CHAPTER THREE – METHODS AND MATERIALS

Two coalfields were chosen to be evaluated and specific seams with in each, namely the No. 4 Seam in the Witbank coalfield and the Upper Ecca Coal Zone in the Waterberg. These coalfields were chosen as they are of great significance for the thermal coal industry and the low grade export coal market. The Free State Coalfield was later added to provide a comparison between the currently researched coals and a high ash content coal from a different coalfield. This is to establish whether the outcomes of the objectives in Section 1.1 could be applied more widely. The samples from the various sources were homogenised to provide composite samples representative of the seams and coalfields from which they came:

	No. of Samples			
Samples	**Witbank No. 4 Seam**	**Waterberg Upper Ecca**	**Free State Top Seam**	**Analysis**
Borehole samples	5	10		washability
Large borehole diameter	2	2		sized washability
ROM Samples	2		2	sizing, sized washability
Plant Product and Discard Samples	2 each			washability
Total No. of Samples	13	12	2	

*2 x product samples and 2 x discard samples each

In this chapter the geological (borehole) samples used in this research are described in Section 3.1.1 and 3.1.2, and the samples taken in the beneficiation plants are described in Section 3.2. Sampling was done by a) Belt-cuts on various sections of the feed conveyor, b) Automatic sampling or c) Falling stream sampling. The ROM sampling was done from conveyor belts as close as possible to the 'source', or in other words, the shaft or pit to establish true reflections of coal feed to plant from each section and to minimize the affect size segregation might have. ISO 13009 sampling was adhered to throughout, especially in an instance where large, representative and accurate samples had to be taken with the vast array of analysis. The segregation of coal particles had to be accounted for and controlled throughout the sampling period, minimising the effect that segregation might have. Note that the

Waterberg samples were comprised of both borehole and large borehole diameter samples, but not beneficiation plant or ROM samples.

In summary, the scope of the project involved:

1. An in-depth geological study of the Witbank (4 seam) and Waterberg (Upper Ecca) coalfields. Especially borehole grade construction evaluations.
2. The sampling of ROM coal was done mainly in operations in the Witbank area (No. 4 seam). An additional Free State (Vereeniging) ROM sample was taken to serve as a high ash content reference sample for modelling purposes.
3. The collection of beneficiation plant samples including product and residue samples that were taken at existing beneficiation plants at various operations. These samples were taken primarily with automatic samplers.
4. The screening of samples. Subsequently floats and sinks analysis was done at each of the identified size fractions (see Table 11).
5. The storage and preparation of samples for various analyses.
6. A full set of the analysis to be done is given in Table 11, using composite and washed samples as an example.

Table 11: *A summary table of single bulk sample analyses for composite and beneficiated samples.*

		Relative Density (RD) Fraction											
	Unit	Composite	1.30	1.40	1.50	1.60	1.70	1.80	1.90	2.00	2.10	2.2	Sinks
Proximate Analysis													
Ash	%	X	X	X	X	X	X	X	X	X	X	X	X
Volatiles	%	X	X	X	X	X	X	X	X	X	X	X	X
Inherent Moisture	%	X	X	X	X	X	X	X	X	X	X	X	X
Fixed (by difference)	%	X	X	X	X	X	X	X	X	X	X	X	X
Calorific Value	MJ/kg (ad)	X	X	X	X	X	X	X	X	X	X	X	X
Mineralogical Analyses													
QXRD	Quantity	X	X	X	X	X	X	X	X	X	X	X	X
QEMSEM	Quantity & Liberation	X	X	X	X	X	X	X	X	X	X	X	X
Petrographic Analysis	ReV	X											
Elemental Analysis													
Ultimate Analysis		X	X	X	X	X	X	X	X	X	X	X	X
Sulphur (Organic, Pyretic)		X	X	X	X	X	X	X	X	X	X	X	X
Trace Element		X											
Size Fractions													
Witbank No. 4 Seam													
-150+50mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-50+12mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-12+0.5mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-0.5mm	%	X	X	X	X	X	X	X	X	X	X	X	X
Waterberg Upper Ecca													
-50+32mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-32+25mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-25+16mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-16+12.5mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-12.5+6mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-6+3mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-3+1mm	%	X	X	X	X	X	X	X	X	X	X	X	X
-1mm	%	X	X	X	X	X	X	X	X	X	X	X	X

The chapter further contains an expansion of the physical analysis methods (Section 3.4.1, 3.4.2) used to obtain the washability and froth flotation fractionated samples, with a description of the conventional to advanced analytical methods employed for coal characterisation purposes (Section 3.4.3-3.4.6). In Section 3.5 the approach for the techno-economic modelling approach is described and referred.

