. To aid in the analysis, it will be important to conduct petrographic analyses on samples sent for Wits-Ehac to test how specific maceral composition correlates, and to determine what macerals are present in the Iron Ore Mine A carbonaceous shale.

The relationship between nitrogen and Wits-Ehac Index found in this research report, was difficult to describe or investigate further since petrography to determine the maceral composition, did not form part of this research. The nitrogen most likely occurs in the organic matter compound and petrography is the best way to investigate the physical maceral composition of the coal (Snyman, 1989; Mayes & Myers, 2014). Future work to include petrography on the carbonaceous shale is therefore suggested.

Additional research work may also be recommended in the coal industry to investigate the role that carbon plays versus the role of sulphur in spontaneous combustion incidents as well as ground reactivity. Learnings from the coal industry may be applied in the Iron Ore Mine A environment with respect to its carbonaceous shale presence. It will be interesting to determine if spontaneous combustion can occur without the presence of sulphur and vice versa. The importance of the presence of pyrite is not apparent in this study, and the presence of siderite in samples with relatively low spontaneous combustion risk might be an interesting avenue to investigate further.

The Han et al. (2005) study at an iron ore mine that experienced ground reactivity in its carbonaceous shale, successfully correlated the shale pH with total sulphur. Analysing the geochemical attributes for the Iron Ore Mine A carbonaceous shale in a similar fashion might be useful as a reactive ground assessment for Iron Ore Mine A.

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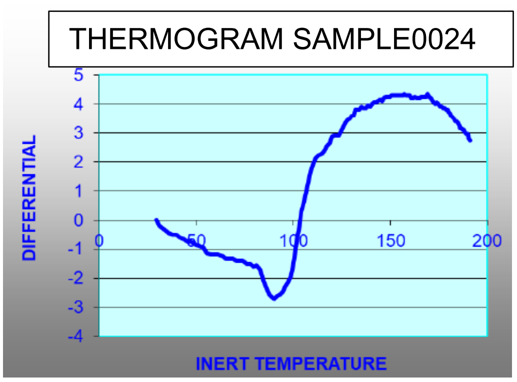
7 APPENDICES

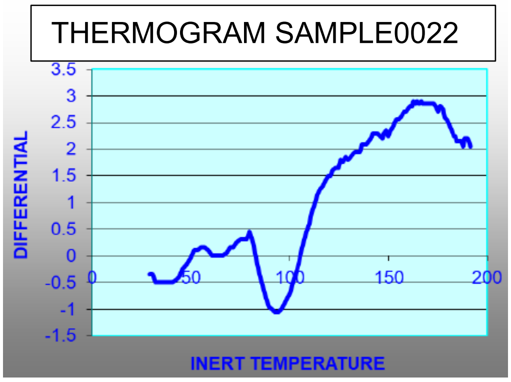
7.1 APPENDIX I

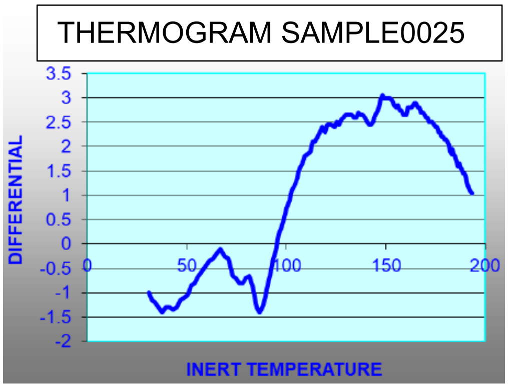
	Mineral Composition (%)											
Sample ID	Quartz	Muscovite	Clay	Chlorite	Kaolinite	Albite	Pyrite	Anatase	Siderite	Pyrophyllite	Feldspar	Total
SAMPLE0001B	56	4	3	-	25	-	2	1	-	9	0	100
SAMPLE0002B	50	6	4	-	31	-	1	1	-	7	0	100
SAMPLE0003B	38	8	4	-	38	-	1	2	-	8	0	100
SAMPLE0004B	43	8	4	-	39	-	1	2	-	4	0	100
SAMPLE0005B	39	9	4	-	43	-	1	2	-	4	0	100
SAMPLE0001C	56	6	2	-	30	-	3	1	-	2	0	100
SAMPLE0002C	56	6	4	-	30	-	3	1	-	1	0	100
SAMPLE0006B	40	7	3	-	37	-	3	2	-	9	0	100
SAMPLE0007B	40	5	2	-	39	-	2	1	3	8	0	100
SAMPLE0008B	74	2	2	3	10	3	Tr	-	5	-	0	100
SAMPLE0009B	29	2	9	-	31	-	8	-	22	-	0	100
SAMPLE0010B	12	1	6	2	21	-	7	-	51	-	0	100
SAMPLE0013B	33	7	-	-	47	-	1	-	12	-	0	100
SAMPLE0014B	10	-	-	-	7	-	Tr	-	82	-	0	100
SAMPLE0018B	39	11	-	2	41	5	2	-	-	-	0	100
SAMPLE0020B	13	-	-	5	17	-	2	-	63	-	0	100
SAMPLE0021B	29	11	17	-	43	-	1	-	-	-	0	100
SAMPLE0023B	19	6	-	-	27	-	4	-	44	-	0	100
SAMPLE0011	37	18	3	-	33	0	1	2	-	5	1	99
SAMPLE0016	38	16	2	-	34	0	1	2	-	5	1	99
SAMPLE0017	36	14	3	-	40	0	1	2	-	-	3	97
SAMPLE0019	41	13	3	-	40	0	1	1	-	-	-	100
SAMPLE0022	31	14	2	-	52	0	-	1	-	-	-	100
SAMPLE0024	34	15	1	-	48	0	1	1	-	-	-	100
SAMPLE0025	53	10	-	-	34	0	1	1	-	2	-	100
SAMPLE0026	37	13	1	-	44	0	2	2	-	-	1	99
SAMPLE0027	36	15	-	6	36	0	1	1	-	4	1	99
SAMPLE0028	39	13	-	6	30	0	-	1	4	5	1	99

