

Coal washability determination using RhoVol technology

SB Bothoko

 [orcid.org/ 0000-0002-5638-5224](https://orcid.org/0000-0002-5638-5224)

Dissertation accepted in fulfilment of the requirements for the degree *Master of Science in Engineering Sciences with Chemical Engineering* at the North-West University

Supervisor: Prof QP Campbell

Co-supervisor: Prof M le Roux

Graduation: Jul - Aug 2023

Student number: 25264354

ABSTRACT

Density is one of the key properties of coal and provides information about its physical and chemical composition. Washability analyses (or densimetric information) are used to specify the yield and quality of clean coal produced in coal processing plants. Traditional coal washability analysis is determined by conducting a sink-float test. However, this method involves environmentally hazardous chemicals, is expensive, time-consuming, and is highly dependent on the expertise and experience of the operator. Therefore, the research to develop an alternative technique for determining coal washability would be highly applicable, especially in a coal-rich country like South Africa.

In this study, an alternative method based on a 3D imaging system, the RhoVol Density Measurement System (De Beers Group Ignite™), where the densities of individual particles are determined using mass and volume measurements, was introduced. This technology can instantly, repeatably, and efficiently produce the density distribution of a batch of particles. Additional data, like shape and particle size, are also calculated and recorded per particle. The RhoVol is equipped with a sorting functionality that enables the operator to sort the sample into bins according to any captured particle parameters such as density, compactness, flatness or elongation.

This study aimed to determine whether RhoVol can be used as an alternative method for coal washability determination. Four different coal samples were taken from the three coal fields (Highveld, Witbank, and Molteno-Indwe) in South Africa. The coal samples (-8 mm +3 mm) were analysed using the traditional zinc chloride float-sink method and RhoVol. Helium pycnometry (true density) was used for the calibration of the RhoVol results.

It was found that the correlation coefficients (R^2) between pycnometry data and the RhoVol were higher than 0.97 after the application of a correction factor. Based on this correlation, a linear correction was applied to the RhoVol output data to estimate the true density distribution.

The washability curves generated using RhoVol were in agreement with the washability information produced by the float–sink test. Our findings showed that RhoVol can be used in the determination of coal washability data in a non-destructive and non-toxic way.

Keywords: Coal, RhoVol, Density, 3D imaging system, Sink-float, Washability, Pycnometry, Correction procedure.

PUBLICATIONS

Botlhoko, S.; Campbell, Q. P.; le Roux, M.; Nakhaei, F., washability analysis of coal using RhoVol: A Novel 3D image-based Method. *mineral processing and extractive metallurgy review* **2022**, 1-13.

Nakhaei, F., Campbell, Q.P., le Roux, M., and Botlhoko, S. 2022 Estimation of coal density using a 3D imaging system: RhoVol. *Journal of the Southern African Institute of Mining and Metallurgy*, vol. 122, no. 8, pp. 443–450

ACKNOWLEDGEMENTS

My heartfelt thanks go to North-West University for making this research feasible. I would like to thank specific individuals in particular, without whom this work could not have been accomplished; your influence and contribution are greatly appreciated.

- First and foremost, I want to thank my supervisor, Professor Quentin Campbell, for his wisdom, guidance, and support. Thank you for your words of encouragement during my studies. Your effort was made possible by using examples, expertise, and mentorship.
- My mentor, Dr Nakhaei, who was always available to assist, without whom the project could not have succeeded. Your contribution is greatly appreciated.
- I would also like to thank my co-supervisor, Professor M. Le Roux, for his unique insight and assistance throughout this course.
- Mr Mgano, the laboratory analyst, and the workshop workers at North-West University for the installation of the equipment. Thank you for your contribution of time and effort.
- De Beers Technologies SA is now known as De Beers Group Ignite for their financial support, which enabled this study to take place and provided access to the RhoVol analyser, which benefited the coal sample analysis.
- To my goddaughter, Kabo Ntwe, this study is only a little part of what I can do to preserve your memory. It is a part of thinking ahead and stretching the boundaries of life as we know it to discover and grow. May your soul find eternal peace.
- To my family and friend thank you for your prayer and for making the journey easier.
- To my one and only Chulo- thank you for making me believe in my ability.
- Finally, with the most profound thankfulness, I express my heartfelt appreciation to our Almighty God Jesus Christ for the magnificent blessings and strength to do the work.

Table of Contents

Abstract	i
Publications	ii
Acknowledgements	iii
Table of content	iv
List of tables.....	vi
List of figures	vii
List of abbreviations	viii
CHAPTER ONE: INTRODUCTION	1
Background and motivation	1
Problem statement	3
Scope and dissertation outline.....	4
CHAPTER TWO: LITERATURE REVIEW	6
Background	6
Coal preparation.....	7
Densities separation methods.....	8
Density.....	8
Density measurement methods	11
CHAPTER THREE: METHODOLOGY	16
General Introduction	16
Safety	16
Material and sample preparation	16
Characterisation	18
Experimental procedures	20
Gas pycnometry	20
Float-Sink Method	22
RhoVol	22

CHAPTER FOUR: RESULTS AND DISCUSSION	25
General Introduction	25
RhoVol and Pycnometry	25
Density correction procedure	30
RhoVol and float-sink test	32
Ash-density relationship	35
CHAPTER FIVE: CONCLUSION	37
RECOMMENDATIONS	38
REFERENCES	39
APPENDIX A	41
APPENDIX B	44

LIST OF TABLES

Table 1: Size distribution and average contents of each coal sample's moisture, volatile and ash.	20
Table 2: The RhoVol test condition for the four types of coals.	23
Table 3: Summary of shape data of four coals	25
Table 4: The comparison of the RhoVol (DR) densities (bulk) to the pycnometer (DP) densities for each bin's contents	30

LIST OF FIGURES

Figure 1: Scope of the project 5

Figure 2. 1: A schematic flowchart depicting the operation of a standard coal cleaning process(Luttrell and Honaker, 2012).....7

Figure 2. 2: Bulk volume illustration..... 9

Figure 2. 3: Photograph of a sample indicating cleats, particularly in the vitrain layers of bright coal; note the blocky nature caused by these smaller cleats. (Merkus, 2009) 11

Figure 2. 4: The 3D representation of particles from seven different cameras 12

Figure 2. 5: A free-body diagram depicting the many forces acting on a particle moving in a liquid 13

Figure 2. 6: Schematic of the μ CT process used for measurement and data acquisition..... 14

Figure 3. 1: (a) Jaw crusher, (b) Screening, (c) sampling; cone and quartered..... 18

Figure 3. 2: Photograph of helium pycnometry process..... 21

Figure 3. 3: Float-sink tests 22

Figure 3. 4: The RhoVol system's multi-stage internal operations 23

Figure 4. 1: Volume calculation scatter plots for coal A-D samples.....26

Figure 4. 2: (a- d) Particle size distributions for four coal samples by sieving and RhoVol methods 28

Figure 4. 3:Pycnometry results with RhoVol data for each bin for Coal A-D 29

Figure 4. 4: Correlation between average RhoVol densities (bulk) and pycnometer densities of the contents of each bin for coals A-D..... 32

Figure 4. 5: Show graph of corrected RhoVol Density distribution and pycnometer distribution for each bin 32

Figure 4. 6: The comparison of the cumulative density distribution obtained by float-sink and RhoVol experiments; Coal A-D 33

Figure 4. 7:Cumulative density distribution derived from sink-float and corrected RhoVol based on static correction factor tests for Coal A-D..... 34

Figure 4. 8: Sink-float and corrected RhoVol (based on linear regression models) cumulative density distributions Coal A-D..... 35

Figure 4. 9: Correlation between Rhovol's bins' average corrected density and ash content. Each point shows the average particle densities in each bin..... 36

LIST OF ABBREVIATIONS

Abbreviation	Description
DMS	dense medium separation
μ CT	Micro X-ray computed technology
VSFD	Vision size frequency distribution
CV	Calorific value
NGMI	near-gravity material index
VOI	volume of interests
DE-XRT	Dual-energy X-ray transmission
AFDMB	The air-dense medium fluidised bed

CHAPTER 1

Chapter one provides background information to ensure a comprehensive understanding of the topic, as well as the investigation's rationale, and numerous objectives. This chapter is divided into the following sections:

- Section 1: Background and Motivation - It briefly describes the investigation, its objective, and a declaration of the study's most critical issues.
- Section 2: Gives a brief description of the problem statement and a general hypothesis for the study. It concerns coal washability and density separation.
- Section 3: Aim of the study is provided in this section, and the objectives to achieve the aim are defined.
- Section 4: Provides the reader with a detailed outline of the dissertation, directing the flow of the dissertation and outlining what is expected in each chapter.

1.1 Background and Motivation

The characteristics of coals from different coalfields differ in rank, type, grade, and, thus, morphology. Most coal processing plants use density separation, like gravity or dense medium separation, to upgrade or beneficiate the coal to some product specification. While it may be easier to wash some coals, others may pose severe challenges related to the density distribution of the particles in the coal stream, also referred to as washability. The washability is related to the rank, grade and morphology, and to what extent crushing had liberated the different density constituents.

Due to the irregular, porous, and heterogeneous nature of coal, the definition of the term “density” should be carefully considered as far as beneficiation is concerned. There are various definitions of densities like envelope density (based on the volume defined by an outer shell or envelope around the particle), true density (which excludes the volume of at least some of the pores in the structure), bulk density (which takes the volume between a collection of particles into account), and others. These density measurements depend on the definitions of the volumes being considered and whether or not pores are included. The bulk coal density may increase with coalification, meaning that high-rank coal is denser than low-rank coal. (Alamooti and Malekabadi, 2018)

In this research, dense medium separation would determine the density distribution of a population of representative particles and identify the cut-point density needed to achieve the

desired product grade. The standard method, such as float and sink in some media, has been used to achieve density separation, but it has received many criticisms because it is time-consuming and costly. In daily production, operators often need rapid information about washability to optimise the production line, but typical results will take few days to be returned, so it is impossible to optimise the plant operation accurately. Furthermore, the sink and float test is a manual procedure highly dependent on the operator's competence and experience, and process repeatability needs to be revised. (High, 2018; Dai *et al.*, 2020)

The washability curves are also used to determine the degree of difficulty of coal gravity separation. One can estimate the clean coal's theoretical yield and ash yield obtained in an ideal gravity separator at various density cut points. Several factors influence coal washability; this includes mineral content, particle size, shape, and quantity of near-gravity particles. The washability curves show the relationship between calorific values (CV), ash yield, and density. As a result, these curves are used to determine plant resource and reserve estimates, design, daily plant control, process optimisation, and assessing possible recovery and coal quality, among others.

Unfortunately, due to the costly nature and toxicity of suitable media utilised (like heavy organic liquids or salt solutions) and the time-consuming process of washing and drying of samples, there is a motivation for developing alternative methods to determine washability curves.

1.1.1 RhoVol

Recently, a new technology (RhoVol Density Measurement System) has been developed to estimate particle densities on an individual basis, and it has the potential to replace sink and float tests for predicting coal washability. This technology was initially designed for diamond application but was later re-purposed for other uses, such as iron ore or coal. RhoVol is a 3D image system invented by the De Beers group of Technologies SA (now known as De Beers Group Ignite™). This technology can repeatedly and efficiently produce the density distribution of a batch of particles and additional data, like shape and particle size distributions. These properties are calculated and recorded per particle and can be collated to represent the sample population. (High, 2018) In addition, the RhoVol machine enables sorting of samples into different bins based on any of the particle's measured parameters, facilitating additional functionality like sample preparation and screening. Three models of RhoVol exist to date. Namely, the ultra-fine RhoVol machine that treats -3 +1mm particles, the fine RhoVol machine that handles -8 +3mm particles and finally, the course RhoVol machine that treats -25 +8mm particles. This technology is suitable for all commodities to be beneficiated using dense medium separation (DMS).

1.2 Problem statement

A previous study on determining coal washabilities using RhoVol showed the possibility of supplementing or replacing traditional float and sink analysis. It was determined that most difficulties arise from the appropriate calibration of data due to the porous nature of coal. Other problems during the study were understanding the density to be measured to predict dense medium separation and which type of density to use during plant production. Such densities include; skeletal, actual, envelope, and submersion, which are all related to specific measurement techniques. The density determination is based on the following aspects:

- Two coals may have the same ash values but differ in porosity; thus, the ash-density relationship for these two coals will differ.
- The particular separation density required to produce clean coal with the desired ash content will differ from one coal to another.
- The ash distributions of two coals with the same density content may differ depending on the mineral constituents.

Although studies on the washability of fine and coarse coal have been conducted using optical imaging, the RhoVol approach is less expensive. It has fewer safety risks than commonly used x-ray imaging techniques.(Nguyen, A. V. Nguyen, *et al.*, 2018; Nie *et al.*, 2018) Therefore, the RhoVol technology has the potential to generate the density distribution of coal samples. The obtained information may be valuable for laboratory and industrial applications.

1.2 Aim and objectives

This study aims to determine if RhoVol can be used to sort coal particles based on density and, to estimate the coal washability, as compared to the conventional sink and float test. The focus is to understand the fundamentals of the RhoVol process, particularly the errors associated with the density measurements and known approaches to minimise errors. The validity and reliability of a camera-based 3D imaging system for coal density measurement are described and focused on improving the accuracy of the RhoVol. The objectives of the research project are as follows:

- To determine the densities of the coal samples using RhoVol and Helium pycnometry
- To evaluate the washability characteristics of the same coal particles through a float-sink test.

- Determine the density profile using RhoVol and compare it to float and sink analysis after calibration.

1.3 Scope and outline of the dissertation

The contents of the project's execution, including methodologies, findings, conclusions, recommendations, and the coal scientific and technical knowledge base, are detailed in chapter figure 1.

The dissertation is arranged into five chapters; chapter one consists of the background, problem statement, and aims and objectives. The second chapter focuses on assessing the literature. First, the coal washability methods are briefly discussed, along with an overview of the main beneficiation procedures used in coal processing. Following that is the complete study of the appropriate coal preparation theory and the pertinent theory underlying the RhoVol machine and its influence on coal beneficiation. Next, density definitions and coal characteristics are discussed, followed by the importance of density in coal processing.

Chapter 3 discusses the methodological approach and equipment used in this study. Chapter 4 comprises the analyses, presentation and interpretation of the findings. Finally, chapter 5 includes final observations and possible development suggestions for the study project.