Materials and facilities required

1. The materials required for the floats and sinks analysis, proximate and ultimate analyses of the samples were available at the respective colliery laboratories.
2. Trace element content were analysed at *UIS (Unique Innovative Scientific)* Laboratories in Centurion, Pretoria.
3. The QEMSEM analysis was conducted at *ESKOM R & D* with Dr. Chris van Alphen.
4. The QXRD (Quantitative X-Ray Diffraction) samples were analysed through *Analytical Consulting cc* with Dr. Sabine Verryn.
5. The petrographic analysis was done through Petrographics SA with Dr. Vivian du Cann.

Data Analysis

A wide range of data had to be analysed. The range and cohesion of the data were critical as all the data required strengthened the base case. The data collected were divided into:

1. **Geological data analysis:** Analysing borehole samples collected at the time of sampling.
2. **Fieldwork data analysis:** This included the recording of the conditions during sampling which could have influenced the data generated. In identifying possible errors, anomalies occurring during sampling had to be identified.
3. **Coal Analyses data analysis:** From the sample analysis summary as illustrated in Table 11, standard representation methods of washability characteristics evaluation were employed.

4. **Environmental data analysis:** The included the (i) prediction of carbon dioxide emissions data due to different species combinations and (ii) acidification properties (based on sulphur content) of different coals.
5. **Financial and economic data analysis:** This involved the analysis of all economic data relevant in doing the techno-economic study.
6. **Statistical data analysis:** This involved geological and test work data collected that were statistically evaluated to identify repeatability and trends. In order to obtain good repeatability and acceptable confidence in the data it was critical that a sufficient number of samples were collected and analysed to improve the statistical evaluation.

3.1 Geological Background

The sections below provide the geological and stratigraphy background of the coalfields under investigation.

3.1.1 Witbank Coalfield No. 4 Seam

Due to coal being heterogeneous in nature in terms of both the organic and inorganic composition, the concentrations of trace elements have been found to vary within the coal seam associated significantly with the sedimentology. Coal seams in the Northern Hemisphere (Carboniferous Coals) are known to be overlain by strata of marine origin, and therefore tend to have higher pyritic concentrations of sulphur and, in general, higher trace element concentrations than those associated with non-marine sediments found mostly in South Africa (Wagner, 2005). The elemental-geological relationships have therefore become important in establishing the sulphur, and hence trace element associations. Earlier investigations by Cairncross (1990) proved that coal-bearing Vryheid Formation (Karoo Sequence) in the Witbank Coalfield was associated with both marine and non-marine depositional events. The trace element distribution in this coalfield is therefore likely to vary significantly and therefore needs to be investigated.

