# Chapter 4: Coal characterisation

## 4.1. Introduction

In this chapter, the origin of the coal, as well as the experimental procedures applied for the characterisation of the coal, is discussed. The origin of the coal sample is briefly discussed in Section 4.2. In Section 4.3 the methods used for the preparation of coal samples for characterisation purposes, are described. A description of the procedures used for the general characterisation of the coal sample is given in Section 4.4. Section 4.5 consists of a detailed discussion regarding the results obtained from the coal characterisation analyses. Section 4.6 contains a brief results summary of the coal characterisation analyses.

## 4.2. Origin of the coal sample

The coal used is a beneficiated coal from the Highveld Coalfield in Mpumalanga. The coal is a washed steam coal with a low-ash content (~10%) and density less than 1 400 kg/m$^3$, which is specifically beneficiated for the export market (Mining Technology, 1997).

The export product is used for this study, since a low-ash content coal is favourable for catalytic gasification studies (Suzuki *et al*., 1984) . The raw coal is transported from the mines to the coal beneficiation site, where the coal is crushed and screened, and stockpiled before further processing. The stockpile is used to feed the beneficiation plant, where the coarser coal is washed in dense-medium cyclones to produce high quality export product (Mining Technology, 1997). The export coal was supplied by SGS laboratories (SGS South Africa (Pty) Ltd) in Secunda. A bulk coal sample of export coal was wet-screened according to specified size fractions, before the coal was air-dried and sealed in plastic containers. Table 4.1 indicates the various size fractions specified for the wet-screening of the export coal sample.

**Table 4.1: Size fractions of export coal sample.**

| Size fractions (mm) | Mass (kg) |
|---|---|
| -5 mm | 75 kg |
| +5 mm – 25 mm | 75 kg |
| +25 mm – 40 mm | 75 kg |
| +40 mm | 75 kg |
| **Total** | **300 kg** |

As seen in Table 4.1, the various size fractions amount to a total of 300 kg of coal. The specific size fractions were selected according to the particle sizes (large particles) which will be used for this investigation.

## 4.3. Sample preparation

The cone-and-quarter method was used to obtain a representative sample from the bulk coal sample supplied by SGS laboratories. The cone-and-quarter method is a sampling method used to obtain a more manageable, representative sample from a bulk coal sample (Allen, 1996; Allen and Khan, 1970). Firstly, the 75 kg coal sample fraction is emptied onto a flat surface and thoroughly mixed, after which it is formed into a cone-shaped pile as seen in Figure 4.1.

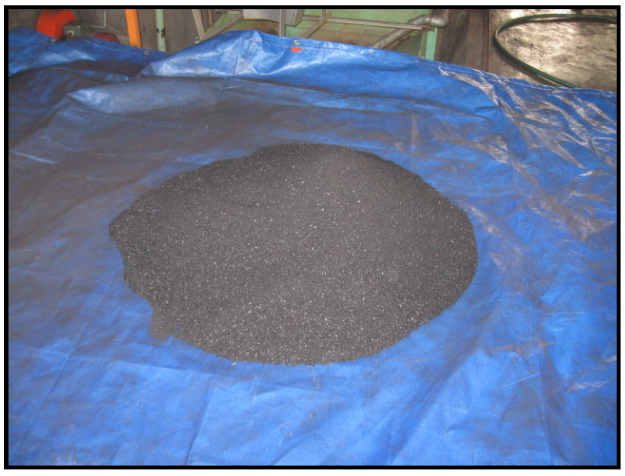

**Figure 4.1: Bulk coal sample in a cone-shaped pile**

The bulk coal sample, as seen in Figure 4.1, is then divided into four equal volumes using a spade. Figure 4.2 illustrates the four cone-shaped heaps.