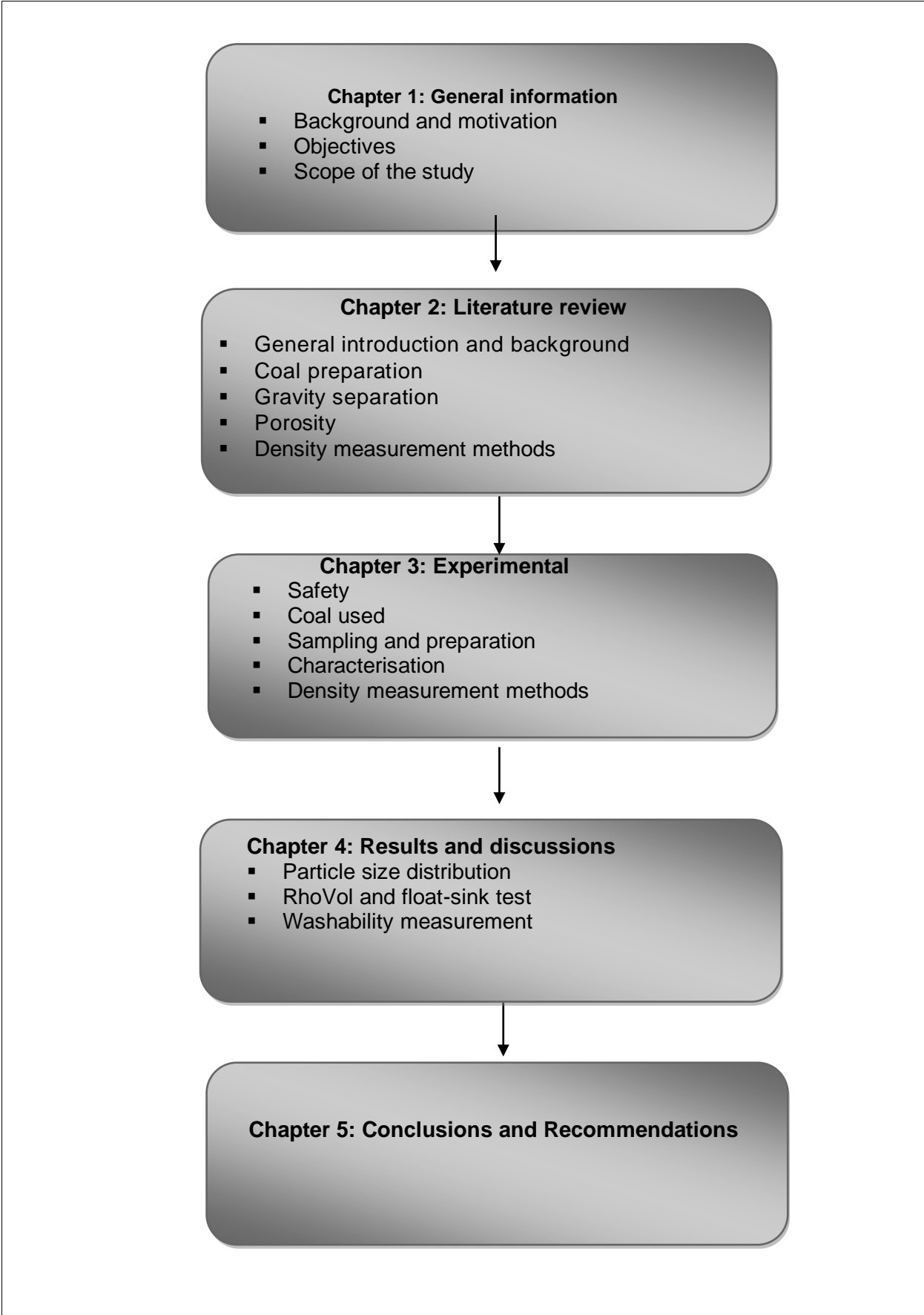


Figure 1: Scope of the project

CHAPTER TWO: LITERATURE REVIEW

Chapter 2 consists of the theory regarding coal preparation, types of densities and methods to determine density. This chapter is divided as follows:

- Section 2.1: Provides a brief description of the study background, followed by a declaration of the main topics for the study.
- Section 2.2: Describes the coal preparation and the processing phases involved.
- Section 2.3: Details the various density separation methods and summaries the current and advancing beneficiation techniques.
- Section 2.4: Gives an in-depth discussion of the definitions of density and their importance in coal beneficiation.
- Section 2.5: Discusses porosity and its effect on density determination.
- Section 2.6: Concludes with the various methods used to measure density and provides a summary of their operation.

2.1 Background

Dense medium separation is widely used in the coal, iron ore, and diamond industries to upgrade run of mine ores into valuable commodities. Generally, DMS is used in both dry and wet beneficiation, and several research studies were conducted on both methods. (Diedericks, Le Roux and Campbell, 2021) These beneficiation techniques separate particles based on the density difference between the high-value materials and the impurities. This can be achieved by developing a specific medium, such as zinc chloride, cesium chloride, and ferrosilicon, among others.

The density difference between different coal particles serves as the fundamental separation principle in the conventional float-and-sink test; thus, the coal particles are grouped into a range of specific gravities after each round of the float-sink test. This process may take time for operations that need to quickly turnover multiple washability results on a given day. Since RhoVol can analyse mass, volume and size of the individual particles, we can predict the density and size of a specific particle and calculate the washability of a specific coal sample.

2.2 Coal preparation

Reducing incombustible inorganic material such as ash and sulphur content during the preparation process helps improve coal quality. As a result, raw coal can be transformed into various feedstock. (Gui *et al.*, 2015)

South Africa uses two steps in coal preparation, namely screening and washing. Coal is first screened to eliminate foreign items before being crushed to reduce the top size. Another screening step separates the coal into desired ranges for direct sale or additional processing through beneficiation.

During the screening phase, vibration screens separate particle sizes into coarser and finer sizes. The coarser particles are treated in static dense medium vessels, while the smaller particles are cleaned in dense medium cyclones and spirals. Ultra-fines cannot be beneficiated using conventional gravity, and density separation methods can be ineffective and eliminated. Fine coal, on the other hand, can be treated in a flotation circuit and, finally, dewatered using a disc vacuum, dryers and filters as shown in figure 2.1. The separation efficiency will vary due to the feed's different coal qualities and particle sizes. (Luttrell and Honaker, 2012; Gui *et al.*, 2015)

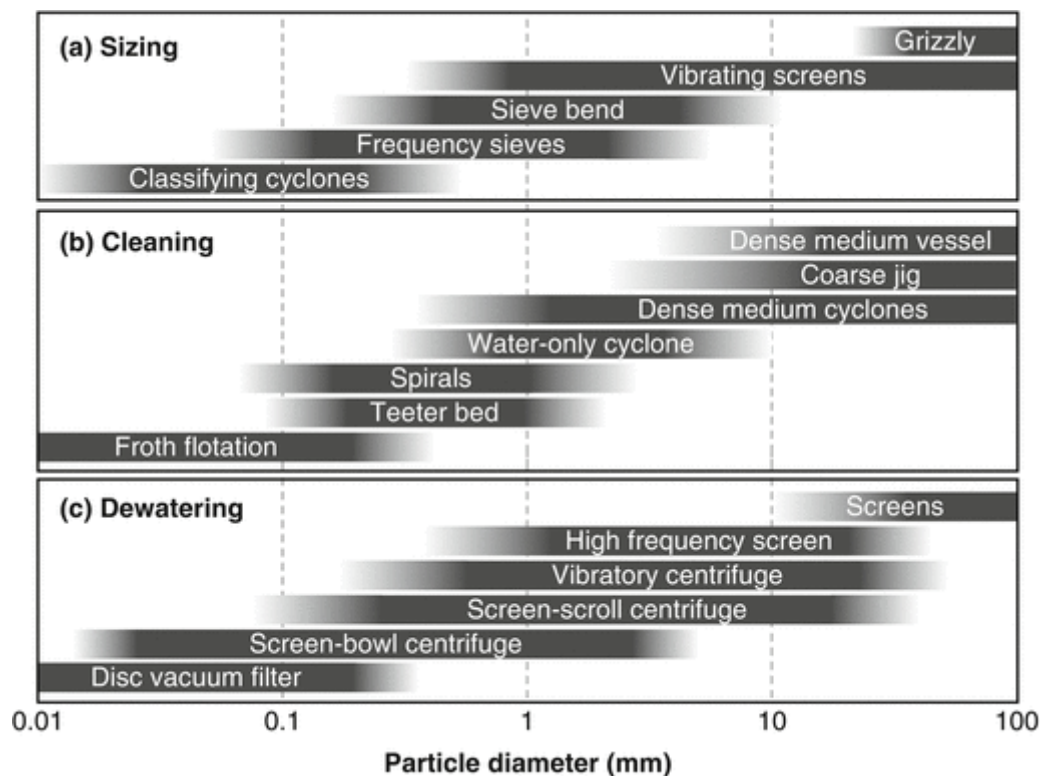


Figure 2. 1: A schematic flowchart depicting the operation of a standard coal cleaning process (Luttrell and Honaker, 2012)

2.3 Density separation methods

Coal washing is accomplished in two ways: wet and dry. The dry method comprises the following: magnetic separation, hand picking, microwave separation, air jigging, electrostatic separators AFDMBs (Air dense medium fluidised bed), and DAFB (dense medium fluidised bed).

Wet separation techniques are much more common and effective than dry beneficiation procedures. Furthermore, wet beneficiation techniques have larger capacities, use less energy, and are more appropriate for the bulk of today's ROM coals. The wet processes comprise of spiral, froth flotation, and DMS; these methods depend on the desired results and the feed particle size. (Pudasainee, Kurian, and Gupta, 2020)

2.4 Density

Density is a crucial factor in mineral separation processes; thus, understanding the density of a material subjected to a metallurgical process is essential. There are different definitions of density based on the method of determination.

a. Bulk density

Bulk density is the mass of a population of material's particles divided by the volume occupied. It is influenced by physical characteristics such as shape, particle size distribution, and the dimensions of the measuring container. If the particles were sieved to be nearly the same size, particle size would not affect bulk density. (Blake and Hartge, 2018)

In the real world, however, a particulate substance always has a range of particle sizes, which heavily influences bulk density. If smaller particles sift downward in a container, filling in the gaps between the larger particles, then bulk density will be higher if there are many smaller particles than larger particles. Depending on the material's storage and particle size, bulk density may range from 700 to 1100 kg/m³. Bulk density is also strongly influenced by shape. If particles are highly angular, the edge tends to fit into the gaps between other particles. However, the gaps between particles tend to remain unfilled when particles are well-rounded shown in figure 2.2. (Merkus, 2009; Mallol *et al.*, 2010)

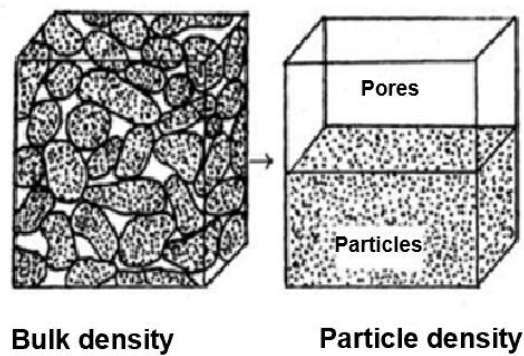


Figure 2. 2: Bulk volume illustration(Blake and Hartge, 2018)

The above factors mean that bulk density is not a very useful measurement to determine a material's properties.

b. True/Absolute density

True or absolute density is defined as the mass of a particle divided by its true volume, with no open and closed pores included, the most common method for measuring the true density is helium pycnometry. As the smallest atom, helium has the greatest chance of penetrating the most significant number of pores. Helium pycnometer uses a sample of the known mass and seals into the compartment at a maintained constant temperature. The pressure in the sample compartment equilibrates when helium is added to the system. (Inglezakis and Pouloupoulos, 2006)

c. Particle density

The particle density is the mass of an object divided by its volume, including cracks and pores. The term "particle" generally refers to a small piece of an object with a specific size, shape, and number of pores. This volume is the same as the envelope volume calculated by RhoVol. It is a highly effective method for characterizing various solid materials across different industries, particularly porous objects of varying sizes and shapes.

d. Submersion density

Submersion density is measured by float and sink (buoyancy). It is suggested that this density is probably between absolute/true and particle density, depending on the crack and pore sizes. This fact forms the basic assumption of the study.

2.5 Porosity

Porosity is the pore space measurement present within a solid and is represented by the volume percentage of void. Since coal is a porous substance, porosity may substantially impact its behaviour during preparation, mining and density determination. Research found that the existence of pores and their size distribution, may be intrinsically related to product quality. To ensure the accuracy of results, open pores are filled with a known liquid. This filling should be considered when figuring out the density of particles. (Yao *et al.*, 2011)

The coal pore system consists of three categories, namely;(Calvo *et al.*, 2005)

- Micropores with a size range of 0.4-1.2 nm
- Mesopores with a range from 2-50 nm
- Macropores with a size >50 nm

Since porosity provides capacity for moisture or gas storage, the density of the material is affected by the nature of the fluid or gas contained within the pores. Pores and their size distributions may be assessed using gas pycnometry with methane, krypton, helium, nitrogen or mercury penetration, small-angle neutron scattering (SANS) and small-angle x-ray scattering (SAXS) techniques. Mercury is utilised to penetrate macro and mesopores. In contrast, helium is employed to measure micropores. The lower rank of high-volatile bituminous and sub-bituminous coals will have high overall of 92–99% porosity and a large fraction of intermediate pore sizes. With high-rank bituminous coals containing no intermediate-sized pores will have a significantly lower microporosity.(Cakmak *et al.*, 2023)

Typically, bituminous coal porosity is also related to cleat within coal seams. The fracture sets and partings along bedding planes give the coal a blocky appearance (figure 2.3). Cleats, which arise from active coalification processes and fluid pressure applied during tectonic events are the most prevalent natural porosity and permeability pathways in coal seams and extensional fractures, particularly in the vitrain layers.(Merkus, 2009; Roux, 2021)

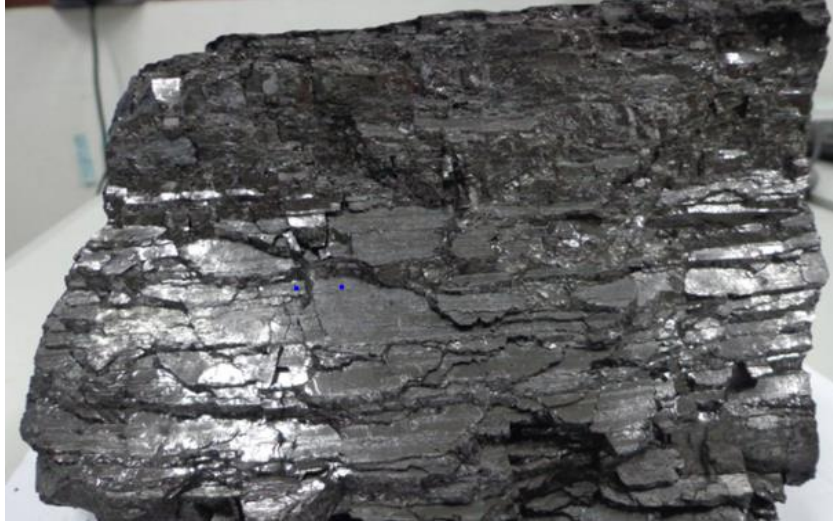


Figure 2. 3: Photograph of a sample indicating cleats, particularly in the vitrain layers of bright coal; note the blocky nature caused by these smaller cleats. (Merkus, 2009)

2.6 Density measurement methods

2.6.1 RhoVol Density Analyser

De Beers Technologies SA, now known as De Beers Group Ignite™, developed an automated densimetric measurement device known as RhoVol. It was introduced commercially a decade ago with two machines available for fine (-8+3 mm) and coarse (-8+25 mm) particles. More recently, the RhoVol ultra-fine has been developed for particles -3mm down to 1mm. The working principle of RhoVol is to measure individual particles' mass, followed by a 3D reconstruction of their volume using the processing of images from multiple cameras. Three-dimensional reconstruction is created by processing a collection of projections at various angles in a full rotation. The projected image is a 3D matrix of the vision size-frequency distribution (VSFD). As demonstrated in Figure 2.4, the reconstruction is directly generated into a 3D image. More recently, the RhoVol ultra-fine has been developed for particles -3 mm down to 1 mm.(Fofana and Steyn, 2019)

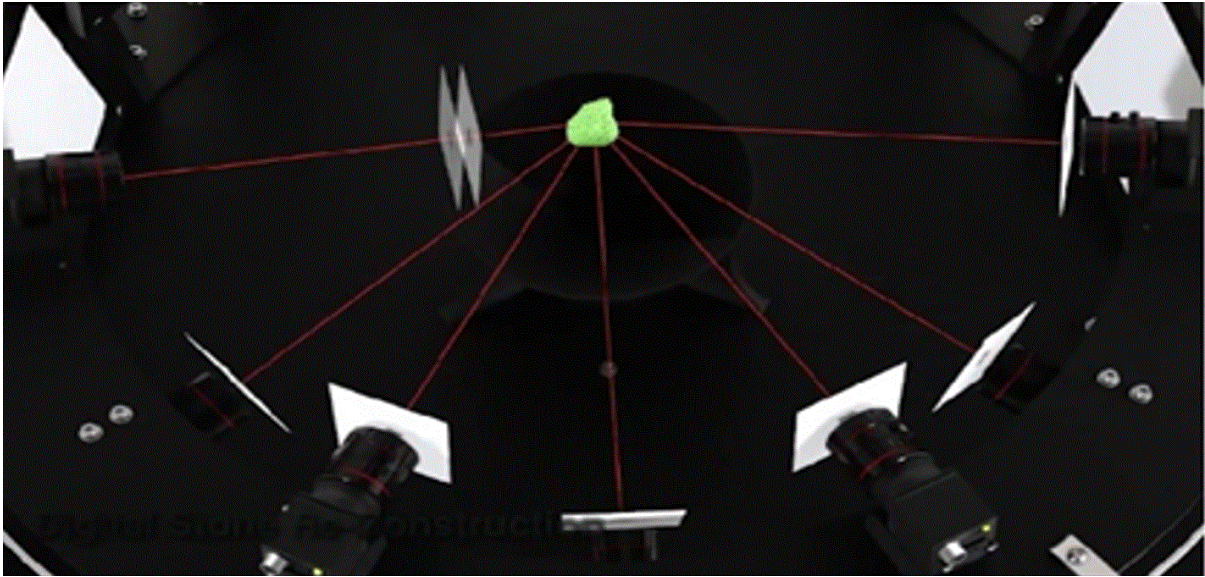


Figure 2. 4: The 3D representation of particles from seven different cameras (Botlhoko *et al.*, 2022a)

a. Correction factor

Error correction methods for coal density obtained by RhoVol are proposed. Accuracy and repeatability of measured volume/density depend on the physical features and the orientation of the particle at the moment when the images are captured with Resolutions of 720 x 480 pixels. From 3D images, the effect of porosity and cracks on the volume of coal samples cannot be determined. In other words, the weakness of this method is that it fails to capture hidden concavities in the sample leading to an overestimation of the coal particle volume. Furthermore, it is impossible to consider the influence of porosity and cracks on the volume of coal samples derived from 2D silhouette images. Therefore, the density calculation based on RhoVol 3D models requires a correction factor (CF). There are two types of CF: static and objective function.

