REVERSE FLOTATION: A NOVEL PROCESS FOR THE BENEFICIATION OF FINE COAL

University of Cape Town

By:

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A thesis submitted to the University of Cape Town in fulfullment of the requirements for the degree of Doctor of Philosophy.

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August, 1991

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ACKNOWLEDGEMENTS

I should like to express my sincere thanks to my supervisor, Prof. J–P Franzidis for his enthusiasm, assistance and guidance in this study.

The help of the following people is also gratefully acknowledged:

The workshop staff in the Chemical Engineering Department of the University of Cape Town and Mr Granville de la Cruz for construction, maintenance and repair of flotation equipment

Mrs Helen Divey and Mrs Lorna Wall for assistance with float/sinks analyses

Mr E.W. Randall for help and advice with various computer-related problems with the production of this thesis

Mr Martin Harris, Mr Sean von Holt and other colleagues in the Chemical Engineering Department of the University of Cape Town for their interest and advice

Mr Stefan Segal and Mr Alan Saul for the use of their computers

Ms Katy van Rooyen for expert help with the typesetting of this thesis

Mrs Diana van Rooyen for pertinent suggestions on presentation and style

Mr Peter Mills for his friendly support and useful suggestions regarding the column flotation testwork

My parents for their encouragement, moral support and financial assistance throughout the last four years.

The support of the following organizations is gratefully acknowledged:

The Foundation for Research Development (FRD) for the award of my postgraduate bursary

the National Energy Council for funding the project.

CERTIFICATION BY SUPERVISOR

During the course of this study, the following papers have been published:

Journals:

Stonestreet, P. and Franzidis, J–P. "Reverse flotation of coal—a novel way for the beneficiation of high ash coal fines". *Minerals Engineering*. Vol. 1, No. 4, pp 343–349. 1988.

Stonestreet, P. and Franzidis, J–P. "Development of the reverse flotation process: Depression of coal in the concentrates". *Minerals Engineering*. Vol. 2, No. 3, pp 393–402. 1989.

Conference proceedings:

Stonestreet, P. and Franzidis, J–P. "Development of the reverse flotation process for the beneficiation of coal fines in column cells". SAIMM International colloquium: "Developments in froth flotation". Gordons Bay, South Africa. 3-4 August, 1989.

Stonestreet, P. and Franzidis, J–P. "Reverse flotation—Exciting new process or academic curiosity?" SAIChE 6th National Meeting. Durban, South Africa. 7-9 August, 1991.

In terms of Paragraph GP 8 of "General Rules for the degree of Ph.D.", I, Assoc. Prof. J–P Franzidis, as supervisor of the candidate, P. Stonestreet, certify that I approve of the incorporation in this thesis of material that has been published.

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NOMENCLATURE

θ	*Contact angle*
γ	*Surface tension (J/m^2 or N/m)*
A	*interfacial area (m^2)*
G	*Gibbs free energy (J)*
C_a	*Carrying capacity (t/h/m^2)*
d_b	*bubble diameter (mm)*
d_p	*particle diameter (mm)*
d_{50}	*50% sample passing given particle diameter*
d_{80}	*80% sample passing given particle diameter*
ε_g	*fraction of gas holdup (collection zone)*
E_k	*collection zone model efficiency term*
E	*Efficiency index*
ε_{g0}	*gas holdup at froth overflow (fraction)*
e_M	*degree of entrainment*
F_M	*true recovery by flotation*
J_B	*Superficial bias rate (cm/s)*
J_g	*Superficial gas rate (cm/s)*
$J_g{}^*$	*Superficial gas rate at standard condititions (cm/s)*
$J_{g(max)}$	*value of J_g at onset of turbulence (cm/s)*
J_W	*Superficial wash water rate (cm/s)*
k_c	*collection zone model rate constant*
K_1	*fraction of monolayer bubble loading*
R_M	*Overall flotation recovery*
Q_t	*Tails volumetric rate (l/min)*
τ	*average residence time (min)*
τ_p	*particle residence time (min)*
W	*water recovery*
W_A	*Work of adhesion (J/m^2)*

LIST OF ABBREVIATIONS

r.o.m.	*run-of-mine*
LAC	*low ash coal*
% quartz/coal	*percentage quartz by mass in artificial mixture; remainder is coal*
PZC	*point of zero charge*
DTAB	*Dodecyl-trimethyl-ammonium bromide*
HPYC	*Hexadecyl-trimethyl-pyridinium chloride*
HTAB	*Hexadecyl-trimethyl-ammonium bromide*
MIBC	*Methyl isobutyl Carbinol*
DIBK	*Diisobutyl Ketone*

SUMMARY

The beneficiation of ultrafine coal by froth flotation is a widely used process, and its importance for the treatment of –0.1 mm coal has been appreciated of late in South African coal washing plants. A number of problems adversely affect the efficiency of this process, such as poor liberation, poor floatability, gangue entraiment/entrapment, and high mass recovery which can lead to throughput limitations in coal flotation equipment.

Reverse coal flotation is a novel process which has been proposed to address three problems occurring in the conventional, "forward flotation" of coal, namely: high mass recovery, gangue entrainment and the variability of floatability of South African coals. In this process, the entire gangue fraction in the coal would be floated in a single step process, with the clean coal product reporting as the tailings. To the author's knowledge, no such process has been investigated previously for coal flotation.

In this thesis, a reverse flotation process for ultrafine coal beneficiation was developed from first principles. In order to obtain a clear understanding and interpretation of experimental flotation results, artificial feed mixtures consisting of blends of "pure" gangue and "pure" coal were used for a large section of the experimental testwork. Having established the technical viability of the process on these artificial mixtures, the flotation work was extended to four South African run-of-mine coals.

The study comprised the selection of suitable reagents for reverse flotation and the evaluation of these reagents experimentally. Experimental testwork comprised batch adsorption tests, and flotation tests in a conventional, sub-aeration laboratory batch cell and in a laboratory column flotation cell. The process was evaluated in terms of coal recovery into the product and gangue removal in the waste stream, and its metallurgical performance was compared to conventional flotation using both the artificial feeds and r.o.m. coals.

The structures of coal and coal mineral matter were considered in order to select suitable reagents for reverse flotation. It was found that a cationic-type surfactant collector would be required to float the naturally hydrophilic coal mineral matter; a depressant would be required to suppress the natural floatability of coal; and a frother would be required to ensure froth phase stability. A reagent suite for reverse flotation was proposed, namely the use of quaternary amine salts to act as ash collector, coal depressant and frother. Three amines were investigated. These were dodecyl-trimethyl ammonium bromide (DTAB), hexadecyl-trimethyl ammonium bromide (HTAB), and hexadecyl-pyridinium chloride (HPYC). Dextrin was proposed as an additional coal depressant and DIBK or MIBC as additional frothers, should they be required.

In order to make a preliminary assessment of the suitability of the reagent suite, the adsorption behaviour of the amines onto low ash washed coal ("pure" coal) and quartz ("pure" gangue) surfaces was investigated. The fact that adsorption occurred on both coal and gangue was encouraging for the amines' proposed use as collector and

depressant. It was inferred from amine adsorption dependence on pH that reverse flotation tests should be conducted between pH=3 and pH=4 in order to eliminate residual collector action on coal. The use of dextrin as a coal depressant was examined and it was found that prior conditioning of coal with dextrin was necessary to ensure that the maximum adsorption occurred. Once the dextrin adsorbed, the amine was not able to dislodge the dextrin attachment to coal.

The adsorption tests indicated that the selected reagent suite did adsorb onto coal and quartz and thus might be suitable for the reverse flotation process. It still needed to be determined whether this adsorption was sufficient to result in flotation of quartz and depression of the coal. This could only be determined by flotation experiments. The experimental testwork was initially restricted to ideal, artificial mixtures consisting of blends of low ash coal and quartz or kaolin in various proportions. The reason for this was to obtain results that were as unambiguous as possible, without being clouded by the additional factor of feed liberation. Experimental work was carried out in a small laboratory sub-aeration flotation cell and in a laboratory scale column cell.

Flotation tests were first performed in a conventional, laboratory size Leeds-type flotation cell, using artificial quartz/coal feeds. The purpose of these tests was to evaluate the selected reagent suite and to identify the optimum flotation conditions and procedures. The quaternary amines were found to be convincing ash collectors and frothers. With kaolin/coal mixtures, gangue selectivity was found to be good in spite the kaolin having an extremely small average particle size, which is unsuitable for flotation. This "worst case" suggested that reverse flotation of kaolin (the dominant coal mineral) in "real", r.o.m. coals should be successful.

Initially, coal depression by the amines was found to be poor. Typically, 25% of the feed coal was lost to the concentrate discard. Investigation showed that entrainment/entrapment, rather than poor depression was largely responsible for product coal loss in the flotation concentrates. A staged reagent addition method was found to reduce the coal loss due to entrainment. The reagent HTAB was found to give the best overall results, using a three stage addition process. This reagent was used in all subsequent testwork. The use of dextrin as an additional coal depressant was found to reduce coal loss even further. This resulted in additional coal recovery in the tails. It was also shown that the feed should be conditioned with dextrin prior to collector addition in order to obtain the maximum coal depression.

Reverse flotation in a laboratory scale column of the same artificial coal/quartz mixtures as in the batch cell was found to be highly successful. Better cleaning was obtained than in the laboratory batch cell. This was due to the reduction of (coal) entrainment by wash water in the froth zone. The metallurgical performance was of reverse flotation in the column cell was compared with forward flotation (on the same artificial mixtures). Although the cleaning obtained in reverse flotation was slightly inferior (it was found that some coal entrainment occurred, which resulted in a loss of coal recovery from the tailings product), a throughput of up to three times greater than that which could be attained in forward flotation, was achieved. This is a major advantage of the process in a column flotation cell.

Up to this point in the study, all the test work had been conducted on ideally liberated feeds containing pure coal and pure gangue. The process was now evaluated for a real-life situation, i.e. reverse flotation of run-of-mine coals. Reverse flotation was performed on four different South African r.o.m coals, from the Rietspruit, Grootegeluk, Ermelo, and Durnacol Collieries, respectively. Tests were performed first in the laboratory batch cell, but did not result in an acceptable degree of beneficiation for any of the coals tested.

The column cell resulted in better cleaning than was obtained in the batch cell. Of the four coals tested, the best separation was achieved with Durnacol coal, which had the best liberation characteristics. The product achieved with this coal represented a substantial reduction of the feed ash content but was not, however, of marketable grade. In contrast, forward flotation of the same coal did yield a product of marketable grade.

The inferior metallurgical performance obtained with reverse flotation of the r.o.m. coals was attributed to the entrainment of clean coal in the discard due to a high froth water recovery, poor floatability of ultrafine ash particles, and to the poor liberation characteristics of the coals.

Although it was not possible to obtain a product of marketable grade with the South African r.o.m. coals tested in this study, the results were encouraging: a reagent suite had been selected for reverse flotation which was shown to be successful for the "perfectly" liberated artificial feeds. It was shown that throughput could be greatly improved in column cells. Thus, one of the proposed advantages of the process is valid. It would be a pity if the process were rejected because the feeds employed in this study were unsuitable for the process. It is recommended that the process be investigated on Northern Hemisphere coals, which generally have much better liberation characteristics and are more floatable than South African coals. Another recommendation is to investigate alternative frothers as a means of reducing high froth water recoveries which could, in turn, reduce entrainment of clean coal in the discard, and thus improve the overall separation of the process.

CHAPTER 1
INTRODUCTION

1. Importance of coal beneficiation

Most of the coal produced throughout the world is used in the country in which it is mined and prepared. However, about 10% (Osborne, 1988) of the total world coal production is traded internationally. The main coal exporting countries are the U.S.A., Australia, South Africa, Poland, U.S.S.R., Canada and Germany, whereas the main importers of coal are Japan, Italy and France. Some coal exporters may still import coal with particular qualities when their own supplies are insufficient.

A large proportion of this export trade consists of coal for metallurgical coke production, but recently there has also been a growing trade in thermal (steam) coal. Coal used for either of these purposes has to meet certain quality specifications. For a specific coal type, there is usually an upper limit set on the ash and sulphur levels and a minimum limit on the calorific value. Frequently the coal producers have to beneficiate the run-of-mine coal in order to comply with the client's specification. In a coal preparation process, beneficiation is the improvement of the quality of a coal by the reduction of its ash and sulphur contents and the increase in its calorific value. This is achieved in some "cleaning" or "coal washing" stage of that process.

Currently, about one third of the worldwide annual coal production is beneficiated (Osborne, 1988). Ever more stringent quality constraints arising from environmental concerns (in the U.S.A. and Europe, in particular) about, for example, the coal sulphur content, have already resulted in a flurry of activity to develop "deep cleaning" technologies and will lead to an increase in the fraction of production that needs to be beneficiated.

1.1 Importance of coal beneficiation in South Africa

In South Africa, the need for coal beneficiation is particularly strong for the simple reason that South African coal is of poor quality. If the size, nature and utilization of South Africa's coal reserves are examined, then the importance of beneficiation, both present and future, can be appreciated.

The total coal reserves of South Africa have been estimated to be 81 000 Mt (Petrick et al., 1975). This value refers to coal seams having economically mineable thickness and lying at a maximum depth of 300 m. Most of the coal is bituminous but a small amount of anthracitic coal (2%) and metallurgical quality coal (1.4%) is also present (Ward, 1984). Of these reserves, about 55 000 Mt is considered extractable by either open cast mining or underground mining methods (DMEA, 1989). This represents about 10% of the world's total hard (anthracite and bituminous) coal reserves and means that South Africa is ranked fifth in terms of the world's hard coal reserves, and seventh in terms of total world coal reserves (BP Statistical Review, 1990).

South Africa's coal reserves consist mainly of low grade (high ash) deposits. Figure 1.1 illustrates the distribution, showing the coal deposits in terms of their in-situ ash content. As can be seen, only 2% of the total reserves are in the 5 to 15% ash range, while at the other extreme, 46% of the coal has greater than 30% ash. On an overall basis, some 88% of the reserves have an ash content of greater than 20%.

If high grade coal were not required in South Africa, then there would be no need for beneficiation. However, a substantial amount of high grade coal is required for both domestic and export purposes. Figure 1.2 shows the breakdown of coal consumption in 1988 by various sectors (DMEA, 1989). The biggest customer was ESCOM (the national Electricity Supply Commission), which consumed 41% of the total amount of coal produced. Since power station furnaces are designed to use low-grade coal, the coal used by this sector is generally not beneficiated. The SASOL oil-from-coal plants, which make up a large part of the local industry consumption of 27%, also do not beneficiate their coal prior to gasification. For the other customers, however, the situation is different.

The export market, for example, was responsible for 23% of coal consumption. Some washing plants in the Witbank area produce two products for export, comprising power station smalls or PSS (thermal coal of 28 MJ/kg or ca. 15% ash) and blend-coking coal with an ash content of 7%, known in South Africa as low ash coal, or LAC. Other plants produce thermal coal or metallurgical coal only. Together with local requirements, the total consumption of high quality coal (15% ash or less) was nearly 30% of total coal production and this is clearly disproportionate to the amount of sub-15% ash coal occurring in-situ. Thus, beneficiation of the raw coal is required and is being carried out to generate saleable products of the desired quality.

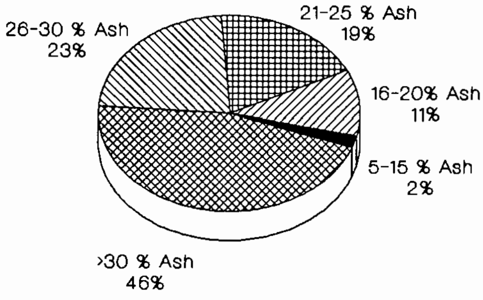

Figure 1.1: In-situ ash distribution of South African coals (after Ward, 1984).

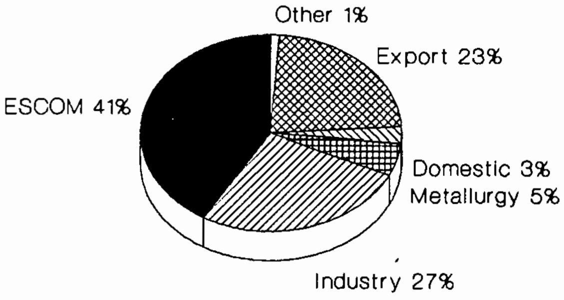

Figure 1.2: South African coal utilization by sector: 1988 (DMEA, 1989).

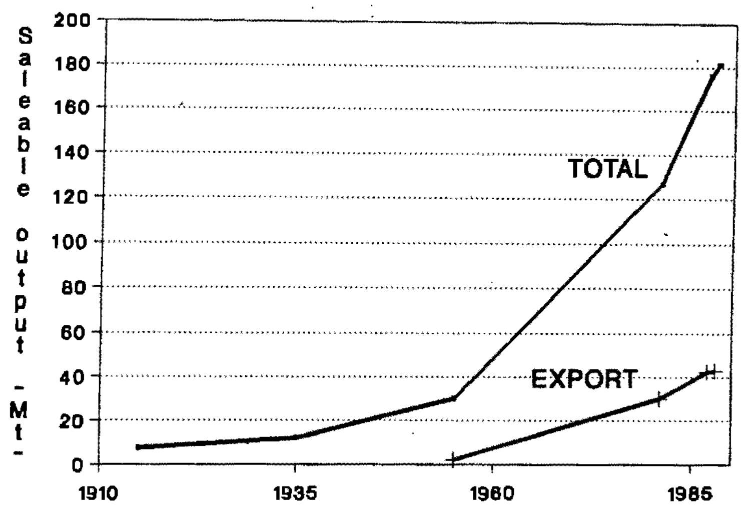

Figure 1.3: Growth of yearly South African coal exports since 1915 (after Ward, 1984; DMEA, 1988).

Furthermore, there is a continuous increase in the demand for coal in South Africa. The current demand is approximately 180 Mt and coal consumption is growing at about 3% per annum (see Figure 1.3). This growth in demand, coupled with the fact that the better quality reserves are being exploited (and are thus being depleted), will make the importance of beneficiation even greater in the future.

