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INVESTIGATION INTO THE BENEFICIATION POTENTIAL OF A LOW-GRADE DISCARD COAL FROM THE WITBANK COALFIELD

MSc (50/50) RESEARCH REPORT

Prepared by

Sancho Nkosilathi Nyoni

743158

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School of Chemical and Metallurgical Engineering, Faculty of Engineering and the Built Environment, University of the Witwatersrand, Johannesburg, South Africa

Supervisor: Dr M. M. Bwalya

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DECLARATION

I, Sancho Nkosilathi Nyoni, hereby declare that this research report contains my unaided work. It is being submitted to the University of the Witwatersrand in partial fulfilment of the requirements for the Degree of Master of Science in Engineering. This work has never been submitted before to any other university for any degree or examination.

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Signature of Candidate

/ / 2019

ABSTRACT

The South African Department of Energy has projected that the usable coal reserves will have depleted within 100 years. This research, in response to the problem, investigated the possibility of expanding the coal resource through beneficiation of low-grade discard coal from the Witbank Coalfield. Amenability to beneficiation was investigated using various analytical tools and through sink-float testing, and flotation separation techniques. Sink-float testing showed that the coal is highly difficult to wash through most gravity methods but indicated that it was amenable to separation using dense medium cyclone (DMC) equipment. For a -1.18 to 4.5mm feed size (38.9% Ash), the test showed that at 1.7 SG, a 45.2% yield would be obtained with a minimum grade of 25.8% Ash. Flotation of the low-grade discard coal, milled to a feed size of -300µm (52.6% Ash), for a period of 1 min using diesel collector at optimum dosage of 14g/kg Coal produced a 51% yield of grade 33.6% Ash, and a clean coal recovery, $r_c = 65.21\%$. Using the Whitten model to estimate partitioning in a DMC, based on data from the sink-float test, it was predicted that when operating at a cut-point of 1.7 SG, the same 51% yield would have a higher grade (27.9% Ash) but a closely-related clean coal recovery, $r_c = 63.74\%$. The DMC model approach thus helped link analytical work in sink-float testing with real performance in a typical DMC when processing the coal. Based on the conditions set in this research, DMC and flotation equipment have shown a comparable capability to upgrade the coal within the respective size classes suited for each technique. An integrated process utilising both separation equipment at appropriate size classes is recommended.

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Special thanks go to my supervisor, Dr Murray Bwalya for his invaluable contributions throughout the research process. Just as importantly, I would like to commend the School of Chemical and Metallurgical Engineering for availing resources timeously to back the research effort and the technical staff for training and general support given during the practical stages of this research. Finally, I wish to express gratitude to Prof. Nicola Wagner for the assistance rendered in petrographic analysis.

DEDICATION

Sincere gratitude goes to my parents, Thabani and Pauline Nyoni, my adorable siblings and extended family. The love that binds us together is my single greatest motivation!

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1
INTRODUCTION

1.1 Background

The term beneficiation, as applied to coal, refers to the upgrading of the coal through the separation of coal macerals from the associated gangue minerals. The dominant coal upgrade practice in South Africa is dense medium cyclone (DMC) separation, a hydraulic or wet beneficiation technology, which uses the density difference between coal and mineral matter to effect separation. Other wet technologies utilised, though to a lesser extent, are jigging, and flotation. Dry beneficiation techniques are largely still under research thus are yet to gain significance in the local industry.

Local research into dry beneficiation has mainly been in magnetic separation and electrostatic separation and to a lesser extent in air dense medium fluidised bed separation. Magnetic separation exploits the difference in magnetism between valuable and gangue particles to effect separation. Electrostatic separation, on the other hand, achieves separation as the coal passes through an electric field; the ash forming minerals, which are usually negatively charged are deflected by the electric field while the coal particles that are slightly positive to neutral proceed without being diverted by the electric field (Bada, Falcon, & Falcon, 2010). Air dense medium fluidised bed separation applies similar principles to hydraulic dense medium separation using a pseudo-dense medium that essentially is a two-phase gas-solid fluid created through the suspension of solid particles in an upward direction using air.

The first step in any beneficiation circuit is size reduction or comminution. Following that the washing characteristics of the coal are investigated by performing density-based separation tests in the laboratory on a sample of coal that is representative of the source using a series of

density baths prepared from organic liquids, aqueous solutions or suspensions according to (SANS 7936, 2010) specifications. Float-sink testing results help determine the optimum size and density for the removal of loosely associated gangue and free dirt. Over time this test has gained recognition as the first basis upon which dense medium separation plants are designed.

For finer coal which cannot be handled by DMS, flotation is increasingly becoming a more attractive alternative due to its inherent capacity to handle small particle sizes, 50-400µm. Low-grade coal beneficiation potential is theoretically augmented by flotation due to the fine grind size requirement which enhances the physical liberation of the coal from its association of coal with ash-bearing minerals, hence separable in flotation medium. Flotation is a hydraulic, physicochemical method that exploits the unique surface properties of the coal to selectively produce a float concentrate that has a low ash content through the separation of the macerals from gangue material when suspended in liquid by adhering to gas bubbles thus gaining selective levitation of the desired solid (Speight, 2015).

Another physicochemical method called oil agglomeration has emerged in recent decades. The principle behind the technique is the formation of an oil coating on the hydrophobic surfaces of coal particles followed by the agglomeration of these particles to form larger particles of significantly reduced density. In terms of separation efficiency with low-grade coal, oil agglomeration is said to be better, producing a product of low ash content and high combustibles recovery(Xia, Xie, & Peng, 2015). This technique becomes more valuable when the coal liberation size tends towards the ultra-fine range. The merit of oil agglomeration is in the beneficiation of ultra-fine, high ash, poor washability coals, and middlings which do not respond satisfactorily to conventional processes. Such tests have been done at bench-scale using coals from problematic seams, washeries. However, the technology is mainly still under research and is yet to gain industry recognition.

Coals of different types, rank, and grade behave differently in any processing and utilisation operation of which flotation is no exception. Therefore, coals from within the same region or from different regions are likely to exhibit different liberation and flotation characteristics. This phenomenon is often studied across coal rank differences, low and high, to determine the optimum separation performance. Free State Coalfields generally have lower-rank coal than Witbank coals, and KwaZulu-Natal (KZN) coals generally have a higher rank (Jeffrey, 2005). Release analysis is used to obtain information useful in kinetics studies and which serves as a key guide in making flotation-based processing decisions (Blondin, Girardeau, & Nomine, 1988).

To develop and maintain an efficient beneficiation process, it is important that characterisation of the coal deposit be done periodically from as early as the planning stage. Such information is useful in determining the processing route for the coal resource, plant design and process optimisation strategies. Surface texture analysis through the study of microlithoypes is one of the most efficient liberation analysis tools, that gives valuable processing insight as the sedimented or layered nature of the coal and its association with mineral inclusions, both syngenetic and epigenetic is better understood (Wagner, Malumbazo, & Falcon, 2018). Additional physical, petrographic, mineralogical and chemical analysis is also essential to gain a more thorough understanding of a coal sample.

Novel and improved approaches to the coal conundrum are continually being developed, necessitated by the impending energy crisis. Research bodies such as the DST/NRF funded SARChl Chair of Clean Coal Technology have been set up with governmental support and with the support of private entities like the Fossil Fuel Foundation to continue to address issues that span the entire coal continuum from coal characterisation to processing and utilisation with the addition of carbon dioxide capture and sequestration. This is evidence of the enduring

dependence on coal in the economy despite the emergence of new alternative energy production technologies.

1.2 Problem Statement and Motivation

Coal continues to be an important energy resource despite attempts to switch to other cleaner resources. The demand shows no prospect of lowering in the coming decades particularly in South Africa and other developing countries. Such is the case in the face of alarmingly diminishing marketable coal resources. Some resource monitoring agencies, governmental and international, claim that it is improbable for the available local reserve to last beyond the century at current depletion rates (Department of Energy, 2019).

In the global energy context, coal is expected to continue to play a major role with reserves expected to last beyond the current century in comparison to natural gas and oil which are forecasted to be exhausted within the next 50-60 years (Chary, Gupta, & Dastidar, 2015). Coal presently powers the South African economy and most developing countries and will continue to dominate the energy sector in upcoming decades. This is due to its availability, the magnitude of infrastructural investment into coal-based technologies and stability in the price.

Among the major challenges facing the coal industry currently is the diminishing available coal reserves due to intensive use of the resource spanning centuries since the first industrial revolution, making lower-grade coal usage inexorable. Technologies consequently must adapt to match this change and face up with increasing socio-political variables governing its use. It is thus imperative to continue to forge ahead both fundamental and applied fields, developing novel and improved methods of characterisation, processing, and utilisation to sustainably satiate both current and future coal needs.

Electricity generation and synthetic fuels production are dominated by coal and the emerging hydrogen economy will also likely utilise coal as the major source of hydrogen (Miller, 2005; Mohr & Evans, 2009). The International Energy Agency, IEA forecasts that by 2035 wind and solar photovoltaic sources will contribute 5.6% and 9.8% each to Africa's total electricity generation(Müller, 2019). Hence, conservation and sustainable energy concerns, though important in the global energy picture, will, in the coming decades continue to play a lesser role in helping meet the energy demands of Africa (Moumakwa O, 2009).

Malumbazo (2016) during a presentation at the 35th International Geological Conference GIFT Workshop submitted that of the 336Mt annual output of mined coal in South Africa, 93.2Mt is consumed in thermal power generation, 42.9Mt is used in synthetic fuels production, 25.0Mt is used in smaller local markets, 78.7Mt is exported and 74.8Mt is non-marketable due to low grade. This low-grade coal can potentially be beneficiated to meet the criterion for some coal markets.

Deposits of acceptable grade are available in Springbok Flats Coalfields in association with uranium deposits which complicates their beneficiation and utilisation (Cairncross, 2001; Jeffrey, 2005). The challenge of beneficiating low-grade coal found in the Witbank No. 4 Seam between seams of high-grade coal presents less of a hazard. This coal is high in ash and moisture, low in volatiles and has a calorific value under 15MJ/kg, hence is categorised as low-grade coal.

Larger low-grade coal deposits also exist in the Molteno-Indwe Seams and the Free State Coalfields (Jeffrey, 2005). In some coalfields, including the Witbank, dolerite included coal has also been found and often rejected to discards during coal preparation. These cumulative discards have over the years created a potentially upgradable resource that is over a 1 000 000 000 metric tons in magnitude (Department of Energy, 2019; Snyman & Botha,

1993). The burden thus lies with the coal processing engineer to design feasible methods or mechanisms through which low grade can be successfully beneficiated. Such a design should be significantly sound both qualitatively and quantitatively to justify its adoption.

By design, only one power station, Lethabo, has the capability to utilise low-grade coal feed. Lethabo has an installed capacity of 3708MW which comprises approximately 8% of Eskom's generating capacity and 7.34% of the total feed to the national grid, inclusive of independent power producer(Jeffrey, 2005) However despite its good tolerance to low-grade coal in comparison with other coal-fired power plants, its lower limit is not sufficiently low to utilise the entire low-rank coal continuum, hence 74.8Mt continue to be discarded per annum.

Considering the increasing energy demand, it is imperative that the capacity for utilisation of low-grade coal is increased. However, decommissioning of utility boilers firing higher grade coal in preference to lower grading coal boilers is not feasible since the country is already experiencing acute power shortages and the cost of installing new boilers systems could be prohibitive. Thus, upgrading low-grade coal would be a viable option.

The challenge or opportunity created by the current drive for sustainable development is that process engineering has evolved and is no longer fixated on high production rates and product quality but is also cognizant of the ecological impact of the process in terms of by-product toxicity, recyclability, and water, power and reagent economy. A review paper by (Xia et al., 2015) on recent advances in beneficiation of low-rank coals indicates that the technologies that are most likely to yield success with high ash material are air dense medium fluidised bed separation, electrostatic separation, flotation, and oil agglomeration.

1.3 Main Objective

This research seeks to investigate the beneficiation potential of low-grade coal in the Witbank Coalfield and to evaluate the techno-economic feasibility of further upgrade of the coal to industrial specifications. This research sought to ascertain whether low-grade run-of-mine (ROM) coal can be beneficiated using conventional coal cleaning technologies particularly to meet power plant boiler feed specifications for South Africa. The key result areas for the study would be the balance between the quantity and grade of the clean coal recovery and, operational cost and complexity. Sink-float analysis was used to determine the separation potential of the coal-based on density. Using a partitioning model, the potential of dense medium cyclone (DMC) usage in cleaning the coal was determined. Flotation was also investigated to determine if there was a need to modify the usual reagent regime used in coal flotation when processing low-grade Witbank coal. Finally, a process flowsheet showing how the low-grade coal can be most effectively beneficiated is proposed.

1.4 Organisation of Research Report

Chapter 1 gives an overview of the coal industry within the South African context, submits the problem statement, aim, research questions, general objectives and research objectives around which this research report was developed. Chapter 2 gives a review of related literature regarding the Witbank Coalfield and other local coalfields. Furthermore, the chapter reviews analytical standards and techniques applied to coal testing and characterisation as well as select beneficiation technologies of interest to this investigation. These technologies are air dense medium fluidised bed separation, the dense medium cyclone, and froth flotation. Chapter 3 provides the experimental methods applied in all testing and characterisation. Where a standard was applied, reference is duly made. Standard procedures were adhered to in float-sink and flotation testing as well as physical, chemical and petrographic analysis of the coal sample.

Chapter 4 then gives data from the experiments conducted and basic analysis results represented in graphical and tabular form. No discussion is made pertaining to the results. Chapter 5 discusses the results and analysis from the preceding chapter and seeks to logically explain the phenomenon experienced during the investigations with the support of the reviewed literature. Finally, Chapter 6 offers a short discussion preluding the conclusion and recommendations and gives suggestions for future work. A list of references and an appendix populated with experimental data from the tests conducted then conclude the document.

CHAPTER 2
LITERATURE REVIEW

2.1 Introduction

Coal is one of the best-studied rock types in geology (Thomas, 2013). It is defined as a composite of the fossilised residue of plant debris or phyterals which have undergone progressive chemical and physical alteration over geological time. Alternatively, it is defined as a brittle, combustible, carbonaceous solid phase material formed through the decomposition and alteration of vegetation by heat, compaction and consolidation under superimposed strata over geologic time(Speight, 2015). Key factors affecting the nature of a coal deposit are plant composition, climate, and depth of burial, sedimentary environment, tectonic control and time. The primary causation of differing rank between coal deposits though are the temperature, load pressure and period of deposition which in combination contribute to the metamorphosis process which results in carbon enrichment, a key indicator of rank change.

Coal has a multi-constituent matrix that exhibits a wide range of chemical and physical properties which are related to its organic or maceral composition and the associated mineral matter. Due to this inherent complexity, it is difficult to correlate composition with behaviour. It is only through an assiduous and careful study using petrographic, mineralogical and chemical analysis tools that a holistic perspective of coal can be achieved and applied to advise beneficiation, utilisation, and legislation.

2.2 South African Coal Deposits

South African coal deposits are situated in a series of basins found in the northern and eastern parts of the country. These coal-bearing sediments are hosted in Ecca Permian (Gondwana) age rocks of the Karoo Supergroup. The Springbok Flats Basin, Main Karoo Basin (MKB),

Ellisras/Waterberg basin which spreads into Botswana, Tuli Basin extending to Botswana and Zimbabwe and the Limpopo Basin which also spreads to Botswana, Mozambique and Zimbabwe are the five major coal hosting basins. 98% of the coal found in the above-mentioned basins is Middle-Rank C bituminous classification (Hancox & Götz, 2014).

Coal deposits found in these basins are limnic, inferring that at the time of peat (partially decomposed plant material) formation, they resulted from mires which accumulated in freshwater environments or in slowly subsiding basins. Deposits existing in marine environments where there is a hydrological connection to the sea are termed paralic (Papp, Hower, & Peters, 1998; Wagner et al., 2018). Peat from which Gondwana Permian coals originate mainly constituted of glossopterids, stunted broad-leaf trees, or shrubs.

South African coal was developed at three stratigraphic points in the Karoo sequence; the Volksrust, Permian Vryheid and the Middle Triassic Molteno Formation. The Vryheid formation has been actively exploited and boasts the most abundant occurrence of coal deposits among the 19 coalfields reported for South Africa, while the remaining formations have barely been exploited (Cadle, Cairncross, Christie, & Roberts, 1993; Wagner et al., 2018). Subbituminous, high volatile bituminous, and anthracitic coals are found which are often affected by dolerite intrusions as well as geothermal gradients.

South African Gondwana coals are mainly of Middle-Rank C Bituminous classification. Dolerite intrusions have partially or completely devolatilised or burnt coals in all the Mpumalanga and KZN coalfields. These intrusions are localised. This was caused by major volcanic activity that happened towards the end of the Karoo sedimentation. Magma intruded into strata below and above and within the coal seams in certain areas, heat affecting the coal and, in some areas, burning out entire seams.

Common in coal deposits within the Vryheid Formation is the presence of low-rank coal in between seams of high-rank coal, a phenomenon that can be attributed to the botanical features of the plant precursors. Most exploited among the coal deposits of South Africa is the Witbank Coalfield of the MKB whose features will be discussed in the following section. Figure 2-1 shows a map of the distribution of South Africa's coalfields according to (Pinetown, Ward, & van der Westhuizen, 2007).

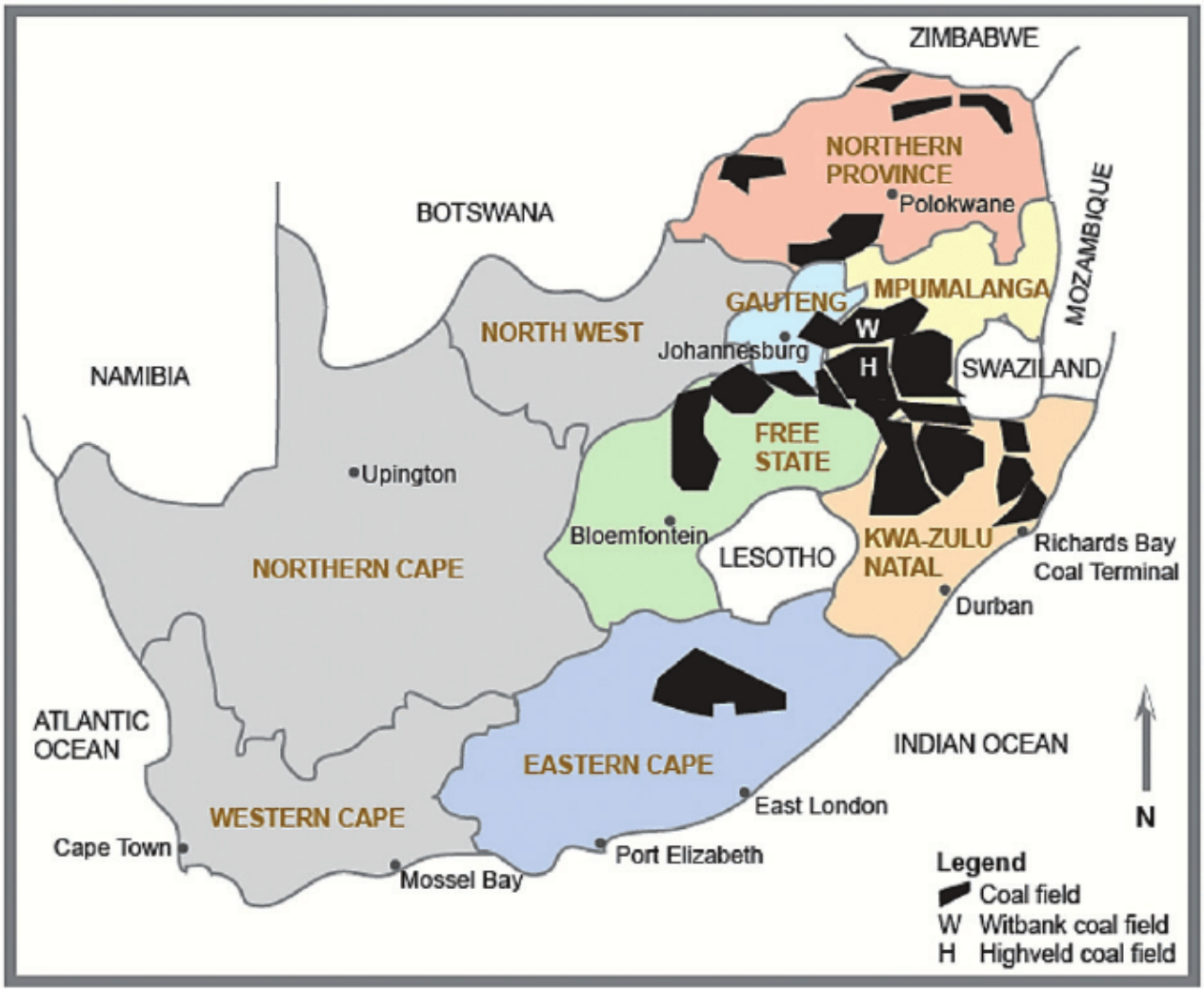


Figure 2.1 Coalfields of South Africa (Pinetown et al., 2007)

2.2.1 The Witbank Coalfield

The Witbank Coalfield of the Mpumalanga province is in the northern part of the MKB, and is South Africa's major coalfield, extending over 568 000ha and producing coal for steam generation and export. Coal-fired thermal power stations in the region are strategically positioned constructed at mining locations to allow for mine-to-mouth supply. Inertinite rich Middle-Rank C bituminous coal of variable ash content is obtained in this coalfield. Dolerite intrusions have affected a major portion of the coalfield subsequently causing devolatisation of the coal. Table 2-1 gives the basic chemical characteristics of coal found in the seams.

Table 2-1 Chemical Characteristics of Witbank Coal Seams (Smith & Whittaker, 1986)

SEAM	ASH (%)	VOLATILE MATTER (%)	INHERENT MOISTURE (%)	CALORIFIC VALUE (MJ/kg)
No. 1	25.4	21	1.7	24
No.2	23.3	21.5	5.3	21.2
No.4	27.6	20.7	2.6	22.2
No.5	13.1	32	2.5	28.7

Of the six seams present, the No.2 Seam is the largest and most utilised. Coal in this seam is distinctly zonal with distinctive quality in each zone. The three basal zones produce export quality steam coal of good washability characteristics and low ash metallurgical coal while the others produce coal for local power stations.

The No.1 and No. 5 seams are an auxiliary supply of coking coal of low phosphorous content. No. 3 and No. 6 seams are generally not mined. The No. 4 seam contains poor quality, dull to

dull lustrous coal with a lot of inorganic intrusions thus is only consumed locally as a power station and domestic steam feedstock (Jeffrey, 2005; Wagner et al., 2018).

2.3 Coal Petrography

Petrology is the earth science that seeks to determine the origin, structure and composition of rocks. The most prominent branch of petrology is petrography which applies microscopy to the description and classification of these rocks (Wagner et al., 2018). To standardise the approach towards petrographic analysis of coal, the International Committee for Coal and Organic Petrology (ICCP) was formed which tracks the progress of petrography over the centuries and monitors the development of new terminology and techniques, to name a few functions.

The applications of coal petrography have expanded over the years to include some of the following; the assessment of physical, inferred chemical and predicted technological properties of the constituents of coal which in combination give a detailed appraisal of the seam's properties; analysis of weathering, oxidation and other anomalous effects either within the coal seam or during storage; accurate profiling of combustion, coking, gasification and other coal conversion processes; a more holistic evaluation of coal as an integral part of the various phases in the coal industry from end-user utilisation going down to marketing, processing and geological exploration (Miller, 2005; Speight, 2012; Taylor et al., 1998; Wagner et al., 2018).

At a macroscopic level coal is classified according to lithotypes. These are bands of at least 5mm which represent changes in organic input and conditions in the peat mires. The criterion for the lithotype identification includes lustre, fracture patterns, texture, and type of stratification, coal colour and the colour of the streak produced by the coal. There exists two main lithotype groups, the non-banded or sapropelic coals and banded/ humic coals. Humic

coals are found in most of the seams in South Africa while sapropelic coals are found in a few seams of economic significance within the region.

(O'Keefe et al., 2013) states that the humic coal lithotype originates from wood, sedge or reed precursors and are a result of the accumulation of humic matter within the proximity of the original peat swamp and is layered with alternating fine bands of variable brightness. Sapropelic coals, on the other hand, are dull, compact, non-banded with even granular surface, have a conchoidal fracture and originate from non-woody, resistant plant fragments such as spores and algae which have been redeposited from the original peat swamp and often have finely dispersed mineral matter.

Petrographic assessments, however, are done at a microscopic level. There are three key assessable parameters at this level, the macerals, coal rank and the degree and type of mineral inclusions that are used to define coal. Macerals are discrete organic constituents which determine the coal type. The degree and type of mineral inclusions are related to the inorganic constituents, these determine coal grade. Coalification progress determines coal rank.

2.3.1 Type (Macerals)

Macerals are entities which evolved from different sections of the parent plant material from primary accumulation to the preliminary stages of biochemical degradation and preliminary coalification (Wagner et al., 2018). Unlike the gangue which is mostly comprised of minerals which have an unchangeable internal structure and homogeneous composition, macerals constitute a mixture of organic compounds whose variability is further augmented by the chemical and physical changes due to coalification. Colour, morphology, shape, the extent of preservation of cell structure, the intensity of fluorescence and reflectance level are the

properties that are used to distinguish between macerals (Mastalerz, Drobniak, Hower, & O'Keefe, 2010).

Biochemical degradation at the peat stage and during the stage of soft brown coal determines the organic composition of coal. The subsequent processes include the partial or complete disintegration of cell structure, peptidisation, plasticization or softening, compaction and homogenisation also play a significant role in establishing the coal type (Teichmüller, 1989). Three classes of macerals, the main coal types, emerge namely vitrinite, liptinite, and inertinite.

Gondwana coals are rich in inertinite which has been attributed to climatic conditions where freeze-drying increased the oxidation of the organic structure, preconditioning the mires to be inertinitic (Taylor et al., 1998). Maceral groups are also unique distinguishable based on their response or change in form or structure when subjected to heat with the inertinite macerals group exhibiting the least reactivity in relation to other groups. When classified according to type, it is the distribution of macerals that is considered.

2.3.1.1 Vitrinite Maceral Group

Vitrinite group macerals originate from humifiable lignocellulosic woody precursors like roots, stems, trunks, branches, and leaves. Subgroups within the vitrinite classification are telovitrinite, detrovitrinite and gelovitrinite (International Commitee for Coal and Organic Petrology (ICCP), 1998). Telovitrinite subgroup macerals tellinite and collotellinite represent intact condensed fragments of woody-origin material of variable degrees of compaction. Detrovitrinite subgroup macerals vitrodetrinite and collodetrinite originate from smaller fragments, exhibit a higher degree of degradation and often serve as groundmass to other macerals (Teichmüller, 1989) Gelification prior to or during coalification produces either

gelinite or corpogelinite macerals of the gelovitrinite subgroup. This process happens under anaerobic or reducing conditions.

The chemical structure of vitrinite is the second most oxidised in comparison to other coal types, the degree of oxidation increasing with rank. Inherently, vitrinite macerals also possess aliphatic groups and aromatic rings which highly influence and affect its industrial application. In Middle-Rank C bituminous coals vitrinite has a dark grey appearance under the microscope with reflectance values midway between those of liptinite and inertinite(Wagner et al., 2018). These values are subject to change with progressive rank and chemical attenuation.

2.3.1.2 Liptinite Maceral Group

Liptinite group macerals are a coal type that exhibits greater chemical resistance to chemical and physical degradation than other maceral types. This is related to their composition which largely constitutes of non-humifiable plant matter like cuticles, pollen, spores, resins, and waxes which also serve as the subdivisions of the maceral group (Mastalerz et al., 2010). Owing to a greater number of aliphatic groups, liptinite macerals like alginate (algae remains) and bituminite are rich in hydrogen. Alginite is often associated with deep water coals while bituminite is associated with sedimentary rocks with shale gas potential. Other macerals within the liptinite classification are sporinite, cutinite, exudatinite, resinite, and suberinite.

Liptinite macerals are generally uncommon in Gondwana Permian coals except in sapropelic bands of coal. Compared to vitrinite, liptinite exhibits lower reflectance at a low and medium rank or thermal maturity and typically show autofluorescence upon illumination with violet, ultra-violet and blue light (Pickel et al., 2017). At ranks above medium-volatile bituminous, liptinite is slowly extinguished with increasing degree of coalification as the chemical reactions during the process made it harder to distinguish them from inertinite and vitrinite due to the

convergence of reflectance of the other maceral groups(Wagner et al., 2018). In low-rank coals, liptinite is fluorescent. The colour of the fluorescence and the intensity helps distinguish the individual macerals of this group.

2.3.1.3 Inertinite Maceral Group

Inertinite macerals originate from plant material that has been subjected to oxidising conditions often with exposure to high temperatures before or during peat formation resulting in strong alteration and degradation of original cellular structure. Gondwana coals have a high inertinite content due to the conditions during peat formation. Processes such as aerobic decomposition, redox reactions, bacterial and chemical attack during coal formation significantly complement inertinite macerals formation.

The term inertinite indicates that the constituents show greater inertness compared to vitrinite and liptinite maceral groups. This is particularly related to the carbonisation process where they show diluent behaviour (ICCP, 2001). Though of similar botanical origin to vitrinite macerals, inertinite group macerals have a chemical structure that shows greater aromaticity and higher carbon content and lower oxygen and hydrogen content because of fusinisation (ultimate carbon enrichment) and the varied oxidation processes mentioned above. This results in maceral forms which are lighter (higher reflectance) in comparison to liptinite and vitrinite macerals of similar rank (Teichmüller, 1989).

Fusinite and semifusinite are in part a result of wildfires, with reflectance values that suggest a direct proportionality to the fire temperature (Mastalerz et al., 2010). In the South African context, semifusinite is further subdivided into reactive and inert semifusinite with the former exhibiting a shade of grey analogous to that of vitrinite in the same unheated sample and possessing comparable technological and chemical properties.

2.3.2 Rank (Coalification)

Coalification is defined as the process of metamorphosis that occurs over long periods of time in conditions of increasing pressure and temperature resulting in the progressive chemical, physical and optical change of the original peat swamp through the lignite, sub-bituminous, bituminous, anthracite, and meta-anthracite stages. (SANS 7404-1, 1994) defines coalification as the process by which sedimented and compacted remains of plant materials are transformed into coal. Rank subsequently is thus the stage at which the coal has reached in the coalification series, otherwise known as the degree of maturation. South African coals predominantly report to the Middle-Rank C bituminous classification.

A major characteristic of coalification is carbon enrichment in the plant remains and decrease of volatile matter. This is linked to an increase in calorific value, a major standard by which coal is traded. With the advancement of coalification, the reflectance of the macerals increases thus the use of reflectance to determine the rank of coal. Vitrinite macerals show the greatest correlation or uniformity between reflectance values and the extent of coalification and thus have been used as reference material in determination of coal rank.

2.3.3 Grade - Inorganic or Mineral Inclusions

While coal type is based on organic or maceral composition, coal grade is based on the inorganic or mineral constituents. Coal grade is based on the inherent ash or mineral matter content of a coal. Apart from calorific value, ash is the second major standard by which coal is traded, hence the importance of grade. Ash is undesirable primarily because it increases the freight costs as well as being detrimental to the thermal and coking properties of coal thus an undesirable constituent that must be minimized in coal utilisation plants.

In coal-fired thermal power generation, ash hinders heat transfer by causing thermal insulation of furnace wall tubes and convective pass tube banks. The rate of heat transfer to the tube surfaces decreases with the accumulation of ash deposits resulting in high flue gas temperatures and thus inefficient transfer of heat to the process. In coking, ash negatively affects the coke strength after reaction (CSR) and results in high slag volumes in the blast furnace. The slag is often acidic, increasing slag viscosity and reducing gas permeability thus necessitating an increase in the amount of the basic fluxes used in the process which consequently increases the cost at this stage and the subsequent stage of hot metal pre-treatment.

Mineral matter is subcategorised as syngenetic and epigenetic in relation to their association with the coal. Syngenetic, intrinsic or inherent inorganic matter originates from the various botanical parts of the original plant and is composed of inorganic compounds or organometallic complexes absorbed from the soil and water in the plant's ecosystem (Wagner et al., 2018). This mineral matter is ultimately trapped in the form of sub-microscopic grains and complexes within the coal, common being pyrite, siderite, and kaolinite.

It is the epigenetic or extrinsic mineral matter that is present in significant quantities to attenuate the coal's properties, often originating from the roof and base of the coal seam and from minerals deposited by precipitation of ground-water solutions into fractures, pores and cavities within the seam long after peatification (Falcon, 2013; Speight, 2015; Wagner et al., 2018). It generally consists of fragments of shale, clay, and stone together with intrusions of inorganic salts like calcite, dolomite, and pyrite.

According to Pinetown et al (2007) coal found in the Witbank and Highveld coalfields contains about 8-35% mineral matter whose constitution is primarily clay and quartz, the former being more difficult to beneficiate than the latter. Coalfields of South Africa generally have kaolinite

clay in abundance, occurring in syngenetic and epigenetic form and have, in lesser abundance, illite and montmorillonite clays.

Clay minerals pose a challenge in all stages of mineral processing, syngenetic clay being the hardest to separate and epigenetic clay still causing significant difficulties. Psychochemical beneficiation through flotation and oil agglomeration is hit hardest due to the very fine particulate nature and different complex chemical structures of clay. Major among the problems faced during flotation, in particular, is the change in froth stability, overconsumption of reagents, increase in pulp viscosity, slime coating, alteration in particle-bubble attachment and mechanical entrainment (Taner & Onen, 2016).

2.4 Analytical Techniques, Standards, and Instrumentation

Due to the need for quality and standardisation across the coal sector, standards have been set governed by international standardisation bodies in collaboration with the local standardisation authorities. The South African Bureau of Standardisation (SABS) is an affiliate of the International Organisation for Standardisation (ISO) whose purpose is the development and assessment of conformity to quality standards across all disciplines, including coal analysis. Where a local standard is unavailable the Australian Standards or the American Society for Testing Materials (ASTM) are used.

2.4.1 Chemical Analysis

For most industrial applications a basic empirical analysis of the calorific value and proximate results often suffices while the more detailed and specific techniques are of importance to coal producers, researchers, and regulators. As an alternative to petrography-based coal ranking is the use of calorific value and proximate analysis. The coal is first assigned a rank using the calorific value (MJ/kg) and then using another parameter, particularly the volatile matter, the

coal is then assigned a subcategory (Li, Whitely, Xu, & Pan, 2005). However, this coal ranking technique is less reliable than the petrographic examination of vitrinite reflectance.

2.4.1.1 Calorific Value

The calorific value is a thermal property of coal whose significance is particularly of interest if coal is to be used for energy generation purposes. Also known as the heating value, the calorific value is defined as the heat released during combustion of a unit mass of coal in an oxygen bomb calorimeter under a specified set of conditions (ASTM D121-15, 2015; SANS 1928, 2009; Speight, 2015).

2.4.1.2 Proximate Analysis

Proximate analysis is an empirical method of coal assay that determines the products of coal combustion under a set of prescribed conditions to quantify four groups namely ash, volatile matter, inherent moisture, and fixed carbon. This assay is usually carried out on an air-dried sample as prescribed by (SANS 17246, 2011). Figure 2-2 below shows the principal features of coal as determined by proximate analysis and how they affect the use of the material in thermal applications.

Figure 2.2 Thermal Impact of the Proximate Parameters of Coal

Inherent moisture is the water that is in surplus of the equilibrium moisture content and is presumed to be held within the capillaries and pore system of the coal. Moisture increases freight cost, reduces the calorific value and enhances heat loss by evaporation and superheating of vapour. However, it is still important in limited quantities as it serves as an aid in radiative heat transfer and to lesser extent assists in the binding of coal (Frayne, 2002). Some coals continue to liberate moisture beyond 110 ºC, the standard temperature when determining inherent moisture. This moisture is in the form of the water of crystallisation and consequently reports to the volatile matter content (Theron & Le Roux, 2015).

Volatile matter is an index of the gaseous fuels. Organic volatile matter significantly contributes to the heating value of the coal and usually comprises methane, short-chain hydrocarbons, hydrogen, and carbon monoxide. The ease of ignition improves with increasing volatile matter content but however, such coal tends to burn quickly and with a long, smoky flame.

Also, coals with higher volatiles content tend to have lower ash fusion temperatures which translate to higher slagging and fouling tendencies, thus demanding operation at lower furnace temperatures which might not be feasible depending on the process. Thus a limit is often put on the amount of volatile matter required based on consumer process (Frayne, 2002; Speight, 2015).

Inert volatiles, the volatile matter which is of inorganic origin as well as incombustible gases like nitrogen and carbon dioxide are present in coal. Such volatile matter is undesirable since it may adversely affect ignition, participate in heat consuming endothermic reactions and does not produce combustible species that contribute to an increase in calorific value. (Theron & Le Roux, 2015).

Minerals in the coal are responsible for the production of these inorganic volatiles, common among them being the carbonates which produce CO_2 produced by the decomposition of carbonates, water of crystallisation by clay minerals, SO_2 by the oxidation of pyrite, FeS_2 and hydrogen chloride gas, HCl(g) from the chloride minerals(Speight, 2015).

Ash results from chemical alteration of mineral matter during the process of combustion. A common generalisation is that ash and mineral matter are interchangeable terms that describe the same matter, but this is inaccurate since the latter is comprised of the full constitution of unaltered inorganic matter in coal while the former only retain a fraction of the mineral matter which is unalterable at the process' furnace temperatures.

Ash is undesirable because it increases freight costs and reduces combustion and boiler efficiency due to the formation of clinker and slag. The accumulation of ash deposits which happens primarily through slagging lowers the rate of heat transfer to the process in boiler

systems and consequently results in high flue gas temperature which can be used as an indicator of process inefficiency (Frayne, 2002).