These correction factors will be used to generate a range of accuracies. The correction factors for the static and objective functions are calculated by comparing the RhoVol density to the pycnometer density for the particles in each bin. The results section will indicate that the objective function factor is determined using a linear regression procedure. The static CF will be the average of all correction factors derived as ratios of RhoVol density to pycnometry density in each density bin and for all coal samples. The first approach in the subsequent study will be to apply a constant correction factor to all coal samples since RhoVol calculates the volume of coal using apparent measurements rather than actual measurements; hence, its accuracy depends on how well the particle volume resembles the true density. (Mangera, Morrison and Voigt, 2017; Grant High, 2018; Fofana and Steyn, 2019)

2.6.2 Float-sink method

Coal washability data is essential for analysing the performance of coal preparation plants. The washability curves are obtained using sink and float tests, and washability can be presented in a graphical format known as a densimetric curve. Coal washability is used to predict optimal plant performance and well as to audit operating plant performance regularly. It is usually presented in conjunction with ash analysis. (Jargalmaa *et al.*, 2015; Adinugraha *et al.*, 2018; Sahu, Chaurasia and Suresh, 2019)

A dense medium fluid is introduced into a separation vessel with material, and the relative velocity of particles in the medium fluctuates in reaction to gravity. The settling theory explains this phenomenon well, i.e., an object submerged in a gas or liquid experiences pressure from all sides while the forces on top and bottom remain constant. Pressure forces from the sides cancel each other while the bottom pressure pushing up is constantly stronger than the top pressure pushing down under the force of gravity. Thus, the object will experience constant acceleration, as illustrated in figure 2.5. It is not easy to precisely characterise the qualities of coal while considering all properties. This is due to particle movement and how the separation process takes place. The bigger particles are more influenced than smaller ones, and the effectiveness of gravity separation rises proportionately as particle size increases. (Majumder and Barnwal, 2004)

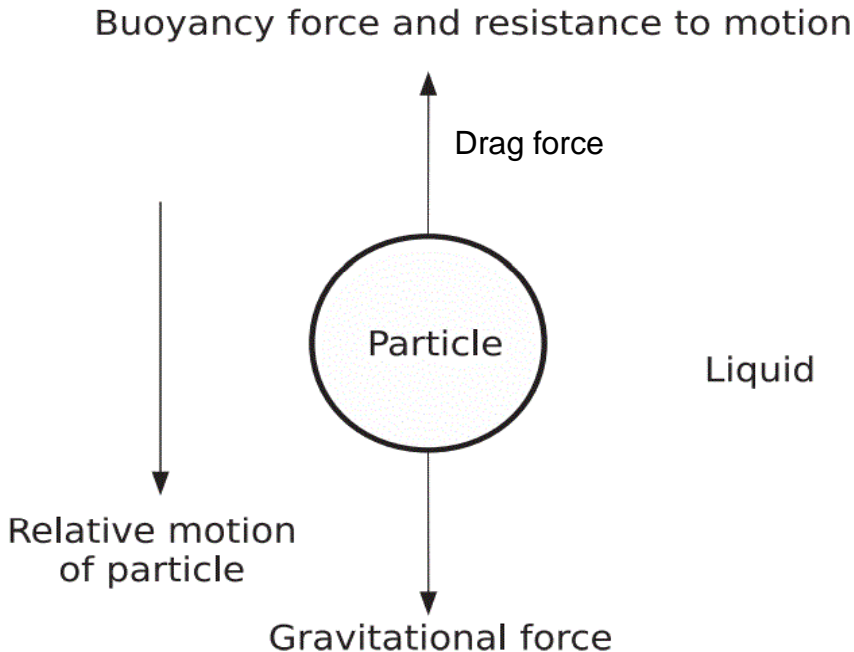


Figure 2. 5: A free-body diagram depicting the many forces acting on a particle moving in a liquid

2.6.3 High-Resolution X-ray microcomputed tomography

The material is exposed to the incoming X-ray beam rotated 180° during acquisition to create a series of projections (typically between 600 - 3600). After reconstruction, these projections build two-dimensional slices of the measured volume using a Scanning Electron Microscope (SEM). The pixels in the 2D slices maintain spatial information about the original volume elements (voxels), allowing for stacking and rendering of the slices to see the sample's 3D volume. The SEM method is also used in mineral differentiation to calibrate the μ CT with pure minerals of known density to connect the attenuation coefficient and the actual apertures of fractures within the coal. (Nguyen, A. V. Nguyen, *et al.*, 2018)

Computed tomography (CT) is one technique that determines density by obtaining three-dimensional images of a particle, including its internal structure, with no destructive sectioning. The primary advantage of CT is its capacity to monitor the 3D internal structure of the ore at resolutions as low as a few microns and remove stereological inaccuracy seen in 2D analysis. The technique consists of μ CT (X-ray computed tomography) that records X-ray attenuation object differences and defines the X-ray proportion that interacts with the object as represented by blue intensities in the reconstructed slice images shown in figure 2.6.

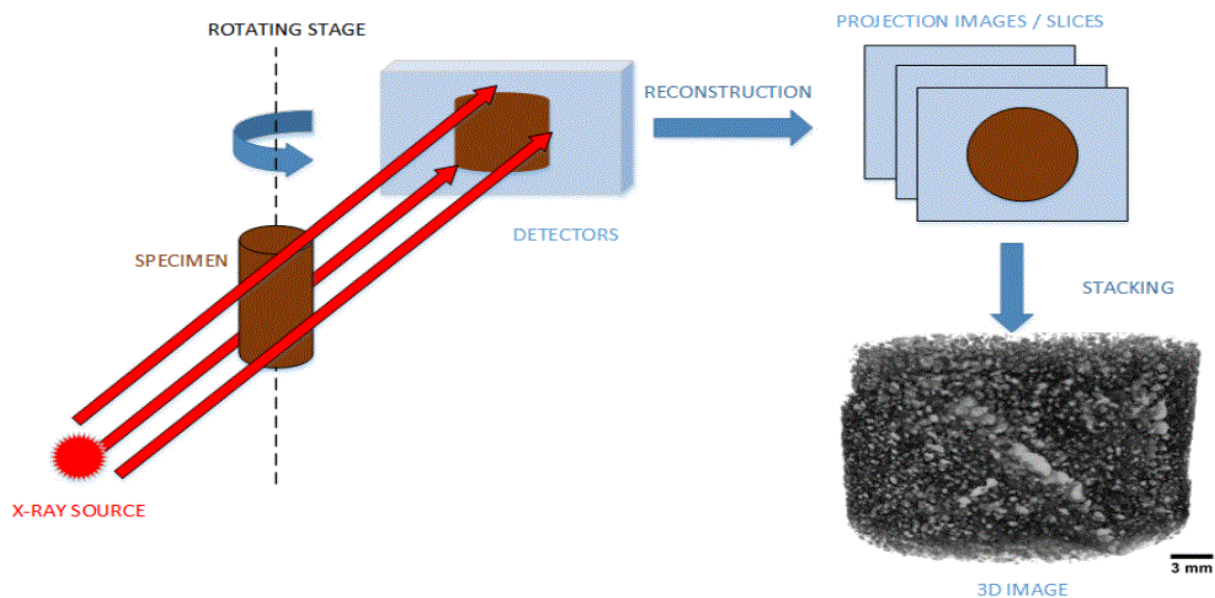


Figure 2. 6: Schematic of the μ CT process used for measurement and data acquisition.

Advanced filtering algorithms are applied to segment the micro-CT image into four distinct phases: resolved fractures, sub-resolution pores and fractures, macerals, and minerals. The application will determine the distribution of resolved aperture size as well as the porosity in coal

samples. However, the disadvantage of this method is the use of X-rays, which are expensive, lengthy testing processes and limited observation accuracy. It is used for one or a few small particles at a time, which is not valid for large populations. (Nguyen et al., 2018)

CHAPTER 3: MATERIALS, EQUIPMENT AND EXPERIMENTAL

Chapter 3 provides a description of the procedure and method of experimentation. This chapter consists of the following sections:

- Section 3.1: Provides an overview of the study and details the necessary materials for the experiments.
- Section 3.2: Describe, the risks in this project, and the safety regulations followed.
- Section 3.3: Discuss, the materials, sample preparation, and coal used during the study.
- Section 3.4: Details the characterisation.
- Section 3.5: Consists of the experimental procedure followed during the investigations, most related explicitly to RhoVol, pycnometry operation and float-sink test required thereafter.

3.1 Chapter overview

A proper experimental design is critical for obtaining accurate and relevant data throughout any research activity. This was done through experimental planning, apparatus construction, and techniques used for analysis, which will be discussed in this section.

3.2 Safety

The risks identified in this project were dust during sample preparation, the electrical connection of the equipment-RhoVol, and the chemical reagents used during the float-sink test. However, on all occasions, personal protective equipment was employed to avoid injury. Before the experiment, equipment training was provided to ensure that all protocol was understood and followed.

3.3 Materials and sample preparation

3.3.1 Coal used

Four coal samples were obtained from South African coalfields—two from the Highveld, one from Witbank and one from Indwe. The Highveld coalfield is located in Mpumalanga and south-eastern Gauteng. This coalfield is the second most operated coalfield in South Africa, with 972.49 Mt of ROM production between 1982 and 2000. This Highveld coalfield consists mainly of gently flat-lying surges with low-grade bituminous coal containing between 18 and 25 MJ/kg CV and 20–40 wt.% ash content(dry basis).

According to South African Coal statistics Marketing and Manual (SACSM), Witbank coal has been mined for blending coke coal and metallurgical applications particularly in the central Witbank area where the coal is of a higher grade. The Witbank coalfield in Mpumalanga Province holds one of the most considerable coal resources in South Africa (8509.3 Mt). (Hartnady, 2010).

The Indwe-Molteno coalfield is distributed throughout the western and southern parts of the Eastern Cape province. It comprises coal and shale with a thickness ranging from 4.3 meters. This coalfield has economic potential, but most of its coal is of low grade. Original mine works were abandoned in the early 1900s, but it was re-opened for commercial production on a small scale. Analyses indicate that it has a high ash content of between 26-27% when washed and 31-51% when unwashed, a low volatile matter (VM) of 7 to 12% dry basis, a calorific value (CV) of 23.9-25.9 MJ/kg dry basis, and high moisture content of 7-11%. This coal has a low density due to its high ash content.

3.3.2 Sampling and preparation

The preparation procedure consisted of crushing, sieving, sampling, and storage (figure 3.1). Crushing and sieving were necessary to acquire the desired coal particle size distribution (PSD). The jaw crusher model 66YROLL from Samuel Osborne (SA) LTD was used for crushing 4 kg of each coarse coal sample to less than 20 mm. The particles sample were then sieved to into the required particle size range of (-8 to +3mm). This was done using a sieve shaker and stack of industrial sieves, from which appropriate over and under sizes were removed and stored in containers. Since it is critical to acquire representative samples for the subsequent studies and experiments, coning and quartering (SANS 195:2006) were employed to obtain those sample.



Figure 3. 1: (a) Jaw crusher, (b) Screening, (c) sampling; cone and quartered

3.4 Characterisation

The properties of each prepared coal sample were established through a series of preliminary studies. Representative samples were generated by coning and quartering, and riffing methods.

3.4.1 Particle size distribution

The aperture sizes of the laboratory test sieves incorporated in the study were 8, 6.7, 5.6, 4.7, 4.0, 3.35, and 2.8 mm. Results are shown in table 1.

3.4.2 Proximate Analysis

Coal samples were subjected to proximate analyses to identify the moisture content, volatile matter, and ash-yield. A detailed procedure of the proximate analyses is in Appendix A.

a. Moisture content

The moisture content test involves evaporating the moisture throughout the heating process. The equipment was run at 105°C for 3 hours as specified by SANS 5924:2009. In this case, the moisture content is determined by calculating the difference between the starting and final weights. The percentage of moisture is then calculated as follows:

- W is the weight of coal before heating.
- W_1 denotes the weight of a cleaned empty crucible.
- W_2 is the total weight of the coal and crucible after heating

$$\% \text{Moisture content} = \frac{W_2 - W_1}{W} \times 100 \quad [1]$$

b. Volatile matter

Volatile matter (VM) is the percentage of mass loss when coal is heated under the atmosphere of pure nitrogen gas. In this experiment, 1 g of the sample was put into a covered crucible, heated to 900 °C for 7 minutes, and then weighed again. The difference is the weight percent lost as emissions during combustion,

$$\% \text{Volatile matter} = \frac{W_2 - W_1}{W} \times 100 \quad [2]$$

- W is the weight of coal before heating.
- W_1 is the weight of a cleaned empty crucible.
- W_2 is the weight of the coal and crucible after heating

c. Ash content

SANS 50:2011 specifies that the ash value is determined in an oven and operated at 900°C for six hours. After complete combustion, the ash value is calculated by dividing the residue remaining by the fixed carbon content. Moreover, the ash % is determined using the following equation: W is the weight of coal before heating; W_1 represents the weight of a cleaned empty crucible + lid, and W_3 is the weight of ash + crucible + lid.

$$\% \text{Ash} = \frac{W_3 - W_1}{W} \times 100 \quad [3]$$

3.4.3 Proximate analyses results

The Molteno-Idwe sample D showed the highest moisture and ash contents. In addition, the results indicate that Witbank is of better quality than the other samples because of low moisture and ash content, and may behave differently during separation. Table 1 only provides the summary results obtained for a samples proximate analysis, however, the details calculations are shown in Appendix A.

Table 1: Size distribution and average contents of each coal sample's moisture, volatile matter and ash.