2. Coal beneficiation in South Africa—methods and problems

South African coals, like other Southern Hemisphere (Gondwana) coals, are more difficult to beneficiate than those of the Northern Hemisphere. One reason is that they generally have much higher ash levels. Also, they differ in the manner in which mineral matter is distributed in the coal structure: South African coals contain much higher proportion of syngenetic mineral matter (Sanders and Brookes, 1986) and this means that the minerals are finely intergrown with the organic coal structure and are thus less easily liberated by crushing and milling. This makes beneficiation difficult, because the mineral and organic matter are still physically associated with each other, even at very fine particle sizes.

2.1 Beneficiation methods employed

Beneficiation methods depend on the particle size of the coal. At South African coal preparation plants, coarse coal (+6.3 mm) is generally cleaned in jig washers or dense medium baths, which rely on differences in relative density between coal and its associated mineral matter or **gangue**. Small coal (–6.3 +0.5 mm) is treated in dense medium cyclones. Dense medium washers use a suspension of very fine magnetite in water to achieve a medium of the required density for separation. Fine coal (–0.5 mm) causes problems with magnetite recovery and is usually screened out of the small coal dense medium cyclone feed. In some plants it is cleaned in spiral concentrators or by froth flotation but on many others it is either discarded or dewatered and added unbeneficiated to a beneficiated product.

For ultrafine coal (–0.1 mm) separation processes based on differences in relative density cannot be used (because of the low particle settling velocities) and processes based on differences in particle surface properties are employed instead. Froth flotation and oil agglomeration both rely on the fact that the organic coal surface is hydrophobic (water repellent) while the gangue surface is hydrophilic (water-loving). However, there are only six coal flotation plants in South Africa and no oil agglomeration plants. All other washing plants discard the ultrafines.

2.2 Importance of fines and ultrafines beneficiation

In South African collieries, the fines may constitute 15% or more of the total run-of-mine production. During the 1980s the continued discarding of this material became a matter of some concern. In 1985, the Department of Mineral and Energy Affairs undertook a survey of colliery discards (DMEA, 1987): of a total raw coal production of 231.3 Mt, some 48.5 Mt (21%) were discarded, of which approximately 10% was –0.5 mm slurry with an average calorific value of over 23 MJ/kg. Discarding this material is a waste of energy, while stockpiling it has practical limitations and adverse environmental effects, such as spontaneous combustion of coal dumps.

Horsfall (1988) has shown that there is considerable economic advantage in beneficiating the fines, in order more effectively to utilize the coal resource. For the typically high inherent ash types of coal that occur in the Witbank region, he recommended the use of spirals to produce steam coal of about 14% ash content from the –0.5 +0.1 mm fines. Since 1984 spiral plants have, in fact, been installed in at least 17 collieries in South Africa to beneficiate the –0.5 +0.1 mm fines, treating over 5 Mtpa.

The study (Horsfall, 1988) also showed that beneficiating the –0.1 mm ultrafines to produce a high grade (low ash) product would result in an improvement in overall plant yields on account of the additional recovery of coal from the +0.5 mm sections of the plants. At five of the six collieries in South Africa *with flotation plants*, spirals have been installed to treat the –0.5 +0.1 mm fines, leaving only the ultrafines to go to flotation. This has resulted in improved washing efficiencies for both size fractions and a greater overall yield of coal (Voges, 1991). At the remaining 12 collieries having spiral plants, the –0.1 mm ultrafines are still being discarded. There is thus still considerable scope for the application of flotation to the beneficiation of ultrafine coal in South Africa.

2.3 The problem of ultrafines beneficiation

Research into the beneficiation of ultrafine coal by froth flotation has been carried out in the Department of Chemical Engineering, University of Cape Town, since 1983. In this research, three major problem areas have been identified, namely poor liberation, poor floatability and gangue entrainment and entrapment.

In an investigation into the flotation of Australian coals, Bustamante and Warren (1983) found that there is a direct link between a fall in flotation recovery and an increase in the proportion of hydrophilic mineral matter in individual coal grains (particles). This proportion of mineral matter in individual coal grains is dependent on the degree of liberation. South African coal, like Australian coal, contains much finely intergrown mineral matter; it is usually necessary to grind the coal to very fine sizes before appreciable liberation occurs. In a study by Fickling (1986) aimed at identifying the most important physical and chemical factors affecting the flotation of South African coals, the most notable feature of the results was that any change in the rate of coal flotation was generally accompanied by a corresponding change in the rate of ash flotation. This was attributed to insufficient liberation of the coal at the particle sizes considered; the smallest of which was 95% –0.3 mm. In a subsequent liberation study by Harris (1987) on a typical Witbank No.2 seam coal, it was shown that a 10% increase in theoretical yield of 7.4% ash product could be obtained by grinding the coal further from 30% –0.15 mm to 90% –0.15 mm. Thus, in South Africa, coal flotation must be directed at the ultrafines and cannot be used to beneficiate the –0.5 mm fines as in the Northern Hemisphere, as little or no yield of useful product would be obtained.

In addition to the problem of liberation, many South African coals, particularly the commercially important Witbank coals, may be termed "poorly floating" because the cleaning obtainable with flotation is much poorer than would be expected from the washability data (which indicates liberation based on gravimetric float and sink analysis). This is because the floatability of a coal is determined not only by the percentage inorganic impurities it contains (which relates to washability, or separability by density) but also by factors affecting its surface properties, such as hydrocarbon structure (related to rank and maceral type) and oxygen functional group content or degree of oxidation (Laskowski, 1986; Aplan, 1988). South African coals are less floatable than Northern Hemisphere coals because they are generally lower in rank and, as a rule, are deficient in macerals of the vitrinite and exinite groups, which are the most naturally floatable (Horsfall and Franzidis, 1988).[1] The discrepancy between the washability and the floatability of a typical South African Witbank coal is illustrated in Figure 1.4. The flotation release curve[2] lies well above the washability curve indicating that below a certain ash content—in this case about 15%—the theoretical yield at a particular ash is considerably poorer for flotation than would be predicted by the float/sinks curve. For example, at 8% ash, the theoretical yield by flotation is 22%, whereas by relative density separation it is 47%. Also, it is not possible to obtain a grade better than 6% ash by flotation.

A third problem encountered in the flotation of South African coals is that of gangue carry-over into the concentrates by entrainment and entrapment. This is common to all

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[1] These twin disadvantages are less apparent in the case of certain coals from the Natal Province. Natal coals more closely resemble Northern Hemisphere coals than coals from the Witbank coalfield, and are therefore more amenable to froth flotation. Three of the six coal flotation plants in South Africa are on collieries in the Natal Province.

[2] A flotation release curve gives the "ideal floatability" of a particular coal. It can be obtained by the Dell method (Dell, 1961), or by differential flotation.

flotation processes, but the problem is particularly pronounced in coal flotation because of the high concentrate mass recovery: 70 to 80% of the feed is typically recovered in the froth product, compared with 2% or less in the flotation of most mineral ores such as pyrite, chalcopyrite (copper) or cassiterite (tin). The effect of entrainment is also illustrated in Figure 1.4, which shows the results of typical laboratory batch flotation experiments carried out on the same Witbank coal. The data points lie above the flotation release curve; the difference between the data points and the release curve is the result of entrainment and entrapment.

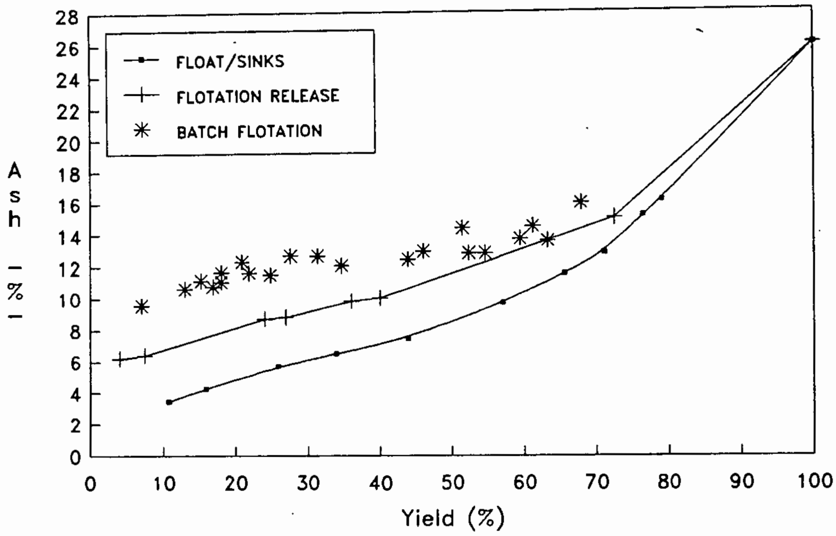


Figure 1.4: Washability, floatability and laboratory batch cell flotation data for a typical Witbank coal (Harris et al., 1986).

A solution to the entrainment problem in coal flotation may be found in the new column flotation technology which, in recent years, has become popular for beneficiating mineral ores (Yianatos, 1989). A column cell differs from conventional sub-aeration cells. It consists of a tall column (8 to 12 m high) of square or circular cross section (up to 6 m in diameter) and has a deep drained froth (0.5 to 3 m) in which entrainment of hydrophilic material is drastically reduced by the addition of wash water near the top of the froth zone. This is designed to give a net downward flow of water in the column, called positive bias, which washes out hydrophilic particles that may be entrained in the froth.

In the last three or four years, column cells have been investigated for coal beneficiation by a number of researchers in the U.S.A. (Parekh et al., 1988), India (Reddy et al., 1988) and Australia (Nicol et al., 1988) on a laboratory or pilot scale level. Generally, better cleaning was obtained than in conventional cells but throughput (capacity) was low when compared to mineral ore flotation. To date, the only industrial plant in operation is installed at the Powell Mountain Coal Co. in Lee County, VA. (Groppo and Parekh, 1990).

Although entrainment is greatly reduced in column flotation cells, the high mass recovery associated with coal flotation still exists and manifests itself as a different problem. In a flotation column there is a limit to the mass of solids that the froth can support. This is called the column carrying capacity and it is expressed in $t/h/m^2$. Carrying capacity is dependent on a number of factors, one of them being particle density (Finch and Dobby, 1990). Because of the low density of coal particles (R.D.=1.2–1.4) the carrying capacity of a coal column is low. Coupled with the high mass recovery inherent in coal flotation, this places a severe limit on the throughput of coal flotation columns.

A possible way of solving both the entrainment problem and the mass recovery problem in coal flotation is by **reverse flotation**. In reverse flotation, the gangue material in an ore is floated, while the valuable (desired) material reports to the tailings. If the gangue material represents the smaller proportion of the feed, then the mass recovery is greatly reduced. An example of reverse flotation carried out industrially is the flotation of heavy minerals from sand. Another example is a process which has been investigated in the U.S. for pyrite removal from coal. This is a two-stage process employing a coal depressant and pyrite collector to remove pyrite from a coal before it is floated in the conventional manner (Miller et al., 1984).

What is proposed here, however, and termed reverse flotation of coal, is that the *entire* gangue fraction be floated in a single step process, with clean coal product reporting as the tailings. To the author's knowledge, no such process has been investigated for coal flotation.

Some preliminary work on reverse flotation of coal has already been done in the Department of Chemical Engineering at the University of Cape Town as a fourth year undergraduate project (Deetleefs and Johnson, 1986). The results were not encouraging. However, a closer examination of the project report would suggest that many aspects were not considered and that the process merits further in-depth investigation.

3. Advantages of reverse flotation of coal

Conventional ("forward") flotation and reverse flotation of coal are compared in Figure 1.5 on the basis of idealized behaviour. In conventional coal flotation, the coal particles are hydrophobic while the gangue (ash) particles are hydrophilic. The relatively small gangue particles and some middlings particles are entrained (or entrapped) along with the collected coal particles in the concentrate.

In reverse flotation, the gangue particles are made hydrophobic and the coal particles are made hydrophilic (depressed). The gangue particles and some middlings particles are collected and thus removed in the concentrate, while some coal particles might be entrained along with them. There are a number of advantages to this mode of operation, and these are discussed below.

3.1 Reduction of mass recovery

In reverse flotation, the gangue material is recovered in the concentrate, while the coal is recovered in the tails. Since the gangue material makes up a smaller proportion of the feed (15 to 30%) than does coal, the concentrate mass recovery is much less than in "forward" coal flotation. An advantage of reduced mass recovery is the fact that higher feed pulp densities could be used in reverse flotation of coal. In conventional, forward coal flotation, low feed pulp densities (5%) are necessary to minimize entrainment (Picard, 1985).

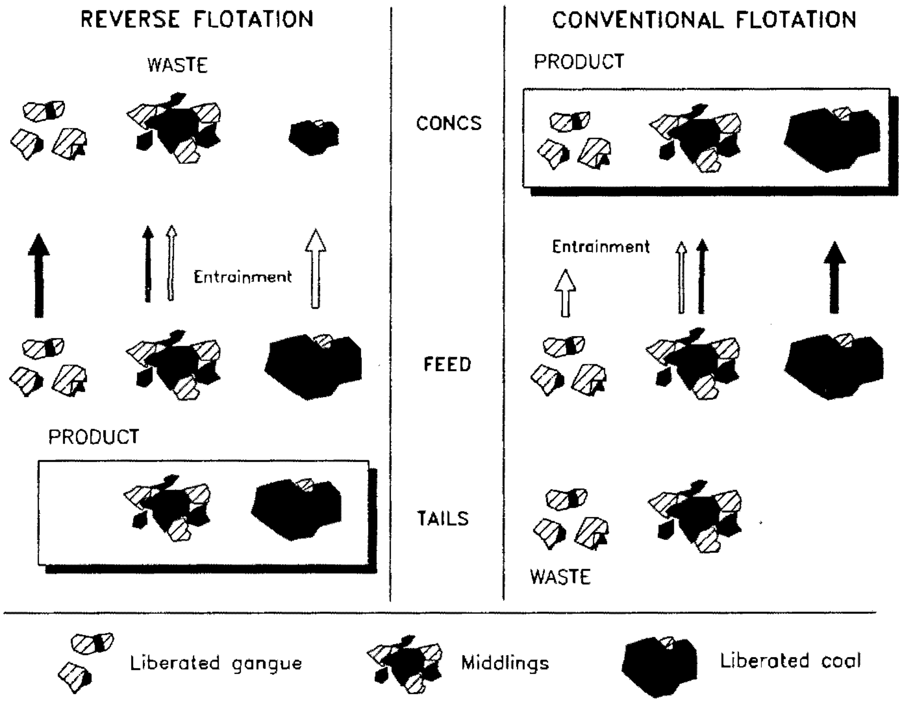


Figure 1.5: Comparison of reverse flotation and conventional "forward" flotation

3.2 Reduction of entrainment

The high mass recovery inherent in forward coal flotation favours entrapment and entrainment of gangue. In the reverse flotation process, however, the gangue material is rising in the pulp, so the process is one of gangue removal from the tails (product) with some coal entrainment into the concentrate. The entrainment of coal particles (along with some middlings) is likely to occur to a lesser extent than the entrainment of gangue particles in forward flotation because of the smaller mass flux, and the fact that coal particles generally have a larger average particle size than the gangue. Thus, if one assumes good collection efficiency in both processes, the grade of the product of reverse flotation should be better than that of forward flotation (see Figure 1.5).

The above discussion reveals an intrinsic difference between the two processes: entrainment occurring in reverse coal flotation results in a loss of coal recovery, whereas with forward coal flotation it results in a decrease in grade.

3.3 Application to column cell technology

The substantial reduction in concentrate mass recovery (for a given feed rate) that would be brought about with reverse flotation is an ideal situation for column cell flotation: for example, if the feed ash of a particular coal is 20%, then there would be a four-fold reduction in mass recovery (if perfect liberation is assumed) in reverse flotation compared to forward flotation. This would mean that, operating the column at the same concentrates recovery rate as forward flotation, a four-fold higher feed rate and hence tails product rate could be attained. For a given column design, fewer columns would be needed to beneficiate a given throughput of coal than for forward coal flotation and this would result in a considerable reduction of plant size and thus capital expenditure.

3.4 Uniformity of gangue floatability

As has already been discussed in section 2.3, flotation of the highly heterogeneous organic coal material is affected by the coal type and other factors affecting coal surface properties, such as oxidation. Coal gangue minerals, on the other hand, are well defined, usually crystalline, structures and there is likely to be far greater consistency in the reverse flotation of different coal types, provided that the gangue material is liberated.

4. Objectives of the research

In this thesis, reverse flotation of coal is investigated as a novel process aimed at solving both the entrainment and mass recovery problems in coal flotation.

The objectives of this research are to develop a reverse coal flotation process for ultrafine coal beneficiation, to investigate the performance of the process in conventional and column flotation cells, and to examine the technical viability of reverse flotation for beneficiating various South African ultrafine coals.

These objectives can be divided into three basic sections:

1. Development of the reverse flotation process. This will include the identification of the gangue in South African coals, the selection of appropriate reagent types for reverse flotation, a decision on which reagent suite to use, and an evaluation of this reagent suite by laboratory adsorption tests.

2. Investigation of reverse flotation in conventional flotation cells. This will involve laboratory batch flotation tests in which the performance of the reverse flotation process is evaluated and compared with forward flotation of the same coals.

3. The extension of flotation tests to a column cell. This will include the comparison of reverse and forward flotation of the same coals in a laboratory column cell, as well as a comparison of these results with those of reverse and forward flotation of the same coals in the batch cell. Performance will evaluated both in terms of coal recovery into the product and gangue removal into the waste stream.

5. Scope

5.1 Scope of the thesis

As the proposed reverse flotation process for ultrafine coal beneficiation is new, it needs to be developed from first principles. In order to obtain a clear understanding and interpretation of experimental flotation results it was decided to use artificial coal feed mixtures as a starting point for the study. These artificial feeds would consist of blends of "pure" coal and "pure" gangue material, which simulates perfect liberation. Although this never occurs in reality, the benefit of using such a feed is that it is easy to define what the ideal separation condition should be and so the flotation performance can be directly evaluated.

Having established the chemical feasibility of reverse coal flotation using the artificial mixtures, and optimized the process with respect to the reagent system, the flotation work would then be extended to various typical South African r.o.m. (run-of-mine) coals to investigate the metallurgical performance achieved.

