A LIBERATION STUDY ON ULTRAFINE SOUTH AFRICAN COALS

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by
IRMTRAUD ERNA BUYS B.Sc.(Hons) (Chemistry) (Cape Town)

Department of Chemical Engineering
University of Cape Town
Rondebosch, 7700
South Africa

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SYNOPSIS

South African coal mines generate large quantities of fines as a result of the increased use of mechanised mining methods. Generally, these fines are discarded. They do however contain relatively large proportions of high grade material, which provides a considerable incentive for their beneficiation. The increasing demand for low-ash and super-low-ash coals is an added price incentive for fines beneficiation.

As coal is a highly heterogeneous substance, it is necessary, in order to optimize beneficiation processes, to have a good understanding of its liberation characteristics. The aim of this thesis is to extend the liberation study of Harris (1987) on Greenside (Witbank No.2 Seam) coal to finer sizes and to investigate the liberation characteristics of two other South African coals, one from the Rietspruit Colliery (Witbank Coalfield) and the other from the Grootegeluk Colliery (Waterberg Coalfield).

Subsamples of each coal were milled to 95 % passing 150, 75 and 45 μm. The unmilled samples and the milled subsamples of each coal were split into +25 μm and -25 μm size fractions. Size analyses were carried out on the -25 μm fractions of all samples and milled subsamples. Each size fraction was then subjected to float and sink analysis, using the new centrifugal method of Harris (1987). In order to verify the separation efficiency of this new method, the results were checked against gravimetric float and sink analysis for the +25 μm material and oil agglomeration for the -25 μm material. Liberation was assessed by means of size and ash analysis, density distributions, washability characteristics and liberation efficiencies.

The size and ash analyses showed that milling preferentially reduced coarse (+25 μm) middlings material to finer sizes (-25 μm). The existing -25 μm material, however, did not become finer on progressive milling. Selective breakage of middlings material was also evident from the density distributions. Low ash material was concentrated in the +25 μm fractions of all three coals.

The density distributions for Greenside and Rietspruit coals showed the characteristic 'middlings hump' (Sanders and Brookes, 1986), which is often associated with poor liberation. Petrographic analysis, however, showed, that both coals contain large quantities of inertinite, which has a relative density between 1,40 and 1,60, and would therefore always be found in the middlings region of the relative density distribution. Grootegeluk contains only very little inertinite and therefore lacks the 'middlings hump'.

The washability study showed that the +25 μm fractions of all coals were better liberated than the -25 μm fractions. However, the washability data for the -25 μm fractions of the Greenside coal especially were unreliable due to exaggerated ash values, especially in the lower relative density regions. Liberation efficiencies, based on the M-curve, were found to be inaccurate.

Hence, a new measure of liberation, based on misplaced maceral and ash material, was proposed. This was done by allocating macerals and mineral matter to different relative density zones and then calculating the amount of misplaced material (based on the R.D. of the respective macerals) in all the zones. According to this measure, the Greenside coal was the most liberated in terms of ash and coal, with Rietspruit second and Grootegeluk last. Milling to 95 % passing 45 μm increased the liberation of the Greenside coal

slightly, that of the Rietspruit significantly, while the Grootegeluk coal remained practically unchanged.

In order to achieve substantial improvement in liberation, much finer grinding is required, possibly using a different type of mill than the one used in this work (rod mill). Going to these finer sizes would make the float and sink method even more inaccurate, and different methods of analysing the separability of the samples (by optical means, or by selective agglomeration) would have to be developed.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Over the last few years the increasing use of mechanised mining methods has resulted in large quantities of fines being generated by the collieries. In most cases, the high ash content of these fines causes them to be discarded. A survey (DMEA, 1987) has shown that South African coal mines discard 3,7 million tons of bituminous slurry annually.

On account of the liberation that takes place with decreasing particle size, however, these fines generally contain relatively large proportions of high grade material. This provides a cost incentive for their beneficiation. Furthermore, in the case of the Witbank coals there is a considerable price incentive for the production of "low-ash coal" (about 7,4 % ash, blend-coking coal) for export and super-low-ash coal (1 to 2 % ash) for direct liquefaction or use in coal-water mixtures.

Recently a comparative study was made of the washability characteristics of coals from Europe, Australia, Botswana, India, Brazil and South Africa (Sanders and Brookes, 1986). It was found that the washability characteristics of the South African coals were indeed improved in the finer sizes. However, the theoretical yields of low ash coal remained poor. The feedstock consisted of +500 μm material, suggesting that in order to obtain better yields of low ash coal, finer particle sizes should be considered.

Subsequently Harris (1987) investigated the liberation characteristics of a typical Witbank No.2 Seam coal, obtained from the Greenside Colliery. A sample of raw coal was milled to 30, 60 and 90 % passing 150 μm, and float and sink analyses were carried out on the milled products. The results were compared with those of a sample of naturally arising fines (thickener underflow) from the same colliery. The liberation efficiencies[1] based on the Mittelwert-curve and the yields of low-ash coal at different particle sizes are given in Table 1 below.

As may be seen, the coal became more liberated on prolonged milling and the theoretical yield of low-ash coal increased markedly, but the overall degree of liberation was still relatively low owing to the presence of high proportions of middling material. Harris (1987) concluded that in order to achieve an appreciable degree of liberation the coal needed to be reduced to a particle size considerably smaller than 150 μm.

The degree of liberation of coal fines, and the proportion of middling material present, are very important factors when considering a beneficiation process for the treatment of these fines. In particular, flotation, which is widely used for the beneficiation of coal fines (and in which there is considerable interest at the moment in South Africa, especially with the advent of column flotation technology) is very sensitive to the presence of middlings. If a coal with a relatively large proportion of middlings material (as is the case with most South African coals) is subjected to flotation, the process cannot differentiate between middlings particles of varying ash contents but similar

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1 A description of how this liberation efficiency is calculated is given in section 2.5.3.

surface properties. A high degree of liberation is therefore required for high organic efficiency.

Table 1

Liberation efficiency, ash content and yield of Low Ash Coal (LAC:7,4 %) of Greenside coal milled to 30, 60 and 90 % -150 μm, and of thickener underflow, by size fraction

Sample	Size Fraction (μm)	Liberation Efficiency[1] (%)	Ash Content (%)	L.A.C. Yield (%)
30% less than 150μm	+150	55,20	20,4	50,6
	-150+ 25	61,74	16,8	65,8
	- 25	60,14	20,8	47,6
	Composite	56,91	20,1	53,2
90 % less than 150μm	+150	67,62	24,1	51,1
	-150+ 25	64,42	19,5	64,3
	- 25	63,06	20,6	49,5
	Composite	64,00	20,2	63,0
Thickener Underflow	+106	59,29	16,0	71,5
	-106+ 25	69,75	19,9	70,3
	- 25	66,69	26,0	41,8
	Composite	64,63	20,6	62,5

from Harris (1987)

In most South African coals the presence of significant proportions of syngenetic mineral matter requires that the particle size be very small indeed before the degree of liberation is substantial and coal of low-ash quality can be produced in reasonable quantities. It is therefore useful to know the degree of liberation of South African coals at

various particle sizes, and whether this may be improved on further size reduction.

A knowledge of liberation characteristics allows one to set goals and targets in plant design (by determining theoretical yield). It also allows one to determine the efficiency of the actual beneficiation process.

Finally, the Coal Research Group in the Department of Chemical Engineering at the University of Cape Town is running a number of flotation and oil agglomeration projects on ultrafine coals (milled to 95 % passing 45 μm). In order to evaluate these results on an absolute basis an independent measure of liberation is needed.

1.2 **AIMS**

The aims of this project were twofold : firstly to extend the liberation study of Harris (1987) on Greenside (Witbank No.2 Seam) coal to even finer sizes; and secondly, to extend the study to two other South African coals. These coals were chosen to be different in several ways from the Greenside coal and were therefore expected to show different liberation patterns on particle size reduction.

The liberation studies were carried out using two fundamentally different processes. Float and sink analysis involves the separation of a sample into a number of fractions according to difference in a bulk property (i.e. density), while oil agglomeration differentiates on the basis of surface characteristics.

In this thesis the origin and major characteristics of South African coals are described briefly in Chapter 2. Liberation theory and various methods that have been

developed to measure coal liberation are described i.e. ways of expressing the results of these measurements by the use of washability curves, M-curves, or the calculation of efficiency indices, are also given. Float and sink analysis, scanning electron microscopy and oil agglomeration are discussed.

In Chapter 3 there is a description of the experimental program undertaken and the experimental techniques and equipment employed. Details of the coal samples used in the testwork are also given. Chapter 4 outlines the result of preliminary work.

The results of the study are presented and evaluated in Chapter 5 and conclusions based on these interpretations are given in Chapter 6.

CHAPTER 2

LITERATURE SURVEY

Coal liberation, compared to the liberation of metalliferous ores, is relatively complicated because coal is an extremely heterogeneous, multi-component substance. In order to gain a better understanding of the liberation characteristics of coal it is important to become familiar with some of the properties of its many constituents.

This chapter begins with a description of the composition of coal and the properties of its constituents. South African coals are described in some detail. There follows a discussion of the theory of liberation and the methods that may be used to measure and assess it. Previous work on the liberation characteristics of South African coals is then reported.

2.1 NATURE OF COAL

2.1.1 Composition

Coal is a heterogeneous mineral consisting of combustible carbonaceous compounds and a variety of inorganic impurities. The carbonaceous constituents or macerals are entities which evolved from different organs or tissues of the original plant material during the course of primary accumulation and the early stages of coalification (maturation). There are three major groups of macerals:

(i) Vitrinite was formed from cell wall material and the cell fillings of the woody tissue of plants.

(ii) Exinite originated from the chemically resistant vegetable matter like spores, cuticles, resins, polymerized waxes, fats and oils.

(iii) Inertinite represents a group of macerals derived from plant material that has been strongly altered and degraded in oxidising conditions in the peat stage of coal formation. Inertinite is derived from the same woody and cellular material as vitrinite.

These groups of macerals have distinct optical, physical and chemical properties and can be identified with relative ease by means of petrographic analysis. A summary of the major characteristics of the three macerals is given in Table 2.1

The most common mineral impurities found in coal are clays, carbonates, sulphides and quartz. These minerals can be associated with coal in two ways:

(i) Epigenetic minerals are minerals which filled cleats and crevices after solidification of the seam. Coals containing epigenetic minerals are easily liberated and beneficiated.

(ii) Syngenetic minerals were deposited at the time of coalification and are thus intimately intergrown with the coal. It is therefore difficult to liberate and to beneficiate syngenetically bound coals.

Table 2.1
Major characteristics of the three macerals of hard coal
(Falcon and Snyman, 1986)

MACERAL GROUP	PLANT ORIGIN	REFLECTANCE			CHEMICAL PROPERTIES		
		DESCRIPTION	RANK	% REFLECTED LIGHT	CHARACTERISTIC ELEMENT	TYPICAL PRODUCTS ON HEATING	
VITRINITE	Woody Trunks, Branches, Stems, Stalks, Bark, Leaf Tissue, Shoots and detrital organic matter gelified/vitrinized in acquatic reducing conditions	Dark to medium grey	Low rank to Medium rank Bituminous	0.5 - 1,1 / 1,1 - 1,6	Intermediate hydrogen content	Light hydrocarbons	Intermediate volatiles decreasing rank
		Pale grey	High rank Bituminous	1,6 - 2,0	-	-	
		White	Anthracite	2,0 -10,0	-	-	
EXINITE	Cuticles, Spores, Resin bodies, Algae accumulating in sub-aquatic conditions	Black-brown	Low rank	-0,0 - 0,5	Hydrogen-rich	Early methane gas	Volatile-rich decreasing with rank
		Dark grey	Bituminous	-0,5 - 0,9 / -0,9 - 1,1		Oil	
		Pale grey	Medium rank Bituminous	-1,1 - 1,6		Condensates wet gases (decreasing)	
		Pale grey (=vitrinite) to white shadows	High rank Bituminous to Anthracite	-1,6 - 10,0	-	-	-
INERTINITE	As for vitrinite, but fusinitized in aerobic oxidizing conditions	Medium grey	Low rank Bituminous	0,7 - 1,6	Hydrogen-poor	-	Low volatiles in all ranks
		Pale grey to white and yellow-white	Medium rank Bituminous to Anthracite	-1,6 - 1,8 / -1,8 -10,0	-	-	

2.1.2 **Origin of coal**

The origin of coal dates back to the Carboniferous, the Permian and the early Cretaceous periods, when decaying plant matter was deposited in swamps, along river beds and in lakes. Over the years these deposits were covered with silt and sandstone and, depending on the nature of the plant

material, and on climatic and tectonic influences, they changed to different types of coal.

As a result of different climatic conditions and vegetation, there are distinct differences between the coals of the Northern and Southern Hemisphere. Northern Hemisphere or Laurasian coals are rich in vitrinite, a highly reactive maceral, while Southern Hemisphere coals, also known as the Gondwana coals, contain mainly inertinite, which is largely unreactive except for semi-fusinite and macrinite. Exinite, a less reactive maceral, is present in small quantities.

There are also differences in mineral association. Northern Hemisphere coals contain mainly epigenetic minerals while Southern Hemisphere coals contain syngenetic minerals. In the Southern Hemisphere clays form about 70 % of the mineral impurities and are of submicron size. The other mineral impurities consist of quartz (20 %), carbonates, sulphides and oxides.

2.1.3 **Characterization**

Coals are generally characterized by proximate and ultimate analysis and petrography. Physical properties, like the Swelling Index, Abrasiveness Index and the Hardgrove Grindability Index are good market indicators and are generally included in the characterization.

The proximate analysis of a coal consists of the determination of inherent moisture, ash, volatile matter and fixed carbon content, the last being calculated by difference (from 100 %).

Ultimate analysis determines the proportions of the main chemical elements (carbon, hydrogen, nitrogen, sulphur and oxygen) contained in the coal.

The Swelling Index is a rank related measure used to determine the coking properties of a coal.

The Hardgrove Grindability Index and the Abrasiveness Index describe the physical nature and condition of a coal.

Petrography is the study of the microscopic constituents (organic and inorganic) in coal and the degree of metamorphosis (rank) to which they have been subjected subsequent to their time of burial. Recently petrographic analyses have been extended to include the physical, chemical and technological properties of these constituents. It is therefore also possible to predict the technological behaviour of a coal from the petrographic composition.

2.2 COAL IN SOUTH AFRICA

2.2.1 Characteristics

South African coals form part of the Gondwana coals which are characterized by large proportions of inertinite and syngenetic minerals (except for Natal coals). Compared to European coals, they contain on average four times more inertinite and syngenetic minerals (see Table 2.2).

Clays like kaolinite, illite and montmorillonite make up between 60 and 70 % of the syngenetic minerals in South African coals. Syngenetic carbonate and sulphide minerals are rare, while quartz occurs in both syngenetic and epigenetic forms.

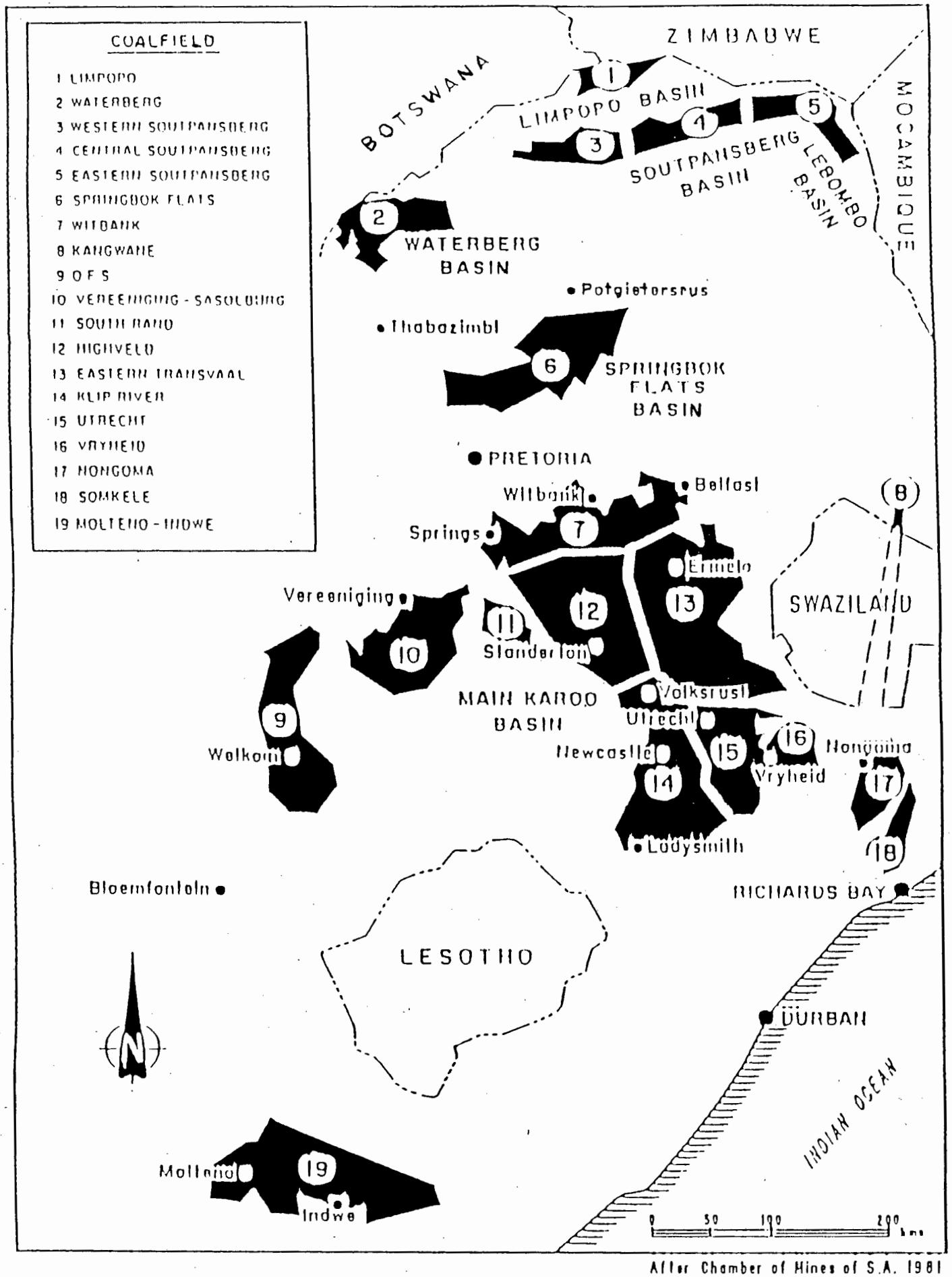
Table 2.2
The average relative proportions (%) of coal constituents in South African and European coals (Falcon, 1978)

	South Africa	Europe
Vitrinite	40	70
Exinite	0 to 5	15
Inertinite	60 (± 20)	15
Syngenetic Minerals	14	3

2.2.2 **Reserves**

South African coal reserves are divided into two regions, the traditional mining region which comprises fields in the Eastern Transvaal (Witbank, Middelburg), Northern Orange Free State and Northern Natal, and the newer mining region consisting of the Waterberg and Soutpansberg Fields in Northern Transvaal and the Springbok Flats in Central Transvaal. The major South African Coal Fields are marked on the map given in Figure 2.1.

By world standards South African coal reserves are relatively large in quantity but poor in quality. About 75 % of the in situ coals have an ash content higher than 21,5 % (Falcon, 1978). Viable exploitation of these reserves therefore depends on efficient liberation and beneficiation techniques. South Africa, nevertheless, is the world's fourth largest exporter of coal, after Australia, U.S.A. and Poland. In 1986 South Africa accounted for 13 % of the world's coal exports and this figure is expected to rise to 15 % by 1990 (Edwards, 1988). The next section details some of the products of the South African coal industry.



After Chamber of Mines of S.A. 1981

Figure 2.1 Major coalfields in South Africa

2.2.3 Products

About 40 Mt of the 176,5 Mt of coal produced in South Africa annually are exported (Harris, 1989). Low-ash metallurgical coal and power station steam coals are the main export products.

The local market requires coal for electricity generation, liquefaction processes, the metallurgical industry and general industry.

Electricity generation accounts for the highest local coal consumption. For this purpose coal is generally unbeneficiated and crushed and screened to a maximum 25 mm top size. ESKOM, the main electricity supplier in the country, has developed a unique method of utilizing poor quality coal efficiently for power generation, and is regarded as a world leader in this respect.

The South African coal liquefaction plants (SASOL I, II and III) are based on the Fischer-Tropsch synthesis and are thus relatively insensitive to the quality of coal. Low grade unwashed coals (ash contents up to 40 %) with particle sizes greater than 3 mm and low swelling and caking indices are suitable for this process.

The metallurgical industry requires good quality coking coal for metallurgical coke production. Since coking coal is expensive and a good export product, alternative routes of steelmaking (employing for instance the direct reduction and Corex processes as the ironmaking step) which require different grades of coal, are also currently used by ISCOR. For the production of ferro-alloys bituminous coals combined with coke and char are used.

2.3 LIBERATION

2.3.1 Mineral liberation

One of the earliest definitions of mineral liberation was published 50 years ago by Gaudin (1939). He defined the degree of liberation of a mineral as the percentage of that mineral occurring as free particles in relation to the total of that mineral occurring in free and locked forms.

Numerous liberation models have been proposed (King, 1975; 1979; Klimpel, 1983) for predicting the increasing liberation of substance "A" occluded in a bulk material "B" with decreasing size. One of the most recent developments is a parameter estimation procedure for a combined size reduction and mineral liberation model having multiple classes of composite particles (Choi et al, 1988).

However, these models were developed mainly for metalliferous ores and are not really applicable to coal because of its complexity. In terms of Gaudin's liberation model, the degree of liberation of most South African coals, when milled to ultrafine particle sizes, would still be nil because the syngenetic minerals cannot be totally removed. In addition, most models have been developed for two component systems and are therefore unsuitable for coal which is a complex multi-component system. The multi-component liberation model of Choi et al (1988) is based on SEM-Image Analysis. This technique is not suitable for coal because it is unable to distinguish between maceral and mineral constituents of a coal particle.

2.3.2 **Coal liberation**

The liberation of coal involves the freeing of coal macerals from the inherent mineral impurities. This is usually achieved by comminution.

In the case of epigenetic minerals, liberation is relatively easy because particles tend to break along the coal-mineral interface. This type of breakage usually occurs during normal mining procedures.

Liberation of coals containing syngenetic minerals is much more difficult and requires fine grinding. In order to liberate such coals in terms of Gaudin's definition, they have to be pulverized and even then liberation may not be complete.

It would thus be more appropriate to speak of the 'degree of liberation' of a particular coal sample rather than liberation.

2.3.3 **Reasons for liberation studies on coal**

In general coal is not crushed with the aim of liberation but rather (if necessary) for marketing purposes. In recent years, however, liberation studies on coal have become important for three reasons:

(i) The poor economic and political climate has put the South African coal mining industry under considerable pressure. As a result, improving the efficiencies of beneficiation processes has become very important. Liberation studies, from which the theoretical yields of different products from a particular coal can be determined, can assist greatly in this regard.

Theoretical yields provide targets against which the **efficiencies** of processes can be measured.

(ii) The increased use of mechanised mining methods by the coal industry has resulted in the production of ever increasing quantities of fines (nominally -500 μm). These fines are usually discarded because of their high ash contents. However, because of their fine size, they also contain large proportions of valuable low ash material and this encourages investigation into the beneficiation of these fines. In order to evaluate the viability of coal fines beneficiation it is important to know the liberation characteristics. These determine the **maximum yields** that are obtainable from a perfect separation process.

(iii) It is believed that there is a potential for the production of **super-low-ash coal** (< 4 %) for processes like direct liquefaction or for use in coal-liquid mixtures. If these high quality products can indeed be achieved through grinding to ultrafine sizes, the market potential of South African coals would increase tremendously. For this application it is important to establish the degree of size reduction necessary for the efficient production of high quality coals, which in turn requires a good understanding of the liberation characteristics.

2.4 MEASUREMENT OF COAL LIBERATION

Over the past decade the increased interest in fine coal beneficiation has encouraged researchers to develop new techniques for measuring coal liberation. Various methods have been used, most of them being variants of the well-known float and sink analysis. This is an indirect method

in which the proportions of the components in a coal particle are related to its density. Other methods, based on optical and surface properties, have also been developed. It is also possible to infer changes in the liberation characteristics from variations in particle size distribution on progressive milling.

2.4.1 **Float and sink separation**

Since coal is a multi-component substance, each component marked by a characteristic density, it is particularly suited to analysis of liberation by a technique based on differences in relative density. Such a technique is the well known float and sink method of analysis. For coarser particles (+75 μm) float and sink analysis can be carried out under gravity in suitable dense liquids. For fine particles (-75 μm) centrifugal density separations are recommended as gravity separations become very time consuming due to long settling times.

The International Organization for Standardization (ISO) (1981) has described a centrifugal method in which glass centrifuge tubes are filled with a coal/dense liquid slurry and centrifuged. Once separation is complete the floats are scooped out of the tube, filtered and dried. This method has two major drawbacks: (i) the high loading rate (approximately 40 % pulp density) can result in entrainment, and (ii) scooping the floated fraction from the surface is fairly difficult to do without contaminating the sinks.

To overcome the inadequacies of the ISO method Franzidis and Harris (1986) designed a special device which fits into a centrifuge tube. A diagram of this device is given in Figure 2.2 below.

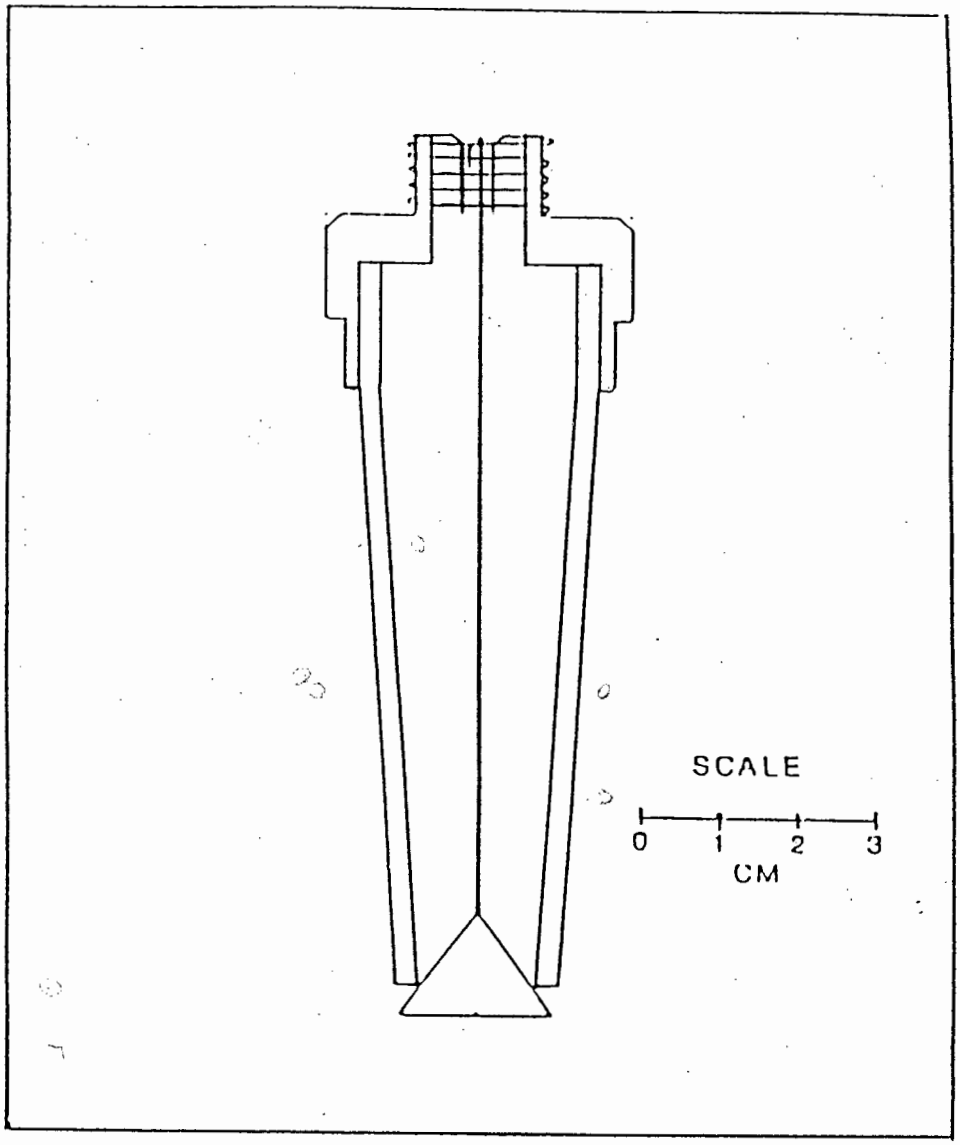


Figure 2.2 A schematic diagram of the new float and sink apparatus (Harris, 1987)

The device consists of a tapered tube, with the bottom sealed by a spring-loaded plug. During centrifugation the plug opens and allows free passage of sink material into the centrifuge tube. Once centrifugation is stopped, the plug pulls closed, resulting in a perfect separation. Other advantages of this method are that it is accurate for particle sizes down to 25 μm and that a very small sample (2 gram) is required for each separation density. This method

is also very quick compared to a gravity separation, which may take days or weeks to separate particles smaller than 25 μm.

More recently, Dumm and Hogg (1988) studied the float and sink characteristics of coals milled to standard respirable dusts (coal powders which were less than 10 μm in size). The density separation technique employed was a slight modification of the ISO method in that it made provision for easier separation of the two fractions. After centrifugation the Teflon FEP tubes holding the separated fractions were pinched at a predetermined level by means of a modified pair of Vice-grip clamps. This allowed the removal of the floats fraction without contaminating the sinks.

Their investigation included a study of the centrifugation times necessary for complete separations of the floats from the sinks. The results of their work are given in Table 2.3 below.

Table 2.3

Mass percent of PSOC1192M coal in specific gravity intervals

	Centrifuge time at 2000 rpm, Hours				
Sp.Gr.	1/4	1/2	1	2	3
1,3 Floats	25,7	20,9	18,4	5,9	1,7
1,3 x 1,4	39,9	43,2	41,8	53,3	60,3
1,4 x 1,5	21,4	21,9	25,5	28,7	24,8
1,5 x 1,6	0,0	3,6	2,1	0,2	0,1
1,6 x 1,7	2,8	0,9	3,7	3,4	2,7
1,7 x 1,8	0,0	0,8	0,3	0,0	2,7
1,8 x 1,9	2,1	0,0	1,3	1,0	0,0
1,9 x 2,0	1,4	3,0	0,8	0,0	1,5
2,0 Sinks	6,7	5,7	6,1	7,5	6,2

From Dumm and Hogg (1988)

A considerable variation in the density distribution with increased centrifugation time was observed in the low relative density fractions. The mass of the floats at 1,30 relative density was reduced from 25,7 % at 15 minutes centrifuging to 1,7 % after 3 hours of centrifugation, while the 1,3 x 1,4 relative density fraction increased from 39,9 % to 60,3 % for the respective centrifugation times. It is therefore important to centrifuge very fine particles (-10 μm) for long periods of time to achieve complete separation in the low density fractions.

Yet another centrifugal method, using 125 ml glass separating funnels with special holders to fit into an IEC Model K centrifuge, was developed at the Electrical Power Research Institute (EPRI) in the U.S.A. (Hervol and Harrison, 1988). The performance of this method was tested on ultrafine coals (-150 +75 μm and -75 μm) and compared to the static-bath method (ASTM, 1985). The static-bath method is a gravity method which is carried out in hour-glass-shaped funnels. The centrifugal method proved more efficient for particle sizes less than 75 μm, but there was no difference in performance for the -150 +75 μm particle size.

Cavallaro and Killmeyer (1988) employed the same method to perform efficient density separations on coals of particle sizes down to -45 μm. The purpose of their work was to refine the procedure for coals of these fine particle sizes in order to eliminate anomalies occurring during low density separations. They investigated the effect of variables like moisture content of the sample, pulp density, dispersant concentration, conditioning time and ultrasonification time. The optimum conditions for their coals were 0 % moisture (bone dry) in the sample, a 5 % pulp density, 15 lb/ton dispersant, 0,1 minutes conditioning time and 120 seconds of ultrasonification.

2.4.2 **Optical methods**

Several methods for evaluating coal liberation exploiting optical properties have been documented in the literature recently. Huggins et al (1982) described a method by which minerals in coal can be quantitatively determined by the combination of Scanning Electron Microscopy-Based Automated Image Analysis (SEM-AIA) and Mossbauer Spectroscopy. This method also facilitates the study of mineral association and could possibly be applied to mineral-maceral associations.

Straszheim et al (1988) used SEM-AIA and energy-dispersive X-ray spectroscopy to characterize mineral matter in sub-bituminous coals. Samples were milled to 75 % passing 75 μm and cleaned by float-sink separations at relative densities 1,40 and 1,38 before being subjected to microscopic analysis. This method is able to show up differences in sample composition with cleaning, but is unable to determine the mode of mineral association i.e. whether the mineral particles are disseminated widely throughout the coal or clustered together.

2.4.3 **Oil agglomeration**

Oil agglomeration is a surface property dependent separation process which functions best on superfines (45 μm and less). It is a beneficiation process, which has been researched for many years, but to date only one plant installation has been reported (Capes, 1988).

The oil agglomeration process depends fundamentally on the formation of hydrocarbon bridges between coal particles by the addition of oil. This facilitates the separation of the hydrophobic and hydrophilic components of a raw coal. A generally accepted sequence for the process (Figure 2.3)

consists of emulsification of the oil, mixing the coal-oil-water system, collisions between oil droplets and coal particles and agglomeration of the oil-coated coal particles. Agglomerates are generally very hard and dry and can be several hundred microns in size.

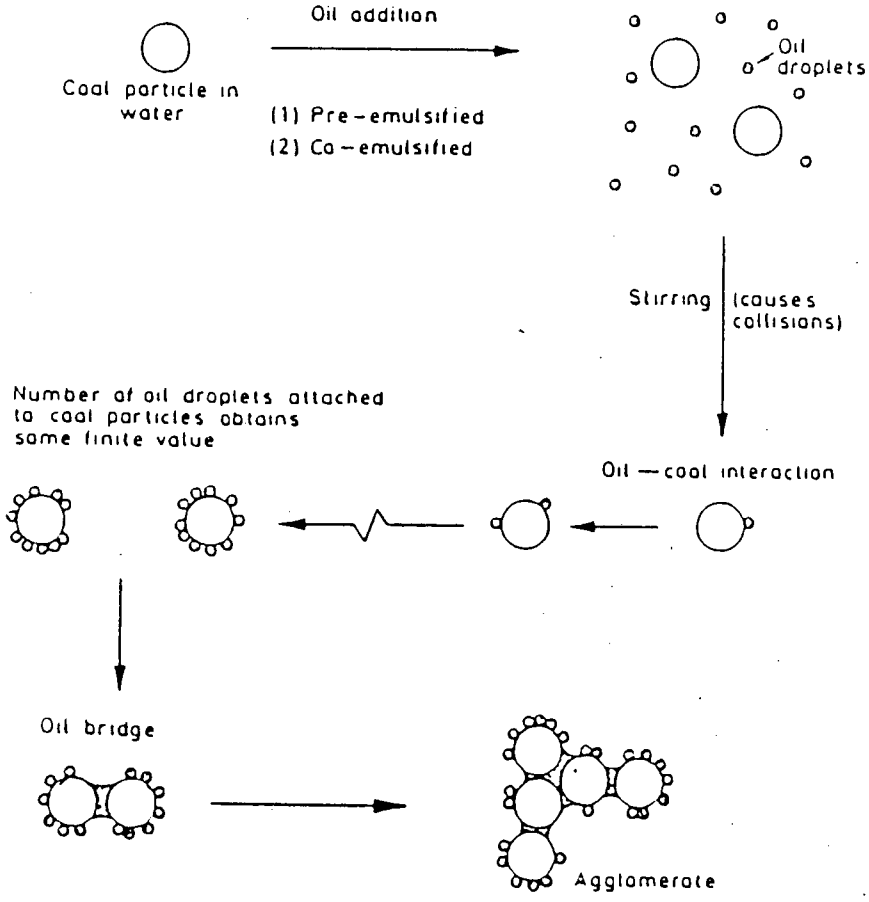


Figure 2.3 Principles of oil agglomeration (Dunsten, 1986)

Bridging oils used in this process vary from highly aromatic to highly aliphatic hydrocarbons. Aromatics are very easy agglomerators while aliphatics are very selective and therefore only agglomerate low ash material. Labuschagne (1989) has described the agglomeration properties of several chemically pure bridging oils in terms of molecular composition and stereochemistry. Commercial oils, like Shellsol AB and Shellsol K (Buys, 1987), diesel (Swanson et

al, 1977) and furnace oil (Rao et al, 1982) are relatively cheap and efficient bridging oils.

Oil agglomeration has not as yet been used for liberation studies but appears to have great potential in this field. In contrast to float and sink analysis, where gradual density changes result in gradual changes in yields, agglomeration is an 'all or nothing' process - yields and recoveries are usually high. For this reason it is difficult to employ agglomeration in the same sense as float and sink analysis, i.e. for drawing up washability curves*, in which a gradual increase in ash content is related to a gradual increase in cumulative product yield.

It is however possible to check the validity of float and sink data with oil agglomeration, especially at low ash values and at particle sizes below 25 μm. This can be done by performing agglomeration and float and sink analysis on the same sample. The position of the agglomeration results (yield and ash %) on the washability curve can be used to corroborate (or cast doubt on) the results of the float and sink separation.

2.4.4 **Size distribution**

Size distributions, especially below 25 μm, can be helpful in inferring liberation characteristics because high ash (i.e. mineral) particles tend to accumulate in the very fine fractions. Changes in the size distribution pattern on grinding can be used to speculate on the behaviour, based on the physical properties, of the macerals and minerals present.

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*Washability curves are described in more detail in section 2.5.2 below.

2.4.5 **Discussion**

Size distribution studies supply information on the physical properties of the coal constituents and therefore aid in predicting liberation characteristics. They are, however, only speculative and have to be substantiated with further analysis.

Optical methods would appear to have great potential for the assessment of liberation characteristics of ultrafine coal, but on close inspection the contrary proved true. Attempts were made to analyse samples of ultrafine coal (particle size less than 25 μm) by SEM-Image Analysis. Sample preparation was very difficult and interpretation of the results extremely subjective. Improvements in technology are required before optical methods can be used reliably for analysis of coal liberation.

The float and sink methods, although indirect, have been the most successful methods used to date to assess the liberation of fine coals. For particle sizes of 150 μm and greater the gravimetric float and sink methods are very reliable but for smaller sizes centrifugation is needed for efficient and fast separations. Liberation assessments of particle sizes down to 25 μm (Franzidis and Harris, 1986) can be made with confidence. For particle sizes finer than 25 μm oil agglomeration can be used to check the efficiency of the float and sink method.

2.5 **ASSESSMENT OF COAL LIBERATION FROM FLOAT AND SINK ANALYSIS**

After splitting a coal sample into a number of relative density fractions by means of float and sink analysis, the mass of each fraction is determined and calculated as a

percentage of the total sample mass. The ash content of each fraction is also determined. From these data the cumulative yield and ash content at any required density can be calculated .

These data can then be used in a number of ways to assess the liberation characteristics of the sample. Some of these ways are described in the sections below.

2.5.1 **Relative density distribution**

The degree of liberation of a coal can be assessed in the first instance by considering the density distribution within the sample. Since relative density is generally indicative of ash content, the distribution pattern exhibited by a particular coal can supply valuable information about the degree of liberation. The presence of intermediate density fractions, for instance, would suggest that the sample is less liberated than a sample with large proportions at the extremes of the relative density range.

The presence of different macerals complicates this analysis. Float and sink analysis is generally carried out in the 1,30 to 2,0 relative densities range. With inertinite having a relative density between 1,40 and 1,60, the presence of material in this relative density range could either be inertinite or unliberated vitrinite (1,2 to 1,30 R.D.). A knowledge of petrographic analysis is thus useful in interpreting relative density distributions.

2.5.2 **Washability curves**

From the float and sink results a number of curves can be plotted relating relative density to cumulative yield and cumulative yield to cumulative ash content of the products. These curves are collectively called the washability curves

and comprise the densimetric, cumulative floats, cumulative sinks and the characteristic ash curve. Sample graphs of the washability curves are presented in Figure 2.4.

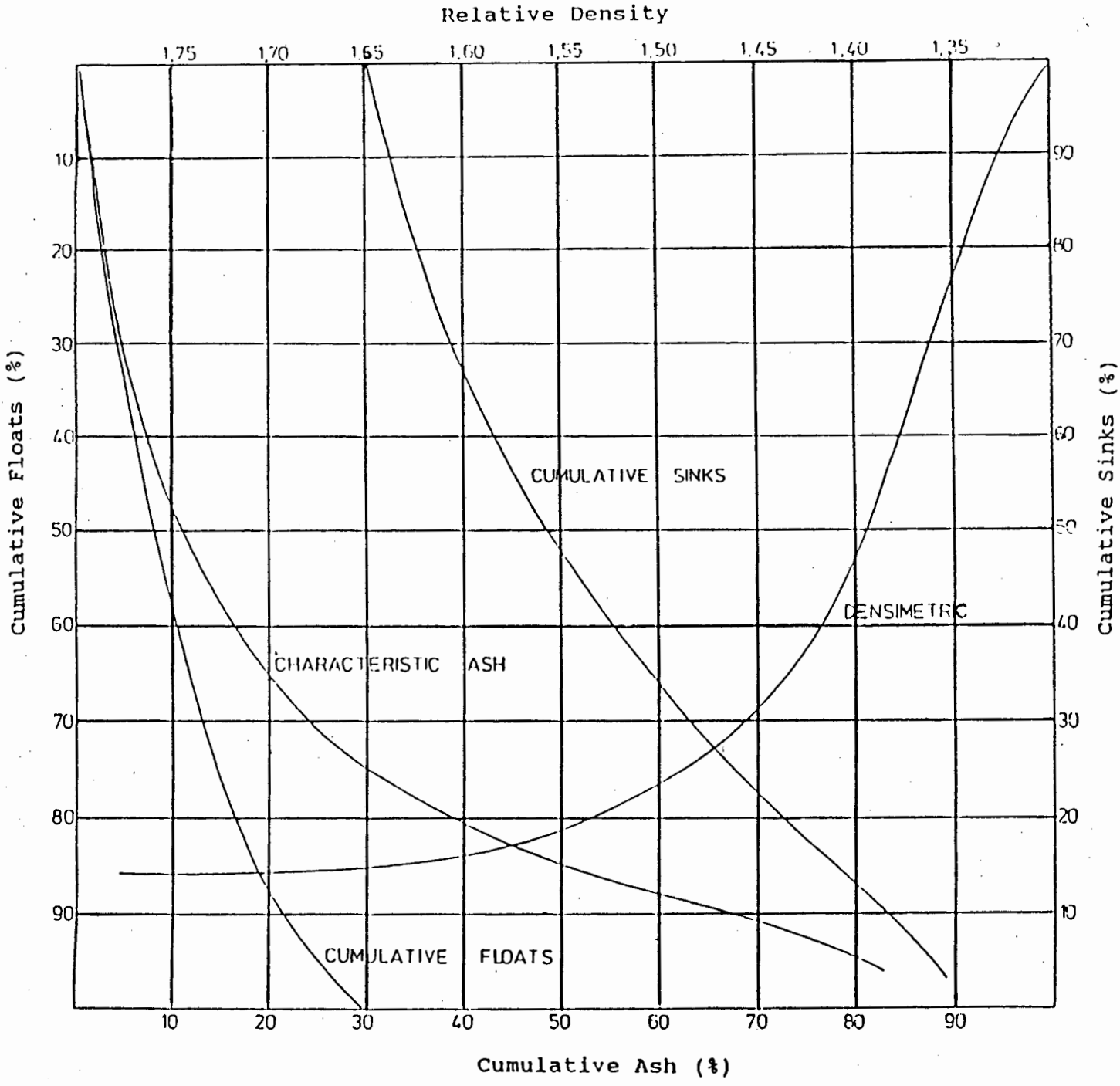


Figure 2.4 Washability curves

The densimetric curve is a graph of relative density against the cumulative yield of clean coal produced. The yield of clean coal (or discard) obtainable at any relative density, or conversely the density of separation required for any yield of either product may be read off this curve.