Sample	D ₅₀ (mm)	Moisture content (wt.%)	Ash content (wt.%)	Volatile matter (wt.%)
Witbank (A)	6.8	0.9	8.92	28.6
Highveld (B)	6.5	1.5	15.76	22.1
Highveld (C)	5.4	1.4	14.66	20.0
Molteno-Indwe (D)	5.0	1.8	28.99	13.4

3.5 Experimental procedure

3.5.1 Density measurement: Gas pycnometry

To evaluate the true density of the particles, in samples measurements were performed. Initially, samples were weighed using an electronic balance with a precision of 10⁻⁴ g. A particle's density is defined as the mass of a unit volume at a given temperature. The bulk density of coal, however, can be determined using equation [4]:

$$V_b = \frac{m}{\rho_b} \quad [4]$$

Where ($\text{g}\cdot\text{cm}^{-3}$) represents the bulk density, m is the mass (g), and V_b indicates the bulk volume. (Strydom *et al.*, 2018) However, it takes time to obtain accurate measurements of a particle's volume, exclusive of its pores, particularly on 2-3 mm coal particles. Gas pycnometry is recognised as a reliable technique for determining particles' true density, as shown in figure 3.2. The process involves purging the sample by degassing it with helium flow. This test is accomplished by pressurising the sample cell and expanding helium gas into a reference

chamber. Gas can easily pass through even the tiniest pores in this condition. Archimedes' fluid displacement and Boyle's law are based on two pressure values. Three measurements were taken, and the mean for each sample was calculated using the equation [5]:

$$V_t = \frac{m}{\rho_t} \quad [5]$$

Where ($\text{g}\cdot\text{cm}^{-3}$) represents the bulk density, m is the mass (g), and indicates the true volume. The standard deviation acquired is less than 0.01 g/cm^3 .

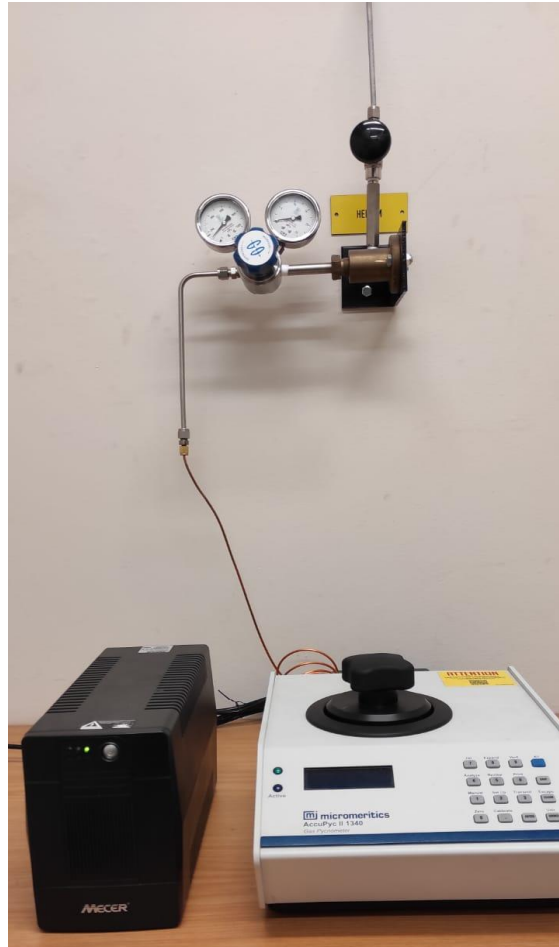


Figure 3. 2: Photograph of helium pycnometry instrumentation

The setup in figure 3.2 calculates helium-based densities using a stereo pycnometer (Quantachrome, MVP-1). It has a digital pressure display resolution of 0.001 psi and an error of 0.2 % when adequately prepared (sample and equipment) and thermally equilibrated. Another gas, such as nitrogen, may also be utilised with little or no measurable difference.

3.5.2 Float-sink tests

The sink-float test was carried out by dipping the sample into a series of liquid solutions of different densities, swirling it for one minute, and leaving it to settle for about five minutes. The solution includes a mixture of distilled water with $ZnCl_2$ (zinc chloride) with varying concentrations, which were used as separating media in all the sink-float tests. Seven consecutive vessels were used, as illustrated in Figure 3.3. The first liquid medium had a density ranging from 1.2 to 1.8 g/cm^3 in 0.1 g/cm^3 increments. The floated coal particles were collected, while sink materials were extracted and washed with tap water before being transferred to the next container with a denser liquid. After each sink-float test, the final sink and float products were washed, dried, and weighed together with all the previous float products. The entire set of collected products was then analyzed.

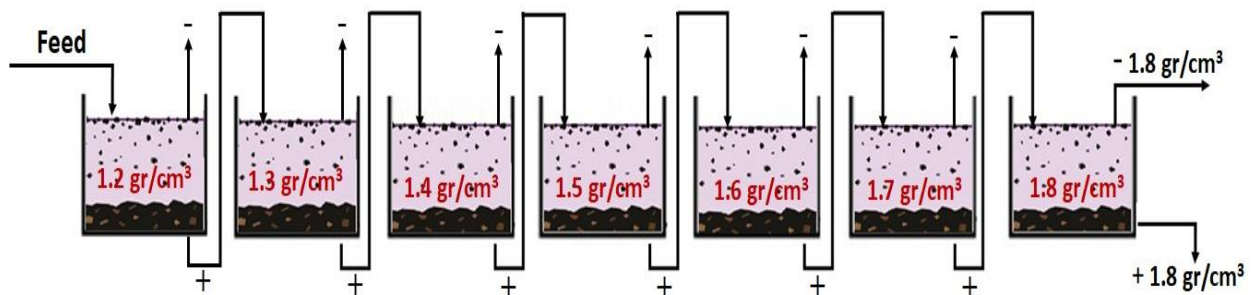


Figure 3. 3: Float-sink tests

3.5.3 RhoVol

RhoVol is an image processing-based system that works in an automated batch process. (Fofana and Steyn, 2019) Individual particles in a batch are weighed and their volumes calculated –using 3D reconstructions; hence any physical dimensions or ratios can be obtained, such as information about the shape factors (elongation, flatness and compactness) or size of the particles. The RhoVol density analyser has a sorting functionality installed to divide a batch of material into 12 bins based on size, density or shape. The machine can process about 800-1200 particles per hour. (Fofana and Steyn, 2019) A total of 275, 247, 302 and 303g of samples (A-D) were loaded into RhoVol. All particles were sorted based on density within a range of 1.1 - 1.7 g/cm^3 , and samples in the failed/recycled bin and under/upper bins were discarded. A correction factor of 1 in the system's software was set to compare the RhoVol findings to sink and float test results, implying that no correction was employed in the machine's analysis. The number of particles from each sample and RhoVol conditions for each test are shown in Table 2.

Table 2: The RhoVol test condition for the four types of coals.

Sample	Valid particles	Mass (g)	Processing time(h)	Lower Sorting Limit (g/m ³)	Upper Sorting Limit (g/m ³)
A	1125	276	1:19:21	1.1	1.7
B	1642	247	2:11:19	1.1	1.7
C	3523	303	5:13:07	1.1	1.7
D	3518	302	6:13:58	1.1	2.0

The multi-stage internal functions of the RhoVol system are shown in Figure 3.4. A minimum of 1000 particles were transferred to primary and secondary feeders, and the mass of each particle sample was captured in the weighing system that uses a high-speed load cell to ensure accurate measurement of each particle. The particle's 3D volumetric representation is created by analyzing photos captured concurrently by seven cameras from different angles.

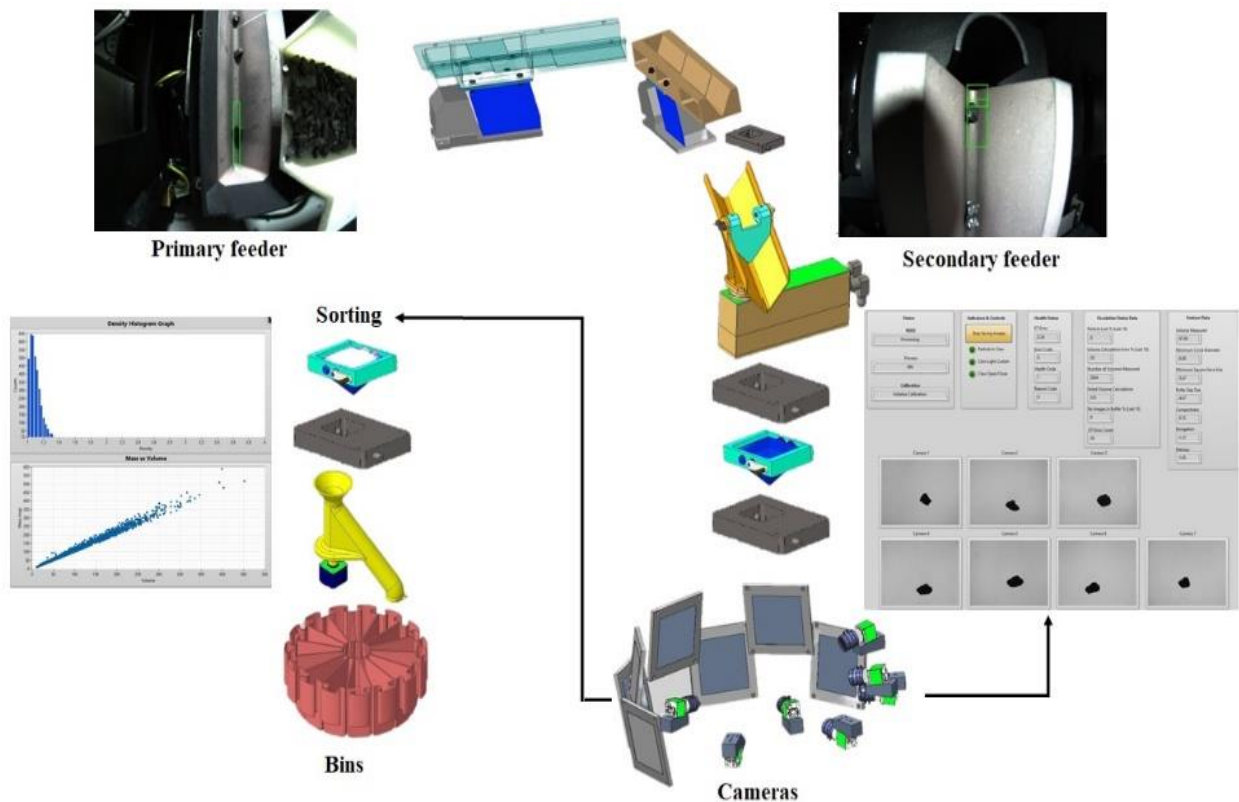


Figure 3. 4: The RhoVol system's multi-stage internal operations(Fofana and Steyn, 2019; Botlhoko *et al.*, 2022b)

a. Operation principle

The operating principle of RhoVol is simple. Operating features has three modes, including single, multi-pass and sorting mode. The feed material enters the system through an intake hopper and settles on the main feeder tray illustrated in figure 3.4. The main tray feeds the material to the open end, which falls into the bowl feeder. A laser distance sensor controls the level in the bowl feeder. Material is fed into the scale gate through the gutter by the bowl feeder, which is regulated on a single-particle basis by the laser sensor. Once the scale has recorded the particle's mass, the chute gate directs the particle into the claw through a light curtain. The claw regulates particle presentation to the VSFD system through a second light curtain. The VSFD measures particle volume and calculates the density of a particle. The cycle is repeated for the next particle until the batch is completed.(Stern and Cronje, 2017; Fofana and Steyn, 2019)

b. Sorting module principle

As discussed above, the claws stop the particle's motion once it has passed through the VSFD mechanism. Once the sorting algorithm has identified the bin position, the sorting funnel is relocated to the right spot. Once fixed in the correct position, it opens to release the particle into the appropriate bin. The light curtain detects successful particle movement, signalling that the sorter is prepared for the next particle.(Steyn and Cronje, 2017)

CHAPTER 4: RESULTS AND DISCUSSIONS

The results obtained are given in this chapter. The chapter is supplemented by an Appendix, which contains the generated results that are not included in this chapter. Chapter four is divided as follows:

- Section 4.1: Presents the results obtained for the RhoVol and Pycnometry test sample as well as the investigation into the effect of PSD, shape and volume.
- Section 4.2: Gives an in-depth description of the correction procedure, and washability curves obtained from RhoVol and float-sink tests as well as the process of achieving the linear regression.
- Section 4.3: Describes the relationship between RhoVol outputs and proximate results to generate the washability data.

4.1 RhoVol and Pycnometry

4.1.1 Particle shape and size distributions

Table 3 details the shape descriptors of four coal samples as obtained from 3D images. Coal A have elongation values ranging from 1.01 to 2.75 and flatness values ranging from 1.01 to 3.30, with an average compactness of 0.73. However, the elongation factor for the last sample in the sample in the table coal D varied widely from 1.01 to 5.10, with the flatness ranging from 1.01 to 8.89 its compactness average is only 0.67. Since Coal A particles had more rounded edges, it resulted in a higher average value of compactness than other coals. Coal (D) had a flatness average of 1.78 and a compactness average of 0.67, its maximum elongation is 5.10, which is higher than other coals. These particles were very angular due to their acute angles, flat surfaces, and edges. RhoVol calculation of size from volume data does not take shape into account, however, it is observed that the flat long particles often have more difficulty passing through a screen.

Table 3: Summary of shape data of four coals

Coal	Elongation				Flatness				Compactness			
	Max	Min	Mean	Std	Max	Min	Mean	Std	Max	Min	Mean	Std
A	2.75	1.01	1.39	0.25	3.30	1.01	1.43	0.29	0.94	0.47	0.73	0.08
B	3.82	1.01	1.47	0.32	5.34	1.01	1.67	0.53	0.94	0.38	0.68	0.10
C	3.99	1.01	1.50	0.35	6.15	1.01	1.69	0.55	0.94	0.33	0.67	0.10
D	5.10	1.01	1.47	0.34	8.89	1.01	1.78	0.69	0.95	0.28	0.67	0.10

The scatter plots of the volume measurements used for density calculation for the four coal samples are shown in Figure 4.1. Comparing the frequency percentage of each sample, there is a difference in the volume of particles. More than half of the particle volume in sample A was higher than 160 mm³ and less than 50 mm³ in samples D and C. The shape descriptors derived from 3D image analysis agreed well with the original images in Figure A1 (appendix A). In that sample A is the most compact with round particles and sample D has elongate particle. These experimental results indicate that RhoVol, as a modern 3D imaging method, is an effective way to quantify differences in coal particle morphology. The random positioning of coal particles against the seven camera viewpoints slightly affects the results of image processing calculations of several geometrical descriptors.