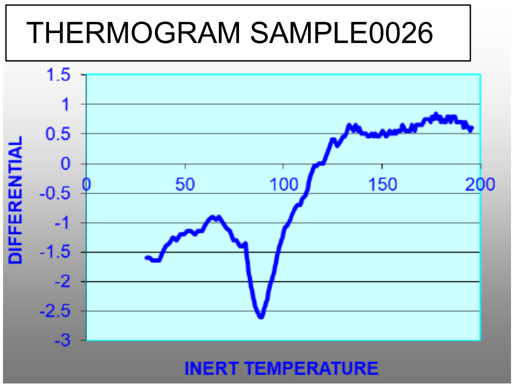
Mineral:	**Approximate chemical formula:**
Quartz	SiO2
Muscovite	KAl2(Si3Al)O10(OH,F)2
Clay	(K,H3O)(Al,Mg,Fe)2(Si,Al)4O10[(OH)2,(H2O)]
Chlorite	(Mg,Fe)3(Si,Al)4O10(OH)2·(Mg,Fe)3(OH)6
Kaolinite	Al2Si2O5(OH)4
Albite	NaAlSi3O8
Pyrite	FeS2
Anatase	TiO2
Siderite	FeCO3
Pyrophyllite	Al2Si4O10(OH)2
Feldspar	CaAl2Si2O8

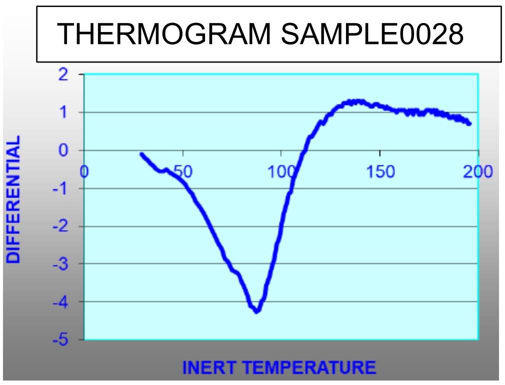
7.2 APPENDIX II

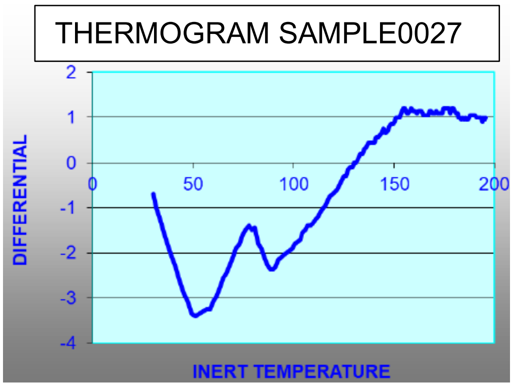


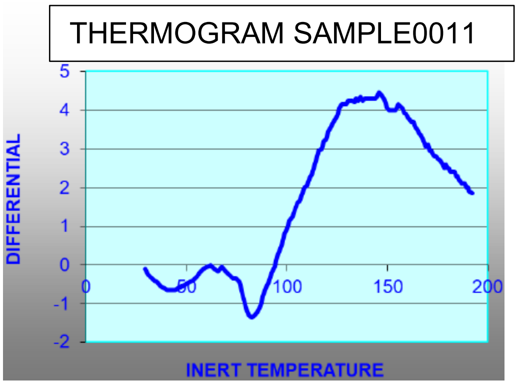








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-2
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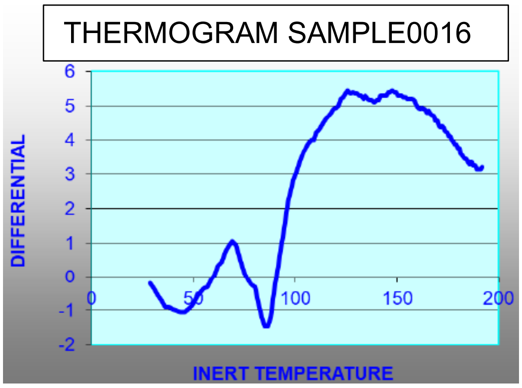
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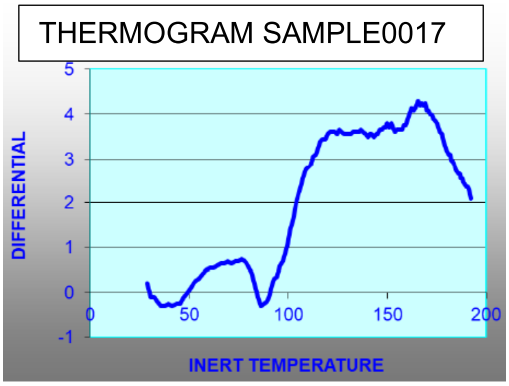
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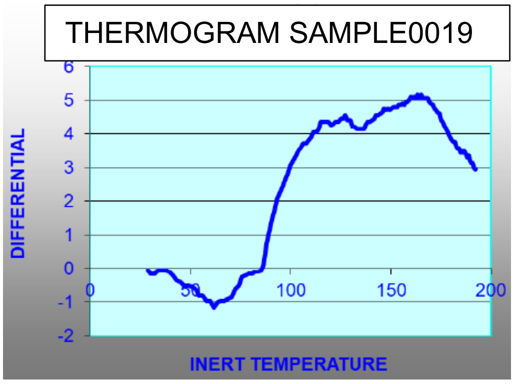
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