The cumulative floats curve relates the ash content of clean coal to the cumulative yield of clean coal. This curve gives the maximum (theoretical) yields at various ash contents.

The cumulative sinks curve (ash content of discard versus yield of discard) gives the ash content of the discard at any yield of discard.

The characteristic ash curve shows the highest ash content of any particle likely to be found in any particular yield of floats. This curve gives an indication how difficult or easy it is to clean a particular coal.

2.5.3 **Mittelwert curve (M-curve)**

Another curve which can be drawn from the results of float and sink experiments is the Mittelwert curve (M-curve). This curve is obtained by plotting the cumulative yield versus the product of the cumulative yield and the cumulative ash content (ash per 100 units of feed). An example of such a curve is given in Figure 2.5, for a coal consisting of 20 % ash.

This curve gives a direct measure of the liberation of coal from ash. For a perfectly liberated sample all the data would lie along lines ABC. For a totally unliberated sample all the data would lie along AC. Hence the degree of liberation, or liberation efficiency of a sample can be estimated by the ratio of the areas ADCA and ABCA. The

liberation efficiency (L) is given by the expression (Birtek and King, 1984).

$$L = \frac{\text{Area ADCA}}{\text{Area ABCA}} * 100$$

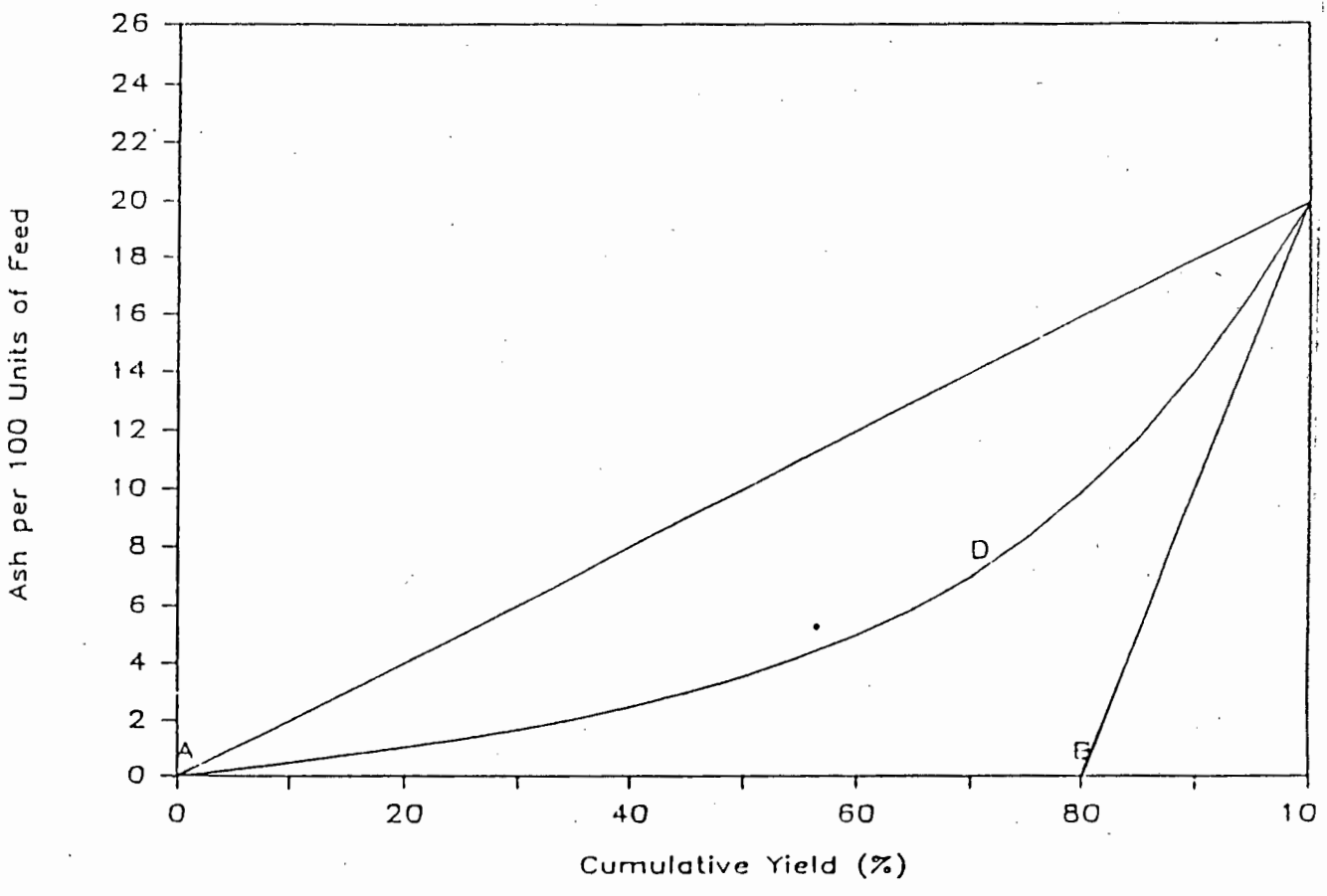


Figure 2.5 The characteristics of a typical M-curve

2.6 LIBERATION CHARACTERISTICS OF SOUTH AFRICAN COALS

It is only with the increased production of coal fines and the development of suitable beneficiation processes that the relevance of liberation studies on coal has become apparent. Since these developments are very recent, little has been reported in the literature on the liberation characteristics of South African coals.

Sanders and Brookes (1986), as part of a comparative study of the washability characteristics of coals from Europe, Australia, Botswana, India, Brazil and South Africa, reported on the liberation characteristics of coals from the Witbank No.2 Seam and from the Ermelo coalfield. The coals were milled down to a top size of 6 mm and analysed by float and sink analysis. This work showed that the washability characteristics were improved in the finer size fractions, but that the yield of low ash material remained poor. To achieve significant improvement in liberation would require still finer grinding.

Birtek and King (1986) studied the liberation behaviour of ash in fine coal from several South African coalfields. The samples were crushed to below 1 mm in size. The -38 +25 μm and -425 +300 μm fractions were subjected to float and sink analysis and analysed for ash content. The maceral compositions of the float and sink fractions were determined by petrography.

The results indicated that the mineral matter was concentrated in the finest size fractions and that crushing had very little effect on the liberation of coal down to 25 μm. All coals exhibited the characteristic Gondwana density distribution patterns, i.e. large proportions of middlings and minimal amounts of low density material. Vitrinite was found to accumulate in the coarser sizes while

inertinite concentrated in the finer sizes. Down to 25 μm there was no liberation of mineral matter within the definition of Gaudin.

Subsequently Harris (1987) investigated the liberation characteristics of a Witbank No.2 Seam coal obtained from the Greenside Colliery. He selected two samples, one of a run-of-mine (r.o.m.) or raw coal and one of thickener underflow. Subsamples of r.o.m. coal were milled to 30, 60 and 90 % passing 150 μm and screened into different size fractions, ranging from +250 μm to -25 μm. The thickener underflow sample was screened into various size fractions without milling. Float and sink analyses were carried out on all size fractions. Petrographic analyses were performed to investigate the liberation of the organic coal components.

The work showed that an increase in size reduction resulted in a relatively small increase in liberation. This can be seen from the liberation efficiencies listed in Table 1, Chapter 1. These were calculated from M-curves according to the method described in Section 2.5.3 above.

Harris (1987) found that the overall results for the sample milled to 90 % passing 150 μm compared well with those of the thickener underflow sample. Figure 2.6 shows the cumulative floats curves of the thickener underflow sample and its size fractions. As may be seen, the curve for the -25 μm size fraction lies well above those of the coarser fractions, indicating a higher overall ash content and the presence of significantly less low ash material.

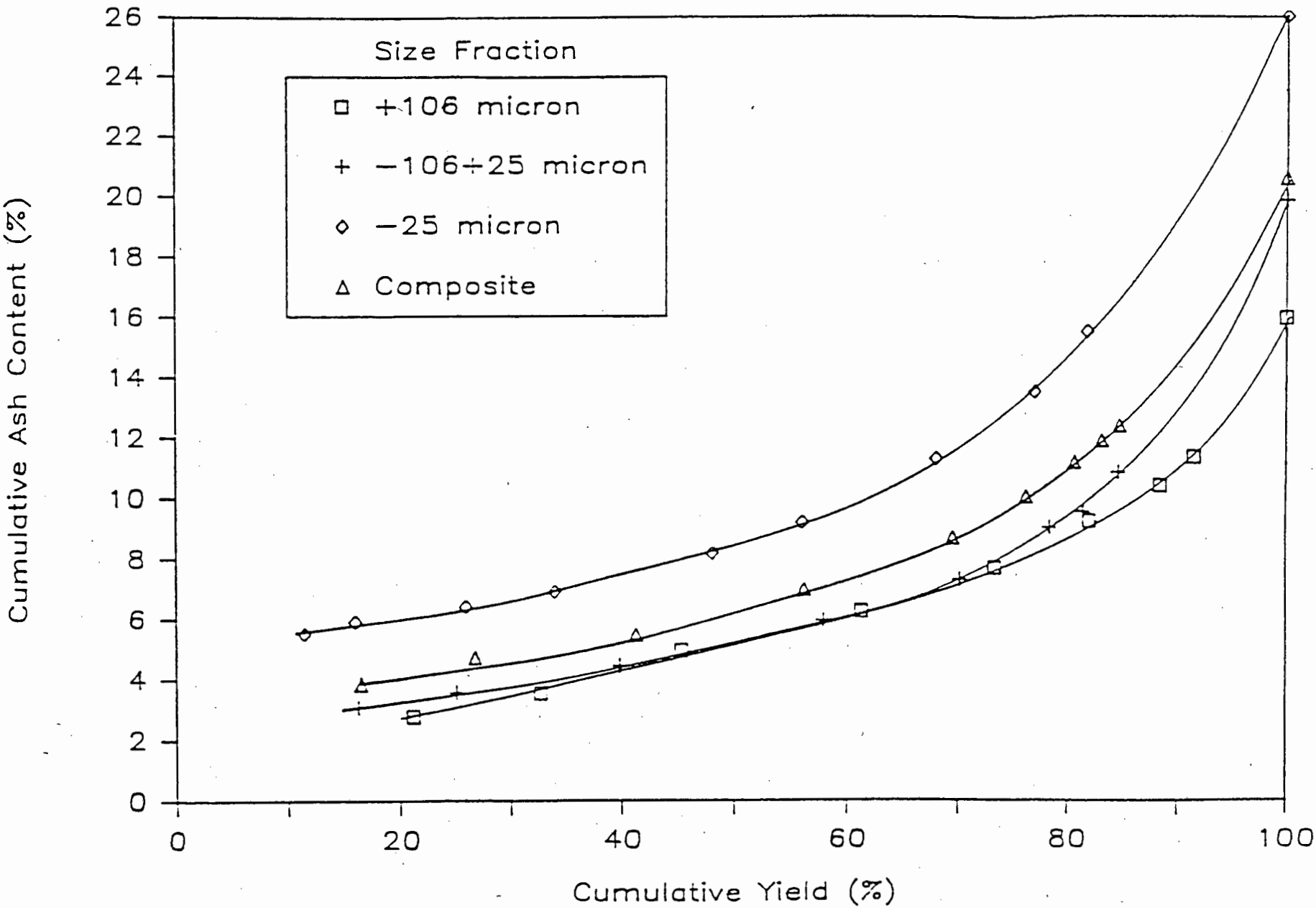


Figure 2.6 Washability curves of the Greenside thickener underflow sample by size fraction (from Harris, 1987)

Harris (1987) attributed the poor washability characteristics of the -25 μm size fraction to a concentration of middling material into the -25 μm size fraction on size reduction. This could also be deduced from the density distributions of the three size fractions (Figure 2.7). In the -25 μm size fraction the proportions of intermediate density material were considerably larger than in the coarser size fractions. The low density material tended to concentrate in the coarser size fractions. Petrographic analysis showed that inertinite concentrated in the -25 μm size fraction and mineral association with this maceral was deduced. Vitrinite was predominantly found in the coarser floats fractions. These results supported and extended the findings of Birtek and King (1986), discussed above.

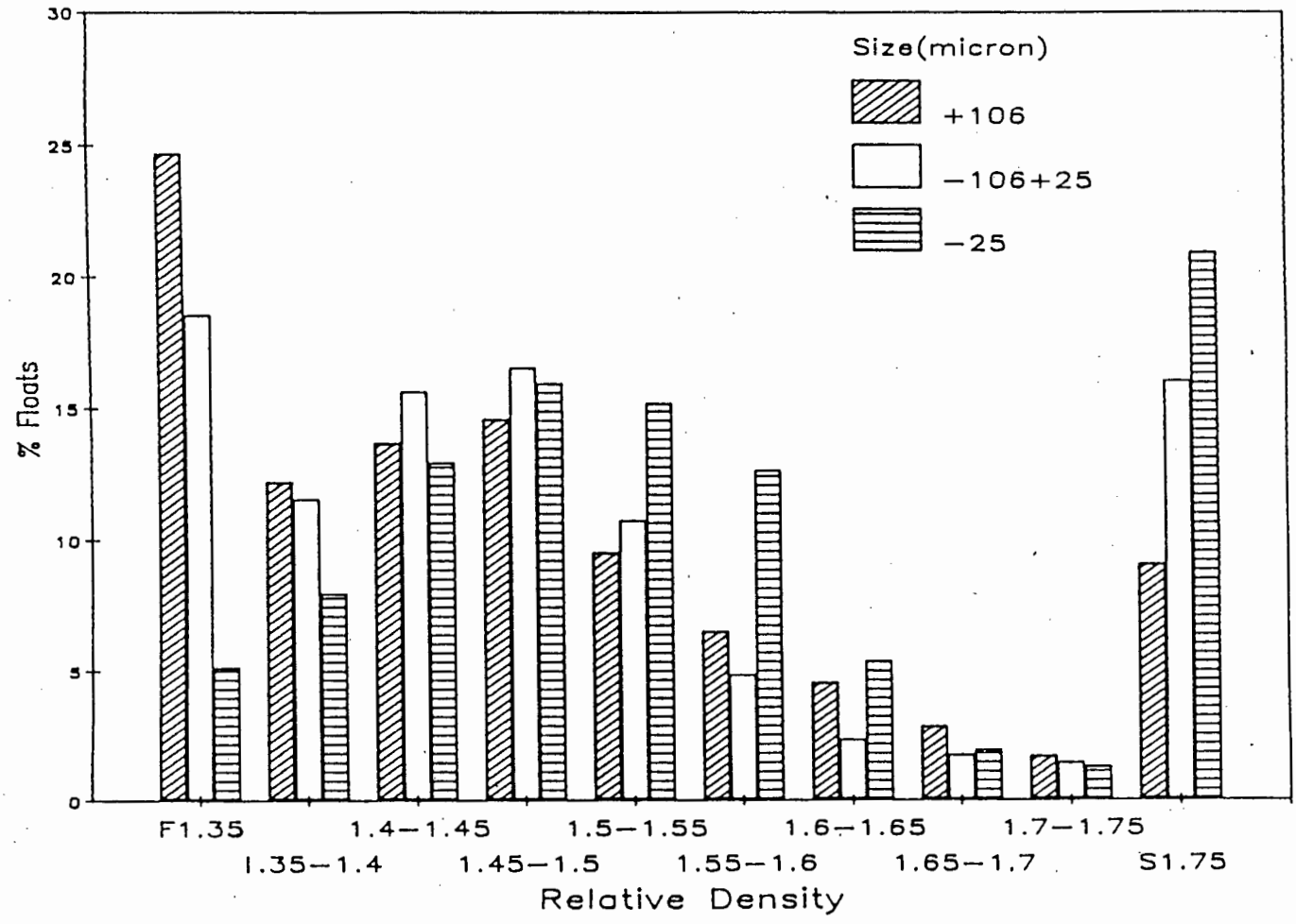


Figure 2.7 Density distribution of Greenside thickener underflow sample by size fraction (from Harris, 1987)

The density distributions of the composite samples, as given in Figure 2.8 showed that there was little change in the distribution pattern with prolonged milling. The characteristic 'middlings hump' of Gondwana coals (Sanders and Brookes, 1986) remained virtually unchanged. Harris concluded that in order to achieve a reasonably good degree of liberation, the coal would have to be ground to much finer sizes than 90 % passing 150 μm.

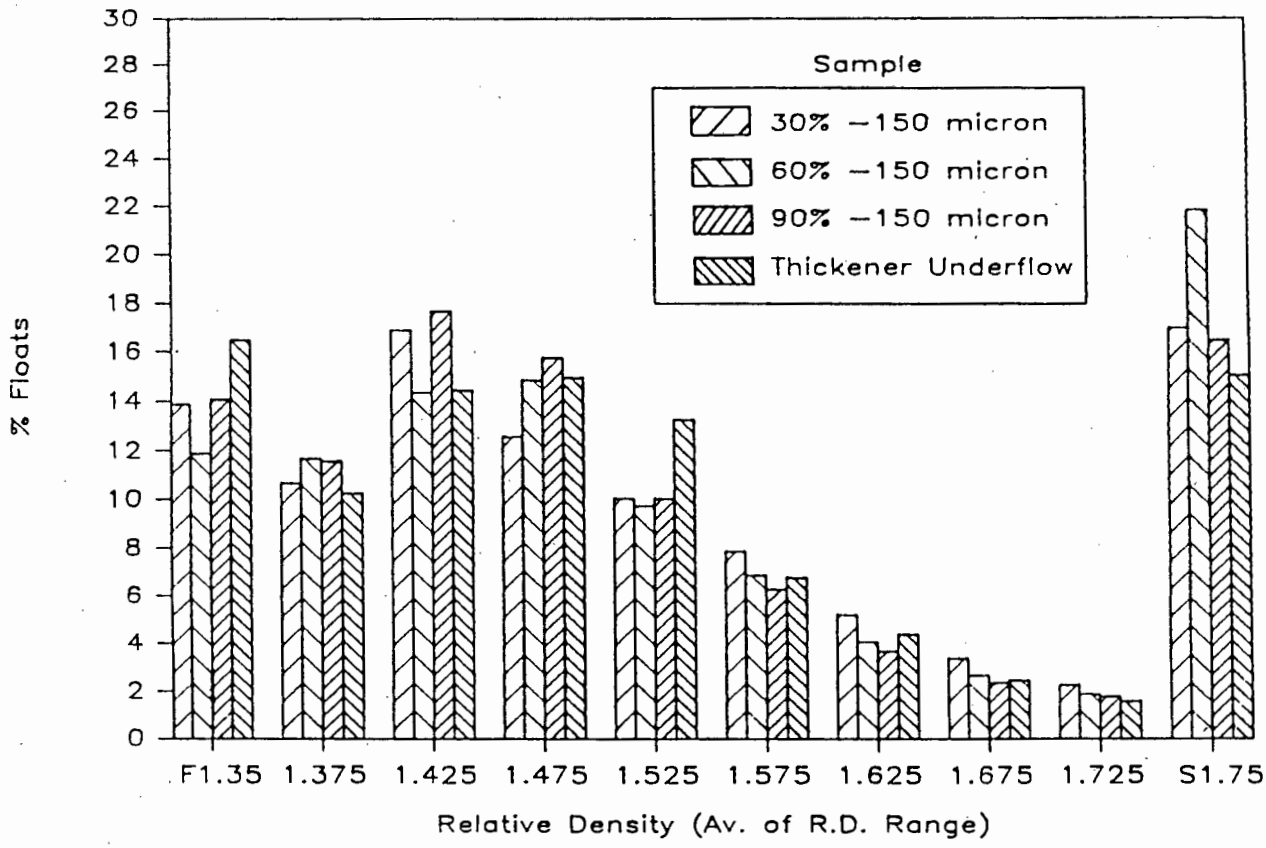


Figure 2.8 Density distribution of Greenside coal milled to various top sizes (from Harris, 1987)

2.7 **SUMMARY**

Coal is a mass of complex organic and inorganic materials bound together during original swamp-forming conditions. The major macerals are vitrinite, exinite and inertinite. The inorganic materials are present in either epigenetic or syngenetic forms.

South African coals are rich in inertinite and contain relatively large proportions of syngenetic clays which require fine grinding for liberation.

Recent developments in the mining industry (technological, economical and ecological) have resulted in a great interest in the beneficiation of fine and ultrafine coals, hence the need for a method for the assessment of liberation characteristics of superfine coals.

The most efficient experimental technique for liberation studies on ultrafines to date is the centrifugal float and sink method of Harris (1987). Oil agglomeration appears to offer a good alternative measure of liberation, while microscopy techniques show great potential for liberation studies but need to be developed further.

Liberation characteristics can be determined by means of washability, difficulty and M-curves, as well as size and density distributions.

Previous work on South African coal fines suggests much finer grinding (finer than 90 % passing 150 μm) is needed to achieve considerable improvement in liberating the organic constituents.

CHAPTER 3

EXPERIMENTAL EQUIPMENT, PROGRAM AND PROCEDURE

This chapter gives details of the experimental work undertaken to extend the study of Harris (1987) to finer sizes and to two other coals. The work involved assessing the extent of liberation of samples of the three coals when milled to various degrees of fineness. The chapter describes the factors considered when choosing the coals, some characteristics of the coals chosen, the experimental program undertaken, the experimental equipment and procedures used, and the analytical and computational methods employed in analysing the data.

3.1 CHOICE OF COALS USED IN THE INVESTIGATION

Two coals from the Greenside and Rietspruit Collieries in the Witbank Coalfield and one coal from the Grootegeluk Colliery in the Waterberg Coalfield were chosen for this investigation. These coals were selected for the following reasons :

(i) The Greenside Colliery is one of South Africa's most important sources of low ash coal for export, and therefore an important revenue earner. An in-depth study of the liberation characteristics would thus be of great interest and value, and could possibly lead to improvements in production. In addition the liberation characteristics of coal fines from seam No.2 of this colliery have previously been investigated (Harris, 1987). From the results it was

evident that the fines would have to be milled to yet finer sizes to achieve good liberation.

(ii) Coal from the Rietspruit Colliery is of interest because it originates from the same coalfield as the Greenside coal but from a different part of the field. The Rietspruit mine produces only one product, namely a steam coal of about 14 % ash. An investigation into the liberation characteristics of the naturally arising fines may reveal a hidden potential for low ash coal production.

(iii) The Grootegeluk Colliery in the Waterberg Coalfield is mining one of South Africa's largest reserves. The coal is very reactive (high vitrinte content) but also contains a very high percentage of ash. If the ash could be removed, possibly by grinding to ultrafine sizes, there is a great potential for direct liquefaction and coal-liquid mixture products.

The samples obtained from the Greenside and Rietspruit Collieries were of thickener underflow while the Grootegeluk sample comprised only the -150 μm fraction of a sample of the thickener underflow (which constitutes the feed to the flotation plant). The separation into +150 μm and -150 μm size fractions was made at the ISCOR pilot plant in Pretoria. The Greenside sample was received air dried in a 20 kg drum lined with plastic and the Rietspruit and Grootegeluk samples were received wet in 300 gram sealed plastic packets.

3.2 **CHARACTERISTICS OF THE COALS CHOSEN**

Representative subsamples of all three coals (as received) were sent to a commercial laboratory for proximate analysis.

Previous work by Harris (1987), Harris et al (1986) and Dimou et al (1986) on the three coals included petrographic analysis. The results of these analyses are described below.

3.2.1 **Proximate analysis**

The proximate analyses of Greenside, Rietspruit and Grootegeluk coals are presented in Table 3.1 below.

Table 3.1
Proximate analysis of the three coal samples

Coal	Moisture (%)	Ash (%)	Volatiles (%)	Fixed Carbon
Greenside	2,3	19,6	14,1	64,0
Rietspruit	3,0	26,3	22,8	47,9
Grootegeluk	1,5	42,0	25,6	30,9

From these analyses it can be seen that all coals contain between 1,5 and 3,0 % moisture. Of the three coals Greenside coal has the lowest ash and volatile matter contents (19,6 and 14,1 % respectively). Rietspruit coal has an ash content of 26,3 % while Grootegeluk contains 42,0 % ash. The volatile matter content in these two coals are 22,8 % and 25,6 %, respectively. The amount of fixed carbon in Greenside coal is 64,0 %, compared to 47,9 % and 30,9 % in the Rietspruit and Grootegeluk coals, respectively.

3.2.2 **Petrographic analysis**

The major organic constituents of Greenside, Rietspruit and Grootegeluk coals are listed in Table 3.2

Table 3.2
Major coal constituents of Greenside, Rietspruit and Grootegeluk thickener underflow samples

Coal	Vitrinite (%)	Exinite (%)	Inertinite (%)	Total Reactants (%)
Greenside	31,3	1,6	67,1	47,3
Rietspruit	29,6	3,8	66,6	46,5
Grootegeluk	82,5	2,4	15,1	87,0

It can be seen that in terms of maceral composition, the Greenside and Rietspruit coals are very similar. The vitrinite content of Greenside is 31,3 % (by volume) and that of Rietspruit coal is 29,6 %. The exinite content is low in both coals; Greenside coal contains 1,6 % and Rietspruit coal 3,8 %. The inertinite content of Greenside and Rietspruit coal is 67,1 and 66,6 %, respectively. The corresponding values for the total reactants in these coals are 47,3 % and 46,5 %.

In the case of Grootegeluk coal, the analysis showed that vitrinite was by far the predominant maceral component, making up 82,5 % of the organic constituents of the coal. Exinite was present at 2,4 % and the remainder (15,1 %) was made up by inertinite. The percentage of total reactants was very high at 87 %.

3.3 **EXPERIMENTAL PROGRAM**

The liberation study carried out on the three coals described above was based mainly on density separations. Each coal was milled to progressively finer particle sizes, namely to 95 % passing 150, 75 and 45 μm, and each milled product was split into a +25 μm and -25 μm fraction. Each of these size fractions was in turn separated into a range

of density fractions by float and sink analysis. Additional particle size analyses were performed on each -25 μm fraction.

For the density separations, the centrifugal float and sink method of Franzidis and Harris (1986) was used. The +25 μm and -25 μm size fractions of the 'as received' sample and of the milled subsamples of each coal were analysed using the cumulative method described in section 3.4.3.2 (a) below.

The -25 μm size fractions were also subjected to sequential centrifugal float and sink analysis (as described in section 3.4.3.1 (b) below) in the relative density range of 1,35 to 1,50. Preliminary experiments (section 4.3) showed that small amounts of misplaced material in the floats could result in grossly erroneous washability data in the low relative density range (1,35 to 1,45), hence the need for sequential float and sink analysis in this density range.

Oil agglomeration, a process extremely suitable for separating ultrafine coals, was used to check the accuracy of the centrifugal float and sink method employed. Oil agglomeration was carried out on the +25 μm and -25 μm fractions of the subsamples of each coal milled to 95 % passing 75 μm and 45 μm.

As a further check on the centrifugal method, some of the +25 μm fractions were sent to an outside laboratory for float and sink analysis by a standard gravimetric method. This method used Certigrav as the dense liquid, and therefore also served as a control for the dense liquid (zinc chloride) in the centrifugal method.

A brief summary of the schedule of work performed on the three coals is given in Table 3.3.

Table 3.3
Experimental schedule for each coal

Particle Size (μm)	+25 μm Fraction	-25 μm Fraction
As Is	-	Size Analysis
	Cum. Float/Sink	Cum. Float/Sink
	-	Seq. Float/Sink
95% -150	-	Size Analysis
	Cum. Float/Sink	Cum. Float/Sink
	Grav. Float/Sink	Seq. Float/Sink
95% - 75	-	Size Analysis
	Cum. Float/Sink	Cum. Float/Sink
	-	Seq. Float/Sink
	Agglomeration	Agglomeration
95% -45	-	Size Analysis
	Cum. Float/Sink	Cum. Float/Sink
	-	Seq. Float/Sink
	Agglomeration	Agglomeration

3.4 **EXPERIMENTAL EQUIPMENT AND PROCEDURES**

3.4.1 **Milling**

Subsamples of the 'as received' Greenside, Rietspruit and Grootegeluk coals were milled to 95 % passing 150, 75 and 45 μm. The milling times and quantities milled in each case are given in Table 3.4 below.

A stainless steel rod mill with a diameter of 31,6 cm was used. The mill operated with 20 rods and varying quantities of air dried coal. The critical speed of the mill was

calculated from the Chemical Engineering Handbook (Perry and Chilton, 1973), as follows:

$$N_c = 76,6/(D^{1/2})$$

where N_c = Critical speed (rpm)
D = Diameter (feet)

For a mill diameter of 31,6 cm (1,04 feet) the formula gives a critical speed of 75,2 rpm. The mill was operated at 0,9 N_c (67 rpm) which is the region of optimum efficiency.

Table 3.4
Milling details for Greenside, Rietspruit and Grootegeluk coals

Coal	Mass (kg)	Milling time (min.)	Final Size (μm)
	1,0	4,0	95 % -150
Greenside	1,0	12,5	95 % - 75
	1,0[2]	65,0	95 % - 45
	1,3[3]	11,8	95 % -150
Rietspruit	0,5[3]	16,8	95 % - 75
	1,0[2]	62,0	95 % - 45
	1,5	3,0	95 % -150
Grootegeluk	1,0	8,0	95 % - 75
	1,0[2]	20,0	95 % - 45

[2] Wet milled with 3 litres of water.

[3] Milled in stages; 0,5 kg of Rietspruit coal milled to 95 % passing 150 μm, was milled for 16,8 minutes.

3.4.2 **Screening**

Each coal, as received, and after milling to 95 % finer than 150, 75 and 45 μm, was split into +25 μm and -25 μm size fractions.

It was not possible to screen the samples with an automatic test sieve shaker because the relatively large proportions of superfines continuously blocked the 25 μm test sieve. Therefore all screening had to be done manually with 200 mm diameter stainless steel laboratory test sieves.

Approximately 50 grams of wetted coal were screened at a time under a jet of water. The screened fractions were filtered and dried at 105^{0}C.

3.4.3 **Float and sink analysis**

Three different methods of float and sink analysis were employed during the course of the testwork :
(a) a cumulative centrifugal method
(b) a sequential centrifugal method
(c) a sequential gravimetric method
The procedures are detailed below, after a brief description of the equipment used.

3.4.3.1 Centrifugal separations

(a) Equipment

The new centrifugal method developed by Franzidis and Harris (1986) was used. The advantage of this method over other density separation methods (discussed in Chapter 2) is that it can perform reliably down to particle sizes of 25 μm, and that it only requires small quantities of sample (2 gram) at

each relative density. A drawing of the separating device appears in Figure 2.2, Chapter 2.

The centrifuge used for this work was a Beckman TJ 6 model with a swing arm rotor. The maximum speed of rotation was 3000 rpm.

Separations were carried out in the relative density (R.D.) range 1,35 to 1,70. A zinc chloride solution in water was used as the dense liquid. A stock solution of zinc chloride (approximately 1,80 R.D.) was prepared by dissolving Technical Grade zinc chloride crystals in water until saturation point. The required densities were then prepared by diluting the stock solution. The relative density of the solutions was measured with a hydrometer.

(b) Procedures

(i) Cumulative Method

Approximately 2 gram of air dried coal was accurately weighed into a specially manufactured 25 ml sample bottle. This consisted of a screw-capped Mc Cartney type bottle with a 4 mm glass outlet tube at the bottom. The tube was fitted with a short length of peristaltic tubing and a clamp. Five millilitres of separating liquid of the required density and a few microlitres of Tween 20 (polyoxyethylene sorbitan monolaurate) were added to the coal in the sample bottle. The bottle was sealed, shaken and placed into an ultrasonic bath for approximately 5 minutes to wet the coal thoroughly.

After wetting, the sample was transferred quantitatively into the perspex separating device, which was then filled to the required level with separating liquid, as was the centrifuge tube (see Figure 3.1 A). The device was then inserted into the centrifuge tube (Figure 3.1 B).

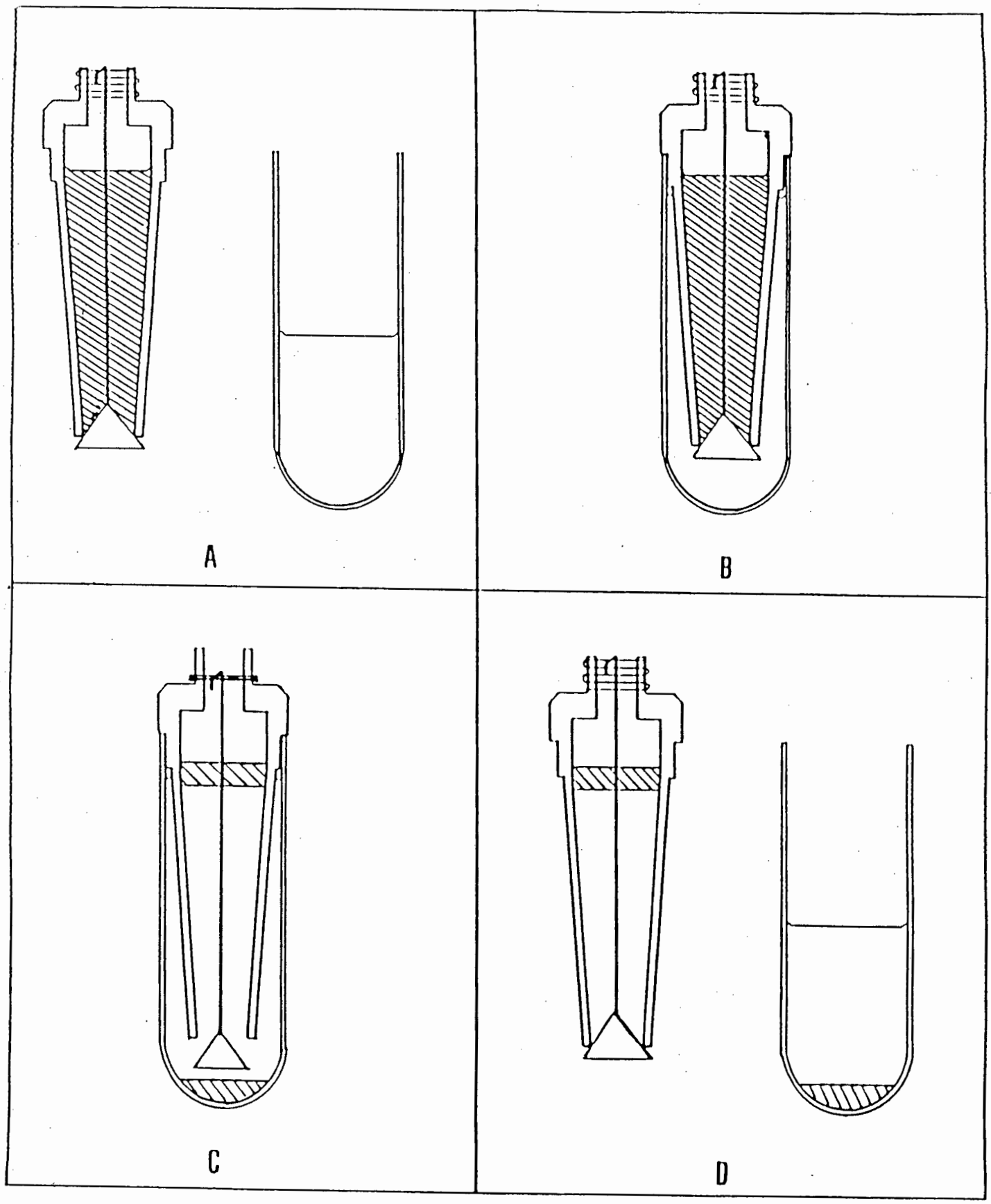


Figure 3.1 The use of the float and sink apparatus (Harris, 1987)

The spring on the device was locked so as to allow the floats and sinks to separate out first in the inner tube. The tube was centrifuged at 3000 rpm for approximately 30 minutes or until a separation within the device was clearly visible. The lock on the spring was then released and the tubes centrifuged for an additional 10 minutes (Figure 3.1 C). After this the perspex device was removed from the outer tube (Figure 3.1 D) and the floats and sinks were filtered with a Buchner funnel using glass fibre filter paper (Whatman GF/B). Each fraction was washed with five aliquots of water and one aliquot of ethanol. The fractions were dried at 105^{0}C, weighed and analysed for ash content.

(ii) Sequential Method

In addition to cumulative float and sink analysis, the -25 μm fractions were subjected to sequential float and sink analysis over the relative density range 1,35 to 1,50. This was done in an attempt to eliminate the misplaced material in the floats at low densities (see Chapter 4, Section 4.3).

Approximately 60 grams of coal were evenly distributed between 8 sample bottles and wetted thoroughly with separating medium of the required density (starting at relative density 1,30 and then increasing by 0,05). Wetting was aided by the addition of small amounts of Tween 20 and ultrasonification. The samples were transferred quantitatively to Teflon centrifuge tubes (capacity 100 ml). These were filled to capacity, sealed and centrifuged at 3000 rpm until separation was complete. The floats were carefully separated from the sinks and both fractions were filtered (Whatman GF/B), washed with water and ethanol, dried at 105^{0}C and analysed for ash content. This procedure was repeated at the next higher density using the sinks as feed.

3.4.3.2 Gravimetric separations

These gravimetric separations were carried out by Mrs. D. Moody of the Department of Metallurgy, University of the Witwatersrand, Johannesburg.

(a) Equipment

Cylindrical glass separating funnels with two-way taps were used for the gravity separations. The dense liquid used was Certigrav, a mixture of perchloroethylene, toluene, methylene bromide and aliphatic naphthals. Depending on the mixture, Certigrav can have a specific gravity from 1,2 to 2,5. Certigrav can be used in glass, teflon and polypropylene. It readily attacks perspex (which is why it could not be used in the centrifugal method) and it is incompatible with aluminium and magnesium.

(b) Procedure

About 300 ml Certigrav of the required density were introduced into a separating funnel. To this 60 gram of coal was added slowly with continuous stirring. The walls of the separating funnel were washed down with an additional 200 ml Certigrav. The coal-Certigrav mixture was stirred and allowed to stand until separation was complete.

As soon as a definite sediment had formed in the bottom of the funnel, this was drawn off to avoid compacting. When separation was complete, the sinks and approximately half the Certigrav were drawn off and filtered (Whatman No.1). The remainder of the Certigrav and the floats were removed from the separating funnel and were also filtered.

The filtered floats and sinks fractions were washed with ethanol and dried overnight at 105^{0}C. The fractions were

weighed and analysed for ash content. An accurately weighed portion of the sinks fraction was introduced into a separating funnel containing 300 ml Certigrav of the next higher density and the above procedure was repeated.

3.4.4 **Oil Agglomeration**

3.4.4.1 Equipment

The agglomeration equipment consisted of a cylindrical glass vessel (250 ml beaker) and a mechanical stirrer. A spatula was inserted into the vessel to serve as a baffle (see Figure 3.2). A 106 μm sieve was used to separate the agglomerates from the gangue.

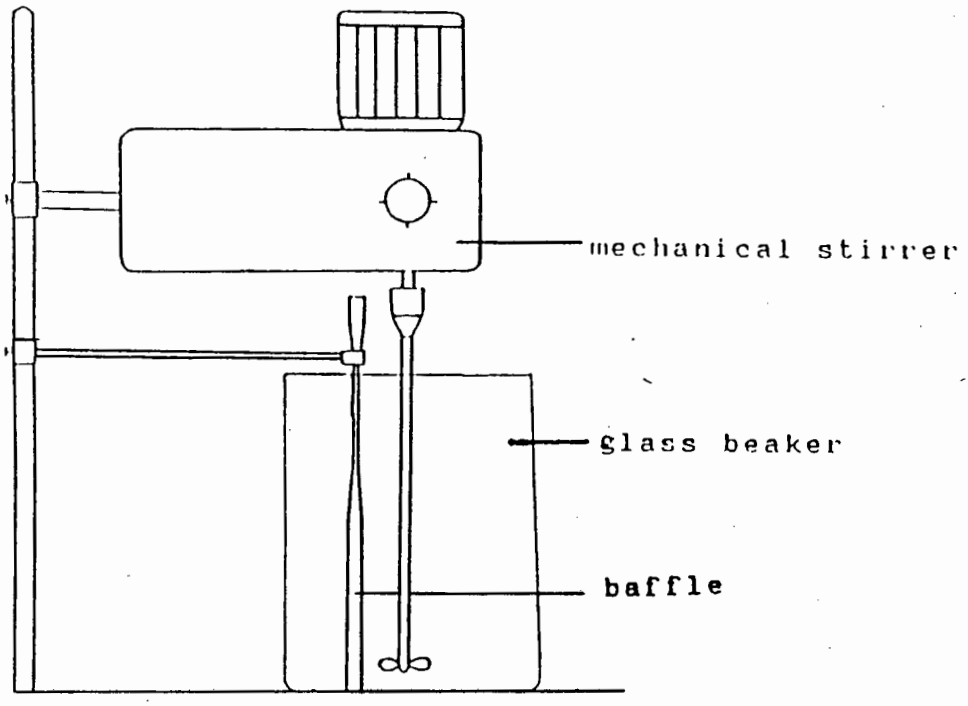


Figure 3.2 Agglomeration apparatus

A variety of bridging oils, ranging from highly selective aliphatic to less selective aromatic oils and mixtures of the two were used. A list of these oils is given below:

Shellsol AB (95 % aromatic hydrocarbons)
Shellsol K (95 % aliphatic hydrocarbons)
Shellsol AB/Shellsol K (50:50) (by mass)
Tetralin (tetrahydronaphthalene)
Benzene/Naphthalene mixtures

3.4.4.2 Method

About 7,5 gram of air-dry coal was weighed accurately and wetted with a minimal amount of water to form a thick paste. This paste was transferred quantitatively into the agglomeration vessel and enough water was added to make up a 10 % (m/v) pulp (7,5 g in 75 ml water). The pulp was agitated for approximately 5 minutes to ensure complete wetting of the coal.