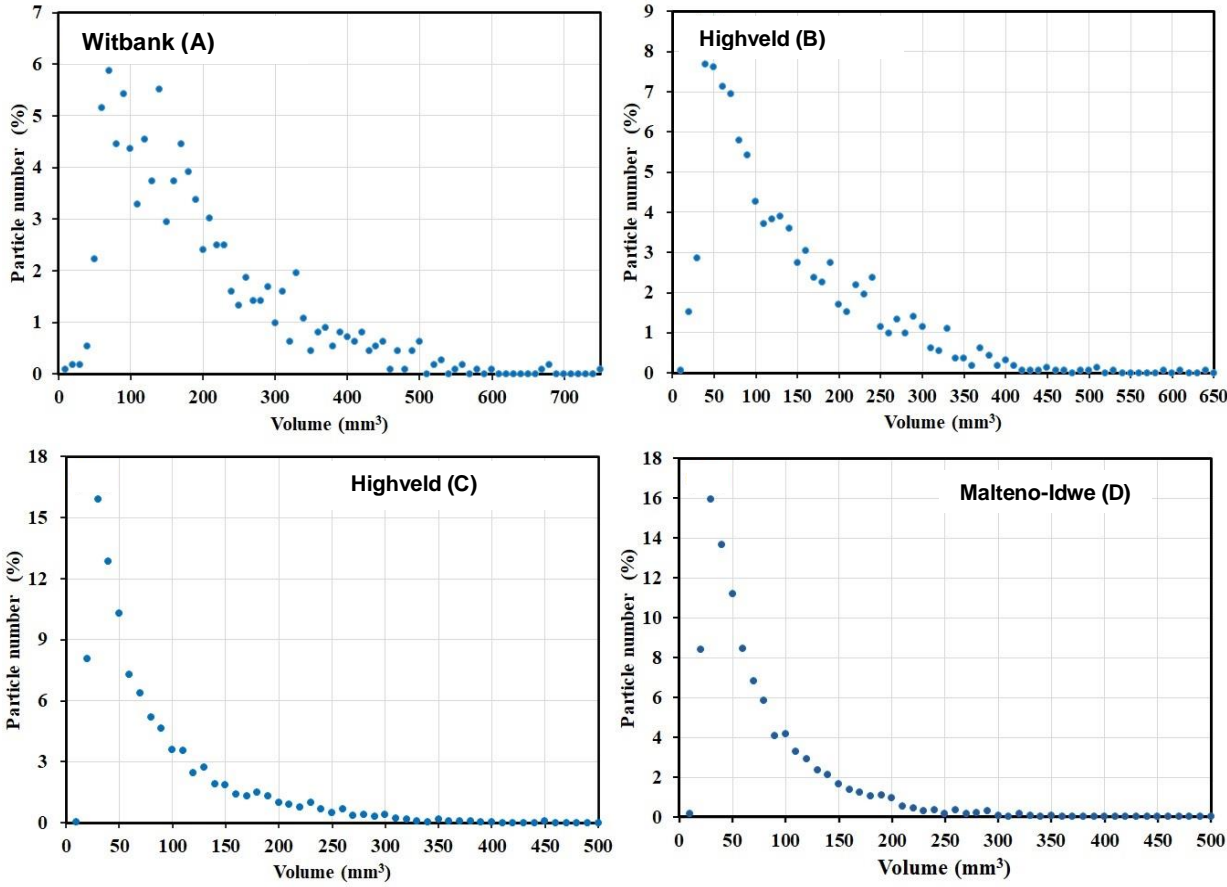
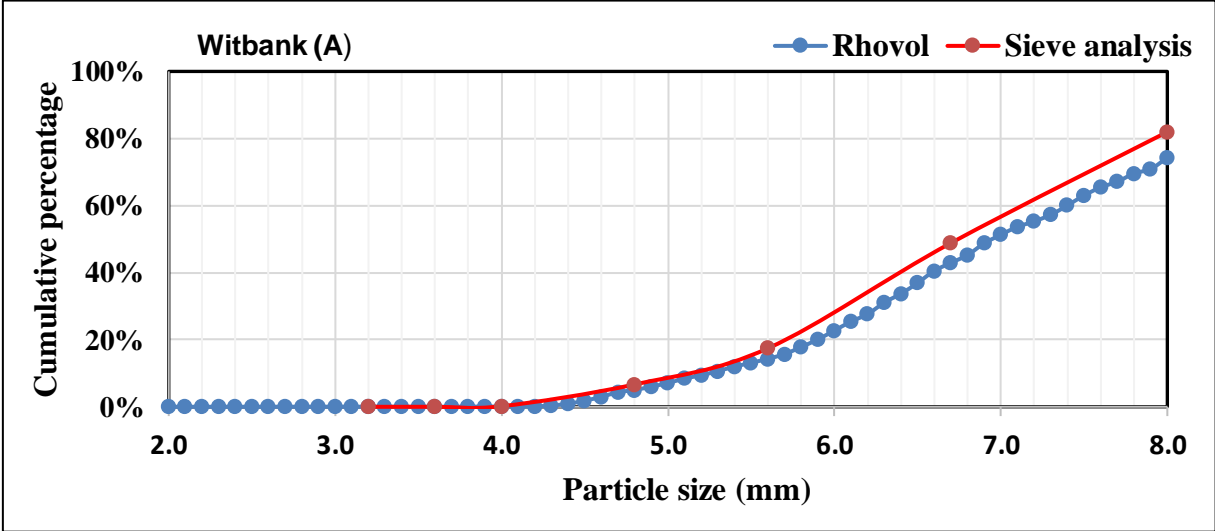


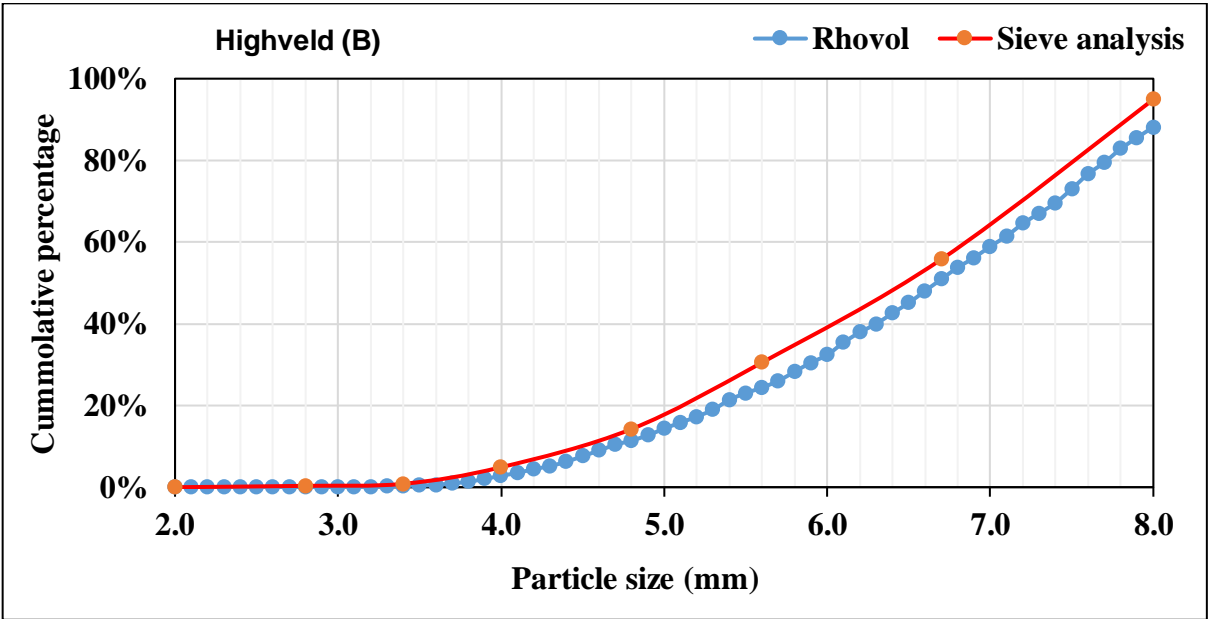
Figure 4. 1: Volume calculation scatter plots for coal A-D samples

The RhoVol and sieving experiments were used to compare the four coal samples' particle size distributions (PSD), as shown in Figure 4.2 (a-d). As measured by the sieves, the distribution shows a slightly coarser distribution than that obtained by image processing in coal A to C. In the case of Coal D, the large discrepancy is probably caused by the longer and flatter particles that do not pass through the screens easily. Similar findings were obtained by Ludwick and

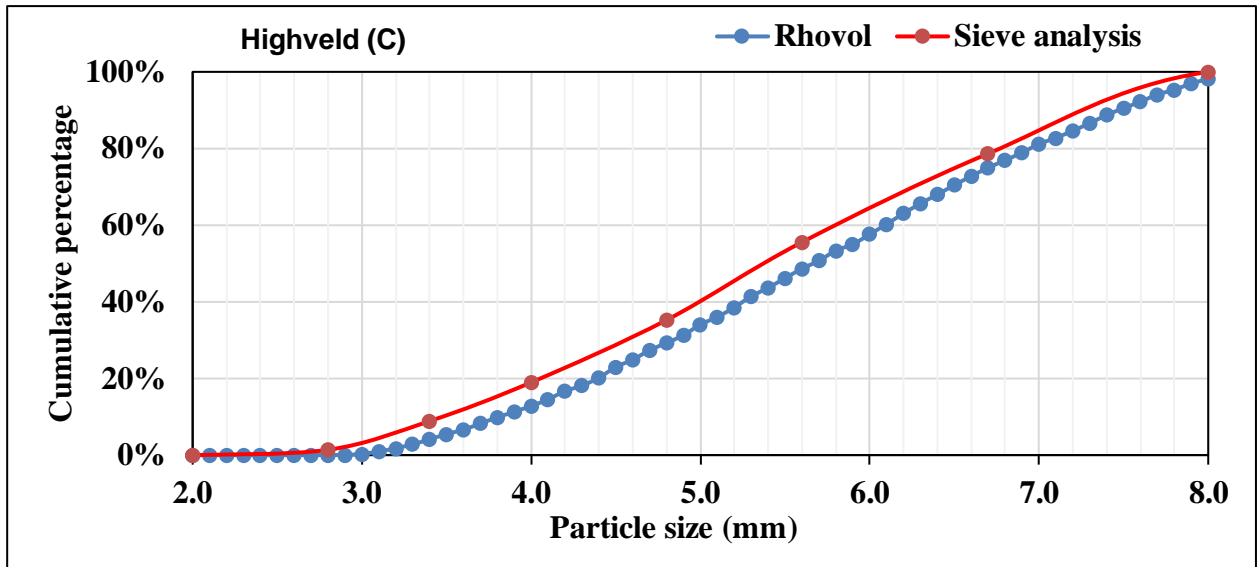
Henderson (1998) and Kim and Dodbiba (2021). As a result, RhoVol is a good reference for establishing the applicability of the image-based technique to particle size measurement, provided the particle shapes are not excessively flat.



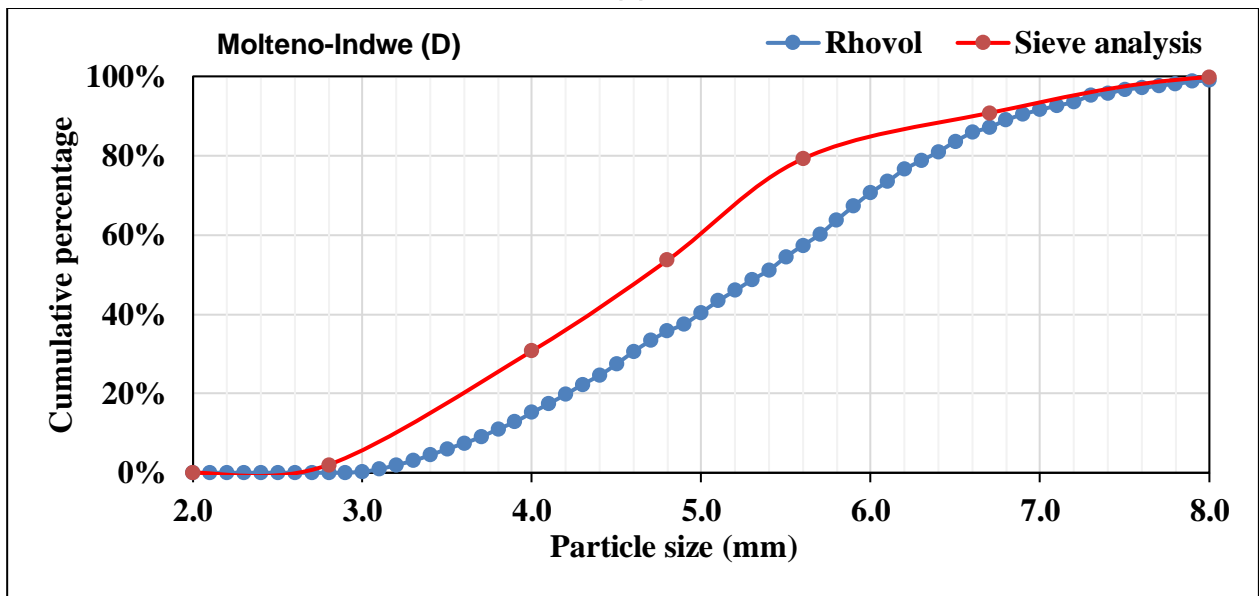
(a)



(b)



(c)



(d)

Figure 4. 2: (a - d) Particle size distributions for four coal samples by sieving and RhoVol methods

4.1.2 Density

Figure 4.3 shows the average density and mass of particles in each sorting bin as determined by the RhoVol. Note that the density values were generated before applying the correction factor. The average raw density values of the bin contents range from 1.05¹ to 2.14 g/cm³. Most particles in sample A have densities in the middle of the range, indicating large presence a larger number of near-dense material. Most Highveld samples (B and C) have densities between 1.05 and 1.24 g/cm³ (bins 1- 3). The average density of 1.05 g/cm³ consist of more than 24 and 50% of particles in samples B and C, respectively. Density value between 1.35 and 1.45 g/cm³ (bins under bin 2) account for more than 65 % of the weight of sample D. In addition, for sample D, there is a relatively broad density distribution. another research study has also found similar results to the ones obtained through XCT image analysis.(Nguyen, A. V Nguyen, *et al.*, 2018) The full description of the density in each bin is in Appendix B.

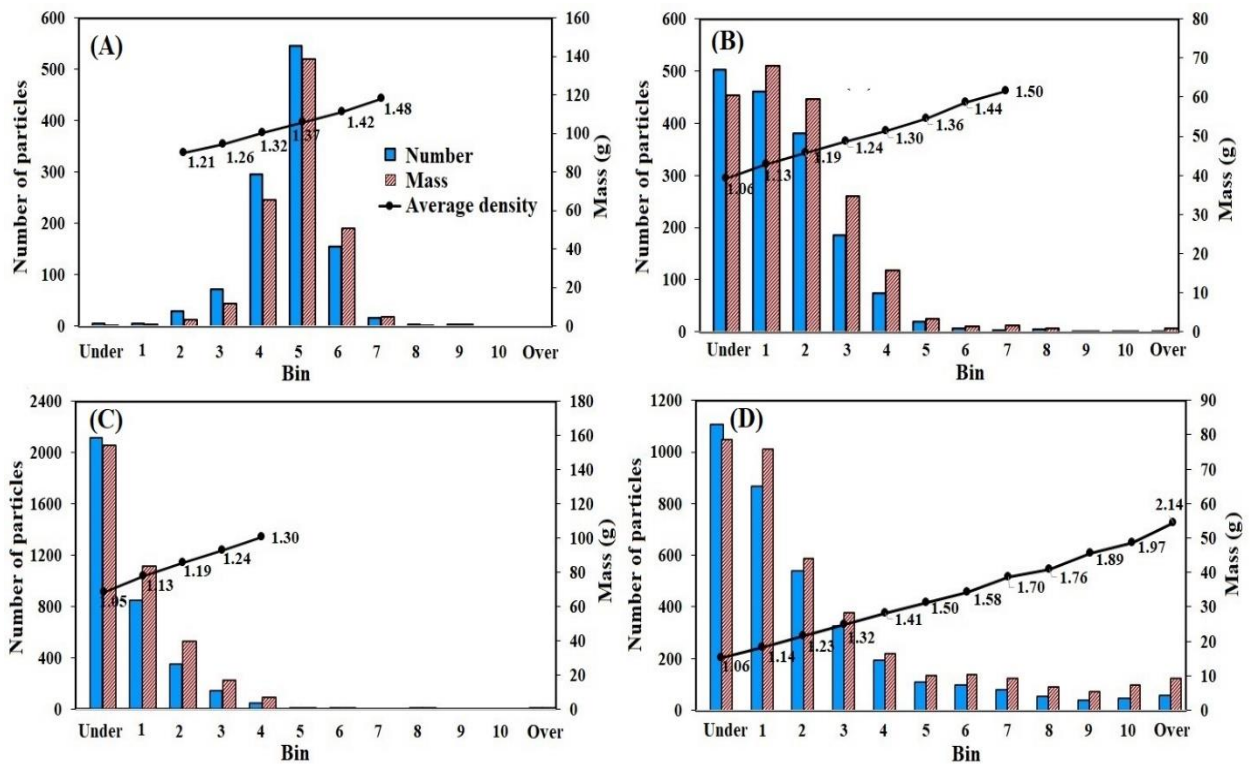


Figure 4. 3: RhoVol data for each bin for Coal A-D

¹ This value is questionable, but it was generated PRIOR to calibration. This clearly shows the need for some back-calibration procedure.