The batch flotation testwork was performed on a conventional 3-litre laboratory flotation cell which has been used extensively in the Department and has been found to give extremely good reproducibility. The column cell flotation testwork was performed on a 5.3 cm i.d. laboratory cell, which was already set up in the Department and had been used successfully for forward coal flotation tests.

5.2 Structure of the thesis

The study has been divided into four sections:

a) identification of a suitable reagent suite,
b) batch flotation testwork using artificial mixtures,
c) column flotation testwork using artificial mixtures, and
d) flotation testwork on several run-of-mine coals.

There is a separate Chapter for each section. Each Chapter contains a review of the relevant literature (where applicable), details of the experimental work, and the presentation and discussion of the results. A final Chapter contains overall conclusions and recommendations of the study. The appendices contain an additional short Chapter; a description of South African coal structures, types and geographical locations.

CHAPTER 2
SELECTION OF REAGENT SUITE

1. Introduction

The beneficiation of minerals by froth flotation is a physico-chemical separation process. It comprises the attachment and aggregation of hydrophobic ore particles in an aqueous medium to air bubbles and the subsequent buoyant transport of these aggregates into a froth zone, from which they are removed as a "concentrate". Hydrophilic ore particles remain in the aqueous medium and report as "tailings".

In the proposed reverse coal flotation process, the mineral matter in coal will be floated and so must be hydrophobic in order to be recovered in the froth zone, while the organic coal material must be hydrophilic in order to remain in the flotation pulp and be recovered as the "tailings".

The objective of this chapter is to identify reagent types that would be likely to render the surfaces of coal mineral particles hydrophobic and those of the clean coal particles hydrophilic; and to make a preliminary assessment of the suitability of these reagents for reverse flotation of South African coals.

This objective is divided into four areas of focus:

1. A review of the basic principles of flotation, including surface chemistry aspects, and an examination of flotation reagent types, and their behaviour and functions.
2. A review of existing conventional (forward) coal flotation practice and problems.
3. A review of the surface chemistry and structure of the coal and mineral matter content of South African coals, and the selection of reagents that would be likely to establish the exclusive hydrophobicity of the mineral matter in a coal flotation feed.
4. Laboratory adsorption tests to investigate the suitability of the selected reagents and to obtain information about their behaviour that might assist in subsequent flotation tests (Chapter 3).

In this chapter, a separate section is devoted to each of the above.

2. Principles of flotation

The key to flotation of a desired mineral is to ensure that the particular mineral is hydrophobic at the expense of all other minerals in the feed ore. Very few minerals are naturally hydrophobic and so the hydrophobicity has to be chemically induced. Once the mineral is hydrophobic, attachment to air bubbles occurs and the mineral is collected in a froth zone. The froth phase needs to be stable and this generally requires chemical assistance in the form of some surface active compound.

The flotation process can thus be conceptualized as consisting of two fundamental steps, namely:

- The establishment or improvement of the exclusive hydrophobicity of the mineral surface to be floated.
- The provision of bubble transport and a stable froth zone for solids accumulation in the concentrate.

Each of these steps requires the controlled addition of the correct amount(s) of the appropriate chemical reagent(s). In order to select such reagents for a particular flotation process and ore type, the nature of mineral-water-air interactions and the properties, behaviour and usage of different flotation reagent types should be known. These topics are addressed in the sections which follow.

2.1 Mineral–water interactions

2.1.1 Free energy and surface tension

The activity of a mineral surface in relation to flotation reagents depends on the forces which operate on that surface. The atoms in the surface layer, as opposed to the atoms in the solid bulk, are not surrounded by atoms from all directions and thus have unsaturated bonds. As a result, the energy of some of the surface layer atoms remains "free" and this energy is called surface free energy. For liquids, a similar situation exists but the free surface energy is called surface tension. Surface free energy is expressed in terms of work-per-unit-area (i.e. J/m^2), while surface tension is more customarily expressed as force-per-unit-length (i.e. N/m).

Thermodynamically, surface tension or free energy tends to be minimized. This is why, for example, water droplets form spheres (to minimize the free surface area of the droplet). For the surface area of a liquid to be increased, work must be performed on the system. Accordingly, the surface tension for a two phase system is defined thermodynamically as: The free energy increase per unit area under conditions of constant pressure, temperature, and composition. In equation (partial differential) form, this is (King, 1982):

$$\partial\gamma = \frac{\partial G}{(\partial A)_{T,P,n}} \qquad \text{[2.1]}$$

The equation simply states the work required to increase the surface area of the liquid isothermally and reversibly. The surface energy for any two contiguous phases is measured on the interface surface (for example, the solid/liquid interface) and is usually given the symbol γ with the subscripts l-s (for solid/liquid interface), l-g (for the liquid/gas interface) and s-g (for the solid/gas interface).

2.1.1.1 Adhesion

The first stage in a molecular reaction between water and a mineral surface is the wetting of the latter by the former. Mutual tension between the molecules of the two phases makes its appearance at the interface. This phenomenon is called adhesion. Tension is exerted by the one phase on the other through their mutual interface and work is required to separate them. This work is called work of adhesion (J/m^2). It is equal to the sum of the surface energies of the two phases taken separately minus the interphase surface energy at the interface. Mathematically, this is represented by the Dupré equation (Davies and Rideal, 1961):

i.e. $$W_A = \gamma_{l\text{-}g} + \gamma_{s\text{-}g} - \gamma_{l\text{-}s} \qquad [2.2]$$

The equation can be explained by the fact that the free interphase energy ($\gamma_{l\text{-}s}$) is the energy "remaining" after the two phases have reached equilibrium and so the energy that is required to separate the two phases again is equal in value to the total of the separate surface free energies LESS the interphase energy.

The equation implies that if $\gamma_{l\text{-}s}$ is large, then the solid interface is not easily wetted by the liquid, because the free energy has not been minimized. Correspondingly, if $\gamma_{l\text{-}g} + \gamma_{s\text{-}g}$ is large compared to the $\gamma_{l\text{-}s}$ term, then the surface is easily wetted by the liquid, since the work of adhesion to separate the two phases is large.

2.1.1.2 Cohesion

The mutual tension between molecules of the same substance, such as water, is called cohesion and is characterized by the work of cohesion. This is the work which must be done on a system to break a column of liquid of unit cross section into two columns of unit cross section.

2.1.2 Contact angle

In many instances it can be observed that a liquid placed on a solid surface will not wet it, but will rather remain as a drop having a definite angle of contact between the liquid and solid phases. This contact angle, θ, is read towards the liquid phase by convention. In a three phase system, such as the solid/air/water system of ore flotation, the contact angle is measured against an air bubble attached to a solid surface as is depicted in Figure 2.1. The contact angle is used to express quantitatively the wettability of a solid surface by a liquid.

The contact angle can be determined by the Young equation (Tsai, 1982):

$$\gamma_{l\text{-}g} \cos\theta = \gamma_{s\text{-}g} - \gamma_{l\text{-}s} \quad \text{or, more generally:} \quad \cos\theta = \frac{\gamma_{s\text{-}g} - \gamma_{l\text{-}s}}{\gamma_{l\text{-}g}} \qquad [2.3]$$

Note: $\gamma_{s\text{-}g} - \gamma_{l\text{-}s} \leq \gamma_{l\text{-}g}$ because $-1 \leq \cos\theta \leq 1$, or $\gamma_{l\text{-}s} \leq \gamma_{s\text{-}g} + \gamma_{l\text{-}g}$ (from eq 2.2).

It can be seen from equation 2.3 that as the liquid-solid free energy ($\gamma_{l\text{-}s}$) approaches zero, the contact angle approaches zero ($\cos\theta \rightarrow 1$) and the solid is wetted. On the other hand, as the $\gamma_{l\text{-}s}$ term becomes significant in comparison to the $\gamma_{s\text{-}g}$ term, θ tends towards 180° ($\cos\theta \rightarrow -1$) and the solid is preferentially "wetted" by air and is thus not wetted by the liquid. In practice, if the contact angle is greater than 90°, the liquid is considered not to wet the solid.

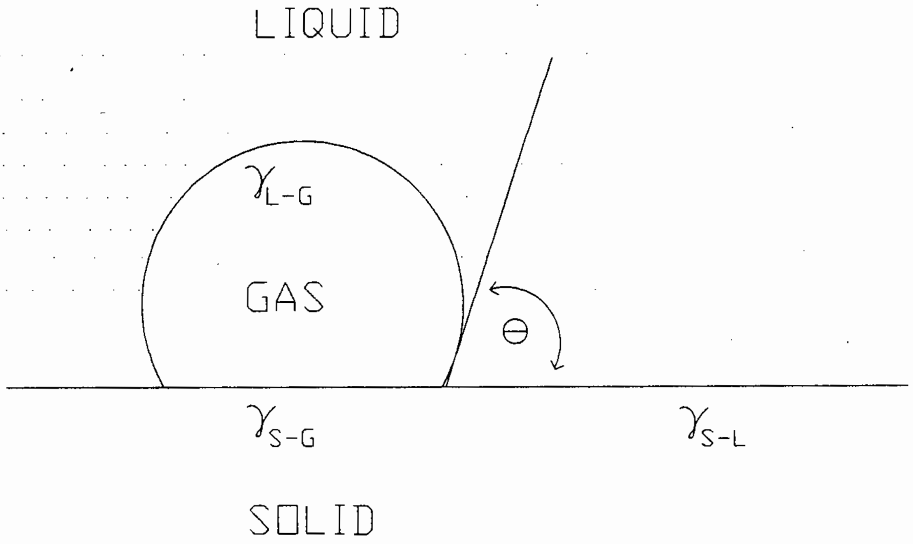


Figure 2.1: Contact angle at a solid surface

2.1.3 Relationship between contact angle and flotation

In a flotation process, the floatability or non-floatability of a mineral is determined by its degree of hydrophobicity. If a solid is **hydrophobic** then non-polar groups characterize its surface. Because water is a liquid of high cohesive energy (due to hydrogen bonding), it interacts with the non-polar groups of a hydrophobic solid only through dispersion or van der Waals forces. These are weak when compared with the cohesive forces of water-to-water interactions, and the work of adhesion is less than the work of cohesion. On the approach of an air bubble, the solid surface will have a finite contact angle because the air-solid interface is favoured in terms of free energy and so the air bubble can attach to the solid surface. The stability of this attachment is determined by the magnitude of the contact angle.

In practice, a contact angle of about 50° to 75° is required for the capture of solid particles by air bubbles (Shaw, 1980). Generally, the higher the contact angle, the higher is the flotation recovery that can be obtained. Hanning and Rutter (1989) have investigated different methods of measuring contact angles on different coal samples. They found that a good relationship exists between contact angle and flotation recovery. This is illustrated in Figure 2.2.

Particle size also influences the relationship between floatability and contact angle. Crawford and Ralston (1988) found with the flotation of various size fractions of hydrophobized quartz that, for a given particle size, there is a unique contact angle below which a particle will not float. Predicting the flotation recovery from the contact angle is thus not straightforward, but measuring the contact angle is a useful way of evaluating both the natural hydrophobicity of a mineral surface and that induced by the addition of reagents (Aplan, 1988).

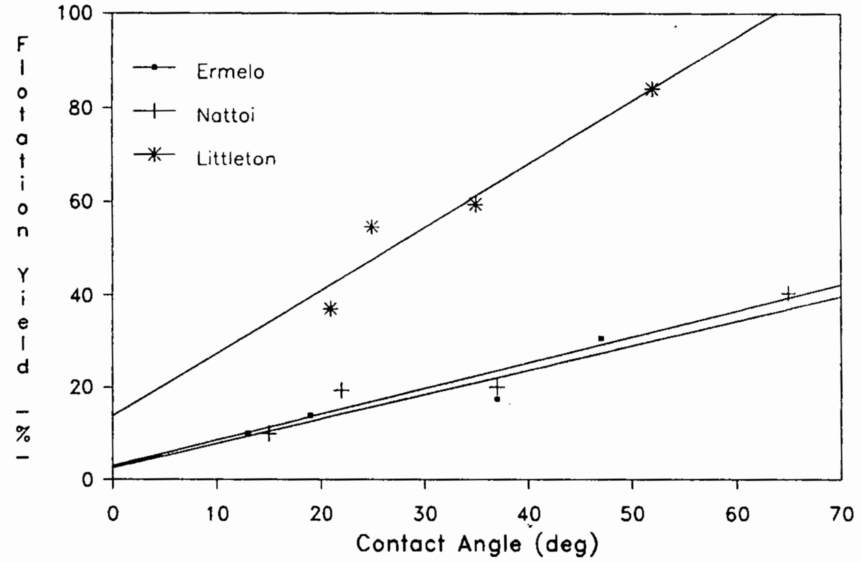


Figure 2.2: Relationship between contact angle and coal flotation recovery (after Hanning and Rutter, 1989).

2.1.4 The electric double layer

Most substances acquire a surface electric charge when brought into contact with a polar medium such as water. For example, the surfaces of finely crushed ore particles in a flotation cell are likely to be charged owing to the unsaturated chemical bonds in the surface layer of atoms of the mineral particles. Ions of opposite charge (counter-ions) in the aqueous phase are attracted towards the solid surface and ions of like charge (co-ions) are repelled away from the surface. As a result of this electrostatic interaction between the surface charge and the ions in solution, the concentration of counter-ions close to the solid surface is considerably larger than at a point which is remote from the surface. Correspondingly, the co-ion concentration is low close to the solid surface.

An electric "double layer" thus forms at the solid/water interface which, at equilibrium, consists of a charged layer on the solid surface and a neutralizing diffuse layer of ions of opposite charge. Thus, although overall neutrality of the system is established, there is a potential as well as a concentration gradient within the diffuse layer. Stern suggested the idea that the diffuse region near the solid surface be divided into two parts (Adamson, 1967, p 75; Davies and Rideal, 1961):

1. An inner layer of ions adsorbed at the surface, forming a compact, double layer called the Stern layer.

2. An outer skin consisting of a diffuse layer of ions.

The two regions are depicted in Figure 2.3. In the Stern plane, the adsorbed ions are not free to move in solution and so form a secondary "interface" or shear plane with the solution. Since the ionic radii have finite dimensions, the width of the Stern layer and hence the distance of the shear plane from the solid surface depends on the type of adsorbed ion.

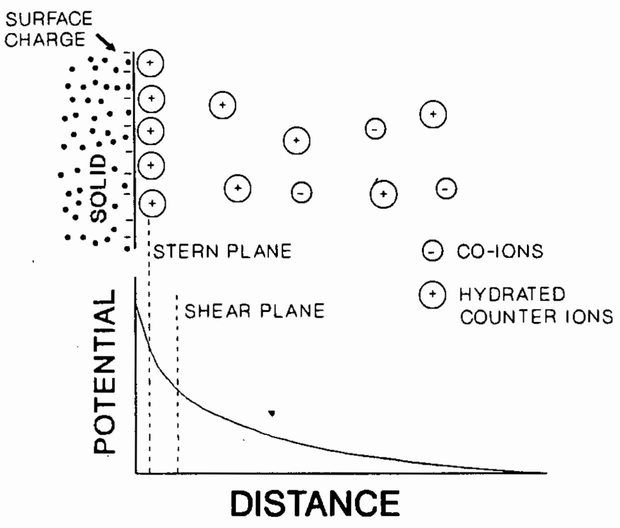


Figure 2.3: The Stern double layer

2.1.5 The Zeta potential and pH considerations

The potential at the shear plane is often assumed to be equal to the "zeta potential". This is the potential induced by the movement of a charged particle through a bulk medium and is measured from electrokinetic experiments. A knowledge of the charge, or zeta potential, at this shear plane is important for selecting flotation reagents. The sign of the zeta potential determines the selection of the type of flotation reagents; i.e. whether they should be cationic or anionic or non–ionic.

Flotation reagents usually adsorb onto the oppositely charged surface of the solid and form a double layer of collector molecules according to the process described above in section 2.1.4. The adsorbed ions in the Stern plane alter the effective charge on the mineral surface and may even cause a charge reversal. This adsorption will be dependent on the existing double layer at the mineral-water interface which will be composed of hydroxyl or hydronium ions which are the potential determining ions.

The zeta potential is strongly affected by the pH of the pulp (pH is defined by the relative concentrations of H^+ and OH^- ions). The pH at which the zeta potential is zero is called the point-of-zero charge or PZC and this parameter is used to select the reagents and possibly the electrolytic conditions of the flotation process.

If an anionic collector is to be used, the pH of the pulp should be maintained below the PZC pH, since a positive surface charge on a solid surface is neutralized at or above the PZC pH by the hydroxyl ions (OH^-) in solution. Below the PZC pH, however, the zeta potential will be positive and the anionic collector can attach itself to the solid without repulsion of surrounding ions (Tsai, 1982).

The reverse is true for cationic reagents; the pulp pH must be kept above the PZC pH because the reagent will "see" a neutral or positive solid surface at or below the PZC pH and will not adsorb. It is thus useful to know the PZC pH for the particular substance to be floated because this will determine the choice of reagent type and/or the pH conditions of flotation.

2.2 Establishing hydrophobicity: reagent types

For a particular mineral to be floated, the surfaces of particles of that mineral must be hydrophobic at the expense of all the other particles in the flotation pulp. Most mineral surfaces are hydrophilic because the solid is held together by ionic and covalent bonds. When minerals are crushed, for example when an ore is milled, these ionic or covalent bonds are broken giving rise to highly reactive, charged sites which readily hydrate in water. These ionic and/or polar sites must be removed (or rendered hydrophobic) if the desired mineral particles are to be floated. At the same time, the floatability of other species must be minimized by ensuring that their surfaces are hydrophilic. These objectives are achieved through the addition of various chemical reagents to the flotation pulp mixture. Such reagents can be classified as collectors, activators, depressants and modifiers.

2.2.1 Collectors

A collector is usually a surface-active, heterogeneous compound that contains an active inorganic group joined to a hydrocarbon chain of variable length. Such a molecule functions by adsorbing electrostatically onto the mineral surface by means of the active inorganic group. The hydrocarbon chain is directed into the aqueous phase and imparts hydrophobicity to the mineral surface due to the non-ionic nature of the chain.

There are four classes of collectors; namely cationic, anionic, amphoteric and insoluble oils (non-surfactants).