Between 25 and 35 % (m/m) of bridging oil was added to the pulp. The agglomeration time (t_A) was monitored visually and was defined as the time taken from the addition of bridging oil to the formation of agglomerates. At time = t_A the agitation was stopped and the agglomerates were separated from the gangue by screening the mixture through the 106 μm test sieve.

The agglomerates were then washed with acetone to remove the bridging oil and dried in a microwave oven. The dry agglomerate and gangue fractions were weighed and analysed for ash content.

The agglomeration parameters that were kept constant throughout the experiments were:

pulp density - 10 % solids
impeller speed - 1660 rpm
natural pH - 7,9 to 8,1

Factors that were varied:

bridging oils (Section 3.4.4.1 above)
bridging oil concentrations - 20 to 35 % (m/m)

3.4.5 **Analytical procedures**

3.4.5.1 Ash content

Ash determinations were performed using a muffle furnace according to SABS Standard Method 926.

3.4.5.2 Particle size analysis

Size analyses on the -25 μm fractions of each sample were performed on a Malvern 2600/3600 Particle Sizer VF.6.

3.4 **LIBERATION ASSESSMENT**

The float and sink data of the +25 μm and -25 μm size fractions of each coal and its milled subsamples were expressed in the form of density distributions, washability graphs and M-curves. An outline of these liberation assessment techniques are given in sections 2.5.1 to 2.5.4.

The M-curves, in combination with a polynomial curve-fitting computer program, were used to determine the liberation efficiencies of numerically reconstituted composite samples of each coal and the milled subsamples.

The overall size distributions of the samples, and of the -25 μm fractions of each coal, were also used to provide information on the liberation characteristics.

CHAPTER 4

PRELIMINARY EXPERIMENTS

Some preliminary experiments were carried out to investigate the possible influence on the coal samples of the reagents used in the float and sink analysis and in the agglomeration testwork. The coals were exposed to zinc chloride, ethanol, bridging oil and acetone for extended periods of time and the changes in mass and ash contents were recorded. Methods and results are reported in sections 4.1 and 4.2 below.

Preliminary investigations were also carried out on the application of the centrifugal cumulative float and sink method on the -25 μm size fractions of the Greenside coal and its milled subsamples. These indicated that this method gave erroneous washability data in the relative density range 1,35 to 1,45. These anomalies were corrected by using sequential float and sink analysis in this density range. The details are discussed in section 4.3 below.

4.1 EFFECT OF ZINC CHLORIDE AND ETHANOL ON COAL SAMPLES

The effect of zinc chloride and ethanol on the mass and ash contents of the +25 μm and the -25 μm size fractions was investigated. Small quantities of the +25 μm and the -25 μm fractions of subsamples of the Greenside, Grootegeluk and Rietspruit coals milled to 95% passing 45 μm were subjected to the following zinc chloride and ethanol treatment:

Five to ten gram of sample was weighed accurately into a 100 ml beaker. A drop of Tween 20 and 80 ml of zinc chloride (R.D. 1,50) were added and the mixture was stirred thoroughly. The mixture was allowed to stand for 3½ hrs

with occasional stirring. The zinc chloride was removed by filtration, the sample washed with approximately 250 ml water and 100 ml ethanol, dried at 105^{0}C and weighed. The ash contents of the treated and untreated sample were determined.

The results of the treatment are listed in Table 4.1 below.

Table 4.1
Zinc chloride / ethanol effect on mass and ash content of each coal

Coal		Mass (g)		Ash (%)	
		Before	After	Before	After
Greenside	(+25)	5,0446	5,0316	12,3	12,2
Greenside	(-25)	10,0618	9,8659	20,7	19,8
Grootegeluk	(+25)	4,9953	4,9805	32,1	32,3
Grootegeluk	(-25)	10,2158	10,0827	45,1	45,3
Rietspruit	(+25)	5,0725	5,0663	17,5	17,6
Rietspruit	(-25)	10,1436	9,9043	27,2	26,7

From these results it can be seen that the mass loss for the +25 μm size fractions was negligible ($\leq$0,30 %). For the -25 μm size fractions, Greenside and Rietspruit coal showed a mass loss of 2 % while the Grootegeluk coal lost only 0,9 % on zinc chloride/ethanol treatment.

It is also apparent that the ash values for the +25 μm fractions of the three coals were not affected by the zinc chloride treatment. In the case of the -25 μm fractions, the ash content of the Grootegeluk coal also remained the same. The ash contents of Greenside and Rietspruit coals, however, decreased from 20,7 % to 19,8 % and 27,2 % to

26,7 %, respectively.

It can thus be concluded that the coarser fractions (+25 μm) in terms of mass and ash contents, are not affected by zinc chloride and ethanol, but the -25 μm size fractions appear to be slightly leached of ash. Consequently the washability curves will predict slightly better density separations than are physically possible.

4.2 EFFECT OF BRIDGING OIL AND ACETONE ON COAL SAMPLES

The effect of bridging oil and acetone on the mass and ash contents of the three coals was investigated by subjecting small quantities of the -25 μm size fractions of the subsamples milled to 95 % passing 45 μm, to these reagents for long periods of time.

7,5 gram of each coal was weighed accurately into the agglomeration vessel and wetted with 75 ml water. After a 5 minute conditioning time, Shellsol AB (35 % m/m) was added. The pulp was agitated for 2 to 6 minutes (until agglomeration was complete) and allowed to stand for 30 minutes before filtering off the water, using a Buchner filter. The filter cake was washed with four 50 ml aliquots of acetone, then submerged in acetone (100 ml) and allowed to stand (covered) for 30 minutes. The acetone was filtered off, the cake washed with several 50 ml aliquots of water, dried at 105^{0}C and weighed. The ash content of the sample before and after the bridging oil/acetone treatment was recorded.

The results of this treatment are presented in Table 4.2 below.

Table 4.2
Effect of bridging oil / acetone on the -25 μm fractions of coals milled to 95 % passing 45 lm

Coal	Mass (g)		Ash (%)	
	Before	After	Before	After
Greenside	7,5021	7,4919	20,7	20,5
Grootegeluk	7,5064	7,5065	45,1	45,1
Rietspruit	7,5028	7,4950	27,2	27,1

It is clear that there is no mass loss (≤0,1 %) on extended exposure of the sample to bridging oil and acetone, nor is there any change in the ash content. It can therefore be concluded that these reagents do not have a leaching effect on the coal samples which could influence the experimental data.

4.3 **WASHABILITY ANOMALY**

In preliminary experiments, an unusual trend was observed in the washability data for the -25 μm fractions of the Greenside coal samples. At low relative densities (R.D.<1,40) the washability curves rose, suggesting that low yields of high ash coal, obtainable also in larger quantities at higher densities, were being obtained.

Agglomeration experiments on the float fractions at of 1,35 R.D. confirmed incomplete separation. A typical example is described below.

A small quantity of the -25 μm size fraction of the subsample of Greenside coal milled to 95 % passing 75 μm,

was subjected to cumulative float and sink analysis (as described in section 3.4.3.1 above). The results are presented in the form of a cumulative floats curve in Figure 4.1. The curve is U-shaped and thus uncharacteristic of a washability curve. Contamination of the floats in the low ash, low yield region of the curve was suspected.

To substantiate this, sufficient float material at 1,35 R.D. was collected by means of the abovementioned float and sink method, and subjected to oil agglomeration (as described in section 3.4.4). Shellsol AB was used as bridging oil at a concentration of 55 % by mass of sample. The results, together with the results of the cumulative float and sink analysis at 1,35 R.D. are given in Table 4.3.

Table 4.3

Agglomeration and float/sink results at relative density 1,35 for the -25 μm fraction of Greenside coal milled to 95 % passing 75 μm

Method	Feed Ash (%)	Float/Aggl. Ash (%)	Sink/Tail Ash (%)	Yield (%)	Discard (%)
F/S	21,5	9,7	23,6	17,41	83,69
AGGL.	9,7	4,6	83,4	93,54	6,46

where F/S = float and sink analysis
AGGL.= oil agglomeration

As may be seen, 93,54 % of the material which floated at 1,35 R.D. was recovered as agglomerates with a much lower ash content (4,6 % ash) than the original floats at 1,35 R.D. (9,7 % ash). The agglomeration showed that 6,46 % of very high ash material (83,4 % ash) was still present in the floats at relative density of 1,35. This represented only about 1 % of the feed mass and consequently affected the cumulative floats curves in the low ash, low yield region to

a much greater extent than in the high ash, high yield region.

The problem was overcome by using the sequential float and sink method, described in section 3.4.3.1 (b) (ii) above. By performing a float and sink analysis at a relative density of 1,30 on a sufficiently large sample, most of the contaminants were removed. Their effect on the ash content of the floated fractions decreased rapidly with increasing density. The results of the sequential method were found to overlap with those of the cumulative method at a relative density of 1,50 (see Figure 4.1 below). Hence it was concluded that the cumulative float and sink method could be used with confidence in the higher relative density range (1,50 to 1,70).

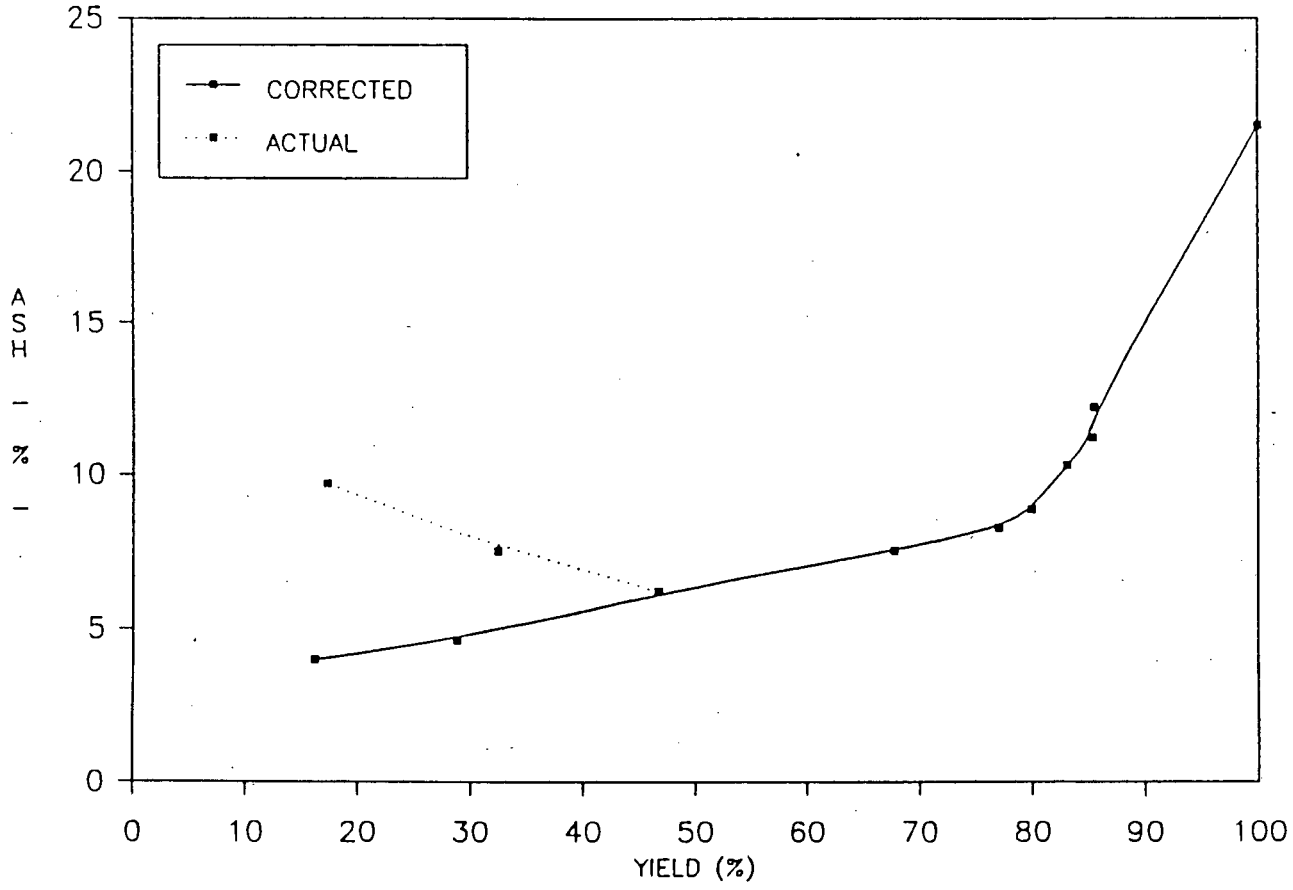


Figure 4.1 Cumulative floats curve for the -25 μm fraction of the Greenside sample milled to 95 % passing 75 μm - anomaly correction

CHAPTER 5

RESULTS AND DISCUSSION

In this chapter, the results of the experimental work carried out on the 'as received' samples of the Greenside, Rietspruit and Grootegeluk coals and on the subsamples milled to 95 % passing 150, 75 and 45 μm are presented. The results are discussed in terms of size and ash distributions, relative density distributions, washability characteristics and liberation efficiencies. The washability characteristics of the -25 μm size fractions are compared with the results of oil agglomeration tests carried out on the same samples, while the washability characteristics of some of the +25 μm size fractions are compared with the results of float and sink analyses carried out by a different method in an independent laboratory.

5.1. SIZE AND ASH DISTRIBUTIONS

The as received ("as is") samples and the milled subsamples of each coal were screened into +25 μm and -25 μm size fractions. Each of these size fractions was analysed for ash content. In addition, detailed size analyses were performed on the -25 μm fractions of all the samples.

The sections below compare and discuss the overall size and ash distributions of the "as is" and milled samples, and the detailed size analyses of the -25 μm fractions, and speculate on the liberation that might be occurring on progressive size reduction.

5.1.1. **Overall comparisons**

The proportions of +25 μm and -25 μm material and the corresponding ash values of the milled and unmilled samples of the three coals are given in Table 5.1 below.

Table 5.1
Size distributions and corresponding ash values for Greenside, Rietspruit and Grootegeluk coals; "as is" and milled to 95 % passing 150, 75 and 45 μm

	Particle	Mass(%)		Ash(%)	
Coal	Size (μm)	+25μm	-25μm	+25μm	-25μm
	As is	67,9	32,1	17,0	25,2
Greenside	95% -150	60,0	40,0	16,5	22,9
	95% - 75	44,7	55,3	16,5	21,5
	95% - 45	17,0	83,0	12,6	21,0
	As is	69,6	30,4	24,6	30,2
Rietspruit	95% -150	60,4	39,6	24,4	29,2
	95% - 75	44,5	55,5	24,1	28,1
	95% - 45	9,1	90,9	17,5	27,2
	As is	58,8	41,2	33,6	53,9
Grootegeluk	95% -150	58,3	41,7	33,8	53,0
	95% - 75	43,8	56,2	33,9	48,5
	95% - 45	25,5	74,5	32,4	45,2

5.1.1.1 Size fractions

A general trend of increasing proportion of -25 μm material with prolonged milling times may be observed in all three coals. It can also be seen that the size distributions for all the milled subsamples were roughly the same for the various degrees of fineness, with the exception of the sample milled to the finest size (95 % passing 45 μm).

Marked increases in the proportions of -25 μm material produced were observed when the coals were milled to 95 % passing 45 μm.

When each coal was milled to 95 % passing 150 μm, about 40 % of the product was finer than 25 μm. This increased to approximately 55 % when the samples were milled to 95 % passing 75 μm. On milling to 95 % passing 45 μm, the proportion of -25 μm material increased dramatically, by between 20 % and 35 %. In the case of Grootegeluk coal the mass (%) increased to 74,5 % while Greenside coal increased to 83 % and Rietspruit coal to 90,9 %.

Table 5.1 also shows how the proportions of +25 μm material decreased on further milling. As each coal was milled to a finer top size, an increasing amount of +25 μm material moved into the -25 μm size fraction. The most noticeable reduction in the +25 μm fraction was observed for Rietspruit coal. On milling to 95 % passing 45 μm, the +25 μm fraction decreased from 69,6 % (for the unmilled coal) to 9,1 %. The corresponding figures for Greenside and Grootegeluk coals are 67,9 % to 17,0 % and 58,8 % to 25,5 %, respectively.

The relatively large proportion of +25 μm material remaining in the Grootegeluk coal can possibly be ascribed to the high ash content (42 %) as well as the high vitrinite content (82,5 % by volume) of this coal. Generally, the ash forming mineral matter in coal is much harder than the organic components and is therefore more resistant to milling than the organic components. Rietspruit coal contains 26 % ash and Greenside coal only 19,6 %.

In terms of maceral content, Greenside and Rietspruit coals contain only (approximately) 30 % vitrinite and 66 to 67 % inertinite. Although vitrinite is hard and brittle, and fractures easily (see Table 2.1) it generally contains very

little occluded mineral matter. The inertinite in South African coals contains much finely disseminated mineral matter (Falcon, 1978) which weakens the lattice and makes it more susceptible to fracture. The Greenside and Rietspruit coals, being inertinite rich, are likely to be more friable than the Grootegeluk coal and to break into smaller fragments. Greenside appears to be harder than Rietspruit coal, and this may be due to its lower ash content (19,6 %). The higher ash content of Rietspruit coal (26 %), if associated with inertinite, may weaken the structure further, making the coal more 'millable' than Greenside.

5.1.1.2 Ash contents

Table 5.1 also shows the ash contents of the +25 μm and the -25 μm size fractions of the milled and unmilled samples. There is a remarkable difference in ash contents between the +25 μm and -25 μm fractions of all three coals.

The ash content of the +25 μm fraction of the "as is" Greenside sample was 17,0 %, while that of the -25 μm fraction was 25,2 %. These values decreased to 12,6 % and 21,0 %, respectively, in the sample milled to 95 % passing 45 μm.

Likewise, in the case of the Rietspruit coal, the +25 μm fraction of the "as is" sample had an ash content of 24,6 % compared to 30,2 % in the -25 μm fraction. These values decreased to 17,5 % and 27,2 % in the respective size fractions of the subsamples milled to 95 % passing 45 μm.

The largest difference in ash between the coarse and fine fractions was observed for Grootegeluk coal. Here the +25 μm fraction of the "as is" sample had an ash value of 33,6 % while the -25 μm fraction contained 53,9 % ash. (This coal could therefore be beneficiated considerably by

classification alone). It is interesting to note that the difference in ash content narrowed from 33,6 % and 53,9 % in the +25 μm and -25 μm fractions respectively of the "as is" sample to 32,4 % and 45,2 % in the respective size fractions of the subsample milled to 95 % passing 45 μm. This may be due to some clean coal preferentially moving into the finer fraction as a result of prolonged grinding.

With prolonged milling the ash contents of the -25 μm fractions decreased gradually. This reflects the movement of "better quality" +25 μm material into the -25 μm size fraction on progressive size reduction. However, the absolute difference in ash content between the +25 μm and -25 μm fractions remained: this suggests the selective breakage of the higher ash material in the +25 μm size fraction into the -25 μm fraction on size reduction. Calculations confirm that the "better quality" material was by no means good quality or low-ash coal; it was material with a marginally higher ash content than the average of the +25 μm fraction. For a starting mass of 100 g of "as is" Greenside coal, ash balance shows that 50,9 gram with an average ash content of 18,4 % transferred from the +25 μm fraction to the -25 μm size fraction by the time the subsample was milled to 95 % passing 45 μm. Out of 100 gram of "as is" Rietspruit coal 60,5 gram with an average ash content of 25,1 % moved from the +25 μm fraction into the -25 μm fraction on milling the sample to 95 % passing 45 μm. For Grootegeluk coal the figure amounted to 33,3 gram of coal containing 34,4 % ash.

As a result of this movement there were steady and noticeable changes in the ash values of the -25 μm fractions as the samples were milled progressively finer. Going from the original sample of the Greenside coal to that milled to 95 % passing 45 μm, the ash contents of the -25 μm fractions decreased from 25,2 to 22,9, 21,5 and 21,0 %; while

Rietspruit and Grootegeluk showed decreases from 30,2 % to 29,2, 28,1 and 27,2 % and 53,9 % to 53,0, 48,5 and 45,2 %, respectively.

In the +25 μm fractions there were only small differences between the ash contents of the original samples and those milled to 95 % passing 75 μm. Further milling to 95 % passing 45 μm brought about considerable changes in the measured ash contents of the +25 μm fraction of the Greenside and Rietspruit coals, but not of the Grootegeluk coal. The ash contents of Greenside decreased from 16,5 % to 12,6 %, and Rietspruit from 24,1 % to 17,5 %. The ash contents of the +25 μm fraction of Grootegeluk coal, however, changed only slightly from 33,9 to 32,4 %. For the Greenside and Rietspruit coals, higher ash material was broken in going from 95 % passing 75 μm to 95 % passing 45 μm, than from going from "as is" to 95 % passing 75 μm.

5.1.2. **Size distribution in -25 μm size fractions**

Size analyses were performed on the -25 μm fractions of the milled and unmilled samples of all the coals, using the Malvern 2600/3600 Particle Sizer VF.6. Detailed results are presented in Table A1 in Appendix A.

In order to observe general size distribution trends within the -25 μm size fractions of the three coals, these results are best expressed in the form of histograms. Figures 5.1 to 5.3 below represent the results contained in Table A1 in histogram form. Each bar represents the mass percent of material found in a particular size fraction of the -25 μm portion of the milled and unmilled samples of the three coals.

From these results it appears that within the -25 μm fractions the proportions of -23,5+10,5 μm material increased with progressive milling while the proportions of the finer fractions (-10,5+5,0 μm and -5,0+1,2 μm) decreased steadily.

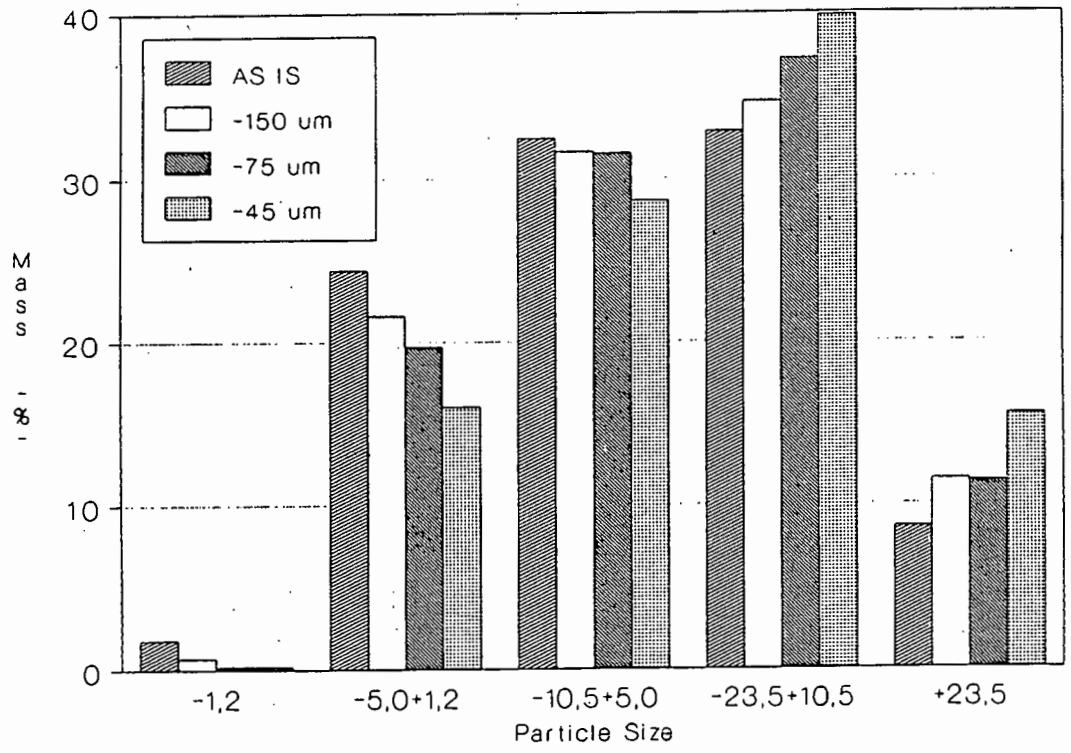


Figure 5.1 Size Distribution of the -25 μm fraction of Greenside coal milled to various top sizes

Figure 5.1 shows the results for the Greenside sample. On milling this sample to 95 % passing 150, 75 and 45 μm, the proportion of -23,5+10,5 μm material, as a percentage of the -25 μm material, increased steadily from 32,8 to 39,8 %. The proportion of +23,7 lm material also increased, from 8,6 to 15,5 %. At the same time, the proportion of -10,5+5,0 μm material decreased gradually from 32,4 to 28,9 % and of -5,0+1,2 μm material from 24,4 to 16,0 %.

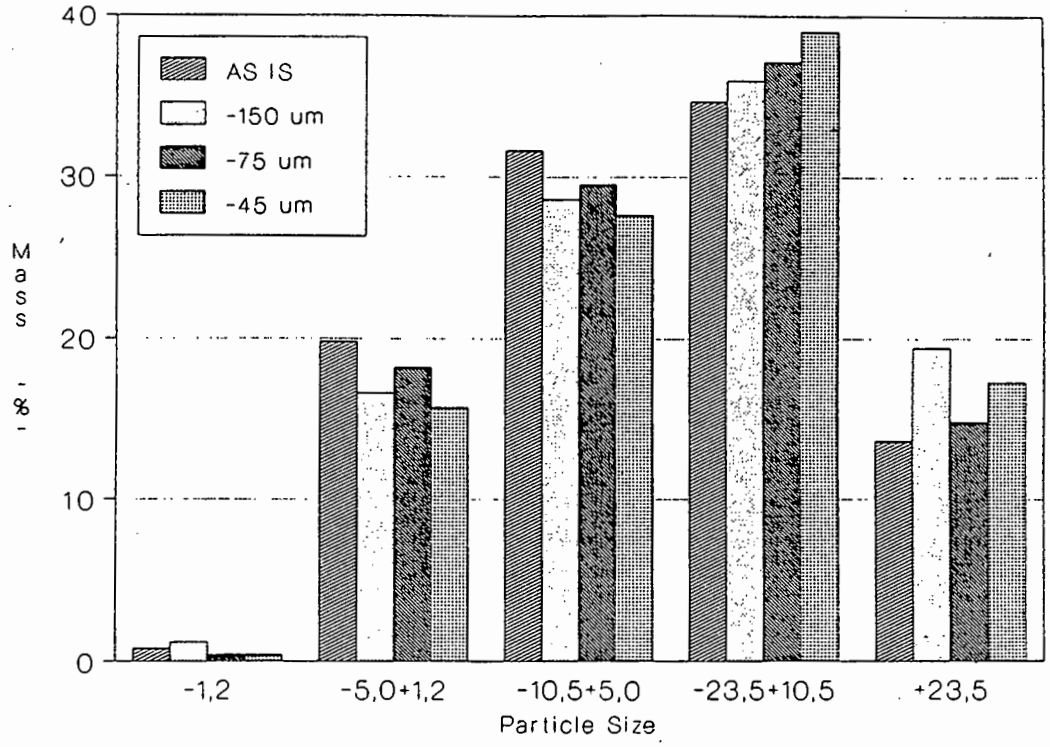


Figure 5.2 Size distribution of the -25 μm fraction of Rietspruit coal milled to various top sizes

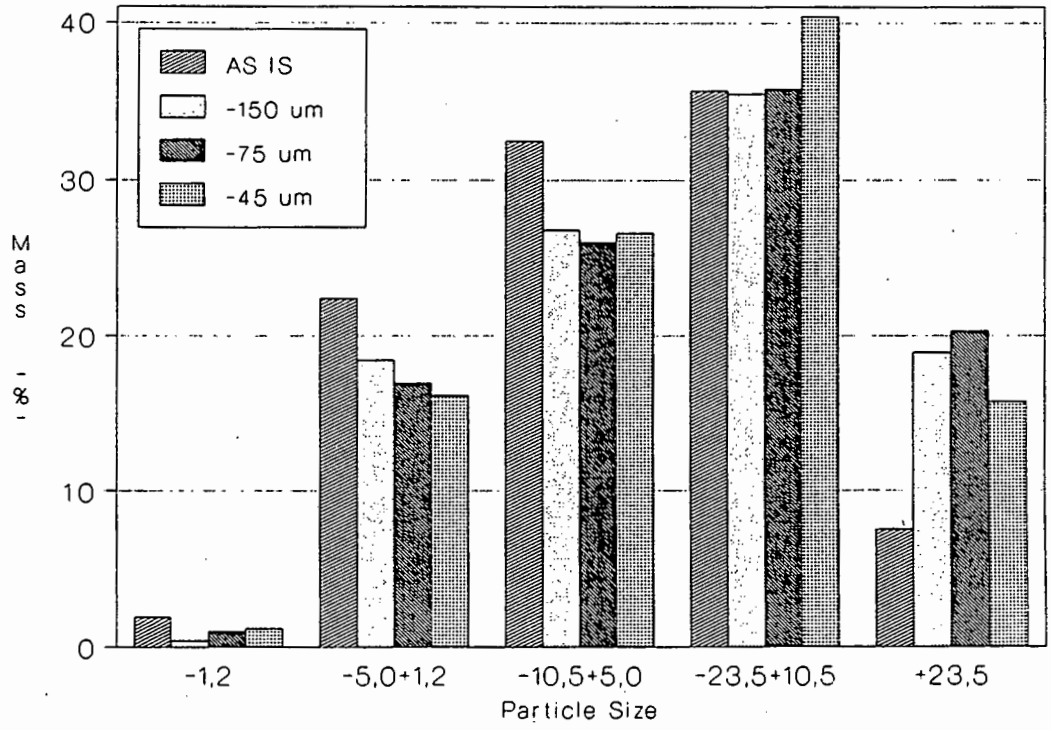


Figure 5.3 Size distribution of the -25 μm fraction of Grootegeluk coal milled to various top sizes

Figure 5.2 represents the size distributions within the -25 μm fractions of the milled and unmilled Rietspruit coal. The pattern is similar to that of the Greenside coal in that the proportion of -23,5+10,5 μm increased steadily with milling, although more gradually (34,4 to 39,0 %), while the proportions in the finer size fractions -10,5+5,0 μm and -1,2+5,0 μm decreased from 31,4 to 27,6 % and 19,8 to 15,7 %, respectively. The proportions of -1,2 μm material, as in the case of Greenside coal, were negligible.

The size distributions in the -25 μm fractions of the Grootegeluk coal are given in Figure 5.3. This indicates that the proportion of -23,5+10,5 μm material remained constant at 35,7 % before increasing to 40,4 % when the sample was milled to 95 % passing 45 μm. The proportions of -10,5+5,0 μm material decreased from 32,5 to 26,8 % on milling to 95 % passing 150 μm and remained virtually the same during further size reduction. The -5,0+1,2 μm fraction decreased steadily with further milling while the -1,2 μm fraction was again of negligible proportion.

The effect of progressive milling on the -25 μm size fractions may be more apparent from the absolute values. As an example the absolute values for Greenside coal will be discussed here. The corresponding values for Rietspruit and Grootegeluk coal may be found in Appendix A.

Consider 100 gram of Greenside ("as is") feed. From Table 5.1, this contains 32,1 gram of -25 μm material. Then, using the data in Table A1, it may be calculated that of the 32,1 gram, 2,76 gram are coarser than 23,5 μm and 10,53 gram fall within the -23,5+10,5 μm size range. The -10,5+5,0 μm and -5,0+1,2 μm size fractions account for 10,40 and 7,83 gram, respectively. Only 0,58 gram fall in the -1,2 μm fraction. These values are listed in Table 5.2 below,

together with the corresponding values for the milled samples.

Table 5.2
Size distribution in the -25 μm size fractions of Greenside coal, based on 100 gram of total feed

Size Fraction (μm)	Mass in Size Fractions (gram)			
	As Is	95 % -150 μm	95 % -75 μm	95 % -45 μm
+23,5	2,76	4,60	6,30	12,87
-23,5+10,5	10,53	13,84	20,57	33,03
-10,5+ 5,0	10,40	12,64	17,42	23,74
- 5,0+ 1,2	7,83	8,64	10,89	13,28
-1,2	0,58	0,28	0,11	0,17
Total (-25 μm)	32,10	40,00	55,29	83,00

From the absolute values in Table 5.2 it may be seen that on milling Greenside coal to progressively finer sizes, the mass increases in the coarser size fractions are considerably larger than those of the finer fractions. The +23,5 μm fraction increases sixfold from 2,76 gram in the "as is" sample to 12,87 gram in the sample milled to 95 % passing 45 μm. The proportion of -23,5+10,5 μm triples from 10,53 to 33,03 gram and the -10,5+5,0 μm fraction doubles from 10,40 to 23,74 gram. The -5,0+1,2 μm fraction increases more gradually, from 7,83 gram in the "as is" sample to 8,64 gram (95 % -150 μm), to 10,89 gram (95 % -75 μm) and finally to 13,28 gram in the sample milled to 95 % passing 45 μm. The amounts of -1,2 μm material remain negligible throughout.

Prolonged milling therefore results in the production of predominantly 10 to 20 μm material but does not reduce the available fines to smaller particle sizes. This may be due to the type of mill (rod mill) used. Since most clay

impurities are 1 to 2 μm in size they cannot be liberated effectively by milling to 95 % passing 45 μm.

The same trends may be observed for the Rietspruit and Grootegeluk coals (Tables A2 and A3, Appendix A). From all these results it would appear that the top size would have to be reduced to considerably less than 45 μm in order to observe marked increases in the proportions of the finer size fractions (-5,0 μm) or a different method of milling would have to be employed.

5.2. RELATIVE DENSITY DISTRIBUTIONS

The density distributions of the original and milled coal samples over the relative density range 1,35 to 1,70 are presented below in the form of frequency histograms. Each coal will be discussed separately. Float and sink analyses were carried out separately on the +25 μm and -25 μm size fractions of each coal. Detailed results are presented in Appendix B (cumulative yields) and Appendix C (cumulative ash contents).

5.2.1. Greenside coal

Figure 5.4 presents the relative density distributions for the +25 μm and -25 μm fractions of the "as is" Greenside coal. Figure 5.5 presents the same relative density distributions for the subsample milled to 95 % passing 45 μm.

The characteristics to be seen in Figure 5.4 are clearly the same as those observed by Harris (1987) - see Figure 2.7 on page 32. At 1,35 relative density, a much greater proportion of the +25 μm size fraction of the "as is"

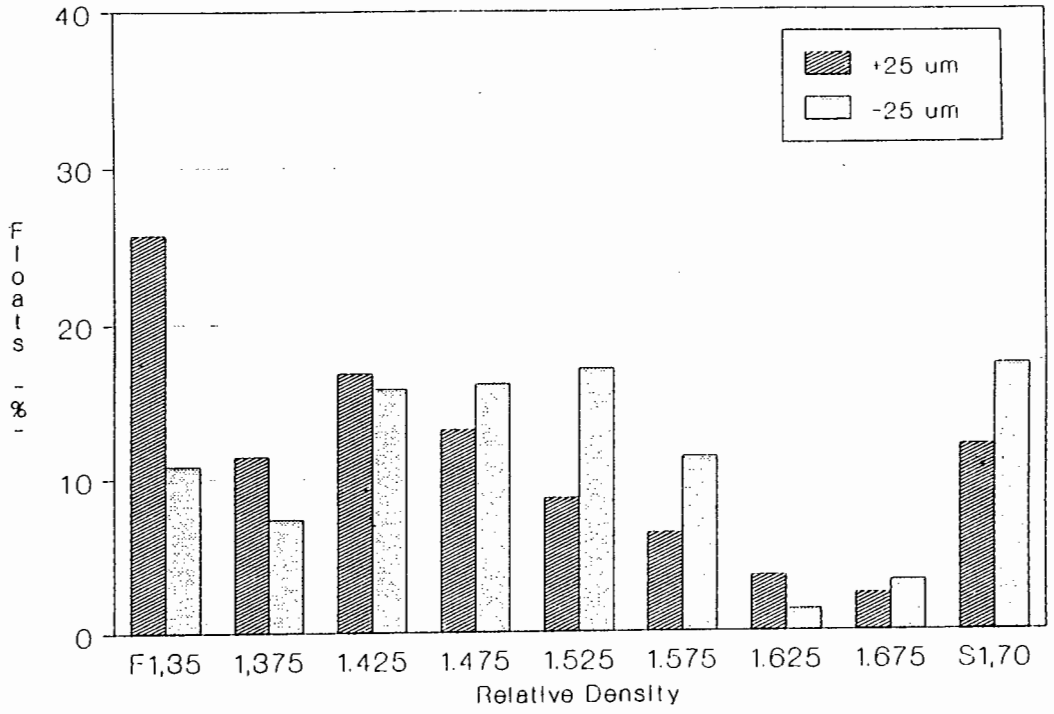


Figure 5.4 Density distributions of the +25 μm and -25 μm fractions of "as is" Greenside coal

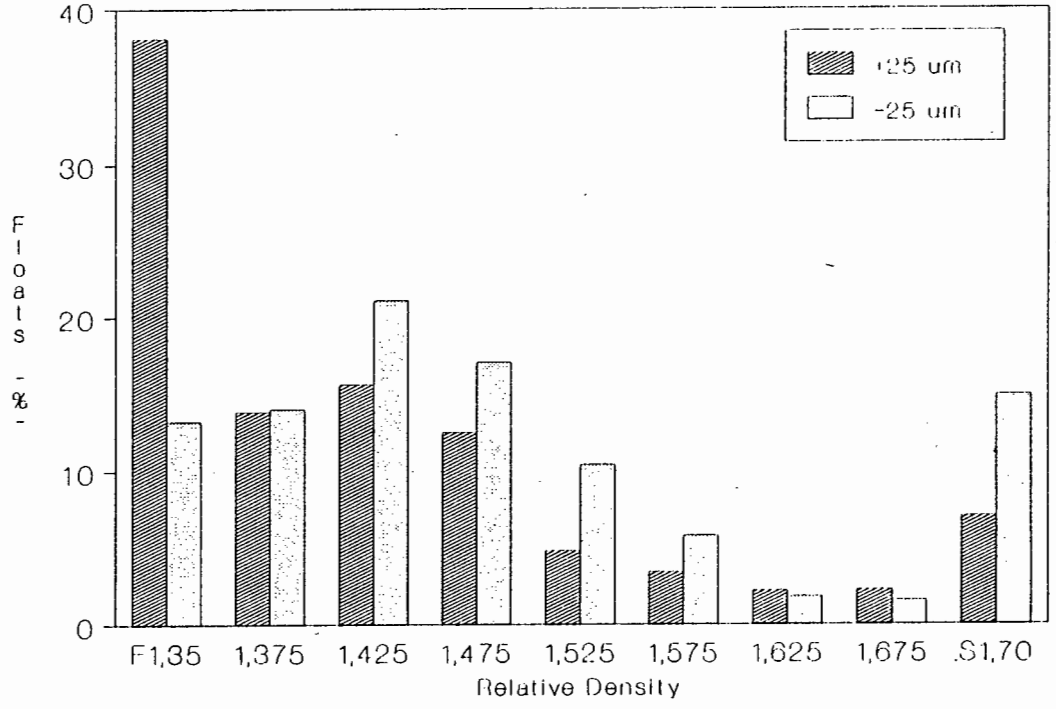


Figure 5.5 Density distributions of the +25 μm and -25 μm fractions of Greenside coal milled to 95 % passing 45 μm

Greenside sample floated than of the -25 μm size fraction. In the 1,35 - 1,40 relative density fraction, the difference was not as great, but there was still more of the +25 μm fraction that floated than of the -25 μm fraction. At 1,40 - 1,45 relative density this difference was almost whittled away to nothing. In the 1,45 - 1,60 relative density range the trend was reversed; in the floats at each of the relative density intervals, a greater proportion of the -25 μm fraction was present than of the +25 μm fraction. A very small proportion of either size fraction was present in the 1,60 - 1,70 relative density range. Of the sinks at 1,70 relative density, the proportion of the -25 μm size fraction present was greater than of the +25 μm size fraction.

Going from Figure 5.4 to Figure 5.5 shows the effect of milling a subsample of the "as is" coal to 95 % passing 45 μm. While at first sight the distributions in the two graphs appear similar, closer observation reveals some interesting differences.

Firstly, the relative difference between the proportions of the +25 μm and the -25 μm size fractions floating at 1,35 relative density was greatly enhanced on reducing the sample to 95 % passing 45 μm. That is, a far greater proportion of the +25 μm size fraction of the milled sample floated at 1,35 relative density than of the +25 μm size fraction of the unmilled sample. This suggests that on milling the Greenside coal, more of the +25 μm material that would float at 1,35 relative density remained unbroken (on average) than material that would appear in other relative density intervals.

The second interesting difference is that the reverse applied to the sinks at 1,70 relative density. This would suggest that on milling, more of the +25 μm material that

would sink at 1,70 relative density was broken (on average) to -25 μm than material that would appear in other density fractions.

Thirdly, on milling, there was a marked shift in the relative density distribution of the middlings of the -25 μm material, to lower relative densities. Whereas, in the unmilled sample, the middlings in the -25 μm size fraction were predominantly in the 1,40 - 1,60 relative density range, in the subsample milled to 95 % passing 45 μm most of the middlings in the -25 μm fraction were in the 1,40 - 1,50 relative density range. This feature is also apparent in the results of Harris (1987), although he did not comment upon it.