Fixed carbon is the key liberator of heat energy during the exothermic reaction of combustion. This value must always be known in any operation to prevent excessive heating that can cause damage to the furnace and associated equipment and to ensure steady energy release. An effective beneficiation process would thus have to increase the fixed carbon content while reducing the mineral matter content per unit mass of the product. Despite its practicality, it is imperative to note that fixed carbon value is not defined on an elemental basis and can be found to contain residual sulphur, nitrogen, hydrogen, and oxygen. It also can be a function of parameters like gas pressure, heating rate and secondary reactions of the volatile content of coal (Theron & Le Roux, 2015). As such ultimate analysis is necessary to further differentiate these species.

2.4.1.2.1 Thermogravimetric Analysis

Thermogravimetric analysis, TGA is a method applicable to proximate analysis that involves the usage of the weight loss of the coal sample when heated in inert and oxidised atmospheres to yield proximate results in a shorter space of time as compared to the muffle furnace-based approach. The standard commonly used is ASTM D7582-15, 2015. This approach yields the proportions of inherent moisture, volatiles content and fixed carbon content. Subsequently, the ash content is then determined using the formula;

$$\%\,ASH = 100 - (\%\,MOISTURE + \%\,VOLATILE\ MATTER + \%\,FIXED\ CARBON)$$

Due to the use of oxygen, complete combustion is achieved more rapidly in comparison to the use of air in the alternative approach to analysis. In addition, a single sample can be used to analyse all three parameters using an inert gas, nitrogen, to avoid oxidation during the

determination of the volatile matter and inherent moisture. Analysis happens in three stages, the first two using nitrogen gas and the third oxygen gas.

Firstly, the TGA furnace's temperature is ramped (raised) to 110°C and held isothermally. The isothermal region's weight loss determines the inherent moisture content of the coal. A second ramping of the temperature to 900°C is then done and isothermally held. The weight loss at this isothermal condition is a direct effect of volatile matter. Fixed carbon is then determined at the third stage when nitrogen gas supply is substituted with oxygen gas to facilitate combustion(Li, Whitely, Xu, & Pan, 2005). The residue post-combustion is the ash content.

2.4.2 Petrographic Analysis

Petrographic analysis is a subdivision of coal petrology concerned with the microscopic description and classification of sedimentary organic matter (coal) based on the concept of macerals and mineral associations. In South Africa, this is done according to (SANS 7404-1, 1994; SANS 7404-2, 2015; SANS 7404-3, 2016; SANS 7404-5, 2016) standard analytical techniques which are developed by the South African Bureau of Standards.

2.4.2.1 The Petrographic Microscope

The instrument used for petrographic microscopy is the reflected light polarising microscope, also known as the metallurgical, epi-illumination or incident light microscopy. It is used for imaging or fluorescence of materials which have been milled to a d_{80} passing size of 30µm. Figure 2-3 shows the typical anatomy of a reflected light microscope

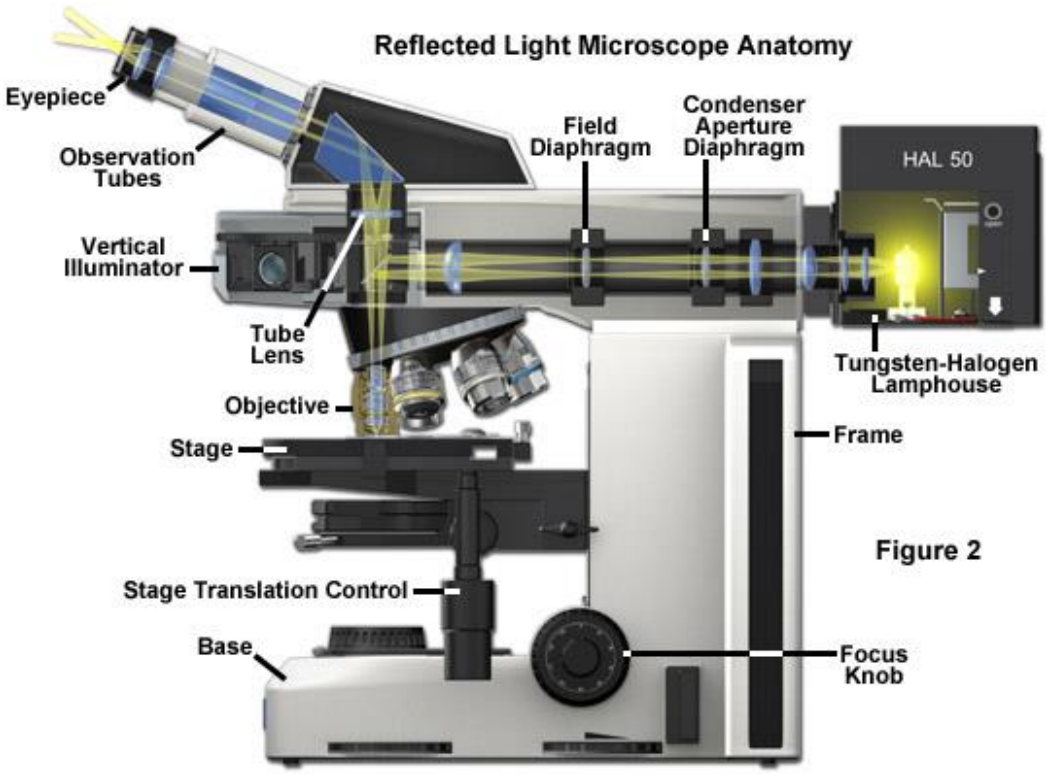


Figure 2.3 Anatomy of a Reflected Light Microscope (Zeiss Microscopy Online, 2019)

The reflected light's optical pathway starts with the illumination of rays that originate in the reflected light lamp housing. This light passes through a collector lens to a vertical illuminator where it is regulated by the aperture and field diaphragms. Upon exit, the light is then reflected by a beam splitter, through the objective, to illuminate the coal specimen. Reflected light from the surface of the coal specimen re-enters the objective and proceeds to the binocular head from where it is either directed to the eyepieces or to a photomicrography port.

In coal petrography, standard practice is to use polished or thin sections of a specimen that have been carefully prepared prior to analysis and an immersion oil objective is used which increases the refraction index in comparison with air, increases the numerical aperture value and has enhanced resolution. In some instances, fluorescent light is used instead of white light to identify certain macerals, especially liptinite.

2.5 Coal Beneficiation Scheme

Beneficiation often follows the sequence; size reduction, screening and then isolation of the gangue constituents from the coal using varied unit operations. Separation of the undesired constituents requires a specialised feasibility study in which probable cleaning methods are identified and the most suitable method chosen based on factors like yield, economic potential, environmental impact and geographical positioning of the identified coal processing site. In the coal instance, the preliminary beneficiation study that is usually carried out seeks to determine the washability or amenability of the coal sample to improvement in grade by float-sink analysis.

Near-perfect separation is achievable in some instances, provided that the gangue is isolable completely from the coal particle by size reduction. It has been observed, however, with most coal that with decreasing particle size the separation potential of float sink analysis progressively diminishes. Due to other technical factors, float-sink analysis is impracticable in an industrial set-up and is thus limited to laboratory use. From the data obtained, information is then generated that advises on the most practicable beneficiation step based on the desired technological application of the product. The subsections that follow discuss varied wet and dry beneficiations technique applicable to coal.

2.5.1 Comminution of Coal

ROM coal can, in some instances, have a diameter of up to 1 metre. Comminution thus involves the crushing and/or milling of mined coal rocks to reduce them to a particles size distribution (PSD) that permits the liberation or occurrence of most of the maceral components of coal and the gangue or mineral constituents occur as separate particles or as determined by the tolerance of the subsequent unit operation. Single-stage open or closed, multi-stage open or closed circuit

configurations are alternatives to comminution which can be selected based on the physical properties of the coal rock and crusher or mill efficiency (Gupta & Yan, 2016).

When size reduction is done in a single pass using a single-stage open-circuit configuration the resulting product is often of a particle size distribution that seldom achieves the targeted degree of liberation. Therefore, multi-stage size reduction is often necessary to gradually reduce the ensuing particle size to a range that will liberate mineral particles to an acceptable degree.

When a closed-circuit configuration is used, a classifier becomes necessary to separate the product into coarse and fine fractions. The coarser fraction is recirculated for further comminution using the same unit. Consequently, the load on the size reduction equipment is increased and a re-circulating load is established, but the required number of units to give the targeted extent of size reduction is lessened(Gupta et al, 2016).

The degree of liberation targeted informs the extent to which size reduction should be done. Laskowski (2001) defines the degree of liberation as the proportion of free particles in relation to the total material. The trend often observed is an increase in liberation with a reduction in the size of the particles present, which can in part be explained to be due to preferential breakage along the planes of contact between the discrete components and secondly because of the reducing likelihood of the occurrence of composites in the material of smaller size.

In the modelling of comminution systems, the basic idea is to obtain a mathematical relationship linking the feed to the product size. For this to be achieved an account of all variables involved in operation inclusive of machine characteristics must be taken into consideration. Comminution is represented by two sub-processes namely selection of a particle for breakage and the determination of the distribution of fragment sizes produced by the broken particles.

During the design of a comminution plant, the power requirement for size reduction and the choice of the crushers and/or grinders to use are very critical. The power or energy requirement is inclusive of the work involved in the crushing and or grinding of the rock and the work required to rotate the mill. Features such as the hardness of the rock, the size of the feed rock and the targeted product size determine the power required in crushing and grinding to realise sensible liberation of the targeted value material from the host rock.

The grindability of the coal is commonly pursued through the batch selection or batch laboratory method, using a model proposed by Bond. In milling operations, the Bond model has been found to be the most realistic of the three, which measures the Bond Work Index W_i, a function determined using the physical properties of the coal rock. From the data obtained scale-up from laboratory to pilot size and ultimately to the full-scale size can be done. The energy required to process a tonne of coal and the power requirement for a specified throughput using the Bond Method is obtained from the equations;

$$\boldsymbol{E = 10W_i\left[\frac{1}{\sqrt{l_{P80}}} - \frac{1}{\sqrt{l_{F80}}}\right]\frac{kWh}{t}} \quad ; \quad \boldsymbol{P = E\left(\frac{kWh}{t}\right) \times F\left(\frac{t}{h}\right)} \quad \boldsymbol{kW}\text{.....Eq 2-1}$$

where l_{P80} and l_{F80} are the respective passing sizes of product and feed, E the energy required to process a tonne of coal, F the feed rate and P the power requirement.

The primary challenge lies in that the model is mostly empirical. Data obtained from this model is estimated to have an accuracy of ±20%. For coal, determination of the Bond Work Index is more complex considering the multiplicity of compounds constituting the coal composite. Details of the process also do not feature in the model. Without the specific details of the process, it is impossible to expect that the model will account sufficiently for all the variables associated with the unit operation.

Coal characteristics exhibit a certain range of dynamicity within the coal body depending on the geology of the seam being mined at that time, therefore a robust model that can tolerate the variability is required. During closed circuit milling, the regrind is even more difficult to model. The Bond Law Model also suffers the handicap of being unable to predict the product particles size distribution henceforth PSD as affected by feed PSD and operating conditions.

Kinetic modelling is a more mechanistic method which has shown greater enhanced accuracy during scale up to pilot and full industrial mill size. This is because it is based on the physics of all major circuit components and is ideal for simulation through steady-state design and dynamic control.

The major parameters in milling kinetics are the selection and the breakage functions. The selection function is a very useful material and size class-specific function which considers the rate of breakage of particles from a discrete size class. The breakage function, on the other hand, defines the fraction of particles which when broken from a particle size interval *j* will reports to a size class *i* before the re-breakage of the fragments. These parameters can be defined through the equations;

$$\frac{d[Wp_i]}{dt} = -s_i Wp_i \quad \text{.....Eq 2-2}$$

W is the total mass of the ore and P_i is the mass fraction of size class i

$$B_{ij}(breakage\ function) = \frac{mass\ of\ particles\ from\ class\ j\ broken\ to\ class\ i, t \to 0}{mass\ of\ particles\ of\ class\ j\ broken, t \to 0} \quad \text{....Eq 2-3}$$

Based on the intended circuit output, the size (weight) of the mill can be modelled based on these functions thus permit the determination of a more realistic power draw requirement. The power draw in this instance is obtained by the equation;

$$P = K(J)\rho_L L D^{2.5} \frac{N}{N_c} sin\theta\ kW \quad \text{.....Eq 2-4}$$

Where $K(J)$ is the mill filling capacity, ρ_L the density of the steel balls, L and D the length and diameter of the mill respectively, $\frac{N}{N_c}$ the critical speed of the mill, and θ the angle of repose.

Batch tests are commonly used in determining the breakage function. Then selection function is often found using data obtained from a continuous mill. However, an alternative method which avoids usage of the selection function exists which uses iteration techniques to solve the population-balance model grinding equation. The inherent weakness associated with assuming first-order kinetics and the complexity associated with determining the actual order of kinetics are thus eliminated resulting in a more accurate model.

Therefore, a detailed relationship amongst various grinding sub-processes like particle breakage kinetics and size reduction, classification of product particle size, and transport of material or particle transport through the mill can be established. Such a detailed relationship between interdependent grinding sub-processes provides a considerable advantage over simplified correlations like reduction in particle size and expended energy.

This information, in turn, can be used to proactively simulate grinding data for mill scale-up design purposes. Population balance modelling also has the advantage of producing good reproducible data in both crushing and milling operations.

2.5.1.1 Size Control during Crushing and Grinding

Screening is the most common form of classification for coal. The primary purpose of classification is to isolate material that has been reduced to the appropriate size from that which is still of larger size and thus needs to be fed back to the comminution circuit. This often requires a single screen with predetermined aperture size. To further distribute the classified product into more defined, narrower particle size distributions, multi-stage screening with decreasing aperture sizes can be applied.

The screening unit operation may also act a preliminary beneficiation step. Clay minerals are often of very fine size and are loosely associated with the coal and thus during screening will report to the smallest PSD. This material can thus be eliminated at this stage thus avoiding the process challenges it poses. Alternatively, a hydrocyclone can be used to achieve classification. Fig 2-4 shows a single-stage milling circuit which utilises a hydrocyclone classifier.

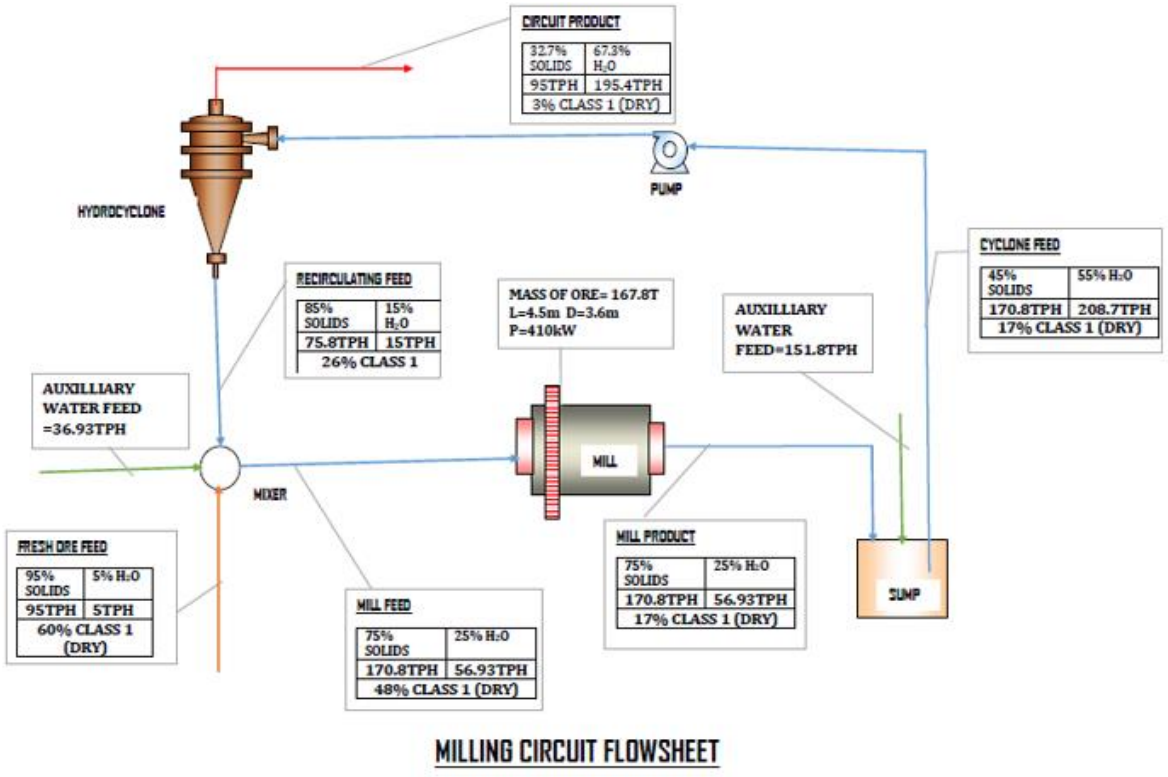


Figure 2.4 Single-Stage Closed Milling Circuit with a Hydrocyclone Classifier

2.5.2 Washability of Coal

Coal washability is the determination of the theoretical parameters for the removal of mineral inclusions from coal by beneficiation practices that effect separation based on specific gravity. At present, density-based methods are the most utilised separation strategy for Gondwana coals. In South Africa, the washability characteristics of coarse coal (size >1mm) have been well investigated but not as much effort has been put into investigating the washability of finer

size material (King, 1990), an omission that is grave considering the large quantities of fines that are produced at local washeries and collieries.

Float-sink testing is the laboratory method used to determine the washability of coal with respect to relative density. The data obtained from the process has gained significant trust among industry players and is extensively used for coal unlike other mineral processing operations because of the availability of a wide range of liquids of suitable density, a fortunate circumstance attributable to the relatively low particle densities for coal(Galvin, 2006).

The conventional approach involves the setting up of baths containing liquid solutions of different density, usually in the relative density range 1.3-2.0 with a 0.1 density variance between successive baths. Accurate determination of density is made possible using a series of hydrometers that cover the density range. A procedure is yet to be designed for determining the washability characteristics especially in relation to low rank, fine coal as the current method has problems related to moisture loss through drying the preparation and analysis of the sample (ASTM D4371-06, 2012).

There are a variety of float mediums usable in float-sink testing, prominent among them being; organic liquids; aqueous solutions; suspensions; autogenous separations/ water fluidisation for jigging; direct measurement of particle density; in-situ measurement of partition curves. The current South African standard (South African National Standards (SANS) 7936, 2010) is based upon the first three methods. This group of methods relies on particles undergoing a sedimentation process to effect separation. If a particle has a density lower than that of the given liquid medium it ultimately floats to the liquid's surface while a particle of higher density ultimately sinks.

At the point of separation, the particle weight is thus entirely supported by the weight of the displaced fluid otherwise known as the buoyancy force of the liquid (Galvin, 2006). Particle size is directly proportional to settling velocity. Thus, with a decrease in particle size, more time should be allowed for settling.

To ensure precise setting of the density of a bath, calculation based on liquid medium data is often insufficient and therefore a hydrometer must be used. The direction chosen in float sink testing is often based on individual choice as the results are often similar. In cases where there is a dependence on the direction taken either from the lowest to the highest density bath or contrariwise, such dependence is termed hysteresis.

The autogenous dense medium method varies from the autogenous suspension method in that the weight of the particle is supported by both buoyancy and drag forces of the fluid thus the separation density is considerably higher than in the latter case. The final method involves the quantification of the mass and then the volume of the solid. To determine the volume of the particle, a vessel of known volume is used into which is added a gaseous or liquid fluid of an amount enough to envelop the particle and fully enter any pores within its matrix. This approach gives a different density form depending on the fluid utilised. Pycnometric devices are often used in this approach.

There are merits and challenges to every approach in float-sink testing. Of the (South African National Standards (SANS) 7936, 2010) given standard approaches to float sink testing, the use of organic liquids has the greatest demerits. Organic liquids, particularly chloroform (Cheremisinoff & Rosenfeld, 2010; Winslow & Gerstner, 1978) poses the challenge of toxicity concerns related to neurological, mutagenic and carcinogenetic concerns. Air drying is necessary in-between successive steps to ensure that pores within the coal particle do not carry over heavy liquid to the next step in the float-sink separation thus eliminating a porosity

associated error in establishing the density set-point for a coal beneficiation plant whose basis is this analysis. Secondary challenges associated with the medium are particle contamination by chlorine and modest viscosity and chemical interaction concerns.

The aqueous solution and suspension approaches to float-sink testing are often co-referred to as water-based systems. The advantage of these systems is that the inorganic compounds forming the aqueous solution or suspension are often recoverable though with varying levels of complexity in the process. Therefore, effluent from these operations is less harmful to the environment.

However, suspensions and solutions requiring the loading of an active component often tend to become significantly viscous when a high density is required which consequently results in low settling rates and slow drainage of the medium from the particles. Therefore further washing of the product to remove the inorganic salt is necessary, thus increasing the cost of salt recovery which is often pursued through evaporative separation (Galvin, 2006).

Contamination issues are more pronounced for aqueous solutions in comparison to suspensions due to the high concentration of inorganic salt required which inherently bring the challenge of the drying of the salt in the pores. Some reactivity might also exist between the coal and the salt by exchange and/or adsorption of ions should the salt breakdown in water. At higher viscosities, liquid uptake may be significantly hindered hence the results might exhibit sensitivity to the time spent in the medium. Finer particles have a larger surface area and thus would require more contact time with the medium than coarser particles.

Zinc chloride, caesium formate, potassium formate, and sodium polytungstate are the most frequently used salts in the preparation of heavy liquids. Using zinc chloride, $ZnCl$ has the demerit that it is a halogen compound hence environmental trepidations emanate. Caesium

formate,$CHCsO_2$ has good solution viscosity of 2-5cp, analogous to that of organic liquids, but is costly thus limiting its use. Potassium formate,$KHCO_2$ on the other hand, is ten times cheaper than caesium formate but has very high viscosity and thus needs to be mixed with caesium formate which complicates the recovery process for the two salts.

Sodium polytungstate (SPT),$Na_6[H_2W_{12}O_6]$ or $3Na_2WO_4 \cdot (WO_3 \cdot H_2O)$ is a relatively new compound with only about 25 years in the industry. The salt dissolves well in water, has no halogen compounds and a low chance of physicochemical interaction with the coal though still requiring careful washing off the salt like the other salts. The major edge of SPT is a wide viscosity range of 1-60cp, a feature that allows it to be utilised over a wide range of bath densities and widens its areas of application though the cost might inhibit frequent use in an industrial set-up (TC-Tungsten Compounds, 2019).

Stability is a concern in the case where solid particles require suspension to form the medium. Haematite, Fe_3O_4 is the most common suspension in use. (Galvin, 2006) states that haematite use poses the challenge that the particles settle forming sediments that are very dense and difficult to suspend again thus requiring that the particles are maintained is suspension continuously, which is another challenge. The major advantage of haematite is that it occurs naturally and thus requires simple processing prior to usage which makes it affordable for industrial application on a continuous basis(Lockhart, 1998).

2.5.3 Coal Processing Unit Operations

Based on float-sink washability testing results, analytical data and the sizing of the coal to be processed, parameters are identified that are used to design the coal processing unit operation. All these unit operations have merits and demerits and thus careful assiduous planning is

required before selecting the best unit operation to invest in. This stage is often shorter for coal than for other resources because of the maturity of the technologies in coal processing.

The Witbank No.2 seam has in the past been the main source of marketable coal in South Africa. Good grade coal (27.5MJ/kg) with good washability characteristics is found in this seam and the fines present (0.1-0.5mm) can be beneficiated using spirals. However, this seam has now been depleted and thus leading to the mining of the No. 4 seam which has lower grade coal and fines which are difficult to beneficiate using spirals. The need to investigate other beneficiation methods is therefore now pertinent due to the significant differences in former and remaining coal resources with lower-grade coal coming into the picture more prominently than before. In this section a few unit operations will be discussed, the fundamentals underpinning their mode of operation and the merits and demerits of their application.

2.5.3.1 Air Dense Medium Fluidised Bed Separation

Air-dense medium fluidised bed separation is a dry beneficiation technique which uses the physical properties of the coal, particularly density, to effect separation. The principle of operation is analogous to that of hydraulic dense-medium separators, with clean coal floating to the surface of a pseudo-dense medium and the rejects sinking. This pseudo-dense medium is essentially a two-phase gas-solid fluid created through the suspension of solid particles in an upward direction using air (Luo, Zhao, Fan, Tao, & Chen, 2006).

Bed density is consistent throughout the region of fluidisation thus solid-phase particles stratify in the bed in order of their individual densities. The mechanism behind the beneficiation process is that the bed density of the pseudo-fluid medium equals the separation density and the pressure distribution in a fluidised bed equals that of static fluid. The forces interacting with

a coal particle in the fluidised bed are gravity, buoyancy and air-coal particle, air-medium and medium-coal particle frictional drag forces (Sahu, Biswal, & Parida, 2009).

To create a fluidised bed of the required density for separation, the equation below is employed.

$$\rho_b = (1-\varepsilon)\rho_s + \varepsilon \cdot \rho_a = \frac{W}{(L \cdot A \cdot g)}$$

where ρ_b is the average bed density $\left({}^{kg}/_{m^3}\right)$, ε the bed porosity, ρ_s and ρ_a the respective densities $\left({}^{kg}/_{m^3}\right)$ of the medium and air, L and A the respective depth (m) and the cross-sectional area of the bed (m^2) , g the acceleration due to gravity $\left({}^{m}/_{s^2}\right)$ and W the total weight of the medium (kg).

Perfect separation using the air dense medium fluidised bed is in accordance with Archimedes' principle which states that the weight of the fluid displaced by an object is proportional to the buoyant force applied to the object. However, in real experimental conditions and in actual practice, the misplacement of particles is observed. Bed viscosity and the velocity of the medium contribute to the misplacing effect (Zhenfu & Qingru, 2001).

When gas velocity is low, the activity of the pseudo-fluid medium is low resulting in lowered bed viscosity and subsequently the misplacing of coal particles. Conversely, when gas velocity gets very high, gas bubbles cause the back mixing of the solids constituting the pseudo-fluid medium thus causing misplacement of coal particles due to attenuation in medium velocity.

A reduction in coal size directly results in an increase in the specific surface which reduces the terminal velocity of the coal particles and increases the drag to gravity ratio exerted on the coal particles thus causing the misplacing effect due to the attenuation of both bed viscosity and medium velocity. Selection of the gas fluidisation velocity, therefore, should be in accordance

with the optimal velocity that can achieve separation of the smallest sized coal particles in the feedstock.

The merits of air dense fluidised bed separation are high thermal efficiency, absence of complexity and cost associated with process wastewater and the fines generated are already suited for fluidised bed combustion. The main advantage nonetheless is that the product is dry with a higher heating value per unit mass of coal. Like other dry coal beneficiation techniques, it, however, has the disadvantage of having less sharp separations than hydraulic beneficiation through gravity separation or flotation. Research into the development of this technique has been mostly nucleated in Canada, China, and India (Sahu et al., 2009).

2.5.3.2 Dense Medium Cyclone

The DMS Cyclone is a wet beneficiation technique developed by Dutch State Mines (DSM) that operates on a similar principle to hydrocyclones where beneficiation occurs based on the subjection of both ore and medium to drag and centrifugal forces with the exception that in the former separation is density and not particle size based. Suspension density is positioned between that of coal and its associated mineral matter. A DMC typically processes ROM coal within the particle size distribution $50mm \leq x \leq 0.5mm$.

The most appealing features of this separation technique are the high capacity, relatively low operating costs and remarkably high separation efficiencies hence its ability to beneficiate difficult to wash coal with a large quantity of near gravity material, NGM (Narasimha, Brennan, & Holtham, 2006). However, increased specialist attention is required as NGM increases. South African coal characteristically is high in NGM, hence the popularity of the DMC in the local coal processing industry.

Experience gained from decades of processing Gondwana coals has shown that NGM is not solely dependent upon the washability characteristics of the feed coal alone thus leading to the redefinition of NGM to $\pm(2 \times E_p)$ from the cut density (Bhattacharya, Maheshwari, & Panda, 2016). This new definition expands the range of variables upon which NGM is dependent to include the process performance of the specific cleaning unit as measured using a Tromp or partition curve and the probable error of separation, E_p thus considering factors like the rate at which coal is fed into the unit, feed particle size distribution (PSD), the state of repair (maintenance) of the unit and the specific conditions for the operation.

Even though as there are various dense medium cyclone models available, the process fundamentals remain unchanged from those deciphered by Dutch State Mines. Beneficiation occurs via the same differential settling principle as in float-sink testing where separation is achieved based on buoyancy differences between dense gangue and light coal particles.

The difference between the DMC and static vessels, the is that the process of separation in the former is hastened because the flow of water inside the cyclone creates a centrifugal force which causes denser gangue particles to move to the walls of the cyclone where they then descend due to axial velocity and are discharged to the underflow while the lighter coal reports to the overflow weir. Due to the enhanced settling rates induced by the conditions in the cyclone, the particle size limit can be extended beyond the usual limit for static vessels. Fig 2-5 gives the principal features of a DMS Cyclone.

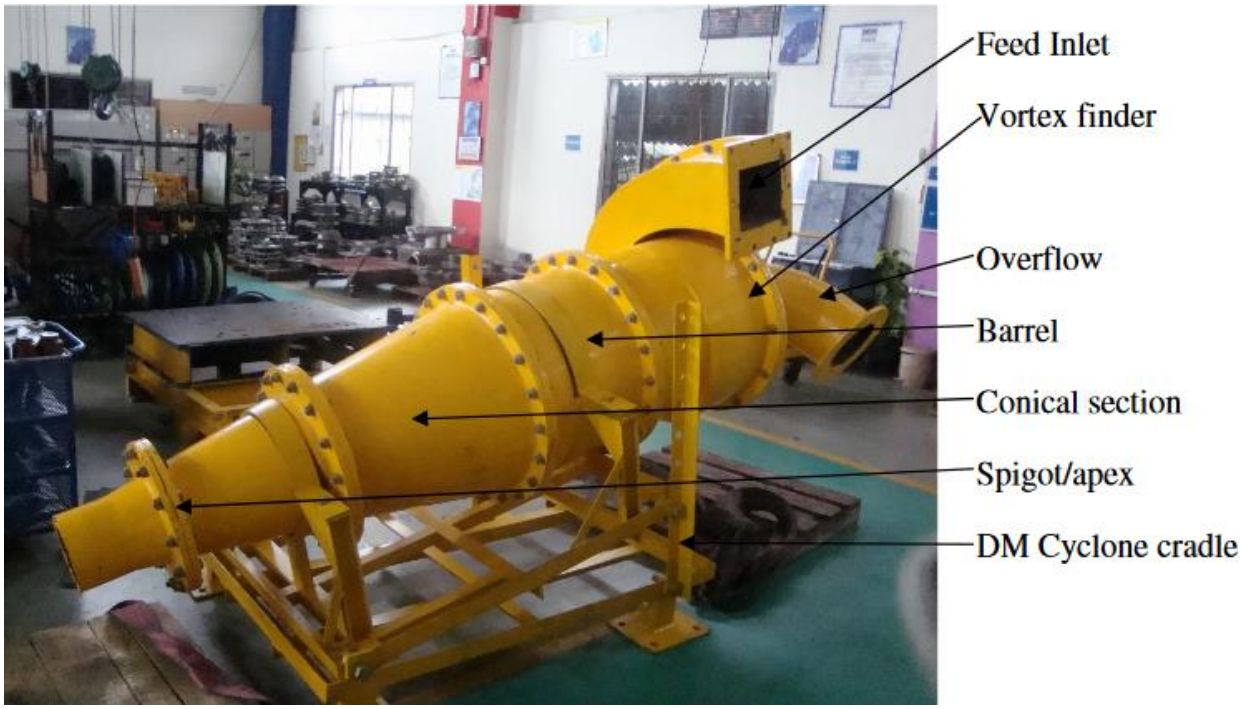


Figure 2.5 Principal Features of a DMS Cyclone Unit

DMS Cyclones utilise fluid pressure energy to create the rotational motion of the dense medium. This rotational motion is produced by the tangential injection of the dense medium and is responsible for effecting a relative motion of the feed suspended in the medium which permits the separation into clean coal and the discard. The feed coal is suspended in ultrafine ferrosilicon, FeSi or magnetite, Fe_3O_4 medium forming a pulp which is fed tangentially into the DMC through to a short cylindrical section carrying the vortex finder.

Separation then happens at the cone-shaped part of the cyclone otherwise known as the frustum. The frustum is extensible through the addition of a barrel section thus increasing the residence time within the cyclone which augments the sharpness of the separation of clean coal from the discard. Discard or sink material exits the cyclone through the spigot and the clean coal exits through the vortex finder.

The separation process occurring inside a DMC is presumed to be influenced by two key forces; the centrifugal force which acts radially outward and the drag force which acts radially inward.

The centrifugal force created inside the cyclone helps accelerate the settling rate of the feed coal's constituent particles, thereby achieving separation based on the specific gravity in the FeSi/ Fe_3O_4 suspension or medium.

The denser constituents of the coal composite are flung to the outer wall of the dense medium cyclone, a region with the lowest settling velocity. This material then spirals down along the wall of the cyclone until it exits at the spigot as an umbrella-shaped spray. A reverse vortex is initiated at the spigot, creating a low-pressure zone (air core) which flows upwards along the cyclone's axis, passes through the vortex finder and finally exits at the overflow.

The less-dense constituents tend to settle less rapidly because of the action of the drag force. As the reverse vortex flows upwards it captures this material thus it exits through the overflow. The density set medium density at which separation occurs is known as the cut-point. However, a proportion of the feed can get trapped in a zero-velocity region or envelope inside the cyclone where the drag force equals the centrifugal force. This material often referred to as near gravity material, NGM has an equal probability of reporting to either the underflow or the overflow.

Fig 2-6 gives a gravity separation circuit which utilises DMC equipment.

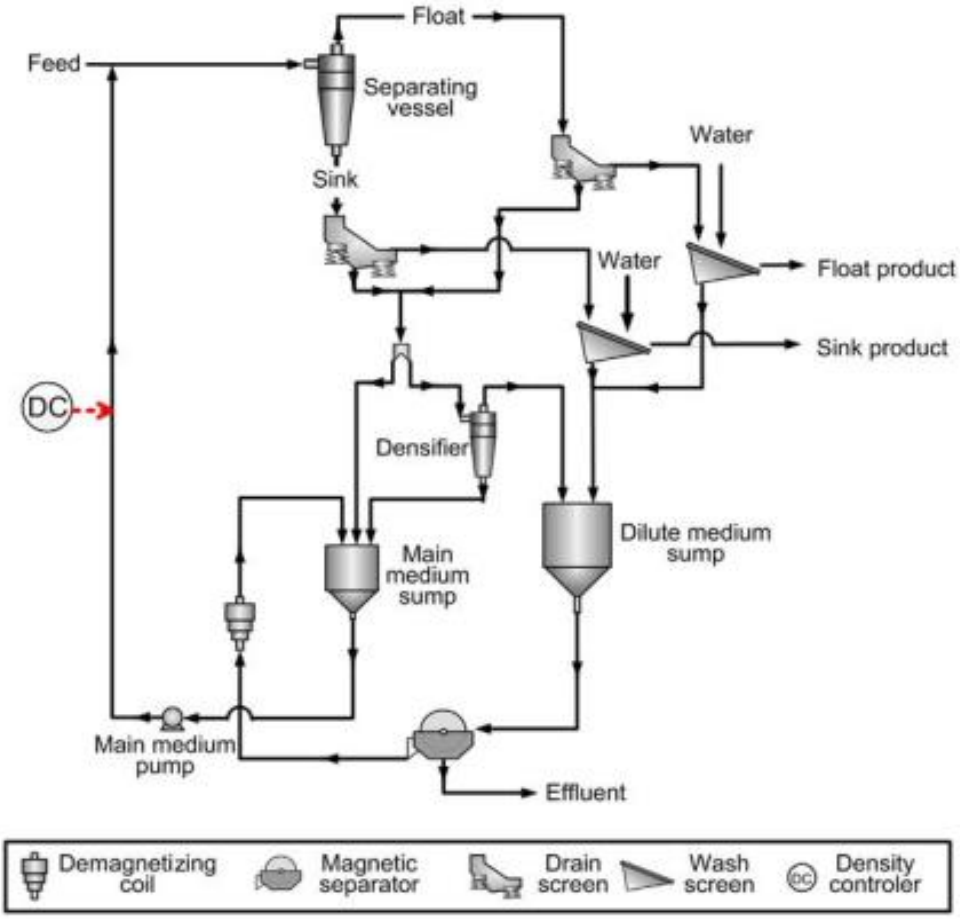


Figure 2.6 Coal Cleaning Circuit using a DMS Cyclone Gravity Separator (Wills & Finch, 2015)

2.5.3.3 Froth Flotation

Flotation is a hydraulic beneficiation technique that can achieve coal separation based on the manipulation of the physicochemical properties of the coal and or its associated mineral matter. Owing to the inability of South African coal washeries, which are gravity separator based, to clean coal of particle size distributions $< 500\mu m$, the prevalent practice has been to either discard it or blend it with steam coal despite the fact that this size class constitutes a likely source of high-grade coal that could find use as blend coking coal if beneficiated to an ash content of 7% (Fourie, Van Der Walt, & Falcon, 1980).