4.2 Density correction procedure

Image processing methods cannot achieve the determination of true volumes of porous particles because the pores and cracks are not accessible with such methods; these are discussed in Chapter 2 Section 2.5. Furthermore, the shape-from-silhouette method does not capture hidden concavities, which can lead to overestimation of particle volume. Without correction, washability data obtained from RhoVol could negatively affect data quality. To eliminate this limitation, a correction factor should be applied to RhoVol data after measurement. This involves fitting a gradient line to all points of the pycnometry dataset.

Table 4 compares each bin's average RhoVol(DR) and pycnometry (DP) densities. The difference between measured RhoVol density and true density(pycnometer) is not constant across all density classes and varies from coal to coal. This difference in density attributed to the different coals' shape, porosity and crack structures.

Table 4: The comparison of the RhoVol (DR) densities (bulk) to the pycnometer (DP) densities for each bin's contents

Sample	Bin's densities												
	Type	Under	1	2	3	4	5	6	7	8	9	10	Over
A	DR	-	-	1.21	1.26	1.32	1.37	1.42	1.48	-	-	-	-
	DP	-	-	1.58	1.6	1.63	1.63	1.65	1.68	-	-	-	-
B	DR	1.062	1.13	1.19	1.25	1.30	1.36	1.44	1.5	-	-	-	-
	DP	1.29	1.40	1.48	1.53	1.57	1.65	1.76	1.80	-	-	-	-
C	DR	1.06	1.13	1.19	1.24	1.30	1.4	-	-	-	-	-	-
	DP	1.29	1.36	1.47	1.54	1.59	1.61	-	-	-	-	-	-
D	DR	1.06	1.14	1.23	1.32	1.41	1.50	1.59	1.70	1.76	1.89	1.97	2.14
	DP	1.32	1.38	1.52	1.61	1.68	1.83	2.01	2.17	2.22	2.38	2.45	2.69

The initial objective is to determine how a CF may be incorporated in the RhoVol data to analyse the Coal's washability and use that technique as an alternative method to the sink and float tests. The first approach was to apply a constant CF to all coal samples as an overall method. This involves finding a correction factor that would result in the minimum difference between the RhoVol and float-sink data. The static CF ratio is defined as the average of all bins in a sample, the CF is 0.82.

4.2.1 Linear regression

Linear regressions were applied to determine the correction between RhoVol and true densities. (Figure 4.4).

The data in table 4 were used to fit a linear correction factor for each coal. The large and positive correlation coefficients, of $R^2 > 0.97$, show that the density offset between the pycnometer and RhoVol densities were close to linear. The density correction function were established as follows:

$$P_{\text{Corrected(A)}} = 0.349 \times \rho_{\text{RhoVol}} + 1.16 \quad [6]$$

$$P_{\text{Corrected(B)}} = 1.137 \times \rho_{\text{RhoVol}} + 0.107 \quad [7]$$

$$P_{\text{Corrected(C)}} = 1.167 \times \rho_{\text{RhoVol}} + 0.097 \quad [8]$$

$$P_{\text{Corrected(D)}} = 1.305 \times \rho_{\text{RhoVol}} - 0.097 \quad [9]$$

However, in comparison to the other samples, Sample A shows a unique curve than the rest of samples with the slope is less than 33% of the other slope and the constant of the curve is more than 10 times the constant of the other. This is a discrepancy between the RhoVol measurement and the sink and float tests for coal A would be due to particle shape, crack, porosity and particle orientation at the time the images were captured.

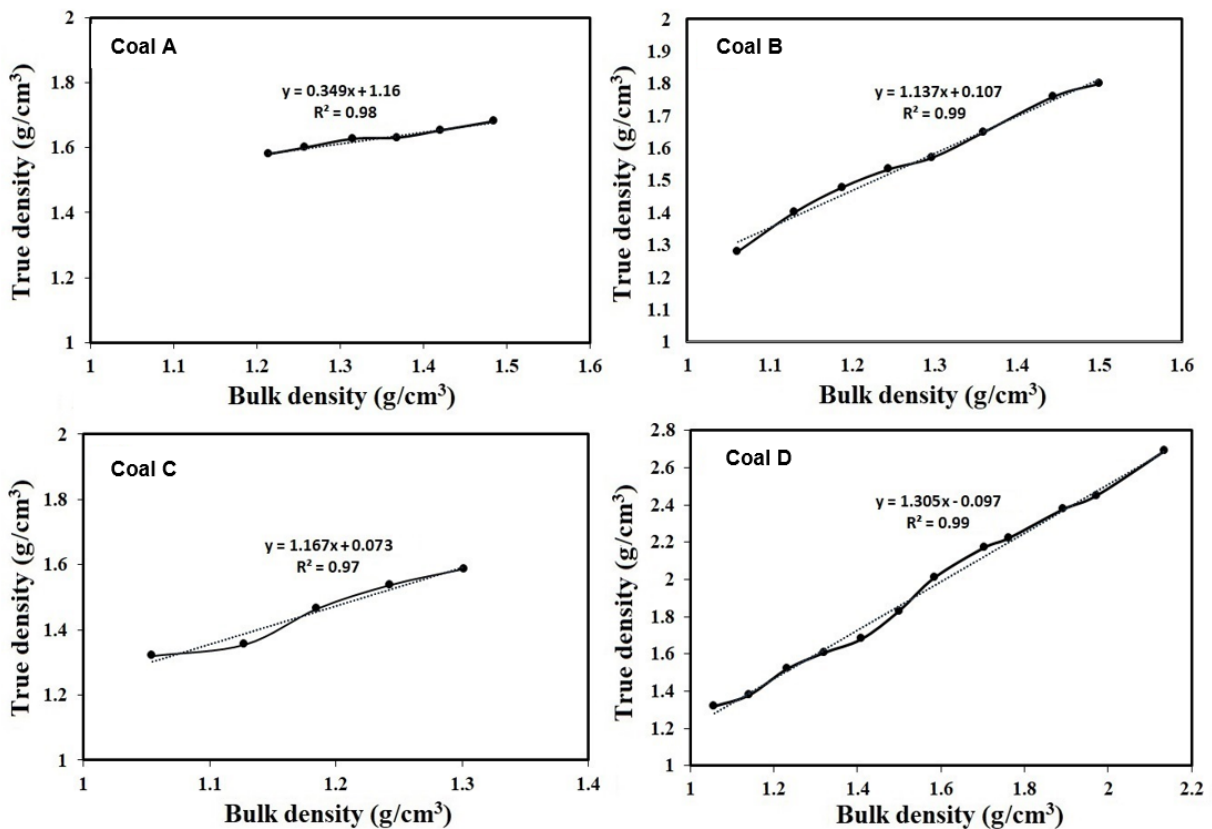


Figure 4. 4: Correlation between average RhoVol densities (bulk) and pycnometer densities of the contents of each bin for coals A-D

For each Coal, the relevant correction function was applied to the RhoVol-generated densities to approximate the true pycnometer density values. Figure 4.5 show corrected RhoVol density distribution vs pycnometer distribution for the various coals as proof of the correction success, due to the close similarity between the histogram in each sample.

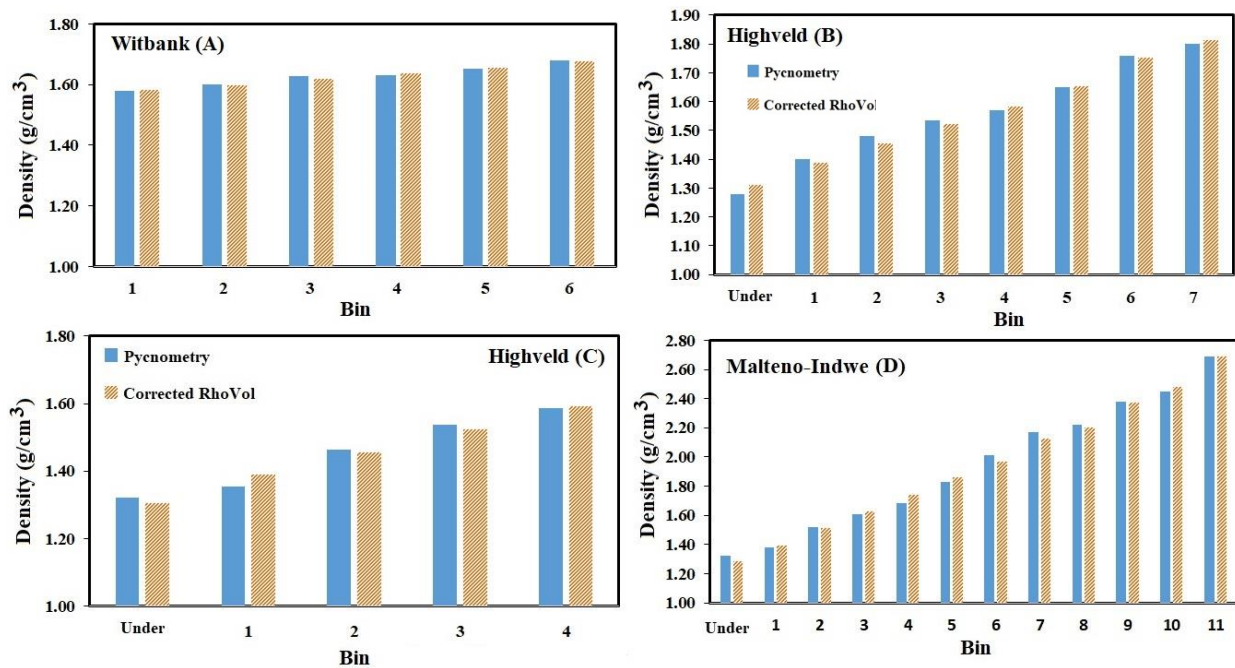


Figure 4. 5: Corrected RhoVol density distribution and pycnometer distribution for each bin

4.2.2 RhoVol and Float-Sink test

As mentioned in sample preparation, all four coal samples were subjected to laboratory density separation using sink–float tests (zinc chloride salt). The washability curves indicate the theoretical yield of clean coal and the operating gravity separation at a given ash content. Furthermore, these curves estimate the yield, quality of waste products and the amount of near-gravity material at the same separation specific gravity.

The float-sink results and the raw RhoVol density distribution (non-corrected) results are shown in Figure 4.6.

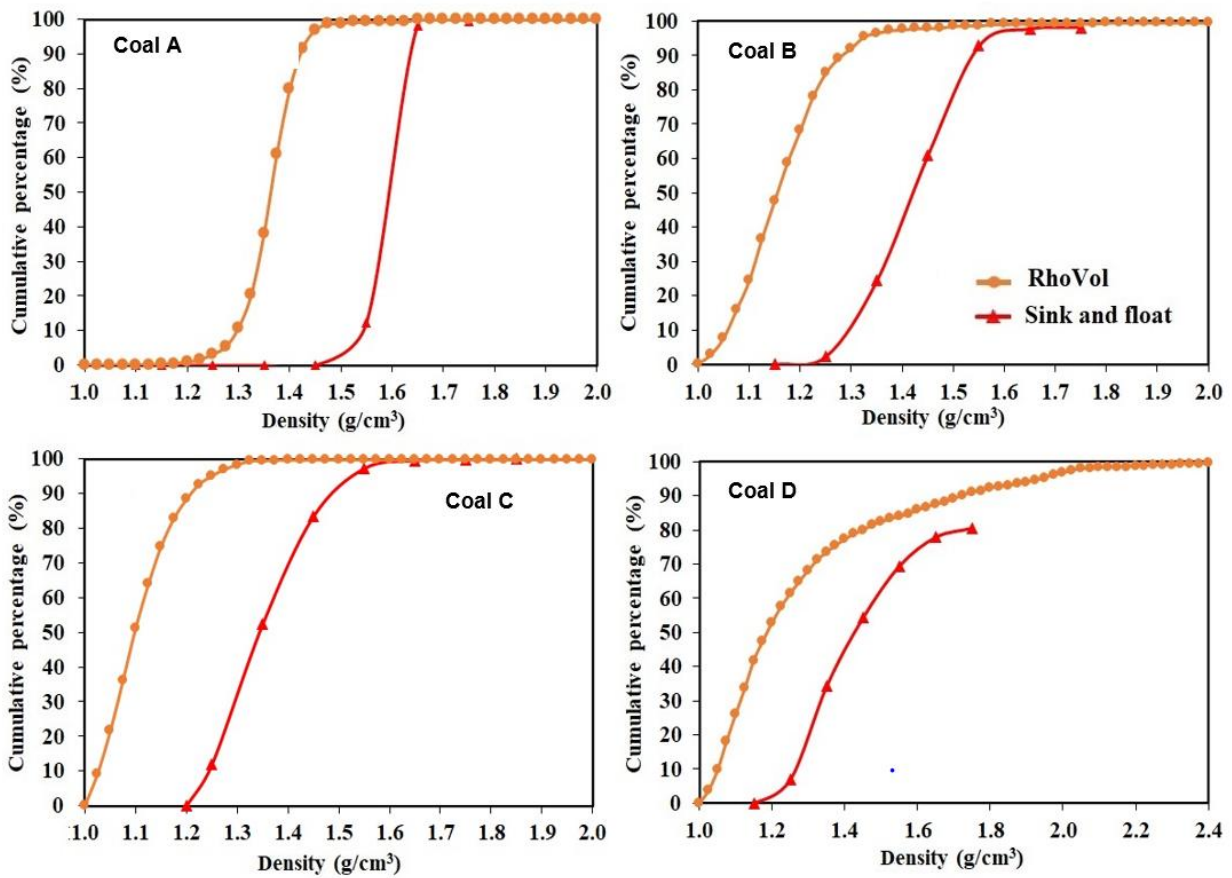


Figure 4. 6: The comparison of the cumulative density distribution obtained by float-sink and RhoVol experiments; Coal A-D (non-corrected)

The RhoVol density curve of coal A indicates most particles with a density under 1.3g/m^3 increase rapidly to 1.4g/m^3 , whereas float-sink data of the sample show little and no particles with density above 1.6g/m^3 . The densimetric curves for coal B-D are essentially identical. This implies that the mineral content of the coal is intrinsic. However, there is poor correspondence between RhoVol (non-corrected data) and float and sink data. This is due to the RhoVol method failing to capture hidden concavities in particle of a sample, leading to an overestimation of the coal particle volume. Furthermore, it is impossible to consider the influence of porosity and cracks on a volume of coal sample derived from 3D silhouette images. Figure 4.7 shows the same data after applying the correction function for each coal's based on the static correction factor.