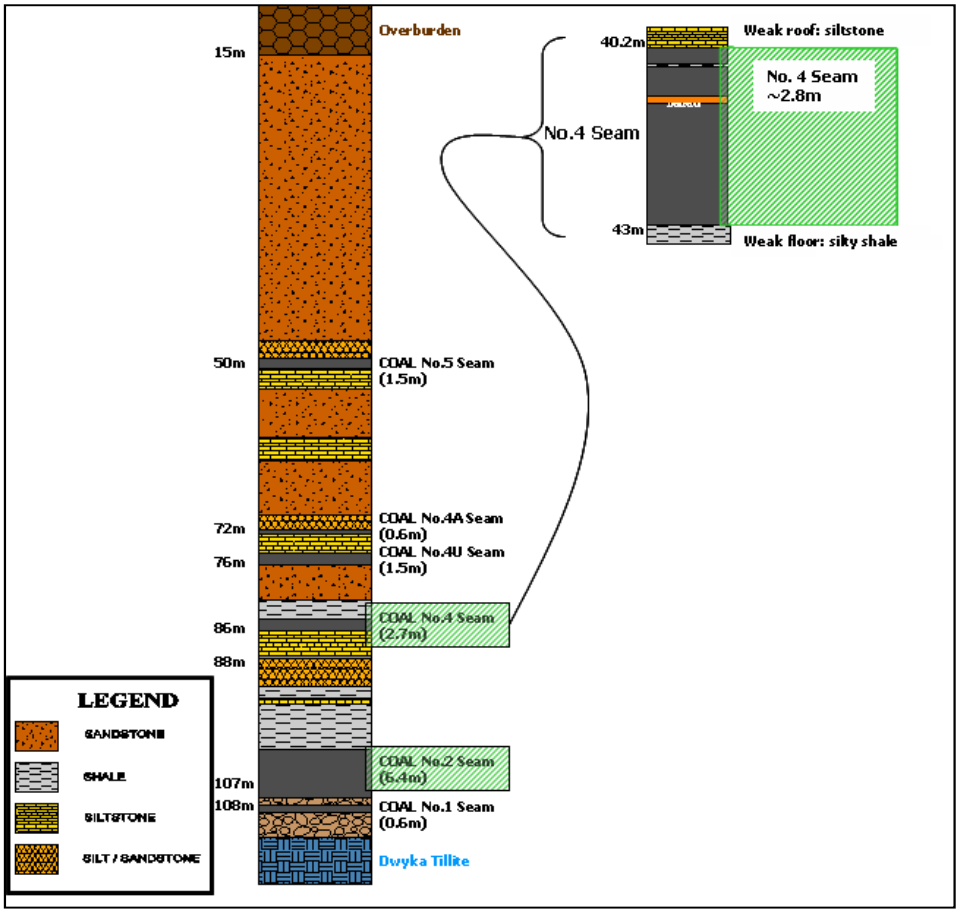

Figure 48: *Witbank coalfield No. 4 seam stratigraphy (Mokele, 2008).*

At the mine where the samples were taken the particular section of the No. 4 Seam ranges in thickness from 2.2 m to 3.3 m, averaging 2.8 m (see Figure 48). In the Witbank coalfield No.4 seam splits exist above the main seam, called the Upper 4 and Upper 4A seams. These are not mined and are not of economic importance. In this instance, an in-seam limonite rich mudstone parting of up to 0,2m in thickness typically separates a lower better quality zone of mainly dull lustrous coal with an average thickness of 1.9 m from an upper poorer quality zone of mixed dull and bright coal averaging 0.7m.

3.1.2 Waterberg Upper Ecca

The description of the Waterberg Upper Ecca, Grootegeluk Formation stratigraphy consists of the following:

The major coal bearing horizons of the Ecca Group are known as Volksrust Formation (55m of intercalated mudstones and coal) and the Vryheid Formation (three major discrete seams of approximately 3m, 9m and 4m, respectively). The most significant difference to the main Karoo Basin is the fact that the Volksrust Formation is carbonaceous with this formation being represented by intercalated carbonaceous shales and coal, (Faure *et al.* 1996[a], and 1996[b]).

The Upper Ecca Group consists of an 80m – 100m thick coal mudstone succession. The coal mudstone succession consists of alternating coal and mudstone layers.The mudtsone layers varying in thickness from a few centimetres up to a few metres.

The lower part of the Upper Ecca Group, approximately 25 meters, is named the Transitional Zone. This succession consists mainly of dull, lustrous coal bands alternating with dark grey, carbonaceous mudstone layers. In many instances, these lithologies grade into one another.

The top 65m to 70m of the Upper Ecca is named the Prime Zone seams. This area consists of bright coal and carbonaceous mudstone layers varying in thickness from a few centimetres to several metres. The contacts between these lithologies are usually sharp. A thin (less than 30cm thick), siltstone or silty mudstone is developed at the contact between the upper and lower portions of the succession. The complete coal mudstone succession has been divided into Prime Lower, Prime Middle and Prime Upper zones. The zones are subdivided into distinctly formed coal layers.