2.2.1.1 Cationic

A cationic collector would be used where the mineral surface has a predominantly negative charge or can be modified to have such a charge. The only cationic collectors used in the industry are amines (King, 1982). Some examples of amine collectors are listed in Table 2.1. The hydrocarbon chains that are attached to the central nitrogen atom are usually long; eight or more carbon atoms in length. Thus, a mineral surface that is receptive to adsorption by these amines should be floatable because of the hydrophobic nature of the long hydrocarbon chains that would characterize the mineral surface after adsorption.

Table 2.1: Amine collector types

Collector	Formula	Ion
1° Amine salt	RNH_3Cl	RNH_3^+
2° Amine salt	$RR'NH_2Cl$	$RR'NH_2^+$
3° Amine salt	$R(R')_2NHCl$	$R(R')_2NH^+$
Quaternary amine	$R(R')_3NCl$	$R(R')_3N^+$
Alkyl Pyridinium salt	R Pr NHCl	R Pr NH^+

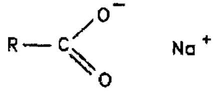
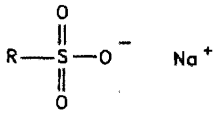
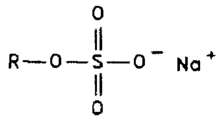
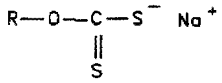
Amines are classified as primary (1°), secondary (2°), tertiary (3°) and quaternary depending on the number of hydrocarbon radicals attached to the central nitrogen atom. The primary, secondary and tertiary amines are weak bases and their ionization is pH dependent. In contrast, the quaternary amines and the alkyl pyridinium salts are strong bases and are completely ionized at all values of pH (King, 1982).

2.2.1.2 Anionic

Anionic collectors are used where the mineral surface charge is positive. The collectors that fall into this category are the carboxylates, sulphonates, alkyl sulphates and sulphydryls and some chelating agents. Table 2.2 shows some examples of anionic collectors.

Solution pH plays an important role in the ionization behaviour of carboxylates and xanthates (sulphydryl), which are weak acids. For the alkyl sulphates and the sulphonates, which are strong acids, this does not occur.

Table 2.2: Anionic collector types

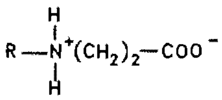
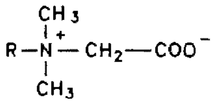
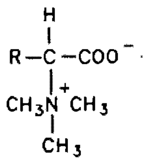
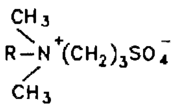
Collector type	Structure
Carboxylate	
Sulfonate	 $R-S(=O)_2-O^-\ Na^+$
Alkyl Sulfate	 $R-O-S(=O)_2-O^-\ Na^+$
Xanthate	 $R-O-C(=S)-S^-\ Na^+$

2.2.1.3 Amphoteric

These substances are heteropolar organic compounds possessing at least two oppositely charged functional groups per molecule. Some examples of amphoteric collectors are given in Table 2.3.

When dissolved in water, the unshared electron pair on the nitrogen atom is capable of accepting a proton in a acid environment. This imparts a positive charge to this site and so the molecule is a cationic collector in acid solution. In an alkaline solution, the molecule is an anion. In the isoelectric region, both positive and negative ions are equal and the molecule is believed to be in the zwitterion form (from Smith, 1988 pp 219–253).

Table 2.3: Amphoteric collector types

Collector type	Structure (zwitterion form)
N-alkyl-2-aminopropionic acids	 $R-N^+H_2(CH_2)_2-COO^-$
n-alkyl-N,N-dimethylglycines	 $R-N^+(CH_3)_2-CH_2-COO^-$
Alkyl betaines	 $R-CH(N^+(CH_3)_3)-COO^-$
N-alkyl-N,N-dimethylammon-iopropane sulphonates	 $R-N^+(CH_3)_2(CH_2)_3SO_4^-$

2.2.1.4 Insoluble

This group consists of the oily collectors used in coal flotation. An insoluble oil, such as dodecane or a paraffin derivative, is dispersed in water and the small oil droplets adsorb onto the partially hydrophobic organic coal surface, enhancing or promoting the hydrophobicity.

2.2.2 Activators, modifiers and other flotation reagents

A successful mineral flotation requires at least one of the minerals in the pulp to be made selectively hydrophobic. Sometimes it is sufficient just to add a collector, but often additional reagents are required to accentuate the difference in the surface chemical properties. Among the more common of these additional reagents are activators, depressants, and modifiers.

An **activator** promotes the adsorption of a collector onto a specific mineral. It functions by adsorbing onto the solid electrostatically and reversing the sign of the Stern potential on the mineral surface. It thus causes the adsorption of collectors that have a charge of the same sign as that of the mineral. Flotation of highly oxidized metallic sulphides with thiol collectors can be enhanced with sodium sulphate addition. Copper sulphate has been used to activate the flotation of ZnS and FeS_2.

A **depressant** is used to inhibit flotation of a particular species by changing the particle surface from hydrophobic to hydrophilic or by preventing the adsorption of collectors on a specific mineral surface. Inorganic depressants usually behave in the latter fashion by reversing the Stern potential of the solid, rendering the potential of the same sign as that of the collector. Organic depressants, such as high molecular weight starch derivatives, function by adsorbing onto the solid surface forming hydrophobic aggregates.

Modifiers are a broad class of compounds that modify the flotation pulp conditions. These include pH modifiers, flocculants and dispersants. A dispersant is sometimes used in flotation to counteract the effect of slime adsorption or to deflocculate materials such as clays.

2.3 Bubble formation and the froth phase

The formation and stabilization of the froth is the second important step in flotation. Once the solid particles have been rendered hydrophobic by adsorbed collector molecules, they must successfully attach themselves to air bubbles and be carried to the froth phase. There they must reside long enough to be scraped into the concentrate.

The addition of a surfactant frother is usually vital for establishing a stable froth phase on top of a flotation pulp. Frother improves the dispersion of air in the flotation cell and reduces the coalescence of individual bubbles. The frother type can also have an important effect on bubble size, which affects mineral recovery.

2.3.1 Frothers

Frothers are surface active agents, usually polar molecules. A frother is used to reduce the bubble size in the pulp and to stabilize the surface froth through the reduction of the surface tension of the air/liquid interface. This is achieved by the heteropolar frother molecules establishing themselves on the wall of the bubbles; the hydrophobic part orientated into the gas phase and the polar part orientated into the water. Non-ionic surfactants, generally slightly soluble monohydroxylated compounds such as cresol, Methyl Isobutyl Carbinol (MIBC), Di-isobutyl Ketone (DIBK) and pine oil are commonly used as frothers.

3. Coal flotation: practice, problems and a proposed new process

Coal is a highly heterogeneous mixture of both organic and inorganic material.

In the conventional (forward) coal flotation process, the practice is to float the organic material off as a concentrate. The usual procedure is to add an insoluble collector oil, such as kerosene, to enhance the natural hydrophobicity of the coal surface. The collector oil is dispersed in the flotation pulp and can adsorb onto the surfaces of coal particles. This adsorption increases the contact angle and the coal particles, which cannot be wetted by water, then attach to air bubbles in the flotation pulp. The air-bubble/coal-particle aggregates are transported to the froth phase and the coal is recovered as a froth concentrate. A surfactant frother, such as MIBC, is added to assist the formation of air bubbles and the stability of the froth zone.

The gangue minerals are naturally hydrophilic; they do not attach to the air bubbles but remain in the pulp and are discarded in the tailings.

3.1 Coal floatability

Although coal possesses a natural hydrophobicity and thus has a natural floatability, the exact nature of the coal surface is highly variable because of the heterogeneous nature of coal structure. Coals of different geographical and/or geological origin thus exhibit different flotation behaviour. Even variations within a specific coal seam, as well as external effects such as weathering (oxidation), will affect flotation.

The natural floatability of a coal is dictated by the hydrophobicity of its surface. Laskowski (1986) has listed three major factors responsible for coal surface properties affecting hydrophobicity:

- The presence of inorganic impurities (mineral matter).
- Hydrocarbon structure (affected by rank and maceral types).
- The oxygen functional groups content.

Coal contains a broad range of mineral matter types, the main ones being the clay minerals (such as kaolinite and illite), quartz and pyrite. These minerals generally have charged or polar surfaces which are hydrophilic. The greater the fraction of mineral matter in a coal, the greater will be the fraction at the surface. Coals with a higher mineral matter fraction (ash content) will generally be less hydrophobic than coals with a lower ash content.

The hydrocarbon structure of the coal also has a strong influence on its natural floatability. As the rank (degree of coalification) of a coal increases, the chemical structure changes. There is a gradual elimination of polar groups, such as hydroxyl and carboxyl, and an increase in the percentage carbon which increases hydrophobicity. Hydrophobicity reaches a peak for bituminous coal, after which there is a slight decline as the rank increases to anthracite (Osborne, 1988; Tsai, 1982). As a rule, low to medium volatile bituminous coals are sufficiently hydrophobic to be floated with frother alone. Generally, however, collector oil is added to improve the flotation recovery; the required quantity increasing with decreasing rank (Aplan, 1988).

Coal hydrocarbon structure and hydrophobicity are also affected by the petrographic composition of the coal, i.e. its maceral content. The macerals are the basic microscopic, physically distinct and chemically different constituents of the carbonaceous matter in coal which originate from material deposited in the primeval swamps (Falcon, 1978). On a macroscopic basis, the macerals occur combined in various proportions to form "lithotypes". The lithotype fusain (containing intertinite group macerals) is generally the least floatable and vitrain (containing vitrinite group macerals) is the most floatable (Osborne, 1988).

The third factor affecting the natural floatability is the degree of oxidation of the coal surface. Oxidation causes the coal to act as if it were of lower rank and this has a detrimental effect on flotation, because the hydrophobicity of the surface is reduced. Increased collector oil additions and even collector promoters (Mishra, 1987) are often needed to improve the flotation performance of oxidized coals.

It is of interest that only **one** of the three factors discussed above affects the propensity of a coal for beneficiation on the basis of relative density, i.e. its washability. This factor is the presence and distribution of the inorganic impurities. There is often a marked difference between the floatability of a coal and its washability. This is illustrated by Figures 2.4 and 2.5 which show the washability determined by the usual float and sink analysis (Osborne, 1988) and floatability (determined, for example, by the method of Dell (1961), of two hypothetical coals. Figure 2.4 shows an "easily floating" coal, while Figure 2.5 shows the case for a "poorly floating" coal. As can be seen, a coal may be "well liberated" in terms of separate "free" ash and coal particles, but may still be poorly floatable. This situation is common with South African coals (see Figure 1.4).

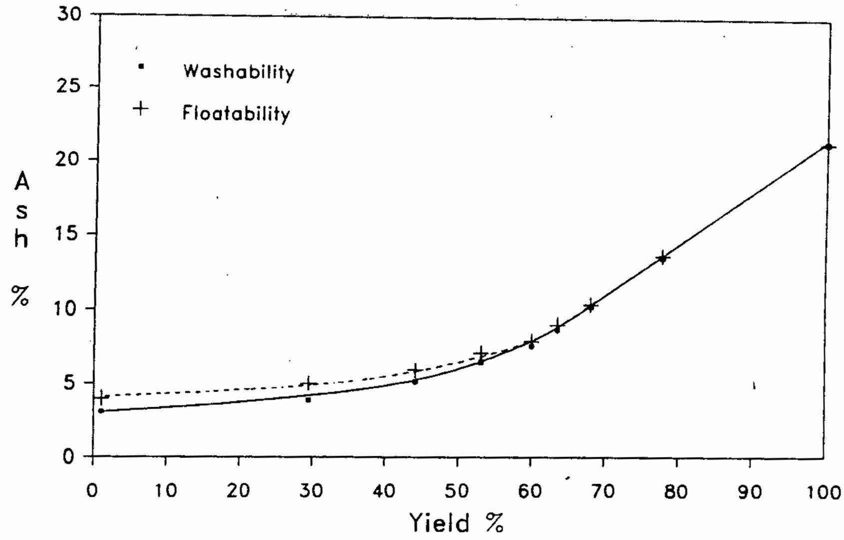


Figure 2.4: Easily floating coal: comparison of washability and floatability

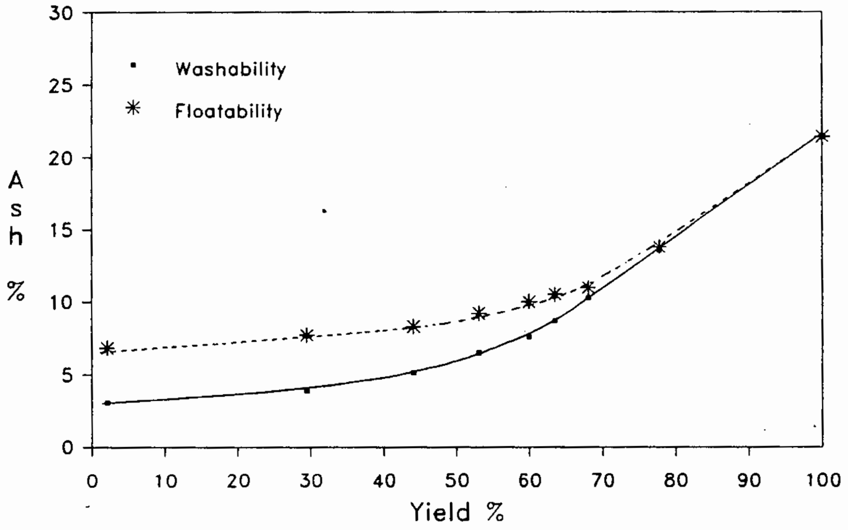


Figure 2.5: Poorly floating coal: comparison of washability and floatability

3.2 Improving flotation efficiency and selectivity

In addition to the inherent hydrophobicity of a coal surface, there are many other factors which affect the recovery of coal in a flotation plant or circuit. These include operational variables such as particle size, pulp density, froth height and residence time; design variables such as flotation equipment and circuit configuration; and chemical factors such as collector or frother types and concentration.

Traditionally, coal has been floated in a single stage circuit (Mishra, 1987). This practice can lead to problems like preferential consumption of flotation reagents by the finer feed size fractions at the expense of the coarser fractions. In such a case, a one stage circuit is inefficient and more complex configurations may be used. For feeds with a significant proportion of finer material, two concepts are popular: primary/scavenger flotation, and separate flotation of classified "coarse" fines (+0.075 mm) and "slimes" (−0.075 mm) (Mishra, 1987).

In the primary/scavenger flotation circuit, the concentrates from the primary stage have a low coarse particle content because of the preferential consumption of the reagents by the slimes. The tailings of the primary stage (containing a high fraction of "coarse" fines) are fed to the scavenger stage where additional reagent is added to recover the coarse material. Flotation of this coarse material is often difficult because the slimes, which help stabilize the froth, are not present in sufficient amounts. This has led to the development of specialized frothers, such as the Dow Chemical Corporation's "DOW 400", which improve the recovery of coarse particles (Klimpel and Hansen, 1987, pp 102–103).

Flotation circuits with feed pre-classification have gained considerable popularity for treating feeds with a high percentage of −0.035 mm material. In such circuits, the coarse and slimes are conditioned separately. They may then floated separately or recombined (after separate conditioning) and floated. In this way, the overall plant recovery is improved. As with the primary/scavenger circuits, the flotation often has to be optimized for the individual size fractions by the use of specialized reagents, such as the use of different frother types for the coarse fines and the slimes (Mishra, 1987).

Another major problem in coal flotation plants has been the presence of very fine clay minerals which leads to a dramatic loss of flotation recovery. This is caused either by the "slime coating" of the coal surface, which reduces hydrophobicity; or by the uptake of reagents (intended for the coal) by the very fine clay particles or slimes. To avoid this situation, various strategies are adopted. One such strategy is to deslime the feed. A second is to add chemical modifiers or desliming agents to reduce the slime coating on coal particles (Mishra, 1987).

The slimes can also enter the concentrate by the mechanism of entrainment, and thereby increase the ash content of the concentrate. Entrainment occurs in conventional sub-aeration flotation cells because of the net upward flow of feed water which "drags" the slimes into the froth phase.[1] In coal flotation, this is aggravated by the high mass recovery (typically 50% to 80%, compared with 2% or less in the flotation of most mineral ores).

One way of reducing entrainment is to operate at very low pulp densities: it is common in coal flotation to operate at a solids content of 5 to 9% (Picard, 1985) (c.f. pyrite flotation where the pulp density is typically 30%). This reduces mass flux and thus entrainment. Water sprinkling of the froth can also help to reduce entrainment (Miller, 1969). An alternative approach is to use column flotation cells. This type of equipment is relatively

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[1] The effect of entrainment in coal flotation has been illustrated in Chapter 1, see Figure 1.4; sec. 2.3

new and consists of tall, vertical columns in which flotation is carried out. A feature is a deep froth zone in which there is a net downward flow of water or "positive bias" which rinses entrained particles out of the froth and thus improves the product grade. Column cells have already been discussed in some detail in Chapter 1 (see section 3).

As mentioned in the previous section, oxidation of a coal surface will reduce its hydrophobicity and thus its potential for flotation. The floatability of oxidized coals may be restored chemically. Methods include the use of cationic amines as collector promoters (in conjunction with the collector oils) and emulsifying agents to improve the collector-coal adsorption. The cationic amine molecules attach to the negatively charged coal surface and improve the surface hydrophobicity with non-polar groups, while the emulsifiers improve the dispersion of the collector oils and induce them to spread across the coal surface (Mishra, 1987).

3.3 Sulphur cleaning

In North America, a major focus of coal cleaning is the reduction of coal sulphur content. Sulphur is inherent to the organic material (organic sulphur), or occurs as pyrite with other coal minerals (inorganic sulphur). In recent years, attempts have been made to design flotation circuits to reduce the pyrite content of fine coal. One of these methods is the two stage reverse flotation process (Miller et al., 1984), which involves depressing the coal with an appropriate depressant and floating off the pyrite with collectors commonly used in sulphide mineral flotation. The coal is then "re-activated" and floated in the normal fashion. This process has, however, been found to be commercially unattractive (Mishra, 1987).

In South Africa, sulphur levels are generally lower than in North American coals (0.5–1.5% compared to 2–3%) and sulphur cleaning has not been practised as yet. However, due to increased concern about air pollution levels this is likely to change, and consequently there will be a future demand to develop technologies for sulphur cleaning.