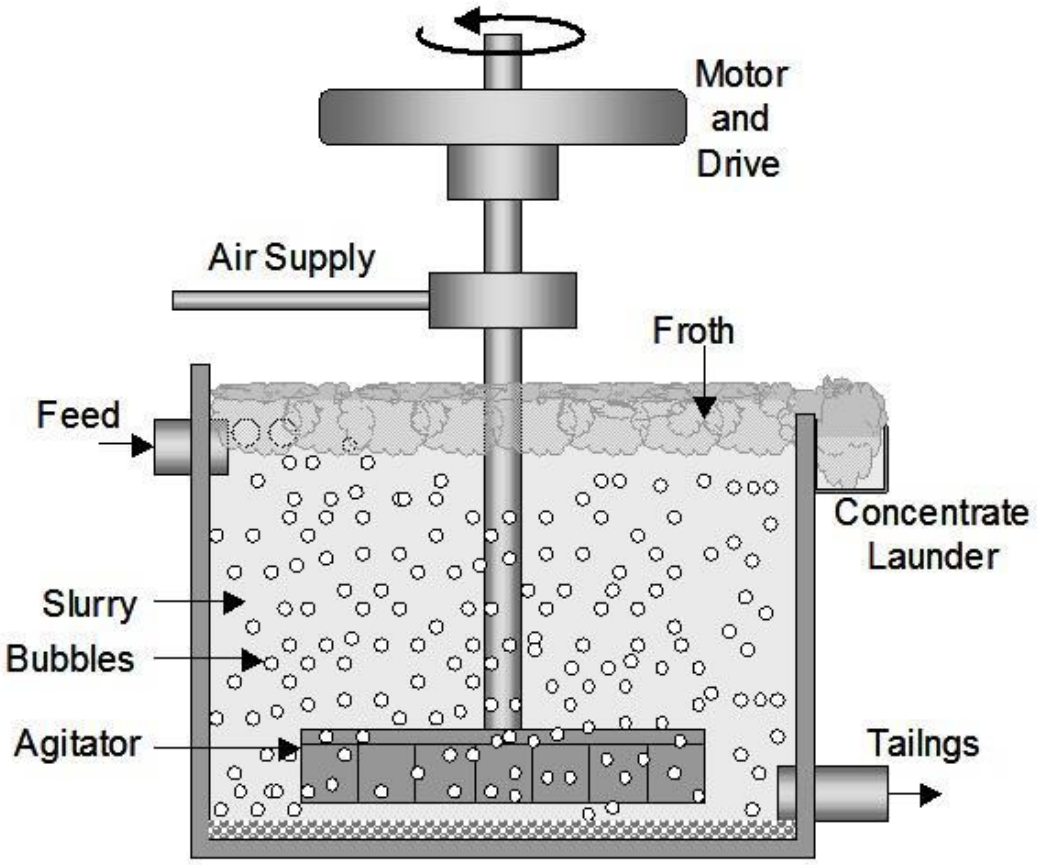


Figure 2.7 Principal Features of a Flotation Unit (Kawatra, 2011)

With coal from the Witbank Coalfield having dropped significantly in quality in recent decades, processing strategies must, therefore, adopt technologies which can effectively beneficiate the above-mentioned variants of coal discards. The best-known pilot plant for coal flotation in South Africa is at Goedehoop Colliery in the Mpumalanga Province which targets Witbank coals (Opperman & Nebbe, 2002). Full-scale operations have since been commissioned within the region.

Key to the flotation process, among other factors, is an understanding of how particles interact with gas bubbles in the pulp (Xing, Gui, Pan, et al., 2017a). Coal flotation is thus based on the preferential attachment of gas bubbles to coal particles (macerals) based on their surface hydrophobicity subsequently rejecting the mineral matter due to its surface hydrophilicity

The underlying principle applied to realise separation is the difference in density between the flotation medium and the air bubbles to which the coal particles and collector droplets attach. The resulting agglomerate is of lower density than the medium in which they are immersed hence it rises to the surface where the clean coal is then recovered through an overflow launder.

Attachment of gas bubbles to the surface is determined by the respective interfacial energies of the gas, liquid and solid phases, as shown by the Young Equation;

$$\gamma_{SG} = \gamma_{SL} + \gamma_{LG}cos\theta \quad \text{.... Eq 2-5}$$

γ_{SG}, γ_{SL} & γ_{LG} are the energies at the interfaces solid-gas, solid-liquid and liquid gas respectively and θ the contact angle at the junction between the gas, solid, and liquid phases.

Due to the strong affinity of the solid phase (coal) for the gas bubbles, the interaction of the solid-gas phase advances replacing the solid-liquid interface which alters the Gibbs free energy, ΔG as is expressed by the Dupré Equation;

$$\Delta G = \gamma_{SG} - (\gamma_{SL} + \gamma_{LG}) \quad \text{.....Eq 2-6}$$

The two equations in combination then give the Gibbs free energy change, per unit area, as;

$$\Delta G = \gamma_{LG}(cos\theta - 1) \quad \text{.....Eq 2-7}$$

This equation, also known as the thermodynamic criterion of flotation shows that ΔG will lower with an increase as the values for γ_{LG} and θ. At $\theta = 0, \Delta G = 0$ thus particle-bubble attachment cannot occur. Particle-bubble attachment is therefore thermodynamically feasible once $\theta > 0$ which makes $\Delta G < 0$. The strength of the particle-bubble attachment, therefore, increases with an increase in the contact angle. A contact angle of 90° is often desirable for most froth flotation practice. Introduction of collector reagent into the system often serves to increase the contact angle between the gas bubble and the surface of the particle.

Reagents are a fundamental part of any flotation process. Collectors augment the hydrophobicity of coal through physisorption, a reversible association on the surface of the coal particle due to the formation of weak Van der Waal forces and hydrogen bonds, thus increasing the separability of the coal from mineral matter which is more hydrophilic and thus has a greater affinity for the aqueous phase. Increased hydrophobicity improves coal particle-gas bubble attachment.

A low dosage means reduced particle-bubble attachment hence the selectivity of the process drops leading to a poor yield of the concentrate and a sink that is still rich in carboniferous material. When the collector dosage in excess this can also contribute to a poorer process performance than that at the optimum dosage. This phenomenon can be interpreted in terms of how the equilibrium between collector at the surface and collector in solution impacts collector adsorption. The form of adsorption is physisorption, a reversible process which requires relatively low activation energy; $E_A = 5 - 40kJ/mol$.

Adsorption occurs readily at low collector dosages due to the presence of excess available coal surface area for attachment. The surface becomes increasingly hydrophobic with the addition of more collector thus leading to an increase in the extent of adsorption. Competition between collector molecules for the available sites increases, forcing the formerly adsorbed species to be positioned in a vertical orientation that promotes close packing of the molecules on the surface, hence exposing surface sites which might have been covered by the initial collector molecules.

Interaction of the hydrophobic tails thus ensues from the close packing resulting in increased stability of the adsorbed collector, a phenomenon known as hemimicelle formation. Sites for adsorption thus become increasingly scarce, thus adsorption starts removing less and less collector till the point where the surface is incapable of accepting more collector, the point of

monolayer coverage. Gangue uptake to the float fraction may begin to happen in some instances.

Hydrocarbon oils are the most common collectors in coal flotation, posing the advantages of selective attachment to the coal particles without altering the surface properties of the associated mineral matter at a relatively cheap cost in comparison with patented non-ionic collectors. In addition, they can be co-used as extenders for more expensive non-ionic collectors due to their synergistic effect (Kawatra, 2011). Diesel, kerosene, and lubricants like bright stock and spindle oil are some of the classic collectors which have been proven through extensive studies to be effective collectors or extenders in coal cleaning (Sönmez & Cebeci, 2006).

Frothers are reagents of low collecting power which are added to the pulp or slurry to produce and stabilise the foam, reduce the surface tension at the gas-liquid interface. They also help prevent the gas bubbles generated from coalescing allowing them to rise to the top carrying hydrophobic coal particles, forming the froth layer which exits at the overflow launder bubble-collector agglomerate hence failure of the clean coal to fully report to the frother phase for collection. Determination of the minimum frother dosage requirement (critical coalescence concentration, CCC) is critical to ensure coalescence is avoided.

Several creosote fractions from coal tar distillation have been found to be able to multi-task as both frothing agents as well as froth stabilisers. Pine oil has been widely used as the frothing agent, in conjunction with petroleum products acting as collecting agents. Another creosote fraction, turpentine, can also be alternatively used for the generation and stabilisation of froth (Babatunde & Adeleke, 2014).

Another group of auxiliary reagents applicable to coal flotation is the modifiers which influence the way in which the collector attaches to the coal particles namely depressants and pH modifiers. To increase the sharpness of the separation, it sometimes is necessary to add a depressant which augments the hydrophilicity of some mineral constituents thus preventing their flotation. Sodium silicate, Na_2SiO_3 is a clay depressant that is commonly used in coal flotation to depress silicate and carbonate minerals.

However use of this reagent requires knowledge of the module number, SiO_2/Na_2O ratio which determines whether its mode of action will be depressant or dispersant (Taner & Onen, 2016).

pH modification is often done to obtain alkaline conditions since most collectors are stable in that region and the corrosion of the flotation cell and associated piping is minimised under alkaline conditions.

Senfroth 200 and Sendep 30F, a frother and a depressant correspondingly are locally manufactured products which theoretically possess potential in coal flotation. Senfroth, a water-soluble frother which is synthesised from ethylene oxide or propylene thus fundamentally should be capable of serving as both a frother and weak collector in coal flotation. Sendep is a polysaccharide-based depressant which acts by inducing hydrophilicity to the gangue and thus retaining the gangue in the aqueous phase (Senmin, 2019).

2.5.4 Chapter Summary

From the complex nature of the coal that has been reviewed, it is not surprising that there are unlimited possibilities on how to beneficiate coal. For this research project, gravity separation and flotation were chosen based on equipment availability and maturity of both technologies.

CHAPTER 3
METHODOLOGY

In this section, a suite of experiments is given which help investigate the research problem. All beneficiation tests were performed in the Peter King Mineral Processing Laboratory and chemical analyses were done in the Coal Laboratory, both of which are within the School of Chemical and Metallurgical Engineering. Petrographic analysis was done at the University of Johannesburg, Department of Geology.

3.1 Sampling and Sample Preparation

A 20 kg sample of a low-grade discard ROM coal was obtained from Ingwe Collieries which is situated within the Witbank Coalfield. The coal had been mined, crushed to $-31.5\ mm$ and deposited in-situ for a period of $+6$ months. Associated chemical analysis results indicated that the coal supplied conformed to the specifications of this investigation. The sample was split into four identical samples using a rotary sampler. One sample was reserved for referral purposes.

All proximate and calorific value analysis samples were size reduced using a jaw crusher and/or a pulveriser to a d_{80} passing size of $< 200\mu m$. The pulverised samples were then mixed thoroughly to ensure homogeneity, after which test portions were then taken for analysis. For petrographic analysis, the samples were milled to a size ranging between $1000\mu m < x < 30\mu m$ and sent for further preparation at the UJ sample preparation laboratory according to (SANS 7404-2, 2015). Flotation feed was milled to a d_{80} passing size of $< 300\mu m$ and float-sink testing material was size reduced to two discrete size fractions of $1180\mu m < x < 4500\mu m$ and $4500\mu m < x < 6500\mu m$ range.

3.2 Material Characterisation Experiments

3.2.1 Particle Size Analysis

A representative 5kg sample was obtained from the 20kg laboratory sample obtained using a sample splitter or riffler. Using a rotary sampler, a 500g representative sample was obtained. Size analysis of the sample was done using a stack of sieves with aperture sizes $9500, 6500, 4500, and\ 1180\mu m$ to determine the mass distribution of the sample and the difference in grade based on particle size. The sieving test was run for 5 minutes before removing the sieve and weighing the mass retained on each screen.

3.2.2 Chemical Analysis

The chemical composition of the samples analysed was determined through calorific value analysis (SANS 1928, 2009) and proximate analysis. Thermogravimetric analysis was used to determine the proximate parameters of the samples.

3.2.2.1 Calorific Value Analysis (SANS 1928, 2009)

A DRYCAL Bomb Calorimeter which measures gross calorific value (GCV) according to (South African National Standards (SANS) 1928, 2009) was used. Duplicate measurements of the GCV were taken to ensure reproducibility of the obtained results. Recalibrations of the instrument were conducted using benzoic acid tablets after every 10 samples to ensure instrument accuracy. Greater detail of the test procedure is given in Section A1 of the Appendix. Fig 3-1 shows the instrument used and its general schematic.

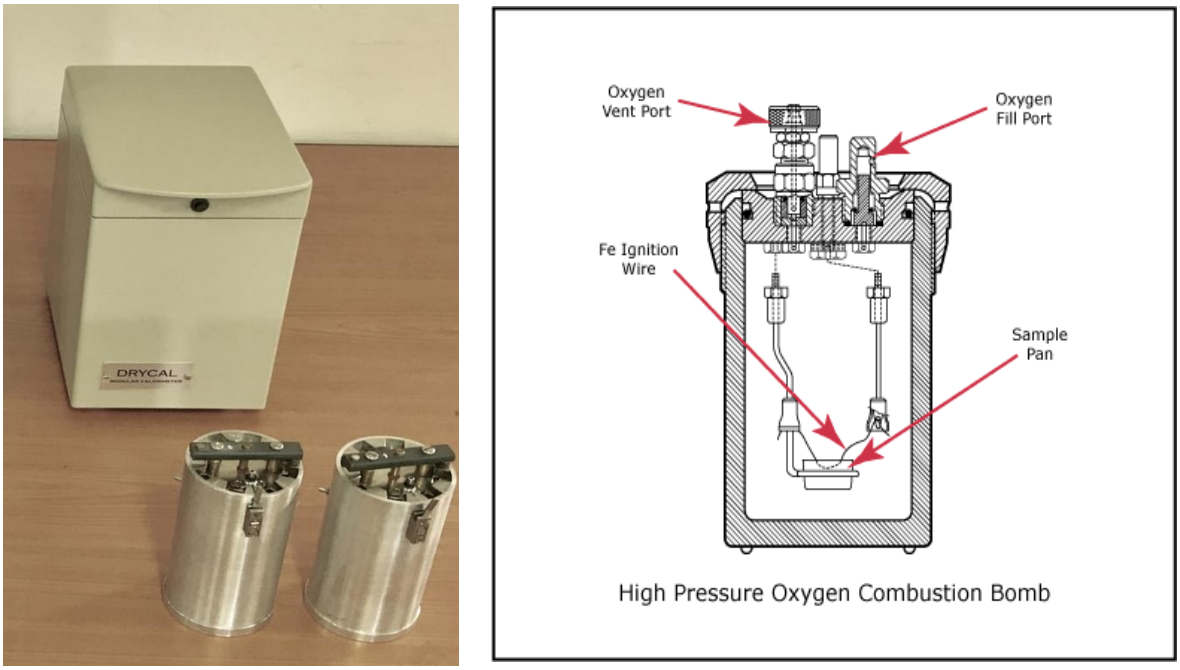


Figure 3.1 General Schematic and Appearance of a Bomb Calorimeter

3.2.2.2 Proximate Analysis (Micro-Thermogravimetric Method)

An SDT Q600 V20.9 Build 20 Thermogravimetric Analyser (TGA) was used in proximate analysis. Analysis for fixed carbon, inherent moisture, volatile matter and ash content was conducted on a 10mg sample according to a set program. This required a total time of 44 minutes to complete. Greater detail of the test procedure is given in Section A1 of the Appendix. Fig 3-2 shows the TGA equipment used.

Figure 3.2 SDT Q600 Simultaneous TGA/DSC Instrument

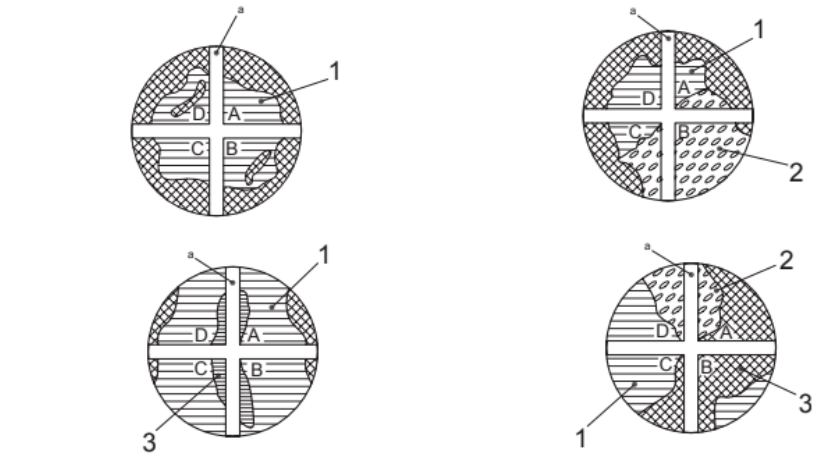
3.2.3 Petrographic Analysis

The petrographic analysis was done according to (SANS 7404-3, 2016; SANS 7404-5, 2016) using a Zeiss AxioImager M2M Reflected Light Polarising Microscope accompanied by Hilgers Diskus Fossil System software and Student Fossil for offline image analysis. The instrument was operated with a total magnification of $500\times$ magnification under oil immersion. The analysis was used for maceral, mineral and rank determination. Such analysis was done on the feed sample and the $1180\mu m \leq x \leq 4500\mu m$ and 14g Diesel/kg Coal products from sink-float and flotation analysis respectively.

3.2.3.1 Maceral and Mineral Groups Determination (SANS 7404-3, 2016)

SANS 7404-3 (2016) was used in maceral analysis which involved determination of the maceral groups present and the mineral groups clay, quartz, carbonates and pyrite. A sample prepared according to (SANS 7404-2, 2015) was levelled, set up on a stage, a drop of immersion medium added onto the block's surface, the microscope focused and then an image was observed. The material lying under the intersection of the crosshairs was then identified using a point count procedure. When the crosshairs went on either vitrinite, liptinite or inertinite the point counter was triggered but when it went on the mounting medium or an empty pore in a maceral void, the point was ignored. In the case that the crosshairs went on a boundary between macerals or maceral-mounting medium boundary, the material immediately adjacent to the intersection of the crosshairs in the top right quadrant going anticlockwise to the top left quadrant was examined and the point counter was focused on the first quadrant without a boundary in it. Block movement was then made by a single step in an L-R direction and counting performed as the specimen was traversed. When the traverse ended, the block would then be advanced in a perpendicular direction by step of the same length from whence the next parallel traverse would start. Step length was chosen to ensure uniform point counting over the

surface. The analysis was considered complete when at least 500 points had been counted, excluding minerals.



Key

1 vitrinite
2 inertinite
3 liptinite

[a] Width of crosshairs exaggerated for clarity.

Figure 3.3 Normal and Boundary Cases Between Macerals or Maceral and Mounting Medium

3.2.3.2 Rank Determination by Vitrinite Reflectance (SANS 7404-5, 2016)

The determination of rank category was conducted on the 1.5 SG sample. No reflectance readings were taken on the original sample or on other products because they had insufficient vitrinite maceral for such analysis. Calibration was done using an yttrium aluminium garnet (YAG) standard with a $0.895 - 0.916\%$ reflectance. A levelled polished block onto which immersion oil has been applied was then placed on the mechanical stage to permit the making of measurements and the microscope focused at one corner of the section to be traversed. The stage was then moved till the crosshairs focused on a carefully selected area with vitrinite which was without maceral boundaries, cracks, mineral inclusions, polishing defects or relief effects. 100 reflectance readings were taken in accordance with SANS 7404-5 (2016).

3.3 Beneficiation Tests

Float-sink testing and flotation experiments were conducted to obtain data of value in determining the response of the associated beneficiation techniques in upgrading the low-grade discard coal.

3.3.1 Float-Sink Test Procedure (SANS 7936, 2010)

An inorganic salt, sodium polytungstate (SPT), $Na_6[H_2W_{12}O_6]$ *or* $3Na_2WO_4 \cdot (WO_3 \cdot H_2O)$ was first used to make aqueous solutions of specific gravity (SG) 1.5; 1.6; 1.7 & 1.8 using $250ml$ beakers as density baths. Suitable hydrometers were used to ensure that the relative density in all the baths was within the range ± 0.002 of the intended bath density throughout the experiment. A $500g$ test sample was selected from $1180\mu m < x < 4500\mu m$ size fraction. The sample was then added in $100g$ increments into the $1.5SG$ bath and agitated gently, taking care not to overload the bath so that separation of entrained near density material would not be interfered with. After 3 minutes, the float fraction was collected and drained, and the sinks agitated to ensure there were no entrained floats after which the procedure was repeated until the test sample was finished. All the float material was washed free of the dense medium, dried in air and weighed. The total sink fractions were drained thoroughly to avoid contamination of the next bath and then transferred into a bath of the subsequent higher density $(1.6SG)$. The float and sinks were treated in a similar manner to the first fraction. This procedure was repeated until the test sample had gone through all the density baths. Following the completion of the test at the above-mentioned size fraction, the relative densities of all the baths were recalibrated to range ± 0.002 of the intended bath density. Thereafter, the process was then repeated using a new $500g$ test sample of size fraction $4500\mu m < x < 6500\mu m$.

3.3.1.1 Gravity Separator and Partition Model Selection

The data obtained from float-sink analysis was used in selecting the most appropriate equipment for gravity separation of the low-grade coal. EXCEL SOLVER was used to determine the mathematical model which best represents partitioning of the coal in the equipment chosen. Real performance upon gravity separation was then determined based on float-sink data which was taken to represent perfect/ideal separation.

3.3.2 Froth Flotation Procedure

A Denver D12 Laboratory Flotation Machine was used in all tests. Impeller speed, flotation cell size, frother (Senfroth) dosage, air flow rate, amount of feed and feed particle size distribution (PSD) were kept constant. Distilled water was first measured, up to the mark, into a $3L$ flotation cell. Thereafter $300g$ of the test sample milled to a $-300\mu m$ size and diesel collector of dosage of $7g/kg\ Coal$ were added to the cell and then the impeller shaft was lowered into the cell. The cell was then agitated at an impeller speed of $1100rpm$ for $4min$ after which $0.125ml$ of frother were added and agitation continued for another minute. Thereafter air was allowed into the cell at an air-flow rate of $38.4ft^3/h\ (^{18L}/_{min})$. Collection of the initial float fraction was done in the first $1min$ followed by collection in the next $2min$ and the last collection in the ensuing $6min$, bringing the total flotation run time to $9min$. The float and sink fractions were then dewatered using a laboratory filter press, dried in an oven operated at 110℃, and then prepared for subsequent analysis. This procedure was then repeated using higher diesel collector dosages of $14g/kg\ Coal$ and $21g/kg\ Coal$. The procedure was then repeated with diesel collector dosage at $14g/kg\ Coal$ but in addition, a depressant, Sendep was used at two different dosages of $5g/kg\ Coal$ and $10g/kg\ Coal$. Another parallel experiment was also performed, maintaining the set constants and using a

more-polar collector, tetrahydrofuran (THF), in place of diesel. Fig 3-4 gives the flotation and drying equipment used.

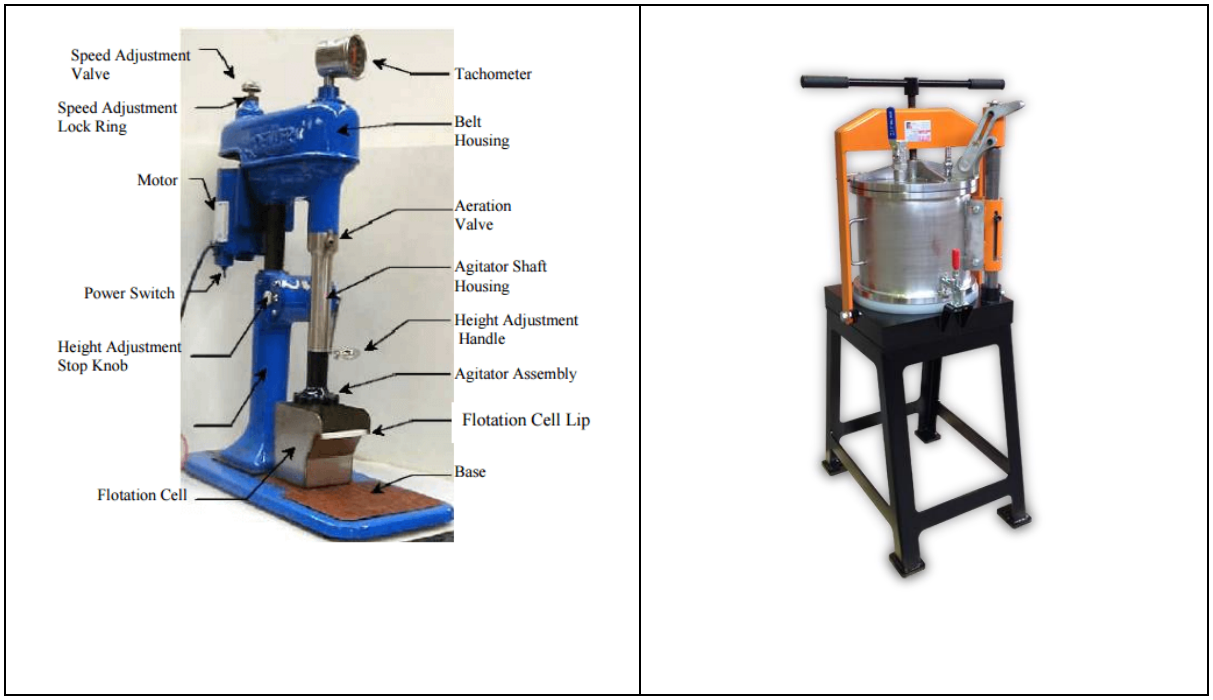


Figure 3.4 (L-R) Denver D12 Laboratory Flotation Unit and Laboratory Filter Press

CHAPTER 4
RESULTS AND CALCULATIONS

Key results are presented for the purpose of the discussion, the rest are included in the Appendix.

4.1 Sample Characterisation

A sample of low-grade discard ROM coal was received from Ingwe Collieries in the Witbank Coalfield. In this section, results that characterise the sample that was received are presented.

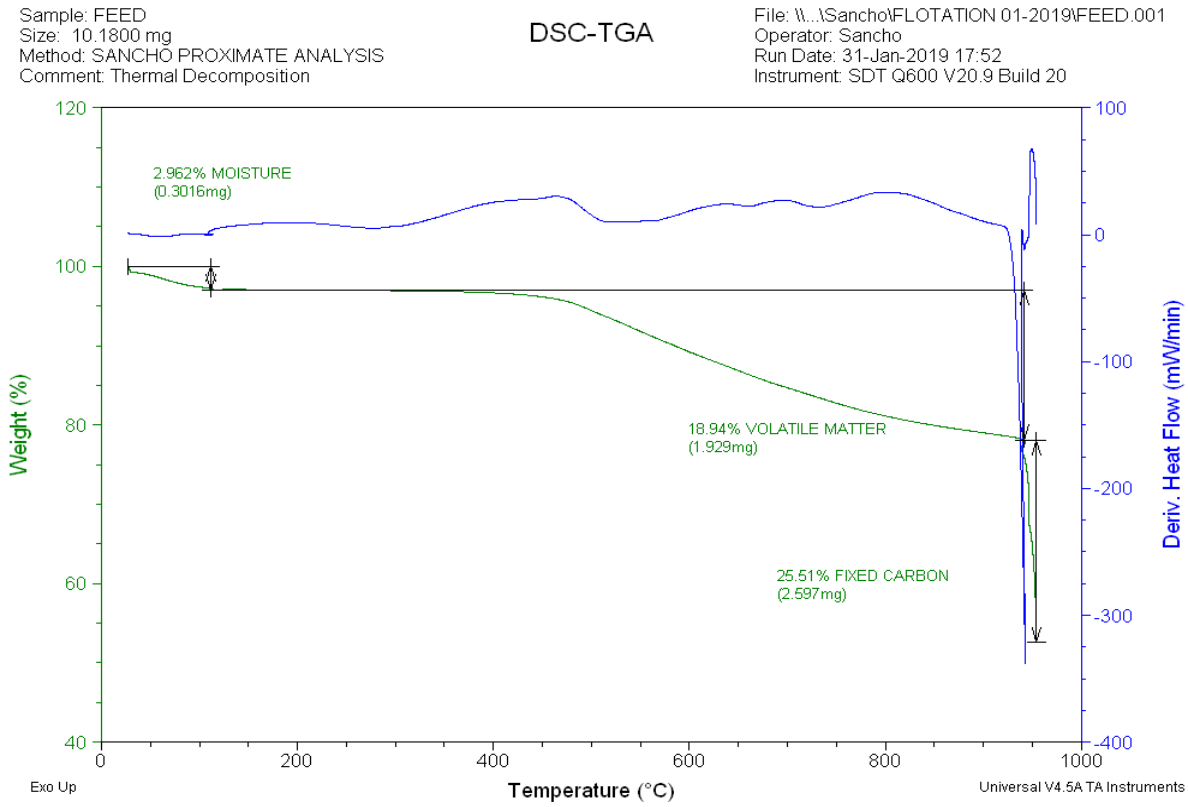


Figure 4.1 TGA Plot Showing Proximate Analysis Results for the Feed Coal

SIZE RANGE (µm)	MASS (%)
-1180µm	3
+1180 -4500µm	27.25
+4500 -9500µm	28
+9500µm	41.75

Figure 4.2 Mass Distribution of Feed Coal

4.2 Gravity Separation Equipment

Based on sink-float data, the dense medium cyclone (DMC) is the most appropriate gravity separator for the coal. Through modelling trials using EXCEL SOLVER, the modified Whitten equation was found to be the best-suited model to represent partitioning in the DMC. Published data on DMC performance was used to estimate the expected performance on the data obtained. Using an Ep value of 0.05 determined from related experimental data and cut-point, $\rho_{50} = 1.6; 1.7$ from current studies, a partition or Tromp curve was drawn, offset using the modified Whiten Equation to best model separation in a DMC. PN_i and ρ_i are the respective ith sink-float fraction density and the corresponding partition number. ρ_{50} is the cut point.

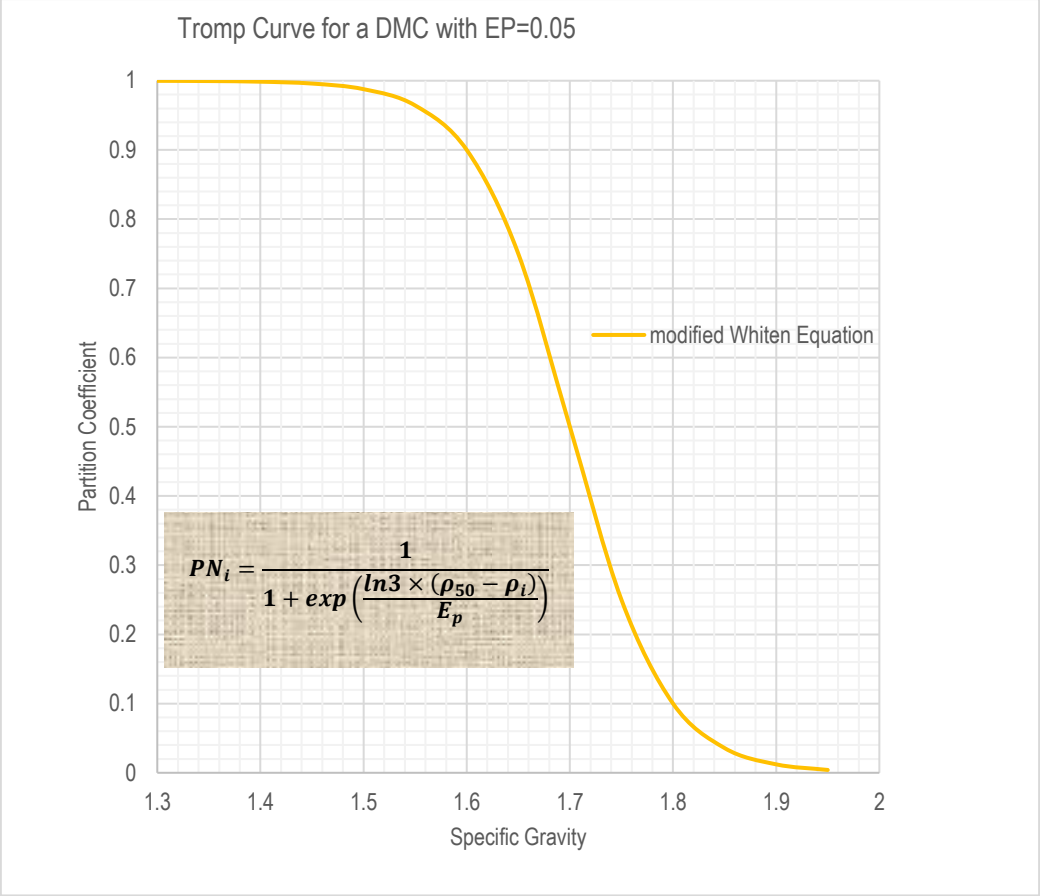


Figure 4.3 Tromp Curve for DMS Cyclone Performance with an Ep=0.05, $\rho_{50} = 1.7$

Table 4-1 DMC Model-Based Partitioning of 1180µm≤x<4500µm Sized Coal at 1.7 SG

Specific Gravity Fraction	Mass	Ash	Mass × Ash	Partition Coefficient	Float Mass	Mass of Ash
[1]	[2] (%)	[3] (%)	[4] [2]×[3]	[5]	[6] [2]×[5]	[7] [3]×[6]
<1.5	6.74	15.90	107.10	100	673.70	10709.81
1.5-1.6	13.66	26.49	361.82	89.99978	1229.27	32563.39
1.6-1.7	24.79	28.14	697.65	50	1239.47	34882.40
1.7-1.8	16.28	39.24	638.72	10.00022	162.79	6387.29
>1.8	38.54	63.32	2439.96	0	0.00	0.00

Table 4-2 DMC Model-Based Partitioning of 4500µm≤x<9500µm Sized Coal at 1.7 SG

Specific Gravity Fraction	Mass	Ash	Mass × Ash	Partition Coefficient	Float Mass	Mass of Ash
[1]	[2] (%)	[3] (%)	[4] [2]×[3]	[5]	[6] [2]×[5]	[7] [3]×[6]
<1.5	9.13	18.31	167.23	100	913.40	16723.44
1.5-1.6	13.94	24.73	344.77	89.99978	1254.69	31029.66
1.6-1.7	18.83	32.63	614.46	50	941.70	30722.96
1.7-1.8	18.51	41.21	762.71	10.00022	185.08	7627.32
>1.8	39.58	63.55	2515.64	0	0.00	0.00

Table 4-3 Theoretical and Predicted DMC Yield and Grade at Select Cut Points

VALUE	Yield				Grade (Ash)			
CUT POINT SG	1.6		1.7		1.6		1.7	
	Theoretical	Predicted	Theoretical	Predicted	Theoretical	Predicted	Theoretical	Predicted
	$\sum[2]$ to SG1.6 (%)	$\frac{\sum[6]}{100}$ (%)	$\sum[2]$ to SG1.7 (%)	$\frac{\sum[6]}{100}$ (%)	$\frac{\sum[4] \text{ to SG1.6}}{\sum[2] \text{ to SG1.6}}$ (%)	$\frac{\sum[7]}{\sum[6]}$ (%)	$\frac{\sum[4] \text{ to SG1.7}}{\sum[2] \text{ to SG1.7}}$ (%)	$\frac{\sum[7]}{\sum[6]}$ (%)
1.18-4.5mm	20.396	23.760	45.185	42.570	22.991	24.229	25.817	26.754
4.5-9.5mm	23.075	24.959	41.909	41.330	22.189	24.306	26.879	27.855

4.3 Flotation Data

Flotation experiments were conducted to test the viability of this technique in upgrading the low-grade coal. From the results comparing recoveries from different diesel dosages and THF dosage, it was observed that diesel performance at a dosage of 14g/kg Coal was the best. This is the result considered in the rest of the evaluations. More results are given in the Appendix.

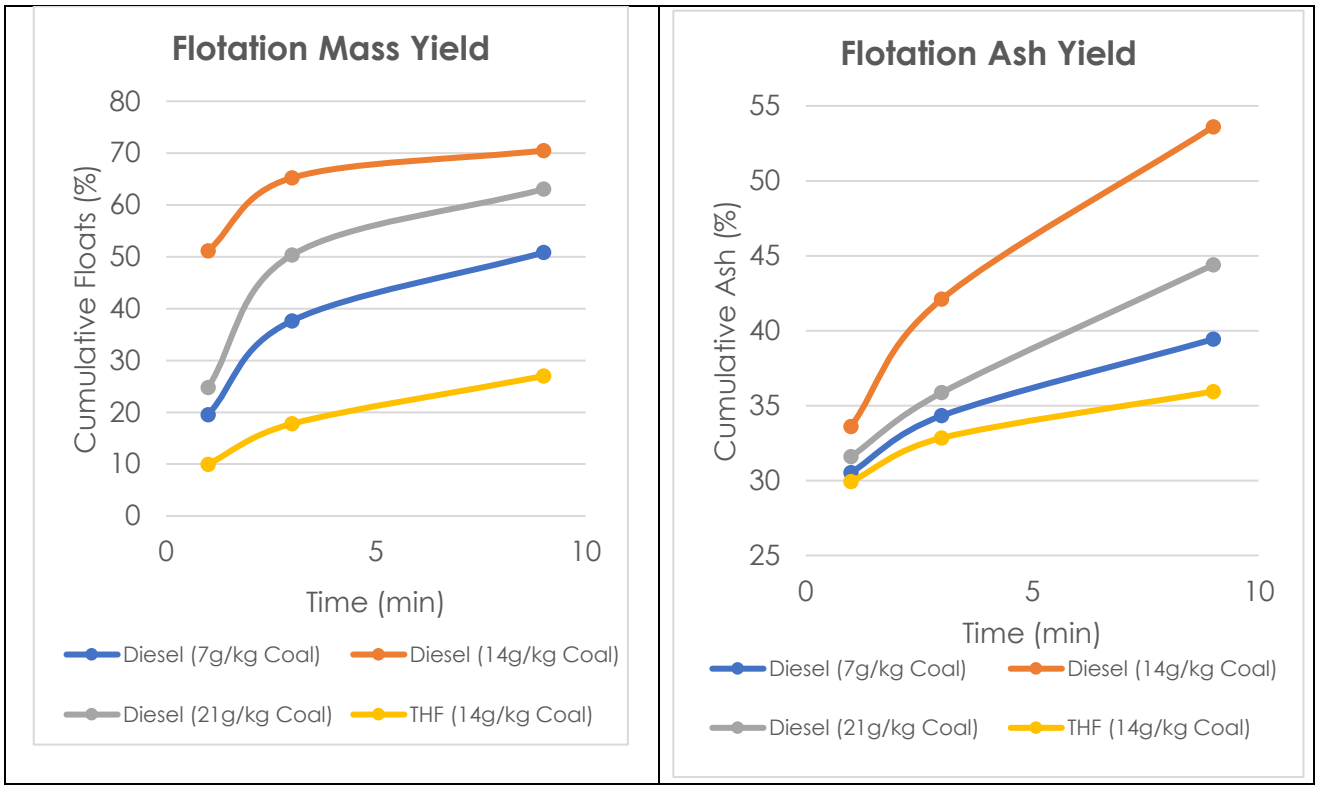


Figure 4.4 Flotation Yield Evaluation

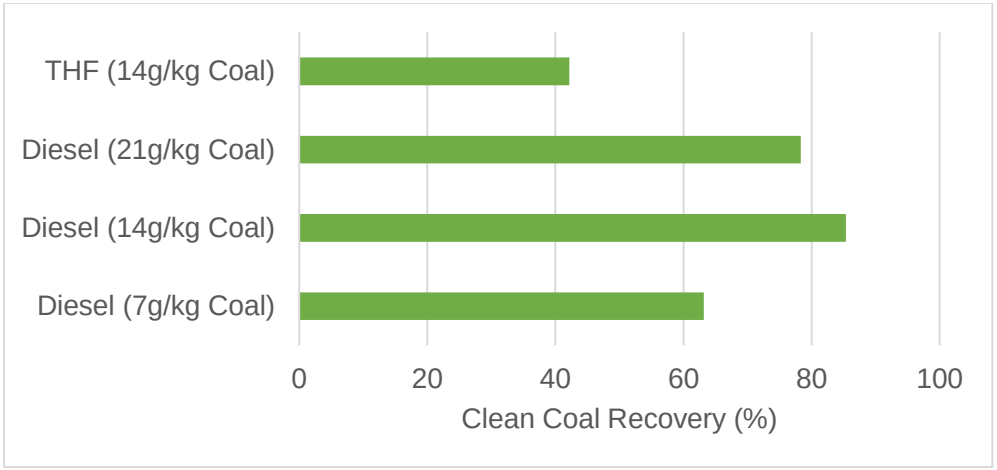


Figure 4.5 Flotation Clean-Coal Recovery Evaluation

CHAPTER 5
DISCUSSION

5.1 The Low-Grade Coal Conundrum

The quality of coal is vital to efficient operations. With increasing consumption over time, the quality of coal found in the Witbank Coalfield of the Mpumalanga province has significantly deteriorated. The impact of the decrease in coal grade is manifested through an upsurge in the cost of equipment maintenance and operational challenges like damage on coal handling and comminution equipment, reduced power plant boiler efficiency and subsequent load loss and an increase in the ash removal burden (Eskom, 2019).