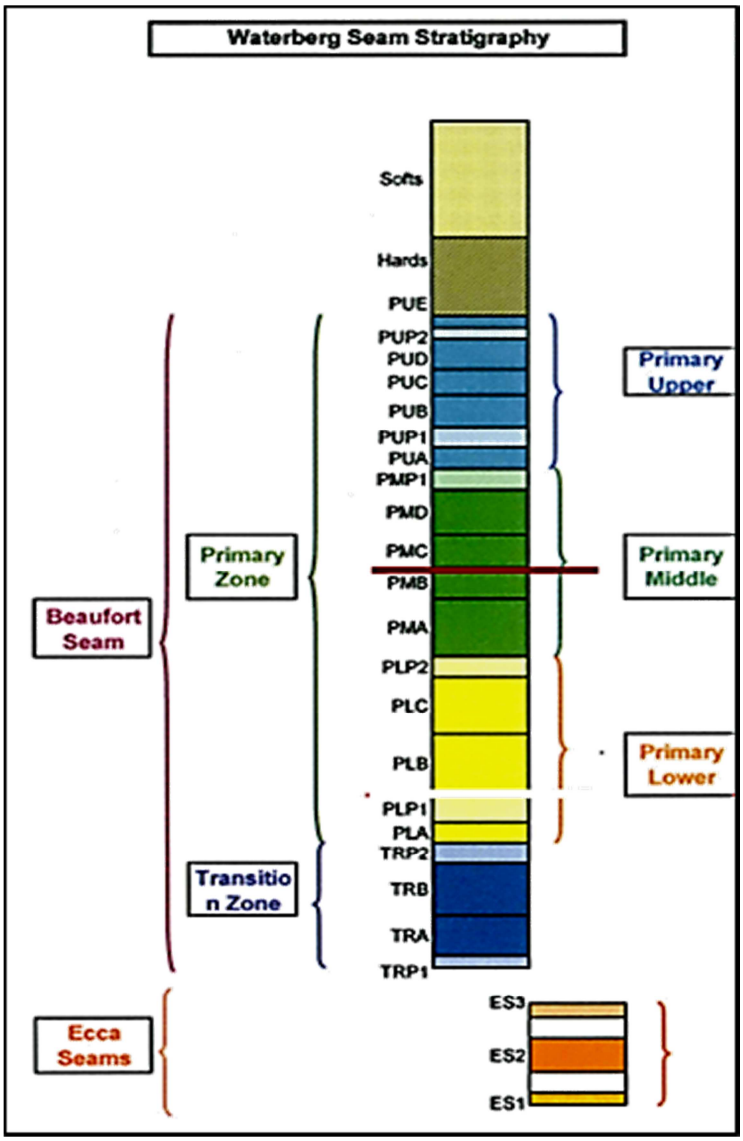

Figure 49: *Waterberg Upper Ecca Stratigraphy (Dorlant, 2012).*

The Swartrant Formation, Middle Ecca Group, contains three seams separated by sandstone and siltstone, namely the ES1, ES2 and ES3. The parting between the ES1 and the ES2 is on average 6.7m thick, with a minimum thickness of 2.3m and a maximum thickness of 20.8m. The parting between the ES2 and the ES3 is on average 5.8m thick, with a minimum thickness of 1.9m and a maximum thickness of 25.2m. The parting (sedimentary succession) between the ES3 and the bottom of the thick interbedded succession, the TRP1 is on average 23.6m thick, with a minimum thickness of 3.7m, and a maximum thickness of 70.2m. There is therefore enough competent material between the Middle Ecca seams so that almost all of it was expected to be extractable by underground mining.

The ES1 is not split into subseams. Where there is distinctive quality zoning in the geophysical logs, the ES2 may be split up into the ES2L (lower), ES2S (select) and ES2U (upper). Samples are then taken according to subseams.

The ES1, ES2 and ES3 are underlain and overlain by sandstone, indicating that strong roof and floor conditions may be expected during mining

The Upper Ecca Group thick interbedded succession consists of 14 subseams and 7 intraseam partings that are all highly correlatable throughout the Waterberg project area using geophysical logs. The quality of the subseams varies throughout (Table XX in the appendix). The best qualities are at the top of the thick interbedded succession. There is an increase in thickness of the Upper Ecca succession from south to north across the Ellisras basin (Faure,1996), as well as an improvement in the quality of the transitional zone. At Grootegeluk Colliery, the coal of the transitional zone contains similar qualities to that of the zones above (Snyman, 1994). A detailed description of the seams, sub-seams and partings is given in Table XX in the appendix.

3.2 Background to Sampling Procedure of Witbank Coalfield No. 4 Seam

Due to coal being heterogeneous, the representative evaluation thereof is extremely difficult. Different coals also have distinct inherent properties even though they may be from the same seam. The presence of extraneous and inherent mineral matter could also vary to such an extent vertically and laterally that variation occurs in between coal bands within the seam. As a result variation may occur from particle to particle when mined. In the case of inherent mineral matter, its presence even in beneficiated coal can cause huge quality variations. In order to understand that not only mineral content, but also macerals and trace element content can affect quality parameters, a broader definition to describe coal has to be applied.

The nature of peat formation and the degree of coalification have a great influence on the physical and chemical properties of a particular coal. These inherent property variances affect the end qualities and assessment of a specific coal. The

heterogeneity caused by these factors alone creates the need for very thorough sampling procedures and reliance on vigorous statistical approaches to coal sampling. In the case of trace element content evaluation, these factors cannot be over emphasised as small influences due to heterogeneous sampling or contamination would have significant consequences. The samples need to be isolated and quarantined properly and analysis and preparation needs to be swift and thorough. These approaches were adopted in the course of sampling and handling of coal during this research.

3.2.1 Witbank No. 4 Seam Sampling Procedures

Two sets of 4 seam ROM were collected (from a mine that produces steam coal from the Witbank coalfield No. 4 seam) taken over a period of 24 hours with the aid of an automated mechanical sampler as per Figure 50. The samples were taken in accordance with SANS ISO 13909-2 Standard - 'Sampling of Moving Streams' for general analysis at a top-size of 150 mm.

Figure 50: *Illustrating the conveyor falling stream mechanical sampler and mechanism.*

It was critical that sufficient sample was taken to satisfy statistical requirements. The calculated mass required for general analysis at a top size of 150mm is 2600 kg. The incremental mass of the sample is in the order of 58 kg; with increments taken every 20 minutes after a period of 24 hours the actual sampled bulk mass was over 4176 kg.