3.4 Flotation of South African coals

South African coals, like other Southern Hemisphere (Gondwanaland) coals, differ from most Northern Hemisphere coals in a number of ways: they have a higher mineral matter content, they have different proportions of maceral types, are generally of lower rank, and they are more difficult to liberate (Falcon, 1977). As a consequence, South African coals, particularly those from the commercially important Witbank/Middelburg coalfield, are "poorly floating".

By way of example, typical washability characteristics of South African coals in relation to other major world coal types are shown in Figure 2.6 below. The diagram shows the cumulative yield/ash curve for a number of European coals (numbers 1–3 on the diagram) as well as for a number of Gondwanaland coals including two South African coals (numbers 7 and 8). As can be seen, the yield that can be obtained for a given ash content is poor for the South African coals indicating poor liberation.

In addition to the problem of poor liberation, South African coals are less floatable than Northern Hemisphere coals because they are deficient in the macerals of the vitrinite and exinite groups, which are the most naturally floatable. Typicaly, South African coals contain 40–80% inertinite, whereas European coals contain 70–90% vitrinite (Falcon, 1978; Sanders and Brookes, 1986).

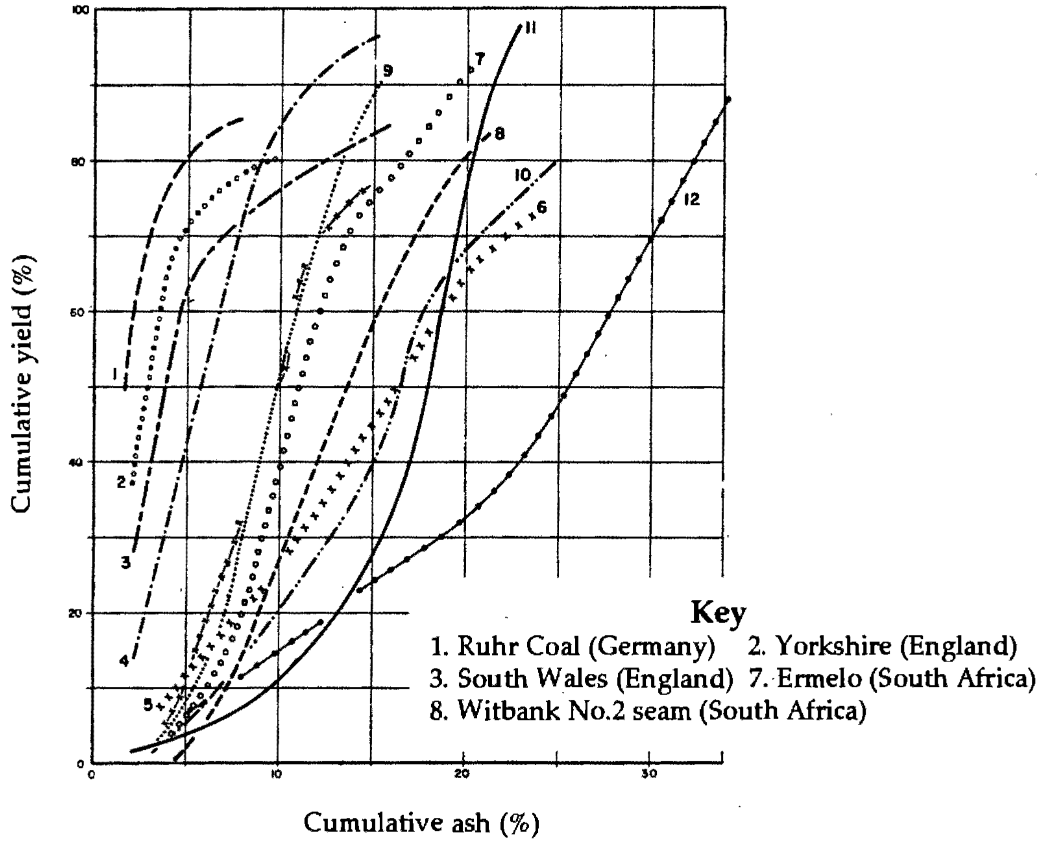

Figure 2.6: Washability of European and Gondwanaland coals (after Sanders and Brookes, 1986).

Because of the difficulty of floating South African coals, very few flotation plants are in existence. The first flotation plant was installed in 1962 in the Natal coalfield (Horsfall and Franzidis, 1988) to treat the −0.5 mm fines. This plant has since closed and today only six flotation plants are in operation, most of which treat the ultrafines (−0.1 mm). None of these are in the Witbank coalfield.

Concern in the 1980s about fines discards has resulted in the use of new methods to beneficiate −0.5 mm coal. A practice which is rapidly gaining popularity on coal washing plants is to use spiral concentrators to beneficiate the −0.5 mm + 0.1 mm fines. There are currently 17 spiral plants in operation. On five of these plants, the ultrafines are beneficiated by flotation; on the other plants the ultrafines are still discarded or stockpiled. In recent years there has been a re-examination of the role of flotation for treating ultrafines (Voges, 1991). New methods, such as column cell flotation, have been examined and there is also interest in oil agglomeration (Franzidis, 1991).

3.5 Reverse coal flotation

The proposed reverse coal flotation process, which has been described in considerable detail in Chapter 1, section 3, is an attempt to look completely afresh at the beneficiation of ultrafine coals: in reverse coal flotation, the mineral matter in coal would be floated off as a **concentrate** which would be **discarded** while the "tailings" would form the clean coal product. The main anticipated advantages of reverse flotation have already been discussed in Chapter 1 (section 3): they may be summarized as follows:

1. *Reduction of mass recovery in coal flotation equipment*: In reverse flotation typically 10 to 30% of the feed mass would be recovered in the concentrates as opposed to 70 to 90% in forward flotation. This would allow operation at higher feed pulp densities and feed rates, and would circumvent the carrying capacity limitations of column cells. This could have a significant effect on reducing the **cost** of flotation equipment.

2. *Reduction of entrainment*: A reduction in mass recovery would reduce the feed water recovery in the froth in sub-aeration cells and thus reduce the tendency for entrainment. This would enable better selectivities to be achieved at higher pulp densities.

3. *Uniformity of gangue floatability*: Reverse flotation of the coal minerals which have well defined structures is likely to be more consistent than forward flotation of highly heterogeneous coal.

The remainder of this thesis is devoted to the development of the new reverse coal flotation process, and its evaluation in sub-aeration and column flotation cells.

4. Selection of reagents for reverse coal flotation

The first stage in the development of a reverse coal flotation process requires the selection of suitable flotation reagents. In order for the (naturally hydrophilic) mineral matter or "ash" in coal to be floated, it must possess a hydrophobic surface, while the (naturally hydrophobic) coal surface must be made hydrophilic so that it does not float. The following reagents are thus required:

- an **ash collector** which can adsorb onto the hydrophilic mineral surface and render it hydrophobic,

- a **coal depressant** which will render the organic coal surface hydrophilic, and

- a **frother** to promote bubble formation and a stable froth phase.

The selection of appropriate reagents with which to carry out testwork to evaluate the new reverse flotation process is made in the sections which follow.

4.1 The mineral surface: selection of an ash collector

4.1.1 The mineral surface

In order to select a collector for the coal ash minerals, the nature of the surfaces of the major mineral constituents must be considered. The clay minerals are by far the most important minerals in coal and, on average, account for 60 to 80 % of the total mineral matter associated with coal (Stach, 1974). The clay minerals are often found as finely dispersed inclusions in the coal. As a rule intergrowths do not exceed several microns in size (Stach, 1974). In South African coals, clays may comprise up to about 70% of the mineral intergrowth, with the major type being kaolinite. Other clay types are illite (muscovite) and chlorite.

The structure of kaolinite is represented in Figure 2.7 below. It is a two layer, sheet-structure aluminosilicate with alternating layers of silica tetrahedra and octahedral aluminium hydroxide. Where the hydroxyl ions of one sheet come into contact with the tetrahedral oxygens of the next sheet, hydrogen bonds hold the structure together. This mineral has a hydrophilic surface because of the broken Si-O bonds on the edges and broken hydrogen bonds on the faces. The double layer charge on the face of such a sheet in an aqueous medium will be determined by the ions H^+ and OH^-, the potential determining ions:

$$\text{Si-OH}_2^+ \overset{H^+}{\leftrightarrow} \text{Si-OH} \overset{OH^-}{\leftrightarrow} \text{Si-O}^-$$

Figure 2.8 shows the zeta potential as a function of pH for a number of coal minerals, including kaolinite (Mishra, 1987). It can be seen that PZC of kaolinite is at pH=3.4 and that the zeta potential of this mineral is negative above this pH which means that the surface is anionic in nature. At the edges, the situation is different. The tetrahedral silica sheets and octahedral alumina sheets are disrupted when primary bonds are broken. Re-adsorption of small amounts of aluminium ions leached out of the clay could give the edges a net positive charge.[1] Generally, however, the kaolinite surface should be

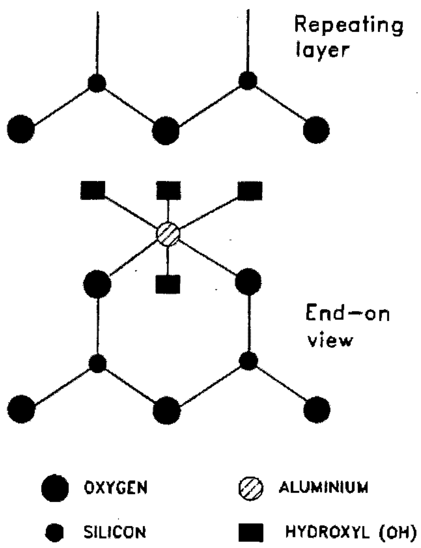

Figure 2.7: Structure of kaolinite

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[1] This usually occurs at low pH values. Under some conditions, the entire surface area may be positively charged (King, 1982)

anionic in nature above the PZC pH because of the much greater area of the face relative to the edge.

Quartz, which makes up about 10 to 20% of the coal mineral matter, is the second most important mineral in coal and occurs in discrete layers or intimately associated with the clays. It is a framework silicate in which fractured Si-O bonds give rise to Si-OH and Si-O$^-$ sites which means the mineral surface is anionic above the PZC pH which occurs in the low pH region of around pH=1.8 (King, 1982; p 30).

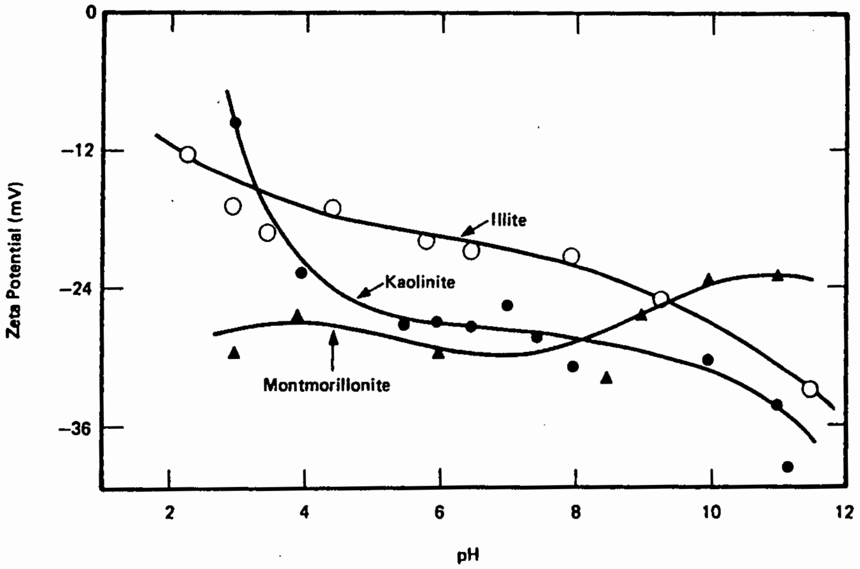

Figure 2.8: Zeta potentials for various minerals
(after Mishra and Klimpel, 1987)

4.1.2 Cationic amine collector types

It is evident from an examination of the mineral surfaces, and a consideration of sections 2.1.5 and 2.2.1.1, that a **cationic** type collector would be required to make both quartz and kaolinite hydrophobic. The most common cationic collectors are the long chain amine salts, such as dodecylamine hydrochloride which has already been found to be successful in the flotation of quartz (Lin, 1974):

$$CH_3\,(CH_2)_{11}\,NH_3{}^{+}Cl^{-}$$

This compound is water soluble and collector action is by adsorption of the cationic end of the molecule to the anionic sites of the mineral surface with subsequent orientation of the hydrocarbon chain into the aqueous medium. The hydration layer around the mineral surface breaks down and the mineral surface becomes hydrophobic.

As mentioned briefly in section 2.2.1.1, the cationic amines can be divided into two groups, according to the number of alkyl groups which are substituted on the nitrogen of the amine:

Group 1 are the primary (1°), secondary (2°) and tertiary (3°) amines, in which at least one hydrogen remains on the nitrogen, e.g. dodecyl-dimethyl-ammonium chloride:

$CH_3(CH_2)_{11}N(CH_3)_2H^+ Cl^-$

These compounds are weak bases and the ionization of the salt is affected by solution pH. They have at least one proton (H^+) to donate and the ionic species can revert to the neutral, insoluble amine by the following reaction:

$$RNH_3^+ + OH^- \leftrightarrow RNH_2 + H_2O \quad \text{[2.4]}$$

The ionic species may also hydrolyze to the weak base by the reaction

$$RNH_3^+ + H_2O \leftrightarrow RNH_2 + H_3O^+ \quad \text{[2.5]}$$

In both cases, the equilibrium is driven to the right as the solution becomes more alkaline. The stronger the base strength of the compound, the higher the pH of the solution must be to favour the first reaction (2.4). This means that the stronger the base, the higher the pH of the flotation pulp can be to still allow maximum flotation. Figure 2.9 is a diagram of log concentration of the ionic and molecular species of 1°, 2°, 3° and quaternary amines possessing 12-carbon length chains. In this case, the 2° amine has the highest pK_a value (11.0), which means that it has the highest base strength. The increase in the molecular species $R(R')NH$ at the expense of the ionic species $R(R')NH_2^+$ occurs at a higher value of pH than for the 1° and 3° amines.

It is clear from the above that the ionization and thus flotation efficiency of this group of collectors is determined by solution pH; this would be an important consideration when selecting them as a collector for the coal minerals.

Group 2 are the quaternary amines, in which all hydrogen atoms on the nitrogen have been substituted by alkyl groups, such as dodecyl-trimethyl- ammonium chloride:

$CH_3(CH_2)_{11}N(CH_3)_3^+ Cl^-$

These molecules are strong bases and, furthermore, do not have a proton to donate; they are completely ionized at all values of pH. The hydrolysis reaction cannot occur, because there is no spare proton:

$R(R')_3N^+ + H_2O = ?$ (c.f. eq 2.5)

and the ionization equilibria favour the ionic species because of the high pK_a value:

$$R(R')_3NH + H_2O \leftrightarrow R(R')_3N^+ + OH^- \quad \text{[2.6]}$$

This behaviour is clearly evident from Figure 2.9.

The fact that the ionization state of the quaternary amine is independent of the pH (except at very high pH values) gives them a clear advantage in terms of their selection as a collector for coal minerals.

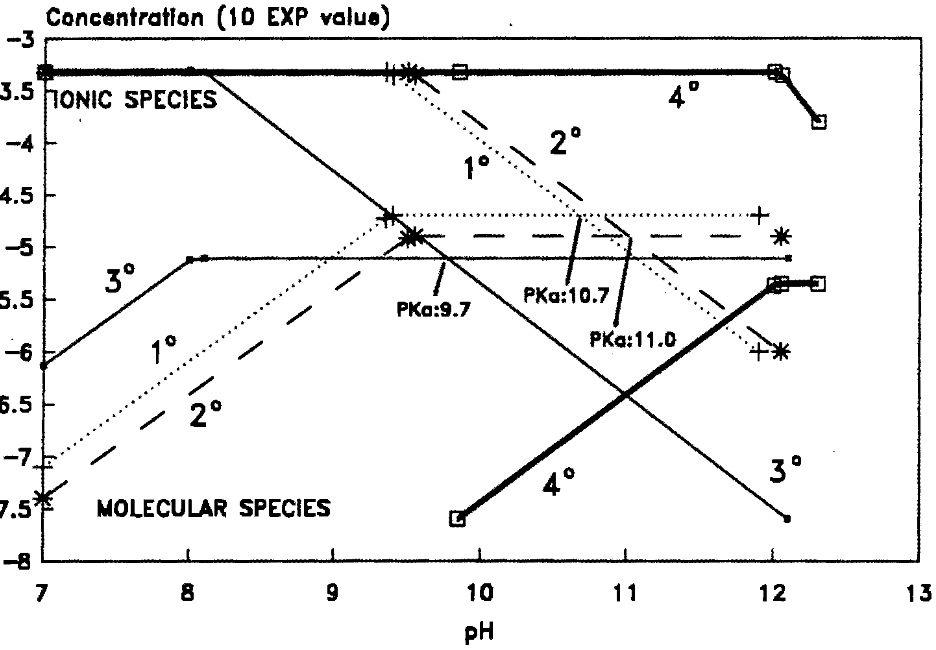

Figure 2.9: Log concentration diagram of 1°, 2°, 3°, and quaternary amines (after Smith, 1988)

4.1.3 Cationic amine collector adsorption mechanism

The adsorption of the cationic amines onto mineral surfaces occurs in two basic steps:

- Concentration of the amine in the double layer,
- Formation of aggregates at the mineral-water interface.

Long chain electrolytes, e.g. amine collectors, can function as counter ions in the double layer (see Figure 2.3) at the mineral-water interface. At low collector concentrations, collector ions adsorb by ion-exchange with other ions present in the diffuse layer, so that there is no net change in the distribution of charge between the Stern and the diffuse layers, and the zeta potential remains constant. At a certain critical collector concentration, there is a sudden change in the zeta potential and a rapid increase in the adsorption density which is attributed to the formation of hydrophobic associations between the adsorbed surfactant ions (Ives, 1984; p 248). Lin (1974) found that in the quartz/dodecylamine hydrochloride system there is a certain collector concentration point beyond which the adsorption density of collector on the quartz surface increases more rapidly than before as the bulk concentration rises. This was attributed to the formation of aggregates of dodecylamine ions on the quartz particles.