The change, on progressively finer milling, in the relative density distributions of the +25 μm and -25 μm size fractions of the Greenside coal may be seen from Figures 5.6 and 5.7, respectively. These Figures reinforce the observations made above, i.e. that the proportions of low density (<1,35 R.D) material were considerably higher (25 to 38 %) in the +25 μm size fractions than in the -25 μm size fractions (11 to 16 %); that the proportions of high density (>1,70 R.D) material were lower (7 to 13 %) in the +25 μm size fractions than in the -25 μm size fractions (15 to 18 %); and that there was a greater concentration of light middlings material in the -25 μm fraction than in the +25 μm fraction.

The steady increase in the proportion of light middlings material in the -25 μm fraction may be seen from the histograms in the 1,35 to 1,45 relative density intervals in Figure 5.7. As the Greenside coal sample was milled to 95 % passing 150, 75 and 45 μm, progressively larger proportions of the -25 μm material reported to these intervals.

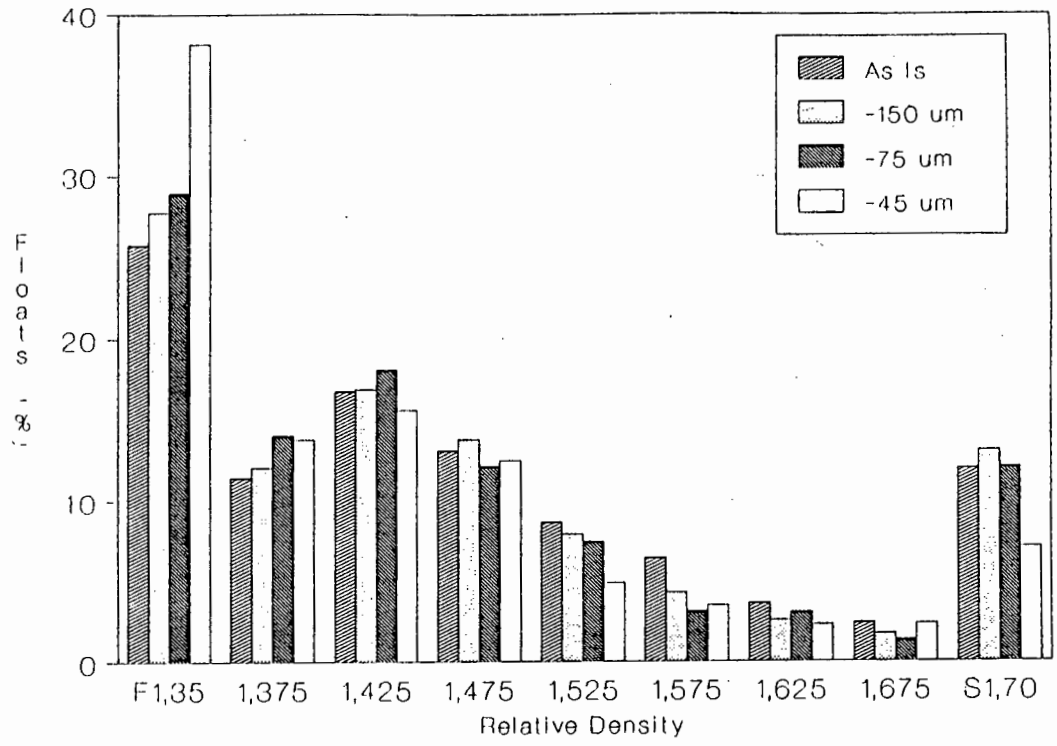


Figure 5.6 Density distributions of the +25 μm fractions of Greenside coal milled to various top sizes

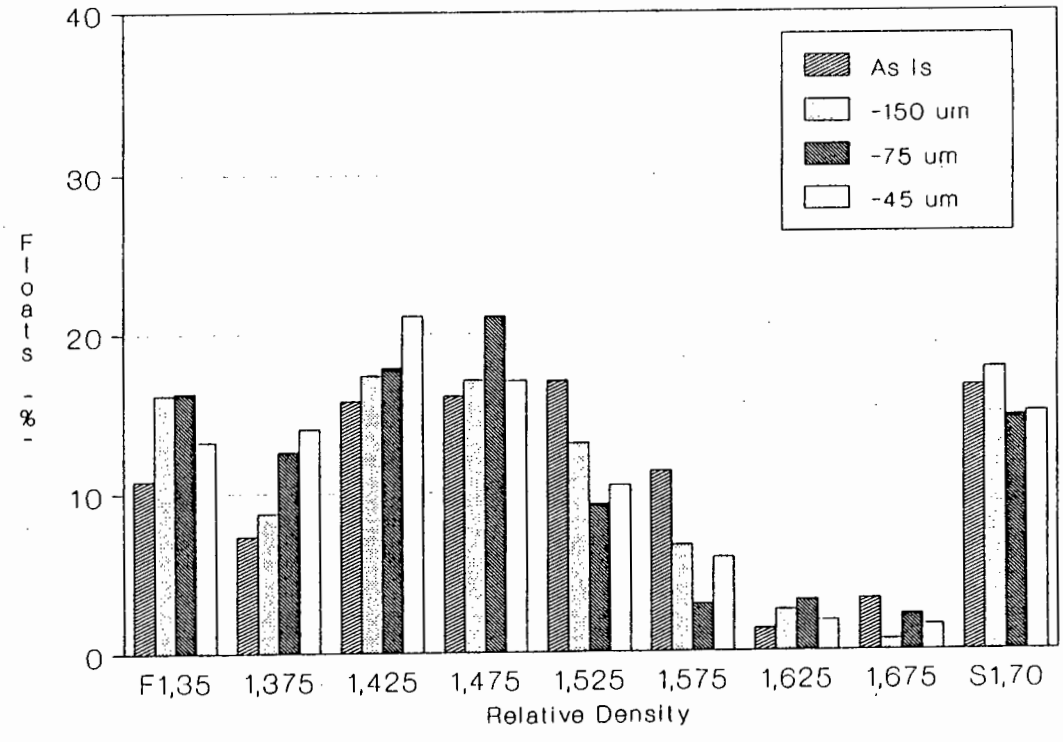


Figure 5.7 Density distributions of the -25 μm fractions of Greenside coal milled to various top sizes

From Figure 5.6 it may be seen that when the coal was milled to finer sizes, the proportions of the +25 μm material in the low relative density intervals increased. The increase in the <1,35 relative density interval was particularly dramatic. At the same time, the proportions in the >1,50 relative density intervals decreased, indicating that this material was selectively being broken into the -25 μm fractions. As relative density is directly related to ash content in coal, this suggests that a greater proportion of the higher ash material in the +25 μm fraction was being broken into the -25 μm fractions, than of the lower ash material. Consequently, one would expect the ash content of the +25 μm fractions to decrease as the Greenside sample was milled finer, a fact which was already observed above. (Section 5.1.1.2 and Table 5.1).

Possibly the most informative way of expressing the relative density distribution data discussed above is in absolute terms, on the basis of a fixed mass of original material. Table 5.3 presents the relative density distributions of the +25 μm and -25 μm fractions of the milled and unmilled samples, based on 100 grams of original "as is" material. These values were calculated in the following way.

From the size analysis of the "as is" sample (Table 5.1) the ratio of the +25 μm and -25 μm fractions was known to be 67,9 : 32,1. Thus, for 100 grams of starting material, there would be 67,9 gram in the +25 μm and 32,1 gram in the -25 μm fraction. The mass of +25 μm material in each relative density interval was calculated by applying the relative density distribution data in Table B1 in Appendix B to 67,9 grams. For example, the mass of +25 μm material in the 1,45 to 1,50 relative density interval was 0,1311 * 67,9 = 8,9 gram. Corresponding values for the -25 μm material were calculated using the other data in Table B1.

Relative Density	"As Is"			95%-150um			95%-75um			95%-45 um		
	+25 um	-25 um	Total	+25 um	-25 um	Total	+25 um	-25 um	Total	+25 um	-25 um	Total
F1,35	17,46	3,47	20,93	16,64	6,47	23,11	12,93	9,00	21,93	6,49	10,97	17,46
1,35 - 1,40	7,77	2,35	10,12	7,26	3,50	10,76	6,28	6,96	13,24	2,35	11,63	13,98
1,40 - 1,45	11,39	5,05	16,44	10,13	6,95	17,08	8,08	9,87	17,95	2,66	17,50	20,16
1,45 - 1,50	8,90	5,15	14,05	8,29	6,83	15,12	5,41	11,65	17,06	2,12	14,17	16,29
1,50 - 1,55	5,86	5,45	11,31	4,73	5,22	9,95	3,30	5,10	8,40	0,82	8,64	9,46
1,55 - 1,60	4,33	3,62	7,95	2,56	2,65	5,21	1,38	1,64	3,02	0,59	4,86	5,45
1,60 - 1,65	2,44	0,44	2,88	1,52	1,02	2,54	1,35	1,75	3,10	0,38	1,54	1,92
1,65 - 1,70	1,62	1,04	2,66	1,01	0,27	1,20	0,58	1,26	1,84	0,39	1,31	1,70
S1,70	8,13	5,53	13,66	7,86	7,09	14,95	5,39	8,07	13,46	1,20	12,38	13,58
	67,90	32,10	100,00	60,00	40,00	100,00	44,70	55,30	100,00	17,00	83,00	100,00

Table 5.3 Proportions of +25 μm, -25 μm and composite fractions of Greenside coal on milling to progressively finer sizes - based on 100 gram of original sample

In the same way, the mass of material in each relative density interval was calculated for the +25 μm and -25 μm fractions of the milled samples, using the size analysis data in Table 5.1 and the relative density distributions in Appendix B. Table 5.3 also shows the changing relative distributions of a whole "as is" sample of 100 gram of Greenside coal (the columns headed "Total") on milling to various sizes. These values were obtained by arithmetically reconstituting the "as is" sample and the milled products from the size analyses and relative density distribution data (i.e. by summing the mass of +25 μm and -25 μm material in a particular relative density interval, for the "as is" sample and the milled products).

Table 5.3 may now be used to follow the simultaneous changes in size and density distributions that would occur on milling 100 gram of "as is" Greenside coal. For example, it may be seen that the original sample would contain 17,46 gram of +25 μm material of <1,35 relative density. On milling to 95 % passing 150 μm, only 16,64 gram of this material would remain. On further milling to 95 % passing 75 μm and 95 % passing 45 μm, the mass of +25 μm material of <1,35 relative density would be reduced to first 12,93 gram, and then 6,49 gram. Thus, in all, 10,97 gram of <1,35 relative density material would be broken out of the +25 μm size fraction. At the same time, in the -25 μm size fraction, the mass of <1,35 relative density material would increase steadily from 3,47 gram in the "as is" sample to 10,97 gram after milling to 95 % passing 45 μm. Thus 7,5 gram of <1,35 relative density material would be broken into the -25 μm size fraction. This analysis is simplistic, as the +25 μm material in a particular relative density interval need not necessarily report to the same relative density interval on being reduced in size to -25 μm; movement to other relative densities is possible if liberation has taken place. Thus the values in Table 5.3 in

reality reflect a net movement of material on progressive milling, by size fraction and relative density interval, as a result of both size reduction and liberation.

The values in Table 5.3 are easier to interpret when presented in histogram form, as in Figures 5.8 to 5.10. Figure 5.8 presents, by density interval, the mass of floats in gram in the +25 μm fractions of 100 gram of "as is" coal or milled product. Figure 5.9 presents the corresponding values in the -25 μm fractions. Figure 5.10 presents the changing density distribution of the whole sample as progressive milling occurred.

Figure 5.8 clearly shows how the mass of +25 μm material in each density interval was reduced as the sample was milled further. The changes were relatively small as the sample was milled to 95 % passing 150 and 75 μm with the biggest reduction in each density interval occurring when the sample was milled to 95 % passing 45 μm. This is necessarily so; the total mass of the +25 μm fraction was reduced from 67,9 to 60,0 to 44,7 to 17,0 gram (starting material 100 gram) with increased milling. Figure 5.9 shows how the mass of -25 μm material in each density interval increased on milling.

It is apparent from Table 5.3 and Figures 5.8 and 5.9 that, in the +25 μm size fractions, the reduction of mass in some density intervals was proportionately more than in others, while in the -25 μm fractions, some density intervals gained proportionately more mass than did others. For example, from Figure 5.8, the reduction in the mass of <1,35 relative density material would appear to be proportionately less than the reduction in mass of >1,70 relative density material. From Figure 5.9 the increase in mass in the 1,35 to 1,45 relative density intervals would appear to be more than in the other intervals.

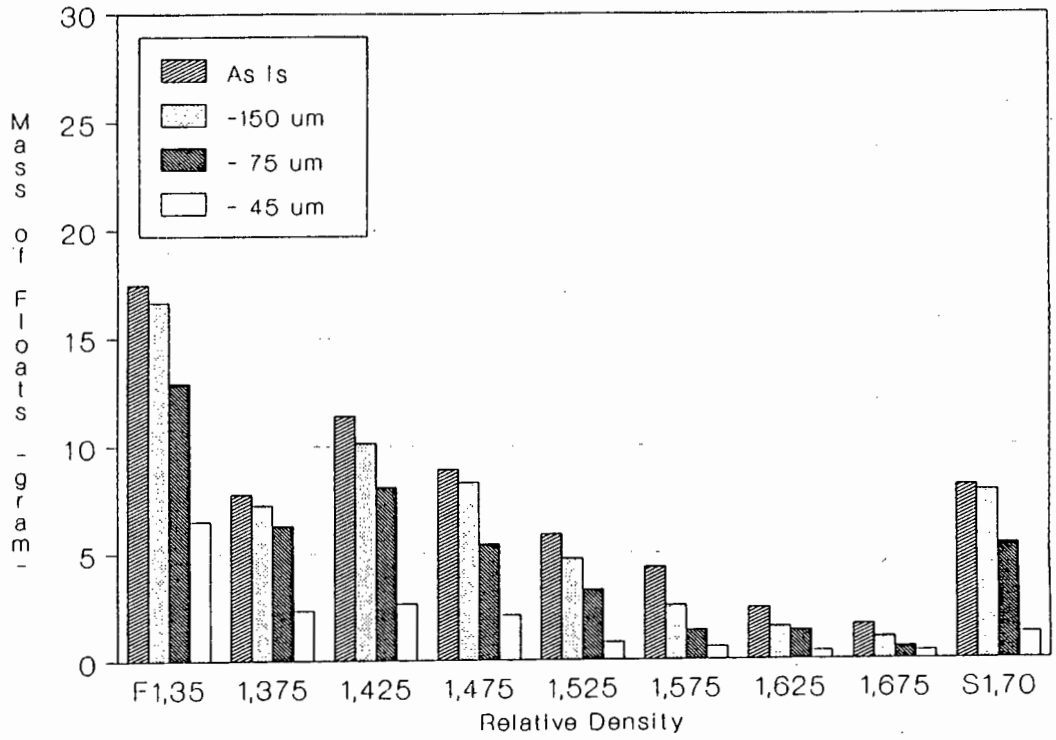


Figure 5.8 Density distribution of the +25 μm fractions of Greenside coal milled to various top sizes (basis: each sample = 100 gram)

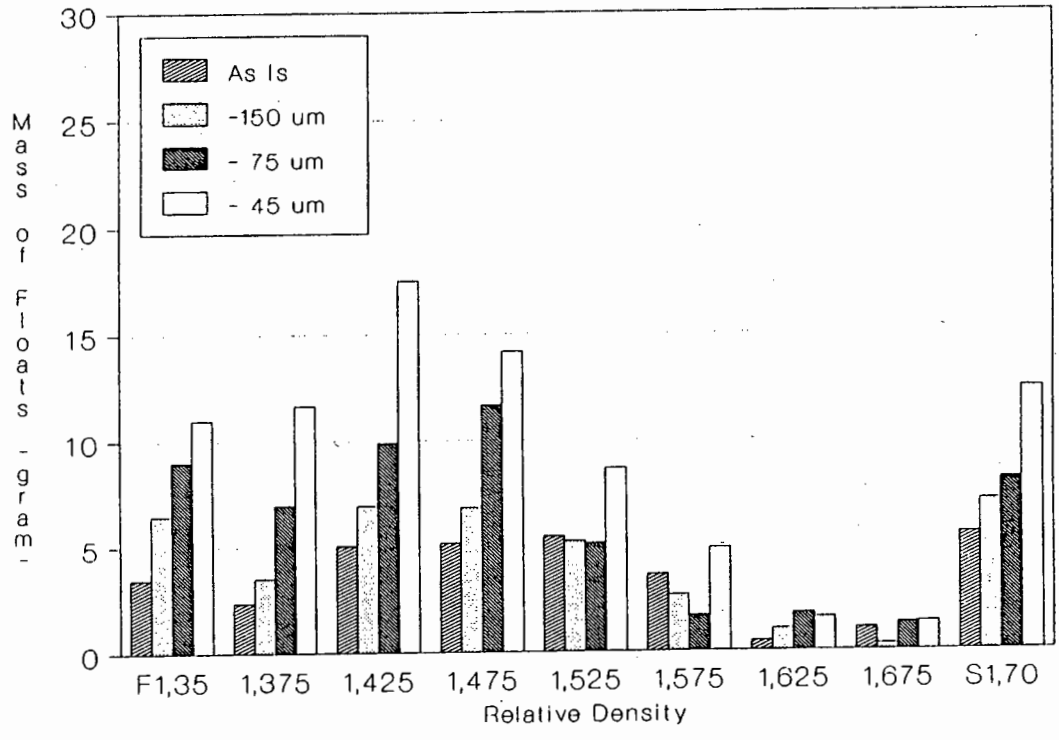


Figure 5.9 Density distribution for the -25 μm fractions of Greenside coal milled to various top sizes (basis: each sample = 100 gram)

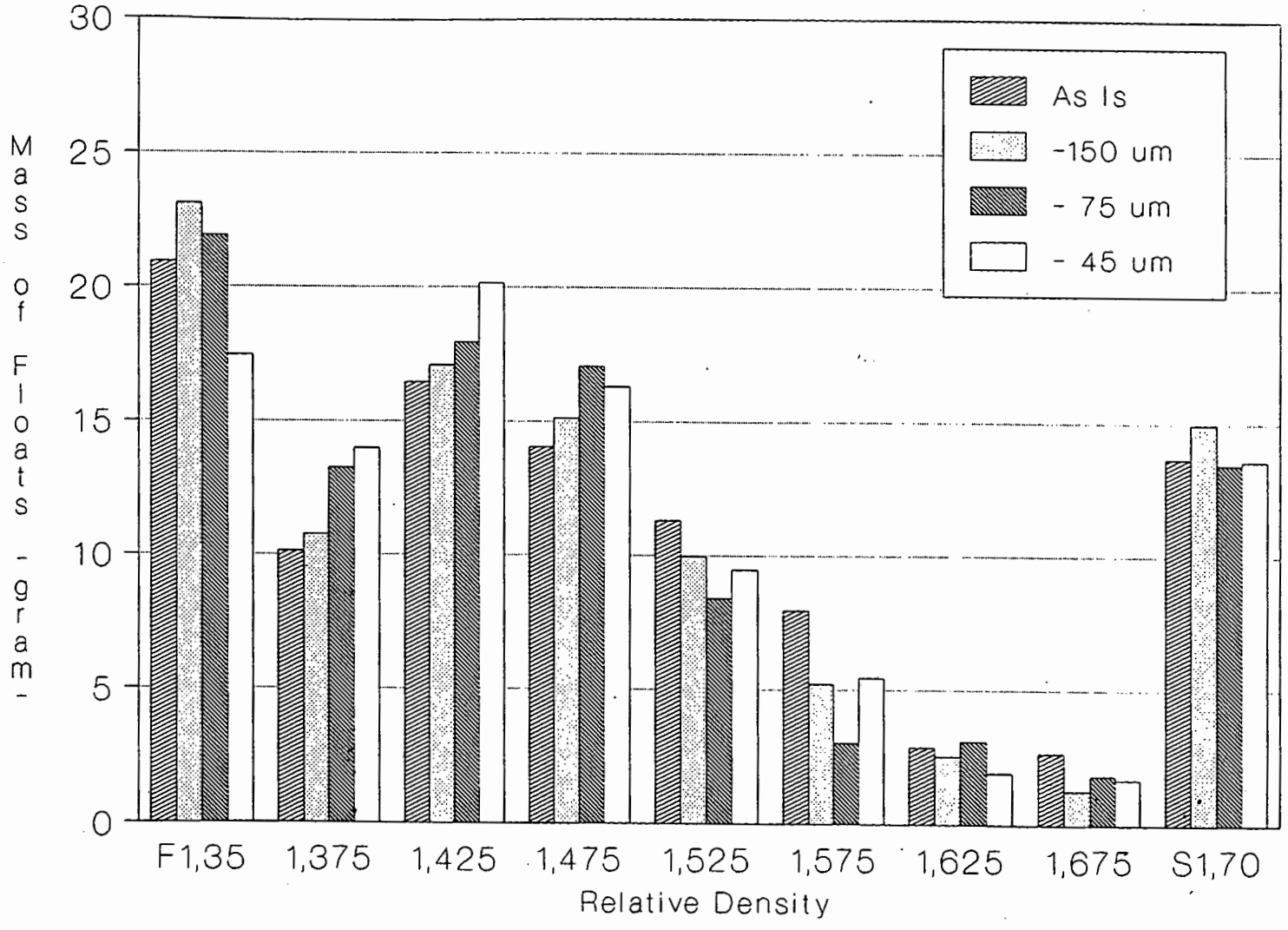


Figure 5.10 Density distribution of Greenside coal milled to various top sizes (basis: each sample = 100 gram)

Table 5.4 presents the calculated proportional decrease in mass of +25 μm material in each relative density interval, together with the proportional increase in mass of -25 μm material.

Table 5.4

Proportional breakage of +25 μm material in Greenside "as is" coal by relative density interval and the proportional increase in mass of -25 μm material on milling to 95 % passing 45 μm

Relative Density	Greenside ("as is") (%) Breakage in +25 μm	Greenside (95 % -45 μm) (%) Increase in -25 μm
F1,35	62,8	216,1
1,35 - 1,40	69,8	394,9
1,40 - 1,45	76,7	246,5
1,45 - 1,50	76,2	175,1
1,50 - 1,55	86,0	58,5
1,55 - 1,60	86,4	34,3
1,60 - 1,65	84,4	250,0
1,65 - 1,70	75,9	26,0
S1,70	85,2	123,9

It is obvious from these figures that the extent of breakage of +25 μm material in the >1,50 relative density intervals was fairly similar - in each case, the mass was reduced to roughly 15 % of the original amount after the sample had been milled to 95 % passing 45 μm. The extent of breakage of material in the 1,40 to 1,45 and 1,45 to 1,50 relative density intervals was lower - in each of these cases roughly 25 % of the original mass remained after the sample had been milled to 95 % passing 45 μm. The extent of breakage of <1,40 relative density material was lower still, with more

than 35 % of the original mass of the lowest density material (<1,35 R.D) remaining after size reduction.

In the -25 μm fraction, the greatest proportional increase in mass occurred in the 1,35 to 1,40 and 1,40 to 1,45 relative density intervals (ignoring the value for 1,60 to 1,65 R.D. which is anomalous). The proportional increase in mass in the 1,50 to 1,70 relative density intervals was particularly low (ignoring, again, the value for 1,60 to 1,65 R.D.). The proportional increase in mass of <1,35 relative density material was significantly higher than for >1,70 relative density material.

This suggests that, taken as a whole, when the original material was milled, +25 μm particles of high density (>1,50 R.D.) were particularly broken, and that the products of breakage appeared in the -25 μm fraction in the 1,35 to 1,45 relative density intervals, more than in others. This may be confirmed by considering Figure 5.10. This shows very clearly that as the sample was milled, the mass of material in the 1,35 to 1,45 relative density interval increased steadily, the mass of 1,45 to 1,50 relative density material also increased, while the mass of <1,35 relative density and 1,50 to 1,70 relative density material decreased. The mass of sinks at 1,70 relative density remained virtually unchanged on milling.

The relative density distributions may also be used, in conjunction with the ash content data from the float and sink analyses, to try to infer the movement of mineral and maceral material as the coal was milled. Table 3.2 gives the proportions of the macerals in the organic part of the coal, in volume percent. These figures can be converted to mass percent, using the known relative densities of the macerals, listed in Table 2.1. Exinite has a relative density of 1,1, while vitrinite has a relative density

between 1,20 and 1,30 and inertinite ranges from relative density 1,40 to 1,60. If an average value of 1,25 for vitrinite, and 1,50 for inertinite are assumed, the maceral content on a mass basis of Greenside coal may be calculated to be 27,64 % vitrinite, 1,24 % exinite and 71,12 % inertinite.

In addition, the average ash content of the Greenside coal calculated by reconstituting the float and sink data of the +25 μm and -25 μm size fractions (see Appendix F) was 19,6 %. Thus, 100 gram of Greenside coal would contain 22,22 gram vitrinite, 1,00 gram exinite and 57,18 gram inertinite. The remaining 19,6 gram constitute ash forming mineral matter. It is assumed in all these calculations that ash is the same as mineral matter. While this is not strictly true, it is used for simplicity and convenience : in addition the error is usually small. This assumption is made consistently throughout this thesis.

Figure 5.10 may now be divided into 'maceral' and 'discard' zones. The <1,35 relative density region may be regarded as the exinite and vitrinite zone, the 1,35 to 1,60 relative density interval the inertinte zone and the >1,60 relative density range the middlings and discard zone. One would expect the <1,35 relative density region to contain mainly liberated exinite and vitrinite and minimal amounts of ash material. The 1,35 to 1,60 relative density interval should contain free (and some locked) inertinite, unliberated vitrinite and ash. The high relative density region (>1,60 R.D.) should contain mainly ash material and unliberated inertinite. Depending on the degree of liberation of the sample, small amounts of unliberated vitrinite may also be present in this zone.

The float and sink data listed in Appendix C (cumulative yield vs. cumulative ash) were fitted using the 'Shareware

Curvefit Version 2.12 A' routine. Smoothed values are given in Appendix E. Using the cumulative yield/cumulative ash data for the +25 μm and -25 μm fractions from Table E1 and the mass fractions from Table 5.3 above, average ash contents and yields were calculated for each of the three zones, based on 100 gram of starting material. The mass of coal in each zone was then allocated to the appropriate maceral, beginning with the <1,35 R.D. zone. The results are presented in Table 5.5 below, for the "as is" sample of Greenside coal, and in Table 5.6 for the sample milled to 95% passing 45 μm. A detailed example of the calculations is given in Appendix F.

Table 5.5

Maceral and ash distribution in Greenside "as is" coal by relative density zone (based on 100 gram of sample)

R.D. Zone	Size Fraction (μm)	Total Mass (g)	Ash Contents (%)	Ash Mass (g)	Coal Mass (g)	Maceral Contents (g)
<1,35	+25	17,46	3,7	0,65	16,81	Ex : 1,00
	-25	3,47	4,9	0,17	3,30	Vit: 19,11
		20,93	4,1	0,82	20,11	20,11
1,35-1,60	+25	38,25	11,8	4,51	33,74	Vit: 3,11
	-25	21,62	15,1	3,26	18,36	In : 48,99
		59,87	13,0	7,77	52,10	52,10
>1,60	+25	12,19	52,2	6,36	5,83	In : 8,19
	-25	7,01	66,4	4,65	2,36	
		19,20	57,3	11,01	8,19	8,19
Totals		100,00	19,6	19,60	80,40	80,40

Table 5.6
Maceral and ash distribution in Greenside coal milled to 95 % passing 45 μm by relative density zone (based on 100 gram of sample)

R.D Zone	Size Fraction (μm)	Total Mass (g)	Ash Contents (%)	Ash Mass (g)	Coal Mass (g)	Maceral Contents (g)
<1,35	+25	6,49	2,9	0,20	6,29	Ex : 1,00
	-25	10,97	3,4	0,37	10,60	Vit: 15,89
		17,46	3,3	0,57	16.89	16,89
1,35-1,60	+25	8,54	11,0	0,94	7,60	Vit: 6,33
	-25	56,80	10,7	6,08	50,72	In : 51,99
		65,34	10,7	7,02	58,32	58,32
>1,60	+25	1,97	51,4	1,01	0,96	In : 5,19
	-25	15,23	72,2	11,00	4,23	
		17,20	69,8	12,01	5,19	5,19
Totals		100,00	19,6	19,60	80,40	80,40

The exinite content is very low and because it has the lowest relative density, it is assumed that exinite will always be in the <1,35 relative density zone. Vitrinite has the next highest density: in both cases it is necessary to allocate some vitrinite to the 1,35 to 1,60 relative density zone. Some inertinite must then be allocated to the >1,60 relative density zone.

It should be stressed that these allocations are speculative, though based on reasonable assumptions. Ultimately, it is impossible to check these calculations as a maceral by definition is greater than 50 μm in size, and particles smaller than 20 μm are being considered. The

analysis is useful, however, for the sake of comparison between samples.

On comparing the values in Tables 5.5 and 5.6, it can be seen that the vitrinite content in the <1,35 relative density zone decreased from 19,11 gram to 15,89 gram on milling. In the 1,35 to 1,60 relative density zone the vitrinite (unliberated) and inertinite increased from 3,11 to 6,33 gram and 48,99 to 51,99 gram, respectively, while the proportion of unliberated inertinite in the >1,60 relative density zone decreased from 8,19 to 5,19 gram.

The decrease in vitrinite content or total coal content in the <1,35 relative density interval on milling to 95 % passing 45 μm, also apparent from Figure 5.10, is difficult to comprehend at first, but may be explained as follows. During float and sink analysis, most particles of the "as is" sample reporting to the float fraction at 1,35 relative density may have consisted of exinite and vitrinite with a low ash content. Some particles however may have been composed of large proportions of clean vitrinite and small proportions of vitrinite with fine mineral intrusions. On milling, these particles may have broken into fragments of clean vitrinite (with a relative density of 1,20 to 1,30) and fragments containing vitrinite with a higher ash content (and, consequently, a higher relative density) than the original particles.

The clean vitrinite fragments would have moved into a lower relative density interval had the density range been extended to values below 1,35. For instance, at a 1,20 to 1,30 relative density interval, the proportions reporting to the floats would probably have shown increases on progressive milling. Depending on the ratio of maceral to mineral content, the 'contaminated' vitrinite with a higher relative density would move into the intermediate relative

density zone. Hence the decrease in total mass in the <1,35 relative density zone.

The figures for ash contents in the <1,35 relative density zone (see Tables 5.5 and 5.6) support the above discussion. The total mass of ash decreased from 0,82 to 0,57 gram, indicating that cleaner coal was present in the milled product. Ash may indeed have been removed in the form of fine mineral intrusions within vitrinite.

The increase in vitrinite content from 9,79 to 13,01 gram in the 1,35 to 1,60 relative density interval confirms the movement of maceral matter out of the <1,35 relative density interval. The increase in inertinite content may be ascribed to the liberation of this maceral from the >1,60 relative density range. The average ash content in the 1,35 to 1,60 relative density region dropped from 13 % to 10,7 %, indicating that macerals within this density region were being liberated.

The movement of the macerals was also apparent from Figure 5.10 which showed that, on milling to progressively finer particle sizes, the proportions of 1,35 - 1,40, 1,40 - 1,45 and 1,45 - 1,50 relative density material increased, the largest increase occuring in the 1,40 - 1,45 relative density interval. The latter resulted from a large increase in the proportion of -25 μm material, as can be seen from Figure 5.9. These increases may therefore have been the result of inertinite being freed from mineral matter in the 1,50 - 1,55 and 1,55 - 1,60 relative density intervals and the 'early' >1,65 relative density region. The decreases in the proportions of 1,55 to 1,70 relative density material in Figure 5.10 would support this theory.

The decrease in the amount of ash material in the <1,35 and 1,35 - 1,60 relative density zones and the increase in the

>1,60 relative density zone upon milling indicate the movement of mineral matter out of the lower relative densities into the high density zone. In the <1,35 and the 1,35 - 1,60 relative density zones the mass of ash decreased from 0,82 to 0,57 and from 7,77 to 7,02 gram, respectively, while in the >1,60 relative density zone the mass of ash increased from 11,01 to 12,01 gram. This suggests that mineral matter has been freed - i.e. the coal has become more liberated on milling to 95 % passing 45 μm.

5.2.2 **Rietspruit coal**

The Rietspruit coal used in this investigation had a higher ash content than the Greenside coal, but a similar maceral composition. The relative density distributions of the +25 μm and -25 μm size fractions of Rietspruit "as is" coal and the subsample milled to 95 % passing 45 μm are presented in Figures 5.11 and 5.12, respectively.

From Figure 5.11 it can be seen that the density distribution patterns of the +25 μm and -25 μm fractions in the unmilled sample were very similar to the respective distribution patterns of Greenside coal (see Figure 5.4). There were differences, however, the major one being that a much larger proportion of both +25 μm and -25 μm material reported to the sinks at 1,70 relative density. This would be expected of a coal with a higher ash content. Other differences were that the proportions in the <1,35 relative density interval and the 1,35 to 1,55 relative density range were smaller, and the proportions in the 1,60 to 1,70 relative density intervals were greater than in Greenside coal.

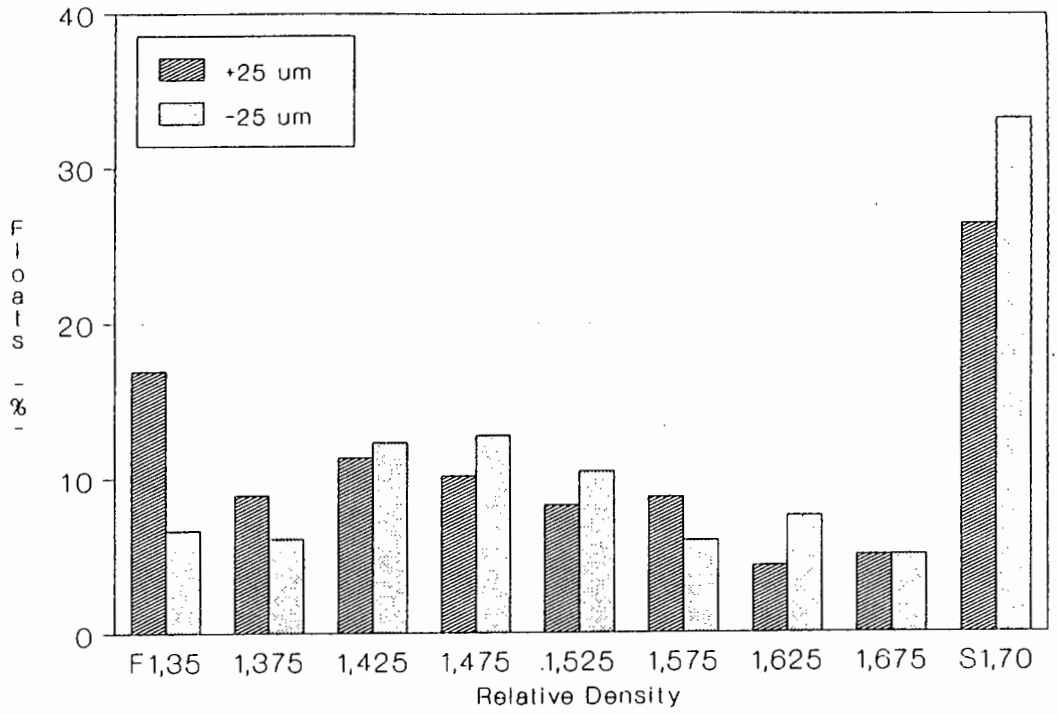


Figure 5.11 Density distribution of the +25 μm and -25 μm fractions of "as is" Rietspruit coal

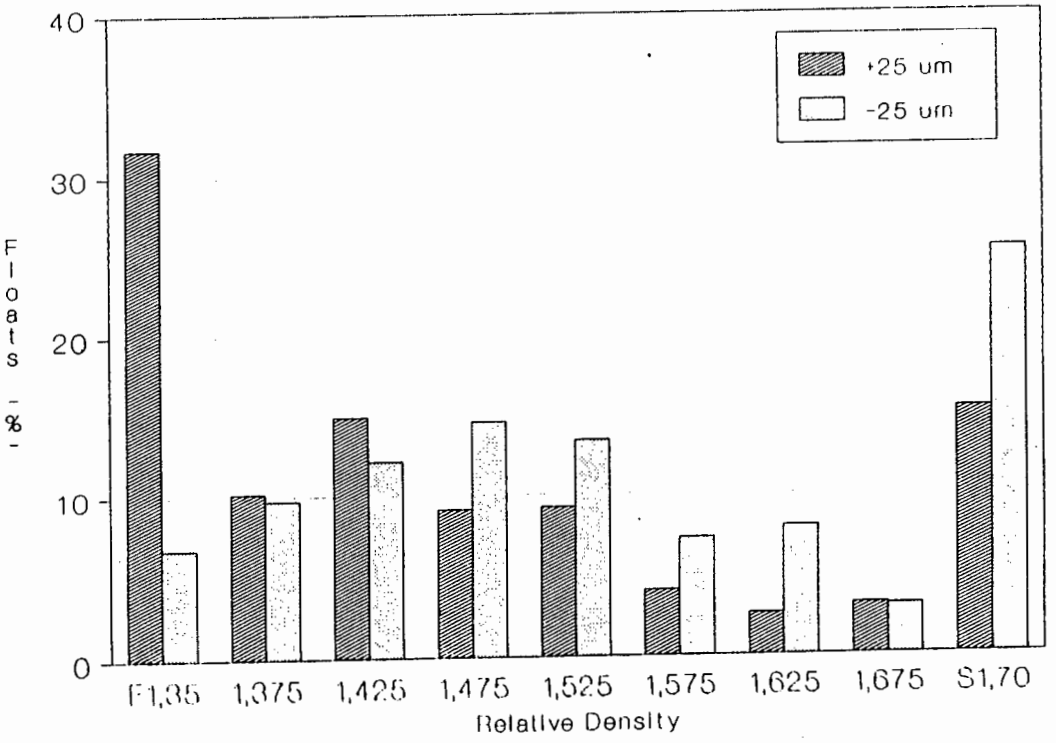


Figure 5.12 Density distribution of the +25 μm and -25 μm fractions of Rietspruit coal milled to 95 % passing 45 μm

Further examination of Figure 5.11 shows that, as with Greenside coal, the proportion of +25 μm material reporting to the floats at 1,35 relative density was much greater than that of the -25 μm fraction. At 1,35 - 1,40 relative density, this feature was much reduced. Between 1,40 and 1,55 relative density a greater proportion of the -25 μm size fraction was present than of the +25 μm material but the differences were small. In the 1,60 - 1,65 relative density interval the -25 μm fraction was greater than the +25 μm fraction, while equal proportions of both fractions reported to the 1,65 - 1,70 relative density range. The sinks at 1,70 relative density contained large proportions of both size fractions, the -25 μm fraction being the greater of the two.

Milling this coal to 95 % passing 45 μm resulted in a marked increase in the proportion of +25 μm material in the floats at 1,35 relative density, and a decrease in the proportion of +25 μm material which sinks at 1,70 relative density. This was also the case with Greenside coal, when milled to the same degree of fineness. This indicates that of the original "as is" sample, more of the +25 μm material that would float at 1,35 relative density remained unbroken (on average) while more of the +25 μm material that would sink at 1,70 relative density was preferentially broken (on average) than material that would appear in other relative density intervals.

Further, in contrast to the Greenside coal, in which a shift in the relative density distribution of the -25 μm material to lower relative densities was observed, in the Rietspruit coal the shift was to higher relative densities. In Figure 5.12, the relative density distribution of the -25 μm material is almost normal in shape between 1,30 and 1,70 relative density, with the mean at 1,45 - 1,50 relative density. In the same relative density range, the

distribution of the +25 μm material is bimodal. Finally, in the sample milled to 95 % passing 45 μm, the proportion of -25 μm material in the 1,55 to 1,65 relative density range was higher than the proportion of +25 μm material in the same interval.

The changes in the distributions of the +25 μm and -25 μm fractions on milling to progressively finer particle sizes are summarized in Figures 5.13 and 5.14 below.

Comparing the density distributions of the +25 μm and -25 μm size fractions, it may be seen that the proportions in the <1,35 relative density interval were far greater (18 to 32 %) for the +25 μm fractions than for the -25 μm fractions (about 7 %). The proportions in the >1,70 relative density interval were smaller in the +25 μm fractions (15 to 27 %) than in the -25 μm fractions (26 to 34 %), and the proportions in the intermediate relative density range (1,40 to 1,60 R.D.) were greater in the -25 μm fractions than in the +25 μm fractions. These distribution patterns are very similar to the corresponding patterns of Greenside coal (see Figures 5.6 and 5.7). Milling, however, resulted in the distribution in the intermediate relative density range of Rietspruit coal to shift slightly to higher densities, while in Greenside coal it clearly shifted to lower densities.