Figure 51: *Illustrating the sampling point from sampler discharge.*

Samples of froth flotation plant feed, product and tailings were also collected by means of mechanical samplers, see Figure 52. These samples were taken in accordance with SANS ISO 20904 'Sampling of Hard Coal Slurry Streams' . The feed samples were used to do bench scale flotation tests. The mass of the samples totalled 51.2 kilograms.

Figure 52: *An illustration of the flotation plant and mechanical slurry stream samplers with which the samples were taken. The plant consists of three modules of three sets of primary and secondary cells each.*

Figure 52 illustrates the method by which the slurry stream samples were taken in order to ensure that there were no outside influences. The plant was running at a steady state before taking the sample. The minimal volume required per increment according to ISO 20904 is 300 mℓ; the current sampler cutter with a width of 10 cm gives on average 500 mℓ.

3.2.2 Borehole samples for borehole correlation liberation data

The samples were collected from the Bethal-Middelburg region and comprised of both borehole samples and large borehole diameter samples. The borehole spacing in the exploration area was determined using the boreholes spacing method of SANS as a guideline. Coal samples are routinely crushed to minus 12.5 mm and washed at several densities to enable evaluation of a full range of products from low relative densities (RDs) to high RDs i.e. 1.3 to 2.0.The borehole sample masses as a collective bulk sample were in excess of 2.5 tons.

3.2.3 Sample preparation

The ROM samples were mixed several times, flattened and cone and quartered to produce a single set of samples for washability analysis. Extreme care was taken to isolate the samples from any possible source of contamination. The samples were transported to the analytical laboratory within 2 hours. The samples were stored at the analytical laboratory in a controlled environment indoors to retain the condition of the coal as sampled. Figure 53 displays the procedure followed during the preparation of the samples for washability, moisture, proximate and ultimate analysis.