Aggregates are formed when the local concentration of the amine in the double layer exceeds both the concentration in the bulk solution and the critical micelle concentration (CMC) of the amine.[1] When this happens highly charged hemi-micelles form, also

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[1] Micelles are the formation of groups or "aggregates" of a particular molecular species in solution. This occurs at a certain critical concentration, the Critical Micelle Concentration.

known as *hemicelles*, which are strongly adsorbed onto the negatively charged mineral surface. The formation of hemicelles is depicted in Figure 2.10.

The formation of hemicelles at the mineral-water interface is generally accompanied by an increase in the hydrophobicity and hence the floatability of the mineral. The adsorbed ions are predominantly orientated with their ionic groups towards the polar/ionic mineral surface and the non-polar group pointing into the aqueous (pulp) phase. It should be noted that too high a collector concentration can actually reduce flotation efficiency because of the adsorption of a second layer of collector molecules with an orientation opposite to that of the first layer (Tsai, 1982, pp 212–220).

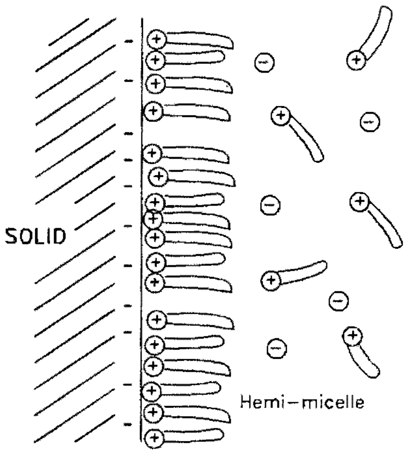

Figure 2.10: Hemi–micelle formation

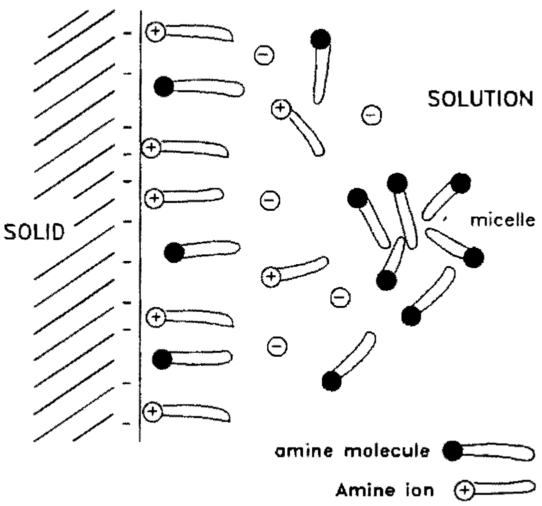

Figure 2.11: Effect of high pH on micelles

Another consideration in the adsorption mechanism is the CMC of the particular amine which is adsorbing. If the bulk concentration of the amine exceeds the CMC, then micelles of amine will form in solution. This is detrimental to the amine adsorption, since the molecules "bunch together" and do not adsorb at the surface. Lin (1974) kept the concentration of the dodecylamine hydrogen chloride collector well below its CMC (0.013 mol.l^{-1} at 25 °C) for the flotation of quartz.

The pH of the flotation pulp solution has an important effect on the adsorption behaviour of the amine collectors: as the pH of the bulk solution rises, there will be an increase in the concentration of the molecular species relative to the ionic species of the amines for all but the quaternary amines (see previous section and Figure 2.9). The CMC of the molecule is much lower than that of the ion because of the lack of repulsion in the micelle. Thus, less adsorption is required for the formation of hemicelles at the surface and furthermore, fewer cations are present as the solution pH rises. This behaviour is represented in Figure 2.11. For 12-carbon chain amines, it has been observed that a rapid drop in contact angle and flotation recovery occurs at pH values greater than pH=11 (Smith, 1988, p237).

4.1.4 Selection of specific collector type

It was decided to investigate the use of the **quaternary amines** as ash collectors for reverse flotation of coal, since the reagent pH dependence would be eliminated as a variable in the process. (This does not, however, imply that pH would not be a consideration in reverse flotation. The flotation pulp pH could affect, for example, the charge on the coal or mineral surfaces (zeta potentials) which would affect reagent adsorption and thus flotation performance.) The quaternary amines were also chosen on the basis of their commercial use. In addition to their surfactant properties, the quaternary amines are strong anti-bacterial agents and are used in this role both in the industrial and the pharmaceutical sector. This would make them cheaper as reagents than the primary, secondary and tertiary amines.

The choice of the specific quaternary amines to be used in the experimental work was based partly on hydrocarbon chain length considerations and partly on the requirements of laboratory adsorption experiments. Their availability from laboratory chemical suppliers was also considered.

The role of hydrocarbon chain length is important because an increase in chain length of the non-polar part of the surfactant increases its adsorption at the liquid-gas interface, thus generally enhancing the flotation of the solids to which the polar ends are attached. It was decided to use amines with as long a hydrocarbon chain as is practical.

In the adsorption tests[1] carried out in the laboratory to investigate the reactions of the quaternary amines with coal and its associated gangue, the (residual) concentration of the amines in solution was measured by UV spectroscopy. Thus it was important to select at least one quaternary amine with a suitable structure that would give rise to an absorbance in the UV spectrum.

Three amines were chosen for investigation. An amine with a 12-carbon chain length and two amines, each with a 16-carbon chain length, were selected:

Dodecyl-Trimethyl-Ammonium-Bromide (DTAB),
Hexadecyl-Trimethyl- Ammonium-Bromide (HTAB) and
Hexadecyl-Pyridinium-Chloride (HPYC).

Of these, HPYC is UV active.

Although not investigated in this thesis, there are many other factors governing the behaviour of the amines and thus the choice of a specific amine collector type for the reverse flotation of coal. These include the number of collector ions that can fit onto the surface of the mineral in a monolayer as well as previously mentioned factors such as the CMC. The aim of this thesis is, however, to demonstrate the reverse flotation process,

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[1] described in section 5 below

or to prove it, and not necessarily to optimize it, and for this purpose the choice of the above three amines was considered appropriate and adequate.

4.2 The coal surface: selection of a coal depressant

Coal is naturally hydrophobic and so for reverse flotation to be successful, the coal surface must be made hydrophilic. This can be achieved by adding a depressant. A suitable depressant for coal should have functional groups that exhibit a preferential attraction for the coal surface. Also, the depressant molecules should not have functional groups that compete with the collector for the surface of the ash-forming minerals to be floated.

Before selecting a suitable depressant to be used in the reverse coal flotation process, the coal surface should be examined.

4.2.1 Chemical structure of coal

The chemical structure of the organic material in coal is rather complex. It is a polymeric solid without repeating monomeric units, the basic structure being a graphite-like, aromatic/hydroaromatic system. The structure consists of three groups, namely: condensed aromatic lamellae, aliphatic and alicyclic interstitials, and bridge groups and heteroatomic interstitials and bridges.

A significant portion of the carbon in coal is contained in condensed aromatic lamellae (Tsai, 1982, pp 93–95). The lamellae become stacked to form larger, imperfect sheets or ring structures. These are buckled due to the presence of hydroaromatic or 5–membered rings. The ring structures are linked by bridges of aliphatic and alicyclic functional groups such as methylene and ethylene. This primarily aromatic hydrocarbon structure imparts a natural hydrophobicity to coal.

There are also heteroatoms present in coal, chiefly oxygen, sulphur and nitrogen. Generally, the polar oxygen functional groups such as methoxyl, carboxyl, carbonyl, hydroxyl, etheric and heterocyclic oxygen play an important role at the surface in terms of hydrophobicity. The greater the proportion of these groups, the less hydrophobic is the surface (Osborne, 1988). As the rank of the coal increases, the oxygen content of coal decreases, the percentage of carbon increases and the coal surface becomes more hydrophobic (Osborne, 1988).

In conventional (forward) flotation of coal, long chain aliphatic or aromatic hydrocarbon collector oils are used to enhance the natural hydrophobicity of coal. The hydrocarbon chains of these oils adsorb onto the hydrocarbon coal surface by van der Waals forces. This increases the solid–water free energy (increases the contact angle) and thus hydrophobicity is increased.

4.2.2 Depressants for coal

Water soluble polymers and polysaccharides such as dextrin (a starch derivative) and guar have been widely used as depressants in the flotation of mineral ores (Lin and Burdick, 1988). Dextrin has also been investigated as a coal depressant in the two-stage pyrite removal process mentioned above (see section 3.3). The hydroxyl groups on these molecules impart strong hydrophilicity but are not surface active like collector ions or molecules (Miller et al., 1983) and so are not likely to compete for the charged coal minerals sites.

A surfactant molecule can also be used as a depressant. Moudgil (1983) has investigated the depression of coal by polar polyacrylamide polymer molecules. The depressant behaviour was explained in terms of the adsorption of the hydrophilic molecules onto the coal which then render the coal surface polar in nature. One problem that may arise with using surfactant depressants in reverse coal flotation is that they will compete with the collector for the mineral surface. If however, the same molecule were used for both coal depression and ash collection, this problem might be avoided.

4.2.3 Selection of a specific coal depressant

The cationic amine surfactants identified above as ash collectors could also be used as depressants for coal. The hydrocarbon chain would adsorb onto the hydrophobic coal surface by van der Waals forces and thus render the surface hydrophilic because of the cation being orientated into the aqueous phase. Furthermore, a long hydrocarbon chain should enhance the adsorption on the hydrocarbon coal surface, thus improving the depressant action. Thus, it was decided **in the first instance** to investigate the use of the long chain quaternary amines selected above (section 4.1.4) as coal depressants as well as ash collectors.

As has been mentioned, many coals possess a strong (natural) hydrophobicity. Thus, it is possible that an additional coal depressant would be required either to enhance the depressant action of the amines or to provide complete depressant action should the amines prove unsuccessful for this purpose. Dextrin was chosen for investigation as an additional depressant. This substance has been used successfully to depress coal and, furthermore, it is unlikely to interfere with the collector action of the amines (see previous section).

4.3 Selection of suitable frother

Although the usual frothers, such as MIBC, are probably suitable for reverse flotation, it is known that long chain amine collectors have frothing properties as well as their collector behaviour. It seems likely, then, that the quaternary amines selected as ash collectors and coal depressants would also function as frothers in the reverse flotation process. The quaternary amines were thus selected for investigation as **ash collector, coal depressant** and **frother** in the reverse flotation of coal process.

If the frothing behaviour of the amines proved unsatisfactory, then traditional frothers such as MIBC or DIBK would be additionally employed.

4.4 Choice of reagent suite

In summary, the reagent suite selected for investigation of the new reverse coal flotation process was as follows:

- In the first instance, the three quaternary amines

  dodecyl-trimethyl-ammonium bromide (DTAB),
  hexadecyl-trimethyl-ammonium bromide (HTAB),
  and hexadecyl-pyrididium chloride (HPYC)

  would be investigated for use as **ash collectors, coal depressants and frothers.**

- The use of dextrin as an additional/alternative coal **depressant** would be examined.

- MIBC or DIBK would be used as additional frothers if required.

4.5 Previous work

Some preliminary work on reverse flotation of coal was already undertaken in the Department of Chemical Engineering at U.C.T. as an undergraduate project (Deetleefs and Johnson, 1986). This project investigated the feasibility of reverse flotation on a sample of coal from a Witbank colliery. Experiments were carried out in a laboratory batch cell. A number of short to medium carbon chain length (6-8 carbons) amines were investigated as gangue collectors in combination with dextrin as an organic coal depressant and MIBC as the frothing agent. The effects of various parameters on flotation were investigated, such as reagent concentration, pH, pulp density and the use of an anti-sliming agent.

The results were generally not encouraging. No significant beneficiation occurred in any of the tests and the effects of most of the parameters were negligible. However, closer examination suggests that the collector type that was selected was not optimal. All the amines used were of the molecular (insoluble) form, rather than of the salt or surfactant form (see section 4.1.2) and had short chain lengths (less than 8 carbon atoms). The choice of the reagents described above is thought to be more appropriate for the application.

It may be concluded that reverse flotation was not adequately investigated in this study and that the process still merits in-depth investigation.

5. Laboratory adsorption tests

The quaternary cationic amines were selected for the dual role of ash collector and coal depressant on account of their molecular structure. It was thought that the cation on the amine molecule would be attracted to the negative gangue surface and would act as a collector, while the hydrocarbon chain of the molecule would adsorb onto the hydrophobic coal surface and act as a depressant. In order to check the validity of these premises, the adsorption behaviour of quaternary amines on coal and gangue surfaces was investigated. In addition, the effect of pH was checked for subsequent flotation testwork.

Of the three amines selected for investigation of reverse flotation, only HPYC was used in the adsorption studies because the presence of the pyridine ring in the molecule gives rise to a peak in the UV spectrum. This makes possible the analysis of the amine concentration in solution by measurement of UV absorbance. HTAB and DTAB are straight-chain compounds and do not show UV activity.

So as to obtain unambiguous information, it was decided to study the adsorption of the amines onto separate samples of "pure" organic coal and "pure" gangue material. Pure, organic coal is hard to define and, in any case, would be impossible to obtain in practice. A low-ash, washed coal product from a colliery was used as a substitute. The ideal gangue material to use in the study would be a sample of pure kaolinite, since this is the dominant mineral in South African coals. A kaolinite sample was available (and used in flotation tests — see Chapter 3) but its particle size distribution was 50% $-2\mu m$ and a coarser sample could not be obtained. This size fraction was found to be unsuitable for use with the adsorption equipment (see below). It was decided to use a sample of pure quartz, which could be obtained at a much coarser size, instead. Although quartz is not the dominant mineral in South African coals, a quartz particle, like a kaolinite particle, would possess a negative surface charge above its PZC (see section 4.1.1), and thus it was thought that the amine adsorption on quartz would give a good indication of the behaviour on kaolinite. [1]

In addition to the use of the amines as ash collectors and coal depressants, the use of dextrin as a coal depressant has also been proposed. Thus it was decided to investigate the adsorption behaviour of dextrin (on the pure coal) and its interaction with the amine (HPYC). Dextrin adsorption could not be measured directly by UV spectroscopy, but its behaviour was inferred by observing the adsorption of HPYC in the presence and absence of dextrin. The adsorption of the HPYC on the separate samples was measured as a function of time as well as of the pH of the bulk pulp solution.

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[1] To some extent this is an oversimpification, because the structures of quartz and kaolin are different, and the kaolinite surface may posess positive charges under certain conditions (see section 4.1.1). This would adversely affect the cationic amine adsorption on the (cationic) kaolinite surface. However, as has already been discussed, the anionic characteritic of kaolinite is expected to predominate, and thus quartz should be a reasonable substitute gangue mineral for kaolinite in the artificial mixtures.

5.1 Experimental samples and apparatus

5.1.1 Feed samples

5.1.1.1 Coal

The coal used in the adsorption testwork was a high volatile, bituminous B rank blend–coking coal from the Witbank No.2 seam, obtained as low ash coal (LAC) product from the Landau Colliery near Witbank. Over the duration of the entire study, two bulk samples had to be obtained from the colliery at different times; the first contained 4.4% ash, and the second contained 9.2% ash. Most of the batch flotation testwork (Chapter 3) was performed on the 4.4% ash sample, but a number of experiments were performed on the 9.2% sample. The adsorption tests were performed on either of the two samples (as indicated below in section 5.2.2) so that the results from the adsorption tests could be related with confidence to the results of the flotation tests.

The coal was received as a −10 mm sample from the colliery. The +5.6 mm material was removed by sieving and was discarded. The −5.6 mm fraction was milled to obtain the desired size fraction for the adsorption experiments (−0.15 mm).

5.1.1.2 Gangue

The quartz used was a sample of "Delmas Silica" and was supplied already milled and packaged into 1 kg bags by MINTEK (Council for Mineral Technology) in Johannesburg. The size distribution of the sample was 98% −0.150 mm; 70% −0.075 mm, making it very similar to the coal.

5.1.2 Milling equipment

The mill used for grinding the coal samples was a laboratory stainless steel rod mill, with a diameter of 31.6 cm. The critical speed was calculated as 75.2 rpm, using the formula (Perry and Chiltern, 1973):

$$N_c = 76.65D^{-0.5} \quad \text{[2.7]}$$

The mill was operated at 90% of critical speed (67 rpm).

5.1.3 Reagents

The HPYC was a 98% pure laboratory grade reagent obtained from the Aldrich Chemical Company.

The sample of dextrin used was a 90% water soluble, laboratory grade, "Type IV" potato dextrin obtained from the Sigma Chemical Company.

Hydrochloric acid was used to modify the pH of the pulp solution in some of the adsorption experiments. This acid was chosen specifically so as not to add any additional ionic species to the solution (the Cl^- ion is common to both the acid and the amine).

The acid used was 32% BDH AnalaR®, analytical grade HCl which was diluted to 1/10 strength using distilled water.

5.1.4 Adsorption apparatus

Adsorption experiments were carried out in a stirred, 1–litre glass vessel immersed in a temperature controlled water bath. The apparatus is illustrated in Figure 2.12. The vessel was fitted with a pH probe and a temperature probe connected to a Hama model HI 8417 digital pH meter.

A small sintered disk filter was immersed into the solution in the vessel and connected to a Watson Marlow 503S peristaltic pump using narrow bore Tygon® tubing in order to remove small samples of the pulp solution for analysis. The filtrate was discharged into 4 ml plastic tubes situated equidistantly on the circumference of a specially constructed rotating disk. The disk consisted of a 40 cm diameter perspex platter fitted to a notched hub which was made to rotate under gravity with a weighted string wound around the hub and passing over a small pulley. An electronic timer device actuated a solenoid plunger which engaged or released against notches in the hub. In this way the disk was able to rotate in discrete steps every 5 s or 10 s (depending on the timer setting) so that a full tube was moved from under the discharge pipe of the pump and an empty tube replaced it. The pump flowrate was set to just fill a sample tube in the 5 s or 10 s time interval.

In order to obtain the flowrate required for the filtrate removal, a Pyrex® sintered disk of porosity 3 had to be selected for the filter bulb. The pore size of this disk is 15 to 40 μm.