Most industries have been invariably affected by the challenges faced by Eskom which can to some extent be attributed to the decrease in coal quality. Mining and metallurgical industries, key contributors to the national gross domestic product, GDP have been inevitably affected by this change with the latter's woes augmented by the increased need for the importation of coking coal thus increasing the cost of local steel and subsequently leading to a reduced ability to compete for the global steel market share. Fig 5-1 shows the key sectors of the economy that need electricity and their respective % consumption in 2012.

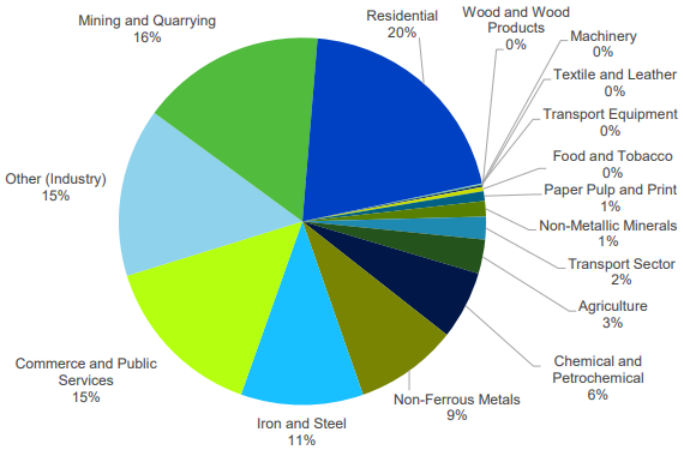


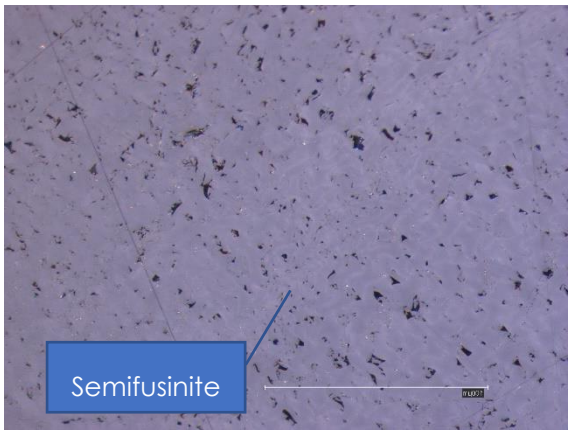
Figure 5.1 Electricity Consumption by Sector, 2012 (Deloitte, 2017)

Low-grade coal beneficiation has thus become an inescapable necessity, with the situation expected to worsen going forward. The aim of this research was the determination of the beneficiation potential of a low-grade discard ROM coal from the Witbank Coalfield. Based on the objectives of this research, it was imperative that the characteristics of the coal and products from beneficiation tests be determined using both petrographic and chemical analysis to fully evaluate process efficiency and predict the potential technological performance of the product.

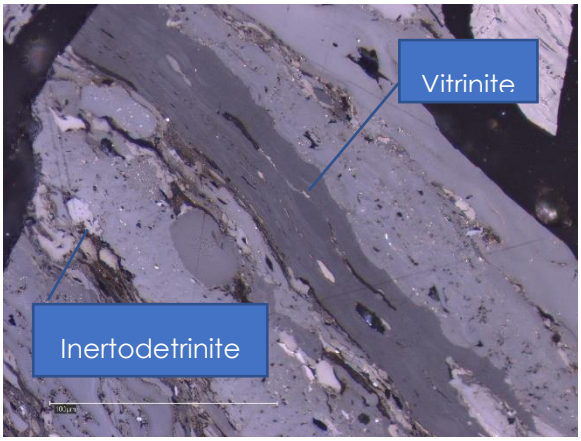
In this section, a brief discussion on the implications of the findings from characterisation experiments conducted on the as-received coal will be ventured into after which the beneficiation techniques utilised, gravity separation and froth flotation will be discussed starting with the selected procedures and then focusing on the results obtained and their inferences.

5.2 Assessment of Feed Coal

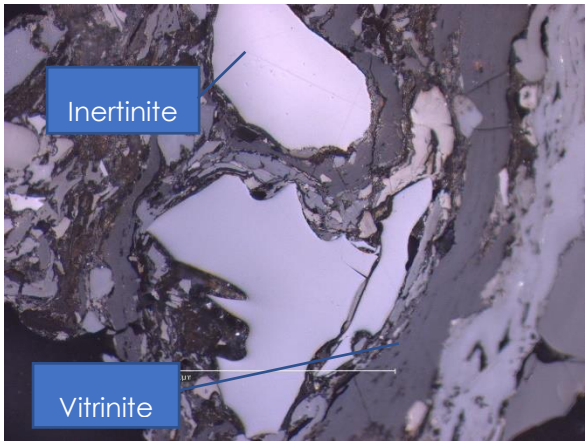
The coal used in this investigation is a discard low-grade ROM coal obtained from Ingwe Collieries in the Witbank Coalfield. Petrographic analysis results, given in Tables A2-11 and A2-12 confirmed that the sample is low-grade, having 46.1vol% minerals. The most abundant maceral determined was inertinite (48.8 vol%) and the rank category is Medium-Rank C Bituminous. RoV analysis was conducted on the 1.5 SG float fraction from float-sink testing since the amount of vitrinite in the feed sample, 4.3vol% was inadequate for analysis but had risen to 14.0vol% in that fraction thus providing an opportunity to get a more-accurate RoV classification. There is very little liptinite, 0.8vol% which firmly suggests that the feed is not sapropelic but rather is humic coal. Fig 5-2 shows petrographic photographs of the coal showing both macerals and minerals present in both excluded and included forms.



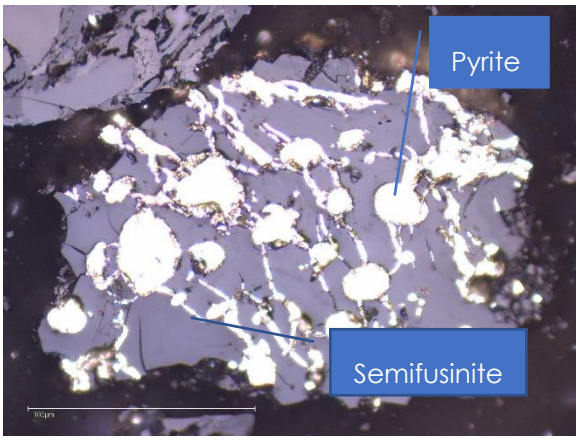
Clean Semifusinite (Inertinite)



Clean Layers of Vitrinite and Inertodetrinite



Vitrinite and Inertinite Macerals with Included Clay and Quartz



Semifusinite with Included Pyrite

Figure 5.2 Petrographic Photographs of Feed Coal [500X, Oil Immersion, Scale Bar 100µm]

From the above photomicrographs, the major processing challenges would be related to the liberation of clays, quartz and pyrite which exist in both included and excluded forms, particularly syngenetic clay which exist as ultra-fine grains. Based on the mineralogical analysis on coals from this region, the clay mineral present is predominantly kaolinite occurring together with montmorillonite and illite in fewer proportions (Pinetown et al., 2007). It was observed that the carbonates in the sample exist mainly in excluded form and are quickly liberated during comminution.

Evidence of partial oxidation was noted on photomicrographs containing carbonate minerals. The coal may be partially oxidised due to exposure to atmospheric conditions in the discard,

the extent being a factor of oxidising conditions and duration of exposure. Figure 5-3 gives photomicrographic evidence of partial oxidation as observed on the carbonates in the coal.

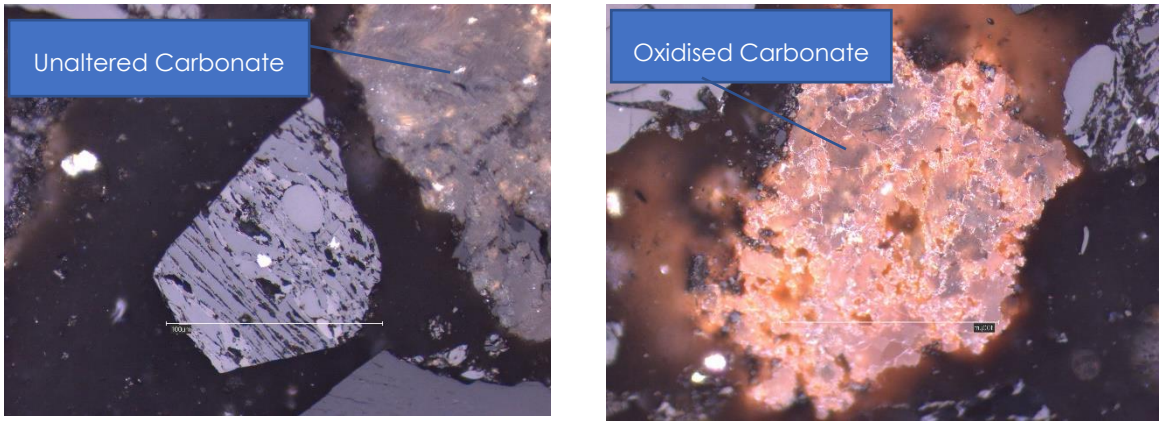


Figure 5.3 Evidence of Partial Oxidation [500X, Oil Immersion, Scale Bar 100µm]

The information obtained on volatile matter and inherent moisture content (Table A2-2) indicates a close synergy of some proximate parameters between the overall feed and the size-classified feed used in gravity separation (sink-float) tests. Fig 5-4 shows the impact of feed size classification on proximate parameters.

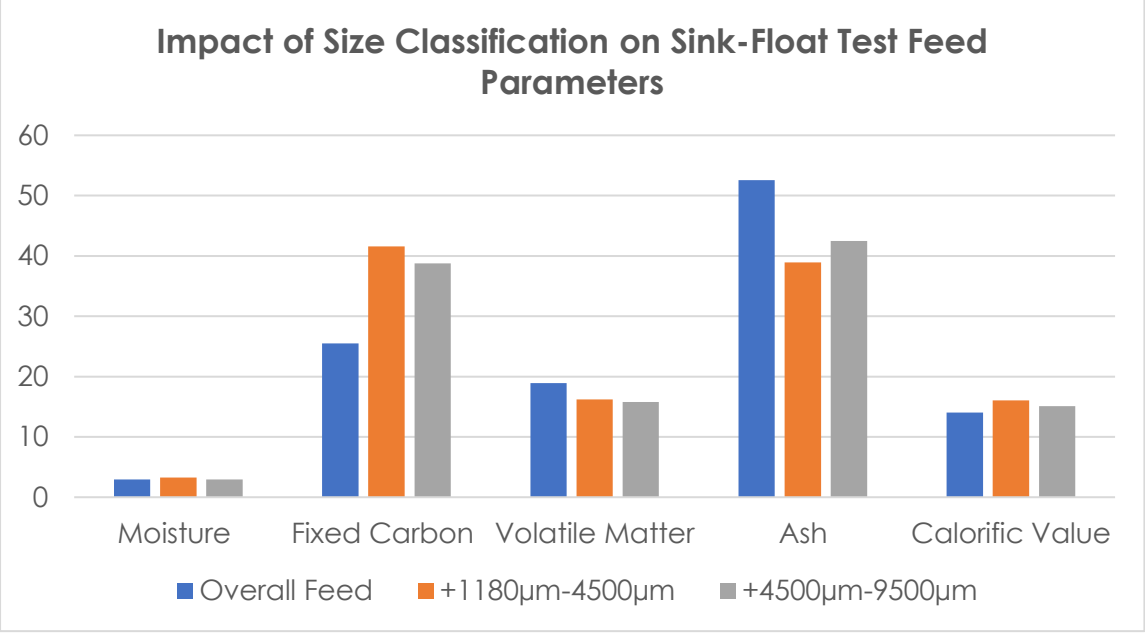


Figure 5.4 Impact of Size Classification on Sink-Float Test Feed Parameters

Volatile matter and inherent moisture parameters are closely related in both size-classified feeds which varies slightly from the data obtained for the overall feed. Considering that moisture is low (<4%) in all cases, ash is, therefore, the primary heat consumer during combustion. Fixed carbon in the overall feed varies by a significant margin (>13%) compared to both size-classified feeds.

Fixed carbon and subsequently ash are not evenly distributed among the size classes. The uppermost size class +9500µm due to having the highest mass proportion (41.75 wt%) contains the greatest quantity of ash despite having the second-lowest grade (49.4 wt%). Though of the lowest grade (56.5 wt%), the -1180µm size class does not contain the most ash due to its lesser mass proportion (3 wt%). With negligible economic impact, this size class can be rejected at this stage to lessen the processing burden.

Petrographic results (Table A2-12) confirm the large abundance of mineral matter (46.10 vol%) in the feed thus confirming that the coal is low-grade. Quartz, the most abundant mineral group in the feed (29.50 vol%) was found primarily in liberated form suggesting that it was either obtained from inorganic intrusions into the coal bed or from the roof of the coal seam. Aluminosilicates or clays also constituted a significant proportion of the feed (8.30 vol%) and were present as fine particulates in both liberated and included form. Present in lesser quantities are the carbonates (5.3 vol%) and pyrites (3.0vol%).

5.2.1 Ash Handling

Based on the feed coal assessment, there is good information on which a plant can be designed; starting from the handling to the comminution and cleaning section. The comminution stage is often the most expensive unit operation in any beneficiation circuit. Due to the abundance of

the mineral matter associated with this coal, the Hardgrove Grindability Index (HGI) is expected to be low indicating greater resistance to crushing and grinding.

Conventional comminution equipment may not be enough and thus selection of equipment with materials of construction which can handle the harsher conditions associated with this coal becomes a critical part of the plant design.

Due to the significantly higher gangue to combustibles ratio, waste handling is another likely challenge that will be faced in the processing stage. The disposal costs to deal with large volumes of discard are also expected to be significant. With dumping also comes environmental legislature concerns thus attracting taxes which increase the operational costs and lower the profitability index. Any sustainable design must, therefore, not solely focus on processing technology but also requires creative consideration of the gangue to make it useful feed material for other processes or functions.

Sulphur removal upgrades the gangue to the quality desired for landfill purposes. Reduction of the calorific value of the gangue to values typically <10MJ/kg makes the material highly suitable for brickmaking and the manufacture of Portland cement (Argiz, Sanjuán, & Menéndez, 2017).

5.3 Evaluation of Beneficiation Strategies

It is important that laboratory data, which often gives information on ideal separation be linked to the most appropriate beneficiation technique to achieve a closely associated real separation. This statement is particularly true for washability studies while the real performance of the DMC, the most utilised beneficiation technique in coal processing, shows variance from the predictions obtained from float-sink testing.

In this section, the performance of gravity and flotation separation is discussed and a comparison of the application of the two techniques conducted upon which technical estimates of the beneficiation potential of the low-grade coal will be drawn.

5.3.1 Gravity Separation

The amenability of coal to separation through gravity-based approaches is often studied through float and sink tests. Based on the product specifications, the specific gravity at which the targeted coal separation occurs is determined. Despite the narrow range of quality specifications for coal consumed by local power stations, it is not feasible to produce coal of fixed quality parameters for supply to several power stations since boilers designs are highly sensitive to coal quality.

The difference is often within 2MJ/kg for calorific value and 2% for volatiles and ash content (Steyn & Minnitt, 2010). Burning coal of inappropriate quality results in reduced plant efficiency and increased maintenance problems. Given in Table 5-1 are the targeted thermal coal specifications within the local context. Eskom, in addition, has boilers systems at Lethabo, Kendal, Matla, Matimba and Majuba power stations which can fire lower grade coal with a calorific value range of 18.3-21MJ/kg. The process parameters for the separation equipment used will, therefore, be adjusted to give products corresponding these grade specifications and thus feasibility will be determined.

The selection of the two size fractions chosen for gravity separation tests was based on the particle size distribution and grade of the test sample received and on the known tolerance or size-class limits for gravity separation. Since most of the coal represented by the low-grade sample tends to be of a finer size, amenability testing was conducted on a relatively fine size range $1180\mu m \leq x < 4500\mu m$ and on a coarser size range $4500\mu m \leq x < 9500\mu m$.

The first size range is relatively fine compared to the feed of most coal cleaning gravity separators. Despite the existence of gravity separators efficient enough to handle materials finer than those investigated (down to 500µm), washability test results are difficult to produce at such fine PSDs hence the selection of 1180µm as the smallest aperture size in preparing samples for testing.

Table 5-1 gives thermal coal product specifications for the South African industry.

Table 5-1 South African Thermal Coal Specifications (Steyn & Minnitt, 2010)

Parameter	Units	Eskom	Domestic	Export
Calorific Value	MJ/kg	21-24	24.5-27.5	27.5
Total Moisture	wt%	8-12	8-12	8-12
Ash	wt%	25-34	15-21	15
Volatile Matter	wt%	20-30	23-30	22-30
Sulphur	wt%	0-2	0-1.5	0-1.0
Hardgrove Index			45-70	45-70
Ash Fusion	ºC			1250
Phosphorous	wt%		0.001-0.01	0.001-0.01
Sizing	Mm	0-40	0-6 6-25 25-40	0-50

5.3.1.1 Washability Curves Analysis

Figure 5-5 shows a group of washability curves obtained from the information in Table A2-5 which indicate the difficulty or ease of the proposed separation for a feed of the size fraction 1180µm≤x<4500µm. Maceral and minerals distribution in the float fractions obtained and in the tails are also included in the evaluation.

Except for the densimetric and near gravity material curves which use the secondary abscissa, the family of washability curves shown in Figure 5-5 below were based on cumulative ash (%) as the abscissa. All the washability curves share a common ordinate axis apart from the cumulative sinks curve which uses the secondary ordinate axis.

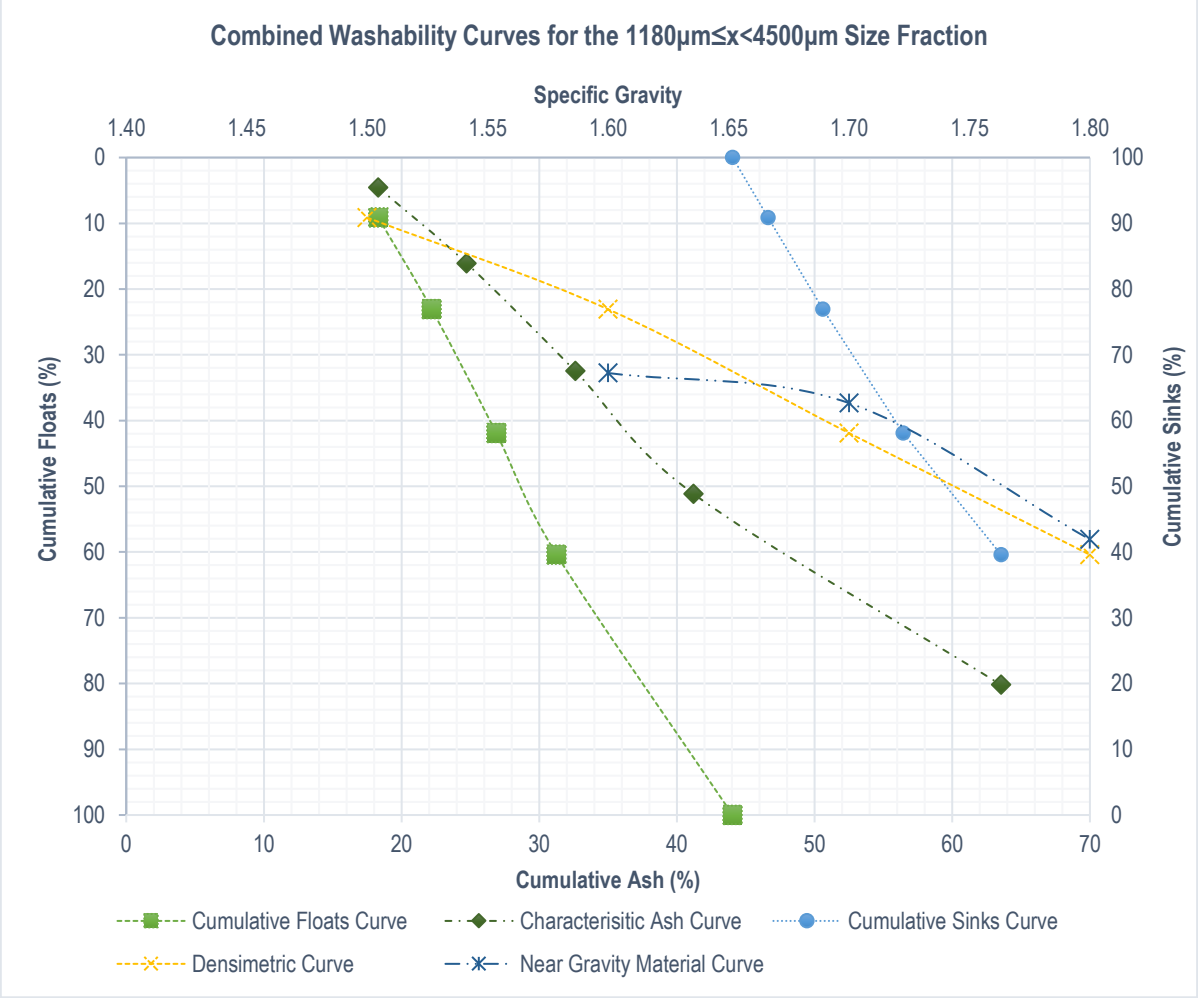


Figure 5.5 Combined Washability Curves for the 1180µm≤x<4500µm Size Fraction

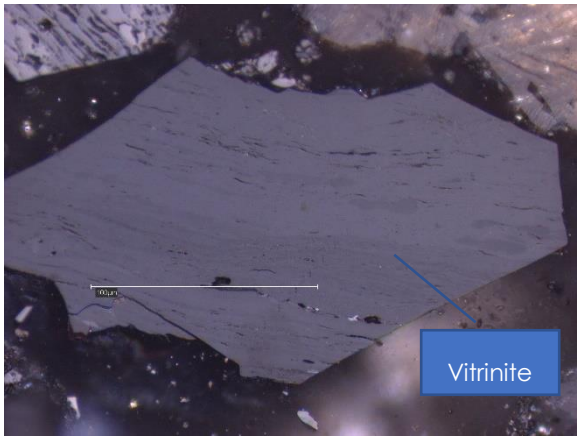
From the cumulative floats curve, it can be deduced that the yield or float mass tends to increase exponentially with a corresponding increase in the set ash target. The graph shows that if the ash content was set at 25% ash, the obtainable float yield will be 34% which corresponds to a separation density of about 1.65SG. It is thus apparent that for separation using gravity methods, the grade or ash specifications for Eskom coal can be met at meaningful

corresponding mass yields. However, the proposed separation is not without difficulty as the amount of near gravity material is already more than 30%.

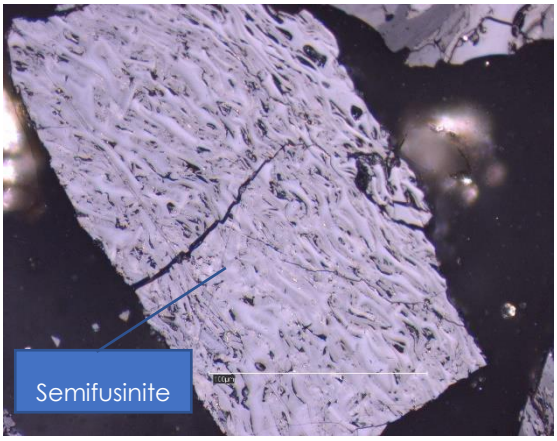
Float fractions at 1.5 SG and 1.6SG had the greatest abundance of vitrinite and greatly reduced pyrite and carbonate mineral groups with abundances of 1.4 and 2.4 vol% respectively (Table A2-12). The cumulative yield, however, increased from 6.74% to 20.4% between the two yields thus a processing decision to set a density cut point at around 1.6 SG would not be economical. At 1.7SG and 1.8SG, the abundance of pyrites and carbonates was like that obtained at the preceding densities and both minerals were mostly present in liberated form. However, there was an increased presence of mineral inclusions in the inertinite.

From a macerals content of 48.8 vol% in the feed coal, ideal separation at 1.8SG would produce a tailings product with only 18.8 vol% macerals. However, when approaching 1.8SG the abundance of macerals begins to decrease, and mineral matter recovery starts to increase rapidly causing a significant reduction in the grade of the product. Therefore, based on both boiler ash specifications and float yield concerns, the ideal optimum cut point should be around 1.65 to 1.7SG. Alternatively, the cut point can be set between 1.7-1.8 SG, but the likely product will be blended with other coals to achieve Eskom specifications.

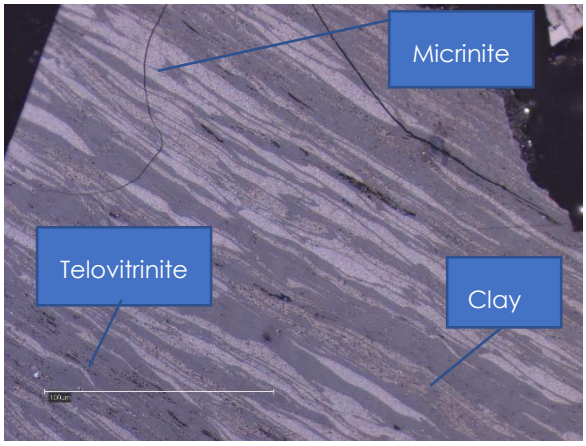
Even at ideal separation conditions, gravity separation is still unable to sufficiently liberate coal from epigenetic matter, particularly the clay minerals. Figure 5-6 shows petrographic photographs of the separation product at 1.5 SG which yielded coal of the highest grade, with an ash content of 15.9%.



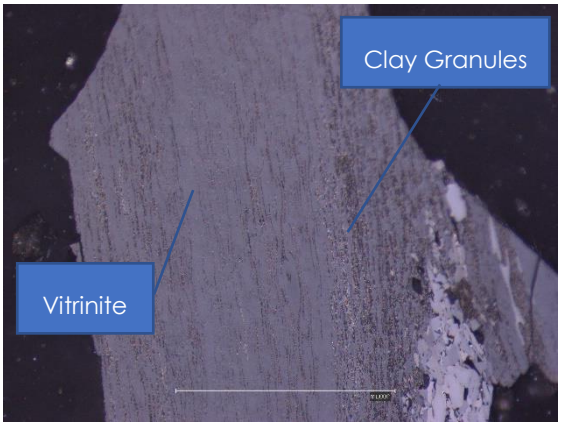
Clean Vitrinite



Clean Semifusinite (Inertinite)



Successive Layers of Telovitrinite, Micrinite (Inertinite) and Included Clay



Vitrinite with Included Fine Granules of Clay

Figure 5.6 Petrographic Photographs of SG 1.5 Float [500X, Oil Immersion, Scale Bar 100µm]

From the photomicrographs, further size reduction to very fine particle sizes will be required to liberate some of the included minerals, especially the aluminosilicates which are finely layered with the maceral. However, there is no economic incentive to perform this operation as the yield is very low to justify the cost of size reduction to such parameters as would be required for the liberation of the maceral from the included mineral. Dust and other occupational health issues also result from the handling of material ground to such size limits.

Fig 5-7 shows a group of washability curves obtained from the information in Table A2-6 which indicate the difficulty or ease of the proposed separation for a feed of the size fraction $4500\mu m \leq x < 9500\mu m$.

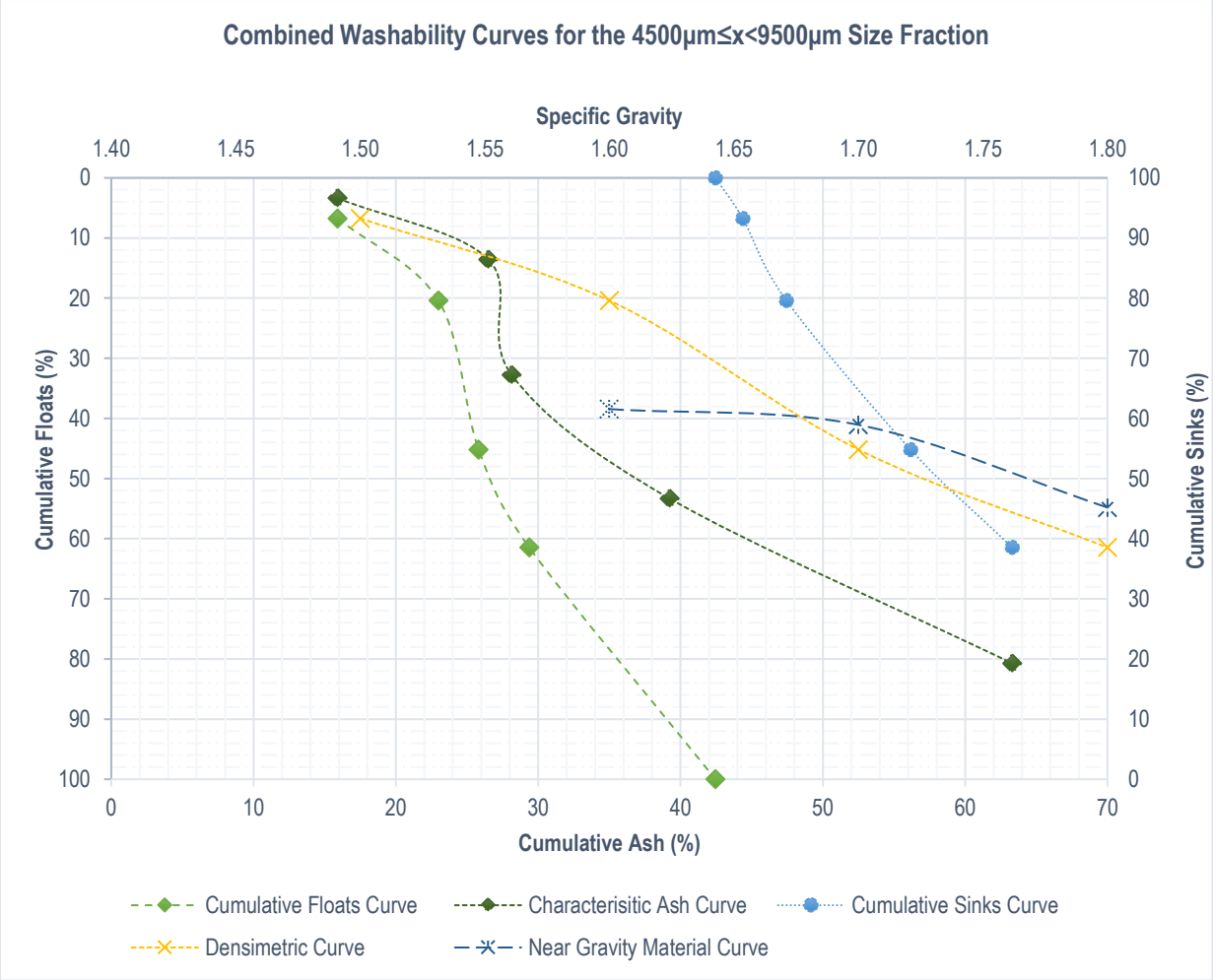


Figure 5.7 Combined Washability Curves for the 4500µm≤x<9500µm Size Fraction

From the cumulative floats curve it can be deduced that for a product with 25% ash, the yield or float mass percent will be slightly higher at 39% which corresponds to a separation density of about 1.67 SG which can be determined by drawing a tie line to the densimetric curve. The proposed separation is, however, quite formidable based on the NGM which is still more than 30%.

Close analysis of the characteristic ash curves for both size classes, between 20-40% cumulative floats, shows a steeper slope for the 4500µm≤x<9500µm size fraction indicating an enhanced rate of change of ash content (ash rejection) and subsequently better product grade. However, the two plots show no significant variance in gravity separation potential based on

the investigated particle size distributions, with the coarser fraction performing slightly better than the finer fraction.

With the increase in NGM, misplacement of material increases resulting in coal losses to the discard and contamination of the clean coal with discard. Gupta et al, (2016) state that the ease of separation using gravity-based approaches of near gravity range >25% is formidable or very difficult. Based on the degree of difficulty, process equipment is selected.

As the ease of separation decreases, the selected equipment for separation must be increasingly more efficient. Gondwana coals, such as are characteristic to South Africa are typically in the NGM range of 25-51% thus coal cleaning operations in the region require highly efficient separation equipment (Bhattacharya, Maheshwari, & Panda, 2016).

5.3.1.2 Dense Medium Cyclone Beneficiation

The DMC is a highly efficient separator developed by Dutch State Mines which has the capability of upgrading coarse and intermediate particles of the size range 0.5-50mm. In South Africa and Australia, the DMC is the most utilised equipment in the beneficiation of coal. Beneficiation occurs via the same differential settling principle as in float-sink testing where separation is achieved based on buoyancy differences between dense gangue and light coal particles. However, the centrifugal forces help to sharpen the separation.

The low-grade sample under investigation primarily originates from the No. 2 Seam in the Witbank Coalfield which averages 50% NGM, with the value fluctuating between 20% and 80% (Fourie et al., 1980). Thus, it was expected that some difficulty would be experienced during the washing of the low-grade coal sample. From the NGM curves shown in Figure 5-4 and Figure 5-5, a gradual increase in middlings is observed progressing from the 1.6SG to the 1.8SG density bath. Since the information obtained shows that the coal sample is high in NGM

with the lowest value more than 30% for both the 1800μm≤x<4500μm and the 4500μm≤x<9500μm size fraction, the most logical choice of a gravity separator would be the DMS Cyclone.

The separation performance of a DMC in beneficiating the low-grade coal sample was predicted using a Tromp curve whose offset is based on the modified Whitten Equation which is reported in numerous publications as highly reliable in DMC modelling (Scott & Napier-Munn, 1992). An E_p value of 0.05 was used based on related literature (Bahrami, Ghorbani, Mirmohammadi, Sheykhi, & Kazemi, 2018).

The conventional Rosin-Rammler function was not used since it has an inherent weakness of dependence upon a normalised density thus necessitating further work in deducing the actual values for the real density scale applied. The model is also less accurate in predicting DMC partitioning compared to the modified Whitten Equation. Fig 4-2 gives the Tromp curve obtained at a cut-point of 1.7 SG. To reverse the partitioning value, PN_i so that it is based on the float or overflow, the Ep was made negative.

South Africa's Gondwana coals typically have a cut point within the 1.6-1.7 SG range due to high NGM content. Partition data was thus generated within this cut point range using Excel Solver at these respective ρ_{50} values. Based on the local redefinition of NGM, $\pm(2 \times E_p)$ (Bhattacharya et al., 2016) from the cut density, at selected cut points of 1.6 SG and 1.7 SG; the respective amounts of NGM remained as obtained originally using the conventional definition of NGM, $\pm(0.1SG)$ (Gupta & Yan, 2016).

Experimental data from the washability tables and partition model data were combined in Tables 4-1 and 4-2 to obtain information presented in Table 4-3 which is of use in predicting the expected mass yield and grade on an ash basis for a DMC. For both 1180μm≤x<4500μm

and 4500μm≤x<9500μm size classes, a variance was observed in theoretical and predicted yields and grade, particularly with the yields where the highest variance obtained was 3.36%. This is evidence of the high separation efficiency of DMC units.

At the 1.6SG cut point, the predicted yields and grade are higher than the theoretical yields thus indicating that the cut point can be slightly lowered to attain a cleaner product. At the 1.7SG cut point, both the predicted yield and grade are slightly lower than the theoretical predictions. Thus, it can be confidently deduced that the cut-point for efficient separation of the low-grade coal from associated gangue materials is within the 1.6-1.7 SG range.

At the 1.6SG cut point 23.76% of the coal reports to the floats with an ash content of 24.23% using a feed of particle size distribution, PSD of 1180μm≤x<4500μm and 24.96% floats yield is obtained with an ash content of 24.31% at the higher PSD of 4500μm≤x<9500μm.