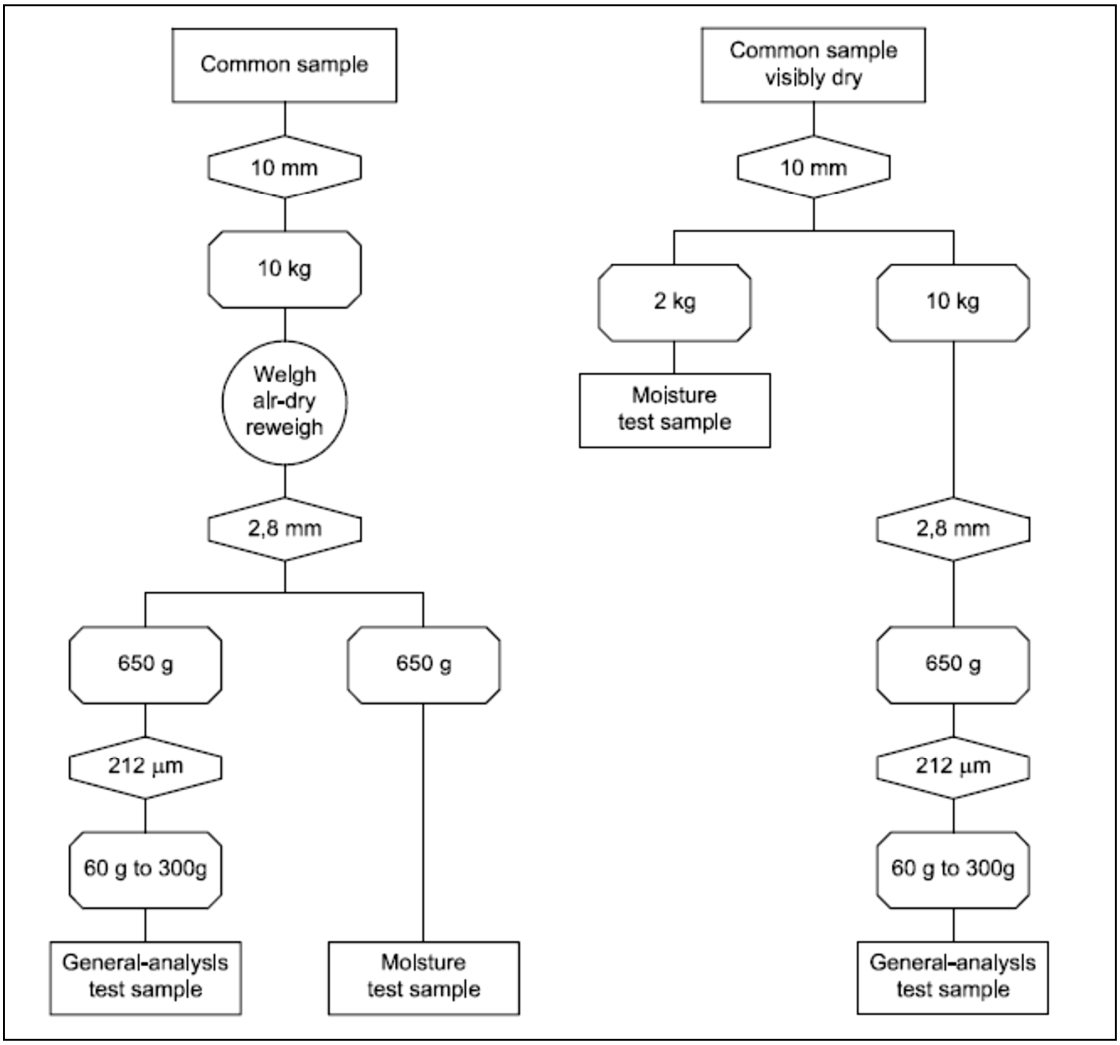

Figure 53: *Flow diagram for division of test samples for analysis from SANS ISO 13909-4.*

3.3 Sampling of Waterberg Upper Ecca

The samples collected from the Waterberg (North) comprised of both borehole samples and large borehole diameter samples. The borehole spacing in the exploration area was determined using the boreholes spacing method of SANS as a guideline. Coal samples are routinely crushed to minus 12.5 mm and washed at several densities to enable evaluation of a full range of products from low relative densities (RDs) to high RDs i.e. 1.3 to 2.0. The borehole sample masses as a collective bulk sample from the Waterberg were in excess of 1.4 tons.

3.4 Analysis

A general summary of the analysis undertaken is shown in Figure 54. Note the samples used for QEMSCAN analysis were not pulverised, but were later pulverised for XRD analysis. The analysis in Figure illustrates the analysis for borehole samples only. In the case large borehole diameter samples, the samples were screened in different size fractions on which washability analysis were carried out.

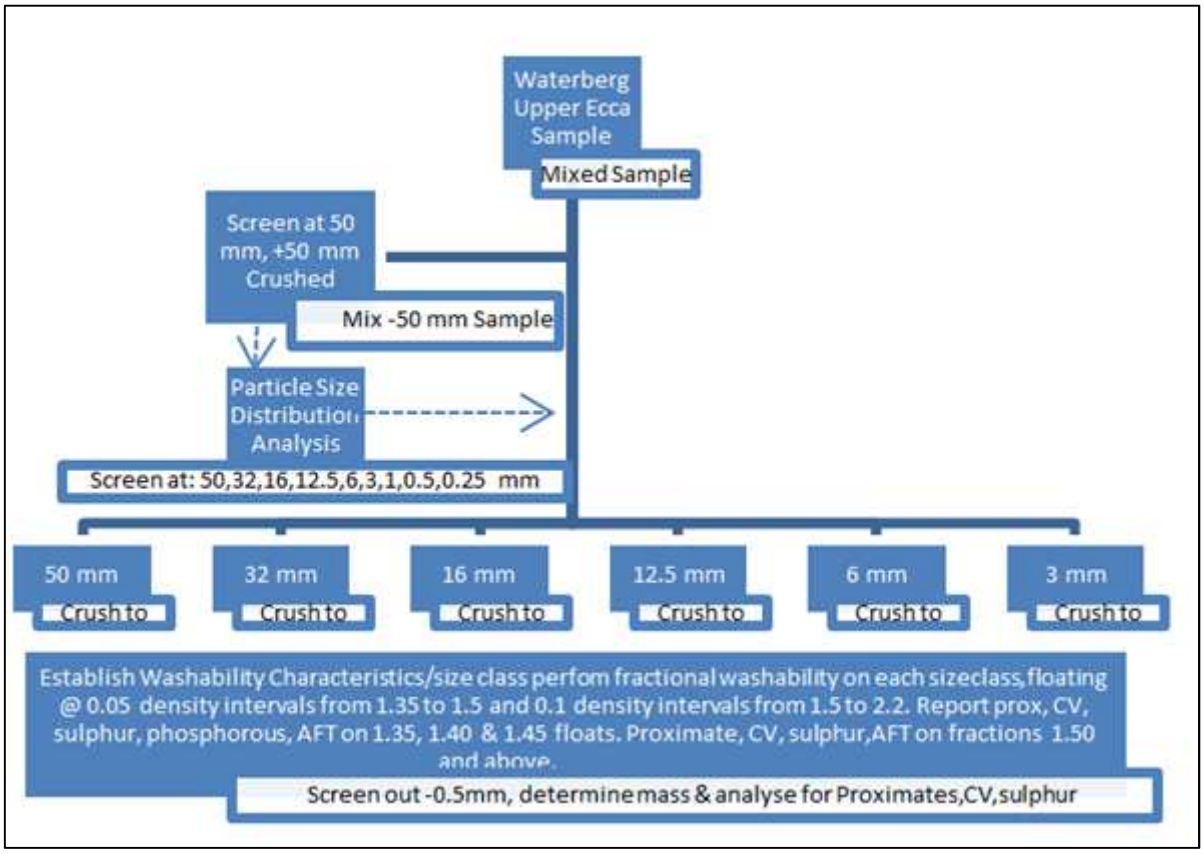

Figure 54: *Illustrating sample splitting and crushing/liberation to facilitate preparation for various analyses.*