**Figure 4.2: Four cone-shaped piles of equal volume**

Two opposite portions are mixed and the whole process is repeated until one sixteenth of the original bulk coal sample remains. This indicates that the entire cone-and-quarter method should be repeated no more than four times (Allen, 1996; Allen and Khan, 1970). Once one sixteenth of the original sample is obtained, the sample is sent through a spinning riffler, as seen in Figure 4.3. A rotary splitter is used to divide the sample obtained from cone-and-quartering into smaller, equal sized portions, while still ensuring that the sample is representative of the original coal sample.

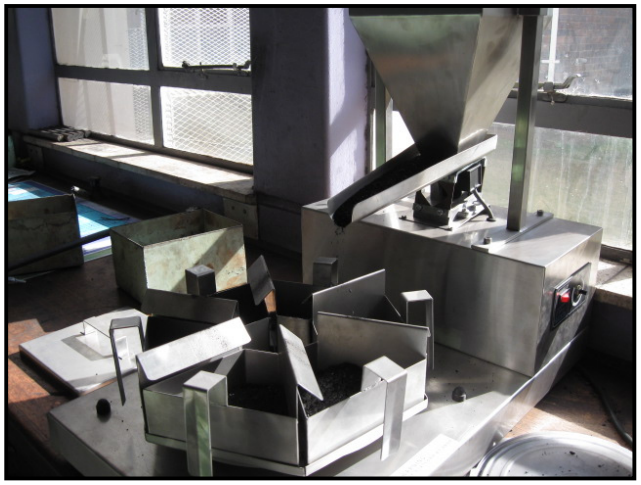

**Figure 4.3: Rotary Splitter**

The coal sample is placed in the hopper, where it is transferred to a series of rotating containers by means of a vibrating chute. With a constant rotating speed, as well as a constant feed flow rate, the containers are filled uniformly (Allen, 1996; Allen and Khan, 1970). The containers are filled until six 500g samples are obtained. Each 500 g coal

sample is stored in an airtight container, under a nitrogen atmosphere, until it is sent for coal characterisation analyses.

## 4.4. Coal characterisation procedures

The characterisation of coal can be divided into three separate categories, namely chemical and mineralogical analysis, petrographic analysis and structural analysis. For the purpose of this study, a general characterisation of the coal, is conducted, which only involves a chemical analysis and a mineral analysis (ash analysis).

### 4.4.1. Chemical analysis

A chemical analysis includes a proximate analysis and an ultimate analysis, as well as the determination of the gross calorific value of the coal sample. Advanced Coal Technology (Pty) Ltd conducted all the coal characterisation analyses (chemical analysis) of the coal sample. Table 4.2 provides a summary of the analytical methods used for the chemical and mineral analysis, along with the ISO standard for each method.

**Table 4.2: Coal characterisation procedures**

| Analysis | Procedure / Method |
|---|---|
| Sample preparation | SANS 18283:2007 / ISO 18283:2006 |
| Proximate analysis - Moisture content (wt.%) | SANS 5925:2007 |
| Proximate analysis - Ash content (wt.%) | SABS ISO 1171:1997 |
| Proximate analysis - Volatile matter content (wt.%) | SABS ISO 562:1998 |
| Proximate analysis - Fixed carbon content (wt.%) | By difference |
| Ultimate analysis | ISO 12902 |
| Ash composition | ASTM D4326 XRF |
| Gross calorific value (MJ/kg) | SABS ISO 1928:1995 |
| Total sulphur (wt.%) | ISO 19579 |

As seen in Table 4.2, the proximate analysis is conducted in order to determine the moisture content, ash content, volatile matter content and fixed carbon content (by difference) of the coal sample, and is reported as a percentage of the weight of the coal sample used. An ultimate analysis is an elemental analysis, which determines the weight % of the carbon (C), hydrogen (H) and nitrogen (N) and sulphur (S) present in the coal. The oxygen (O) is calculated by difference from the results obtained for the ultimate analysis. The gross

calorific value (GCV) was determined by using a bomb calorimeter (SABS ISO 1928:1995). The total sulphur content (by weight %) of the coal sample was determined with a LECO (S144) analyser via infrared (IR) spectrometry (ISO 19579).