This implies a slightly improved yield with little decrease in grade between the coarser and the finer sized feed, a result which is in congruence with literature which states that within the particle size tolerance limits of the DMC, separation efficiency often improves with increasing feed size (Fourie et al., 1980). The deductions which can be drawn at the 1.7SG cut-point when comparing equivalent parameters concur with those drawn at the 1.6SG cut point.

Furthermore, selection of the cut-point SG for the coal washing operation can be made based on thermal coal specifications. For the 4500μm≤x<9500μm size fraction, yields of 24.96% and 41.33% corresponding to ash contents of 24.31% and 27.86% can be obtained at 1.6SG and 1.7SG cut points respectively. With the 11800μm≤x<4500μm size fraction, yields of 23.76% and 42.57% corresponding to ash contents of 24.23% and 26.75% are obtained at the same respective densities. These changes in yield are due to the positive shifting of the Tromp curve along the abscissa as the cut-point is varied.

The graph below shows how the partition curve shifts when the cut point is changed to 1.6 from 1.7 which is shown in Fig 4-2.

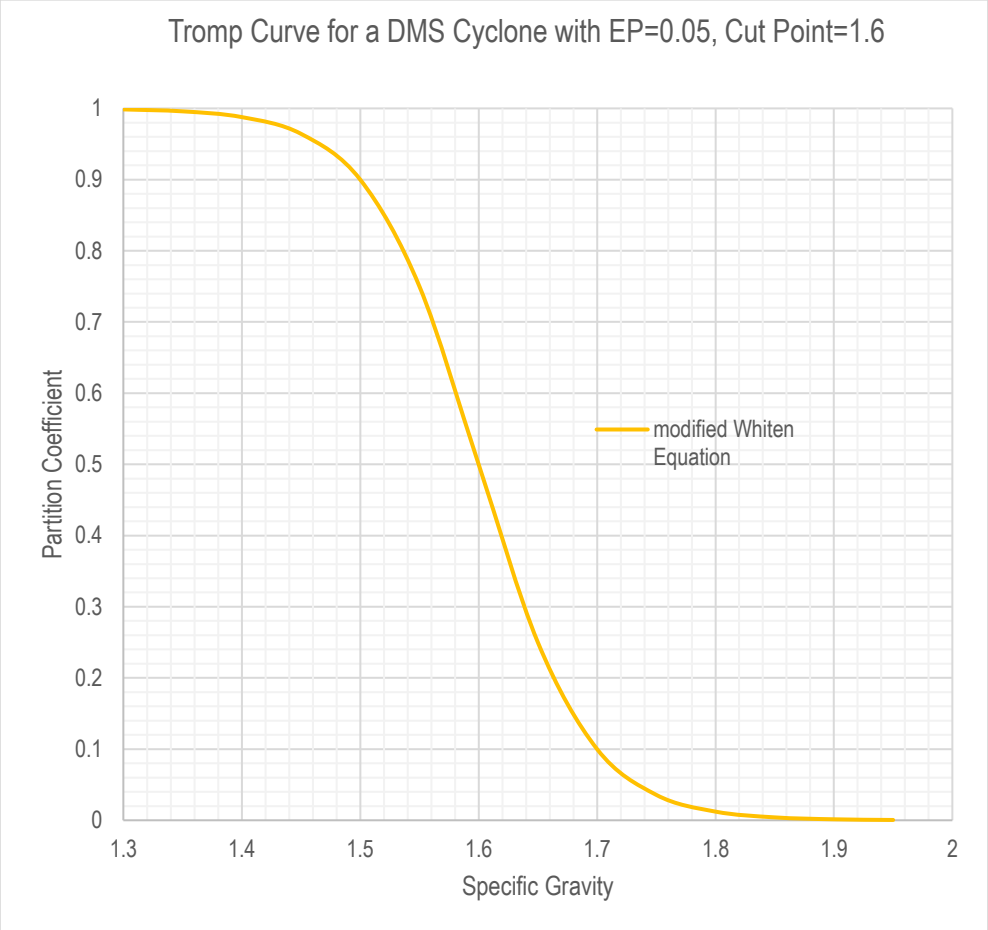


Figure 5.8 Model Partition Curve at SG=1.6

Figure 5-8 shows that that the separation gradient is quite steep, especially in the 1.5-1.7 SG range, a feature which proves that the DMC is highly efficient. For a higher cut-point of 1.7SG, the steep portion of the slope shifts positively as is shown in Fig 4-2. Based on the combined information obtained from experimental and model partition data which is presented in Table 4-3, it can be deduced that when progressing from a cut point of 1.6 to 1.7, a significant improvement in yield by close to 20% is expected while the grade decreases slightly as the ash content increases by about 2%.

Since both cut-points yield coal whose grade is compliant with the ash specifications for Eskom coal, the separation density for the coal washing should, therefore, be around an SG of 1.7 or slightly higher to maximise on the mass yield and subsequently the profitability of the coal washing operation without deviation from the power plant coal feed specifications.

5.3.2 Separation by Flotation

The separation efficiency of gravity-based coal cleaning units is known to decrease with decreasing particle size especially as the feed size approaches 500µm. This size class naturally falls within the domain of flotation. The reagent regime, predominantly collectors are the principal bedrock of the flotation process. The thermodynamic and economic feasibility of flotation is often determined by the collector or set of collectors identified for the composite to be processed. The response or sensitivity of the flotation process to the collector selected is highly significant that it is often impossible to manipulate other components of a flotation process to overcome a poorly selective collector.

Despite the natural hydrophobicity of desired organic macerals, the valuable component targeted in coal flotation, collectors are still required to increase the sharpness of the separation and improve flotation kinetics thus making it possible to treat more feed for less equipment capacity. To meet the objectives of this research, the target was therefore to analyse flotation response to variations in the reagent regime.

Considering that the low-grade coal sample had experienced surface oxidation, the use of an oily collector is likely to be inadequate, thus need was a need to select an alternative reagent whose selectivity is not hindered by the oxidation. Hence, tetrahydrofuran was selected since it bears an oxygen atom in its molecular structure. Abarca, Ali, & Pelton (2018) submit that the difficulty experienced in the floating of low-grade coal is because of the presence of

hydrophilic functional groups predominantly the hydroxyl, OH^-, carboxyl, COO^- and the carbonyl, $^{R''}_{R'}> [C = O]$ groups which reduce the adhesion of the oily droplets onto coal thus hindering flotation.

Figure 5-9 is an image taken during a flotation experiment.

Figure 5.9 Froth Flotation of Low-Grade Coal Sample using a Denver Machine

5.3.2.1 Flotation Beneficiation Analysis

Based on related literature, it is expected that the amount of collector required to achieve a recovery equivalent to that obtained in higher-grade coal processing is larger when processing low-grade coal. Three different dosages of diesel were thus chosen to help in determining the effect of concentration on the collecting ability of the reagent. Table A2-7 provides information yielded from all the reagent configurations investigated.

From a diesel dosage of 7g/kg Coal to 14g/kg Coal an increase in concentrate ash from 34.18% to 36.79% was observed. However, the clean coal recoveries for the respective dosages were 63.19% to 85.37%, suggesting that the flotation kinetics at the 14g/kg Coal dosage were superior to those at 7g/kg Coal. Flotation performance at the highest dosage investigated of 21g/kg Coal was found to be inferior to that at the preceding dosage of 14g/kg Coal, yielding a clean coal recovery of 78.30% against a yield of 63.05%, thus indicating that raising the dosage to 21g/kg Coal was now more than the optimum collector dosage.

It was also observed during the experiment that the froth bubbles were significantly smaller at a diesel dosage of 21g/kg Coal. This is because excess collector makes the micelle to become more hydrophilic and stop floating. In terms of surface phenomena, excess collector causes multilayer adsorption which decreases hydrophobicity thus adversely affects clean coal recovery (Xing, Gui, Pan, et al., 2017b). Therefore, the reduction in clean coal recovery by 7% at 21g/kg Coal when compared to the preceding dosage is thus possible.

Despite having the lowest enrichment ratio of 1.39 against 1.44 and 1.41 among the diesel collector dosage configurations investigated, the 14g/kg Coal dosage performed better than the 7g/kg Coal and the 21g/kg Coal dosages respectively because it had a significantly higher yield. However, the uptake of significant amounts of ash matter at all dosages indicates that diesel collector might not be the best-suited reagent for floating the test sample and in extension, to the processing of low-grade coal from the Witbank Coalfield and other coalfields of analogous characteristics.

When Sendep, the depressant was added to a cell using diesel collector at the identified optimal dosage of 14g/kg Coal, the recovery dropped immensely. Despite having a grade in close range to that at the optimal diesel dosage, the obtained ash contents of 38.75% and 35.95% which correspond to depressant dosages of 5gSENDEP/kg Coal and 10gSENDEP/kg Coal, there is

no matching float yield. At both depressant dosages, the float yield is very low ($< 10\%$). This renders the process inefficient, as is indicated by the respective clean coal recoveries, r_c of 11.16% and 7.15%.

From both float yield and grade results obtained, it is apparent that the use of Sendep has a negative impact on the beneficiation of this material. The results can thus be interpreted to mean that the depressant used has a poor selectivity for the coal, inducing hydrophilicity to both maceral and mineral constituents of the coal composite (Kawatra, 2011).

Tetrahydrofuran exhibited reasonable collecting ability, producing a concentrate with the lowest amount of ash reporting to the concentrate, 32.81% and the highest enrichment ratio of 1.46. However, the float yield was quite low at 26.99% and consequently the clean coal recovery, r_c was only 42.20%. Howbeit, the results are a good indicator that the low-grade coal sample is partially oxidised as suspected.

Xing et al (2017) state that increasing the length of the hydrophobic chain of a collector is more effective in enhancing flotation recovery in comparison to increasing collector dosage. However, longer chains are also known for reducing selectivity. Therefore, esterification of THF to form an ester with a longer aliphatic hydrocarbon chain can potentially increase the hydrophobicity of the material, enhancing the collecting ability of the compound with relation to low-grade, partially oxidised coals (Jia, Harris, & Fuerstenau, 2002).

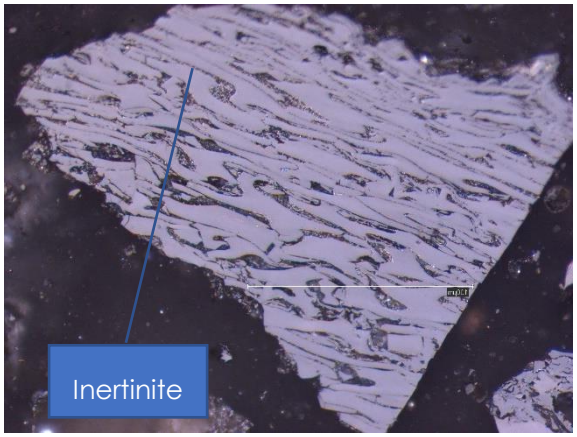
Alternatively, an investigation can be made into the use of THF as a jointly with an oily collector to maximise on the yield and types of organic macerals than can be gleaned from the coal composite during beneficiation.

From all the exploratory data gathered and analysed, it is apparent that the best performing flotation unit based on chemical parameters is that which uses diesel collector at a dosage of

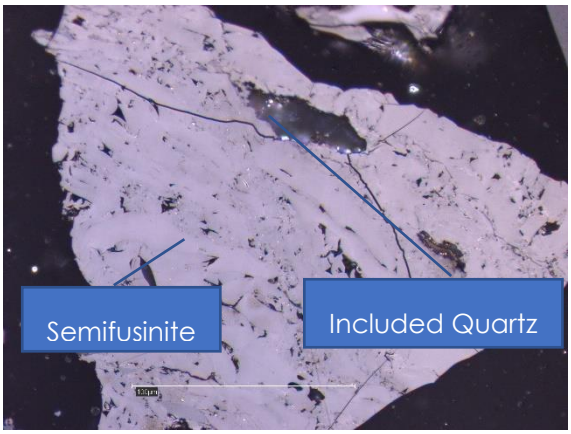
14g/kg Coal. Figure 5-10 shows a photograph of the float yields and sinks after dewatering and drying.

Figure 5.10 Flotation Products at Diesel Dosage 14g/kg Coal

The sequence in Fig 5-10 above shows a decrease in colour intensity with each succeeding fraction which corresponds the decrease in grade shown by ash contents of 33.61%, 42.10%, 53.61% and 73.79% respectively. Though colour can vary a lot with coal, for this case, a darker colour indicates a richer concentrate. Colour becomes progressively lighter with an increase in the proportion of inorganic compounds comprising the gangue or decreasing proportion (%) of carbon which is dark in colour. Fig 5-11 contains petrographic photographs of the highest-grade concentrate from flotation (T=1) and the tailings from the process.



Inertodetrinite (T=1)



Semifusinite with Included Quartz (T=1 FLOAT)

Excluded and Included Quartz (Tails)

Excluded Clay and Quartz (Tails)

Figure 5.11 Petrographic Photographs of Flotation Products [500X, Oil Immersion, Scale Bar 100µm]

From Fig 5-11 above, size reduction to <100µm will have significantly improved liberation. Regrinding and floating of the $T = 1$ product will improve upgrade and still maintain a reasonable yield or alternatively the product can be floated again to further reject excess coal. However, the economic benefit of such measures should be properly evaluated since the product cost should remain low. At 73.79% Ash corresponding to the extent of ash rejection shown above, there remains insufficient maceral content to warrant further processing.

The information obtained from flotation showed a sharp decrease in clean coal recovery corresponding to a decrease in grade. The process goes from being highly efficient at $T = 3, r_c = 80.66\%$ and only a further 4.70% is recovered between 3 and $9min$. There is, therefore,

no technological or economic incentive to proceed with flotation past $T = 3min$. Tailings from the process have an ash content of 73.79% which converts to a beneficiation margin of approximately 21% from the starting ash content of 52.59%.

Maceral and mineral group analysis show a trend like the one obtained during proximate and float yield analysis. The amount of inertinite was 70.1 vol% at $T = 1$, decreased slightly to 61.1 vol% at $T = 3$, followed by an exponential decrease to 26.8 vol% between 3 and $9min$ (Table A2-12). Vitrinite and liptinite macerals also trend similarly but are less abundant thus their technological impact are less significant than that of inertinite. If the process float time is limited to $T = 3min$, the rate of combustion will, therefore, be highest in the upper end of the furnace thus the coal may require longer residence times or higher oxygen input.

Despite both separation techniques having a reasonable and comparable capacity to reject quartz and clay and carbonates, the rejection of pyrites is better performed by gravity separation compared to flotation. The maximum amount of pyrites found in the float products from gravity separation is around 0.2 vol% while for flotation this value rises to 5.4 vol%. However, if flotation time is set at $T = 3\ min$, the expected pyrite content in the float product reduces to 1.0 vol%.

A family of curves describing flotation at a diesel dosage of 14g/kg Coal are given in Figure 5-12 which is derived from the information obtained in Table A2-9. Though a limited number of dosages was used there is a good indication that optimum flotation time is around 3 min. Thereafter there is very little change in yield and past 6 min the ash starts to increase exponentially. In other words, the optimum residence time is between 3 min and 6 min. Based on ash specifications for Eskom's pulverised fuel boilers $(25 - 34\%)$, a residence time of T=1 min is acceptable since it yields a concentrate with 33.61% Ash. This also corresponds to a

float yield of 51.16% and a clean coal recovery, $r_c = 65.21\%$ thus making the process sustainable from a production standpoint.

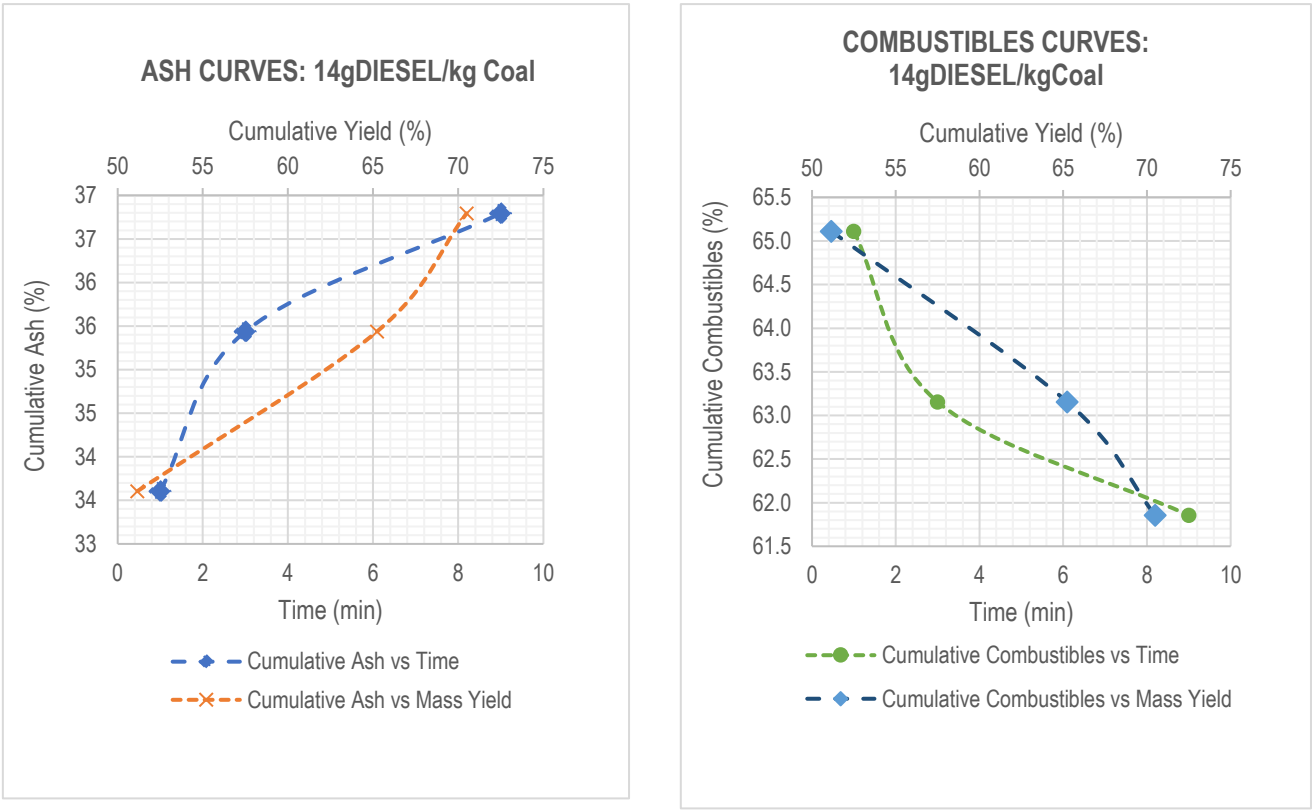


Figure 5.12 Flotation Curves at a Diesel Dosage of 14g/kg Coal

In terms of operating costs, the decision to use an optimum residence time of around 3min also serves to lower energy expenditure. Further work, however, will be required in studying the kinetics of the flotation process to obtain the rate constant, k which can then be applied during upscale to plant and full-scale operation.

5.4 Process Flowsheet

Figure 5-13 gives a proposed pathway for the cleaning/beneficiation of the low-grade coal.

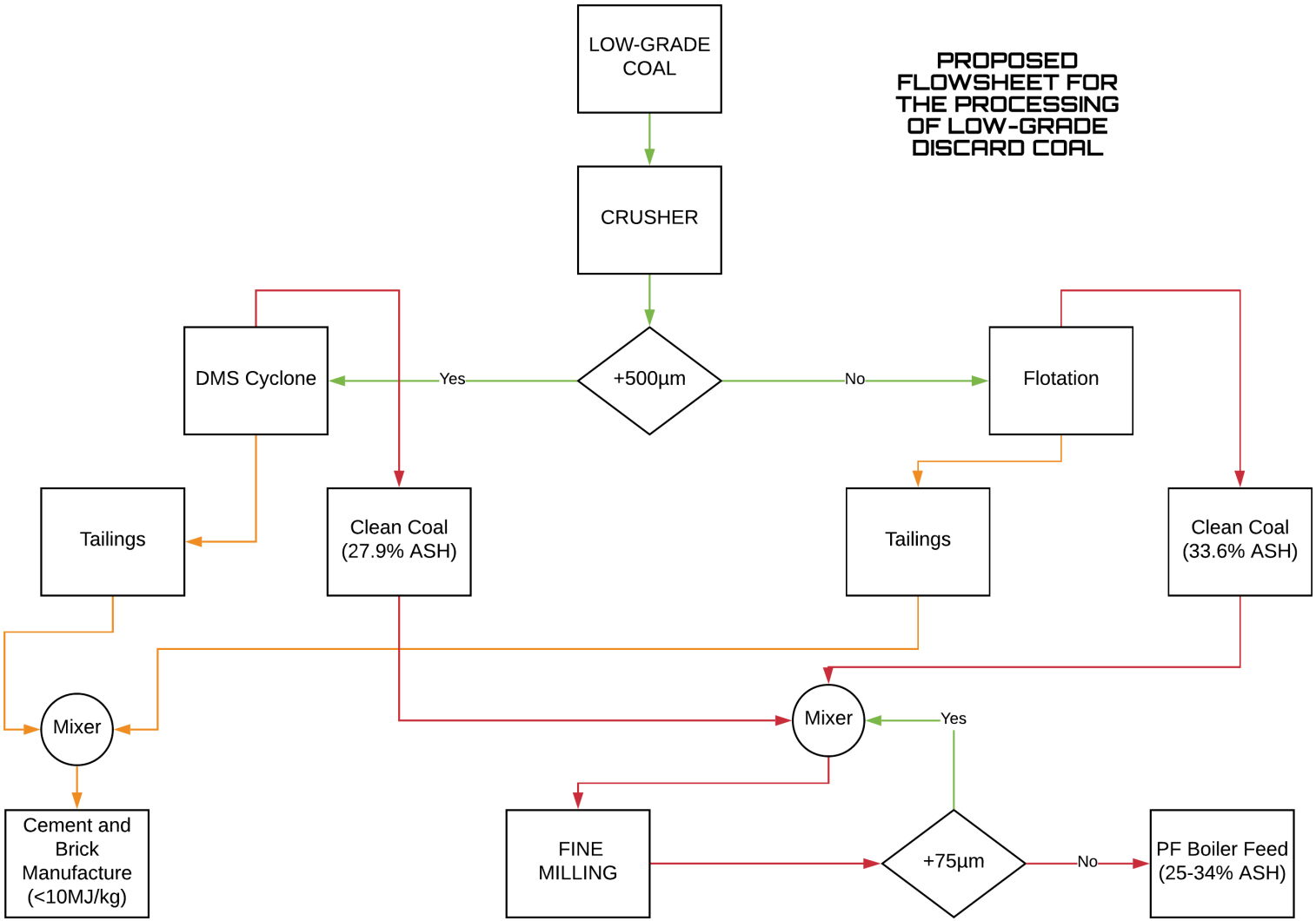


Figure 5.13 Proposed Flowsheet for the Beneficiation of Low-Grade Discard ROM Coal

CHAPTER 6
CONCLUSION AND RECOMMENDATIONS

The intent of this research was to evaluate the possibility of upgrading low-grade coal from the Witbank Coalfield. Thus, a sample of low-grade coal was obtained from Ingwe Collieries which was taken through a set of analytical and process tests to determine the amenability to flotation. Given in this section are the conclusions and recommendations resulting from this research. Direction for future work is also extended.

Petrographic and chemical analysis showed that the coal was high ash (53 wt%), low CV (14MJ/kg) Medium-Rank C bituminous classification and abundant in inertinite group macerals (48.8 vol%), quartz (29.5 vol%) and clays (8.3 vol%). Quartz, the most abundant mineral group in the coal and was present in both syngenetic and epigenetic form. Clays, the second most abundant mineral group, occurred in similar forms to quartz. Carbonates and pyrites were found in smaller quantities.

Gravity separation was investigated on two size classes; $1180\mu m < x \leq 4500\mu m$ and $4500\mu m < x \leq 9500\mu m$. The amount of near gravity material (NGM) was obtained to be 32% and 58% respectively thus indicating that the coal is very difficult to wash. Through modelling work, it was determined that the coal can be upgraded successfully using a dense medium cyclone (DMC). At a cut-point of 1.7 SG, a 43% yield was obtainable with a grade of 26.75% Ash for the $1180\mu m < x \leq 4500\mu m$ size class. An almost equivalent grade and yield are obtainable for the larger size class.

Flotation separation also showed the capability to upgrade coal in the -300µm size class. From the flotation experiments conducted, the best performance was by the first fraction collected at

$T = 1\ min$ using diesel (dosage 14g/kg Coal) which had the best balance between grade and yield. A 51% yield was obtained with a grade of 33.61% Ash resulting in a clean coal recovery of 65.21%.

The partitioning model used for gravity separation (Whitten Model) predicted that a 51.1% yield obtained using a DMC will have a grade of 27.92% Ash and a clean coal recovery of 63.74%. The clean coal recovery for flotation though slightly higher yielded a slightly lower grade than that for the DMC. This indicates that the separation potential of for both flotation and gravity separation through the DMC is closely related for the low-grade coal. Thus, technique selection is less a function of efficiency than it is of the feed size class.

Size analysis showed that the low-grade coal was well distributed over a wide size range, including both fine and coarse particles. Therefore, both DMC and flotation are required to create an integrated process for the upgrade of low-grade coal. Both beneficiation techniques managed to meet the grade specifications for pulverised fuel boiler feed in thermal power generation.

Preference for flotation will result in comminution costs which reduce the economic feasibility of the process while that for the DMC would face technical and efficiency constraints with $-500\mu m$ sized coal. However, an exception to this condition arises when mineral groups in the feed coal happen to be highly epigenetic and finely distributed. In such a situation flotation becomes the beneficiation technique of preference and the comminution costs associated with size reduction are validated.

The role of analytical work in guiding processing decisions was found to be highly significant in this research. Modelling also played a key role in expanding the limits of this research through helping create a larger comparative basis upon which a conclusion can be drawn. This

research, therefore, submits that an integrated process utilising both DMC and flotation separation equipment at appropriate size classes is essential in beneficiating the low-grade coal obtained from the Witbank Coalfield. However, a detailed techno-economic analysis which was not within the scope of this research is still required to determine the economic potential in upgrading the coal.

6.1 Direction for Future Work

From the research conducted, new challenges and opportunities emerged that could be the basis for future research. Firstly, there is a need to expand the sources of low-grade coal for similar research to beyond the Witbank Coalfield to the Molteno-Indwe and the Free State Coalfields which have been reported to host a significant proportion of low-grade coal.

Secondly, concerning the DMC, it is imperative that a laboratory or pilot scale plant be constructed to allow for more in-depth analysis of the techno-economic feasibility of this technique in beneficiating low-grade coal. With regards to flotation, the research ascertains that oily collector dosage requirements are quite high for low-grade coal beneficiation, and the ash rejection is still inadequate, thus necessitating the need for further research into the development of an efficient reagent regime. There is also a need for the erection of a pilot plant to determine process response to scale-up and gather valuable techno-economic data in preparation for full-scale implementation of this critical beneficiation technique.

An interesting possibility proceeding from this research is the potential for use of diesel and tetrahydrofuran, THF as joint collectors. Investigation can also be made into the effect of increasing the length of the hydrophobic part of the THF molecule which potentially could enhance clean coal recovery for South African low-grade coals.

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APPENDIX

Appendix 1: Analytical Procedures

Calorific Value Test Procedure

A DRYCAL Bomb Calorimeter which measures gross calorific value (GCV) according to (South African National Standards (SANS) 1928, 2009) was used. Prior to starting the experiment, the following procedure was conducted; the opening of the water, the opening of oxygen lines and setting the pressure to $2500kPa$; switching on the computer and calorimeter controller; logging onto a data-logging program, Super-Cal, and raising of the jacket temperature to 36.3℃. A daily check of all the valves was conducted and calibration was run using benzoic acid in exactly the same way the experiment would be run. A thread of cotton was then cut, and a loose loop tied which was then put into a crucible. The crucible and cotton were then measured on an analytical balance and then a $(0.5 \pm 0.01)\ g$ tablet of benzoic acid was weighed into the crucible, covering the cotton loop. The other end of the cotton thread was then looped around a firing wire and the crucible then loaded into the bomb-cap ring. After that, the bomb-cap was lowered and locked into position in a clean bomb without any unburnt or residual sample. The bomb was then filled with $O_2(g)$ to $2500kPa$. Now pressurised, the bomb was carefully carried and placed inside a calorimeter. The set-up was then secured by shutting the lid of the calorimeter which automatically triggered the test program. The test went through a stabilising stage where jacket and bomb temperature were being equalised after which the bomb was fired starting the main period of the GCV analysis which ran for the next $320s$. When the test program had reported that the analysis was complete, the bomb was carefully removed, depressurised and then left to cool. A duplicate measurement was done to ensure reproducibility of the results.

Proximate Analysis Test Procedure

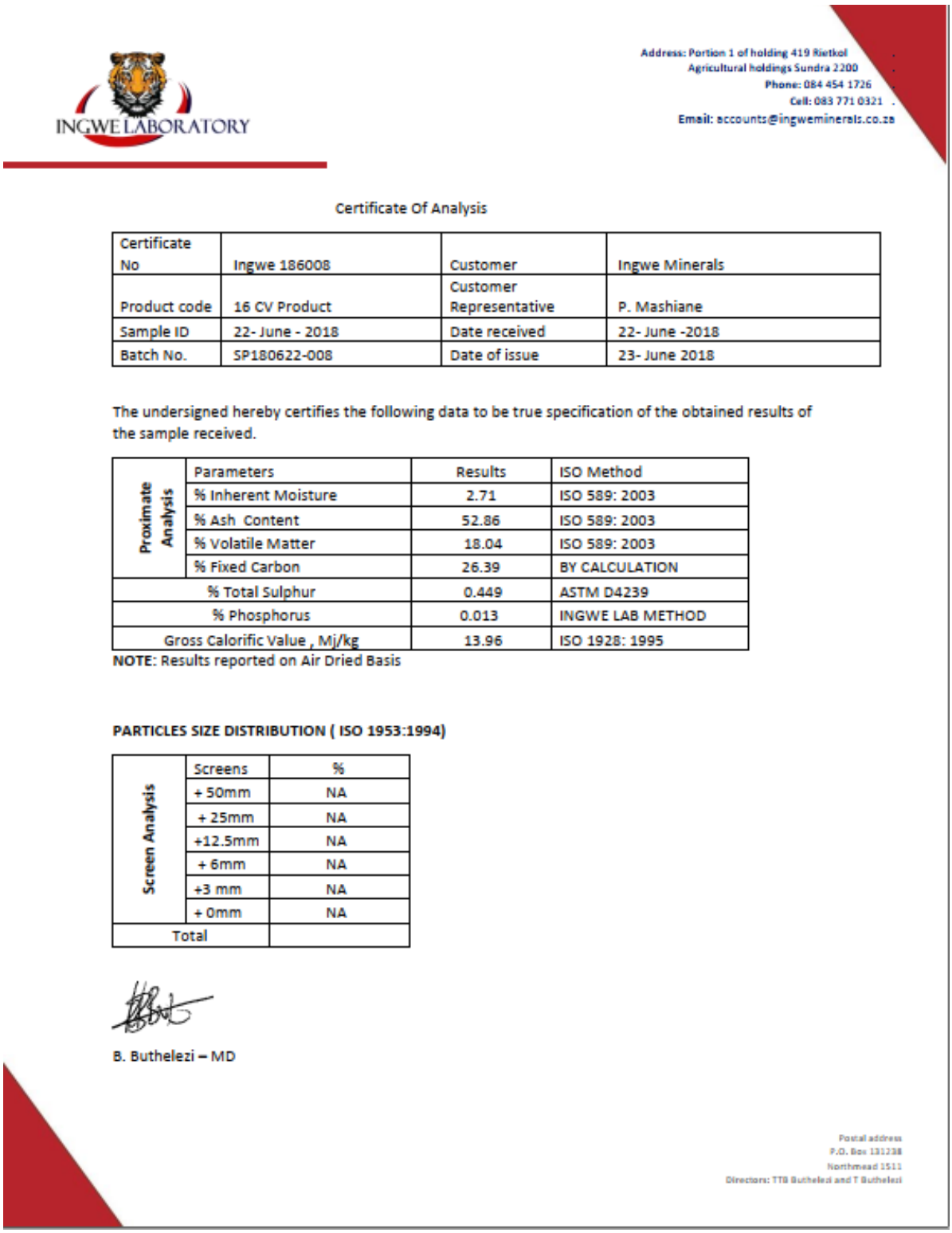
An SDT Q600 V20.9 Build 20 Differential Scanning Calorimeter (DSC) - Thermogravimetric Analyser (TGA) which provides a concurrent measurement of true differential heat flow (DSC) and weight change (TGA) on the sample from ambient temperature to $1300^{\circ}C$ was used for proximate analysis. Prior to running the experiment, the sample crucible was cleaned and calibrated against the reference crucible inside the furnace. The sample crucible was then removed and 10mg of the sample was then measured into it using an analytical balance. The sample crucible was then returned, and the furnace closed. A program for proximate analysis of coal was then run.

Table A1- 1 TGA Program

Method log:

1. **Select Gas:1**
2. **Mass flow 45.0mL/min**
3. **Data Storage: On**
4. **Isothermal for 4.00 min**
5. **Ramp 20.00°C/min to 110°C**
6. **Isothermal for 5.00 min**
7. **Ramp 80.00°C/min to 950.00°C**
8. **Isothermal for 15.00min**
9. **Select Gas: 2**
10. **Mass flow 80.0mL/min**
11. **Isothermal for 25.00min**
12. **Air cool: On**
13. **Air cool: On**
14. **End of method**

The system produced a graph of weight change (%) on the primary y-axis, derivative heat flow $\left({}^{mW}/_{min}\right)$ on the secondary x-axis and temperature (℃) on the primary x-axis. The curve for weight change was then integrated to give inherent moisture, volatile matter and fixed carbon $wt\%$ compositions in succession. Heat flow analysis was only done qualitatively.



INGWE LABORATORY

Address: Portion 1 of holding 419 Rietkol
Agricultural holdings Sundra 2200
Phone: 084 454 1726
Cell: 083 771 0321
Email: accounts@ingweminerals.co.za

Certificate Of Analysis

Certificate No	Ingwe 186008	Customer	Ingwe Minerals
Product code	16 CV Product	Customer Representative	P. Mashiane
Sample ID	22- June - 2018	Date received	22- June -2018
Batch No.	SP180622-008	Date of issue	23- June 2018

The undersigned hereby certifies the following data to be true specification of the obtained results of the sample received.