3.4.1 Washability analysis

A conventional float and sink analysis using Zinc Chloride (ZnCl) as the dense medium was carried out at the analytical laboratory. The dense medium separation characteristics of bulk samples are determined by float and sink analysis also referred to as washability analysis. The SGs used varied from 1.30RD to 2.00 RD.

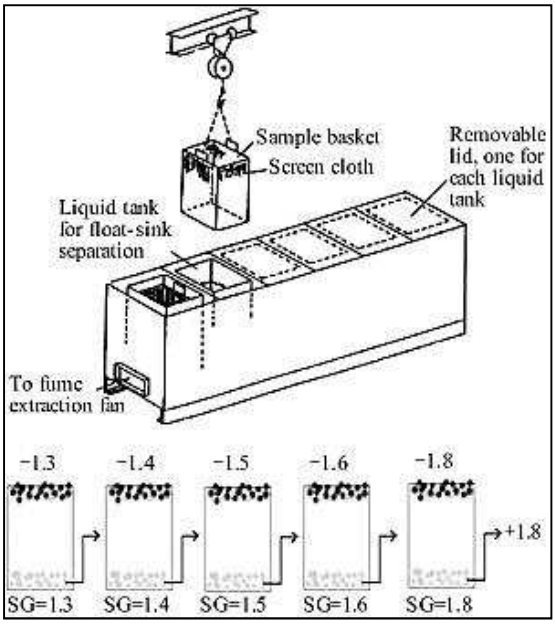

Figure 55: *Washability equipment and testing method (from Habetinejad et al. 2012)*

The total sample including the fines fraction (-0.5 mm), were used in the washability tests. The separate density fractions were then split to allow different sets of analyses of the samples. The complete set of analysis as described in Table 11 were determined for each density fraction.

Figure 56: *An illustration of the dense medium washing baths used at ALS (previously ACCL).*

The potential effect on the accuracy of the trace element analysis due to contamination by any residual ZnCl particles was taken into account. Zinc and

chloride would have the greatest effect, whilst other trace elements would only be present in parts per billion.

3.4.2 Floatability analysis

For the bench scale flotation tests a conventional Denver froth flotation cell was used. Each test consisted of 200 grams of minus 200 µm coal and 3 litres of water.

Figure 57: *An illustration of the Denver flotation cell utilised to do the bench scale flotation tests.*

This slurry mixture was preconditioned without air and reagent for a period of 60 seconds. Figure 57 and 58 illustrates the bench scale flotation cell used in the analysis.

The reagents used in the test work were a combination of 80% collector (mainly kerosene) and 20 % frother (glycol and emulsifiers), 0.3mℓ reagent was added to the slurry and a further conditioning time of 30 seconds was allowed. Upon the addition of air, a further 2.5 minutes were allowed for flotation with scraping done to remove the froth every 20 seconds. The full set of analysis as per Table 11 were carried out at the analytical laboratory on each of the flotation fractions feed, tailings and product. The trace element content of the reagent used could have played a role in the final trace element concentration in the concentrate. This was neglected based on the low dosage of the reagent and the low trace element content in the reagent. The influence was considered to be minimal on the sample trace element concentration, and based on calculation, cannot exceed an impact more than in the order of parts per billion (ppb).