From Figure 5.13 it can be seen that there was a great concentration of +25 μm material in the floats fraction at 1,35 relative density even prior to milling. On milling to 95 % passing 45 μm the proportion of this material increased steadily to almost double its initial value. The proportions of +25 μm material in the 1,35 - 1,40 and 1,40 - 1,45 relative density intervals also increased steadily, though by not as much. In contrast, the proportions of material reporting to the higher density intervals (>1,50 R.D.) decreased steadily. This means that milling

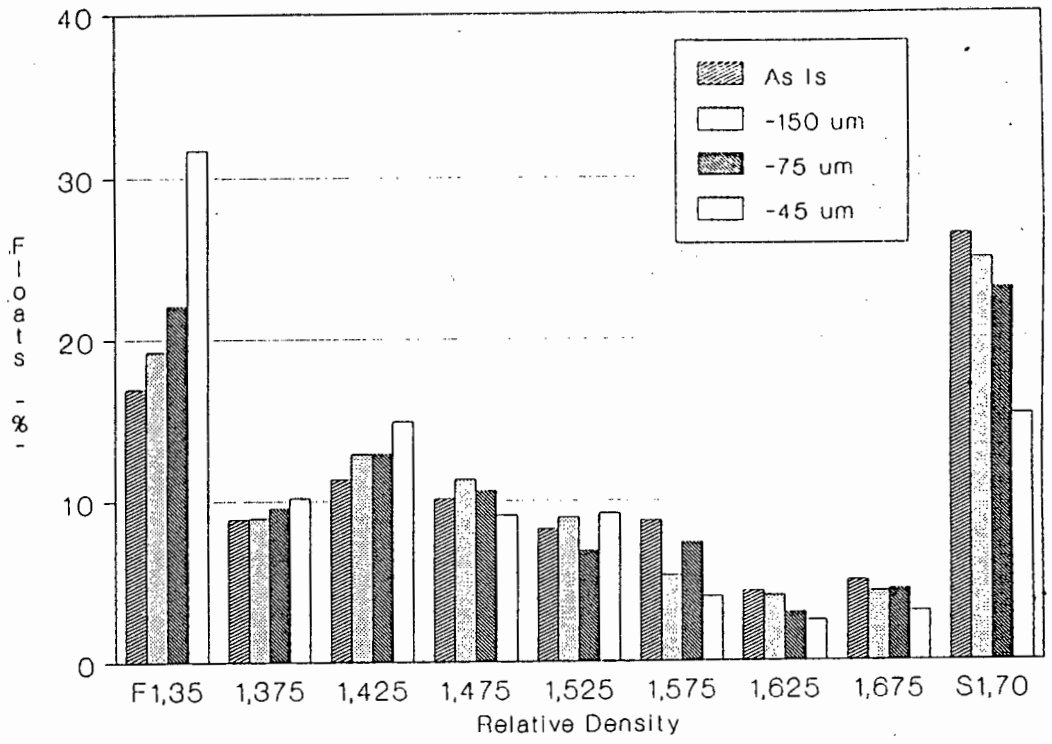


Figure 5.13 Density distributions of the +25 μm fractions of Rietspruit coal milled to various top sizes

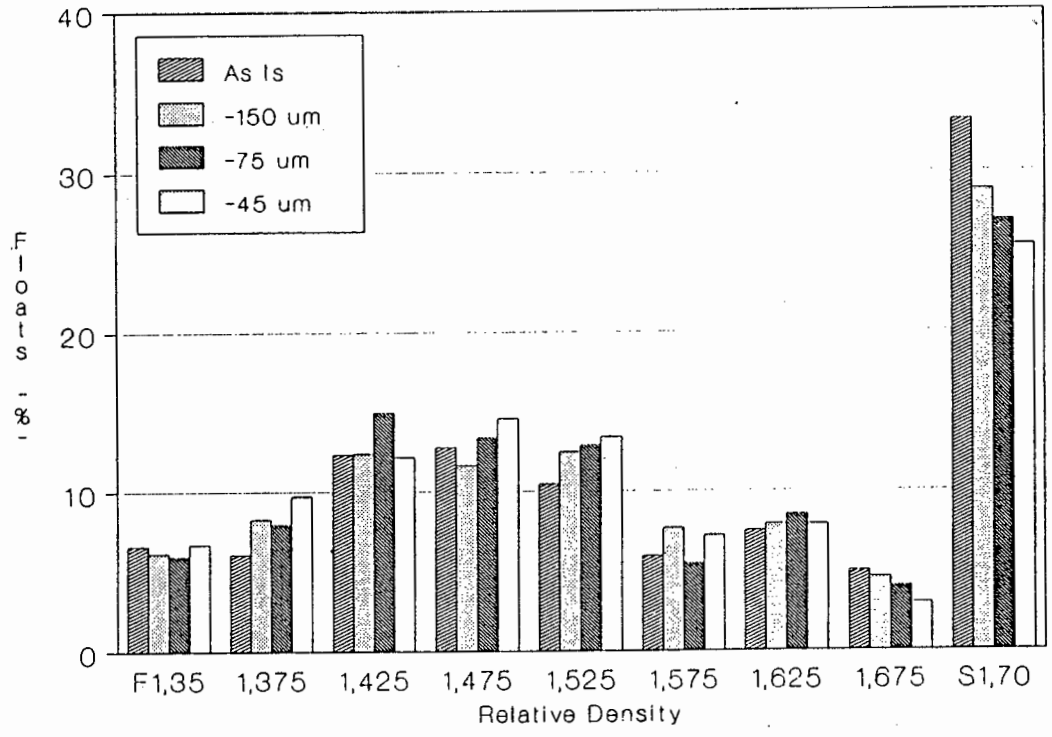


Figure 5.14 Density distributions for the -25 μm fractions of Rietspruit coal milled to various top sizes

selectively broke high density (high ash) material into -25 μm particles rather than reduce lower ash coal to finer particle sizes. Similar observations were made with Greenside coal.

Expressing the data on a basis of 100 gram of feed helps to clarify the changes in the distribution pattern on milling. The absolute values were calculated in the same way as for the Greenside coal (section 5.2.1). They are presented in Table 5.7 and Figures 5.15 to 5.17.

Table 5.7 clearly shows the disappearance of +25 μm material on milling, and its reappearance in the 1,35 to 1,55 relative density intervals in the -25 μm size fraction.

Figure 5.15 also shows how, except for the floats at 1,35 relative density, the +25 μm material was milled away almost to nothing. In the higher relative density intervals (1,60 to 1,70) the reductions appeared proportionately larger than in the lower relative density intervals. A significant decrease was also noted in the sinks material at 1,70 relative density.

Turning to Figure 5.16, especially large increases in the mass of material in the intermediate relative density range (1,40 to 1,60) and in the sinks at 1,70 relative density appeared in the -25 μm size fraction as the "as is" sample was milled to 95 % passing 45 μm. The increase in the sinks fraction, however, appeared proportionately smaller than the increase in the intermediate density range. The proportion of material in the <1,35 relative density interval also increased, but not as much as the proportions in the intermediate density range.

Relative Density	"As Is"			95%-150um			95%-75um			95%-45 um		
	+25 um	-25 um	Total	+25 um	-25 um	Total	+25 um	-25 um	Total	+25 um	-25 um	Total
F1,35	11,78	2,02	13,80	11,61	2,45	14,06	9,82	3,33	13,15	2,89	6,10	8,99
1,35 - 1,40	6,19	1,86	8,05	5,39	3,28	8,67	4,27	4,43	8,69	0,93	8,85	9,78
1,40 - 1,45	7,90	3,74	11,64	7,80	4,90	12,70	5,76	8,19	13,95	1,36	11,06	12,42
1,45 - 1,50	7,06	3,88	10,94	6,86	4,60	11,46	4,74	7,42	12,16	0,83	13,24	14,07
1,50 - 1,55	5,72	3,18	8,90	5,42	4,94	10,36	3,07	7,15	10,22	0,84	12,15	12,99
1,55 - 1,60	6,10	1,80	7,90	3,23	3,05	6,28	3,28	3,06	6,34	0,36	6,58	6,94
1,60 - 1,65	3,01	2,30	5,31	2,45	3,14	5,59	1,35	4,73	6,08	0,23	7,18	7,41
1,65 - 1,70	3,47	1,53	5,00	2,61	1,83	4,44	1,97	2,25	4,22	0,27	2,72	3,00
S1,70	18,36	10,10	28,46	15,03	11,41	26,44	10,24	14,94	25,15	1,39	23,02	24,39
	69,60	30,40	100,00	60,40	39,60	100,00	44,50	55,5	100,00	9,10	90,90	100,00

Table 5.7 Proportions of +25 μm, -25 μm and composite fractions of Rietspruit coal on milling to progressively finer sizes - based on 100 gram of original sample

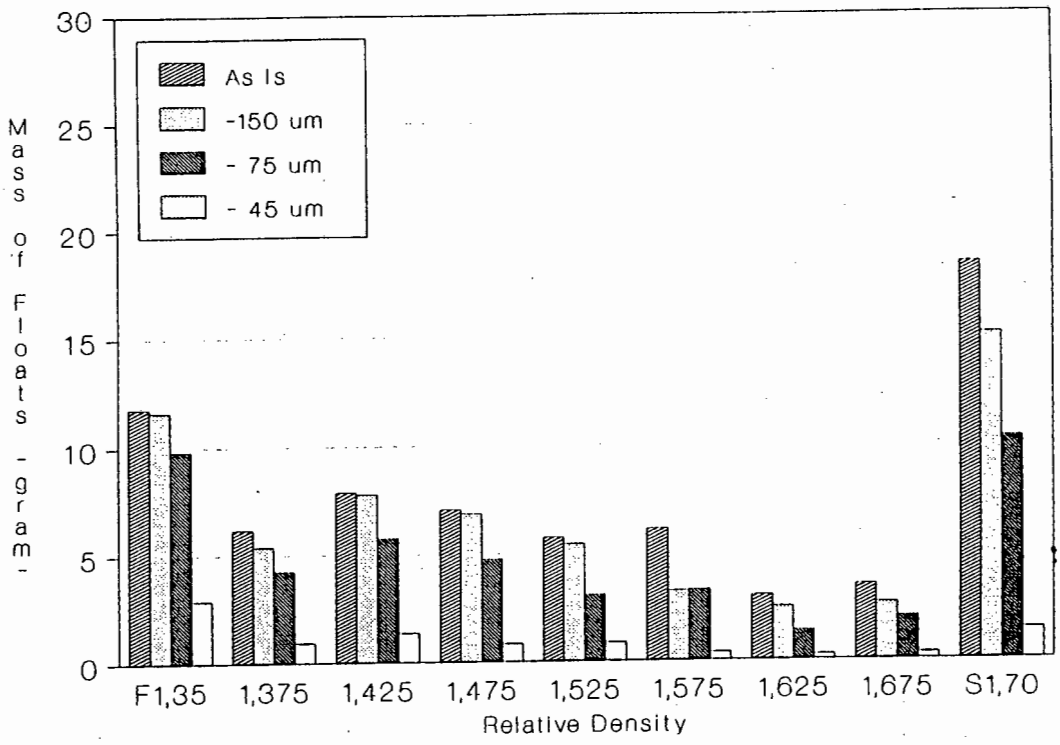


Figure 5.15 Density distributions of the +25 μm fractions of Rietspruit coal milled to various top sizes (basis: each sample = 100 gram)

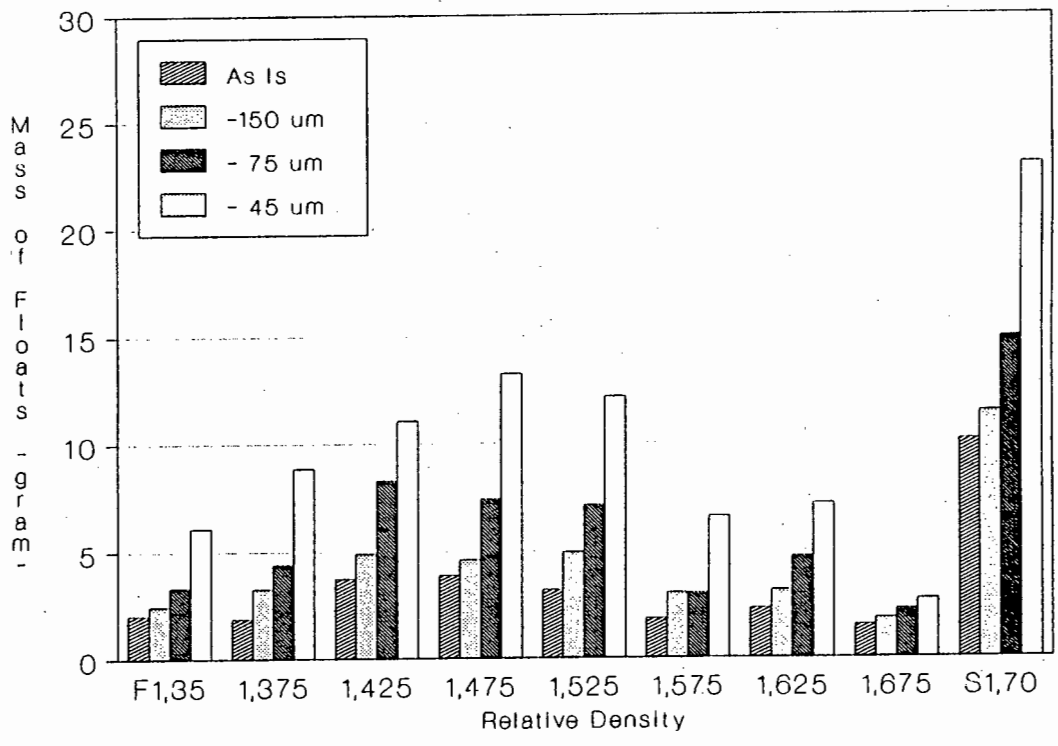


Figure 5.16 Density distributions of the -25 μm fractions of Rietspruit coal milled to various top sizes (basis: each sample = 100 gram)

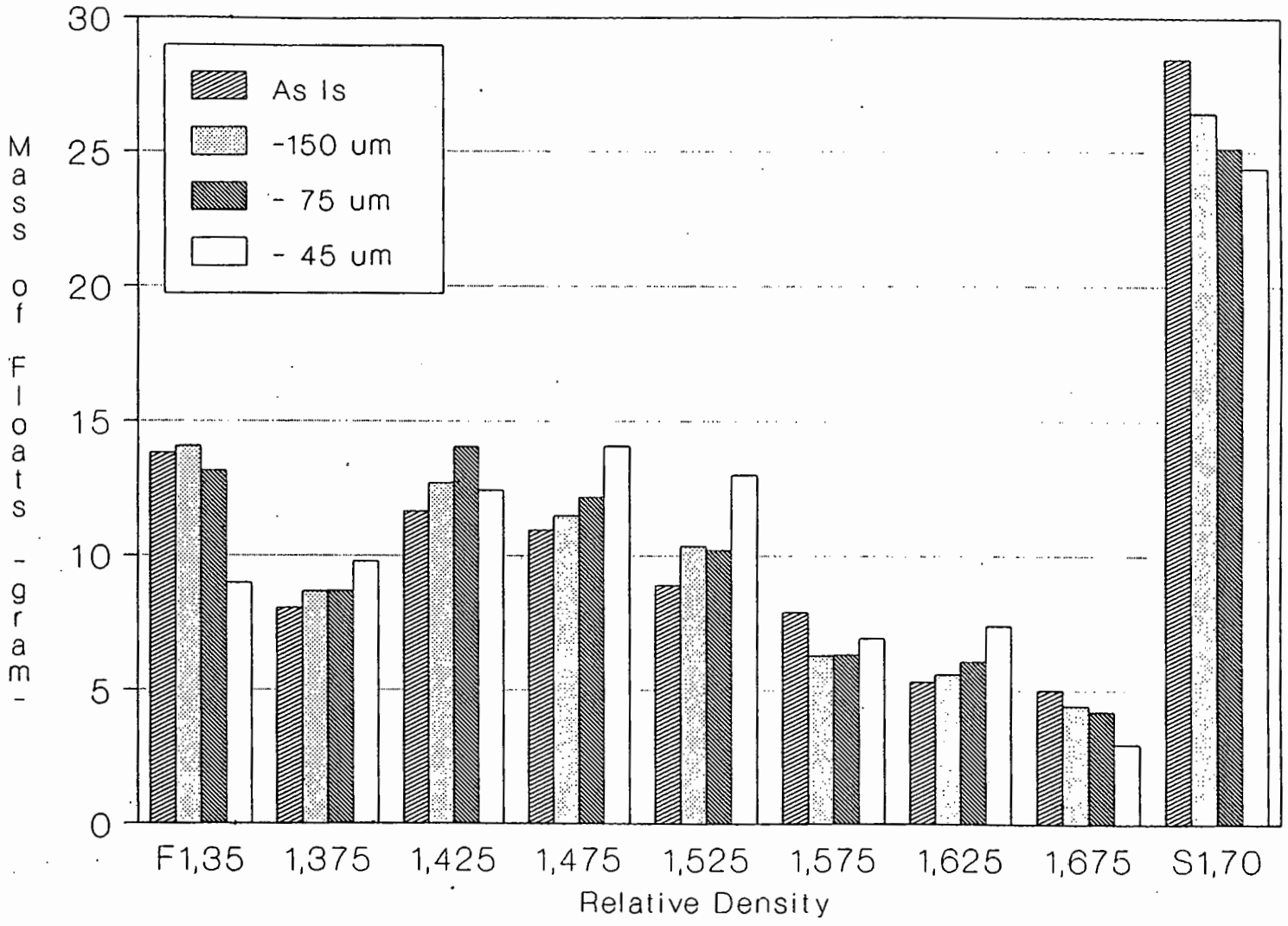


Figure 5.17 Density distributions of Rietspruit coal milled to various top sizes (basis: each sample = 100 gram)

The proportional decrease in mass of +25 μm material in each density interval, together with the proportional increases in mass in the -25 μm fractions, resulting from milling this coal to 95 % passing 45 μm, give an indication of the movement of particles between densities. These values are presented in Table 5.8.

Table 5.8
Proportional breakage of +25 μm material in Rietspruit "as is" coal by relative density interval and the proportional increase in mass of -25 μm material on milling to 95 % passing 45 μm

Relative Density	Rietspruit("as is") (%) Breakage in +25 μm	Rietspruit(95 % -45 μm) (%) Increase in -25 μm
F1,35	75,5	202,0
1,35 - 1,40	85,0	375,8
1,40 - 1,45	82,8	196,0
1,45 - 1,50	88,2	241,2
1,50 - 1,55	85,3	282,1
1,55 - 1,60	94,1	263,5
1,60 - 1,65	92,4	212,2
1,65 - 1,70	92,2	78,4
S1,70	92,4	127,7

It can be seen very clearly from these figures that over 90 % of the +25 μm material in the higher relative density region (>1,55 R.D) broke on milling the "as is" sample to 95 % passing 45 μm. In the 1,35 to 1,55 relative density range on average 85 % broke and in the <1,35 relative density interval only 75,5 % of the +25 μm material was reduced to particle sizes smaller than 25 μm.

In the milled product the proportion of -25 μm material in the 1,35 - 1,40 relative density interval increased nearly four fold. The next highest increase was observed in the 1,45 - 1,65 relative density interval (on average 250 %). This was followed by the <1,35 relative density interval with 202 %, the 1,40 - 1,45 interval with 196 % and lastly the 1,65 - 1,70 and the >1,70 relative density intervals with 78,4 and 127,7 %, respectively.

It is of interest to compare, in passing, these figures with those observed previously for Greenside coal (Table 5.4). The pattern of breakage of Rietspruit coal by relative density intervals was the same, but more intense, while the products of breakage were quite different. For Greenside coal, the proportional increase in 1,50 to 1,65 relative density material in the -25 μm size fraction was low (26 to 58 %) while for Rietspruit coal the corresponding figures were significantly higher (212 to 282 %). The proportional increase in <1,35 and >1,70 relative density material were approximately the same, and in both coals the greatest increase was seen in the 1,35 - 1,40 relative density interval - nearly fourfold in each case.

From the values in Table 5.8 the following breakage pattern may be proposed for Rietspruit coal: particles of densities higher than 1,55 fracture first, followed by particles of 1,35 to 1,55 relative density and lastly by particles in the <1,35 relative density interval. The product consists predominantly of 1,35 - 1,40 relative density material and 1,45 - 1,65 relative density material. This is also evident from Figure 5.17, which presents the relative density distribution of the whole Rietspruit sample as progressive milling occurred. The proportions in the <1,35 and >1,70 relative density intervals decreased on milling while the proportions in the 1,35 to 1,65 relative density range increased.

As with Greenside coal, this result appears strange according to conventional liberation theory but an explanation may be sought in terms of possible movement of maceral and mineral material as milling proceeded. The petrographic analysis of Rietspruit was similar to that of Greenside coal. The maceral proportions listed in Table 3.2 may be converted to mass percent and combined with the average ash content (from Appendix E) to calculate the mass of each maceral and of ash in 100 gram of Rietspruit coal. In 100 gram of feed, Greenside coal contained 22,22 gram vitrinite, 1,00 gram exinite and 57,18 gram inertinite, while Rietspruit coal contained 19,33 gram vitrinite, 2,18 gram exinite and 52,19 gram inertinite.

As before (see page 80) the 1,35 to 1,70 relative density range may be divided into 'maceral zones' and a maceral distribution pattern may be proposed. Tables 5.9 and 5.10 list the total mass (gram/100 gram of feed), ash content (%), mass of coal (gram/100 gram of feed) and maceral content (gram/100 gram of feed) in each relative density zone of the "as is" sample and the sample milled to 95 % passing 45 μm.

The figures in these tables were calculated using values from Table 5.7 above and Tables E5 and E8 in Appendix E, in the same way as for Greenside coal (see Appendix F for sample calculation). Once again it was assumed that the exinite was totally contained within the <1,35 relative density zone, some vitrinite had to be allocated to the 1,35 - 1,60 relative density zone, and some inertinite to the >1,60 relative density zone.

On milling, the total coal content of the <1,35 relative density decreased from 13,22 to 8,69 gram, and the average ash content from 4,2 to 3,3 %, respectively. This may again

Table 5.9

Maceral and ash distribution in Rietspruit "as is" coal by relative density zone (based on 100 gram of sample)

R.D. Zone	Size Fraction (μm)	Total Mass (g)	Ash Contents (%)	Ash Mass (g)	Coal Mass (g)	Maceral Contents (g)
<1,35	+25	11,78	3,8	0,45	11,33	Ex : 2,18
	-25	2,02	6,2	0,13	1,89	Vit: 11,04
		13,80	4,2	0,58	13,22	13,22
1,35-1,60	+25	32,97	13,7	4,52	28,45	Vit: 8,29
	-25	14,46	11,3	1,64	12,82	In : 32,98
		47,43	13,0	6,16	41,27	41,27
>1,60	+25	24,84	48,9	12,14	12,70	In : 19,21
	-25	13,93	53,3	7,42	6,51	
s		38,77	50,5	19,56	19,21	19,21
Totals		100,00	26,30	26,30	73,70	73,70

be the result of particles breaking into fragments of clean vitrinite and vitrinite with fine mineral intrusions ('contaminated' vitrinite). The decrease in ash content indicated that the material in the <1,35 relative density had become cleaner. The mass of vitrinite in the <1,35 relative density interval decreased from 11,04 gram to 6,51 gram, supporting the idea of 'contaminated' vitrinite moving out of the <1,35 relative density interval and reappearing in the intermediate relative density region (1,35 to 1,60). The increase in the vitrinite content (8,29 gram to 12,82 gram) in the 1,35 to 1,60 relative density interval supported this. In the Greenside coal (section 5.2.1), similar changes in the maceral distribution in the <1,35 relative density interval were observed.

Table 5.10

Maceral and ash distribution in Rietspruit coal milled to 95 % passing 45 μm (based on 100 gram of sample)

R.D. Zone	Size Fraction (μm)	Total Mass (g)	Ash Contents (%)	Ash Mass (g)	Coal Mass (g)	Maceral Contents (g)
<1,35	+25	2,89	3,4	0,10	2,79	Ex : 2,18
	-25	6,10	3,3	0,20	5,90	Vit: 6,51
		8,99	3,3	0,30	8,69	8,69
1,35-1,60	+25	4,32	10,9	0,47	3,85	Vit: 12,82
	-25	51,88	10,0	5,19	46,69	In : 37,72
		56,20	10,1	5,66	50,54	50,54
>1,60	+25	1,89	54,0	1,01	0,87	In : 14,47
	-25	32,92	58,7	19,33	13,59	
		34,81	58,5	20,34	14,46	14,47
Totals		100,00	26,30	26,30	73,70	73,70

In the 'inertinite zone' or intermediate relative density region, the total coal content increased from 41,27 to 50,54 gram and the average ash content decreased from 13,0 % to 10,1 %. The vitrinite content increased from 8,29 gram in the unmilled sample to 12,82 gram in the milled sample. At the same time the amount of inertinite increased from 32,98 gram to 37,72 gram. Inertinite was therefore being liberated from the >1,60 relative density zone and may have been partly responsible for the large increases in -25 μm material in the 1,40 to 1,60 relative density region (see Figure 5.16). The inertinite content in the >1,60 relative density interval decreased from 19,21 to 14,47 gram, indicating that inertinite had moved into the intermediate density region.

The movement of mineral matter out of the vitrinite and inertinite zones into the middlings zone, already observed in the Greenside coal (see page 83), is also apparent from the maceral and mineral distribution calculations for Rietspruit coal. On milling, the mass of ash in the <1,35 and the 1,35 - 1,60 relative density intervals decreased from 0,58 to 0,30 gram and from 6,16 to 5,66 gram, respectively. At the same time the mass of ash in the >1,60 relative density interval increased from 19,56 to 20,34 gram.

Thus, 0,78 gram of ash material, which constitutes 2,97 % (0,78/26,3 * 100) of the total ash content of Rietspruit coal, has moved out of the maceral zones, into the heavy middlings. In Greenside coal (see Tables 5.5 and 5.6), however, 1,00 gram of ash forming material, or 5,10 % of the total ash moved out of the maceral zones during milling. Thus proportionately more ash material was released in Greenside coal than in Rietspruit.

The milled product exhibited a relatively even distribution of material over the intermediate relative density range (see Figure 5.17) compared to a skewed distribution for the milled product of Greenside coal (see Figure 5.10). This may be due to the higher ash content of Rietspruit coal and also to a greater association of ash material with vitrinite. For example, in the sample of Greenside coal milled to 95 % passing 45 μm, 46,0 % (13,01/28,27 * 100) of the total vitrinite content was present in the intermediate relative density region, compared with 78,7 % (19,21/24,40 * 100) in Rietspruit coal. The milled product of Rietspruit therefore contained much more vitrinite in the middlings than Greenside coal.

Of the total amount of inertinite in Rietspruit coal, 68,4 % (31,33/45,8 * 100) was present in the intermediate relative

density region. In Greenside coal, however, 89,7 % of inertinite was present in the 1,35 to 1,60 relative density region.

Rietspruit contained 31,6 % of its total inertinite content in the heavy middlings (>1,60 R.D), while Greenside coal contained only 10,3 %. Therefore considerably less inertinite was freed from mineral matter on milling Rietspruit coal than was in the case of Greenside coal.

5.2.3 **Grootegeluk coal**

Grootegeluk coal has distinctly different characteristics from Greenside and Rietspruit coals. The proximate and petrographic analyses, described in sections 3.2.1 and 3.2.2, differed greatly and the results of the size and ash analyses in section 5.1 indicated distinctly different milling characteristics.

The density distribution pattern was also found to be very different from the other two coals. Figures 5.18 and 5.19 present the density distributions of the +25 μm and -25 μm size fractions of "as is" Grootegeluk coal and the product milled to 95 % passing 45 μm.

A very characteristic feature of this coal were the large proportions of <1,35 and >1,70 relative density material. In the "as is" sample (Figure 5.18) 65 to 70 % of the material reported to these relative densities combined, compared with 28 to 38 % for Greenside coal, and 40 to 43 % for Rietspruit coal. This is the result of the high vitrinite content of the Grootegeluk coal (82,5 %) and the high ash content (42 %).

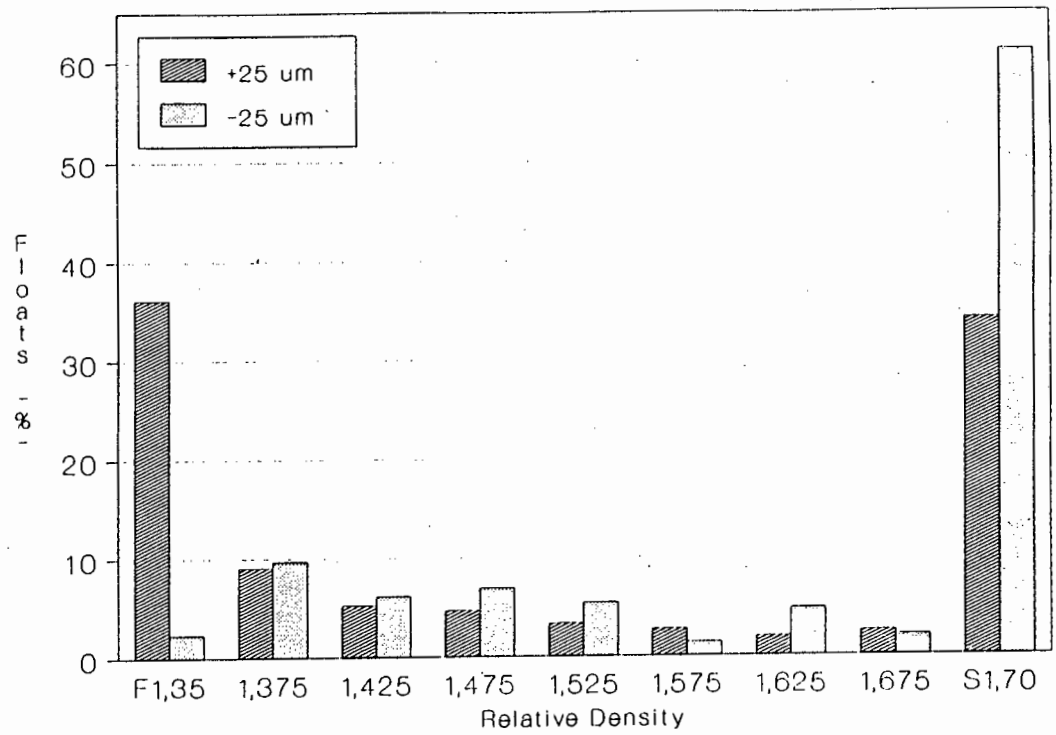


Figure 5.18 Density distributions of the +25 μm and -25 μm fractions of "as is" Grootegeluk coal

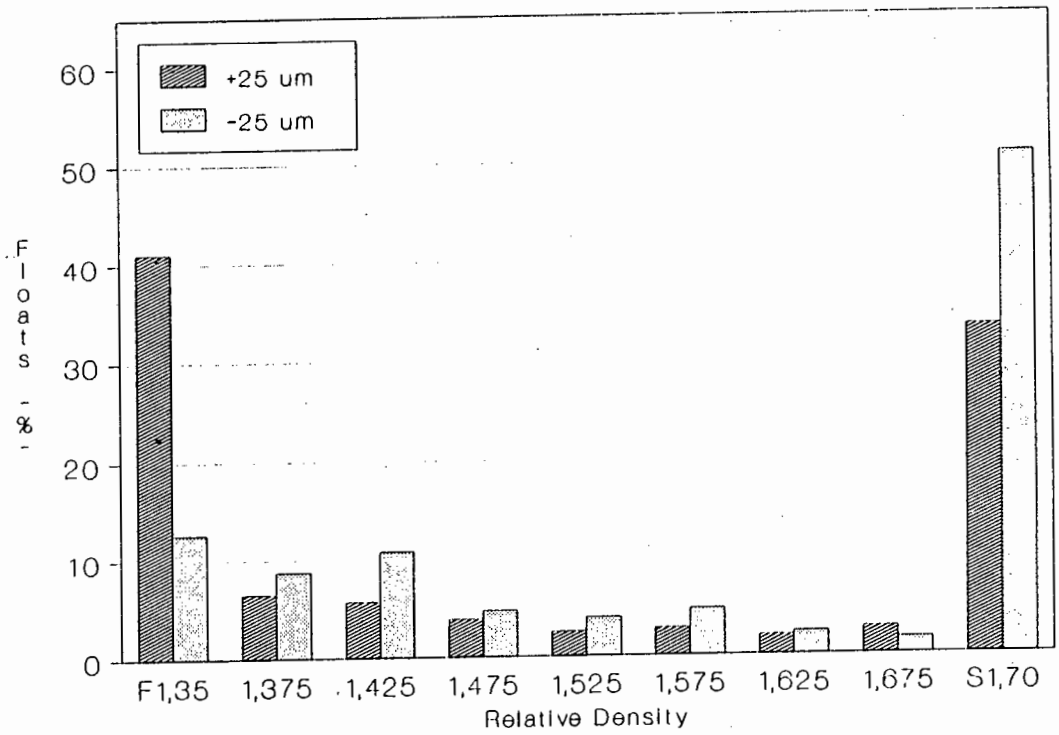


Figure 5.19 Density distributions of the +25 μm and -25 μm fractions of Grootegeluk coal milled to 95 % passing 45 μm

Within the "as is" sample, the trends in the relative density distribution were similar to those observed in the case of the other two coals (cf. Figures 5.4 and 5.11), but exaggerated at the extremes of the distribution. In the <1,35 relative density interval the proportion of +25 μm material (about 37 %) exceeded the proportion of -25 μm material (about 3 %) by far, while in the >1,70 relative density interval the -25 μm fraction (about 62 %) was much greater than the +25 μm fraction (about 35 %). In the intermediate intervals (1,35 to 1,70 R.D.) the proportions of either size fraction were very small, the proportions in the -25 μm fractions being marginally greater than the proportions in the +25 μm fractions in most cases.

On milling to 95 % passing 45 μm, a slight increase was noted in the proportion of +25 μm material reporting to the <1,35 relative density interval, while the proportion of +25 μm material in the >1,70 relative density interval remained roughly the same. This indicates that the <1,35 relative density material was slightly more resistant to breakage than the rest of the +25 μm fraction. In the -25 μm size fraction, a marked increase was noted in the proportion of floats at 1,35 relative density, and a decrease in the proportion of sinks at 1,70 relative density. Increases may also be seen in the proportions in the 1,40 - 1,45 and 1,55 - 1,60 relative density intervals.

These changes on progressive milling are more readily apparent from Figures 5.20 and 5.21. Just looking at the relative density distributions of the +25 μm fractions, there is a small increase in the proportion of <1,35 relative density material, small decreases in the 1,35 to 1,55 relative density intervals, negligible changes in the 1,55 to 1,70 relative density intervals and a small decrease in the proportion of >1,70 relative density material. In the -25 μm fractions, the <1,35 and the 1,40 - 1,45 relative

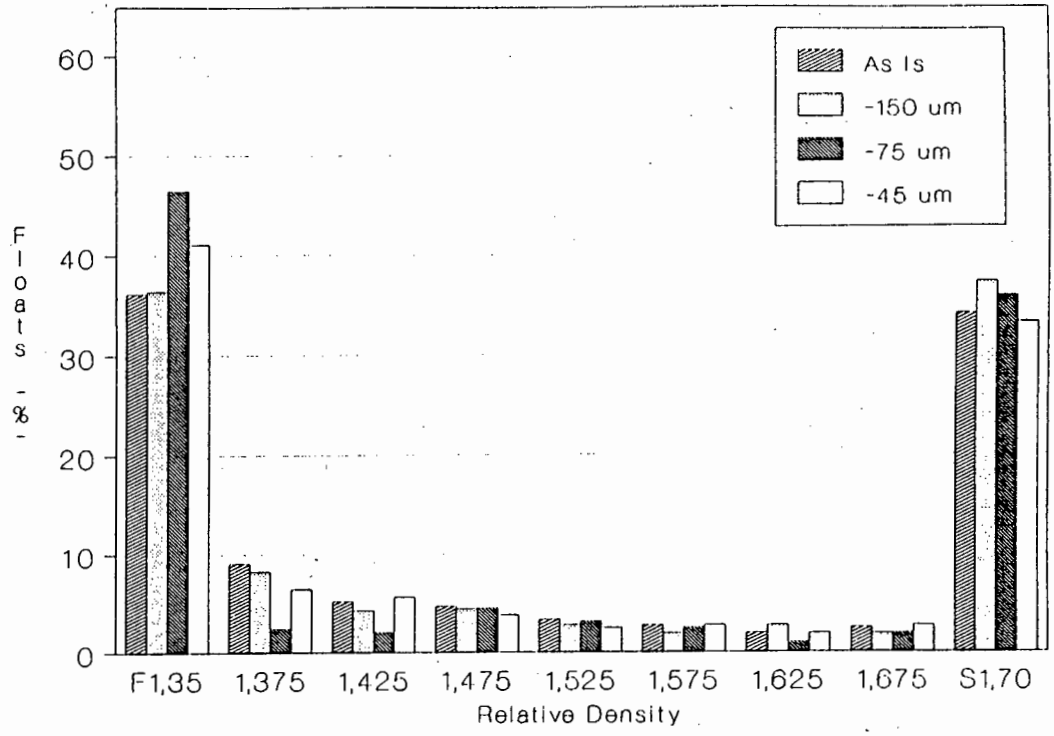


Figure 5.20 Density distributions of the +25 μm fractions of Grootegeluk coal milled to various top sizes

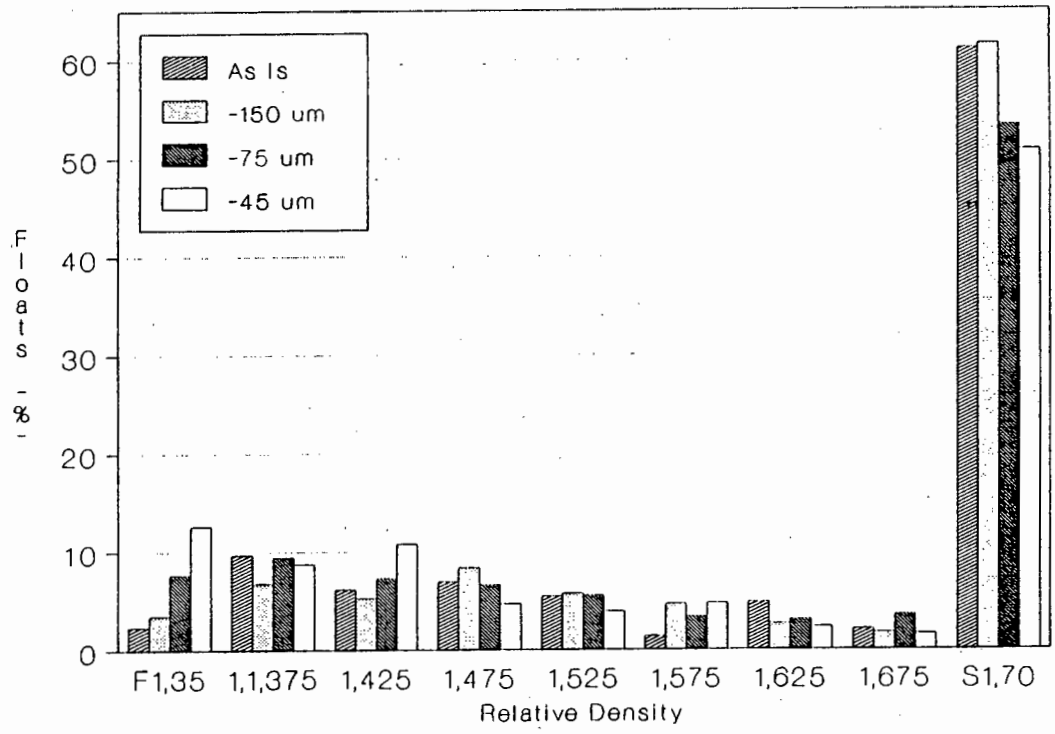


Figure 5.21 Density distributions of the -25 μm fractions of Grootegeluk coal milled to various top sizes

density intervals showed proportionately large increases on milling, while the proportion of material in the 1,55 - 1,60 relative density interval decreased slightly, the proportion in the >1,70 relative density interval decreased considerably. The proportions in the other intervals tended to decrease slightly.

Comparing Figures 5.20 and 5.21, the differences at the low and high densities are evident. As with the Rietspruit coal the proportions in the <1,35 relative density interval were far greater (36 to 46 %) for the +25 μm fractions than for the -25 μm fractions (2 to 12 %). In the >1,70 relative density interval, the proportions were much smaller (33 to 37 %) for the +25 μm fractions than for the -25 μm fractions (51 to 61 %). Between 1,35 and 1,70 relative density, the proportions in the -25 μm fractions tended to be greater than in the +25 μm fractions. This indicates that good quality coal preferentially reported to the coarser (+25 μm) sizes, while high ash material reported to the superfines (-25 μm). This explains the very big difference in ash content between the +25 μm and -25 μm fractions of the Grootegeluk coal, noted above (Table 5.1).

Considering these density distributions on a basis of 100 gram of feed (Table 5.11 and Figures 5.22 to 5.24), considerable changes in the proportions of the <1,35 and >1,70 relative density material in the +25 μm fraction were very obvious. From Table 5.11 it can be seen that the proportion of <1,35 material decreased from 21,28 gram in the +25 μm fraction of the "as is" sample to 10,47 gram in the finest milled sample, and the proportion of >1,70 relative density material decreased from 20,04 gram to 8,46 gram. In the 1,35 to 1,70 relative density range, approximately equal proportional decreases were observed in each relative density interval. These changes can be seen most readily from Figure 5.22.