### 4.4.2. Mineral analysis

The mineral analysis of a coal sample involves the analysis of the incombustible matter, i.e. the ash, in order to determine the mineral composition of the sample. XRF analysis is a non-destructive elemental analysis which can qualitatively and quantitatively measure certain periodic table elements present in the ash sample. XRF analysis was conducted by UIS Analytical Services. The specific standard method, according to which the XRF analysis was performed, is shown in Table 4.2. A brief description of the procedure used by UIS Analytical Services for XRF analysis, follows:

Step 1: Pulverisation - ash sample is thoroughly mixed to ensure the homogeneity of the sample.

Step 2: Ignition - ash sample is ignited at 815 °C for a period of 1 hour, to ensure that the ash sample is in the same state as directly after the ashing process.

Step 3: Weighing of sample – 1 g of the ash sample is weighed, to which 1 g of ammonium nitrate (oxidising agent) and 9 g of flux (66 % Lithium Tetraborate: 34 % Lithium Metaborate) are added.

Step 4: Sample (11 g) is transferred into a platinum (Pt) crucible. The sample is fused on a semi-automated fusion machine at approximately 1000 °C. The molten sample is cast into a Pt mould. Once the sample cools, it forms a glass disk or bead, which is analysed on an XRF instrument. The instrument used for XRF is a Thermo Fischer analyser, which is calibrated for 15 elements using various certified standards from national and international origin (Schoeman, 2010).

## 4.5. Results and discussion

The results obtained from the chemical analysis and the mineral analysis of the coal sample are presented and discussed in this section.

### 4.5.1. Chemical analysis of coal

The results obtained for the chemical analysis of the coal sample are provided in Table 4.3. The results for the chemical analysis include those for the proximate analysis and the ultimate analysis, as well as the total sulphur content and the gross calorific value. All percentage values given in Table 4.3 are given as weight percentage (wt. %).

**Table 4.3: Chemical analysis of coal sample**

| Characterisation analysis | Coal sample (air-dried basis)* | Coal sample (d.a.f)* |
|---|---|---|
| Proximate analysis | | |
| Inherent moisture content | 5.0 | - |
| Ash content | 12.6 | - |
| Volatile matter | 27.0 | 32.8 |
| Fixed carbon (by difference) | 55.4 | 67.2 |
| Ultimate analysis | | |
| Carbon content | 67.1 | 81.4 |
| Hydrogen content | 4.2 | 5.1 |
| Nitrogen content | 1.7 | 2.1 |
| Oxygen content (by difference) | 8.8 | 10.7 |
| Total sulphur | 0.68 | 0.82 |
| Gross calorific value (MJ/kg) | 26.6 | - |

(* - all values are given as wt.% unless indicated otherwise)

#### 4.5.1.1. Proximate analysis

During the proximate analysis, the inherent moisture content, ash content and volatile matter content are determined using the standard methods shown in Table 4.2. The fixed carbon content of the coal sample is consequently calculated by subtracting the above-mentioned values from the total mass (100 %) of the coal sample. The values obtained for the proximate analysis are comparable with the values obtained by Pinheiro (1999), for this

specific South African coal. According to Pinheiro (1999), the ash values for South African coals range between 6.8 wt. % and 42.7 wt. % (air-dry basis). As seen in Table 4.3, the ash value of the washed coal sample is 12.6 wt. %, which is a relatively low ash content compared to South African ROM coals. However, according to Lang (1986), an ash content of 12.6 wt. % is advantageous for catalytic steam gasification. The results for the proximate analysis indicate that the coal sample has an ash content of 13.2 wt. % and a volatile matter content of 28.5 wt. %, reported on a dry basis. According to Suzuki (1984), coals with ash content values of 7.2-14.7 dry wt. % and volatile matter content values of 31.1-46.0 dry wt. % gave optimum results when used in catalytic gasification studies.