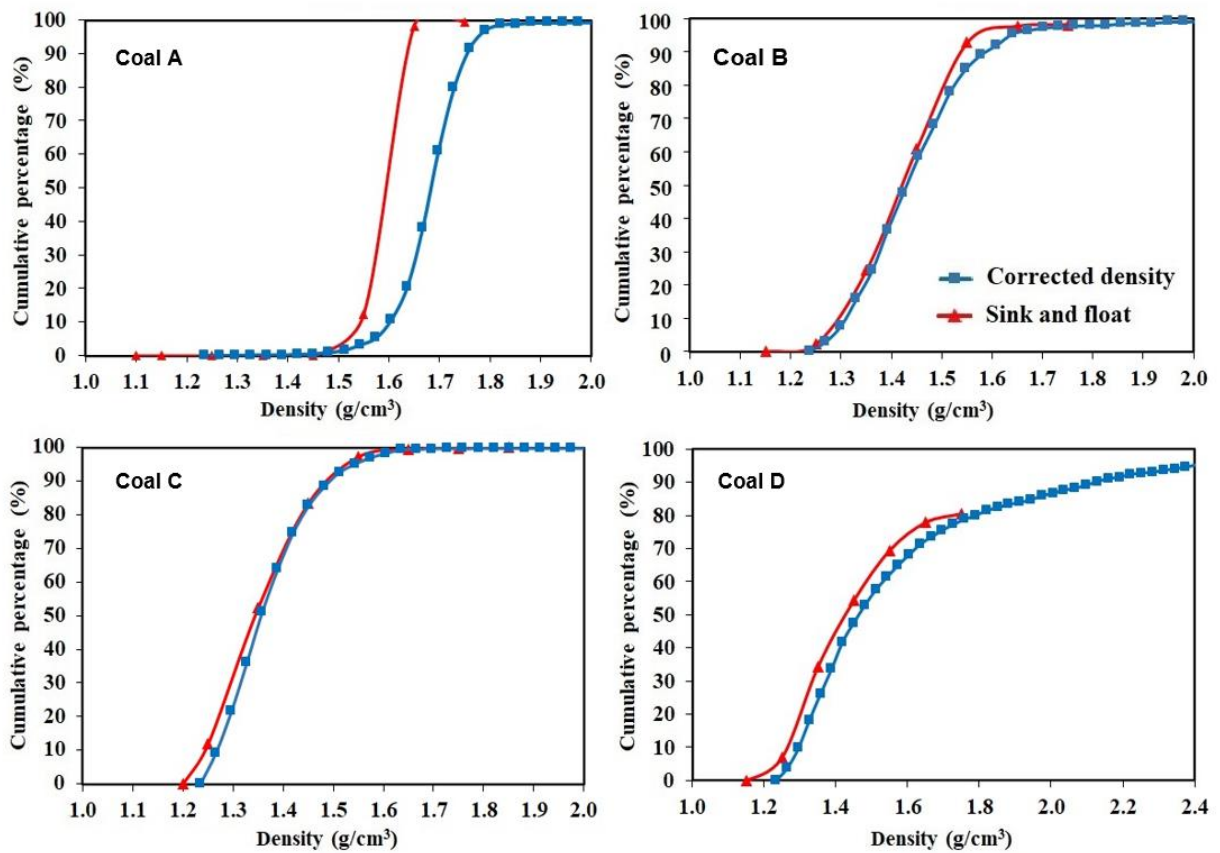


Figure 4. 7: Cumulative density distribution derived from sink-float and corrected RhoVol based on static correction factor tests for Coal A-D

Figure 4.7 shows the Cumulative density distribution derived from sink-float and corrected RhoVol based on static correction factor. It is observed that RhoVol is more efficient for sample with large number of particle (Coal B-D, chapter 3 Table 2). Particles with density much greater than the separation density will report to underflow, while less dense particles will report to overflow all with high efficiency. Coal A also shows more particles with lower density ($<1.2\text{g/m}^3$) than other Coal samples, which may be due to the residence time particle inside the medium and the ash content in coal.

Figure 4.8 presents a similar plot to figure 4.7 with the exception that the linear correction functions are used to generate the density distributions. The densimetric plots for sample A coal A shows better similarities in this figure compared with value in figure 4.7. Density distributions for coal B-D remain identical to distribution in figure 4.7.

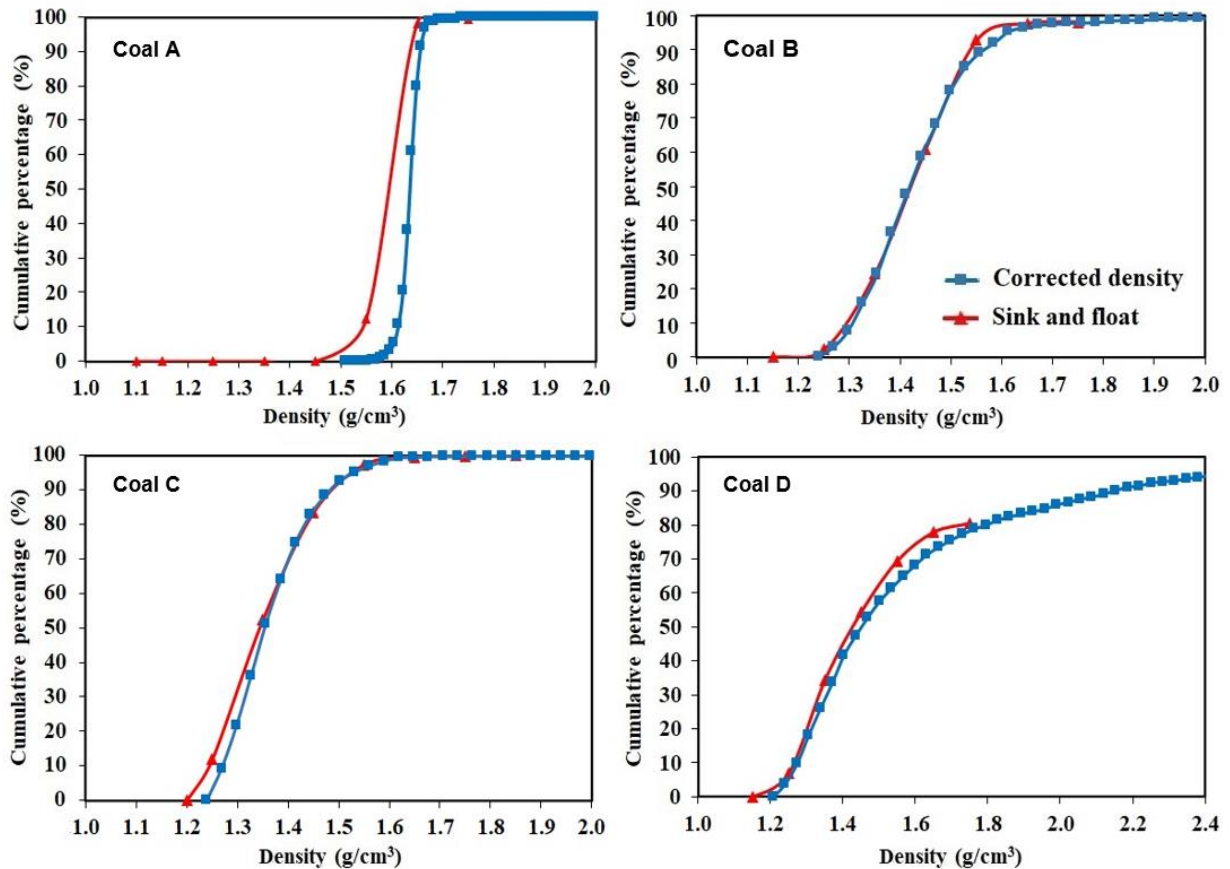


Figure 4. 8: Sink-float and corrected RhoVol (based on linear regression models) cumulative density distributions Coal A-D.

Float-sink tests will not give many resolutions around the d_{50} . Hence curve tends to be inaccurate in that area for instance Coal A. In contrast, (figure 4.8) RhoVol can give a much better densimetric curve showing a more accurate estimate of d_{50} .

4.3 Establishing the ash–density relationship

The washability curves determine the theoretical balance of coal-washing products and the appropriate separation density for a coal preparation plant's maximum efficiency. Because coals are made up of a range of macerals and may contain multiple types of gangue material, the relationship between particle density and ash level varies from coal to coal.

The relationships between RhoVol density outputs and ash contents were examined to generate the washability data. Figure 4.9 depicts the relationship between ash content and corrected RhoVol density data for each coal sample. The linear regression indicates a good correction between the average density of each bin and the ash content as seen with ($R^2 >$

0.89). The ash content of the float fractions increases as the density increases. This is because the organic fraction in Coal has a lower density of hydrocarbons compared to the density of gangue minerals. As coal density increases, the amount of carbonaceous mineral in coal decreases while the amount of mineral matter characterised by ash content increases.

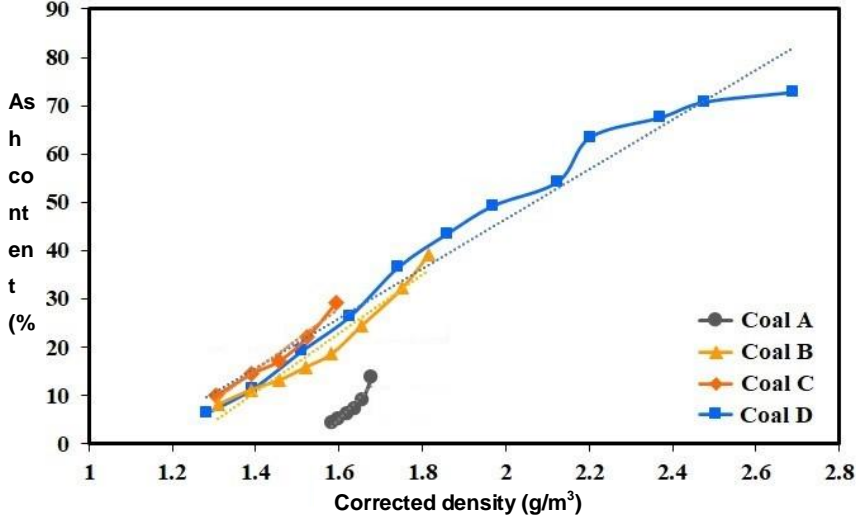


Figure 4. 9: Correlation between RhoVol's bins' average corrected density and ash content. Each point shows the average particle densities in each bin.

The RhoVol data can be used to plot washability curves without performing the float-sink test, saving significant time, labour, and heavy liquid amounts. RhoVol can sort coal particles across a wide range of densities, unlike in float-sink tests, which are typically performed only at limited density ranges 1.2 to 1.4g/m³. Therefore, the RhoVol technique is then a viable alternative to float-sink tests.

CHAPTER 5: CONCLUSIONS

- Traditional coal washability analysis is established by conducting the float-sink test, which is time-consuming and destructive to the integrity of the original coal as it disintegrates somewhat when immersed. In addition, the float-sink test uses environmentally hazardous chemicals. In this study, an alternative technique based on machine vision (3D imaging system) called RhoVol was also used. In this device, the densities of individual particles are measured by separate mass and volume measurements, which were used to determine coal washability curves
- Four different types of Coal were taken from three coal mines (Highveld, Witbank, and Molteno-Indwe) in South Africa. The coal samples (-8 to +3mm) were sorted based on density, using zinc chloride for float-sink, over density range 1.2 to 1.8 g/cm³ and RhoVol up to 2.4g/m³.
- RhoVol could estimate the PSD fairly accurately, provided that not too many elongated and flat particles were present.
- The shape-from-silhouette method as it is used in RhoVol failed to capture the hidden concavities in a particle leading to an overestimation of the particle volume. Moreover, the 3D pictures generated by RhoVol could not include the effect of porosity and cracks on the volume of coal samples. The densities achieved by RhoVol were then corrected using a linear correction function determined from true density measurements using a pycnometer.
- The results showed an excellent agreement between the RhoVol measurements and sink and float test data AFTER applying a correction function, determined from pycnometer measurements, to the raw RhoVol density data.
- It was well understood from the experimental findings that the RhoVol had the potential to be an alternative to conventional float and sink analysis, if that data generated are calibrated correctly.

RECOMMENDATIONS

Future work can be aimed at exploring additional ways such as line neural network models or nonlinear regression methods to decrease the density estimation errors of coal samples when using RhoVol. The work can also be applied to a larger dataset to ensure that the method is robust.

REFERENCES

- Adinugraha, W. Sulaksana, N. Hendarmawan, H. Santoso, B. Umar, D. F. Amalia, F. (2018) 'Washing test of Kendilo coal using a sink-float method to improve its quality', *Indonesian Mining Journal*, 21(1), pp. 35–44. doi:10.30556/imj.vol21.no1.2018.408.
- Alamooti, A.M. and Malekabadi, F.K. (2018) 'An Introduction to Enhanced Oil Recovery', in *Fundamentals of Enhanced Oil and Gas Recovery from Conventional and Unconventional Reservoirs*. Gulf Professional Publishing, pp. 1–40. doi:10.1016/B978-0-12-813027-8.00001-1.
- Blake, G.R. and Hartge, K.H. (2018) 'Particle density', in *Methods of Soil Analysis, Part 1: Physical and Mineralogical Methods*. Wiley, pp. 377–382. doi:10.2136/sssabookser5.1.2ed.c14.
- Botlhoko, S. Campbell, Q. P. le Roux, M. Nakhaei, F. (2022a) 'Washability Analysis of Coal Using RhoVol: A Novel 3D Image-based Method', *Mineral Processing and Extractive Metallurgy Review* [Preprint]. doi:10.1080/08827508.2022.2027769.
- Botlhoko, S. Campbell, Q. P. le Roux, M. Nakhaei, F. (2022b) 'Washability Analysis of Coal Using RhoVol: A Novel 3D Image-based Method', *Mineral Processing and Extractive Metallurgy Review* [Preprint]. doi:10.1080/08827508.2022.2027769.
- Cakmak, E. Annamraju, A. Mathews, J.P. He, Lilin. Gallego, Nidia. Lara-Curzio, E. (2023) 'Characterization of Porosity and Pore Accessibility of Vitrinite-Rich Bituminous and Subbituminous Coals by Small-Angle Neutron Scattering, Mercury Intrusion Porosimetry, and Low-Pressure N₂ Adsorption', *Energy & Fuels*, 37(1), pp. 191–203. doi:10.1021/acs.energyfuels.2c02814.
- Calvo, M. García, R. Arenillas, A. Suárez, I. Moinelo, Sabino R. (2005) 'Carbon foams from coals. A preliminary study', *Fuel*, 84(17), pp. 2184–2189. doi:10.1016/J.FUEL.2005.06.008.
- Dai, W. Zhang, L. Fu, J. Chai, T. Ma, X. (2020) 'Model-data-based switching adaptive control for dense medium separation in coal beneficiation', *Control Engineering Practice*, 98, p. 104241. doi:10.1016/j.conengprac.2019.104241.
- Diedericks, E., Le Roux, M. and Campbell, Q. (2021) *Dry beneficiation of +0.5mm-5.6mm South African coal using an air dense medium fluidization bed*. doi:10.21203/rs.3.rs-678819/v1.

Fofana, M. and Steyn, T. (2019) 'Monitoring the performance of DMS circuits using RhoVol technology', *Journal of the Southern African Institute of Mining and Metallurgy*, 119(2), pp. 133–138. doi:10.17159/2411-9717/2019/v119n2a5.

Grant High (2018) 'Debtech's RhoVol: real-time sample density measurement', (April), p. 2018.

Gui, X. Liu, J. Cao, Y. Miao, Z. Li, S. Xing, Y. Wang, D. (2015) 'Coal preparation technology: Status and development in China', *Energy and Environment*, 26(6–7), pp. 997–1013. doi:10.1260/0958-305X.26.6-7.997.

Inglezakis, V.J. and Pouloupoulos, S.G. (2006) 'Heterogeneous Processes and Reactor Analysis', *Adsorption, Ion Exchange and Catalysis*, pp. 57–242. doi:10.1016/B978-044452783-7/50003-3.

Jargalmaa, S. Gerelmaa, T. Baterdene, E. Tsatsral, G. Avid, B. Purevsuren, B. Dugarjav, J. (2015) 'Washability of Coal from Seams IV and VIII of the Tavantolgoi Deposit', *Natural Resources Research*, 24(2), pp. 189–195. doi:10.1007/s11053-014-9245-9.

Luttrell, G.H. and Honaker, R.Q. (2012) 'CoalcoalPreparationcoalpreparation BT - Encyclopedia of Sustainability Science and Technology', in Meyers, R.A. (ed.). New York, NY: Springer New York, pp. 2194–2222. doi:10.1007/978-1-4419-0851-3_431.