5.2 Experimental method

5.2.1 Sample preparation

The coal used in the testwork was obtained as a 200 kg bulk sample from the colliery. The sample was supplied wet and, before milling, a sub-sample of approximately 10 kg was removed from the drum and air dried overnight on a large table. The +5.6 mm fraction was then removed by sieving and the −5.6 mm material was divided into approximately 1 kg samples for milling in the rod mill.

Milling was carried out batchwise: 1 kg of coal was milled at a time, using 20 rods. All milling was done dry. The milling time for the −5.6 mm coal to reach 98% −0.150 mm was 20 minutes (see Appendix II for the milling times). Milling was timed manually, using a stopwatch.

The quartz sample was obtained already milled and pre-packaged into 1 kg samples, so no milling was performed on this material.

In order to reduce the tendency for clogging in the sintered glass filter (which had a maximum pore size of 40 μm), the −0.075 mm size fraction was removed from both the coal and the quartz samples by wet sieving. The samples were then filtered and dried overnight in an oven at 80 °C.

After drying, both the coal and the quartz samples were (separately) divided by hand from a bulk sample (about 3 kg) into approximately 80 g samples for the adsorption experiments. These samples were then weighed on a two figure balance to the exact required mass for the adsorption experiments (75 g) and each sample was stored in a sealed plastic bag until required.

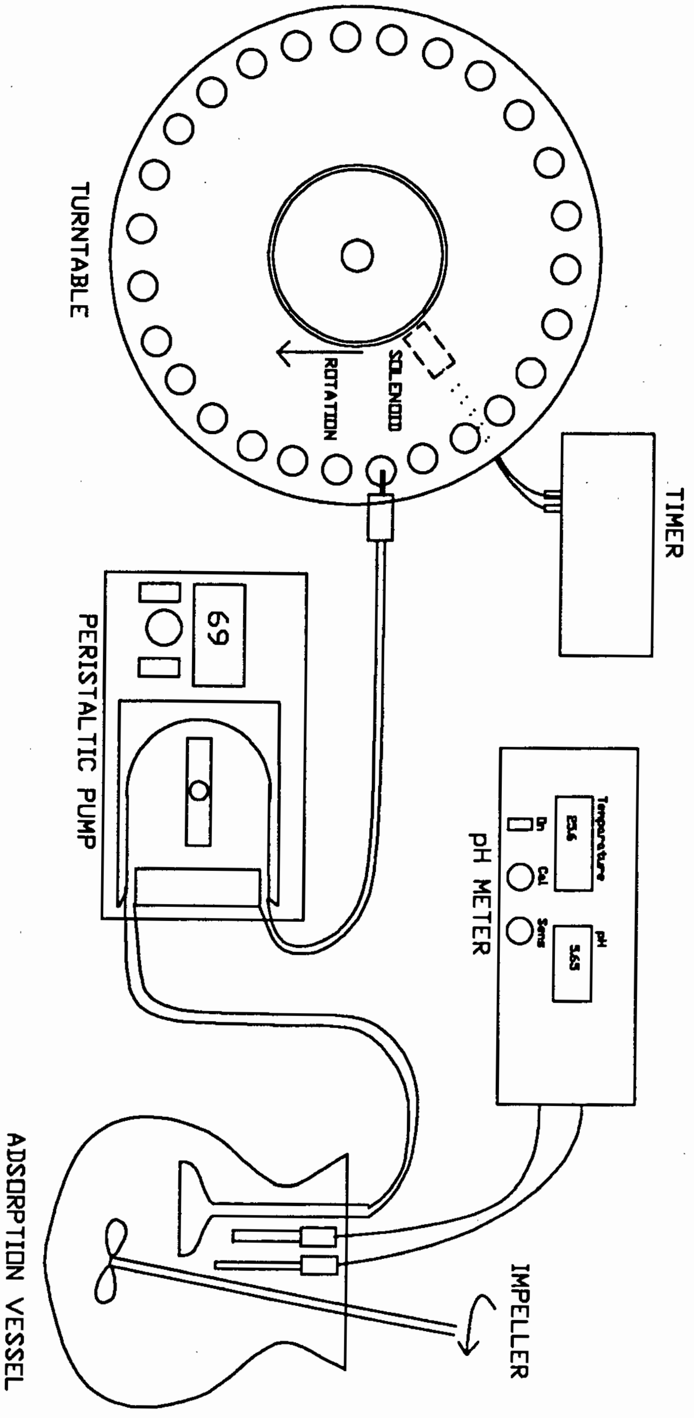

Figure 2.12: Diagram of experimental apparatus

5.2.2 Adsorption procedure

The adsorption work was divided into two parts:

- Part 1: Investigation of HPYC adsorption density on coal and quartz (separately) as a function of time and pulp pH.
- Part 2: Investigation of HPYC adsorption behaviour on coal with the use of dextrin as an additional coal depressant.

All runs were conducted at 25 °C at a pulp density of 10% solids. The pulp was agitated at 500 rpm. In some of the experiments, dilute HCl was added dropwise, from a burette, to obtain pH values ranging from pH = 7.6 (pH of Cape Town tap water) down to pH=2.5.

For part 1, runs were performed as follows. The reaction vessel was filled with 750 ml of tap water using a measuring cylinder. The stirrer and baffle, the temperature and pH probes, and the filter and tubing were then fitted, using a retort stand to secure them all. The agitation rate was set to 500 rpm and the water was allowed to reach the water bath temperature (usually about 20 minutes). The pH of the water was set to the desired value by adding HCl and checking the pH on the pH meter.

An exact mass of (crystalline) HPYC was weighed out on a four–figure Mettler AE50 electronic balance to make a known initial concentration of approximately 0.02 g/l for the quartz experiments and 0.025 g/l for the coal experiments (5.65 x 10^{-05} mol/l and 7.06 x 10^{-05} mol/l respectively). The HPYC was added to the water in the vessel and the solution allowed to recirculate by means of the pump for five minutes to ensure a uniform concentration throughout the entire vessel/filtration system. During this period, the pH was rechecked a number of times and (when necessary) HCl was added to restore the pH to the desired value.

At the end of the five minute period, the pump was adjusted to a flowrate so as just to fill the 4 ml tubes in the desired sampling period (either 5 or 10 s). The filtrate was then allowed to discharge into the sample tubes. The turntable timer was activated and the pre-weighed coal or quartz sample (75 g) was added to the vessel as quickly as possible. This was taken as time zero. During the adsorption run, the pH was checked and adjusted periodically to ensure the pulp pH remained at the desired value throughout the run.

The coal used in these experiments was the sample containing 4.4% ash.

For part 2, two types of runs were performed. The first type was identical to part 1, except that the requisite amount of dextrin was added along with the amine in the reaction vessel. The dextrin concentration was 500 g/t (based on the dry coal mass).

In the second type, the amine was injected into the reaction vessel which already contained the coal in suspension. The requisite mass of (crystalline) amine was placed in a 10 ml glass syringe, diluting water was added and the resulting solution was then injected into the pulp. The dextrin was added in two ways: it was either conditioned with the coal for five minutes prior to amine addition (with filtrate recirculation), or was added simultaneously with the amine by means of the syringe. The run was performed by starting the turntable and injecting the solution at the beginning of a new sample period, usually after 2 or 3 tubes had been filled. This was taken as time zero.

The coal used in these experiments was the sample containing 9.2% ash.

5.2.3 Analysis of samples

The adsorption of the amine onto the solids was determined by measuring the residual amine concentration in the filtrate samples by UV absorbance using a Varian UV Spectrophotometer. The HPYC molecule gives rise to a peak at 258.8 nm which was determined experimentally (see Appendix III).

The adsorption of dextrin could not be measured by UV spectrophotometry. Although there is an absorbance of the molecule in the UV spectrum, the intensity was found to be too small to be useful at the dextrin concentration used in the adsorption experiments. Its behaviour and interaction with the amine adsorption could, however, be inferred from the HPYC adsorption behaviour.

5.2.4 Representation of data

Collecting a sample for a fixed period of 5 or 10 s meant that the measured concentration of the sample represented a time average. Although not strictly correct, this measured adsorption value was taken to be the concentration of the solution at half the time interval. For each 4 ml sample to be time representative of the bulk solution, two factors had to be considered: Firstly that the bulk pulp solution had to be well mixed to ensure a uniform amine concentration at all times throughout the reaction vessel, and secondly that the flow behaviour in the filter/tube system had to approximate plug flow (minimum mixing).

In order to satisfy the first condition, the pulp agitation rate was set to 500 rpm and a baffle inserted into the reaction vessel to improve the mixing and to avoid a "whirlpool" effect which might entrain air into the solution. In order to satisfy the second condition, the following steps were taken: the bulb of the filter disk was specially constructed so be as small as possible and so minimize holdup, and the total tube length was kept as short as was practical. The flow behaviour was then checked by injecting amine into the reaction vessel with no coal added. In this way, a step change in the concentration in the vessel was made and the response was measured by collecting solution as described in the section on adsorption procedure. Figure 2.13 is a residence-time distribution (RTD) plot of these tests (the data and calculations can be found in Appendix III). As can be seen from the graph, the behaviour of the response (given by the "J(theta)" curve on the graph) is not ideal plug-flow, but is similar to the model for segregated flow in a tubular reactor (Smith, 1984; Levenspiel, 1979). In this model (given by the "F-curve"), the flow is assumed to be laminar; dispersion due to molecular diffusion is neglected and mixing occurs only as a result of the velocity profile in the tube. Although this is not the ideal situation and the obtained data does not represent accurate values, it can, however, be used quite adequately for comparison of the adsorption behaviour of different runs.

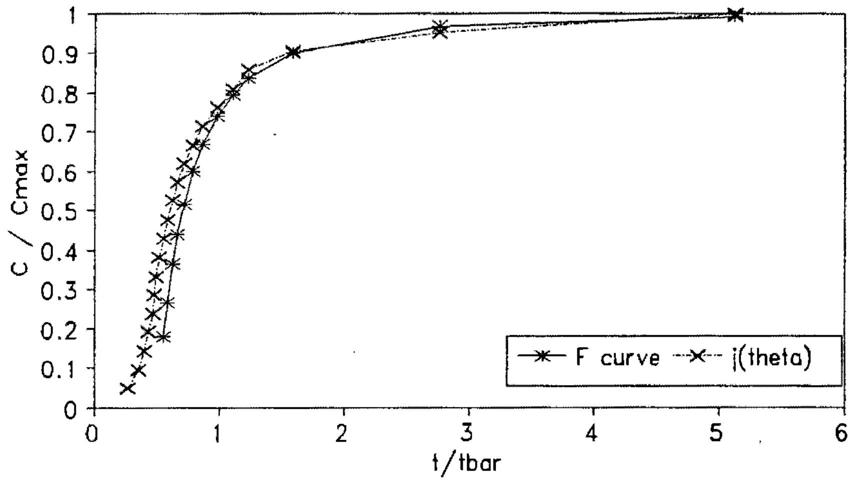

Figure 2.13: RTD analysis of adsorption apparatus
(step input test)

5.3 Discussion of results

5.3.1 Part 1: HPYC adsorption on coal and quartz

The first set of runs was performed with HPYC on quartz. The initial HPYC concentration was 0.02 g/l and the pH was varied in the range 2.5 to 6. The adsorption was measured for a period of 60 s. The second set of experiments was performed with HPYC on coal. The initial concentration of HPYC was 0.025 g/l and the pH was varied from 2.5 to 7.6 (the pH of Cape Town tap water). In these experiments, the adsorption was measured for a period of 100 s. The detailed results of all these experiments can be found in Appendix IV.

Figure 2.14 shows the extent of adsorption of the HPYC onto quartz at various pulp pHs, while Figure 2.15 shows the situation for coal. What is immediately obvious is that adsorption occurs both on the coal and on quartz (as expected). For the same size fraction and same mass of feed, the adsorption density on coal is greater than on quartz. However, it must be remembered that quartz has a density greater than that of coal, so for the same mass of solids, the coal has more surface area for adsorption because of the greater number of particles. Also, coal has a porous structure and this might also account for the greater adsorption of the amine.

As can be seen from Figure 2.14, the adsorption of HPYC on the quartz is strongly pH dependent. This suggests an electrostatic adsorption mechanism which may be explained as follows: as the pulp becomes more acidic, the increased concentration of H^+ ions reduces the negative zeta potential on the surface of the quartz particles and even reverses it (Smith, 1963). When this happens, the tendency for the cation of the amine to adsorb on the quartz becomes less. This is illustrated very clearly by Figure 2.14: as the pulp pH decreased, the adsorption density at any given time was reduced and below pH=3.0, almost no more amine was adsorbed.

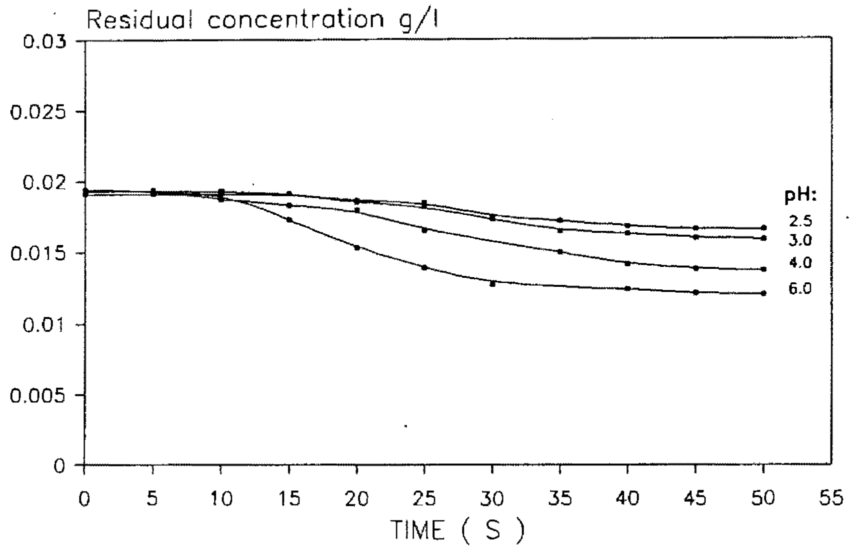

Figure 2.14: HPYC adsorption on quartz as a function of pH.

Over the range pH=3.0 to pH=6.0, it was observed at the end of the adsorption runs that the entire mass of quartz was suspended as a foamy aggregate on the surface of the pulp in the adsorption vessel. Below pH=3.0, however, most of the quartz remained in the pulp phase. This would indicate that the quartz surface would not be sufficiently hydrophobic for flotation purposes if the pulp pH were below pH=3.0. Thus, for reverse flotation purposes it would be essential to keep the pulp pH above pH=3.0 to ensure sufficient collector adsorption on the quartz.

The trends observed here are consistent with those found by other researchers. For example, Smith (1963) investigated the contact angle on quartz as a function of pH using dodecylamine (a primary amine) as a collector. He found that the contact angle was a fairly strong function of pulp pH, and that it dropped off sharply below pH=3.5. Since the contact angle is dependent on collector adsorption, these results are an indication of the adsorption density of the collector, i.e. that the adsorption density is a function of pH.

As can be seen from Figure 2.15, the amine adsorption density on the coal is pH dependent above pH=4.0: the adsorption density increased as the pulp became less acidic. Below pH=4.0, adsorption still occurred but was independent of pH. This suggests that the mechanism for adsorption of amines below pH=4.0 is one which is not affected by ionic attraction, i.e. that it is one of hydrophobic adsorption (by van der Waals forces) of the hydrocarbon chain of the amine on the coal surface. At low pH, hydronium ions are likely to replace the amine cations in the double layer on the coal surface and the hydrophobic mechanism of the amine is favoured. Hydrophobic adsorption would result in the cation of the amine molecules being directed into the aqueous phase and thus the coal surface would be wetted, i.e. the coal particles would be depressed..

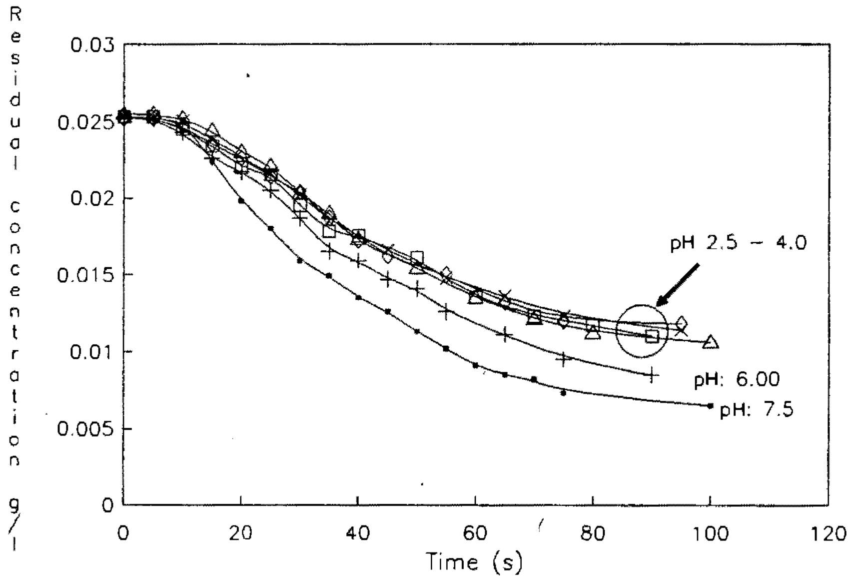

Figure 2.15: HPYC adsorption on coal as a function of pH

Another explanation is that the "adsorption" that is observed might actually be the result of diffusion into pores on the coal surface (some coals are highly porous).

Above pH=4.0 the increase in the HPYC adsorption is probably the result of electrostatic attraction between cations on the amine molecules, and negative charges on the coal surface. The nature of the charge on the surface of coal is indicated by the zeta potential of the coal. Figure 2.16 shows zeta potential data for various coal types as a function of pH (data of Onlin and Aplan; from Aplan, 1988, p103). It can be seen that a particular coal has a negative zeta potential above its PZC pH. A cationic collector molecule would be expected to adsorb onto the coal surface above the PZC. Figure 2.16 shows that a high-volatile bituminous coal or "HVB" coal, such as the LAC coal used in the adsorption experiments, would have a PZC pH at pH=4.0 (see Figure 2.5). This value is in good agreement with the observed adsorption behaviour, and provides evidence that the HPYC did, in fact, adsorb electrostatically above pH=4.0 (due to the LAC having a negative zeta potential).