Relative Density	"As Is"			95%-150um			95%-75um			95%-45 um		
	+25 um	-25 um	Total	+25 um	-25 um	Total	+25 um	-25 um	Total	+25 um	-25 um	Total
F1,35	21,28	0,97	22,25	21,24	1,46	22,70	20,35	4,32	24,67	10,47	9,39	19,86
1,35 - 1,40	5,36	4,00	9,36	4,78	2,81	7,59	1,08	5,33	6,41	1,66	6,51	8,17
1,40 - 1,45	3,09	2,29	5,38	2,52	3,57	6,09	0,92	4,11	5,03	1,45	8,02	9,47
1,45 - 1,50	2,79	1,45	4,24	2,58	2,11	4,69	1,98	2,73	4,71	0,97	3,48	4,45
1,50 - 1,55	1,97	3,93	5,90	1,64	2,38	4,02	1,38	4,12	5,50	0,63	2,90	3,53
1,55 - 1,60	1,61	0,57	2,18	1,13	1,92	3,05	1,11	1,91	3,02	0,70	3,52	4,22
1,60 - 1,65	1,17	1,99	3,16	1,60	1,08	2,68	0,46	1,73	2,19	0,49	1,71	2,20
1,65 - 1,70	1,48	0,86	2,34	1,10	0,70	1,80	0,84	1,98	2,82	0,69	1,16	1,85
S1,70	20,04	25,16	45,20	21,72	25,65	47,37	15,69	29,96	45,65	8,46	37,81	46,27
	58,80	41,20	100,00	58,30	41,70	100,00	43,80	56,20	100,00	24,50	74,50	100,00

Table 5.11 Proportions of +25 μm, -25 μm and composite fractions of Grootegeluk coal on milling to progressively finer sizes - based on 100 gram of original sample

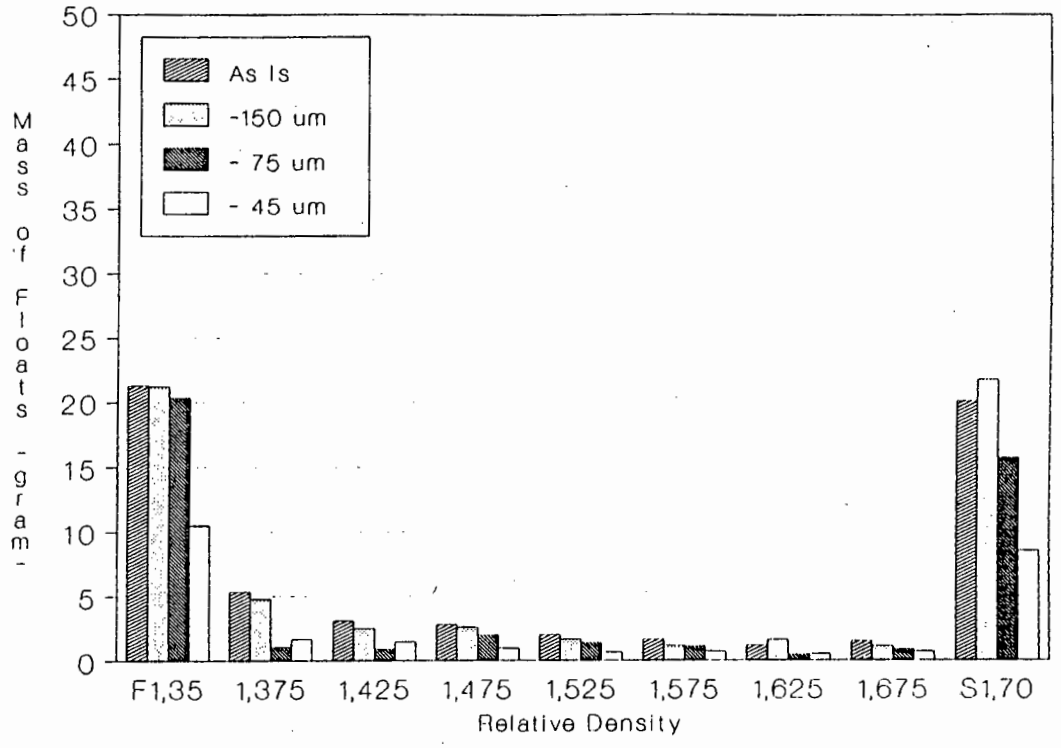


Figure 5.22 Density distributions of the +25 μm fractions of Grootegeluk coal milled to various top sizes (basis: each sample = 100 gram)

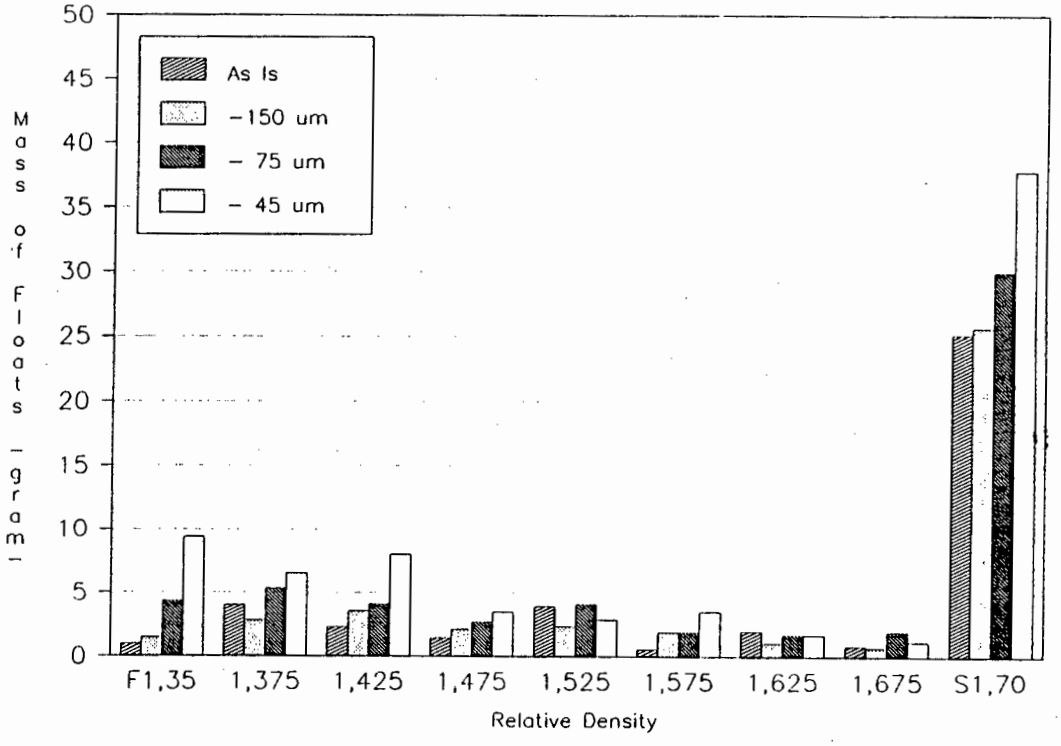


Figure 5.23 Density distributions of the -25 μm fractions of Grootegeluk coal milled to various top sizes (basis: each sample = 100 gram)

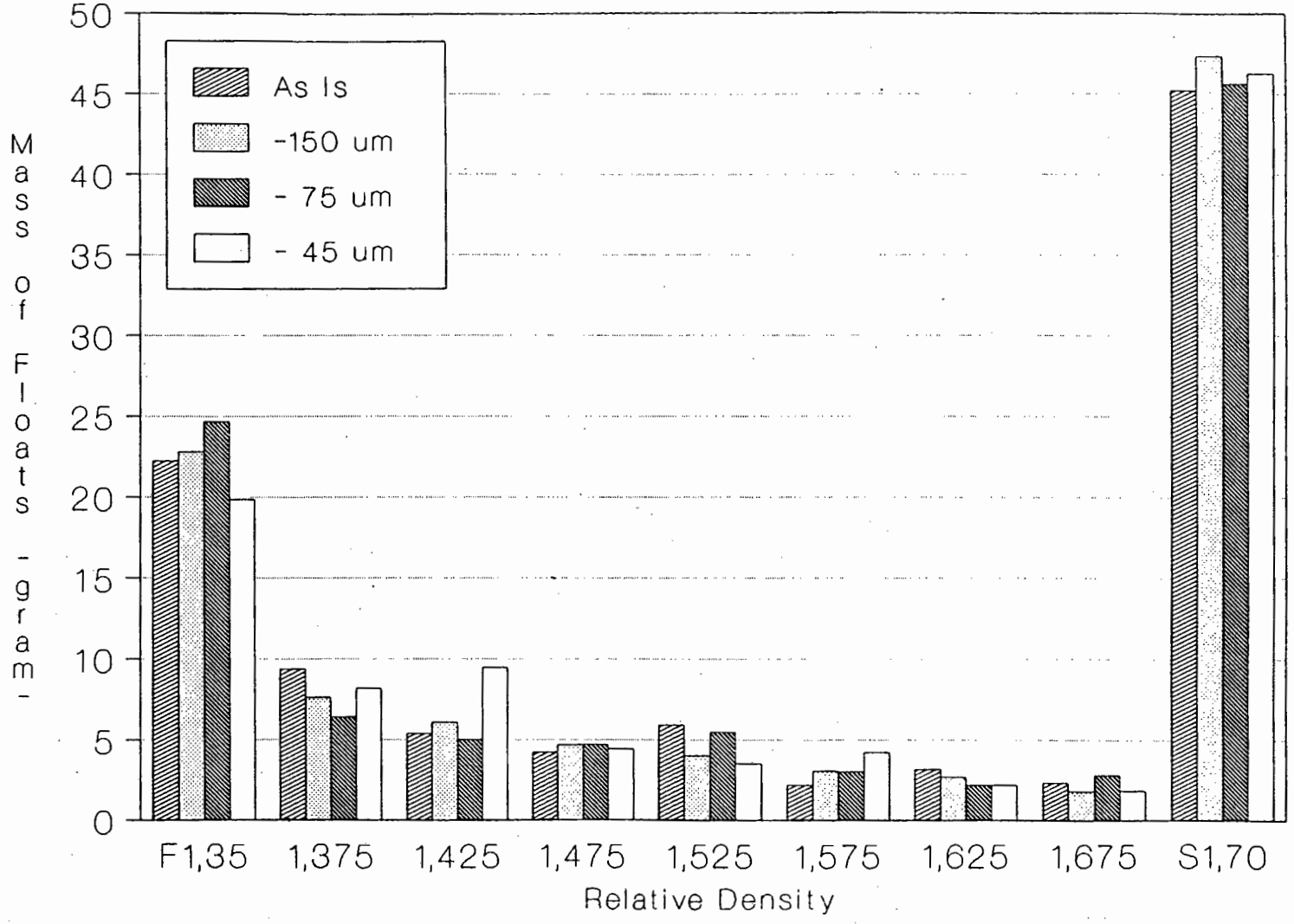


Figure 5.24 Density distribution of Grootegeluk coal milled to various top sizes (basis: each sample = 100 gram)

In the -25 μm fractions large increases were observed in the <1,35, >1,70 and 1,40 - 1,45 relative density intervals. The proportion of <1,35 relative density material increased from 0,97 gram to 9,39 gram, when the sample was milled to 95 % passing 45 μm. The increases in the proportion of 1,40 - 1,45 relative density material were less drastic, but still high. The mass of >1,70 relative density material, already 25,16 gram, increased to 37,81 gram. In all other density intervals the increases were smaller. These changes are shown in histogram form in Figure 5.23.

More information maybe obtained from the calculated proportional breakages of +25 μm material on milling and the increases in -25 μm material, listed in Table 5.12.

Table 5.12
Proportional breakage of +25 μm material in Grootegeluk "as is" coal by relative density interval and the proportional increase in mass of -25 μm material on milling to 95 % passing 45 μm

Relative Density	Grootegeluk("as is") (%) Breakage in +25 μm	Grootegeluk(95 % -45 μm) (%) Increase in -25 μm
F1,35	50,8	868,0
1,35 - 1,40	69,0	62,8
1,40 - 1,45	53,1	250,2
1,45 - 1,50	65,2	140,0
1,50 - 1,55	68,0	26,2
1,55 - 1,60	56,5	517,5
1,60 - 1,65	58,1	14,1
1,65 - 1,70	53,4	34,9
S1,70	57,8	50,3

The values in Table 5.12 show that between 50 and 60 % of +25 μm material in the <1,35, 1,40 - 1,45 and 1,55 to 1,70 relative density intervals broke, and between 65 and 69 % of material in the 1,35 - 1,40, 1,45 - 1,50 and the 1,50 - 1,55 relative density intervals. No distinct pattern of breakage was evident from these figures; and indeed, the figures are so similar as to suggest that the +25 μm particles broke uniformly, regardless of density.

The increase in <1,35 relative density material in the -25 μm fraction on milling was exceptionally high at 868 %. Other big proportional increases were in the 1,40 to 1,50 relative density intervals and in the 1,55 - 1,60 relative density interval.

Considering these figures together with the histogram in Figure 5.24, which shows the change in the density distribution of 100 gram of "as is" material on progressive milling, some deductions on the milling characteristics of this coal may be made. The small net decrease in the <1,35 and 1,35 - 1,40 relative density material was balanced by the increase in the 1,40 - 1,45 relative density interval. Similarly, the small overall decrease in the 1,50 - 1,55 relative density interval was made up in the 1,55 - 1,60 relative density interval. The changes in the other density intervals were marginal. It may be deduced that the large increase in the proportions in -25 μm material (see Table 5.12) in the <1,35 relative density interval on milling was the result of +25 μm material in that density interval being reduced to -25 μm material; and that only small amounts of material moved between densities.

These observations are borne out when the allocations of macerals to the different density zones is made, as shown in Tables 5.13 and 5.14. The values in these tables were

calculated from Table 5.11 above and Tables E9 and E12 in Appendix E (see Appendix F for a sample calculation).

The maceral content of Grootegeluk coal differs greatly from the other two coals. On a mass basis, the non-mineral content of Grootegeluk coal is 80,3 % vitrinite, 2,1 % exenite, and 17,6 % inertinite. Therefore in 100 gram of Grootegeluk coal, there would be 46,58 gram vitrinite, 1,19 gram exinite, 10,23 gram inertinite and 42,00 gram ash forming mineral matter.

Grouping the relative density intervals into maceral and mineral zones, exinite, vitrinite and inertinite were assigned in the same way as for Greenside coal (see page 80). For the "as is" sample, all the exinite (1,19 gram) and some vitrinite (20,38 gram) was assigned to the vitrinite zone. The inertinite zone was filled with vitrinite (22,99 gram), while the remainder of vitrinite (3,21 gram) and the whole mass of inertinite (10,23 gram) was placed in the heavy middlings zone. The maceral allocations in the sample milled to 95 % passing 45 μm were made on the same basis.

These allocations may seem unlikely, but they are the most realistic in view of the very high vitrinite content of this coal which forces vitrinite into the >1,60 relative density zone. Any allocation of inertinite, however small, into the 1,35 - 1,60 relative density zone, would simply increase the amount of vitrinite in the >1,60 relative density zone.

From Tables 5.13 and 5.14, it can be seen that there are no major changes in the three relative density zones in either the coal or the ash distribution. Going from the "as is" sample to the sample milled to 95 % passing 45 μm, the total amount of coal (in 100 gram) in the <1,35 relative density interval decreased from 21,57 to 19,19 gram. In the 1,35 to 1,60 and the >1,60 relative density intervals, the total amount of coal increased from 22,99 gram to 25,26 gram and

from 13,44 to 13,55 gram (negligible), respectively. The decrease in the <1,35 relative density interval and the increase in the 1,35 to 1,60 relative density range may be ascribed to clean fragments of vitrinite breaking away from vitrinite with mineral intrusions. The latter would have a higher density and would therefore move into the next density interval.

The ash contents of the material in these density intervals changed only marginally. The only real movement was the net gain of 0,49 gram of ash by the 1,35 to 1,60 relative density zone from the >1,60 relative density interval.

Table 5.13

Maceral and ash distribution in Grootegeluk "as is" coal by relative density zone (based on 100 gram of sample)

R.D. Zone	Size Fraction (μm)	Total Mass (g)	Ash Contents (%)	Ash Mass (g)	Coal Mass (g)	Maceral Contents (g)
<1,35	+25	21,28	3,0	0,64	20,64	Ex : 1,19
	-25	0,97	3,4	0,04	0,93	Vit: 20,38
		22,25	3,0	0,68	21,57	21,57
1,35-1,60	+25	14,82	16,7	2,47	12,35	Vit: 22,99
	-25	12,24	13,0	1,60	10,64	In : none
		27,06	15,0	4,07	22,99	22,99
>1,60	+25	22,69	73,4	16,65	6,04	Vit: 3,21
	-25	28,01	73,5	20,60	7,40	In : 10,23
		50,70	73,5	37,26	13,44	13,44
Totals		100,00	42,00	42,00	58,00	58,00

Table 5.14

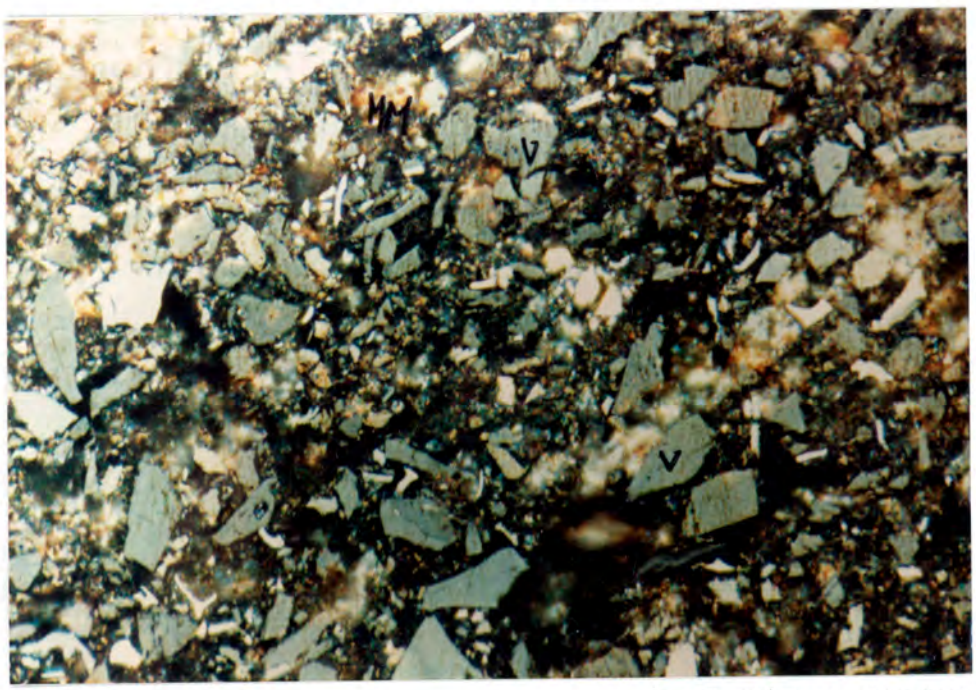
Maceral and ash distribution in Grootegeluk coal milled to 95 % passing 45 μm (based on 100 gram of original sample)

R.D Zone	Size Fraction (μm)	Total Mass (g)	Ash Contents (%)	Ash Mass (g)	Coal Mass (g)	Maceral Contents (g)
<1,35	+25	10,47	3,3	0,35	10,12	Ex : 1,19
	-25	9,39	3,4	0,32	9,07	Vit: 18,00
		19,86	3,4	0,67	19,19	19,19
1,35-1,60	+25	5,41	17,8	0,96	4,45	Vit: 25,26
	-25	24,43	14,8	3,62	20,81	In : none
		29,84	15,3	4,58	25,26	25,26
>1,60	+25	9,64	72,6	6,98	2,65	Vit: 3,32
	-25	40,68	73,2	29,78	10,90	In : 10,23
		50,32	73,3	36,77	13,55	13,55
Totals		100,00	42,00	42,00	58,00	58,00

Hence, as there was no movement of ash material out of the lower relative density zones into the heavy middlings zone, no maceral liberation had taken place (in fact, the coal was becoming less liberated).

Turning to the maceral distribution, in the <1,35 relative density zone the mass of vitrinite decreased slightly (20,38 to 18,00 gram) on milling and reappeared in the 1,35 to 1,60 relative density zone (22,99 to 25,26 gram). In the >1,60 relative density zone the mass of vitrinite was unaltered (3,21 vs. 3,32 gram). The whole mass of coal in the >1,60 relative density zone remained the same, suggesting that no coal was freed on milling to 95 % passing 45 μm.

A photograph (see Figure 5.25) of the -25 μm fraction in the "as is" sample confirms the presence of an abundance of very small vitrinite particles (grey) between smaller mineral particles. Whether the vitrinite particles had fine mineral intrusions is not visible from the photograph, but this was suspected from the maceral distribution. From the photograph it can also be seen that only very little inertinite (light grey) was present in this sample.

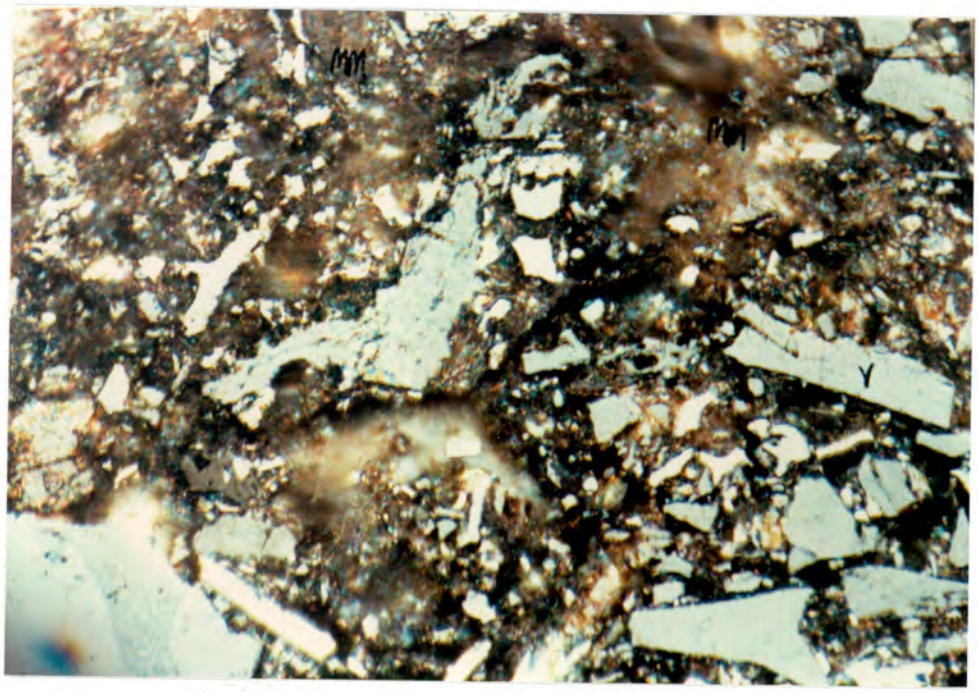


from Dimou (1986)

Figure 5.25 Petrographic photograph of the -25 μm size fraction of "as is" Grootegeluk coal

Another photograph (Figure 5.26) of the material in the >1,52 relative density region of the "as is" sample showed a very large proportion of small vitrinite particles. This supports the idea of relatively large proportions of vitrinte in the 1,35 - 1,60 relative density zone, proposed in the maceral distribution above. A larger vitrinite particle in the center of the photograph appeared to have a high mineral content (dark spots) and may be a typical particle found in the intermediate relative density range,

which on breaking would fracture into particles of roughly the same density.



from Dimou (1986)

Figure 5.26 Petrographic photograph of the >1,52 relative density fraction of "as is" Grootegeluk coal

From these findings one may therefore conclude that by milling Grootegeluk coal to 95 % passing 45 μm, no liberation was achieved; milling merely reduced +25 μm material to -25 μm material of the same composition/density.

5.2.4 **A new measure of liberation**

From the observed coal and ash distributions by density interval of Greenside and Rietspruit coals, the movement of ash material out of the maceral zones into the heavy density zone, on milling each coal to 95 % passing 45 μm, was clearly visible (see section 5.1.1, Table 5.1). Therefore ash material was freed on milling, suggesting that the degree of liberation was increased. For Grootegeluk coal,

the reverse was true : the combined mass of ash material in the <1,35 and the 1,35 - 1,60 relative density zones increased (by 0,5 gram), indicating a decrease in the degree of liberation.

From these observations a new measure of liberation can be proposed. For "overall" liberation, the total amount of misplaced material (maceral and mineral) in all the relative density zones must be considered. For example, in the "as is" sample of Greenside coal (Table 5.5), the misplaced material is made up of 0,82 gram ash in the <1,35 relative density zone, 7,77 gram ash and 3,11 gram vitrinite in the 1,35 - 1,60 relative density zone and 8,19 gram inertinite in the >1,60 relative density zone (total = 19,89 gram). Therefore, 19,89 gram in 100 gram of sample are misplaced; conversely, 80,11 gram are in the correct place, i.e. 80,11 % of this coal is liberated.

Similarly, the amounts of misplaced material in the Rietspruit and Grootegeluk coals can be calculated. Values for the milled and unmilled samples of all three coals are listed in Table 5.15 below.

Of the three coals, Greenside coal has the least misplaced material and is thus the most liberated of the three coals (80,11 to 80,89 % of the macerals and mineral are in the correct relative density zones). Rietspruit coal is less liberated (65,76 to 66,75 %), while Grootegeluk coal is the least liberated of all (58,82 to 55,94 %).

The figures also show that the increase in overall liberation of the Greenside coal is slight (0,78 %). On milling to 95 % passing 45 μm, the increase in the liberation of the Rietspruit coal is higher (0,99 %), while the overall liberation of Grootegeluk coal decreases (-2,88 %).

Table 5.15
Amounts of misplaced and liberated material in Greenside, Rietspruit and Grootegeluk coals milled to various top sizes (based on 100 gram of original sample)

Coal	Particle Size (μm)	Misplaced Material (gram)	Liberated Material (gram)
Greenside	"as is"	19,89	80,11
	95 % - 45	19,11	80,89
Rietspruit	"as is"	34,24	65,76
	95 % - 45	33,25	66,75
Grootegeluk	"as is"	41,18	58,82
	95 % - 45	44,06	55,94

If liberation is expressed in terms of ash and "coal" only, with "coal" being the sum of the macerals, the values in Table 5.16 ensue. These were calculated by assuming that any ash found in the <1,60 relative density zone was misplaced, as was any coal found in the >1,60 relative density zone. In these terms, Greenside coal is still the most liberated, but now Grootegeluk coal occupies second position, with Rietspruit coal last. Upon milling to 95 % passing 45 μm, the liberation of the Grootegeluk coal decreases slightly, and that of Greenside coal increases by 2 %, while the increase in liberation in Rietspruit coal is still the highest at over 5,5 %.

Table 5.16
Amounts of misplaced ash and "coal" and liberated material in Greenside, Rietspruit and Grootegeluk coals (based on 100 gram of original sample)

Coal	Particle Size (μm)	Misplaced Ash and "coal" (gram)	Liberated Material (gram)
Greenside	"as is"	16,78	85,22
	95 % - 45	12,78	87,22
Rietspruit	"as is"	25,95	74,05
	95 % - 45	20,43	79,57
Grootegeluk	"as is"	18,19	81,89
	95 % - 45	18,80	81,20

5.3 **WASHABILITY CHARACTERISTICS**

In this section the washability characteristics of each coal are discussed and the washability of the +25 μm and -25 μm fractions compared. Relative densities are now left out of the picture, and the yields at different ash values are examined. Tables containing the raw data for each coal are given in Appendix C. These data are presented in graphical form (cumulative yield vs. cumulative ash) below.

The accuracy of the centrifugal float and sink analysis method used to obtain the results (section 3.4.3.1) was checked using gravimetric float and sink analysis and oil agglomeration. These results are also reported in this section.

5.3.1 **Cumulative floats data**

5.3.1.1 Greenside coal

Figure 5.27 shows the cumulative yield versus cumulative ash points for the +25 μm and the -25 μm size fractions of the Greenside "as is" coal sample and the milled subsamples.

From the figure it is immediately apparent that the data lie in two bands, with the cumulative floats points for the -25 μm (open symbols) fractions always lying above those of the +25 μm (shaded symbols) fractions (for the same sample). This implies that higher yields of low ash coal (7,4 %) can be obtained from the +25 μm fractions than from the -25 μm fractions. The +25 μm fractions thus exhibit better washability characteristics than the -25 μm fractions. This was not unexpected from the density distribution patterns, discussed in Section 5.2.1.

Closer comparison of the washability data with the density distribution does however give some cause for disquiet. The density distribution of the "as is" sample (see Figure 5.4) showed that the proportion of +25 μm material in the <1,35 relative density interval exceeded the proportion of -25 μm material by about 15 %, while in the sample milled to 95 % passing 45 μm (see Figure 5.5) the difference between these proportions was 25 %. The cumulative floats points in Figure 5.27, however, show greater differences. For example, at an ash content of 5 %, the difference in yield between the +25 μm and -25 μm fractions of the "as is" sample is approximately 35 %. The same is true at other low ash values.

A possible reason for this may be that the observed ash values for the -25 μm fractions are too high. High ash values at low relative densities may result from inefficient separation in the float and sink analysis of very fine

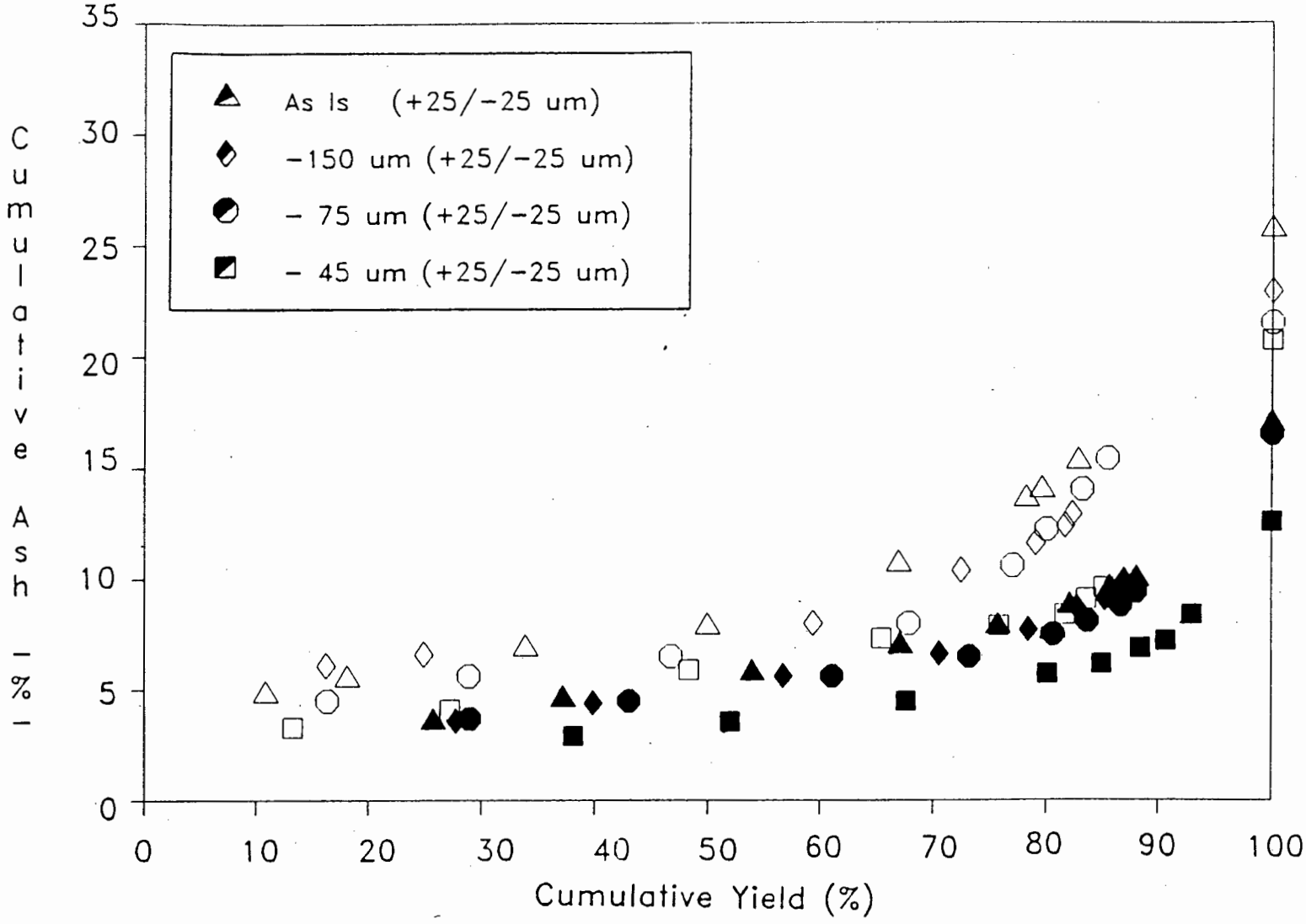


Figure 5.27 Cumulative floats data for the +25 μm and -25 μm fractions of Greenside coal milled to various top sizes

particles of high ash content. This was already observed in the preliminary work and was the reason for conducting the float and sink analyses on the -25 μm fraction sequentially (section 4.3). This point will be discussed again below (section 5.3.2).

5.3.1.2 Rietspruit coal

The washability characteristics of the +25 μm and -25 μm fractions for the milled and unmilled samples of Rietspruit coal follow similar trends to those of Greenside coal, as can be seen in Figure 5.28.

The +25 μm band of points lies below the -25 μm band, implying that the former has better washability characteristics than the latter. The bands lie closely together and overlap in the central portion where there is a high concentration of data points. The +25 μm fraction of the sample milled to 95 % passing 45 μm exhibits the best washability characteristics, while the -25 μm fraction of the unmilled coal is represented by the uppermost set of points and therefore has the worst washability characteristics.

From the density distributions of the "as is" sample of Rietspruit coal and the sample milled to 95 % passing 45 μm (Figures 5.13 and 5.14), the differences in the proportions of +25 μm and -25 μm material reporting to the floats at <1,35 relative density, are 10 % for the "as is" sample and 25 % for the milled sample. One would therefore expect to find relatively small differences between the yields at any ash value of the +25 μm and the -25 μm fractions in the "as is sample, but quite large differences in the corresponding values of the sample milled to 95 % passing 45 μm. The cumulative floats data show that this is indeed so. The points for the +25 μm and the -25 μm fraction of the "as is"

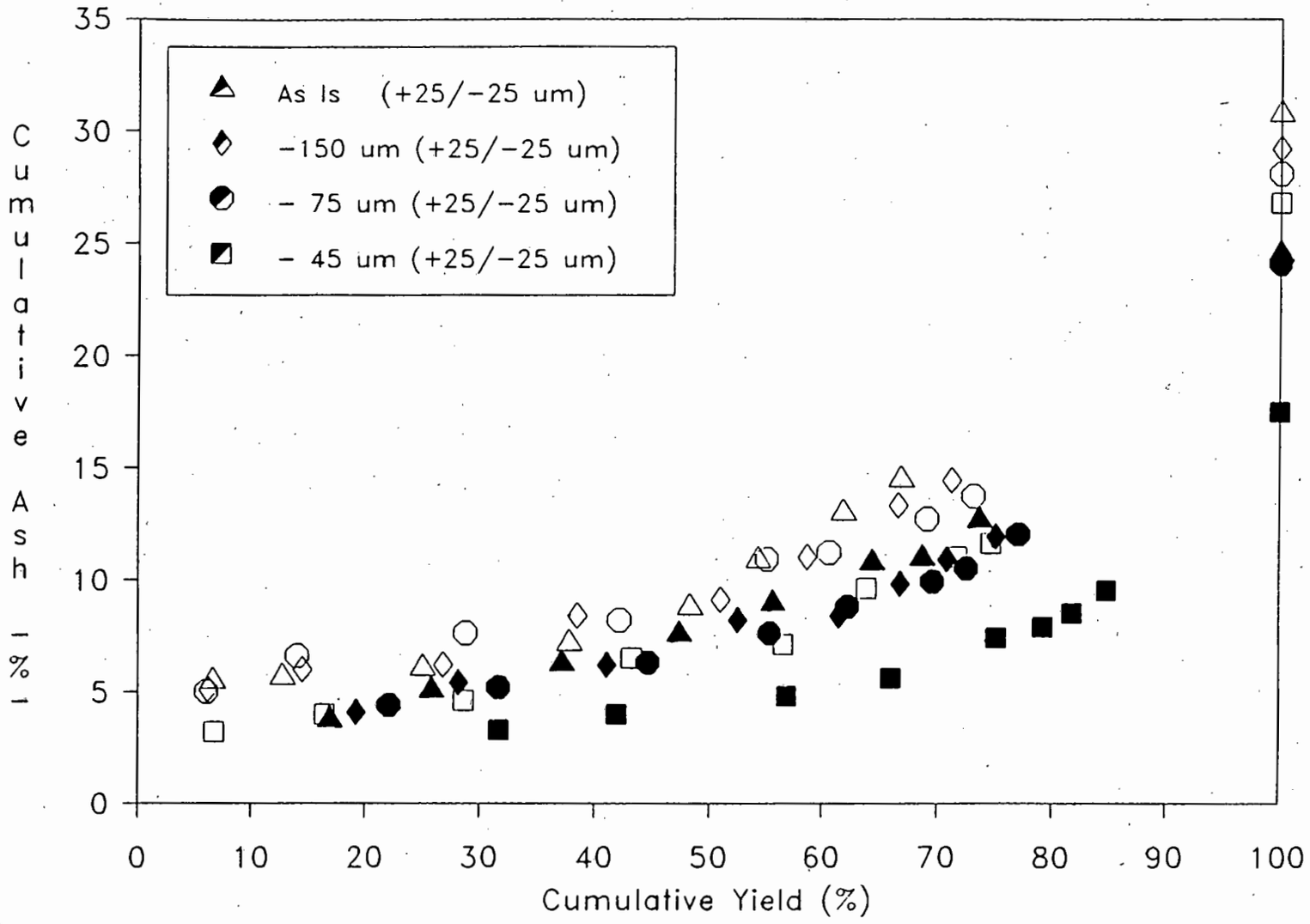


Figure 5.28 Cumulative floats data for the +25 μm and -25 μm fractions of Rietspruit coal milled to various top sizes

sample are close together, whereas the respective points for the sample milled to 95 % passing 45 μm are far apart (about 30 %). This suggests that the ash value of the -25 μm floats fraction may be slightly exaggerated, but not as much as for the Greenside coal.

5.3.1.3 Grootegeluk coal

The global results for the "as is" Grootegeluk coal and the milled samples are presented in Figure 5.29. It can be seen that there is a great difference in the washability characteristics of the +25 μm and the -25 μm fractions. The cumulative floats points for the -25 μm fractions of the "as is" sample and the samples milled to 95 % passing 150, 75 and 45 μm lie well above the points of the +25 μm fractions of the same samples.

It may also be seen that the band for the +25 μm fractions is very narrow. This shows that the washability characteristics of the +25 μm fractions remain virtually unchanged on progressive milling. The washability characteristics of the -25 μm fractions, however, are affected by milling. This is evident from the broader band for this size fraction. In this band, the points of the sample milled to 95 % passing 45 μm have the lowest ash values, implying that this sample has the best washability characteristics. The progressive "sweetening" of the -25 μm fractions on progressive milling is clearly evident from Figure 5.29, reflecting the movement of better quality material from the +25 μm fraction.

The density distributions for the "as is" sample and the sample milled to 95 % passing 45 μm in Figures 5.18 and 5.19 indicated very large differences in the proportions of +25 μm and -25 μm material in the <1,35 relative density interval. In the "as is" sample the difference was more

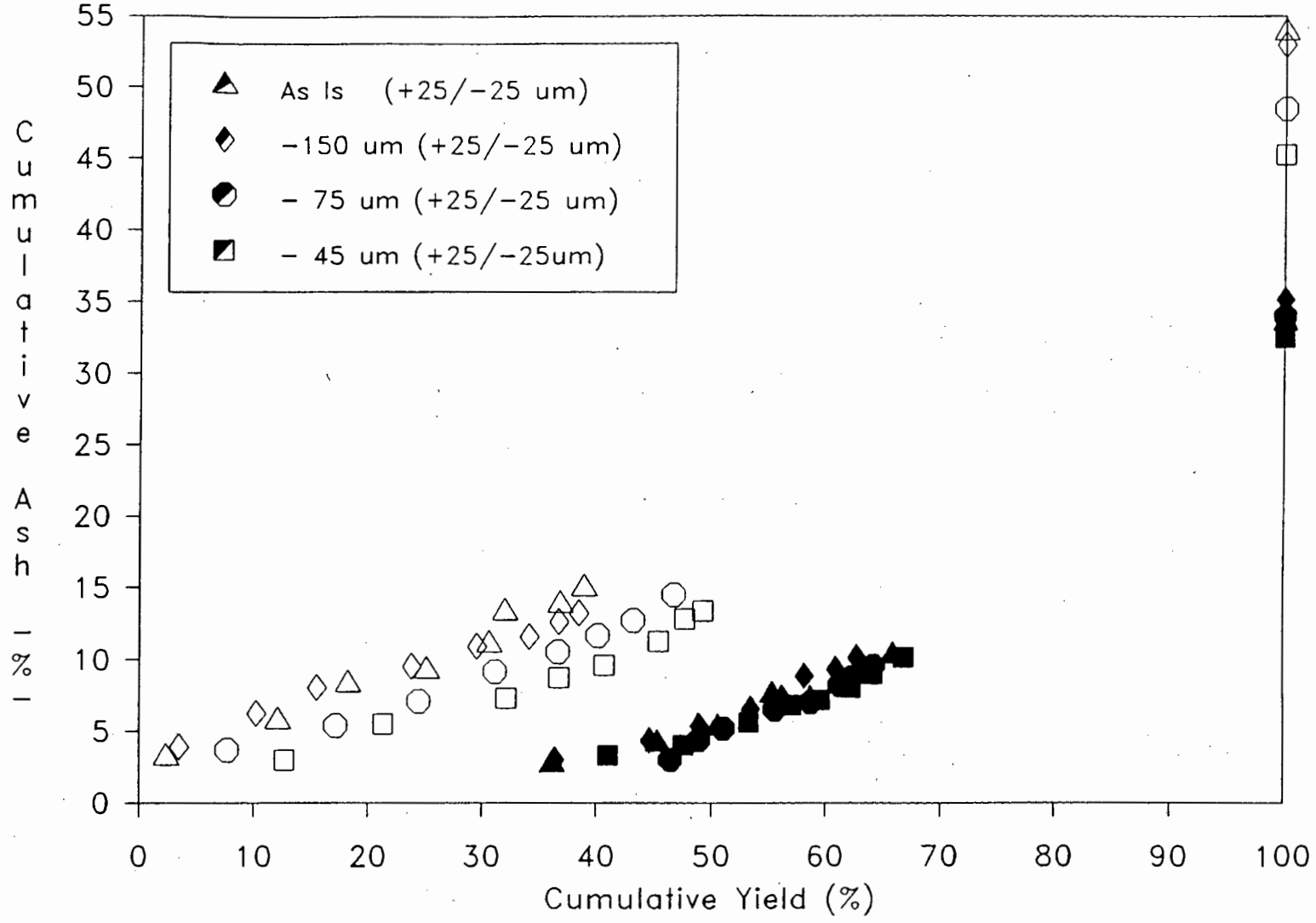


Figure 5.29 Cumulative floats data for the +25 μm and -25 μm fractions of Grootegeluk coal milled to various top sizes

than 30 %, while in the milled sample it was nearly 30 %. One would therefore expect a 30 % difference in yield at any ash value. The cumulative floats points in Figure 5.29 clearly show a 30 % difference between the +25 μm and the -25 μm fractions. It is therefore expected that, unlike the Greenside and Rietspruit coals, the observed ash values in the floats fraction of the -25 μm samples are a good measure of the true values.

5.3.1.4 Discussion

The examination of the washability data of all three coals shows that the +25 μm fractions in all cases have better washability characteristics than the corresponding -25 μm size fractions. This was expected from the comparison of the density distributions in section 5.2. However, for the Greenside and Rietspruit coals, the difference in theoretical yields of products of low ash values (8 % ash and less) is greater than expected. This raises questions as to the validity or accuracy of the ash contents of the low density floats fractions of these coals. For this reason, the relative density distribution of the "as is" and each of the milled samples of each coal were not combined into overall washability curves, as this would have been misleading.

The accuracy of the float and sink results obtained on the +25 μm and -25 μm size fractions of each coal was checked by independent gravimetric float and sink analyses, and by oil agglomeration. This work is described in the next section.

5.3.2 **Corroboration**

Since the centrifugal float and sink method employed in this thesis was relatively new and had not been used extensively on -25 μm material, it was considered important to try to

check the results obtained. A gravimetric method was chosen for the comparison of float and sink analyses on the +25 μm fractions of selected samples of each coal. This method is discussed in section 3.4.3.2 of this thesis.