Majumder, A.K. and Barnwal, J.P. (2004) 'Development of a new coal washability index', *Minerals Engineering*, 17(1), pp. 93–96. doi:10.1016/j.mineng.2003.10.005.

Mallol, G. Llorens, D. Boix, J. Aguilera, M. Foucar, L. Arnau, J. (2010) 'Rapid, harmless, and non-destructive measurement of ceramic tile bulk density', *Boletín de la Sociedad Española de Cerámica y Vidrio*, 49.

Mangera, R., Morrison, G. and Voigt, A. (2017) 'Particle volume correction using shape features', in *2016 Pattern Recognition Association of South Africa and Robotics and Mechatronics International Conference, PRASA-RobMech 2016*, pp. 1–6. doi:10.1109/RoboMech.2016.7813181.

Merkus, H.G. (2009) 'Particle Size, Size Distributions and Shape', in *Particle Size Measurements*. Springer, Dordrecht, pp. 13–42. doi:10.1007/978-1-4020-9016-5_2.

Nguyen, T.D., Nguyen, A. V, Lin, Chen L. Mi, Jan D. (2018) 'Application of high-resolution X-ray microcomputed tomography for coal washability analysis', *Minerals Engineering*, 124, pp. 137–148. doi:10.1016/j.mineng.2018.05.027.

Nie, X. Chen, J. Cao, Y. Gong, D. Deng, H. (2018) 'Analysis of Coal Swelling Deformation Caused by Carbon Dioxide Adsorption Based on X-Ray Computed Tomography', *Geofluids*. Edited by U. Tinivella, 2018, p. 6939827. doi:10.1155/2018/6939827.

Pudasainee, D., Kurian, V. and Gupta, R. (2020) '2 - Coal: Past, Present, and Future Sustainable Use', in Letcher, T.M. (ed.) *Future Energy (Third Edition)*. Elsevier, pp. 21–48. doi:https://doi.org/10.1016/B978-0-08-102886-5.00002-5.

Roux, L. (2021) 'Density - A contentious issue in the evaluation and determination of Resources and Reserves in coal deposits ', *Journal of the Southern African Institute of Mining and Metallurgy* . scieloza , pp. 227–250.

Sahu, D., Chaurasia, R.C. and Suresh, N. (2019) 'Mineralogical characterization and washability of Indian coal from Jamadoba', *Energy Sources, Part A: Recovery, Utilization and Environmental Effects*, 41(5), pp. 517–526. doi:10.1080/15567036.2018.1520336.

Strydom, C. Mthombo,S. Bunt, J.R. Neomagus, H. (2018) 'Some physical and chemical characteristics of calcium lignosulfonate-bound coal fines.', *Journal of the Southern African Institute of Mining and Metallurgy*, 118, pp. 1277–1283. doi:10.17159/2411-9717/2018/v118n12a6.

T Steyn (De Beers Group Excite), and Cronje, N. (2017) 'RHOVOL_manual.pdf'. Johannesburg, p. 99.

Yao, S. Jiao, K. Zhang, Ke. Hu, WenXuan Ding, H. Li, M. Pei, W. (2011) 'An atomic force microscopy study of coal nanopore structure', *Chinese Science Bulletin*, 56(25), p. 2706. doi:10.1007/s11434-011-4623-8.

APPENDIX A

The procedure of proximate analysis

Moisture Content (M)

In a silica crucible, 1 gram of finely pulverised (212 microns) air-dried coal sample is weighed and then placed in an electric hot air oven. It's kept at 1100 degrees Fahrenheit (593^o C). The crucible containing the coal sample is kept in the oven for 1.5 hours before being removed with tongues, cooled in a desiccator for roughly 15 minutes, and weighed. Moisture is the term used to describe weight loss.

Ash Determination procedure

An empty silica crucible was initially heated for one hour in a muffle furnace. The weight was obtained after being allowed to cool at room temperature. One gram of coal sample was weighed in the crucible and heated for 30 minutes at 450 °C in the muffle furnace. The furnace's temperature was then adjusted to 900 °C and maintained for another 24 hours. The crucible was then removed and weighed after being placed in the desiccator. On a percentage basis, the residue was reported as ash percentage.

Presented below are the data generated from proximate analysis.

Table A1: Proximate data results for all four coals. (a-d)

Witbank (Coal A)	Density			
	1.55	1.65	1.75	1.80
Crucible	8.37	8.42	8.38	8.58
Particles	1.026	1.08	0.99	1.02
After 105 (particle)	0.99	1.064	0.98	1.01
volatile crucible	11.50	10.40	10.75	10.97
volatile crucible +particle	12.48	11.47	11.73	11.98
After 900 (volatile crucible +particle)	12.30	11.39	11.63	11.79
After 900 (particle)	0.146	0.08	0.09	0.18
Volatile matter	14.23	7.71	9.43	18.41
Ash crucible	11.46	10.40	10.75	10.98
After 1100 (crucible +particle)	11.51	10.49	10.89	11.21
After 1100 (particle)	0.05	0.096	0.13	0.23
Ash%	4.97	8.85	13.22	22.56

(a)

Highveld (Coal B)	Density						
	1.25	1.35	1.45	1.55	1.65	1.75	1.80
Crucible	8.582	8.38	8.56	8.25	8.56	8.25	8.25
Particles	1.042	1.00	1.027	1.012	1.05	1.06	1.02
After 105 (particle volatile crucible	1.01	0.99	1.023	0.978	1.023	1.04	0.99
volatile crucible +particle	10.98	11.46	11.55	11.332	11.55	14.99	11.05
After 900 (volatile crucible +particle)	11.99	12.45	12.5728	12.31	12.57	16.02	12.05
After 900 (particle)	11.571	12.11	11.30	12.09	12.37	15.9	11.97
Volatile	0.41	0.34	0.27	0.21	0.20	0.13	0.07
Ash crucible	39.83	33.87	26.27	20.85	19.33	11.94	7.23
After 1100 (crucible +particle)	10.98	11.46	11.55	11.33	11.55	14.99	11.05
After 1100 (particle)	11.03	11.56	11.67	11.49	11.74	15.30	11.42
Ash%	0.047	0.10	0.12	0.16	0.20	0.31	0.38
	4.51	9.70	11.72	15.81	18.67	29.21	37.24

(b)

Highveld (coal C)	Density						
	1.25	1.35	1.45	1.55	1.65	1.75	1.80
Crucible	8.46	8.47	8.25	8.38	8.38	8.31	8.52
Particles	1.06	1.04	1.10	1.03	1.01	1.02	0.51
After 105 (particle volatile crucible	1.06	1.03	1.08	1.01	0.98	1.06	0.50
volatile crucible +particle	10.56	10.56	11.34	10.79	10.21	14.23	11.40
After 900 (volatile crucible +particle)	11.61	11.58	12.41	11.79	11.19	15.28	11.90
After 900 (particle)	11.22	11.05	12.12	11.56	10.98	15.01	11.76
Volatile	0.39	0.53	0.29	0.23	0.21	0.27	0.14
Ash crucible	36.88	51.29	26.59	22.50	20.41	26.45	27.95
After 1100 (crucible +particle)	10.56	11.50	11.34	10.79	10.21	14.23	11.40
After 1100 (particle)	11.62	11.69	11.52	11.00	10.48	14.60	11.66
Ash%	0.06	0.14	0.18	0.21	0.278	0.37	0.25
	5.83	13.03	16.49	20.76	27.56	36.53	49.22

(c)

Maltene (Coal D)	Density						
	1.25	1.35	1.45	1.55	1.65	1.75	1.80
Crucible	8.52	8.38	8.27	8.10	8.10	8.53	8.42
Particles	1.08	1.01	1.05	1.01	1.03	1.00	1.00
After 105 (particle)	1.01	0.99	0.99	0.99	1.01	0.98	0.98
volatile crucible	10.75	10.21	11.04	11.59	11.59	10.79	10.40
volatile crucible +particle	11.76	11.21	12.03	12.58	12.61	11.76	11.38
After 900 (volatile crucible +particle)	11.37	10.83	11.83	12.34	12.46	11.58	11.23
After 900 (particle)	0.39	0.38	0.19	0.21	0.15	0.18	0.15
Volatile matter	37.02	37.48	18.75	21.01	14.69	18.75	15.02
Ash crucible	10.753	10.214	11.0367	11.594	11.594	10.7881	10.40
After 1100 (crucible +particle)	10.80	10.30	11.19	11.81	11.86	11.18	11.10
After 1100 (particle)	0.05	0.08	0.15	0.22	0.26	0.39	0.69
Ash%	4.65	8.25	14.90	21.70	25.65	38.67	68.92

(d)

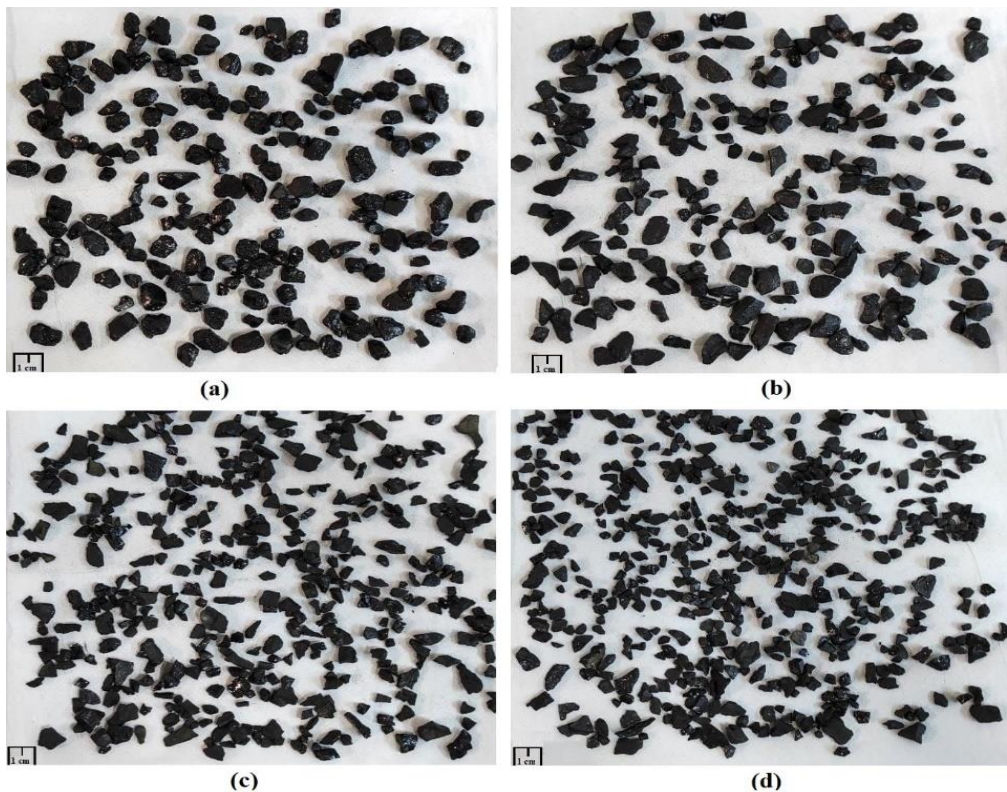


Figure A 1 Photograph of four samples of coal; Coal A-D

APPENDIX B

Presented in this appendix are the results of RhoVol data from all coal sample

Table A 2: RhoVol data results for Coal A

	Circulation 1	Circulation	Circulation	Circulation	Circulation 5								
Valid Captures:	1125	0	0	0	0								
Fed Particles:	1197	0	0	0	0								
	Failed Volumes :	31	0	0	0								
	Process Time:	1:19:21	0:00:00	0:00:00	0:00:00								
Mass (g):	276	0	0	0	0								
Sort According To:	Density												
Upper Sorting Limit:	1.7												
Lower Sorting Limit:	1.1												
	Bin 1	Bin 2	Bin 3	Bin 4	Bin 5	Bin 6	Bin 7	Bin 8	Bin 9	Bin 10	Over	Under	Total
Counts:	5	29	72	295	545	155	16	2	2	0	0	4	1125
Volume (cm3):	0.5	2.8	9.3	49.7	101.1	35.6	3.3	0.2	0.5	0	0	0.3	16.94167
Mass (g):	0.6	3.4	11.7	65.4	138.4	50.6	4.9	0.3	0.8	0	0	0.3	23.03333
Average density (gr/cm3)	1.20	1.21	1.26	1.32	1.37	1.42	1.48	1.50	1.60	NA	NA	1.00	1.36

Table A 3: RhoVol data results for Coal B

	Circulation 1	Circulation	Circulation	Circulation	Circulation 5								
Valid Captures:	1642	0	0	0	0								
Fed Particles:	2046	0	0	0	0								
	139	0	0	0	0								
	2:11:19	0:00:00	0:00:00	0:00:00	0:00:00								
Mass (g):	247	0	0	0	0								
Sort According To:	Density												
Upper Sorting Limit:	1.7												
Lower Sorting Limit:	1.1												
	Bin 1	Bin 2	Bin 3	Bin 4	Bin 5	Bin 6	Bin 7	Bin 8	Bin 9	Bin 10	Over	Under	Total
Counts:	460	380	185	75	19	6	4	5	1	2	2	503	1642
Volume (cm):	60.1	50	27.8	12.1	2.5	0.9	1.1	0.6	0.2	0.1	0.5	56.9	17.73333
Mass (mg):	67.9	59.4	34.6	15.7	3.4	1.3	1.6	1	0.3	0.2	0.8	60.4	20.55
Average density (gr/cm3)	1.13	1.19	1.24	1.30	1.36	1.44	1.45	1.67	1.50	2.00	1.60	1.06	1.16

Table A 4: RhoVol data results for Coal C

	Circulation 1	Circulation	Circulation	Circulation	Circulation 5								
Valid Captures:	3523	0	0	0	0								
Fed Particles:	5183	0	0	0	0								
Failed Volumes :	307	0	0	0	0								
Process Time:	5:13:07	0:00:00	0:00:00	0:00:00	0:00:00								
Mass (g):	303	0	0	0	0								
Sort According To:	Density												
Upper Sorting Limit:	1.7												
Lower Sorting Limit:	1.1												
	Bin 1	Bin 2	Bin 3	Bin 4	Bin 5	Bin 6	Bin 7	Bin 8	Bin 9	Bin 10	Over	Under	Total
Counts:	850	353	141	45	8	2	0	2	0	0	3	2119	1125
Volume (cm):	74.3	33.5	13.6	5.3	0.5	0.1	0	0.1	0	0	0.1	146.5	22.83333
Mass (mg):	83.8	39.7	16.9	6.9	0.7	0.2	0	0.1	0	0	0.3	154.5	25.25833
Average density (gr/cm3)	1.13	1.19	1.24	1.30	1.40	2.00					NA	1.05	1.11

Table A 5: RhoVol data results for Coal D

	Circulation 1	Circulation	Circulation	Circulation	Circulation 5								
Valid Captures:	3518	0	0	0	0								
Fed Particles:	5150	0	0	0	0								
Failed Volumes :	89	0	0	0	0								
Process Time:	6:13:58	0:00:00	0:00:00	0:00:00	0:00:00								
Mass (g):	302	0	0	0	0								
Sort According To:	Density												
Upper Sorting Limit:	2												
Lower Sorting Limit:	1.1												
	Bin 1	Bin 2	Bin 3	Bin 4	Bin 5	Bin 6	Bin 7	Bin 8	Bin 9	Bin 10	Over	Under	Total
Counts:	868	541	327	193	109	99	80	55	37	45	58	1106	1125
Volume (cm):	66.5	35.7	21.5	11.7	6.8	6.5	5.4	3.8	2.8	3.7	4.4	74.4	20.26667
Mass (mg):	75.9	44	28.4	16.5	10.2	10.3	9.2	6.7	5.3	7.3	9.4	78.7	25.15833
Average density (gr/cm3)	1.14	1.23	1.32	1.41	1.50	1.58	1.70	1.76	1.89	1.97	NA	1.06	1.24