	Parameters	Results	ISO Method
Proximate Analysis	% Inherent Moisture	2.71	ISO 589: 2003
	% Ash Content	52.86	ISO 589: 2003
	% Volatile Matter	18.04	ISO 589: 2003
	% Fixed Carbon	26.39	BY CALCULATION
% Total Sulphur		0.449	ASTM D4239
% Phosphorus		0.013	INGWE LAB METHOD
Gross Calorific Value , Mj/kg		13.96	ISO 1928: 1995

NOTE: Results reported on Air Dried Basis

PARTICLES SIZE DISTRIBUTION (ISO 1953:1994)

	Screens	%
Screen Analysis	+ 50mm	NA
	+ 25mm	NA
	+12.5mm	NA
	+ 6mm	NA
	+3 mm	NA
	+ 0mm	NA
Total		

B. Buthelezi – MD

Postal address
P.O. Box 131238
Northmead 1511
Directors: TTB Buthelezi and T Buthelezi

Figure A2- 1 Ingwe Certificate of Feed Analysis

Table A2- 1 Gross Calorific Value Data

ENERGY INSTRUMENTATION DRYCAL MODULAR CALORIMETER			
DATE	**SAMPLE ID**	**GCV (MJ/kg)**	**AVERAGE GCV (MJ/kg)**
2/12/2019 13:12	CAL5	26.447	**26.447**
2/12/2019 13:29	FEED	13.98	**14.0435**
2/12/2019 13:42	FEED_2	14.107	
2/12/2019 13:58	THF_1_1	19.919	**19.973**
2/12/2019 14:10	THF_1_2	20.027	
2/12/2019 14:23	THF_3_1	19.492	**19.561**
2/12/2019 14:36	THF_3_2	19.63	
2/12/2019 14:49	THF_9_1	18.783	**18.7975**
2/12/2019 15:02	THF_9_2	18.812	
2/12/2019 15:17	THF_TAILS_1	11.593	**11.5425**
2/12/2019 15:30	THF_TAILS_2	11.492	
2/12/2019 15:46	14gDIESEL/kg Coal_1_1	20.159	**20.141**
2/12/2019 15:58	14gDIESEL/kg Coal _1_2	20.123	
2/12/2019 16:09	14gDIESEL/kg Coal _3_1	18.639	**18.5775**
2/12/2019 16:21	14gDIESEL/kg Coal _3_2	18.516	
2/12/2019 16:34	14gDIESEL/kg Coal _9_1	16.443	**16.46**
2/12/2019 16:46	14gDIESEL/kg Coal _9_2	16.477	
2/13/2019 9:40	14gDIESEL/kg Coal _TAILS_2	9.223	**9.1945**
2/13/2019 9:58	14gDIESEL/kg Coal _TAILS_3	9.166	
2/13/2019 10:16	5gSENDEP/kg Coal_1_1	16.47	**16.4485**
2/13/2019 10:27	5gSENDEP/kg Coal _1_2	16.427	
2/13/2019 11:24	5gSENDEP/kg Coal _3_1	17.095	

2/13/2019 11:35	5gSENDEP/kg Coal _3_2	17.06	**17.0775**
2/13/2019 11:47	5gSENDEP/kg Coal _9_1	17.285	**17.242**
2/13/2019 11:59	5gSENDEP/kg Coal _9_2	17.199	
2/13/2019 12:11	5gSENDEP/kg Coal _TAILS_1	12.237	**12.1805**
2/13/2019 13:14	5gSENDEP/kg Coal _TAILS_2	12.124	
2/13/2019 13:27	1.18-4.5 HMS FEED_1	16.098	**16.059**
2/13/2019 13:40	1.18-4.5 HMS FEED_2	16.02	
2/13/2019 13:56	4.5-9.5 HMS FEED_1	15.043	**15.0965**
2/13/2019 14:08	4.5-9.5 HMS FEED_2	15.15	
2/13/2019 14:19	9.5+ HMS FEED_1	11.989	**11.936**
2/13/2019 14:29	9.5+ HMS FEED_2	11.883	
2/14/2019 11:51	<1.18MM FINES_2	8.466	**8.523**
2/14/2019 12:14	<1.18MM FINES_3	8.58	
2/14/2019 14:03	(4.5-9.5mm)SG1.5_1	25.081	**24.963**
2/14/2019 14:23	(4.5-9.5mm)SG1.5_2 REP	24.845	
2/14/2019 14:36	(4.5-9.5mm)SG1.6_1	22.125	**22.11**
2/14/2019 14:48	(4.5-9.5mm)SG1.6_2	22.095	
2/14/2019 15:18	(4.5-9.5mm)SG1.7_1_1	19.761	**19.703**
2/14/2019 15:43	(4.5-9.5mm)SG1.7_2_1	19.645	
2/14/2019 15:57	(4.5-9.5mm)SG1.8_1	16.683	**16.6215**
2/14/2019 16:10	(4.5-9.5mm)SG1.8_2	16.56	
2/14/2019 16:22	(4.5-9.5mm)SG TAILS_1	8.051	**8.0315**
2/14/2019 16:36	(4.5-9.5mm)SG TAILS_2	8.012	

Table A2- 2 Characterisation of the Feed Coal Based on Particle Size

Size Fraction	Mass (kg)	Mass (wt%)	Moisture (wt%)	Ash (wt%)	Volatile Matter (wt%)	Fixed Carbon (wt%)	Calorific Value (MJ/kg)
$0\mu m < x < 1180\mu m$	0.02	3	2.671	56.549	19.22	21.56	8.523
$1180\mu m \leq x < 4500\mu m$	0.14	27.25	3.267	38.943	16.22	41.57	16.059
$4500\mu m \leq x < 9500\mu m$	0.14	28	2.934	42.486	15.8	38.78	15.0965
$\geq 9500\mu m$	0.21	41.75	2.174	49.366	17.66	30.8	11.936
Overall Feed			2.962	52.588	18.94	25.51	14.0435

Table A2- 3 Characterisation of the Yields of Float-Sink Analysis

Size Fraction	Specific Gravity	Mass (g)	Mass (wt%)	Moisture (wt%)	Ash (wt%)	Volatile Matter (wt%)	Fixed Carbon (wt%)	Calorific Value (MJ/kg)
4500μm≤x<9500μm	1.5	33.69	6.74	3.41	15.90	24.65	56.04	24.96
	1.6	68.29	13.66	3.13	26.49	18.90	51.48	22.11
	1.7	123.95	24.79	3.28	28.14	17.60	50.98	19.70
	1.8	81.39	16.28	2.84	39.24	15.70	42.22	16.62
	Tails	192.68	38.54	1.79	63.32	13.72	21.17	8.03
1180μm≤x<4500μm	1.5	45.66	9.13	3.58	18.31	22.40	55.71	
	1.6	69.68	13.94	3.28	24.73	19.13	52.86	
	1.7	94.15	18.83	3.77	32.65	16.43	47.15	
	1.8	92.51	18.51	3.64	41.21	15.63	39.52	
	Tails	197.86	39.58	1.88	63.55	13.28	21.29	

Table A2- 4 Sink and Float Data and Calculations for the Size Fraction 1180µm≤x<4500µm

SAMPLE: LOW-GRADE COAL FROM INGWE MINERALS **SIZE FRACTION:1180µm≤x<4500µm** **PROPORTION OF TOTAL FEED: 27.25%**												
[1]		[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]	[10]	[11]	[12]	[13]
RELATIVE SPECIFIC GRAVITY FRACTIONS		**SIZE FRACTION BASIS**											
		MASS	**ASH**	**PROPORTION OF ASH**	**RELATIVE SG**	**CUMULATIVE FLOATS**			**CUMULATIVE SINKS**			**CUMULATIVE MASS TO MIDDLE FRACTION**	**MASS OF NEAR GRAVITY MATERIAL**
						MASS	**PROPORTION OF ASH**	**ASH**	**MASS**	**PROPORTION OF ASH**	**ASH**		
				[2] × [3]		∑ [2] ↓	∑ [4] ↓	[7]/[6]	∑[2]↑	∑[4]↑	[10]/[9]	[6]-[2]/2	(±0.1SG)
SINKS	**FLOATS**	(%)	%			(%)		%	(%)		%	(%)	(%)
	1.5	`	15.90	107.10	1.50	6.74	107.10	15.90	100.00	4245.23	42.45	3.37	
1.5	1.6	13.66	26.49	361.83	1.60	20.40	468.92	22.99	93.26	4138.13	44.37	13.57	38.45
1.6	1.7	24.79	28.14	697.64	1.70	45.19	1166.56	25.82	79.60	3776.31	47.44	32.79	41.07
1.7	1.8	16.28	39.24	638.72	1.80	61.46	1805.28	29.37	54.82	3078.67	56.16	53.32	54.82
1.8		38.54	63.32	2439.95		100.00	4245.23	42.45	38.54	2439.95	63.32	80.73	
TOTAL		100.00	42.45										

Table A2- 5 Sink and Float Data and Calculations for the Size Fraction 4500µm≤x<9500µm

SAMPLE: LOW-GRADE COAL FROM INGWE MINERALS **SIZE FRACTION:4500µm≤x<9500µm** **PROPORTION OF TOTAL FEED: 28.00%**												
[1]		[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]	[10]	[11]	[12]	[13]
RELATIVE SPECIFIC GRAVITY FRACTIONS		**SIZE FRACTION BASIS**											
		MASS	**ASH**	**PROPORTION OF ASH**	**RELATIVE DENSITY**	**CUMULATIVE FLOATS**			**CUMULATIVE SINKS**			**CUMULATIVE MASS TO MIDDLE FRACTION**	**MASS OF NEAR GRAVITY MATERIAL**
						MASS	**PROPORTION OF ASH**	**ASH**	**MASS**	**PROPORTION OF ASH**	**ASH**		
				[2]×[3]		∑[2]↓	∑[4]↓	[7]/[6]	∑[2]↑	∑[4]↑	[10]/[9]	[6]-[2]/2	(±0.1SG)
SINKS	**FLOATS**	(%)	%			(%)		%	(%)		%	(%)	(%)
	1.5	9.13	18.31	167.23	1.50	9.13	167.23	18.31	100.00	4404.83	44.05	4.57	
1.5	1.6	13.94	24.73	344.77	1.60	23.08	512.01	22.19	90.87	4237.59	46.64	16.10	32.78
1.6	1.7	18.83	32.63	614.46	1.70	41.91	1126.47	26.88	76.93	3892.82	50.60	32.49	37.34
1.7	1.8	18.51	41.21	762.71	1.80	60.42	1889.18	31.27	58.09	3278.36	56.43	51.16	58.09
1.8		39.58	63.55	2515.64		100.00	4404.83	44.05	39.58	2515.64	63.55	80.21	
TOTAL		100.00	44.05										

Table A2- 6 Data and Calculations from Chemical Analysis of Flotation

Chemical Reagent	Dosage (g/kg Coal)	Float Fraction	Mass Yield (%)	Cumulative Mass (%)	Ash Content (%)	Cumulative Ash (%)	Fixed Carbon (%)	Volatile Matter (%)	Cumulative Combustibles (%)	Inherent Moisture (%)	Gross Calorific Value (MJ/kg)
Diesel	7	T=1	19.54	19.54	30.52	30.52	47.81	20.29	68.10	1.39	
		T=3	18.12	37.66	34.32	32.35	44.71	19.17	66.07	1.80	
		T=9	13.16	50.82	39.43	34.18	40.36	18.29	64.15	1.92	
		TAILS	49.18	100.00	60.43	47.09	23.49	15.12	51.59	0.96	
	14	T=1	51.16	51.16	33.61	33.61	46.25	18.86	65.11	1.29	20.14
		T=3	14.08	65.24	42.10	35.44	38.86	17.19	63.15	1.85	18.58
		T=9	5.25	70.49	53.61	36.79	29.65	16.08	61.86	0.66	16.46
		TAILS	29.51	100.00	73.79	47.71	12.39	12.94	51.08	0.89	9.19
	21	T=1	24.79	24.79	31.59	31.59	47.11	19.45	66.56	1.85	
		T=3	25.56	50.35	35.86	33.76	44.23	18.75	64.74	1.16	
		T=9	12.70	63.05	44.39	35.90	36.16	17.88	62.59	1.57	
		TAILS	36.95	100.00	69.68	48.39	15.57	14.03	50.40	0.72	
Tetrahydrofuran	14	T=1	9.99	9.99	29.92	29.92	48.37	19.24	67.61	1.39	19.97
		T=3	7.80	17.79	32.84	31.20	46.38	18.34	66.34	1.80	19.56
		T=9	9.21	26.99	35.94	32.81	43.63	17.87	64.69	1.92	18.80
		TAILS	73.01	100.00	52.30	47.04	29.67	16.42	51.11	0.96	11.54
SENDEP 30F (+ Diesel 14g/kg Coal)	5	T=1	2.29	2.29	39.35	39.35	39.90	18.39	58.29	2.36	16.45
		T=3	1.55	3.84	37.85	38.75	40.77	18.57	58.71	2.81	17.08
		T=9	5.29	9.13	38.76	38.75	40.38	18.45	58.78	2.41	17.24
		TAILS	90.87	100.00	51.49	50.33	29.41	17.60	48.08	1.50	12.18
	10	T=1	1.18	1.18	37.72	37.72	41.03	18.39	59.42	2.87	
		T=3	1.43	2.61	36.98	37.31	41.54	18.73	59.89	2.75	
		T=9	2.88	5.50	34.72	35.95	43.07	19.39	61.24	2.82	
		TAILS	94.50	100.00	52.06	51.17	30.02	16.27	47.11	1.65	

Table A2- 7 Chemical Analysis-Based Performance Evaluation of Flotation

Reagents	Dosage	Float Yield	Concentrate Ash A_c	Concentrate Combustibles c	Clean Coal Recovery $r_c = \frac{Cc}{Ff}$	Enrichment Ratio $\frac{c}{f}$
	(g/kg Coal)	(%)	(%)	(%)	(%)	
Diesel	7	50.82	34.18	64.15	63.19	1.44
	14	70.49	36.79	61.86	85.37	1.39
	21	63.05	35.90	62.59	78.30	0.62
Tetrahydrofuran	14	26.99	32.81	64.69	42.20	1.46
SENDEP 30F (+ Diesel 14g/kg Coal)	5	9.13	38.75	58.78	11.16	1.32
	10	9.21	35.95	61.24	7.15	1.38

Table A2- 8 Yield-Based Analysis of Flotation (14g DIESEL /kg Coal)

Time	Float Yield	Concentrate Ash (Grade)	Concentrate Combustibles	Clean Coal Recovery	Enrichment Ratio
(min)	(%)	(%)	(%)	(%)	
1	51.16	33.61	65.11	65.21	1.46
3	14.08	42.10	56.05	15.45	1.26
9	5.25	53.61	45.73	4.70	1.03

Table A2- 9 Comparison of DMS Cyclone and Froth Flotation Performance

Cleaning Unit	Operating Conditions	Concentrate Yield (%)	Concentrate Ash (%)	Concentrate Combustibles (%)	Clean Coal Recovery r_c	Enrichment Ratio
DMS Cyclone	PSD 1.18-4.5mm; SG=1.753	51.10	27.92	72.08	63.74	1.21
Froth Flotation	Diesel14g/kg Coal; T=1min	51.16	33.61	65.11	65.21	1.46

Table A2- 10 Rank Classification by Vitrinite Reflectance Analysis (RoV%)

SAMPLE IDENTIFICATION		SG=1.5 FLOAT
VITRINITE REFLECTANCE	**Rmax**	
	st. dev.	
	Rrandom	0.66
	st. dev.	0.089
	maximum reading	1.89
	minimum reading	0.49
	number of points	102
	RANK CATEGORY	Medium-Rank C

Table A2- 11 Petrographic Comparison of Maceral and Mineral Composition (vol %)

SEPARATION TECHNIQUE			GRAVITY (1180µm≤x≤4500µm)					FLOTATION (DIESEL 14g/kg Coal)			
SAMPLE IDENTIFICATION		FEED	FLOAT SG				TAILINGS	TIME (min)			TAILINGS
			1.5	1.6	1.7	1.8		1	3	9	
MACERAL GROUP	VITRINITE	4.3	14.0	10.2	3.6	4.7	1.8	5.9	5.9	1.6	0.8
	LIPTINITE	0.8	2.5	2.2	3.0	2.5	0.4	1.6	0.8	1.4	0.0
	INERTINITE	48.8	79.4	79.2	81.9	76.4	16.6	70.1	61.1	26.8	10.8
MINERAL GROUP	CLAYS	8.3	0.8	3.2	2.2	2.1	11.8	4.6	3.7	5.7	9.0
	QUARTZ	29.5	1.9	2.8	6.5	12.1	41.2	14.2	23.2	49.0	64.8
	PYRITE	3.0	0.2	0.2	0.2	0.2	16.4	0.8	1.0	4.9	5.4
	CARBONATE	5.3	1.2	2.2	2.6	2.0	11.8	2.8	4.3	10.6	9.2
	OTHER MINERALS	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

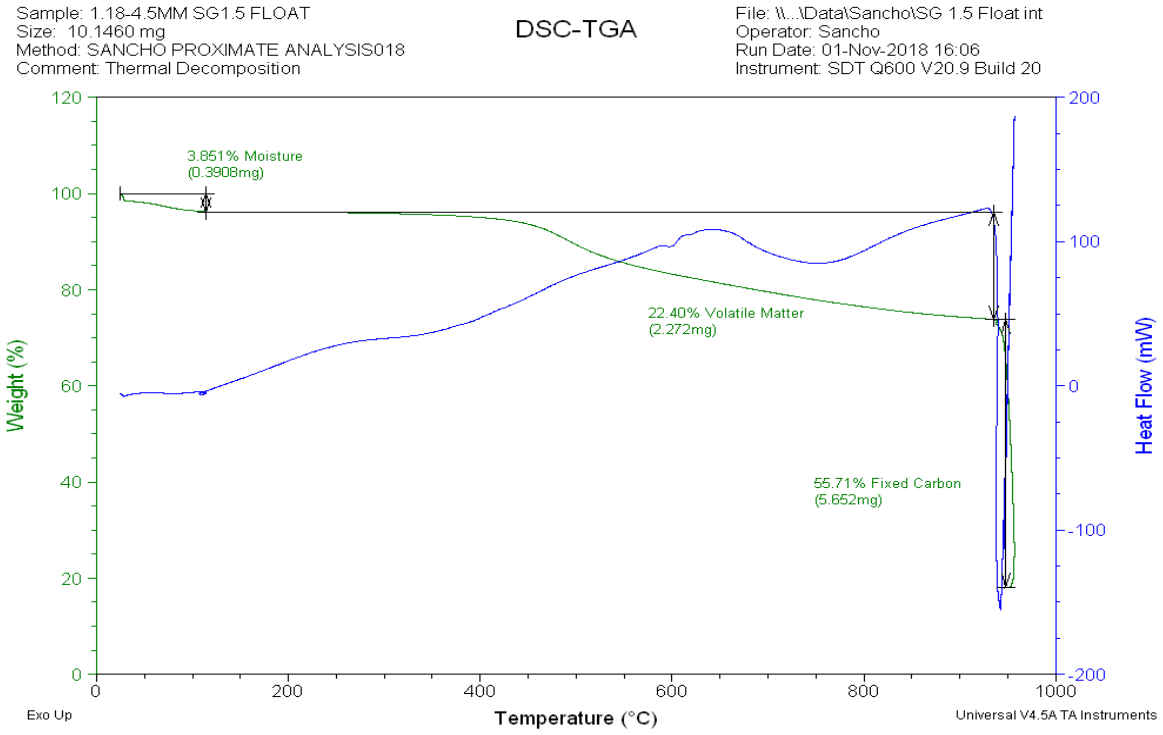


Figure A2- 2 DSC-TGA Plot at SG=1.5 (1.18-4.5mm)

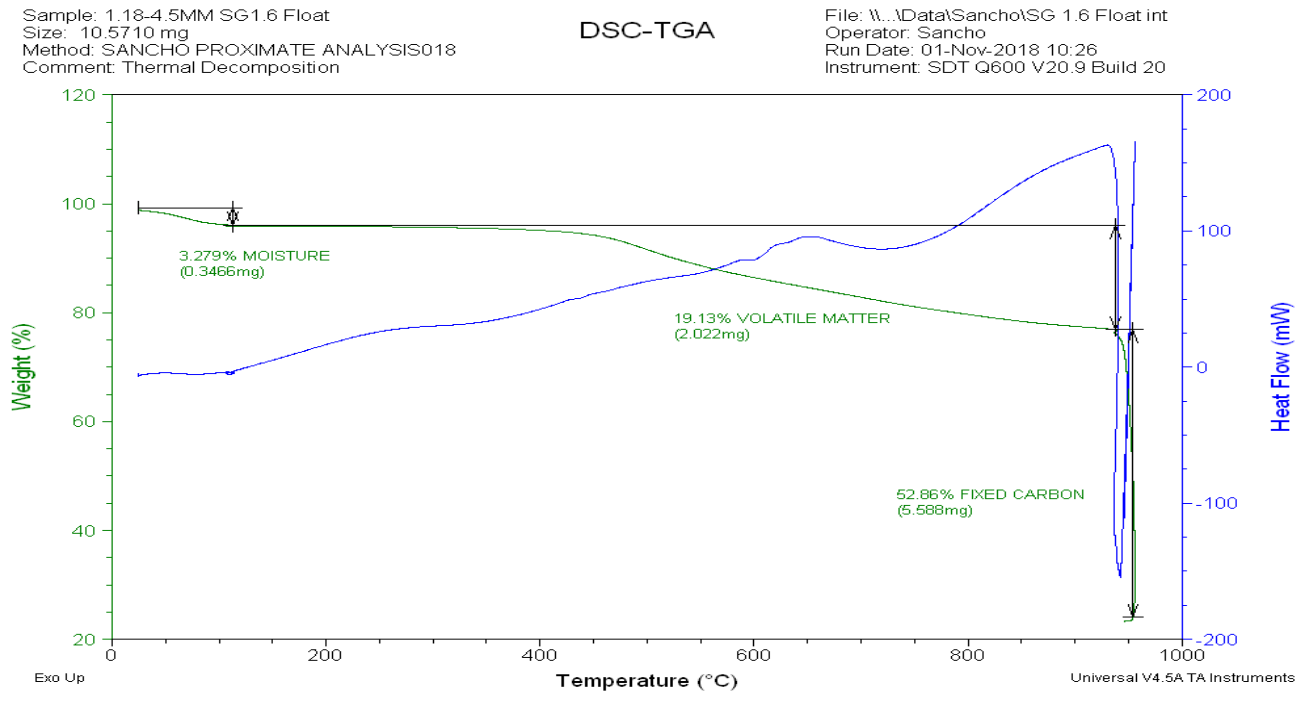


Figure A2- 3 DSC-TGA Plot at SG=1.6 (1.18-4.5mm)

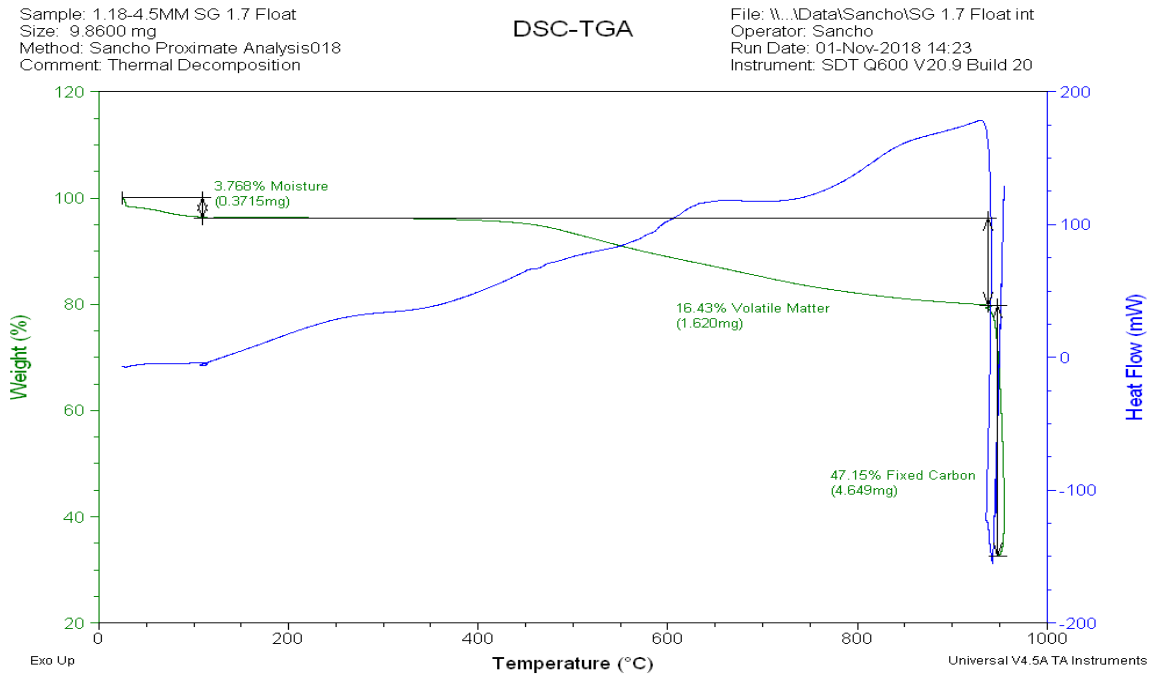


Figure A2- 4 DSC-TGA Plot at SG=1.7 (1.18-4.5mm)

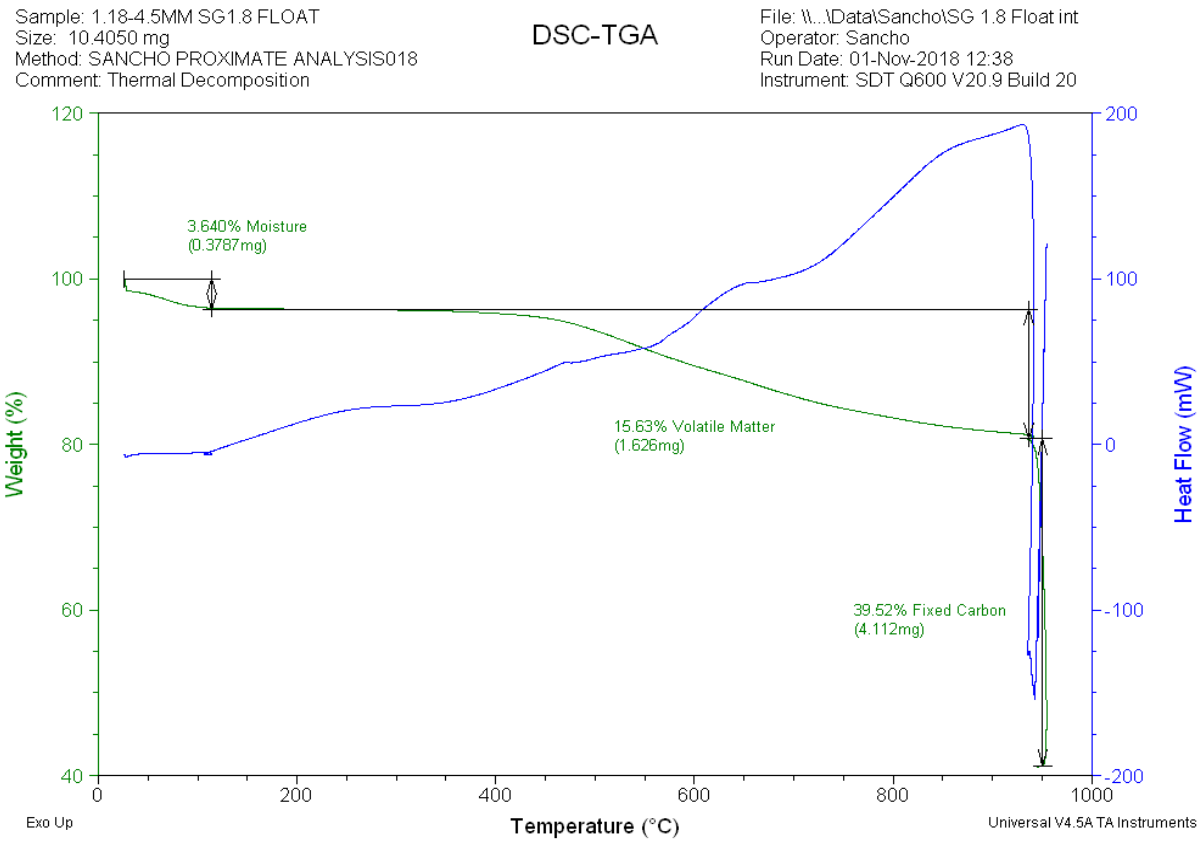


Figure A2- 5 DSC-TGA Plot at SG=1.8 (1.18-4.5mm)

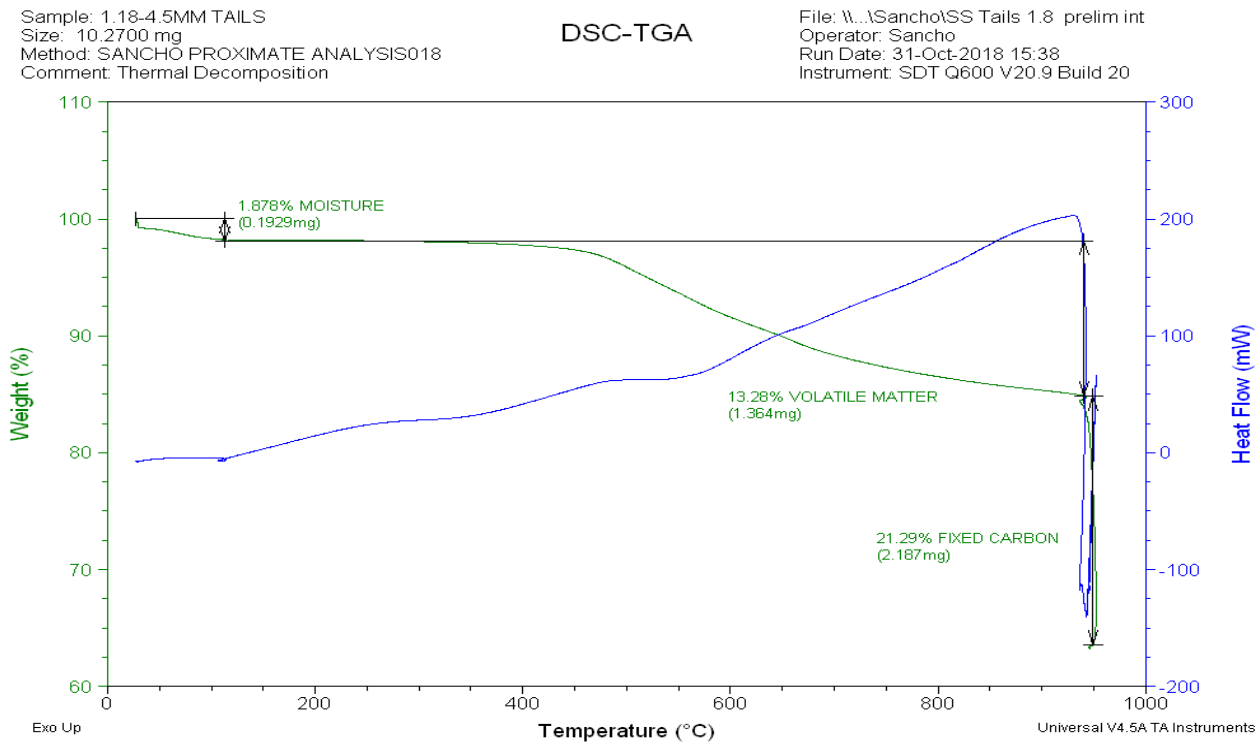


Figure A2- 6 DSC-TGA Plot at SG>1.8 (1.18-4.5mm)

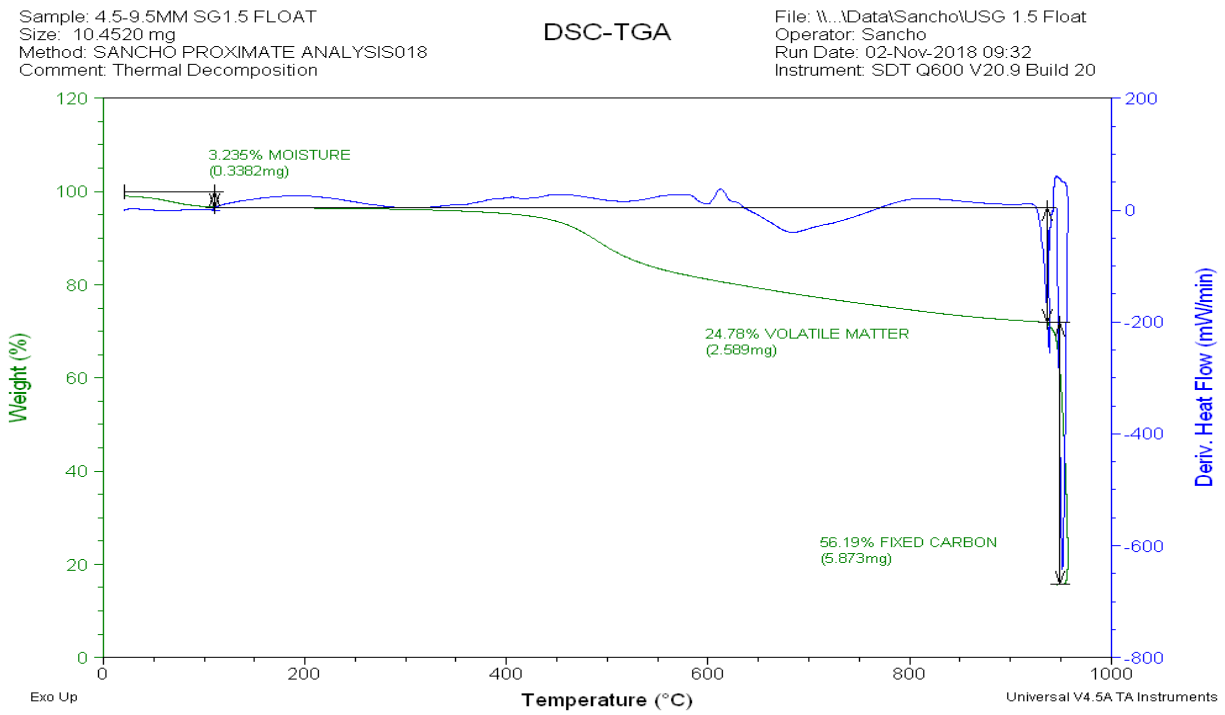


Figure A2- 7 DSC-TGA Plot at SG=1.5 (4.5-9.5mm)

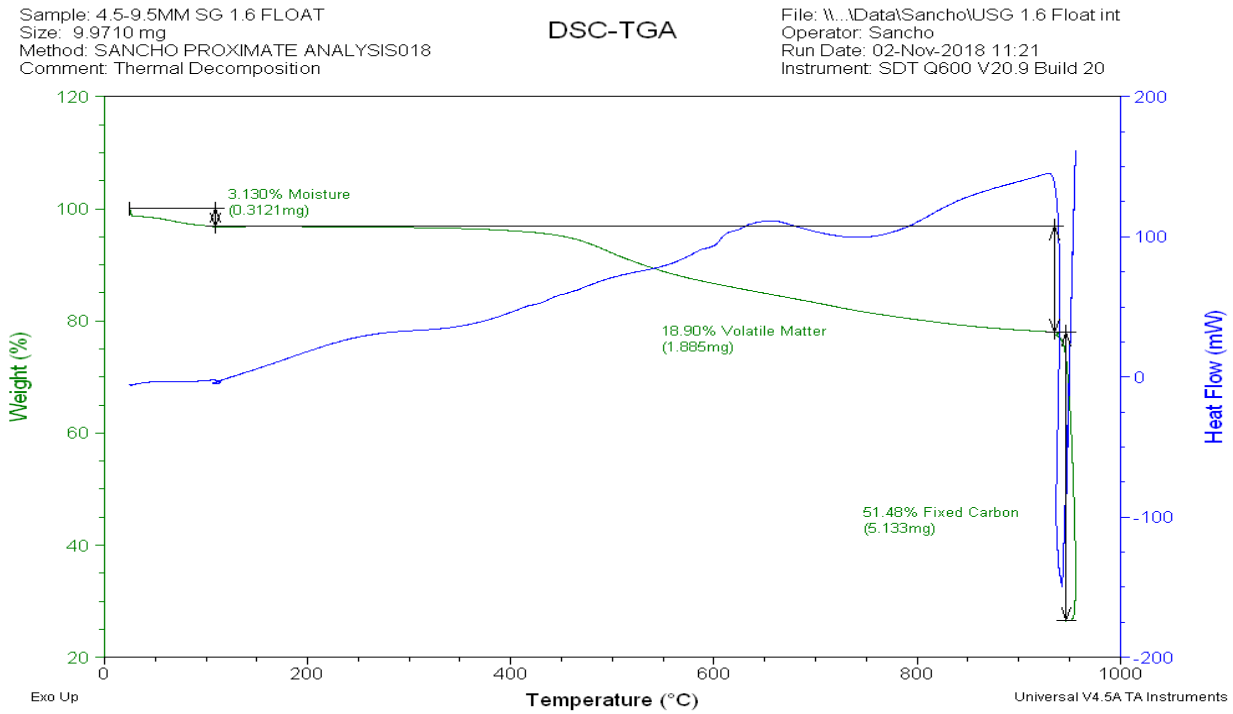


Figure A2- 8 DSC-TGA Plot at SG=1.6 (4.5-9.5mm)

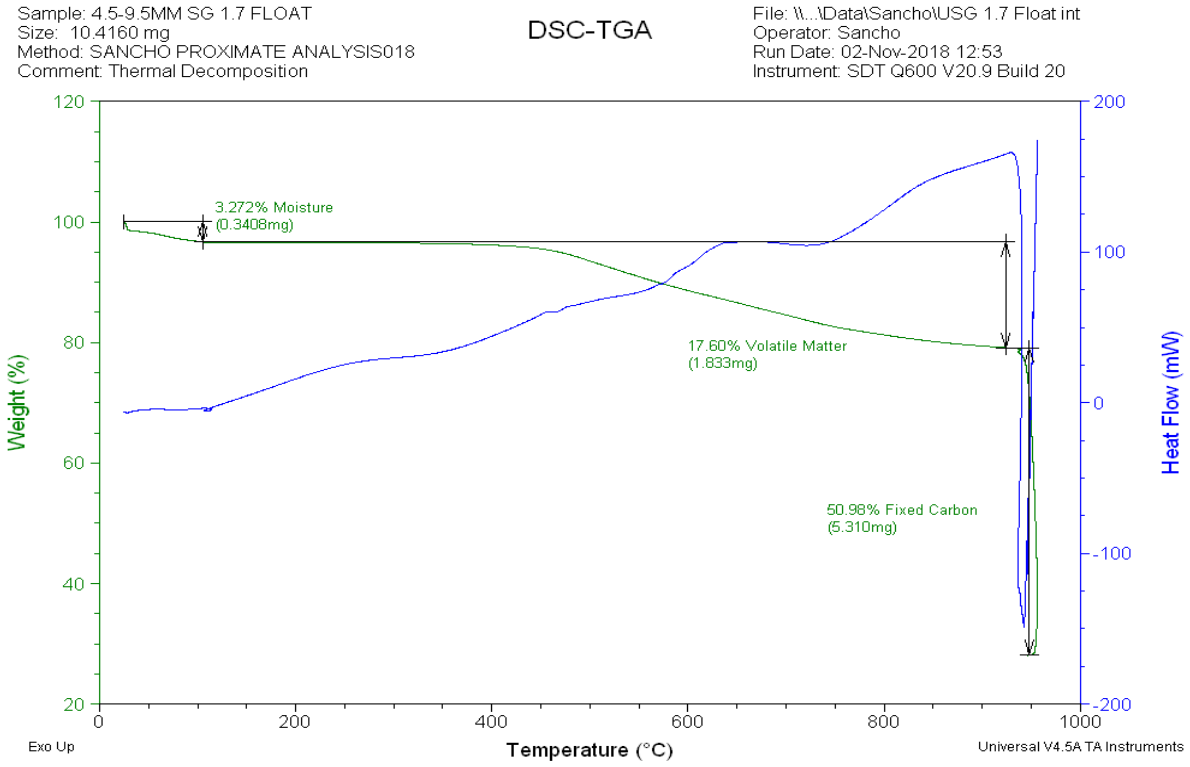


Figure A2- 9 DSC-TGA Plot at SG=1.7 (4.5-9.5mm)