As there is no approved float and sink method for particle sizes smaller than 25 μm, oil agglomeration, an extremely efficient process for separating clean coal from mineral matter at ultrafine particle sizes, was used to check the results on the -25 μm fractions of selected samples of each coal. The method used is described in section 3.4.4 of this thesis. Oil agglomeration tests were also carried out on some of the +25 μm fractions, out of interest.

5.3.2.1 Gravimetric float and sink analysis on +25 μm material

The +25 μm size fractions of the Greenside, Rietspruit and Grootegeluk coals milled to 95 % passing 150 μm were subjected to gravimetric float and sink analysis at relative densities of 1,45, 1,50 and 1,60. The raw data may be found in Appendix C.2.

Figures 5.30 to 5.32 present the results of the gravimetric float and sink analyses together with the respective cumulative floats versus cumulative ash points obtained using the centrifugal float and sink method.

From these figures it can be seen that there is very good agreement between the two sets of data for all three coals. In each case the gravimetric data points are in close proximity to the cumulative floats curve obtained by centrifugal float and sink analysis of the same coal sample. This indicates that the centrifugal method gives a reliable

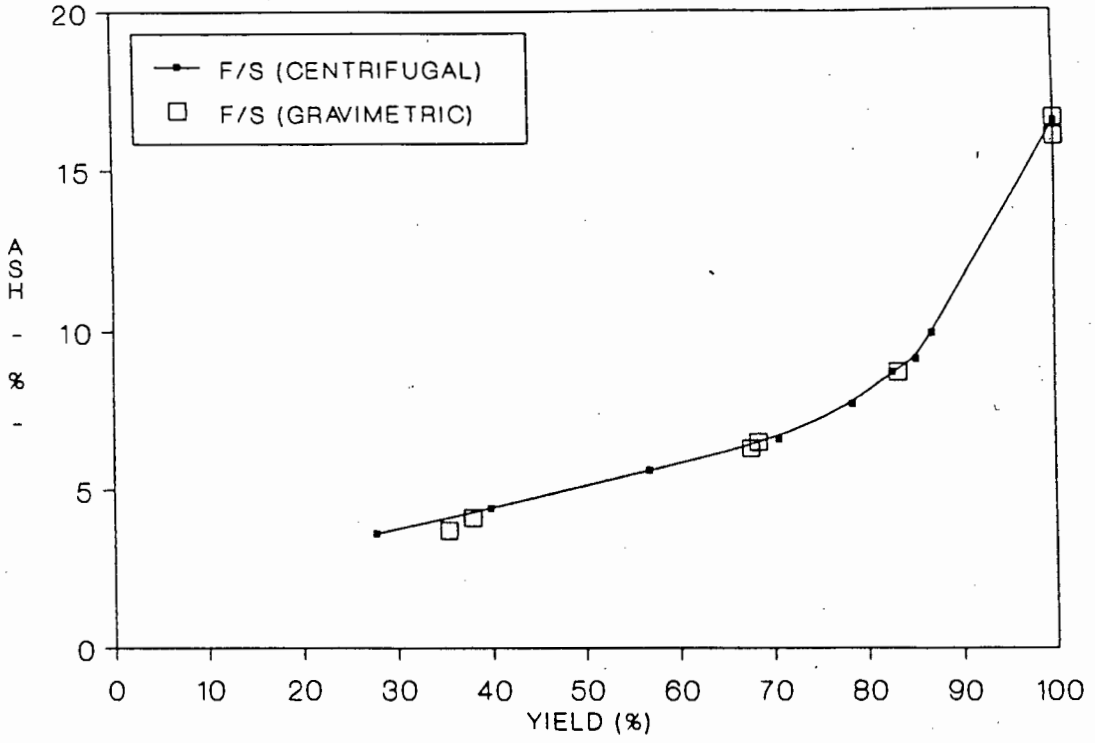


Figure 5.30 Gravimetric vs. centrifugal float and sink data for the +25 μm fraction of Greenside coal milled to 95 % passing 150 μm

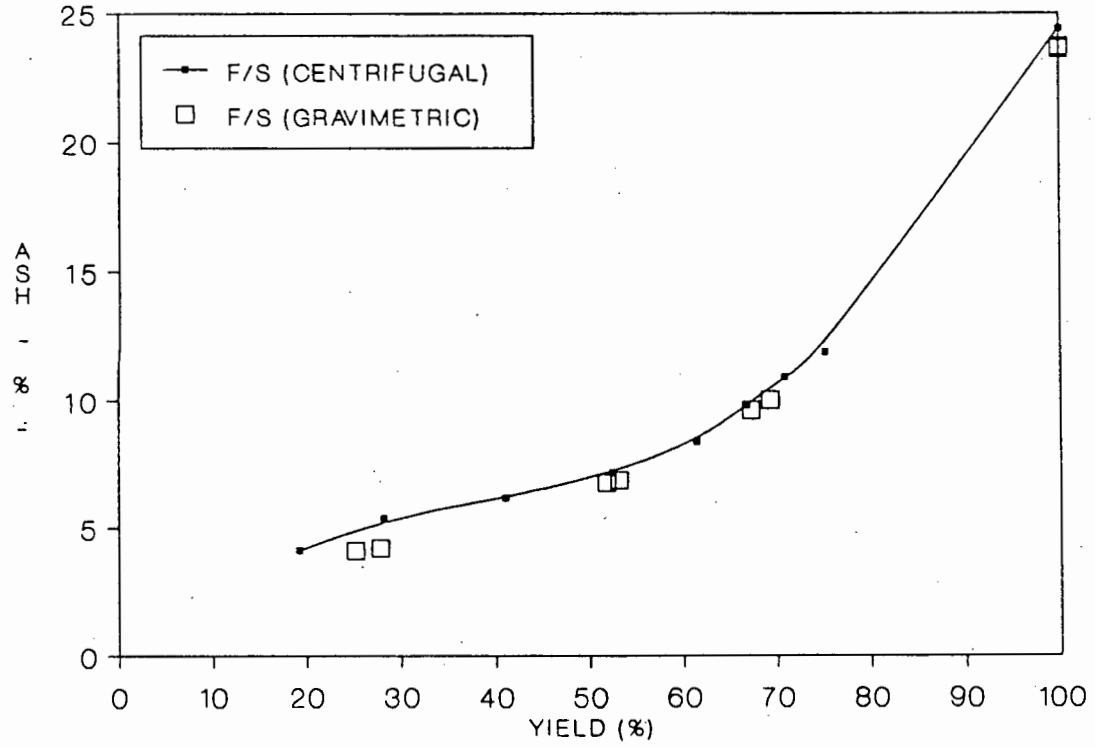


Figure 5.31 Gravimetric vs. centrifugal float and sink data for the +25 μm fraction of Rietspruit coal milled to 95 % passing 150 μm

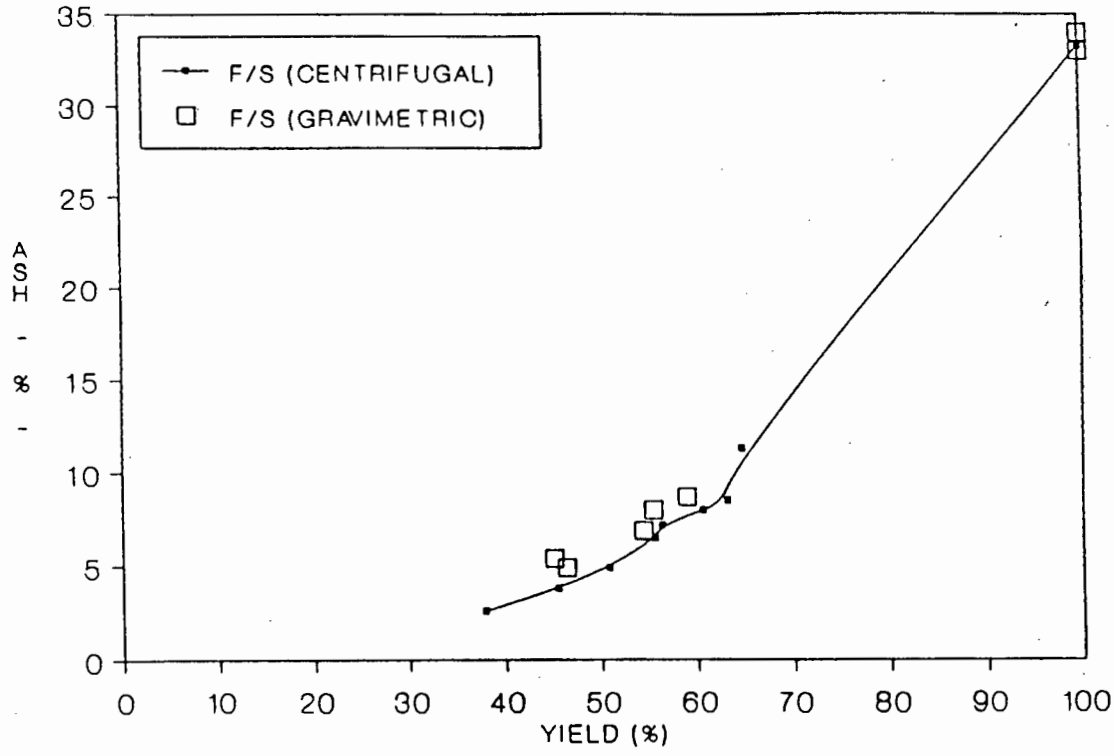


Figure 5.32 Gravimetric vs. centrifugal float and sink data for the +25 μm fraction of Grootegeluk coal milled to 95 % passing 150 μm

assessment of the separability of a coal sample of particles greater than 25 μm in size.

5.3.2.2 Oil agglomeration tests

Oil agglomeration experiments were carried out on the +25 μm and -25 μm fractions of samples of Greenside, Rietspruit and Grootegeluk coal milled to 95 % passing 75 and 45 μm. Tables of the resulting data can be found in Appendix D. The agglomeration results on the two size fractions of the coals milled to 95 % passing 75 μm followed the same trends as those for the samples milled to 95 % passing 45 μm ; thus, only the results for the sample milled to 95 % passing 45 μm material will be discussed here.

In the sections below, the agglomeration data are presented together with the respective cumulative floats data for each coal. Each coal will be discussed separately.

(a) Greenside coal

Figures 5.33 and 5.34 exhibit the data for the +25 μm and -25 μm fractions, respectively, of Greenside coal milled to 95 % passing 45 μm.

From Figure 5.33 it can be seen that the agglomeration points for the +25 μm fraction lie well above the cumulative floats curve. This indicates that better separations were achieved with the float and sink method than with oil agglomeration. For example, up to 92 % yield of 8 % ash coal was recovered on float and sink testing, whereas only 82 % yield of the same quality was obtained with oil agglomeration. It may be deduced that the separability of a coal sample of particles greater than 25 μm in size is better indicated by float and sink analysis than by oil agglomeration.

The agglomeration points for the -25 μm fraction, however, as given in Figure 5.34, fall well below the cumulative floats points. Here an 80 % yield of 6 % ash coal was obtained by oil agglomeration compared to approximately 55 % of the same quality coal by float and sink analysis. Yields of 5 % ash coal produced by oil agglomeration and float and sink analysis were 62 % and 38 %, respectively. Thus, oil agglomeration appears to give a better indication of separability of -25 μm particles than float and sink analysis.

The washability data for the -25 μm fraction of the Greenside coal are therefore not reliable, as was suspected

from the discussion in section 5.3.1.1. In that section, the greater than expected differences in yield at any ash value between the +25 μm and -25 μm fractions were ascribed to exaggerated ash values in the low density floats fractions of the -25 μm size fractions. Small amounts of ultrafine ash material reporting to the floats at low relative densities can have devastating effects on the washability curve, as was already noted in the discussion of the preliminary experiments in section 4.3.

The unrealistically high ash values at lower relative densities (yields) could cause almost the entire cumulative floats curve to be displaced. Because of the shape of the cumulative floats curve of the Greenside coal (owing to the presence of such a large proportion of light middlings material), a 1 % error in ash content results in a 20 to 30 % error in theoretical yield at any ash content. Consequently, the agglomeration points lie far below the cumulative floats points. The relative density distribution data (section 5.2), however, would be little affected by small errors in the ash values; the conclusions made in section 5.2 would remain valid.

It may be deduced that if the volume to surface area ratio in a coal sample is large (large particles) then density separations are preferred. When the volume to surface area ratio is small (small particles), density separations are hindered by large amounts of near density material and physical forces. Accurate density separations would require very long centrifugation times and high speeds (see Table 2.3 and accompanying discussion). Therefore, for particle sizes finer than 25 μm, oil agglomeration is the more efficient separation method.

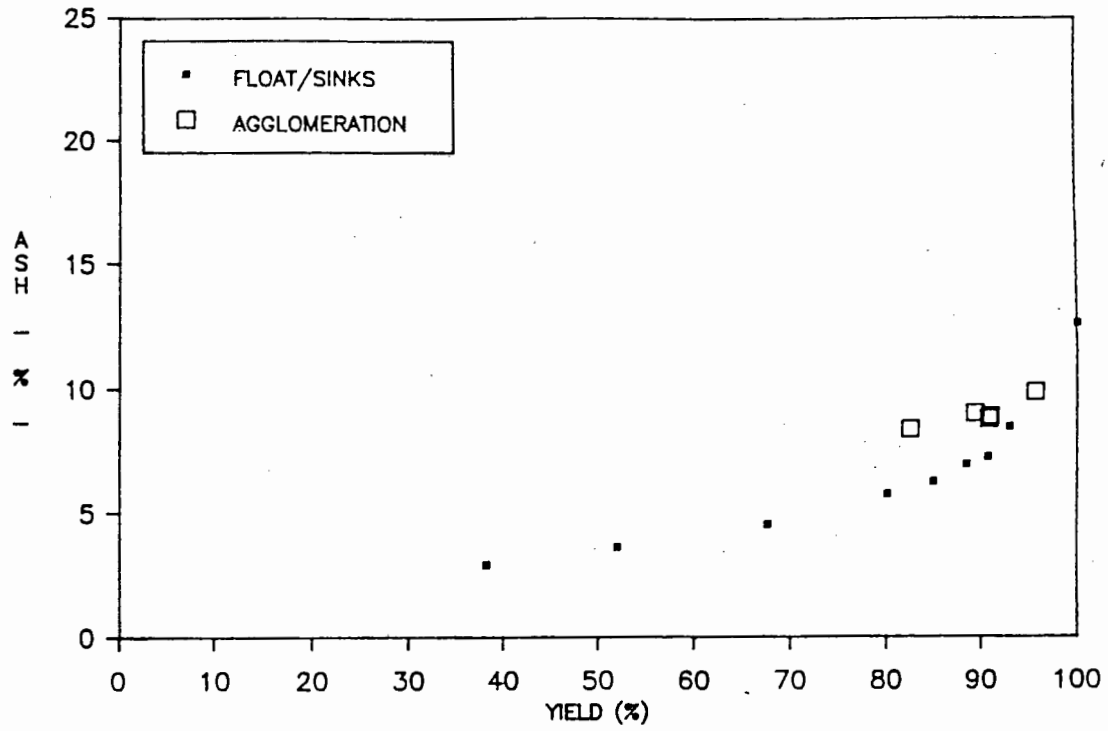


Figure 5.33 Cumulative floats data with agglomeration points for the +25 μm fraction of Greenside coal milled to 95% passing 45 μm

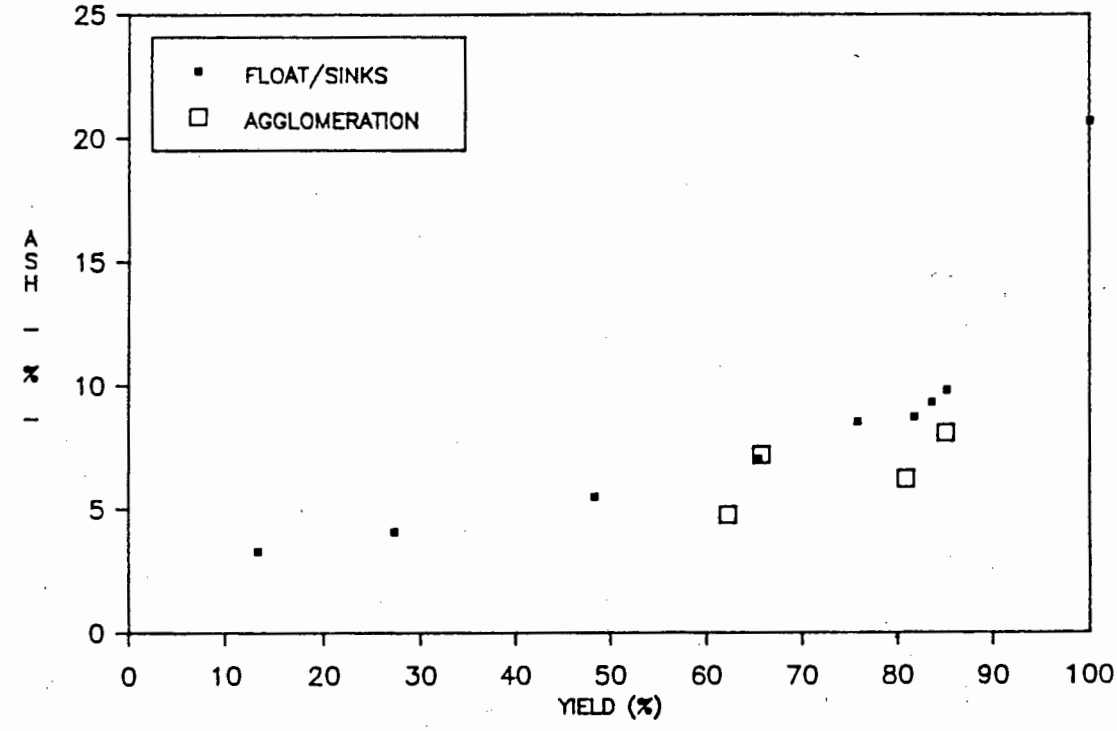


Figure 5.34 Cumulative floats data with agglomeration points for the -25 μm fraction of Greenside coal milled to 95% passing 45 μm

(b) Rietspruit coal

Figure 5.35 represents the agglomeration points and the cumulative floats data for the +25 μm size fraction of Rietspruit coal milled to 95 % passing 45 μm. Better separations were obtained by float and sink analysis than by oil agglomeration since the agglomeration points lie far above the cumulative floats points. The best grade achieved by agglomeration is a 11 % ash product (70 % yield). The cumulative floats points, however, indicate that good yields (60 %) of higher grade material (7 % ash) could be obtained by float and sink analysis.

For the -25 μm size fraction of Rietspruit coal, given in Figure 5.36, better separations could be achieved with agglomeration than with float and sink analysis. About 60 % yield of 7 % ash coal was obtained by agglomeration while the maximum yield of the same product obtainable by float and sink analysis, was 42,5 %. This difference is not as pronounced as with the Greenside coal. In the low ash, low yield region of the chart the efficiency of separation for float and sink analysis and oil agglomeration was the same.

These observations bear out the conclusions made in (a) above, that in general, the separability of +25 μm particles of coal is best determined by float and sink analysis, and of -25 μm by oil agglomeration.

(c) Grootegeluk coal

The cumulative floats data and the agglomeration points for the +25 μm and -25 μm size fractions of Grootegeluk coal milled to 95 % less than 45 μm are presented in Figures 5.37 and 5.38.

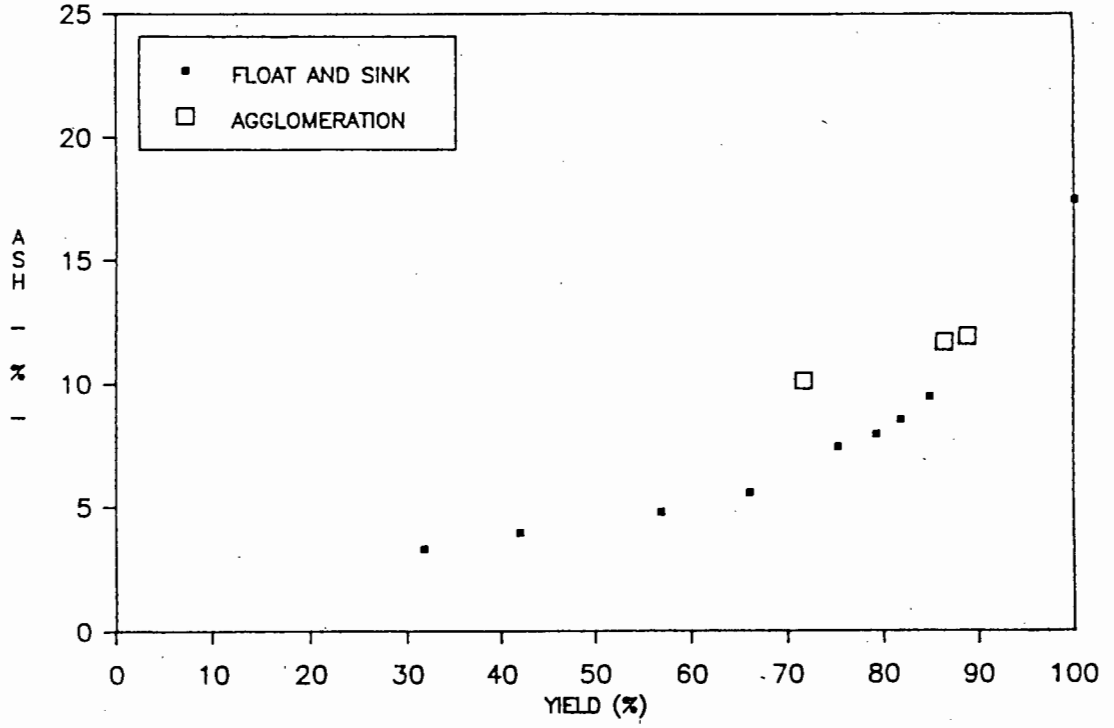


Figure 5.35 Cumulative floats data with agglomeration points for the +25 μm fraction of Rietspruit coal milled to 95% passing 45 μm

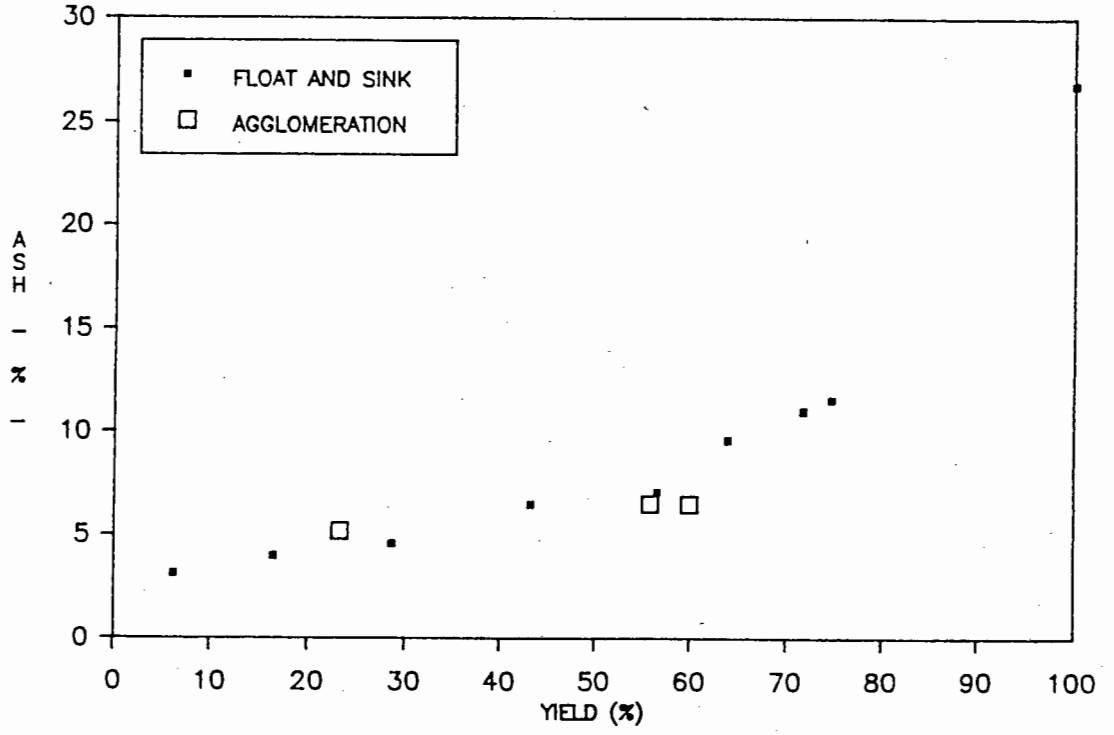


Figure 5.36 Cumulative floats data with agglomeration points for the -25 μm fraction of Rietspruit coal milled to 95% passing 45 μm

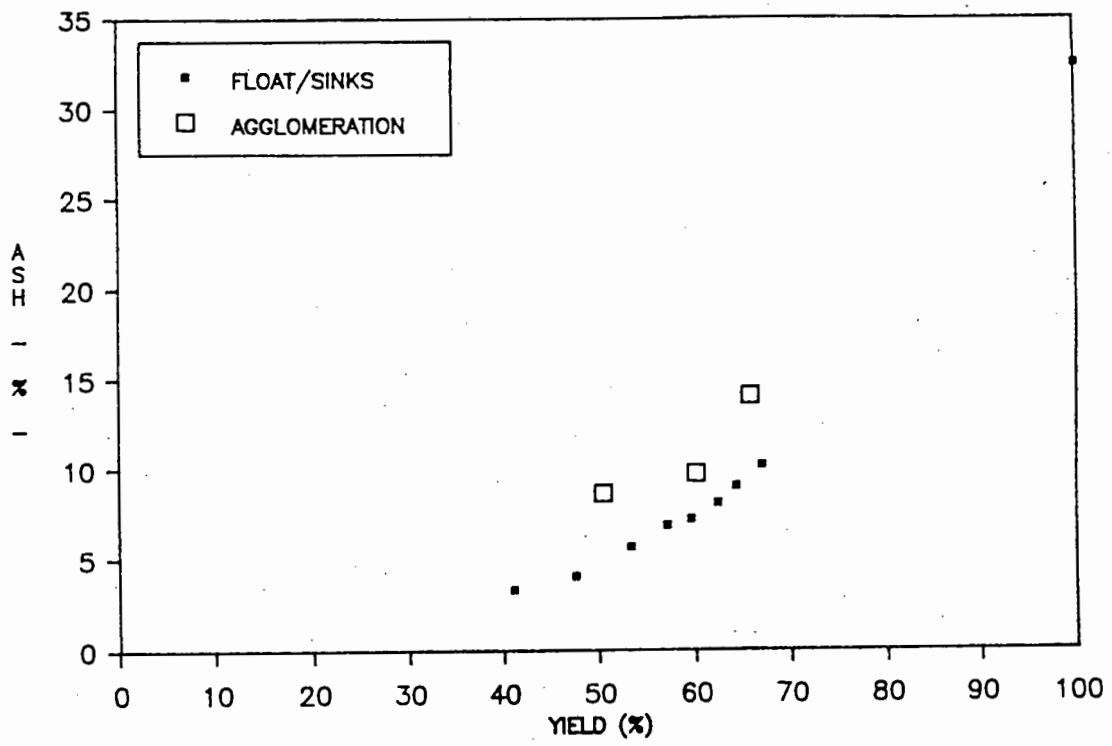


Figure 5.37 Cumulative floats data with agglomeration points for the +25 μm fraction of Grootegeluk coal milled to 95% passing 45 μm

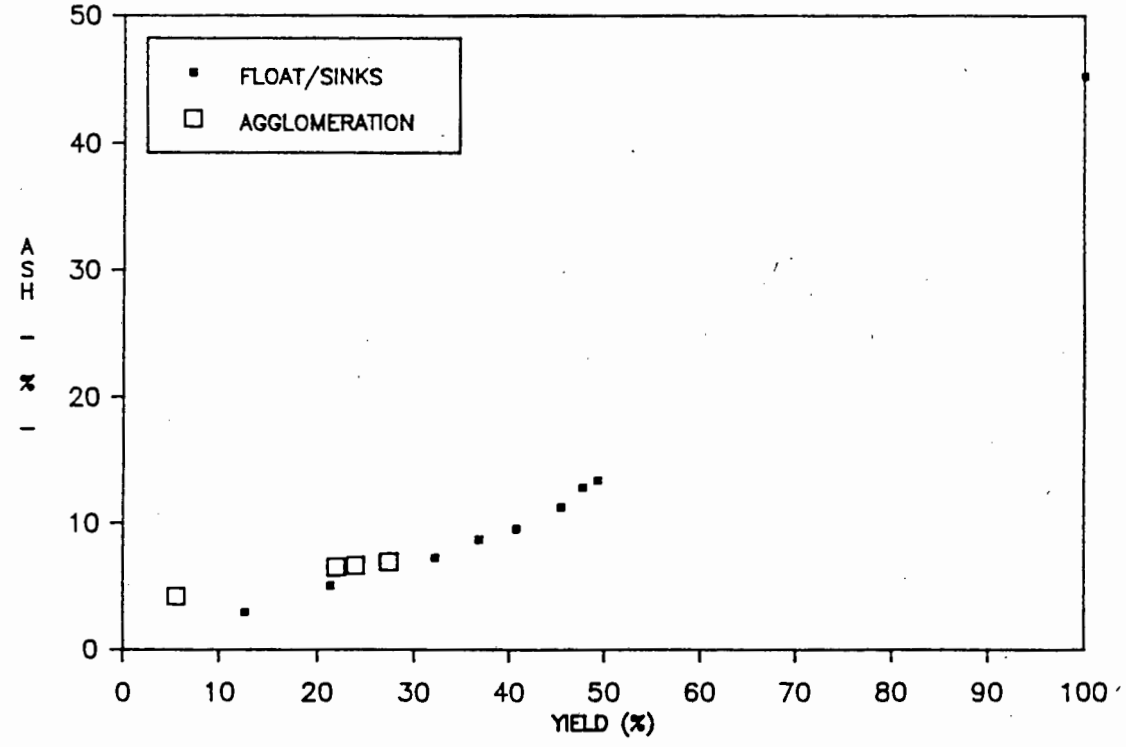


Figure 5.38 Cumulative floats data with agglomeration points for the -25 μm fraction of Grootegeluk coal milled to 95% passing 45 μm

As in the Greenside and Rietspruit coals, the agglomeration data for the +25 μm fraction of Grootegeluk coal (Figure 5.37) lie above the cumulative floats data, thereby making the float and sink method the more efficient separation technique.

From Figure 5.38 it can be seen that the agglomeration points for the -25 μm material are in close proximity of the cumulative floats curve, implying that the float and sink method is as efficient as oil agglomeration, as would be expected from the washability characteristics (section 5.3.1.3).

It must be remembered, however, that Grootegeluk coal, due to its very high vitrinite content, is more easily oxidized than the Rietspruit and Greenside coals and thus less sensitive to oil agglomeration. Precautions were taken to keep oxidation at a minimum, but the actual state of oxidation was not known. In view of this, the agglomeration results for Grootegeluk coal may be poorer than they might otherwise have been.

5.3.2.3 Conclusions

Gravimetric float and sink analysis confirmed the results of centrifugal float and sink analysis on +25 μm samples. Thus, centrifugal float and sink analysis can be used with confidence on coals of particle sizes down to 25 μm.

Below 25 μm, oil agglomeration is a more efficient process for separating clean coal from mineral matter. Oil agglomeration showed that the ash values obtained for some of the floats fractions of the -25 μm size fractions were incorrect. This is thought to be the result of small amounts of ultrafine high ash in the floats, which can result in enormous errors in the theoretical yield at any

ash content. Errors were detected in the Greenside and Rietspruit coals. These errors would not have any significant effect on the relative density distributions, however, and the discussion and conclusion of section 5.2 remain valid.

5.5 LIBERATION EFFICIENCY

The degree of liberation for each coal and its milled subsamples was assessed by calculating the liberation efficiencies as described in section 2.5.4. The cumulative floats data of the +25 μm and the -25 μm fractions of the unmilled and milled samples of each coal were smoothed and reconstituted in the correct mass proportions to yield cumulative floats points of the composite samples ("as is" and milled to 95 % passing 150, 75 and 45 μm). M-curves were prepared from the data and the degree of liberation was assessed by calculating the liberation efficiency L using the formula in section 2.5.3. The smoothed cumulative floats and M-curve data and tables of polynomial constants can be found in Appendix E.

Table 5.17 lists the computed liberation efficiencies of the milled and unmilled samples of Greenside, Rietspruit and Grootegeluk coals.

From Table 5.17 it can be seen that the liberation efficiency values for Greenside and Rietspruit coals were similar and lay between 62 % and 73 %, while the values for the Grootegeluk coal were much higher and ranged from 85 % to 92 %. This indicated that the Grootegeluk coal was more liberated than the Greenside and Rietspruit coals, regardless of the degree of milling.

Table 5.17
Calculated liberation efficiencies og Greenside, Rietspruit and Grootegeluk coals milled to various top sizes

Particle Size (μm)	Coals		
	Greenside	Rietspruit	Grootegeluk
As Is	64,3	62,4	91,9
95 % -150	64.8	66,4	87,6
95 % - 75	64,6	66,7	87,0
95 % - 45	71,6	73,3	85,2

For Greenside coal the liberation efficiencies of the unmilled sample and the samples milled to 95 % passing 150 lm and 75 μm were 64,3, 64,8 and 64,6 %, respectively. This shows that milling to these sizes did not increase liberation in this coal. Only by milling to a considerably finer top size of 45 μm, did the liberation efficiency increase to 71,6 %.

The liberation efficiencies of Rietspruit coal exhibited a similar trend to Greenside coal. The unmilled sample had a liberation efficiency of 62,4 %, whereas the samples milled to 95 % passing 150 and 75 μm had liberation efficiencies of 66,4 and 66,7 %,respectively. Reducing the particle size to 95 % passing 45 μm increased the liberation efficiency to 73,3 %.

Grootegeluk coal, as was apparent from size and density distributions (Section 5.1 and 5.2) differed greatly from the Greenside and Rietspruit coals. This is also reflected in the liberation efficiencies. The unmilled sample and the samples milled to 95 % passing 150 and 75 μm had liberation efficiencies between 87,0 and 91,9 % making this the most liberated of the three coals (by this definition). The liberation efficiency for the sample milled to 95 % passing

45 μm was only 85,2 %, reflecting a decrease in liberation on grinding: this was to be expected from the relative density distributions (section 5.2.3).

Comparing these calculated liberation efficiencies with the percentage liberation determined from the amounts of misplaced material (section 5.2.4 and Tables 5.15 and 5.16) shows some marked anomalies, however. Based on the amount of misplaced ash and "coal" (Table 5.16) Greenside is the most liberated of the three coals, followed by Grootegeluk and Rietspruit. Based on the amount of misplaced ash and macerals (Table 5.15) the Greenside coal is still the most liberated by far, with Rietspruit next and Grootegeluk a poor third.

Strictly speaking, the calculated liberation efficiencies and the percentage liberation based on misplaced material are incorrect for the Greenside and Rietspruit coals - as was seen in section 5.3.2.2. Oil agglomeration tests showed the washability characteristics of the -25 μm fractions of these two coals to be much better than indicated by the float and sink analysis. This means that some of the ash (about 2 to 3 gram) that was found in the <1,60 relative density zones should have reported to the >1,60 relative density zone. The effect of this would be to reduce the amount of misplaced material by 2 to 3 % and increase the percentage or overall liberation by the same amount.

Thus, based on the amount of misplaced ash and macerals, the Greenside coal would remain the most liberated by far, followed by Rietspruit and Grootegeluk. Based on misplaced ash and "coal", Greenside coal would still be ahead, with the Rietspruit coal possibly being more liberated than the Grootegeluk coal when milled to 95 % passing 45 μm. (The values for Grootegeluk would be unchanged as the

agglomeration tests did not give any better results than the float and sink analyses).

Unfortunately, it is impossible to predict what the effect on the calculated liberation efficiencies would be of correcting the washability data of the -25 μm fractions of the Greenside and Rietspruit coals. However, it is not unlikely that the values would overtake that of Grootegeluk confirming the order as calculated on the basis of misplaced material. Thus it would appear that until a better method is devised to measure the separability of -25 μm coal, the new measure of liberation based on misplaced material gives a better idea of the liberation of a sample than the value of liberation efficiency L calculated from the formula.

5.5.1 **Conclusions**

The liberation efficiencies, calculated from the M-curve, for the Greenside and Rietspruit coals were considerably smaller than for Grootegeluk, implying that the latter was the most liberated coal.

This contradicts the results from the density distribution studies in section 5.2, which showed Grootegeluk coal to be the least liberated and the Greenside coal to be the most liberated coal. However, the errors in the washability determination of the -25 μm fractions of the Greenside and Rietspruit coals suggest that the measure of liberation determined from the density distribution results is the more accurate.

5.6 **SUMMARY**

5.6.1 **Size analyses**

A general trend of increasing proportions of -25 μm material with prolonged milling times was observed in all three coals. The milling characteristics of all three coals were similar for the "as is" samples and the samples milled to 95 % passing 150 and 75 μm, but differed greatly in the samples milled to 95 % passing 45 μm, Grootegeluk coal being the least millable. This was ascribed to the exceptionally high vitrinite and ash content of this coal.

The ash content of the +25 μm fraction of each coal was lower than the ash content of the -25 μm fraction. The largest difference in ash between the coarse and fine fractions was observed for Grootegeluk. The gradual decrease in ash content of the -25 μm fractions with prolonged milling was ascribed to the movement of "better quality" coal from the coarser fractions into the finer sizes. The ash content of the +25 μm fractions also improved with milling, indicating that while this "better quality" coal had a lower ash content than the -25 μm fraction in each case, it had a higher ash content than the average of the +25 μm fraction.

The size distributions within the -25 μm fractions were similar for all three coals. It was evident from the results that prolonged milling produced predominantly 10 to 20 μm material but did not reduce the available fines to smaller particle sizes. This was probably due to the type of mill (rod mill) being used; a different type of mill (eg. ball mill) would be needed to reduce the particles to even smaller sizes.

5.6.2 **Density distributions**

The relative density distributions showed that the Greenside coal contained a fairly large proportion (about 20 % by mass) of low density (<1,35 R.D.) material, a very large proportion (about 67 %) of light middlings (1,35 - 1,60 R.D.) material and a somewhat smaller proportion (about 13 %) of heavy middlings (>1,70 R.D.) material. The +25 μm size fractions contained a disproportionate amount of <1,35 relative density material, while light and heavy middlings were concentrated in the -25 μm size fractions.

On milling, +25 μm particles of >1,50 R.D. were selectively broken into -25 μm particles of <1,45 R.D. The density distribution of a fixed mass of starting material was observed to change as follows on progressive milling to 95 % passing 150, 75 and 45 μm : the mass of material in the 1,35 to 1,45 R.D. interval increased steadily, the mass of 1,45 to 1,50 R.D. material also increased, while the mass of <1,35 relative density and 1,50 to 1,70 R.D. material decreased. The mass of sinks at 1,70 relative density remained virtually unchanged.

Maceral allocations into specific R.D. zones, although speculative, showed clearly that mineral matter was moving out of the <1,35 and 1,35 - 1,60 R.D. zones, while maceral material was moving out of the >1,60 R.D. zone. The Greenside coal was therefore becoming more liberated in terms of ash and "coal" content upon milling to finer and finer sizes.

The relative density distributions of the +25 μm and -25 μm fractions of the Rietspruit coal were very similar to those of the Greenside coal. The proportions of >1,70 R.D. material were much larger in both fractions though, (up to 33 %) because the Rietspruit coal had a much higher ash

content than Greenside coal. Also, the distribution over the 1,35 to 1,60 R.D. range was more even and flatter than in the Greenside coal.

Compared to the Greenside coal, the pattern of breakage of the Rietspruit coal by relative density was the same, but more intense, while the products of breakage were quite different. The products consisted predominantly of 1,35 - 1,40 and 1,45 - 1,65 R.D. material. Overall, the proportions in the <1,35 and >1,70 R.D. intervals decreased on milling while the proportions in the 1,35 to 1,65 R.D. range increased.

Mineral and maceral allocations into different zones of the R.D. distribution clearly indicated the movement of mineral material out of the vitrinite and inertinite R.D. zones into the heavy middlings zone, as well as some movement of maceral material out of the heavy middlings zone into the maceral zones.

The density distribution pattern of Grootegeluk coal was distinctly different from the Greenside and the Rietspruit coals. In the +25 μm fraction of the original sample of Grootegeluk coal, the proportions of <1,35 and >1,70 R.D. material were very large (35 % or more), while the proportions in the intermediate R.D. intervals (1,35 to 1,60 R.D.) were very small (mostly less than 5 %). The -25 μm size fractions were dominated by >1,70 R.D. material (50 to 60 %), while the proportions in the other R.D. intervals were low ($\leq$ 10 %).

The distribution pattern changed only slightly on milling to progressively finer sizes. No particular pattern of breakage of the +25 μm material was observed. The largest increase in -25 μm material was observed in the <1,35 R.D. interval. Generally, the +25 μm material within each R.D.

interval was reduced to -25 μm material, with a minimum of movement of material between R.D. intervals.

These observations were also reflected in the mineral and maceral allocations to R.D. zones. The maceral allocations were partly confirmed by photographs showing unliberated vitrinite in the >1,52 R.D. interval.

From the maceral and mineral allocations a new measure of liberation, based on the amount of misplaced material in the respective R.D. zones, was proposed. According to this method, Greenside coal was the most liberated coal, followed by Rietspruit coal and lastly by Grootegeluk coal. In terms of misplaced "coal" and ash, however, Greenside coal would be the most liberated, followed by Grootegeluk and Rietspruit coal.

5.6.3 **Washability characteristics**

The +25 μm size fractions of all three coals exhibited better washability characteristics than the -25 μm fractions, as was expected from the density distributions. The cumulative floats points for Greenside coal seemed distorted, however, when compared with the relative density distributions, due to exaggerated ash values in the -25 μm fractions. This was ascribed to inefficient separation by float and sink analysis. The cumulative floats points for the Rietspruit and Grootegeluk coals were reliable and compared well with the density distribution results.

Corroboration of the washability results from the new float and sink method with a standard gravimetric float and sink method for the +25 μm size fractions and oil agglomeration for the -25 μm size fractions showed that the new method is reliable down to particle sizes of 25 μm. For particle sizes finer than 25 μm, oil agglomeration separates clean

coal from ash material more efficiently than the centrifugal float and sink method. It is thought that ultrafine ash reporting to the floats fractions at low relative densities is the cause of these errors.

5.6.4 **Liberation efficiencies**

The liberation efficiencies, calculated from the M-curve, for the Greenside and Rietspruit coals were considerably smaller than for Grootegeluk, implying that the latter was the most liberated coal. This contradicts the results from the density distribution studies which showed Greenside coal to be the most liberated.