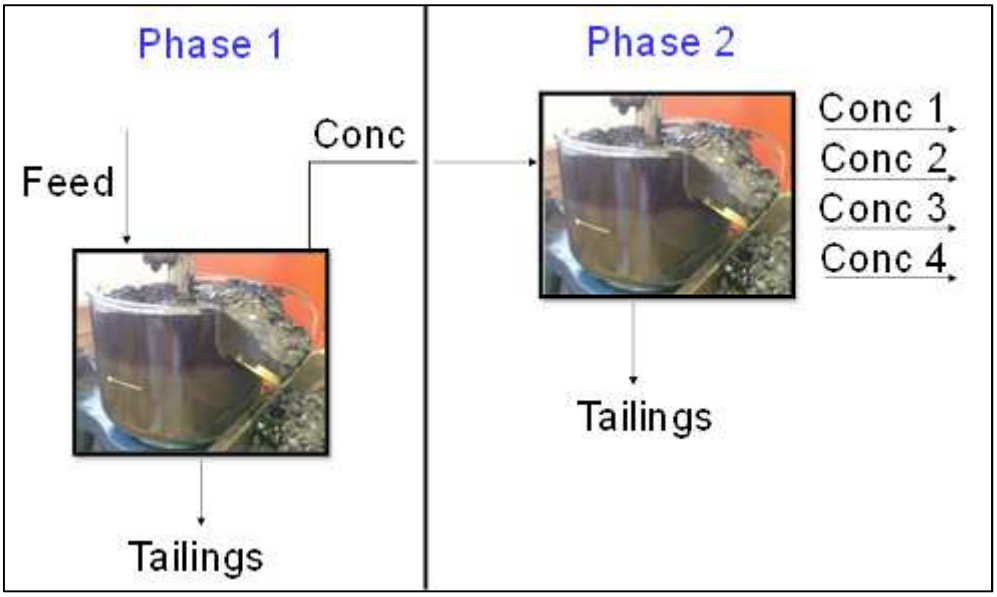

Figure 58: *Experimental procedure during release analysis*

3.4.3 Particle Size Distribution (PSD) Analysis

Particle Size Distribution (PSD) analysis were carried out to (a) determine a size distribution for process design purposes and (b) to do size by size washability for liberation analysis purposes. Screening was conducted at a local laboratory and on the mine. Dry screening of some samples was conducted at the mine laboratory through a conventional vibrating shaker. Dry and wet screening of the large samples were outsourced to the local laboratory. The various screening fractions identified for analysis were based on the conventional sieve series.

3.4.4 Proximate and Calorific Value Analysis

The proximate analyses are a combination of the proportions of ash, inherent moisture, and volatile matter and by difference fixed carbon by percentage. The samples were crushed to 3mm, homogenised and then pulverised down to 212µm for proximate analysis and calorific value analysis.

3.4.5 Mineral Matter and Liberation Analysis

The XRD (X-Ray Diffraction) analysis was outsourced to Dr. Verryn for the identification and quantification of mineral species. The method of Quantitive Evaluation of Materials by Scanning Electron Microscopy (QEM SEM) or QEMSCAN was undertaken at a well-known research & development establishment. Figure 59 illustrates the QEMSEM microscope. The method used by Dr. van Alphen for the QEMSEM analysis is consistent with the method describe in the thesis (van Alphen, 2005).

Figure 59: *The QEMSCAN microscope used for the minerogical investigations.*

3.4.6 Trace Element and Sulphur Form Analysis

The trace element analysis, and total sulphur and forms of sulphur (organic, pyritic and sulphur from sulphates) contents were done at an advanced analytical services laboratory namely UIS (Unique Innovative Scientific) Laboratory. The trace element analysis comprised of microwave digestion followed by Inductively Coupled Plasma Mass Spectroscopy.

Figure 60: *Illustration of the ICP MS spectrometer at UIS analytical services.*

Figure 61: *Illustrating the microwave digestion experimental setup.*

In the microwave digestion test a mixture of nitric, hydrochloric and hydrofluoric acid were used. The samples were heated to approximately 210°C at 20 atm pressure. After a first stage of digestion a second stage digestion was performed to ensure that all the material was dissolved. Stock solutions were used for the determination of a variety of trace elements in the digests by ICP-MS as per Figure 61.

3.4.7 Petrographic Analysis

Petrographic analysis was performed by Petrographics SA (Du Cann, 2011). The sample preparation and analyses used in the petrographic assessment was done in accordance with SANS ISO Standard 7404: parts 1 to 5. Maceral analysis (to determine the petrographic compositions of the coals) and reflectance measurements (to determine the rank) were carried out. The petrographic analyses of each density fraction comprised of vitrinite reflectance for the determination of rank, organic composition (macerals and microlithotypes), mineral groups and condition analysis. The equipment is illustrated in Figure 62. The detailed petrographic analysis report is given in Appendix F.

Figure 62: *Illustration of the vitrinite reflectance and vitrinite reflectance measurement configuration respectively.*

3.5 Techno-economic, Mineralogical and Process modelling methods

The method for mineralogical modelling is described in Section 2.2 and Section 6. The approach used for process modelling is described in Section 8, with background parameters for different equipment described in Section 2.3. The cut point density, epm's and other modelling parameters are given in Appendix G. The method for economic analyses is presented in Section 10.3 and in greater detail in Section 10.5.1. The reason for not including these methodologies in this section is in the interest of non-duplication because the methods and application require to be integrated in the chapters referred to above.