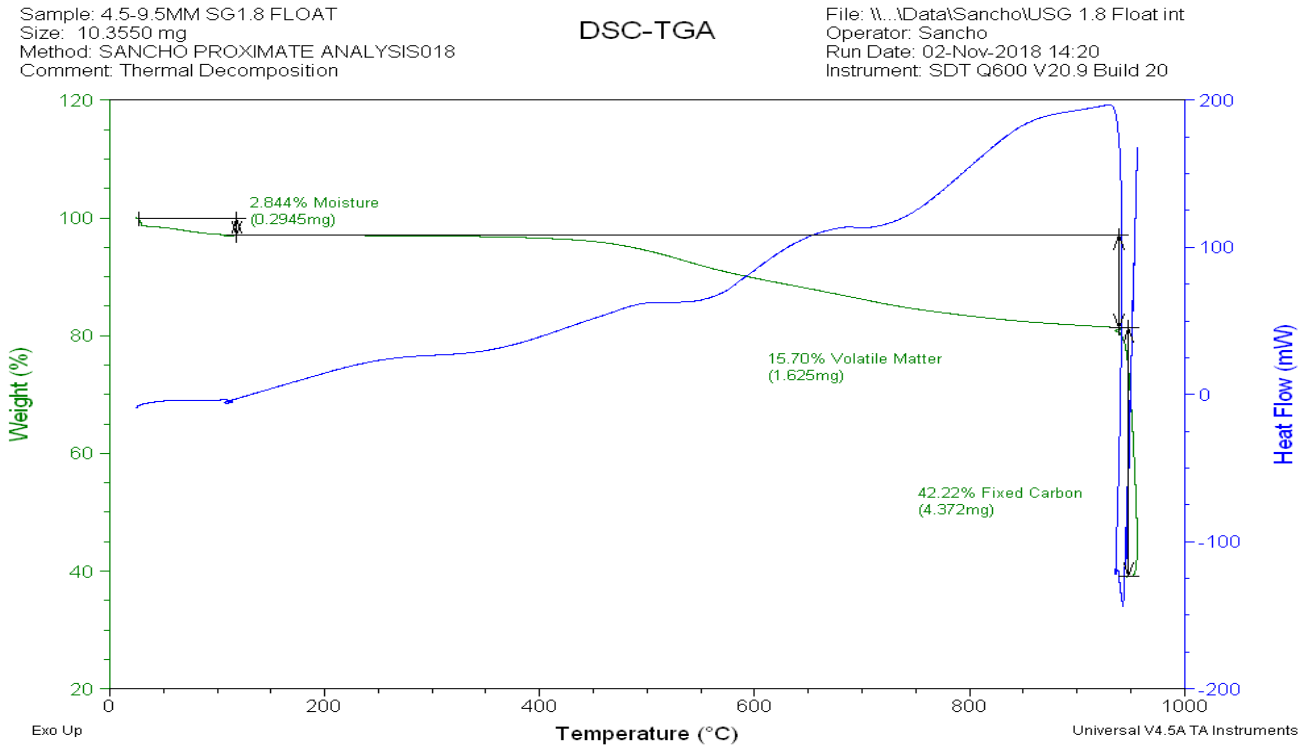


Figure A2- 10 DSC-TGA Plot at SG=1.8 (4.5-9.5mm)

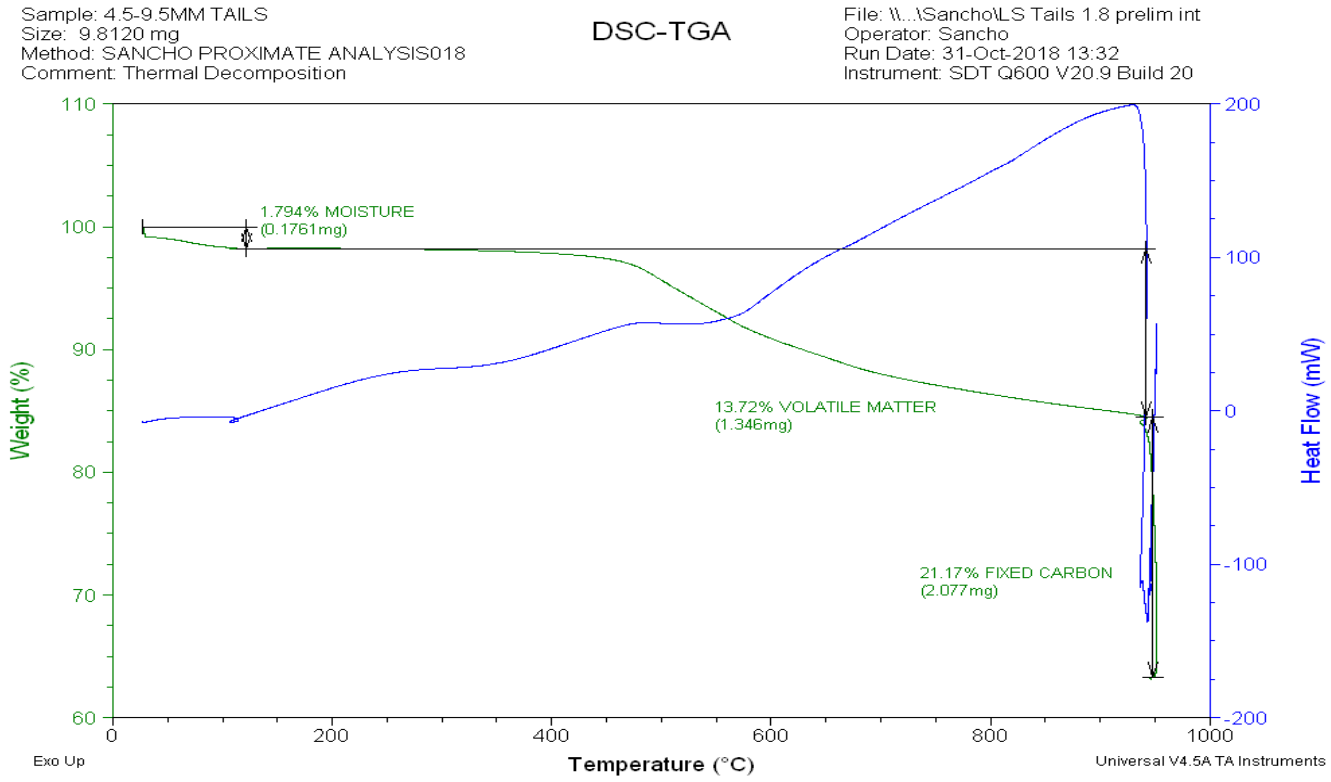


Figure A2- 11 DSC-TGA Plot at SG>1.8 (4.5-9.5mm)