Errors in the float and sink analysis of the -25 μm fractions of the Greenside and Rietspruit coals, in which fine high ash material reported incorrectly to the floats fractions at low relative densities, would seriously impact on the calculated liberation efficiencies, while not affecting the new measure of liberation (based on misplaced material) by much. Thus, in the determination of overall liberation at the ultrafine sizes, it is suggested that this latter method be used.

CHAPTER 6

CONCLUSIONS

This work showed that all three coals would have to be milled to much finer sizes in order to increase liberation substantially. Milling to a top size of 45 μm improved the liberation in the Greenside and Rietspruit coals to a certain degree, but did not improve the liberation of the Grootegeluk coal. A different method of size reduction (eg ball milling) would have to be used to produce partices of finer sizes than investigated in this thesis.

The density distributions of the Greenside and Rietspruit coals indicated the presence of large proportions of intermediate density material, often referred to as the 'middlings hump' (Sanders and Brookes, 1986) and associated with poor liberation. Petrographic analysis, however, showed that both coals had a high inertinite content. As inertinite has a relative density between 1,40 and 1,60, these coals would therefore be expected to have large proportions of material in the intermediate relative density region. Grootegeluk coal had only small proportions of intermediate material because it contained relatively little inertinite.

Macerals were allocated to the vitrinite and exinite, the inertinite and the heavy middlings zones of the relative density distributions of each coal. The amounts of misplaced maceral and ash material in each zone were then added up to give an indication of liberation. Greenside coal was the most liberated, Rietspruit coal came second, and Grootegeluk coal was the least liberated.

For all three coals the washability characteristics of the +25 μm size fractions were better than those of the -25 μm fractions. The new centrifugal float and sink method was found to be reliable for the +25 μm size fractions, but gave erroneous ash values for the -25 μm size fractions. This is thought to be due to ultrafine ash reporting to the floats fraction at low relative densities. As a result, it is not possible to calculate the theoretical yield of low-ash coal in the reconstituted samples. The centrifugal method needs to be improved or modified for -25 μm material, or new methods, based on oil agglomeration or optical analysis for example, need to be developed.

Liberation efficiencies, based on the M-curve gave misleading results, because of erroneous ash values in the washability data. The new measure of liberation, based on misplaced ash and maceral matter in the respective 'maceral' density zones, gives more realistic results.

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APPENDIX A

SIZE DISTRIBUTION DATA

Table A.1

Size distribution in the -25 μm fraction of Greenside, Rietspruit and Grootegeluk coals, when milled to various sizes.

Particle size (μm)	Mass (%) As is	Mass (%) 95% -150 μm	Mass (%) 95% -75 μm	Mass (%) 95% -45 μm
Greenside:				
+23,5	8,6	11,5	11,4	15,5
-23,7+10,5	32,8	34,6	37,2	39,8
-10,5+ 5,0	32,4	31,6	31,5	28,6
- 5,0+ 1,2	24,4	21,6	19,7	16,0
- 1,2	1,8	0,7	0,2	0,2
Total	100,0	100,0	100,0	100,0
Total Fraction*	32,1	40,0	55,3	83,0
Rietspruit:				
+23,5	13,6	18,0	14,8	17,3
-23,7+10,5	34,4	35,9	37,1	39,0
-10,5+ 5,0	31,4	28,6	29,5	27,6
- 5,0+ 1,2	19,8	16,6	18,2	15,7
- 1,2	0,8	0,9	0,4	0,4
Total	100,0	100,0	100,0	100,0
Total Fraction*	30,4	39,6	55,5	90,9
Grootegeluk:				
+23,5	7,5	18,9	20,3	15,7
-23,7+10,5	35,7	35,5	35,8	40,4
-10,5+ 5,0	32,5	26,8	26,0	26,6
- 5,0+ 1,2	22,4	18,4	16,9	16,1
- 1,2	1,9	0,4	1,0	1,2
Total	100,0	100,0	100,0	100,0
Total Fraction*	41,2	41,7	56,2	74,5

* Total on the basis of 100 parts of sample before screening into +25 μm and -25 μm fractions.

Table A2
Distribution in the -25 μm size fractions of Rietspruit coal, based on 100 gram of feed

Size Fraction (μm)	Mass in Size Fraction (gram)			
	As Is	95 % -150 μm	95 % -75 μm	95 % -45 μm
+23,5	4,13	7,13	8,21	15,73
-23,5+10,5	10,46	14,22	20,59	35,45
-10,5+ 5,0	9,55	11,33	16,37	25,09
- 5,0+ 1,2	6,02	6,56	10,10	14,27
- 1,2	0,24	0,36	0,22	0,36
Total (-25 μm)	30,40	39,60	55,50	90,90

Table A3
Distribution in the -25 μm size fractions of Grootegeluk coal, based on 100 gram of feed

Size Fraction (μm)	Mass in Size Fraction (gram)			
	As Is	95 % -150 μm	95 % -75 μm	95 % -45 μm
+23,5	3,09	7,88	11,41	11,70
-23,5+10,5	14,71	14,80	20,12	30,10
-10,5+ 5,0	13,39	11,18	14,61	19,82
- 5,0+ 1,2	9,23	7,67	9,50	11,99
- 1,2	0,78	0,17	0,56	0,89
Total (-25 μm)	41,20	41,70	56,20	74,50

APPENDIX B

DENSITY DISTRIBUTION DATA

TABLE B1

Density distribution data of Greenside ("as is") coal

Relative Density	+25 µm Fraction Fract. Yield (%)	+25 µm Fraction Cum. Yield (%)	-25 µm Fraction Fract. Yield (%)	-25 µm Fraction Cum. Yield (%)
F 1,35	25,72	25,72	10,81	10,81
1,35 - 1,40	11,45	37,17	7,32	18,13
1,40 - 1,45	16,77	53,94	15,74	33,87
1,45 - 1,50	13,11	67,05	16,05	49,92
1,50 - 1,55	8,63	75,68	16,99	66,91
1,55 - 1,60	6,38	82,06	11,28	78,19
1,60 - 1,65	3,59	85,65	1,38	79,57
1,65 - 1,70	2,38	88,03	3,24	82,81
S 1,70	11,97	100,00	17,19	100,00

TABLE B2

Density distribution data of Greenside (95 % - 150 µm)

Relative Density	+25 µm Fraction Fract. Yield (%)	+25 µm Fraction Cum. Yield (%)	-25 µm Fraction Fract. Yield (%)	-25 µm Fraction Cum. Yield (%)
F 1,35	27,74	27,74	16,18	16,18
1,35 - 1,40	12,10	39,84	8,75	24,93
1,40 - 1,45	16,89	56,73	17,37	42,30
1,45 - 1,50	13,81	70,54	17,07	59,37
1,50 - 1,55	7,88	78,42	13,05	72,42
1,55 - 1,60	4,26	82,68	6,63	79,05
1,60 - 1,65	2,53	85,21	2,56	81,61
1,65 - 1,70	1,68	86,89	0,67	82,28
S 1,70	13,11	100,00	17,72	100,00

TABLE B3

Density distribution data of Greenside (95 % - 75 μm) coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35-	28,93	28,93	16,28	16,28
1,35 - 1,40	14,05	42,98	12,58	28,86
1,40 - 1,45	18,07	61,05	17,85	46,71
1,45 - 1,50	12,11	73,16	21,06	67,77
1,50 - 1,55	7,39	80,55	9,22	76,99
1,55 - 1,60	3,08	83,63	2,96	79,95
1,60 - 1,65	3,01	86,64	3,17	83,12
1,65 - 1,70	1,29	87,93	2,27	85,39
S 1,70	12,07	100,00	14,61	100,00

TABLE B4

Density distribution data of Greenside (95 % - 45 μm) coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35	38,14	38,14	13,22	13,22
1,35 - 1,40	13,83	51,97	14,01	27,23
1,40 - 1,45	15,62	67,59	21,09	48,32
1,45 - 1,50	12,49	80,08	17,07	65,39
1,50 - 1,55	4,84	84,92	10,41	75,80
1,55 - 1,60	3,47	88,39	5,85	81,65
1,60 - 1,65	2,25	90,64	1,86	83,51
1,65 - 1,70	2,30	92,94	1,58	85,09
S 1,70	7,06	100,00	14,91	100,00

TABLE B5

Density distribution data of Rietspruit ("as is") coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35	16,93	16,93	6,63	6,63
1,35 - 1,40	8,90	25,83	6,11	12,74
1,40 - 1,45	11,35	37,18	12,31	25,05
1,45 - 1,50	10,14	47,32	12,75	37,80
1,50 - 1,55	8,23	55,55	10,45	48,25
1,55 - 1,60	8,76	64,31	5,96	54,21
1,60 - 1,65	4,33	68,64	7,55	61,76
1,65 - 1,70	4,98	73,62	5,02	66,78
S 1,70	26,38	100,00	33,22	100,00

TABLE B6

Density distribution data of Rietspruit (95 % - 150 μm) coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35	19,22	19,22	6,19	6,19
1,35 - 1,40	8,93	28,15	8,29	14,48
1,40 - 1,45	12,90	41,05	12,37	26,85
1,45 - 1,50	11,36	52,41	11,62	38,47
1,50 - 1,55	8,98	61,39	12,47	50,94
1,55 - 1,60	5,75	66,74	7,70	58,64
1,60 - 1,65	4,06	70,80	7,93	66,57
1,65 - 1,70	4,31	75,11	4,62	71,19
S 1,70	24,89	100,00	28,81	100,00

TABLE B7

Density distribution data of Rietspruit (95 % - 75 μm) coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35	22,06	22,06	6,00	6,00
1,35 - 1,40	9,59	31,65	7,99	13,99
1,40 - 1,45	12,94	44,58	14,76	28,75
1,45 - 1,50	10,66	55,24	13,37	42,12
1,50 - 1,55	6,90	62,14	12,89	55,01
1,55 - 1,60	7,36	69,50	5,51	60,52
1,60 - 1,65	3,03	72,53	8,53	69,05
1,65 - 1,70	4,45	76,98	4,06	73,11
S 1,70	23,02	100,00	26,89	100,00

TABLE B8

Density distribution data of Rietspruit (95 % - 45 μm) coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35	31,71	31,71	6,71	6,71
1,35 - 1,40	10,21	41,92	9,74	16,45
1,40 - 1,45	14,90	56,82	12,16	28,61
1,45 - 1,50	9,12	65,94	14,56	43,17
1,50 - 1,55	9,23	75,17	13,37	56,54
1,55 - 1,60	4,01	79,18	7,24	63,78
1,60 - 1,65	2,51	81,69	7,90	71,68
1,65 - 1,70	3,06	84,75	2,99	74,67
S 1,70	15,25	100,00	25,33	100,00

TABLE B9

Density distribution data of Grootegeluk ("as is") coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35	36,19	36,19	2,35	2,35
1,35 - 1,40	9,12	45,31	9,71	12,06
1,40 - 1,45	5,26	50,57	6,16	18,22
1,45 - 1,50	4,74	55,31	6,97	25,19
1,50 - 1,55	3,35	58,66	5,47	30,66
1,55 - 1,60	2,74	61,40	1,38	32,04
1,60 - 1,65	1,99	63,39	4,82	36,86
1,65 - 1,70	2,52	65,91	2,08	38,94
S 1,70	34,01	100,00	61,06	100,00

TABLE B10

Density distribution data of Grootegeluk (95 % - 150 μm) coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35	36,43	36,43	3,49	3,49
1,35 - 1,40	8,29	44,72	6,75	10,24
1,40 - 1,45	4,31	49,03	5,26	15,50
1,45 - 1,50	4,43	53,46	8,38	23,88
1,50 - 1,55	2,81	56,27	5,70	29,58
1,55 - 1,60	1,94	58,21	4,61	34,19
1,60 - 1,65	2,74	60,95	2,60	36,79
1,65 - 1,70	1,89	62,84	1,69	38,48
S 1,70	37,16	100,00	61,52	100,00

TABLE B11

Density distribution data of Grootegeluk (95 % - 75 μm) coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35	46,45	46,45	7,69	7,69
1,35 - 1,40	2,46	48,91	9,48	17,17
1,40 - 1,45	2,09	51,00	7,32	24,49
1,45 - 1,50	4,53	55,53	6,64	31,13
1,50 - 1,55	3,16	58,69	5,55	36,68
1,55 - 1,60	2,53	61,22	3,40	40,08
1,60 - 1,65	1,05	62,27	3,08	43,16
1,65 - 1,70	1,91	64,18	3,53	46,69
S 1,70	35,82	100,00	53,31	100,00

TABLE B12

Density distribution data of Grootegeluk (95 % - 45 μm) coal

Relative Density	+25 μm Fraction Fract. Yield (%)	+25 μm Fraction Cum. Yield (%)	-25 μm Fraction Fract. Yield (%)	-25 μm Fraction Cum. Yield (%)
F 1,35	41,04	41,04	12,60	12,60
1,35 - 1,40	6,52	47,56	8,74	21,34
1,40 - 1,45	5,68	53,24	10,77	32,11
1,45 - 1,50	3,79	57,03	4,67	36,78
1,50 - 1,55	2,46	59,49	3,89	40,67
1,55 - 1,60	2,74	62,23	4,72	45,39
1,60 - 1,65	1,92	64,15	2,30	47,69
1,65 - 1,70	2,69	66,84	1,56	49,25
S 1,70	33,16	100,00	50,75	100,00

APPENDIX C

WASHABILITY DATA

C.1 Centrifugal float and sink analysis :

TABLE C1

Washability data for the +25 μm and -25 μm fractions of Greenside ("as is") coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	25,72	3,6	10,81	4,8
1,40	37,17	4,6	18,13	5,5
1,45	53,94	5,8	33,87	6,9
1,50	67,05	7,0	49,92	7,9
1,55	75,68	7,9	66,91	10,7
1,60	82,06	8,8	78,19	13,6
1,65	85,65	9,6	79,57	14,0
1,70	88,03	10,0	82,81	15,3
S1,70	100,00	17,0	100,00	25,2

TABLE C2

Washability data for the +25 μm and -25 μm fractions of Greenside (95 % - 150 μm) coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	27,74	3,6	16,18	6,1
1,40	39,84	4,4	24,93	6,6
1,45	56,73	5,6	42,30	6,9
1,50	70,54	6,6	59,37	8,0
1,55	78,42	7,7	72,42	10,4
1,60	82,68	8,7	79,05	11,6
1,65	85,21	9,1	81,61	12,4
1,70	86,89	9,9	82,28	12,9
S1,70	100,00	16,5	100,00	22,9

TABLE C3

Washability data for the +25 μm and -25 μm fractions of Greenside (95 % - 75 μm) coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	28,93	3,7	16,28	4,5
1,40	42,98	4,5	28,86	5,5
1,45	61,05	5,6	46,71	6,5
1,50	73,16	6,5	67,77	8,0
1,55	80,55	7,5	76,99	10,6
1,60	83,63	8,1	79,95	12,2
1,65	86,64	8,8	83,12	14,0
1,70	87,93	9,4	85,39	15,4
S1,70	100,00	16,5	100,00	21,5

TABLE C4

Washability data for the +25 μm and -25 μm fractions of Greenside (95 % - 45 μm) coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	38,14	2,9	13,22	3,3
1,40	51,97	3,6	27,23	4,1
1,45	67,59	4,5	48,32	5,9
1,50	80,08	5,7	65,39	7,3
1,55	84,92	6,2	75,80	7,9
1,60	88,39	6,9	81,65	8,4
1,65	90,64	7,2	83,51	9,1
1,70	92,94	8,4	85,09	9,6
S1,70	100,00	12,6	100,00	21,0

TABLE C5

Washability data for the +25 μm and -25 μm fractions of Rietsruit ("as is") coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	16,93	3,8	6,63	5,7
1,40	25,83	5,1	12,74	6,1
1,45	37,18	6,3	25,05	6,7
1,50	47,32	7,6	37,80	7,2
1,55	55,55	9,0	48,25	8,8
1,60	64,31	10,3	54,21	10,9
1,65	68,64	11,9	61,76	13,0
1,70	73,62	12,7	66,78	14,5
S1,70	100,00	24,6	100,00	30,2

TABLE C6

Washability data for the +25 μm and -25 μm fractions of Rietspruit (95 % - 150 μm) coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	19,22	4,1	6,19	5,1
1,40	28,15	5,4	14,48	6,0
1,45	41,05	6,2	26,85	6,2
1,50	52,41	8,2	38,47	8,4
1,55	61,39	8,4	50,94	9,1
1,60	66,74	9,8	58,64	11,0
1,65	70,80	10,9	66,57	13,3
1,70	75,11	11,9	71,19	14,4
S1,70	100,00	24,4	100,00	29,2

TABLE C7

Washability data for the +25 μm and -25 μm fractions of Rietspruit (95 % - 75 μm) coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	22,06	4,4	6,00	5,0
1,40	31,65	5,2	13,99	6,6
1,45	44,58	6,3	28,75	7,6
1,50	55,24	7,6	42,12	8,2
1,55	62,14	8,0	55,01	10,9
1,60	69,50	9,9	60,52	11,2
1,65	72,53	10,5	69,05	12,7
1,70	76,98	12,0	73,11	13,7
S1,70	100,00	24,1	100,00	28,1

TABLE C8

Washability data for the +25 μm and -25 μm fractions of Rietspruit (95 % - 45 μm) coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	31,71	3,3	6,71	3,2
1,40	41,92	4,0	16,45	4,0
1,45	56,82	4,8	28,61	4,6
1,50	65,94	5,6	43,17	6,5
1,55	75,17	7,4	56,54	7,1
1,60	79,18	7,9	63,78	9,6
1,65	81,69	8,5	71,68	11,0
1,70	84,75	9,5	74,67	11,6
S1,70	100,00	17,5	100,00	27,2

TABLE C9

Washability data for the +25 μm and -25 μm fractions of Grootegeluk ("as is") coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	36,19	2,8	2,35	3,2
1,40	45,31	4,2	12,06	5,7
1,45	50,57	5,3	18,22	8,3
1,50	55,31	7,3	25,19	9,2
1,55	58,66	7,6	30,66	11,1
1,60	61,40	8,2	32,04	13,3
1,65	63,39	9,4	36,86	13,8
1,70	65,91	10,3	38,94	15,0
S1,70	100,00	33,6	100,00	53,9

TABLE C10

Washability data for the +25 μm and -25 μm fractions of Grootegeluk (95 % - 150 μm) coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	36,43	3,0	3,49	3,9
1,40	44,72	4,3	10,24	6,2
1,45	49,03	5,3	15,50	8,0
1,50	53,46	6,5	23,88	9,5
1,55	56,27	7,3	29,58	10,9
1,60	58,21	8,8	34,19	11,6
1,65	60,95	9,3	36,79	12,6
1,70	62,84	10,2	38,48	13,2
S1,70	100,00	33,8	100,00	53,0

TABLE C11

Washability data for the +25 μm and -25 μm fractions of Grootegeluk (95 % - 75 μm) coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	46,45	3,0	7,69	3,7
1,40	48,91	4,4	17,17	5,4
1,45	51,00	5,2	24,49	7,1
1,50	55,53	6,5	31,13	9,1
1,55	58,69	7,0	36,68	10,5
1,60	61,22	8,2	40,08	11,6
1,65	62,27	8,8	43,16	12,7
1,70	64,18	9,5	46,69	14,5
S1,70	100,00	33,8	100,00	48,5

TABLE C12

Washability data for the +25 μm and -25 μm fractions of Grootegeluk (95 % -45 μm) coal

Relative Density	+25 μm Fraction Cum. Yield (%)	+25 μm Fraction Cum. Ash (%)	-25 μm Fraction Cum. Yield (%)	-25 μm Fraction Cum. Ash (%)
F1,35	41,04	3,3	12,60	3,0
1,40	47,56	4,0	21,34	5,5
1,45	53,24	5,6	32,11	7,3
1,50	57,03	6,8	36,78	8,7
1,55	59,49	7,2	40,67	9,6
1,60	62,23	8,0	45,39	11,3
1,65	64,15	9,0	47,69	12,8
1,70	66,84	10,1	49,25	13,4
S1,70	100,00	32,4	100,00	45,2

C.2 Gravimetric float and sink analysis :

TABLE C13

Washability data for the +25 μm fraction of Greenside (95 % -150 μm) sample

Relative Density	Fract. Yield (%)	Cum. Yield (%)	Cum. Ash (%)	Fract. Ash (%)
F1,45	35,4	35,4	3,7	3,7
1,45 - 1,50	32,2	67,6	6,3	9,2
1,50 - 1,60	15,7	83,3	8,7	18,9
S1,60	16,7	100,0	16,0	52,5

TABLE C14

Washability data for the +25 μm fraction of Greenside (95 % -150 μm) sample (repeat run)

Relative Density	Fract. Yield (%)	Cum. Yield (%)	Cum. Ash (%)	Fract. Ash (%)
F1,45	37,9	37,9	4,1	4,1
1,45 - 1,50	30,5	68,4	6,5	9,6
1,50 - 1,60	13,1	81,5	9,5	25,2
S1,60	18,5	100,0	16,6	48,0

TABLE C15

Washability data for the +25 μm fraction of Rietspruit (95 % -150 μm) sample

Relative Density	Fract. Yield (%)	Cum. Yield (%)	Cum. Ash (%)	Fract. Ash (%)
F1,45	25,2	25,2	.4,1	4,1
1,45 - 1,50	26,6	51,8	6,8	9,4
1,50 - 1,60	17,5	69,3	10,0	19,4
S1,60	30,7	100,0	54,2	23,6

TABLE C16

Washability data for the +25 μm fraction of Rietspruit (95 % -150 μm) sample (repeat run)

Relative Density	Fract. Yield (%)	Cum. Yield (%)	Cum. Ash (%)	Fract. Ash (%)
F1,45	27,8	27,8	4,2	4,2
1,45 - 1,50	25,3	53,1	6,9	9,9
1,50 - 1,60	14,1	67,2	9,6	19,7
S1,60	32,8	100,0	23,7	52,8

TABLE C17

Washability data for the +25 μm fraction of Grootegeluk (95 % -150 μm) sample

Relative Density	Fract. Yield (%)	Cum. Yield (%)	Cum. Ash (%)	Fract. Ash (%)
F1,45	45,1	45,1	5,4	5,4
1,45 - 1,50	10,3	55,4	8,0	19,5
1,50 - 1,60	3,5	58,9	9,5	33,2
S1,60	41,1	100,0	34,0	69,1

TABLE C18

Washability data for the +25 μm fraction of Grootegeluk (95 % -150 μm) sample (repeat run)

Relative Density	Fract. Yield (%)	Cum. Yield (%)	Cum. Ash (%)	Fract. Ash (%)
F1,45	46,4	46,4	4,9	4,9
1,45 - 1,50	8,0	54,4	6,9	18,8
1,50 - 1,60	4,5	58,9	8,7	30,5
S1,60	41,1	100,0	33,0	67,8

APPENDIX D

AGGLOMERATION DATA

Table D1

Data for the +25 μm fraction of Greenside coal (95 % -45 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (sec)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B5	S'SOL AB	35	100	9,8	68,8	95,7
B6	S'SOL K	35	660	8,8	44,1	90,9
B22	BENZ/NAP	40	150	9,0	36,6	89,4
B24	S'SOL K	25	540	8,3	29,4	82,6
B48	S'S AB/K	35	240	8,7	40,5	90,8

Table D2

Data for the -25 μm fraction of Greenside coal (95 % -45 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B3	S'SOL AB	35	90	8,0	88,0	85,0
B4	S'SOL K	35	1320	6,2	78,8	80,8
B20	BENZ/NAP	35	60	7,2	46,1	65,8
B23	S'SOL K	35	870	4,8	46,7	62,3

Table D3

Data for the +25 μm fraction of Greenside coal (95 % -75 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B9	S'SOL AB	35	100	12,4	67,1	93,5
B10	S'SOL K	35	670	10,1	51,9	85,8
B53	S'S AB/K	35	60	9,1	38,4	74,4
B54	S'SOL K	35	510	7,1	22,6	40,7

Table D4

Data for the -25 μm fraction of Greenside coal (95 % -75 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B7	S'SOL AB	35	90	10,6	87,9	86,4
B8	S'SOL K	35	1380	6,3	56,6	70,2
B55	S'SOL K	35	760	4,7	28,1	29,9

Table D5

Data for the +25 μm fraction of Rietspruit coal (95 % -45 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B62	S'S AB/K	35	90	10,1	35,1	71,6
B63	S'SOL AB	35	180	11,7	53,8	86,4
B65	TETRALIN	35	300	11,9	61,0	88,8

Table D6

Data for the -25 μm fraction of Rietspruit coal (95 % -45 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B43	TETRALIN	35	270	6,5	56,2	59,9
B44	S'SOL AB	35	190	6,5	52,7	55,9
B45	S'S AB/K	35	200	5,2	32,9	23,3

Table D7

Data for the +25 μm fraction of Rietspruit coal (95 % -75 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B76	S'SOL AB	35	90	14,5	57,6	76,6
B77	S'S AB/K	35	90	13,6	56,5	75,7
B78	S'S AB/K	25	90	10,9	37,2	49,6
B79	TETRALIN	35	60	13,0	50,7	70,0

Table D8

Data for the -25 μm fraction of Rietspruit coal (95 % -75 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B71	S'SOL AB	35	180	8,6	48,8	52,0
B72	S'S AB/K	35	540	6,7	35,9	27,1
B73	TETRALIN	35	390	8,9	54,9	58,0
B75	S'SOL AB	25	240	9,8	39,8	38,9

Table D9

Data for the +25 μm fraction of Grootegeluk coal (95 % -45 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B38	S'S AB/K	35	240	6,9	41,8	27,4
B39	S'SOL AB	35	180	8,5	54,9	50,5
B41	S'SOL K	35	1860	14,0	64,7	65,7
B42	TETRALIN	35	300	9,7	66,3	60,1

Table D10

Data for the -25 μm fraction of Grootegeluk coal (95 % -45 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B34	TETRALIN	35	510	6,5	56,1	22,0
B35	S'SOL AB	35	480	6,7	57,0	24,0
B37	S'S AB/K	35	1020	4,2	47,4	5,6

Table D11

Data for the +25 μm fraction of Grootegeluk coal (95 % -75 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B15	S'SOL AB	35	120	15,9	74,9	69,9
B16	S'SOL K	35	1500	24,4	69,0	79,6
B17	S'SOL K	25	2460	23,6	77,2	80,8
B56	S'S AB/K	35	240	7,8	47,1	34,1

Table D12

Data for the -25 μm fraction of Grootegeluk coal (95 % -75 μm)

Run No.	Bridging oil	Oil conc. (%)	T_A (min)	Ash (%) Aggl.	Ash (%) Tails	Yield (%)
B14	S'SOL AB	35	660	8,0	61,9	25,4
B33	TETRALIN	35	510	6,5	54,3	12,0
B57	S'S AB/K	35	510	6,6	50,4	4,4

Note: S'SOL AB = Shellsol AB
S'SOL K = Shellsol K
S'S AB/K = Mixture (50g:50g) of Shellsol AB and Shellsol K
BENZ/NAP = Benzene/Naphthalene mixture

APPENDIX E

DATA FOR LIBERATION EFFICIENCY DETERMINATIONS

TABLE E1

Greenside coal ("as is")

Relative Density	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	25,72	3,7	10,81	4,9	20,93	4,1	0,86
1,40	37,17	4,4	18,13	5,4	31,06	4,7	1,45
1,45	53,94	5,6	33,87	6,5	47,50	5,9	2,79
1,50	67,05	6,9	49,92	8,2	61,55	7,4	4,52
1,55	75,68	8,2	66,91	10,9	72,86	9,1	6,59
1,60	82,06	9,3	78,19	13,7	80,82	10,7	8,66
1,65	85,65	10,1	79,57	14,1	83,70	11,4	8,97
1,70	88,03	10,6	82,81	15,2	86,35	12,1	10,44
S1,70	100,00	17,0	100,00	25,2	100,00	19,6	19,63

TABLE E2

Greenside coal (95 % -150 μm)

Relative Density	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	27,74	3,7	16,18	6,2	23,12	4,7	1,08
1,40	39,84	4,2	24,93	6,4	33,88	5,1	1,73
1,45	56,73	5,4	42,30	7,1	50,96	6,0	3,08
1,50	70,54	6,8	59,37	8,3	66,07	7,4	4,90
1,55	78,42	8,0	72,42	10,1	76,02	8,8	6,72
1,60	82,68	8,8	79,05	11,5	81,23	9,9	8,05
1,65	85,21	9,4	81,61	12,2	83,77	10,5	8,83
1,70	86,89	9,9	82,28	12,4	85,05	10,9	9,30
S1,70	100,00	16,5	100,00	22,9	100,00	19,4	19,36

TABLE E3

Greenside coal (95 % - 75 μm)

Relative Density	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	28,93	3,8	16,28	4,6	21,93	4,3	0,93
1,40	42,98	4,3	28,86	5,3	35,17	4,8	1,70
1,45	61,05	5,4	46,71	6,5	53,12	6,0	3,20
1,50	73,16	6,6	67,77	9,1	70,18	8,0	5,61
1,55	80,55	7,8	76,99	11,0	78,58	9,6	7,50
1,60	83,63	8,4	79,95	11,8	81,59	10,3	8,38
1,65	86,64	9,1	83,12	12,8	84,69	11,1	9,43
1,70	87,93	9,4	85,39	13,7	86,53	11,8	10,18
S1,70	100,00	16,5	100,00	21,5	100,00	19,3	19,27

TABLE E4

Greenside coal (95 % - 45 μm)

Relative Density	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	38,14	2,9	13,22	3,4	17,46	3,3	0,58
1,40	51,97	3,5	27,23	4,0	31,44	3,9	1,24
1,45	67,59	4,6	48,32	5,3	51,60	5,2	2,68
1,50	80,08	6,0	65,39	7,0	67,89	6,8	4,62
1,55	84,92	6,7	75,80	8,5	77,35	8,2	6,33
1,60	88,39	7,5	81,65	9,5	82,80	9,2	7,60
1,65	90,64	8,0	83,51	9,9	84,72	9,6	7,85
1,70	92,94	8,8	85,09	10,6	86,42	10,3	8,88
S1,70	100,00	12,6	100,00	21,0	100,00	19,6	19,57

TABLE E5

Rietspruit coal ("as is")

Relative Denaity	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	16,93	3,8	6,63	5,8	13,80	4,4	0,61
1,40	25,83	4,7	12,74	6,2	21,85	5,2	1,14
1,45	37,18	6,3	25,05	6,0	33,49	6,2	2,08
1,50	47,32	7,7	37,80	7,3	44,43	7,6	3,36
1,55	55,55	9,2	48,25	9,3	53,33	9,2	4,91
1,60	64,31	11,1	54,21	10,7	61,24	10,6	6,71
1,65	68,64	12,1	61,76	13,0	66,55	12,4	8,23
1,70	73,62	13,8	66,78	14,7	71,54	14,1	10,06
S1,70	100,00	24,6	100,00	30,2	100,00	26,3	26,30

TABLE E6

Rietspruit coal (95 % -150 μm)

Relative Density	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	19,22	4,4	6,19	5,2	14,06	4,8	0,67
1,40	28,15	5,0	14,48	5,7	22,74	5,3	1,22
1,45	41,05	6,1	26,85	6,6	35,43	6,3	2,37
1,50	52,41	7,5	38,47	7,8	46,89	7,6	3,81
1,55	61,39	9,0	50,94	9,6	57,25	9,2	5,53
1,60	66,74	10,1	58,64	11,1	63,53	10,5	6,94
1,65	70,80	11,1	66,57	13,0	69,12	11,8	8,61
1,70	75,11	12,2	71,19	14,9	73,56	13,3	10,03
S1,70	100,00	24,4	100,00	29,2	100,00	26,3	26,32

TABLE E7

Rietspruit coal (95 % - 75 μm)

Relative Density	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	22,06	4,6	6,00	5,6	13,15	5,1	0,67
1,40	31,65	5,1	13,99	6,0	21,85	5,6	1,22
1,45	44,58	6,1	28,75	7,0	35,79	6,6	2,37
1,50	55,24	7,3	42,12	8,4	47,96	7,9	3,81
1,55	62,14	8,4	55,01	10,4	58,18	9,5	5,53
1,60	69,50	10,0	60,52	11,4	64,52	10,7	6,94
1,65	72,53	10,7	69,05	13,4	70,60	12,2	8,61
1,70	76,98	12,0	73,11	14,6	74,83	13,4	10,03
S1,70	100,00	24,1	100,00	28,1	100,00	26,3	26,32

TABLE E8

Rietspruit coal (95 % -45 lm)

Relative Density	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	31,71	3,4	6,71	3,3	8,99	3,3	0,30
1,40	41,92	3,9	16,45	3,8	18,77	3,8	0,72
1,45	56,82	4,9	28,61	4,7	31,18	4,7	1,46
1,50	65,94	5,8	43,17	6,0	45,24	6,0	2,72
1,55	75,17	7,2	56,54	7,9	58,24	7,8	4,56
1,60	79,18	7,9	63,78	9,3	65,18	9,1	5,96
1,65	81,69	8,5	71,68	11,2	72,59	10,9	7,93
1,70	84,75	9,6	74,67	12,0	75,59	11,8	8,93
S1,70	100,00	17,5	100,00	27,2	100,00	26,3	26,32

TABLE E9

Grootegeluk coal ("as is")

Relative Density	+25 µm Cum. Yield (%)	+25 µm Cum. Ash (%)	-25 µm Cum. Yield (%)	-25 µm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	36,19	3,0	2,35	3,4	22,25	3,2	0,71
1,40	45,31	4,1	12,06	5,7	31,61	4,7	1,49
1,45	50,57	5,2	18,22	7,3	37,24	6,0	2,23
1,50	55,31	6,5	25,19	8,4	42,90	7,3	3,13
1,55	58,66	7,6	30,66	10,7	47,12	8,9	4,19
1,60	61,40	8,6	32,04	11,3	49,30	9,7	4,79
1,65	63,39	9,4	36,86	12,2	52,45	10,6	5,55
1,70	65,91	10,5	38,94	13,1	54,79	11,6	6,36
S1,70	100,00	33,6	100,00	53,9	100,00	41,9	41,92

TABLE E10

Grootegeluk coal (95 % -150 µm)

Relative Density	+25 µm Cum. Yield (%)	+25 µm Cum. Ash (%)	-25 µm Cum. Yield (%)	-25 µm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	36,43	3,0	3,49	4,7	22,69	3,7	0,84
1,40	44,63	4,3	10,24	5,7	30,28	4,9	1,47
1,45	48,94	5,3	15,50	7,0	35,00	6,0	2,10
1,50	53,37	6,6	23,88	8,7	41,07	7,5	3,08
1,55	56,18	7,6	29,58	10,4	45,08	8,8	3,95
1,60	58,12	8,3	34,19	12,0	48,14	9,8	4,74
1,65	60,86	9,4	36,79	13,0	50,82	10,9	5,53
1,70	62,75	10,2	38,48	13,6	52,62	11,6	6,12
S1,70	100,00	33,8	100,00	53,0	100,00	41,8	41,80

TABLE E11

Grootegeluk coal (95 % -.75 μm)

Relative Density	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	46,45	3,5	7,69	3,8	24,67	3,7	0,91
1,40	48,91	4,1	17,17	5,3	31,07	4,8	1,48
1,45	51,00	4,7	24,49	6,9	36,10	5,9	2,14
1,50	55,53	6,1	31,13	8,2	41,82	7,3	3,05
1,55	58,69	7,3	36,68	9,5	46,32	8,5	3,96
1,60	61,22	8,3	40,08	10,7	49,34	9,7	4,78
1,65	62,27	8,8	43,16	12,9	51,53	11,1	5,72
1,70	64,18	9,6	46,69	14,4	54,35	15,8	8,58
S1,70	100,00	33,9	100,00	48,5	100,00	42,1	42,12

TABLE E12

Grootegeluk coal (95 % - 45 μm)

Relative Density	+25 μm Cum. Yield (%)	+25 μm Cum. Ash (%)	-25 μm Cum. Yield (%)	-25 μm Cum. Ash (%)	Composite Cum. Yield (%)	Composite Cum. Ash (%)	Ash/10 units of fee
F1,35	41,04	3,3	12,60	3,4	19,84	3,4	0,68
1,40	47,56	4,2	21,34	4,8	28,02	4,6	1,29
1,45	53,24	5,4	32,11	7,3	37,49	6,8	2,54
1,50	57,03	6,5	36,78	8,6	41,94	8,1	3,39
1,55	59,49	7,3	40,67	9,9	45,46	9,3	4,21
1,60	62,23	8,3	45,39	11,7	49,68	10,8	5,36
1,65	64,15	9,0	47,69	12,6	51,88	11,7	6,05
1,70	66,84	10,2	49,25	13,2	53,73	12,4	6,68
S1,70	100,00	32,4	100,00	45,2	100,00	42,0	41,98

TABLE E13

GREENSIDE t/u RECONSTITUTED SAMPLES
POLYNOMIAL CONSTANTS FROM M-PLOTS AND LIBERATION EFFICIENCIES

SAMPLE	A(6)	A(5)	A(4)	A(3)	A(2)	A(1)	A(0)
GS(AS IS)	0.00E+00	1.89E-08	-4.01E-06	3.14E-04	-9.58E-03	1.41E-01	-0.005018
GS(-150)	0.00E+00	1.64E-08	-3.35E-06	2.55E-04	-7.79E-03	1.29E-01	-0.001622
GS(-75)	0.00E+00	1.31E-08	-2.61E-06	1.97E-04	-5.80E-03	1.01E-01	-0.00151
GS(-45)	5.08E-10	-1.31E-07	1.29E-05	-5.89E-04	1.28E-02	-6.74E-02	0.0014492

SAMPLE	FA(ACT)	FA(CALC)	TOT.AREA	INTEGRAL	LOWER	UPPER	MID	LIB.EFF.
GS(AS IS)	19.79	19.77	988.32	478.33	195.46	510.00	282.87	64.32
GS(-150)	19.36	19.35	967.66	461.92	187.31	505.74	274.62	64.81
GS(-75)	19.27	19.26	963.13	460.51	185.55	502.62	274.96	64.64
GS(-45)	19.32	19.09	954.44	401.73	182.16	552.71	219.57	71.57

TABLE E14

RIETSPRUIT t/u RECONSTITUTED SAMPLES
POLYNOMIAL CONSTANTS FROM M-PLOTS AND LIBERATION EFFICIENCIES

SAMPLE	A(6)	A(5)	A(4)	A(3)	A(2)	A(1)	A(0)
RS(AS IS)	0.00E+00	-2.34E-09	6.85E-07	-3.73E-05	1.60E-03	2.75E-02	0.0034786
RS(-150)	0.00E+00	1.43E-09	4.04E-08	-3.17E-06	7.42E-04	3.69E-02	0.0026261
RS(-75)	0.00E+00	4.72E-09	-6.56E-07	4.63E-05	-7.04E-04	5.44E-02	-0.000772
RS(-45)	0.00E+00	7.36E-09	-1.11E-06	7.18E-05	-1.24E-03	4.23E-02	-0.005338

SAMPLE	FA(ACT)	FA(CALC)	TOT.AREA	INTEGRAL	LOWER	UPPER	MID	LIB.EFF.
GS(AS IS)	41.92	26.48	1324.29	716.51	350.66	607.78	365.85	62.42
GS(-150)	42.56	26.30	1315.24	672.12	345.90	643.12	326.22	66.35
GS(-75)	42.12	26.32	1316.08	669.78	346.44	646.30	323.34	66.65
GS(-45)	41.98	25.95	1297.08	593.43	336.62	703.65	256.81	73.26

TABLE E15

GROOTEGELUK t/u RECONSITUTED SAMPLES

POLYNOMIAL CONSTANTS FROM M-PLOTS AND LIBERATION EFFICIENCIES

SAMPLE	A(6)	A(5)	A(4)	A(3)	A(2)	A(1)	A(0)
GG(AS IS)	0.00E+00	8.66E-09	-1.56E-06	1.35E-04	-2.81E-03	4.25E-02	0.0011401
GG(-150)	0.00E+00	2.98E-09	-6.96E-07	1.07E-04	-3.01E-03	5.79E-02	0.0008512
GG(-75)	0.00E+00	0.00E+00	-4.85E-08	5.97E-05	-1.72E-03	4.47E-02	0.0043282
GG(-45)	0.00E+00	8.80E-10	-1.88E-07	6.01E-05	-1.16E-03	3.49E-02	0.0009717

SAMPLE	FA(ACT)	FA(CALC)	TOT.AREA	INTEGRAL	LOWER	UPPER	MID	LIB.EFF.
GS(AS IS)	41.92	41.92	2096.21	977.20	878.78	1119.02	98.42	91.92
GS(-150)	42.56	42.56	2127.90	1057.30	905.56	1070.60	151.74	87.59
GS(-75)	42.12	42.12	2106.23	1045.97	887.06	1060.26	158.91	86.97
GS(-45)	41.98	41.98	2099.16	1061.05	881.26	1038.11	179.80	85.24

APPENDIX F

SAMPLE CALCULATIONS

CALCULATION OF TABLE 5.5 VALUES

In Table 5.5 the values for the +25 μm size fraction in the 1,35 to 1,60 relative density range were obtained as follows:

(i) 'Total Mass' is the sum of the proportions (see Table 5.3) in the intervals within the specified relative density range - i.e. 7,77 + 11,39 + 8,90 + 5,86 + 4,33 = 38,25.

(ii) The 'Ash Content' is the fractional ash content the material within a specified relative density range and was calculated from the following formula :

$\Sigma(Y_F * A_F)/Y_C = A_C$

where Y_F = Fractional yield = 56,34 % (82,06 - 25,72)

Y_C = Cumulative yield = 82,06 %

A_C = Cumulative ash = 9,3 %

A_F = Fractional ash = calculated at 11,80 %

(iii) The 'Coal Mass' = $M_T - (M_T * A_F/100)$

where M_T = Total Mass (gram)

In this case 'Coal Mass' = 38,25 - (38,25 * 11,8/100)

= 33,74 gram

(iv) The 'Maceral Content' is calculated by difference :

Total Vitrinite content (in 100 gram)	= 28,27 gram
Vitrinite in <1,35 R.D. interval	= 18,48 gram
Vitrinite in 1,35 - 1,60 R.D. interval	= 9,79 gram

Total coal mass in this interval	= 52,10 gram
Inertinite in 1,35 - 1,60 R.D. interval	= 42,31 gram
The total maceral content therefore	= 52,10 gram