#### 4.5.1.2. Ultimate analysis

The values (wt. %) for the carbon content, hydrogen content, nitrogen content and total sulphur content are determined using the standard methods indicated in Table 4.2, after which the oxygen content is calculated according to ISO 1170:2008. Equation (11) is the standard equation used for the calculation of the oxygen content on an air-dried basis.

$$\%\,\text{Oxygen} = 100 - (\%\text{Ash} + \%\text{Moisture} + \%\text{Total Sulphur} + \%\text{Carbon} + \%\text{Hydrogen} + \%\text{Nitrogen}) \quad \text{(Equation 4.1)}$$

All values used in (11) are on an air-dried basis. As can be seen from the above equation, the ash content and moisture content, which were determined by proximate analysis, were used to calculate the oxygen content.

The results for the ultimate analysis are shown in Table 4.3, and indicate a high carbon content. Similar results were obtained by Wagner and Hlatshwayo (2005) and Van Niekerk *et al.* (2008), for the ultimate analysis of Highveld coal. It was found that these values are typical for coal originating from the Highveld region (Wagner and Hlatshwayo, 2005; Van Niekerk *et al*., 2008; Pinheiro, 1999).

#### 4.5.1.3. Gross calorific value

As can be seen from the chemical analysis results in Table 4.3, the coal sample has a gross calorific value (GCV) of 26.6 MJ/kg (air-dried basis). The coal sample was graded based on the gross calorific value. With reference to CKS 561-1982, the coal sample is graded as a grade B coal, with a gross calorific value in the range of 26.5 – 27.5 MJ/kg (air dried basis).

### 4.5.2. Mineral analysis

The results obtained from the XRF analysis are presented in Table 4.4. The mineral analysis determines which mineral species are present in the ash sample obtained from the original coal sample. All values reported in Table 4.4 are based on weight percentages (wt. %) on an ash basis.

**Table 4.4: XRF analysis of ash sample**

| Inorganic species | Ash sample (wt. %) |
|---|---|
| $Al_2O_3$ | 29.0 |
| CaO | 7.9 |
| $Cr_2O_3$ | 0.1 |
| $Fe_2O_3$ | 4.3 |
| $K_2O$ | 0.5 |
| MgO | 2.7 |
| MnO | 0.1 |
| $Na_2O$ | 0.7 |
| $P_2O_5$ | 1.5 |
| $SiO_2$ | 45.0 |
| $TiO_2$ | 1.7 |
| $V_2O_5$ | 0.1 |
| $ZrO_2$ | 0.1 |
| Ba | 0.3 |
| Sr | 0.6 |
| $SO_3$ | 2.8 |
| Other | 2.7 |

From the XRF results shown in Table 4.4, it can be seen that the ash of the coal sample consists of significant quantities of $SiO_2$ and $Al_2O_3$, while the CaO and $Fe_2O_3$ species are present in lesser amounts. It can also be seen that the ash sample contains a relatively small amount of the potassium (K) species, in the form of potassium oxide ($K_2O$). The exceptionally low $K_2O$-content of the ash sample is especially advantageous from an impregnation perspective. Since the ash sample, and consequently the raw coal sample, has a low potassium content, the effect of any added potassium catalyst will be easily discernible. According to Pinheiro (1999), the results obtained from the XRF analysis are

similar to the mineral analysis for other washed South African export coals. The results are also comparable with the XRF values obtained by Oberholzer (2009), for Highveld coal.

## 4.6. Summary

A general characterisation of the coal sample, which included a chemical and mineral analysis, was conducted. A summary of the results obtained from the coal characterisation analysis, follows:

The proximate analysis confirmed the coal sample to be a low-ash coal, with an ash content of 12.6 wt.% (air-dried basis). The coal sample was graded as a grade B coal, based on a gross calorific value of 26.6 MJ/kg (air-dried basis). The results acquired from the XRF analysis determined that the ash obtained from the coal sample was rich in $Si_2O_3$ and $Al_2O_3$. The XRF analysis also indicated that the ash sample has a low potassium oxide content of 0.53 wt. %.