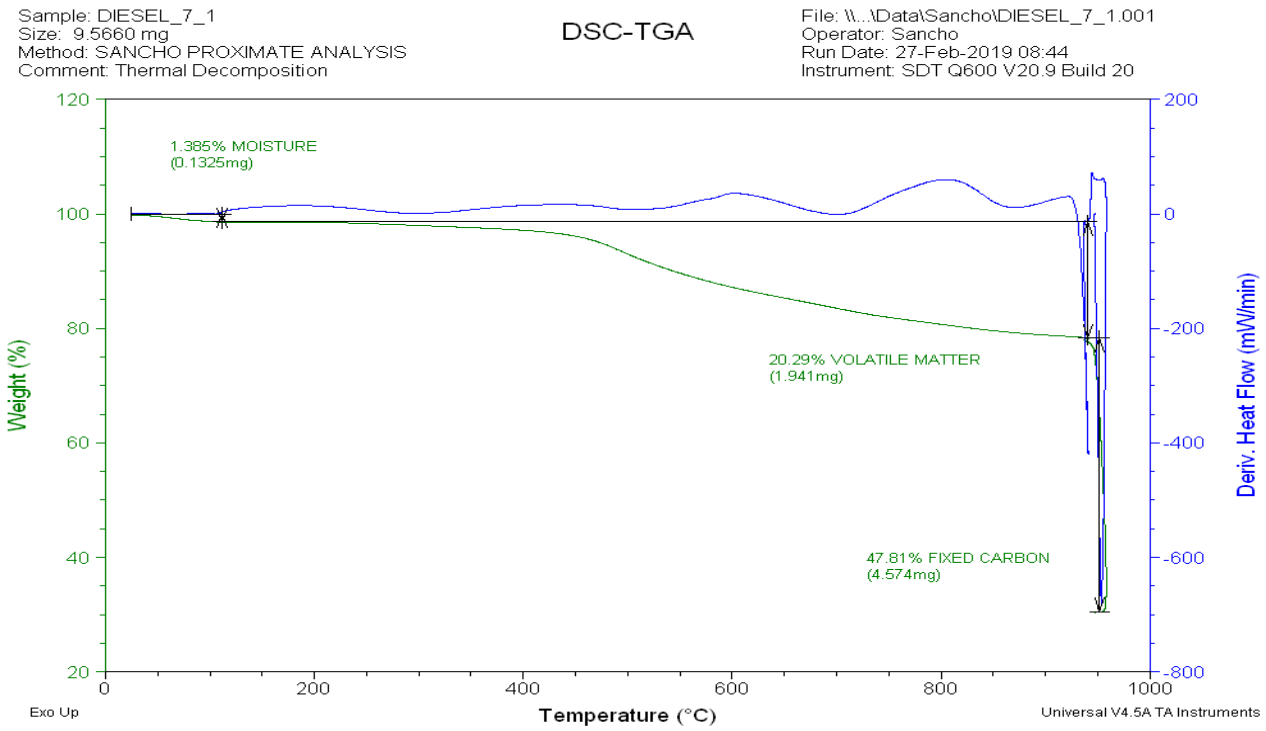


Figure A2- 12 DSC-TGA Plot at T=1: 7gDiesel/kg Coal

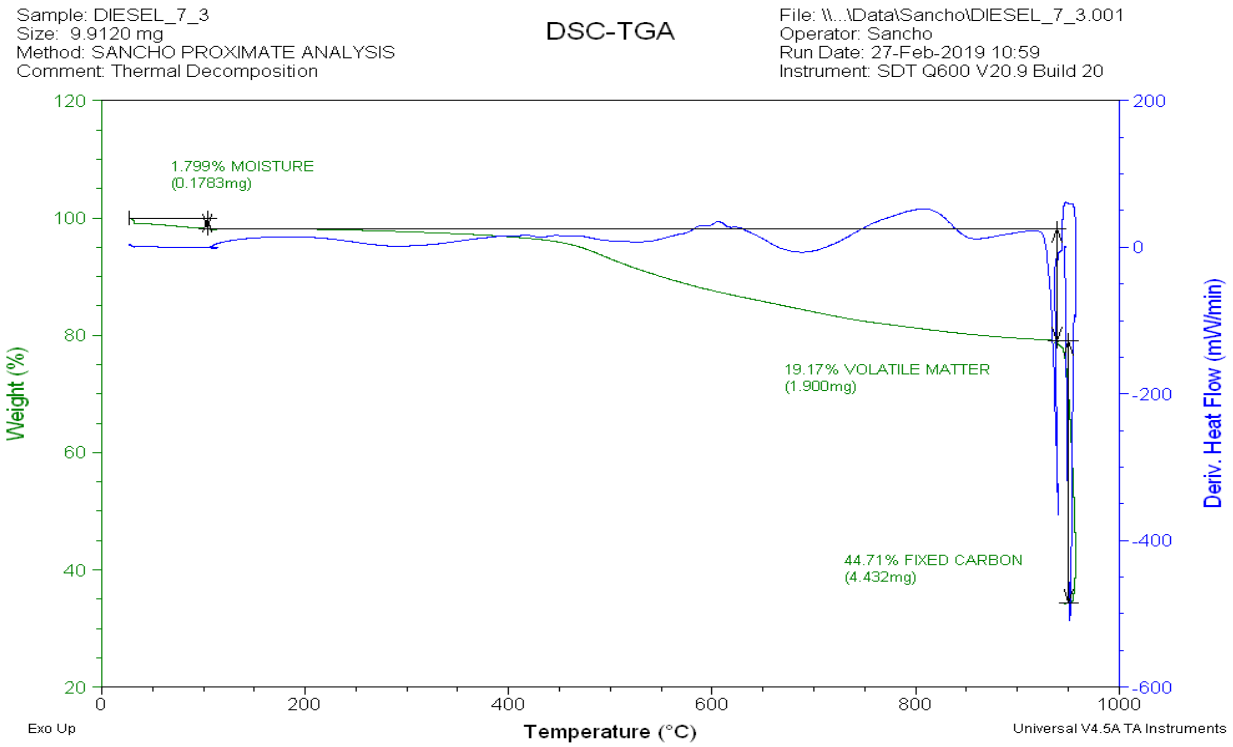


Figure A2- 13 DSC-TGA Plot at T=3: 7gDiesel/kg Coal

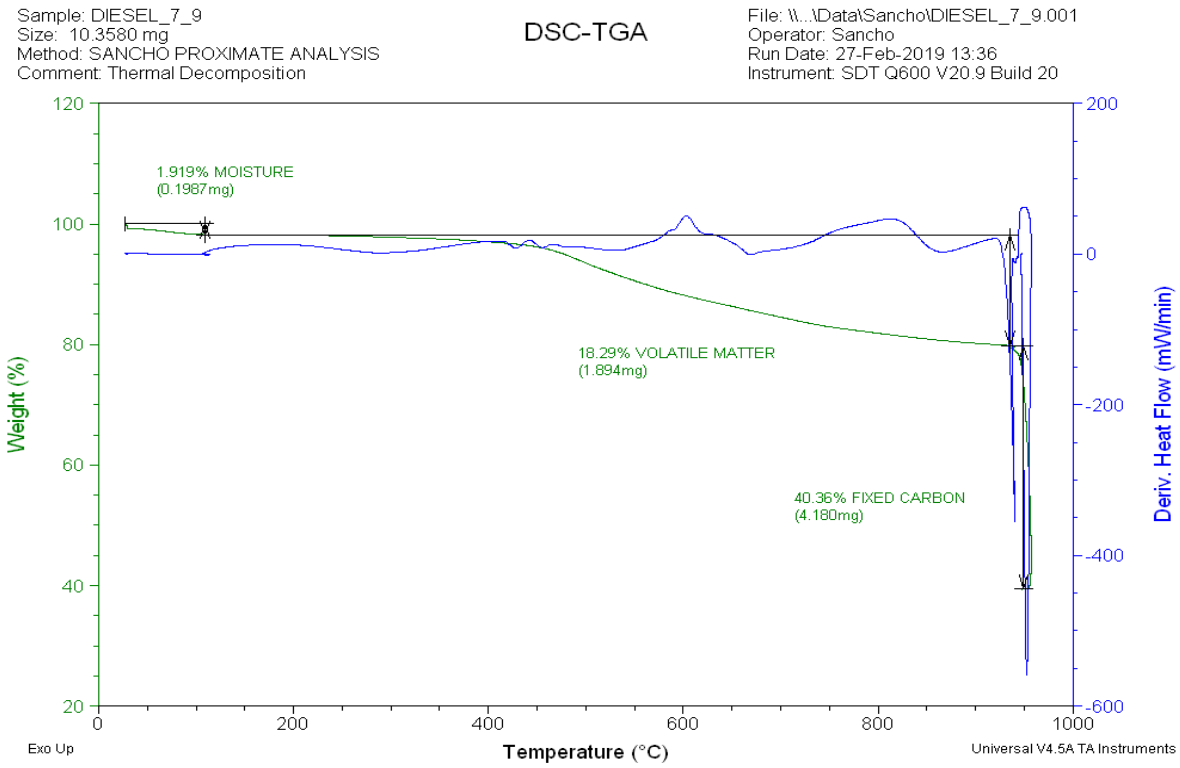


Figure A2- 14 DSC-TGA Plot at T=9: 7gDiesel/kg Coal

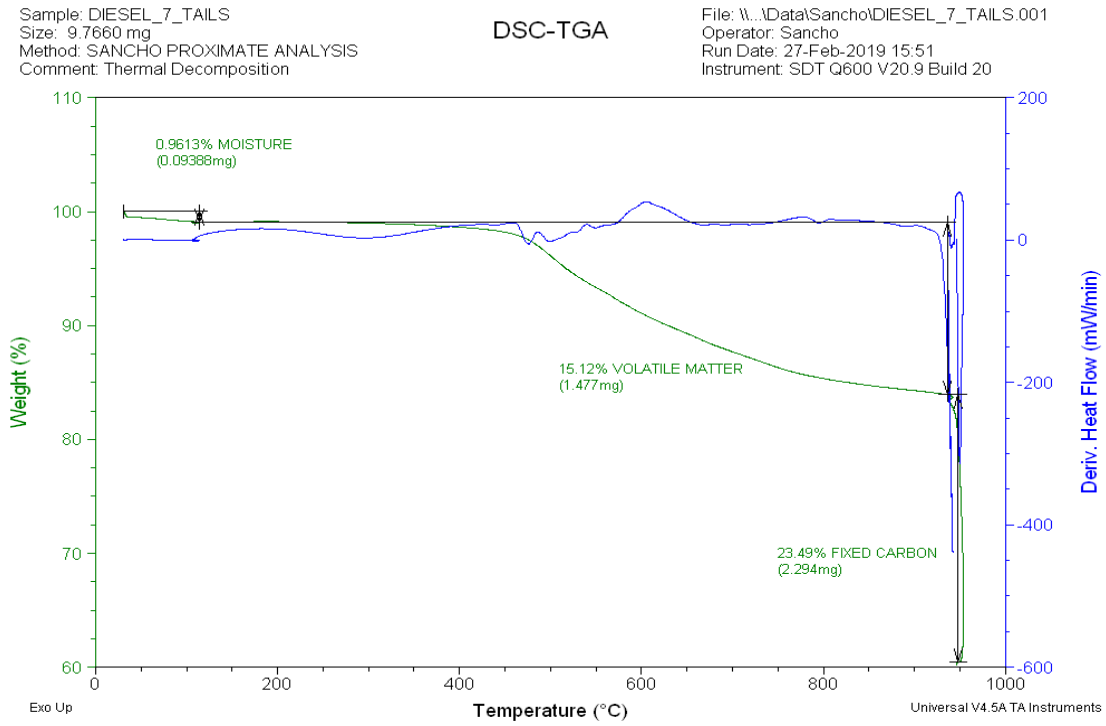


Figure A2- 15 DSC-TGA Tails Plot: 7gDiesel/kg Coal

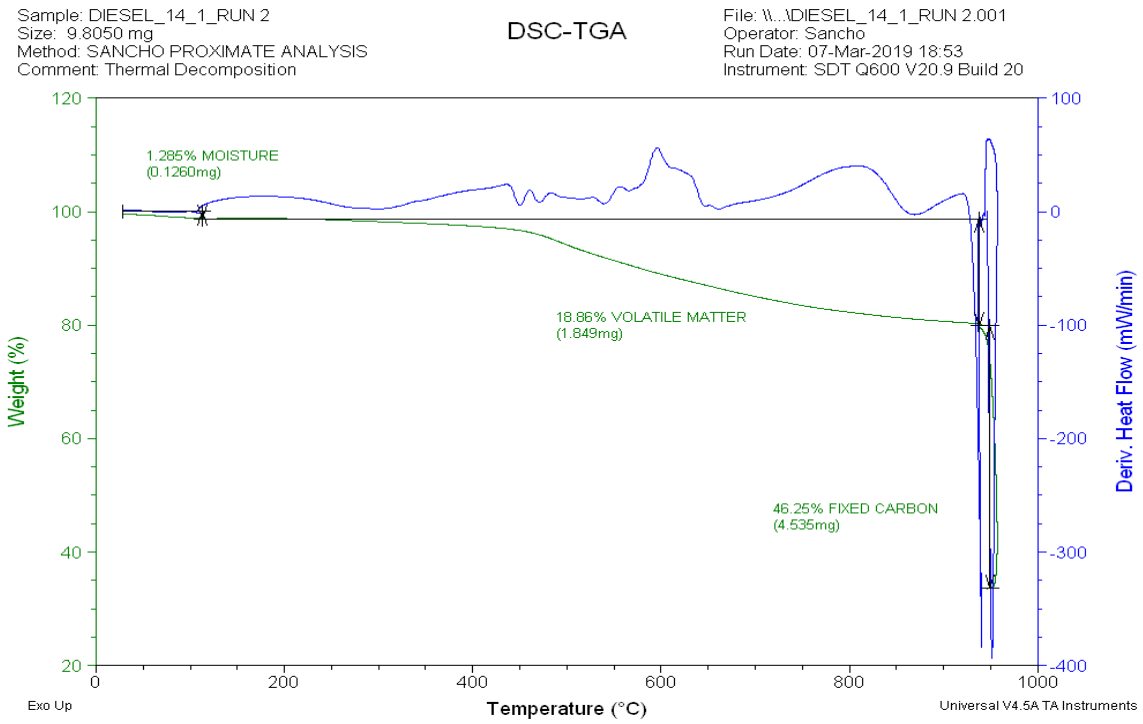


Figure A2- 16 DSC-TGA Plot at T=1: 14gDiesel/kg Coal

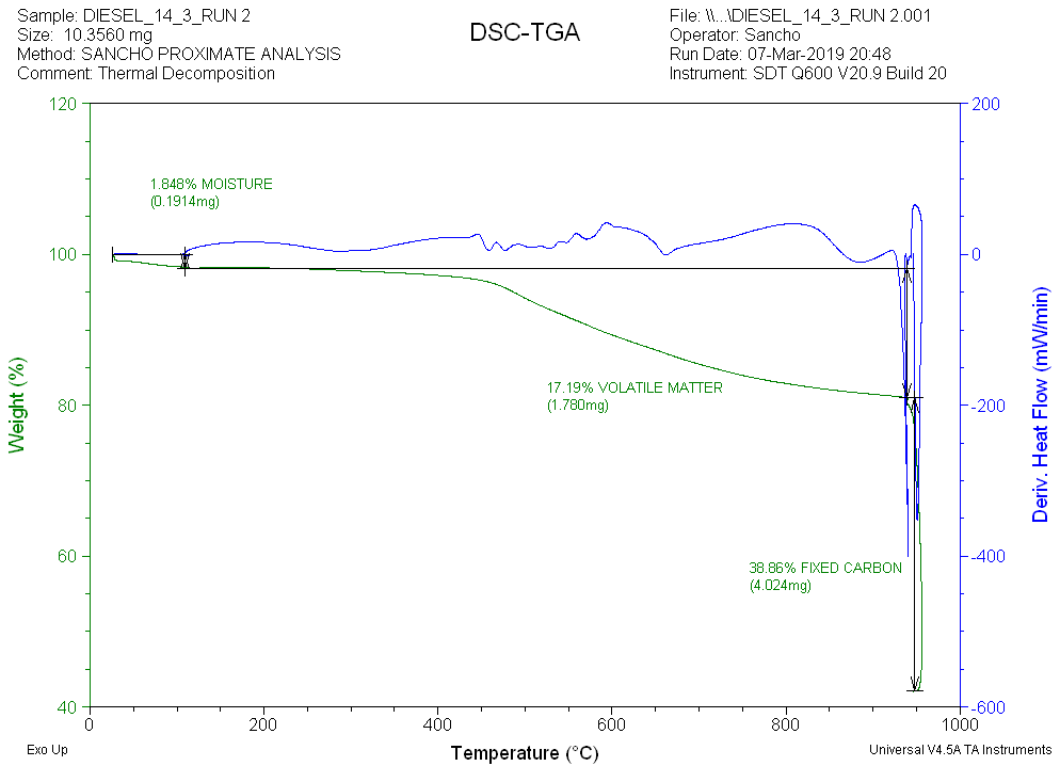


Figure A2- 17 DSC-TGA Plot at T=3: 14gDiesel/kg Coal

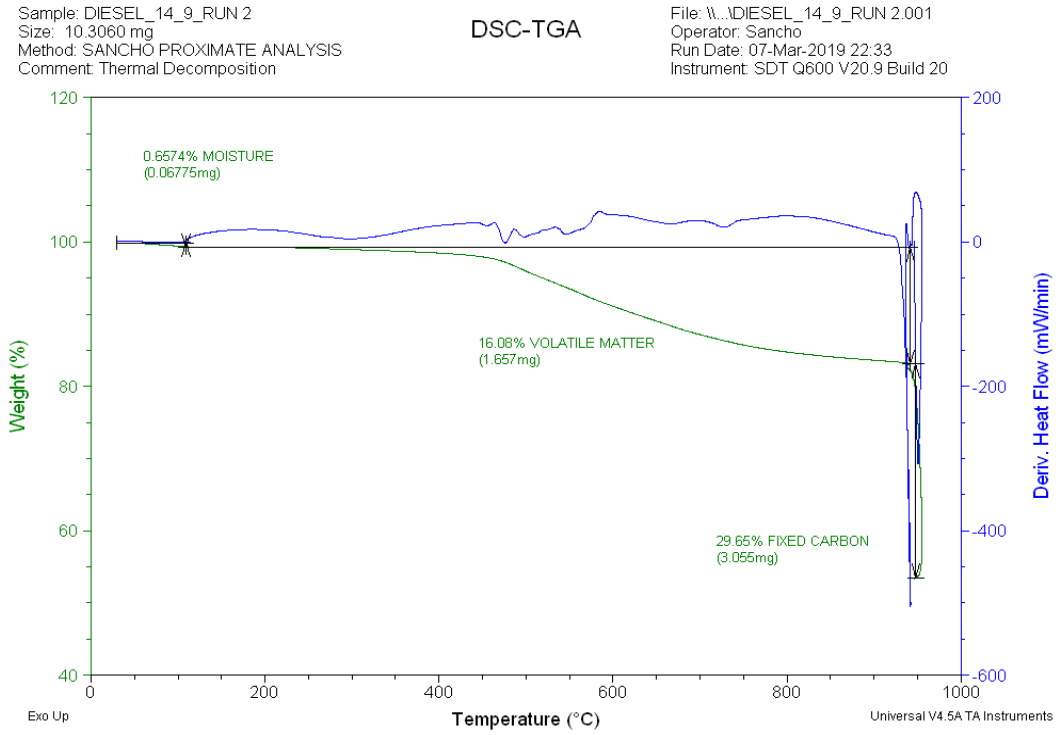


Figure A2- 18 DSC-TGA Plot at T=9: 14gDiesel/kg Coal

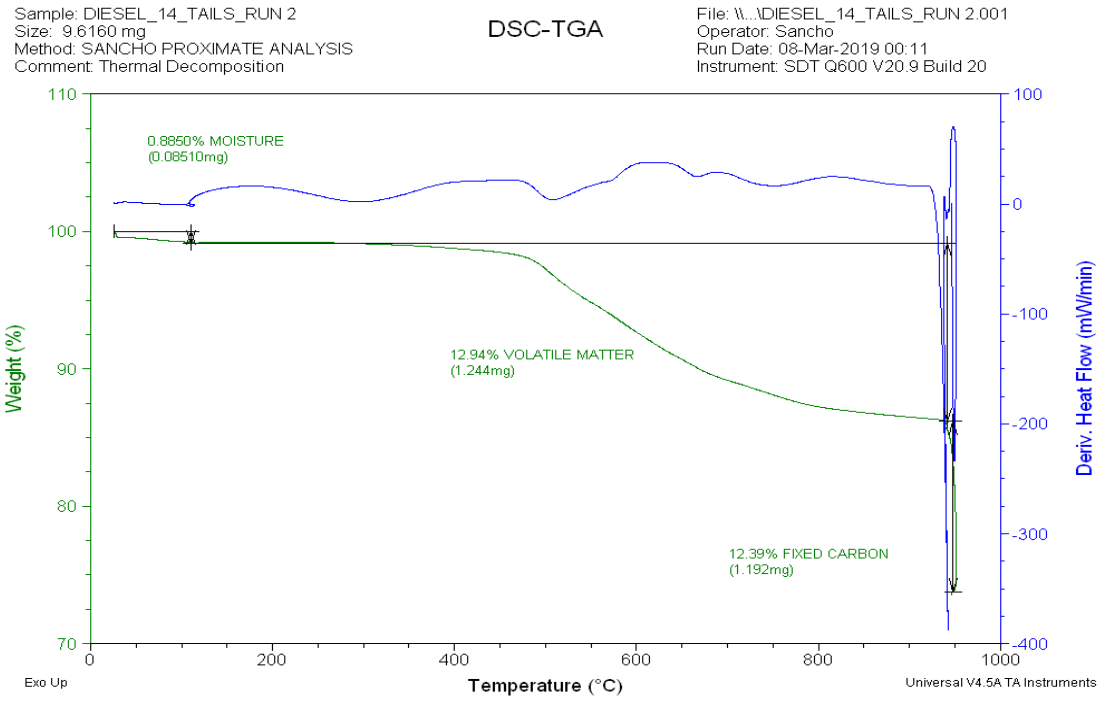


Figure A2- 19 DSC-TGA Tails Plot: 14gDiesel/kg Coal

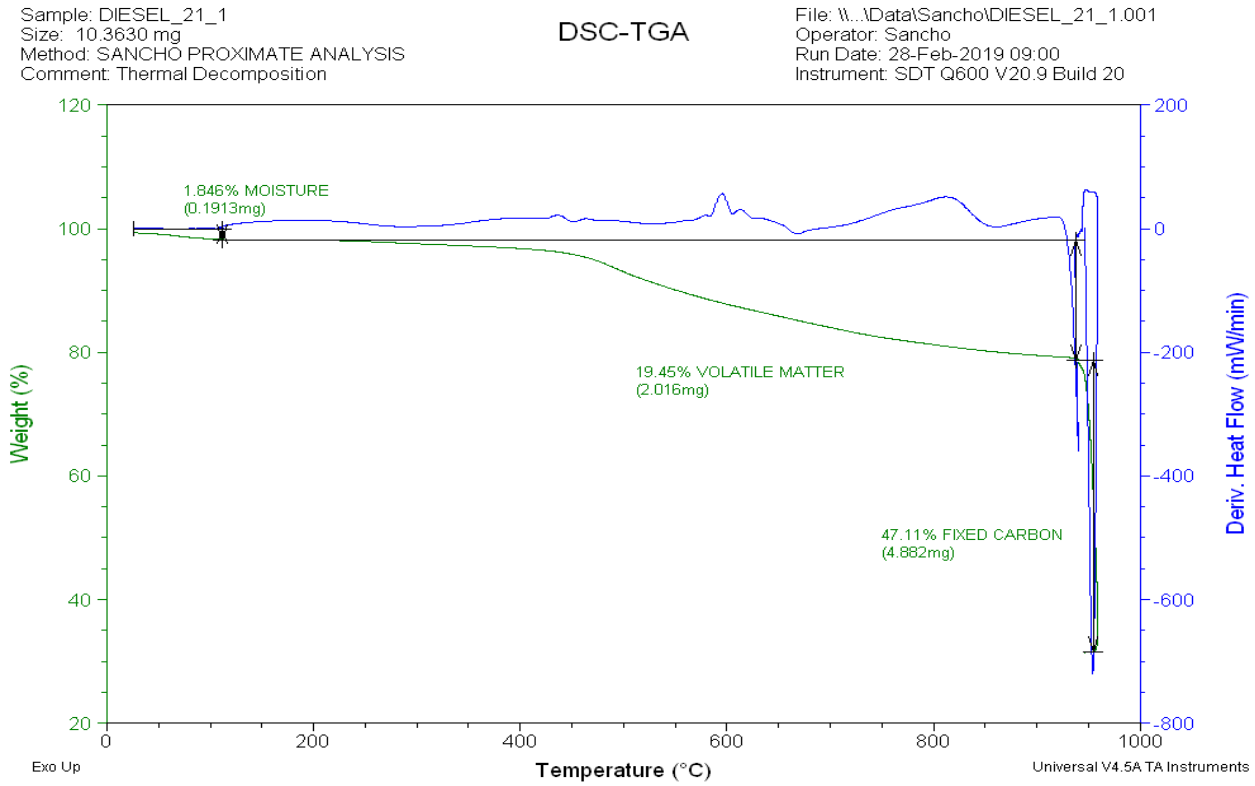


Figure A2- 20 DSC-TGA Plot at T=1: 21gDiesel/kg Coal

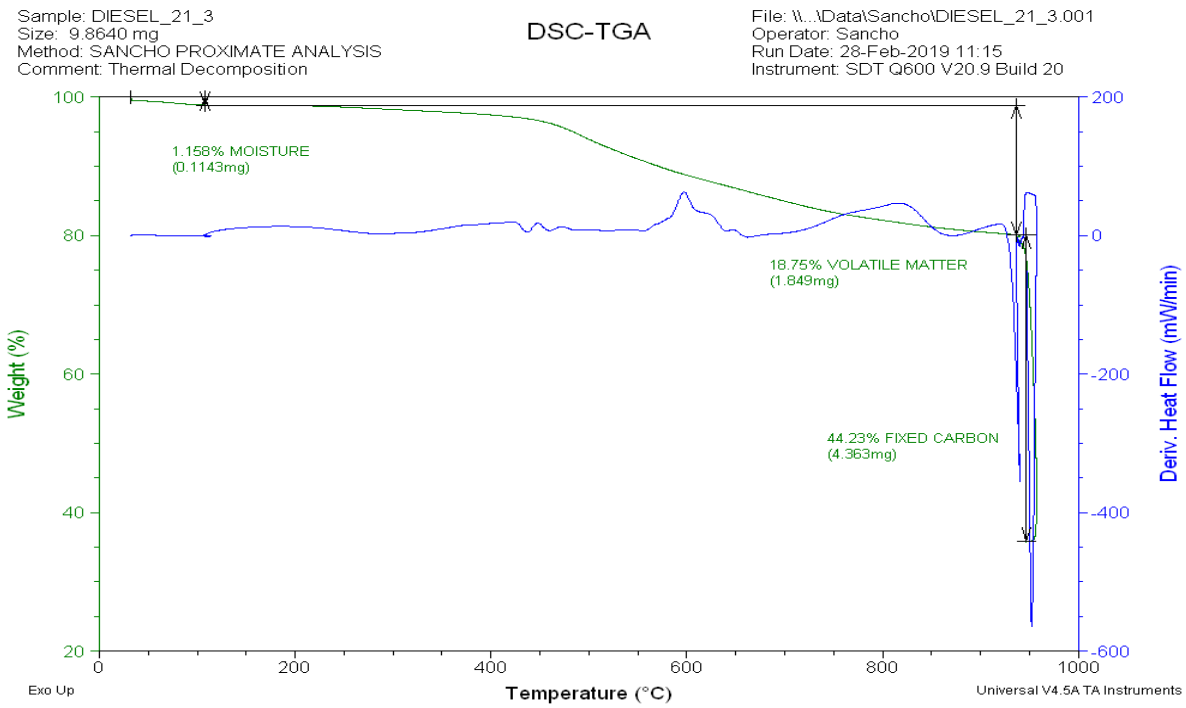


Figure A2- 21 DSC-TGA Plot at T=3: 21gDiesel/kg Coal

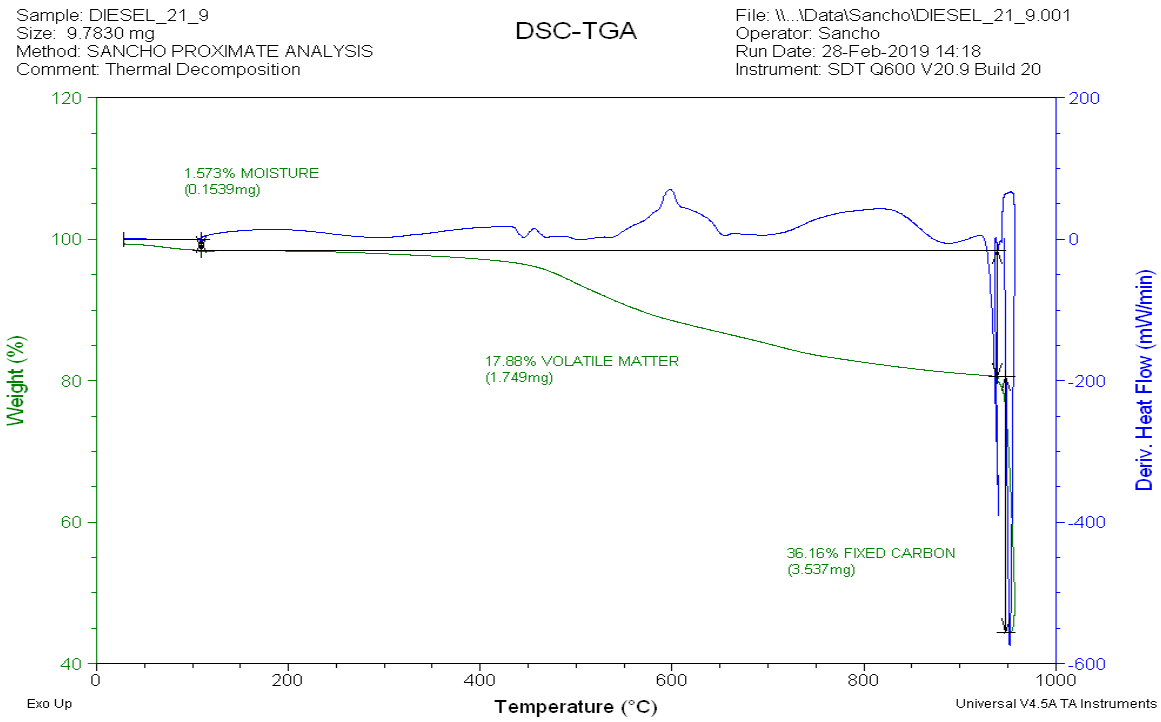


Figure A2- 22 DSC-TGA Plot at T=9: 21gDiesel/kg Coal

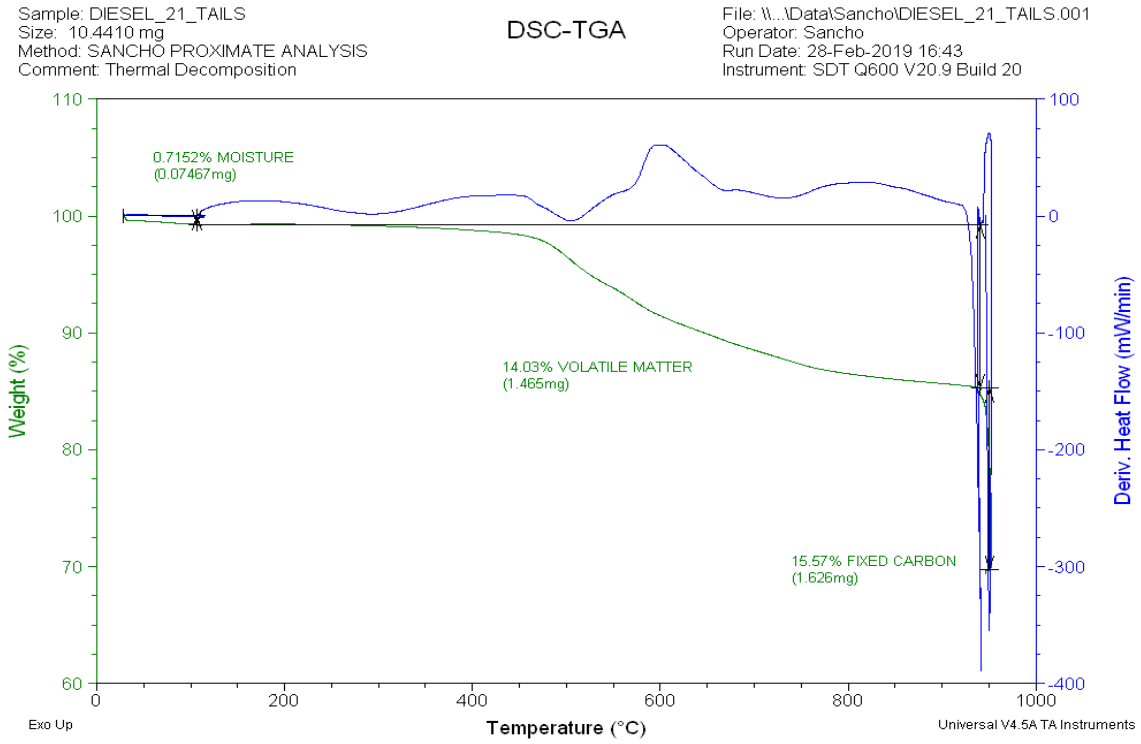


Figure A2- 23 DSC-TGA Tails Plot: 21gDiesel/kg Coal

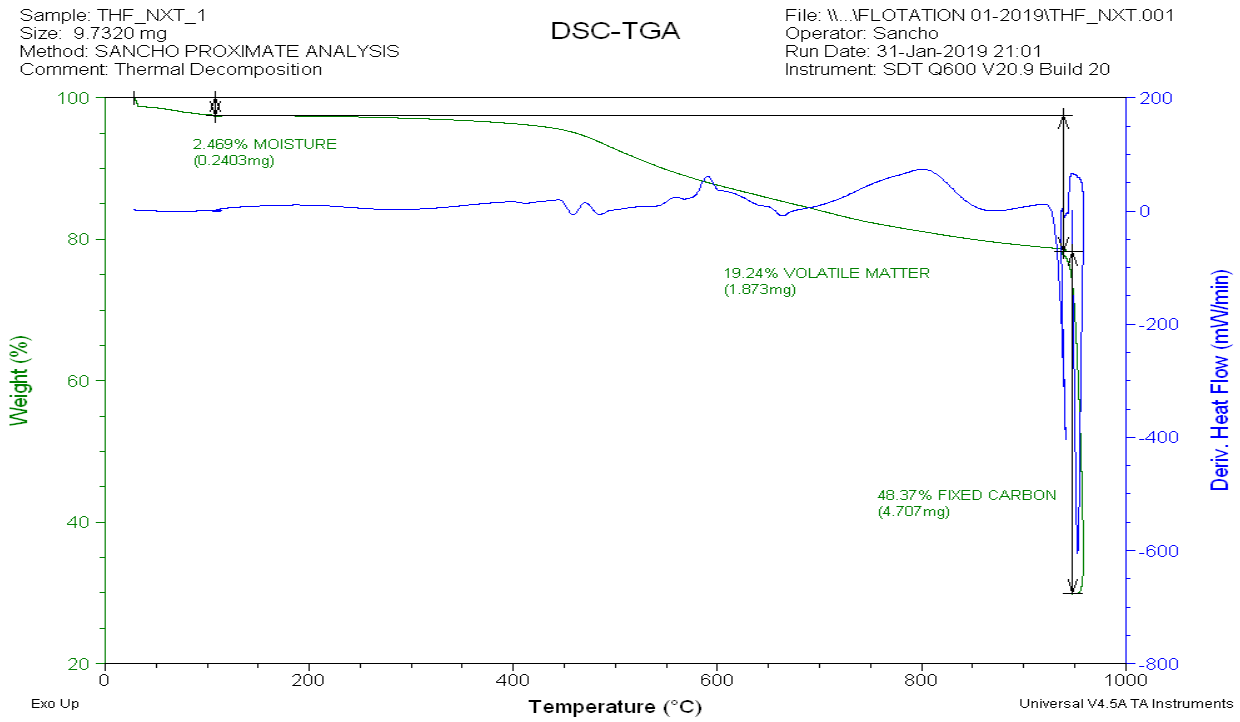


Figure A2- 24 DSC-TGA Plot at T=1: 14gTETRAHYDROFURAN/kg Coal

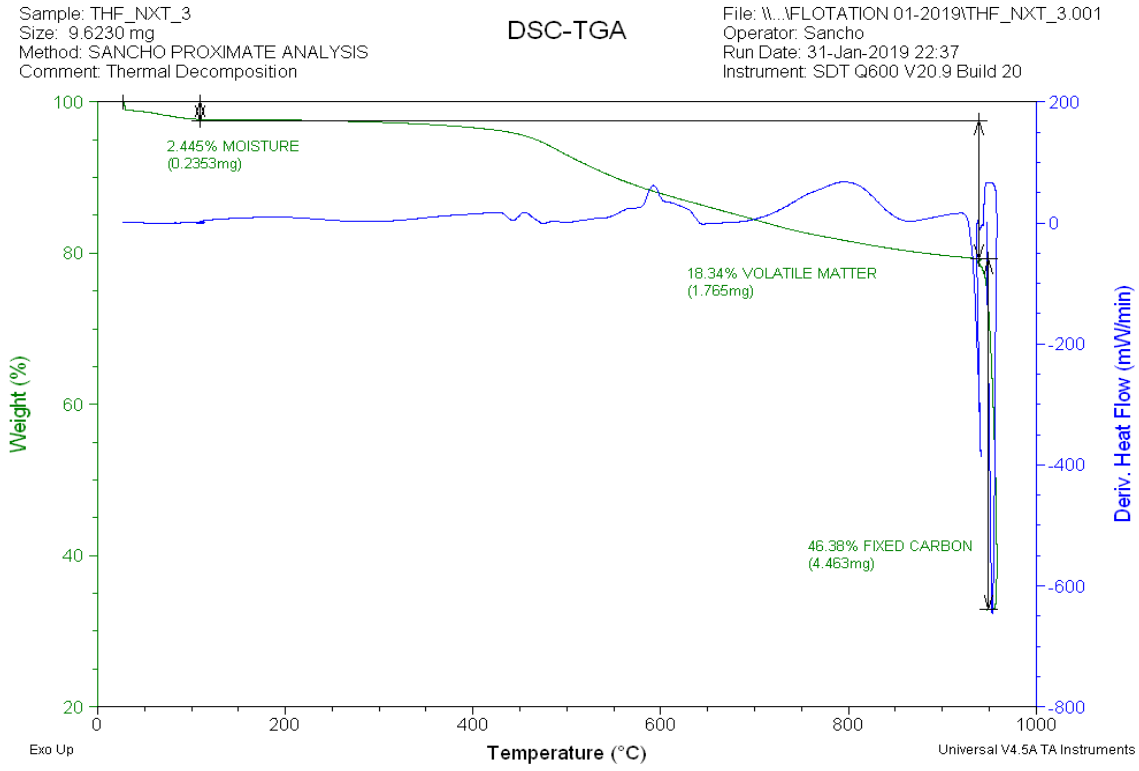


Figure A2- 25 DSC-TGA Plot at T=3: 14gTETRAHYDROFURAN/kg Coal

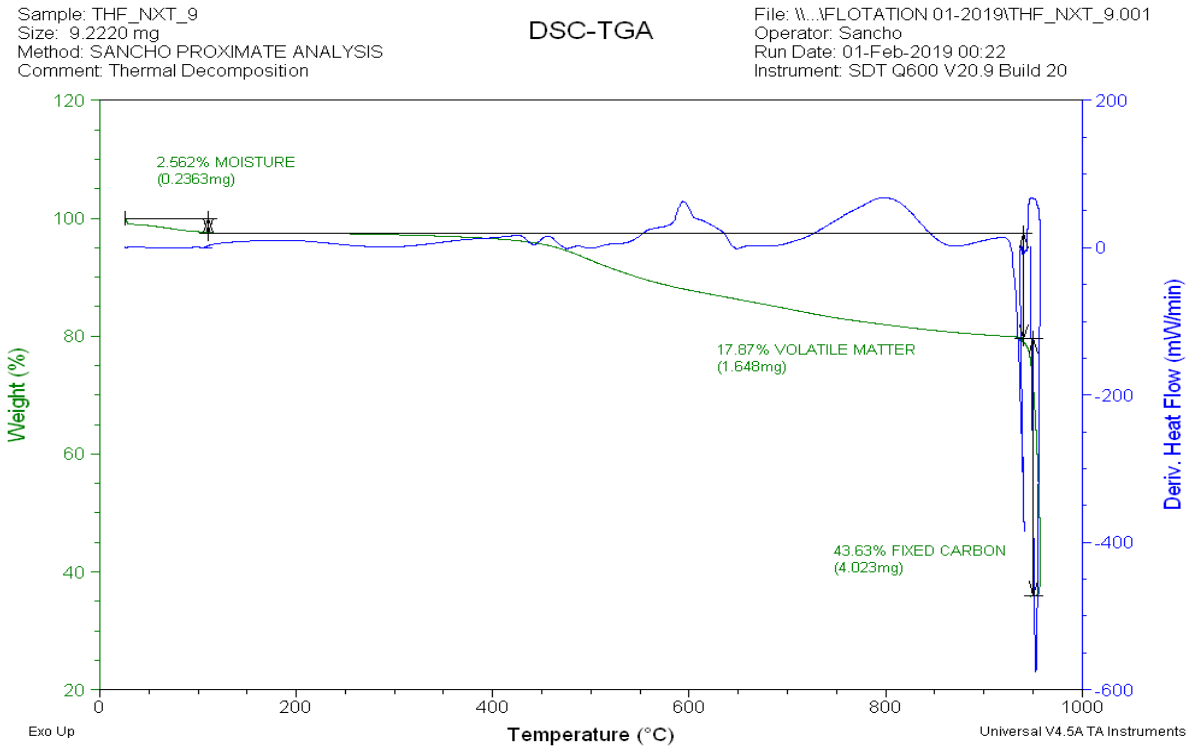


Figure A2- 26 DSC-TGA Plot at T=9: 14gTETRAHYDROFURAN/kg Coal

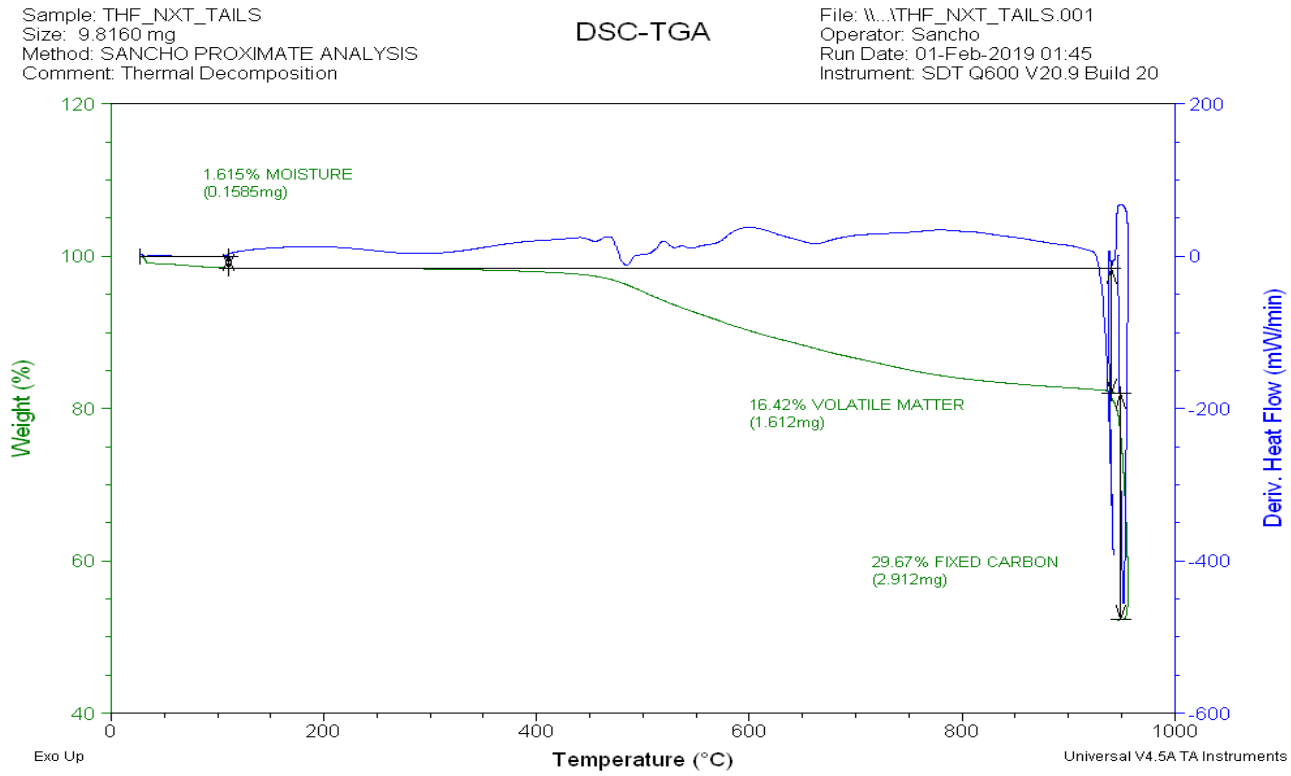


Figure A2- 27 DSC-TGA Tails Plot: 14gTETRAHYDROFURAN/kg Coal

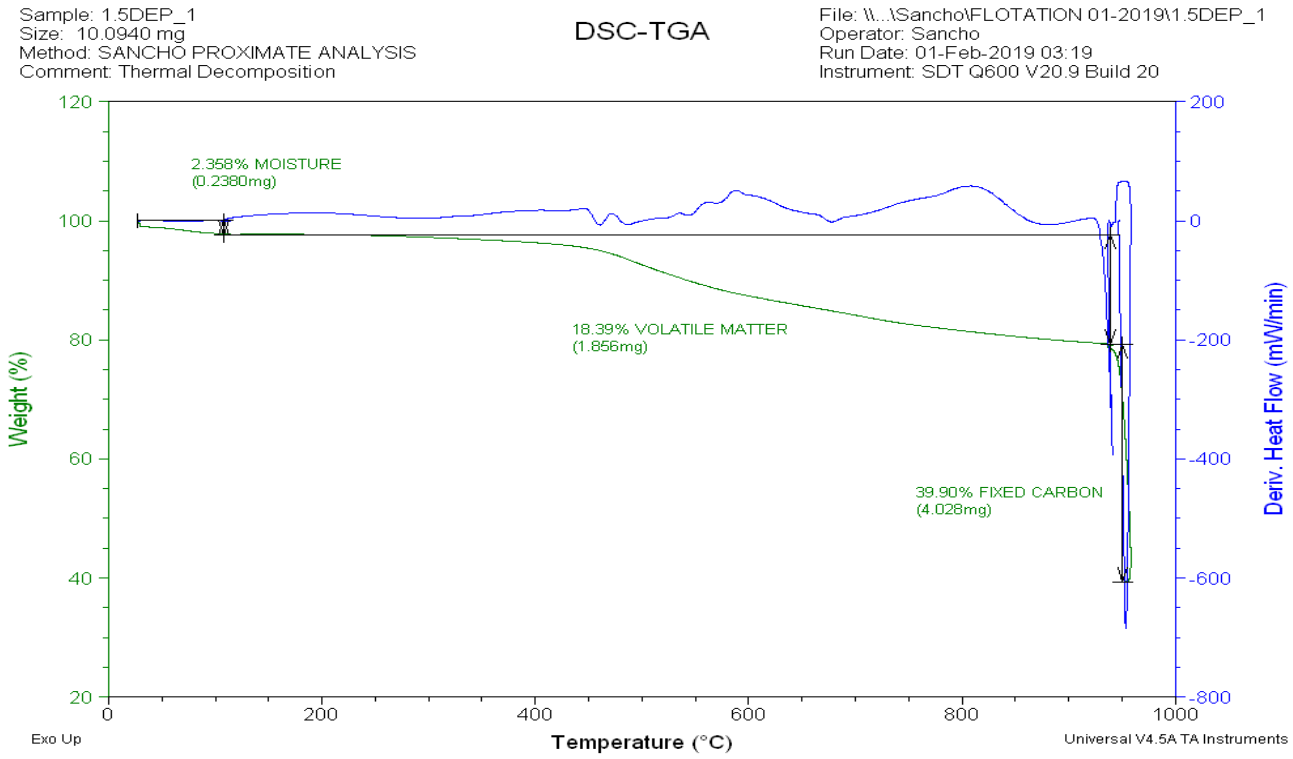


Figure A2- 28 DSC-TGA Plot at T=1: 5gSENDEP/kg Coal; 14gDIESEL/kg Coal

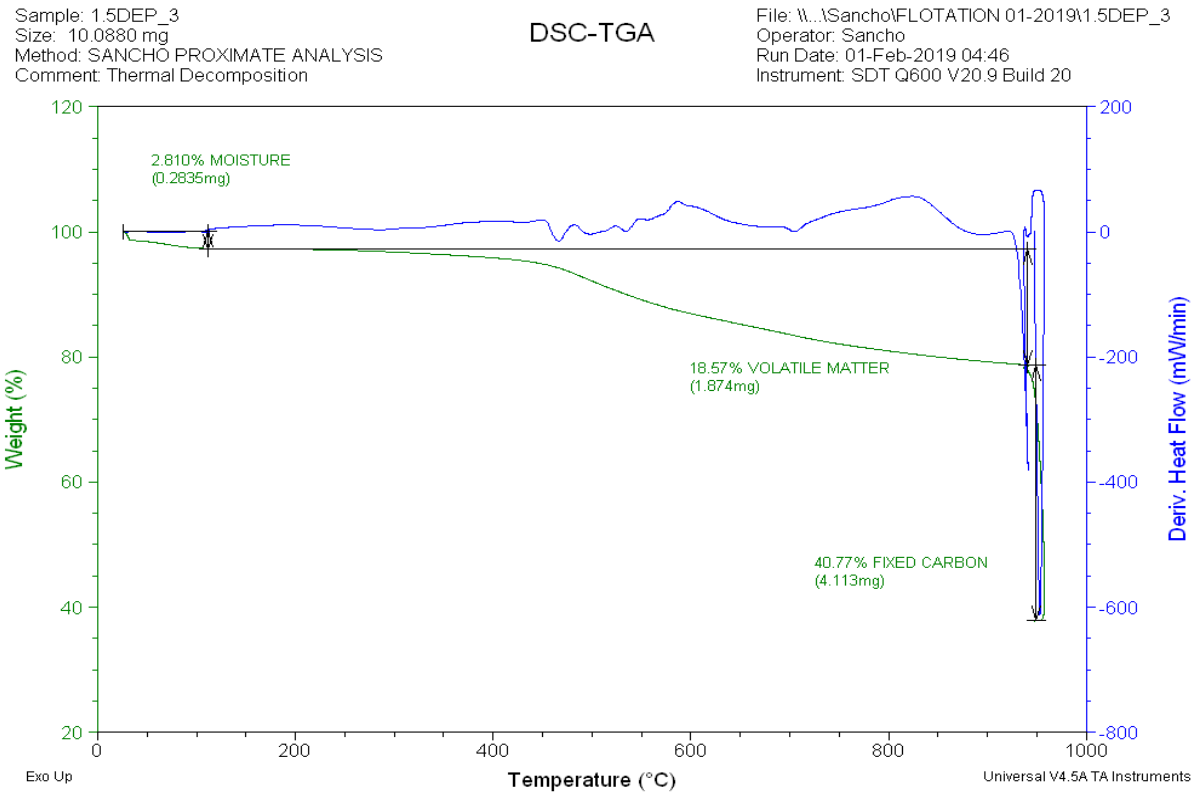


Figure A2- 29 DSC-TGA Plot at T=3: 5gSENDEP/kg Coal; 14gDIESEL/kg Coal

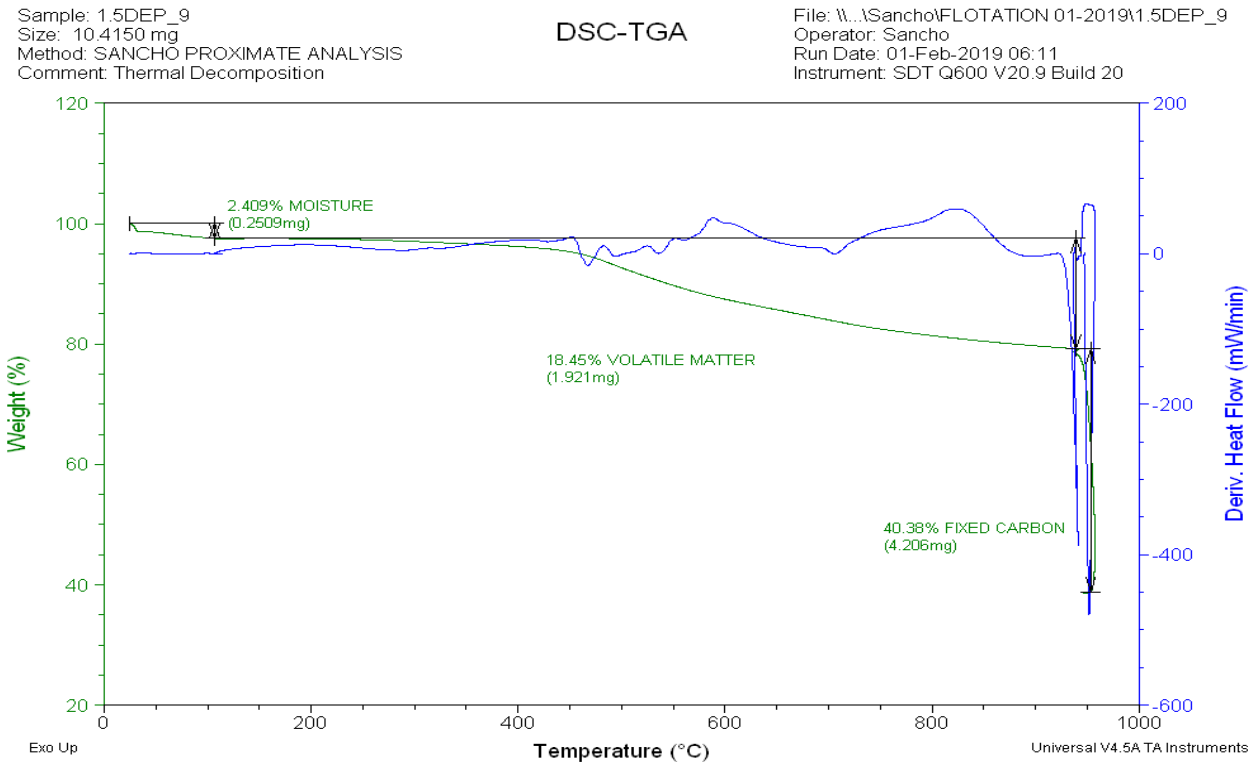


Figure A2- 30 DSC-TGA Plot at T=9: 5gSENDEP/kg Coal; 14gDIESEL/kg Coal

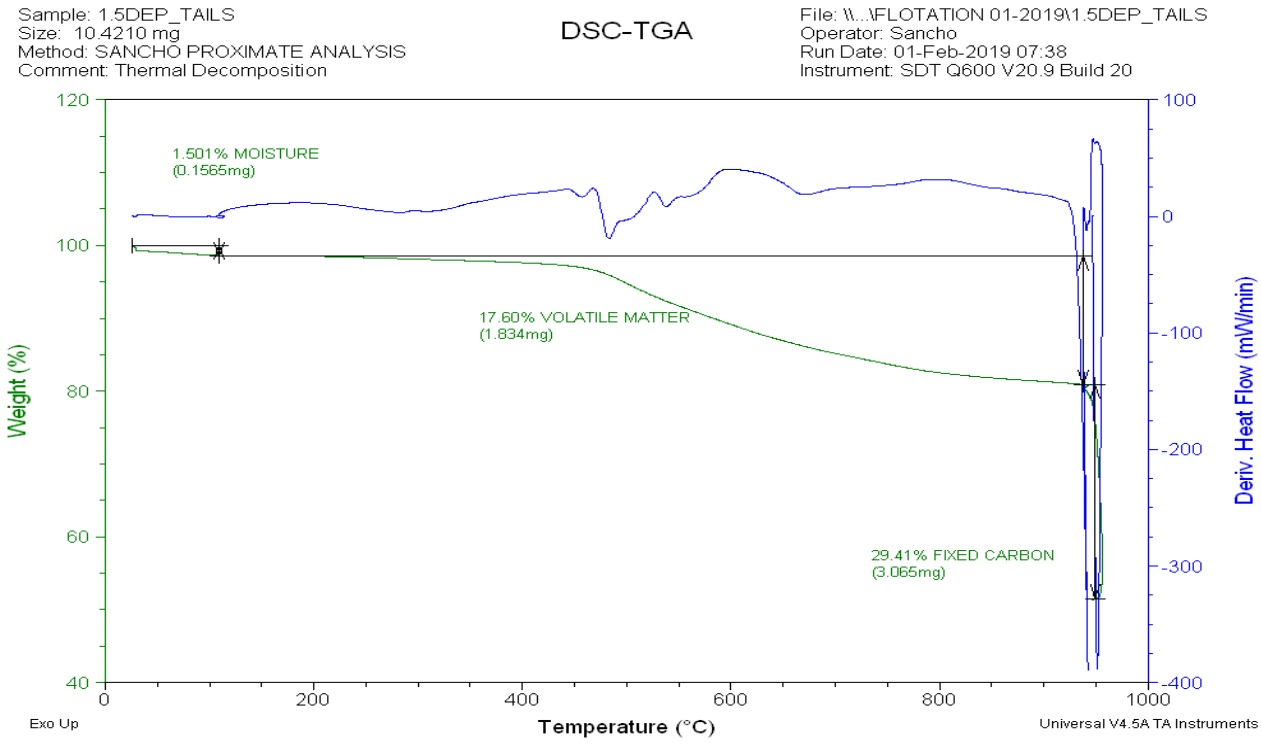


Figure A2- 31 DSC-TGA Tails Plot: 5gSENDEP/kg Coal; 14gDIESEL/kg Coal

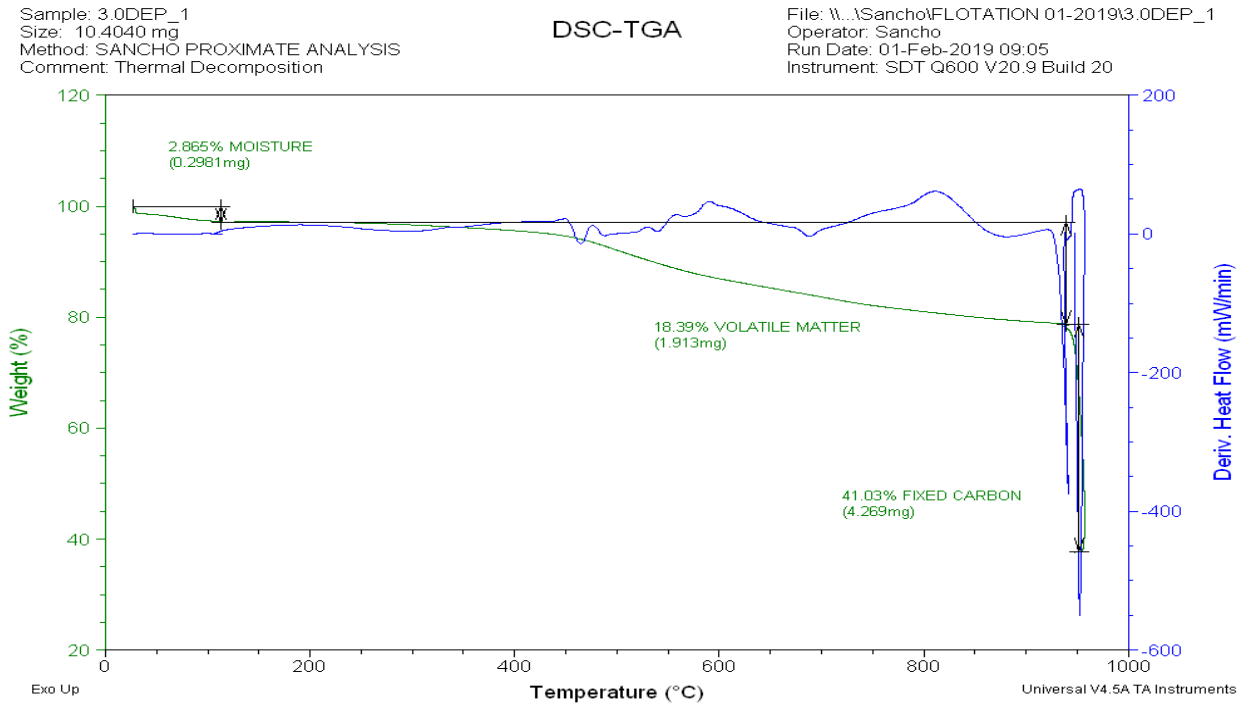


Figure A2- 32 DSC-TGA Plot at T=1: 10gSENDEP/kg Coal; 14gDIESEL/kg Coal

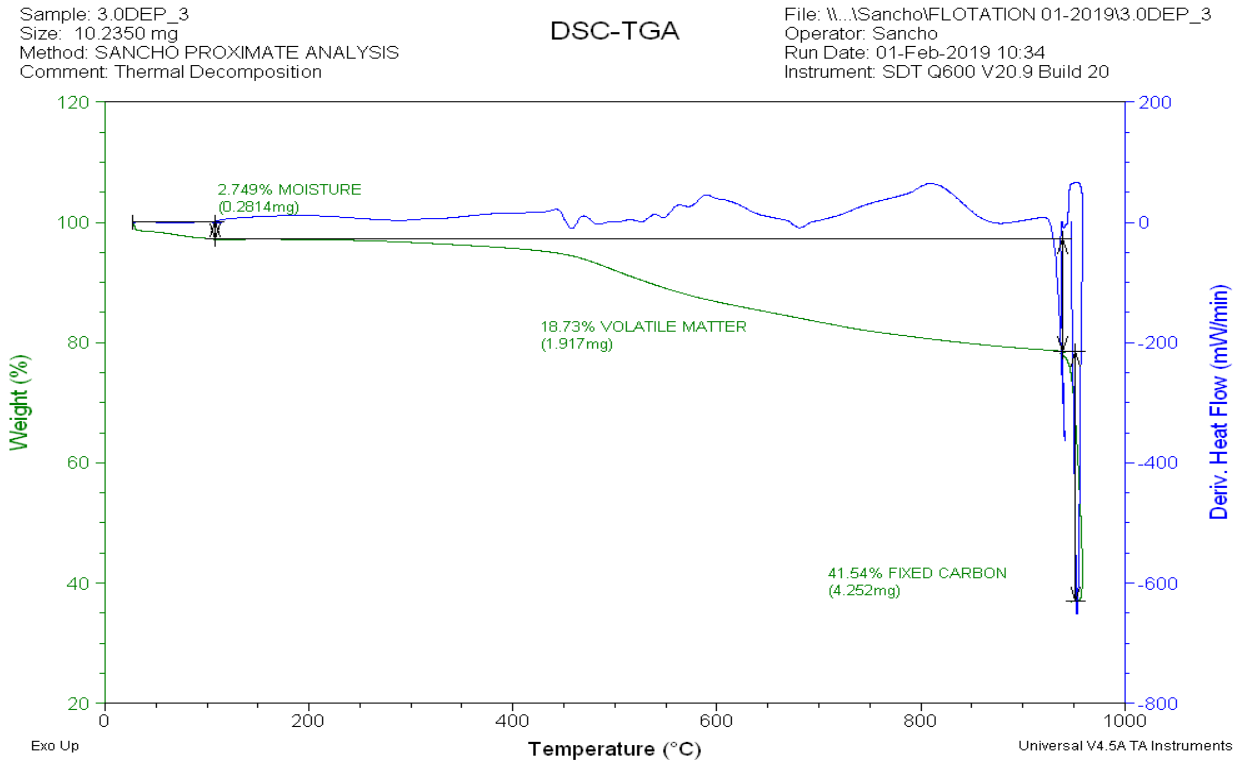


Figure A2- 33 DSC-TGA Plot at T=3: 10gSENDEP/kg Coal; 14gDIESEL/kg Coal

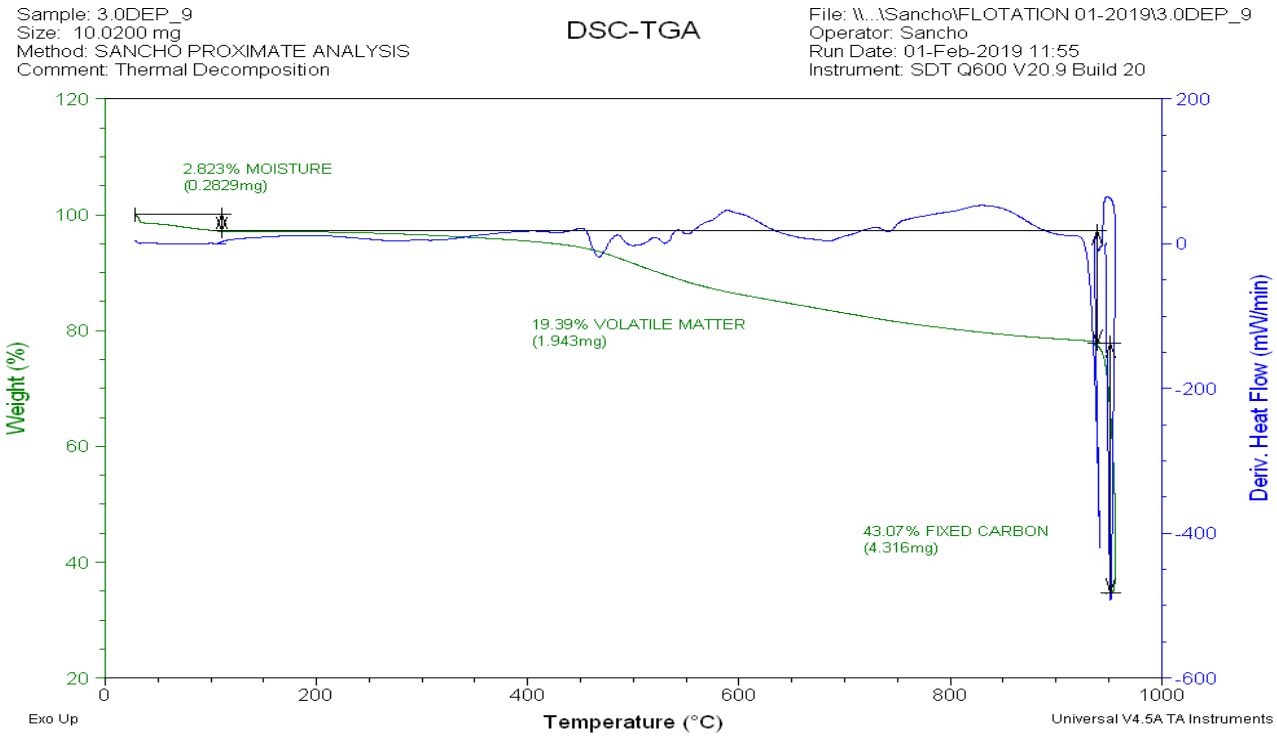


Figure A2- 34 DSC-TGA Plot at T=9: 10gSENDEP/kg Coal; 14gDIESEL/kg Coal

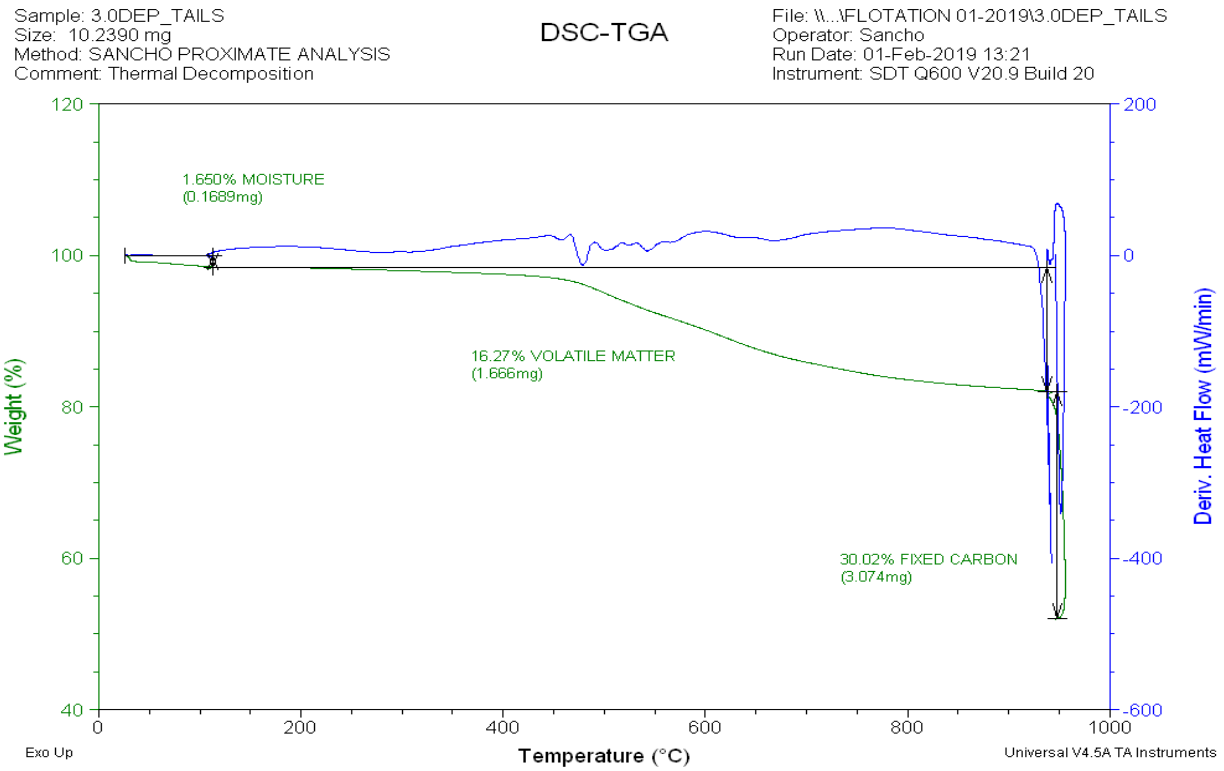


Figure A2- 35 DSC-TGA Tails Plot: 10gSENDEP/kg Coal; 14gDIESEL/kg Coal