In reverse flotation, this adsorption would be undesirable, since it might result in the coal being collected (instead of being depressed).[1] It would appear, then , that the pulp pH of reverse flotation should be kept below pH=4.0 so that amine adsorbs only hydrophobically, and does not act as a coal collector. Alternatively, one could use an additional coal depressant (dextrin) which would adsorb onto the coal in preference to the amine and thus prevent any amine collector action on the coal. This would also obviate the need to adjust the pH.

5.3.2 Part 2: addition of dextrin

The first set of experiments was performed with dextrin together with HPYC on coal. The initial dextrin concentration of the dextrin was 0.05 g/l (500 g/t, based on the coal mass) and the HPYC 0.018 g/l. In all of the runs, the pH was kept constant at 3.4. The adsorption was measured for a period of 100 s as before. The detailed results can be found in Appendix IV.

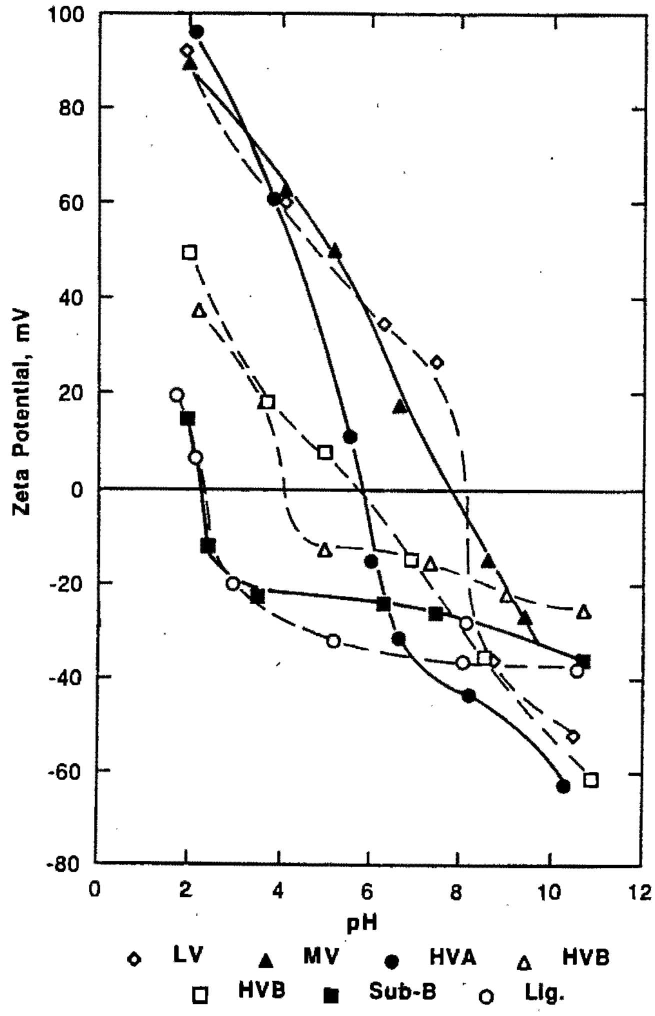

Figure 2.16: Zeta potential and point-of-zero charge (PZC) of coals of various ranks (data of Onlin and Aplan; Aplan, 1988).

Figure 2.17 shows the adsorption of HPYC onto coal in the presence and absence of dextrin. In these runs, coal was added to the reaction vessel which already contained the amine and dextrin. The pH was kept constant in all the runs at pH=3.4, in order to prevent any additional adsorption on amine as found in Part 1 (section 5.3.1 above).

As can be seen, very little change in the adsorption density of the amine occurred when dextrin was added compared to when no dextrin was present. This is contrary to what one would expect: the dextrin should adsorb onto the coal simultaneously with the amine and thereby reduce the amine adsorption. The possibility exists that the amine adsorbs more readily onto the coal than does the dextrin, which is then not able to adsorb onto

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[1] Cationic amines are sometimes used as collector promotors for oxidised coals, i.e. coals which have a significant negative surface charge (Mishra, 1987).

the coal surface already coated with amine. If the dextrin were allowed to adsorb onto the coal prior to the amine, this situation might be avoided.

This theory was tested by altering the adsorption experiments so that the dextrin was conditioned with the coal in the reaction vessel before adding the HPYC. Instead of adding coal to the solution at time zero, the HPYC was injected into a coal pulp and this was taken as time zero. Three types of runs were performed: a blank run without dextrin, simultaneous addition of dextrin and HPYC, and pre-conditioning of the coal with dextrin. In this way, the three situations could be compared directly.

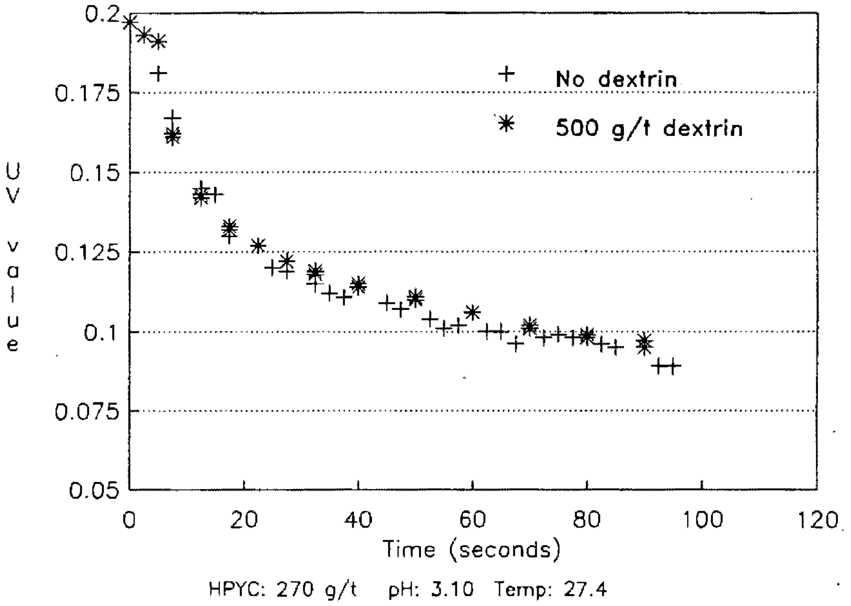

Figure 2.17: Adsorption of HPYC on coal in the presence and absence of dextrin.

In these experiments, the conditions were identical to the preceding set, except that the adsorption was measured for a longer period of 140 s. The results can be found in Appendix IV. Figure 2.18 shows the results of these runs. It is clear from these curves that the HPYC adsorbs less readily onto the coal when there is prior conditioning of the coal with dextrin. Where the HPYC is added simultaneously, the adsorption is only marginally less than for the case where no dextrin was added. This means, in turn, that the dextrin is able to adsorb to a greater extent if it is allowed to come into contact with the coal prior to the amine. It does, indeed, seem that the amine adsorbs more readily onto coal than dextrin and thus prevents effective dextrin adsorption. When dextrin is allowed to adsorb first the amine, once added, does not seem to be able to displace the adsorbed dextrin. If dextrin were to be used as a coal depressant in conjunction with the quaternary amines as ash collectors, it would seem to be essential to first condition the flotation pulp with dextrin before collector is added. This will be tested in the flotation experiments in Chapter 3.

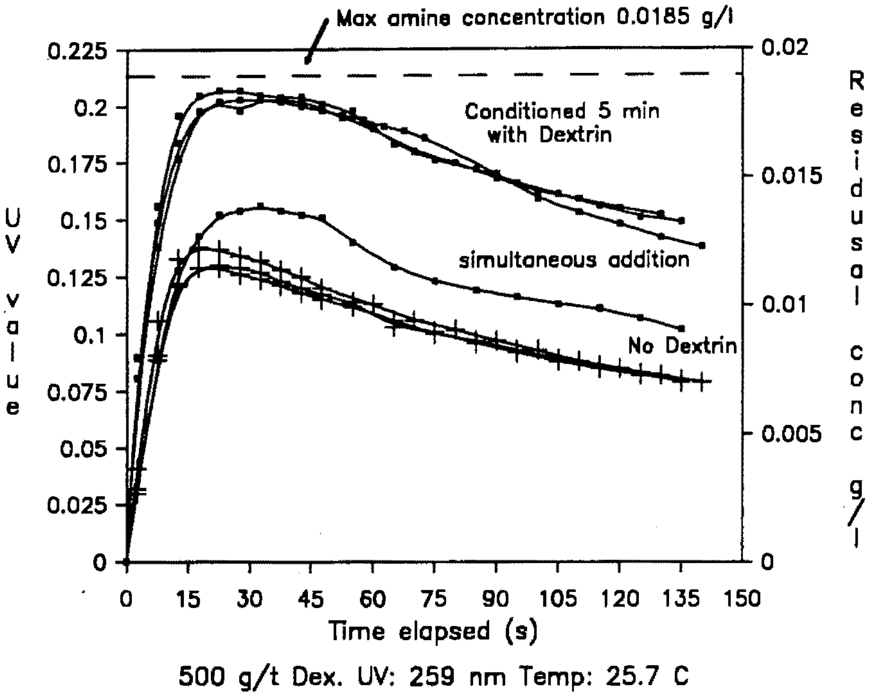

Figure 2.18: Effect of pre-conditioning with dextrin.

6. Summary and Conclusions

Reverse coal flotation is a novel process which has been proposed to address three problems occurring in the conventional, forward flotation of coal, namely: high mass recovery, slimes entrainment and the variability of floatability of South African coals. The purpose of this chapter was to identify a reagent suite for reverse flotation and to make a preliminary assessment of the suitability of the reagents by experimental laboratory adsorption studies. The findings are as follows:

1. A literature survey identified a number of ways of improving flotation performance. These included alterations to flotation circuits and the use of specialized reagents. Reverse flotation has been proposed as a fundamentally different process from all the existing methods.

2. The structures of coal and its associated mineral matter were considered in order to select suitable reagents for reverse flotation. It was found that a cationic-type collector would be required to float the naturally hydrophilic coal mineral matter; a depressant would be required to suppress the natural floatability of coal; and a frother would be required to ensure froth phase stability.

3. A reagent suite for reverse flotation was proposed, namely the use of quaternary amine salts to act as ash collector, coal depressant and frother. Dextrin was proposed as an additional coal depressant and DIBK or MIBC as additional frothers, should they be required.

4. The adsorption behavior of the amines onto "pure" coal and "pure" gangue surfaces was investigated. The fact that adsorption occurred both coal and gangue was encouraging for their proposed use as collector and depressant.

5. It was inferred from amine adsorption dependence on pH that flotation tests should be conducted between pH=3 and pH=4 in order to eliminate any residual collector action on coal.

6. The use of dextrin as a coal depressant was examined and it was found that prior conditioning of coal with dextrin was necessary to ensure that the maximum adsorption occurred. Once the dextrin adsorbed, the amine was not able to dislodge the dextrin attachment to coal.

The adsorption tests have indicated that the reagent suite selected does adsorb onto coal and quartz and thus might well be suitable for the reverse flotation process. What must be determined, however, is whether this adsorption is sufficient to result in flotation of quartz and depression of the coal. This can only be determined by flotation experiments. The next step is thus to confirm the suitability of the reagent suite and also to establish optimal conditions by conducting flotation tests. This is the subject of the next chapter.

CHAPTER 3
CONVENTIONAL BATCH FLOTATION TESTWORK

1. Introduction

In Chapter 2 a reagent suite was proposed for the reverse coal flotation process. Long chain, quaternary amine salts were chosen as ash collectors, coal depressants and frothers, with dextrin as an additional coal depressant (if required). Preliminary studies (adsorption tests) confirmed the suitability of this reagent suite. This chapter presents the results of reverse flotation experiments performed in a laboratory batch scale flotation cell in order to establish the technical feasibility of the reverse coal flotation process.

Since little or no previous information exists on the use of quaternary amines as ash collectors and coal depressants, flotation tests were designed to give results that were as unambiguous as possible. For this reason, the flotation experiments were carried out on artificial mixtures composed of various proportions of free gangue material (quartz or kaolin) and low-ash, washed coal. These artificial mixtures simulate perfect liberation of coal and ash and allow the performance of the reagents to be established without the additional factor of the degree of feed liberation.

The objectives of this chapter are: to assess the suitability of the reagent suite for the reverse coal flotation process, by means of batch flotation testwork using artificial coal/gangue mixtures; to determine the optimal flotation procedure and operating conditions for the process; and to compare the performance of the process with that of conventional "forward" flotation.

In the flotation tests detailed below, the amines are investigated *in the first instance* as ash collectors, coal depressants and frothers. Subsequently, dextrin is investigated as an additional coal depressant. The chapter begins with a look at the samples used in the batch flotation tests. Details of the experimental equipment and procedures are presented. Thereafter the results of the testwork are presented and discussed.

2. Experimental feed samples, equipment and procedures

2.1 Flotation samples

The flotation tests reported in this chapter were all performed on artificial feed mixtures composed of various proportions of quartz or kaolin and low-ash, washed coal. The sections which follow describe the components of these artificial mixtures.

2.1.1 Coal

Three samples of "pure" coal were used in making up the artificial mixtures used in the flotation testwork. All the samples were high volatile bituminous B rank blend coking coals from the Witbank No.2 seam, obtained from the same colliery. Unfortunately they varied in ash content; the samples contained 4.4%, 7.2% and 9.2% ash, respectively. This variation in ash content, though undesirable, was unavoidable. It was also not possible to blend the samples in order to obtain a uniform ash content for all the flotation work since they were obtained at different times. However, within a particular set of flotation runs, the same coal sample was used.

The samples were all received as −10 mm washed coal ("low ash coal" or LAC) product from the Landau Colliery. One of the samples (4.4% ash) was the same as that used in the adsorption experiments, described in Chapter 2. As with the adsorption experiments, the +5.6 mm fraction of each sample was removed by sieving and was discarded; the −5.6 mm fractions were milled to 98% −0.15 mm. Unlike the adsorption tests, however, the −0.075 mm fraction of the sample was not removed prior to flotation, the entire −0.15 mm milled sample was used.

2.1.2 Gangue

The gangue materials used in the artificial mixtures were samples of pure quartz and pure kaolin.

The quartz sample was the same as that used in the adsorption experiments, described in Chapter 2 (see section 5.1.1.2). Although not the dominant mineral in South African coals, the quartz was used in most of the flotation experiments involving artificial mixtures because the size fraction was similar to that of the coal. As explained in Chapter 2, the surface properties of quartz are similar to those of kaolin, and the results obtained are expected to be representative of those that would be obtained with kaolin.

The kaolin was obtained from the nearby Serina Mine in Fish Hoek as a 98% (–0.015 mm; 50% –0.002 mm sample. Usually, such fine material is undesirable for flotation (the process is inefficient for such small particle sizes), but a coarser sample could not be obtained. Notwithstanding, the sample was used in some of the experiments because kaolin is the dominant mineral in South African coals and thus its flotation behaviour with respect to reverse flotation needed to be assessed.

2.2 Flotation equipment

The flotation cell used in the batch testwork was a 3–litre laboratory cell based on the "open top" Leeds cell design which has been shown to give highly reproducible results (Dell and Bunyard, 1972; Fickling, 1986). The cell is depicted in Figure 3.1 below.

The cell was a "bottom driven" type in which the impeller is driven from the base of the cell. The variable speed impeller motor was fitted with a speed controller. The pulp level was controlled by a constant head device. This gives more stable control than the standard diaphragm level controller (Fickling, 1986). The air flow rate was set by means of a pressure regulator and a rotameter. Froth was removed by manual scraping and reproducibility was maintained by removing concentrates at set intervals.

The cell was supplied with filtered municipal tap water (pH 7.6). A rinse pipe allowed material adhering to the cell walls at the end of a run to be washed into the tailings, which were drained into a bucket situated under the cell.

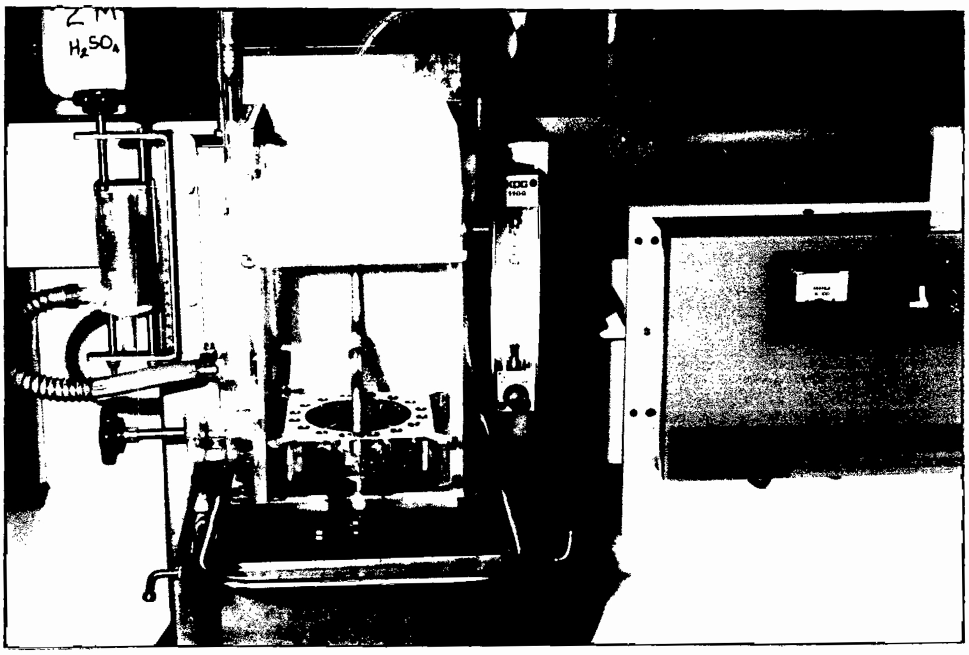

Figure 3.1: The flotation cell used in the batch flotation testwork.

2.3 Milling equipment

The mill used for preparing the coal samples is the same as that described in Chapter 2. The same milling conditions as described in Chapter 2 (section 5.2.1) were used to obtain 98% −0.150 mm samples for flotation.

2.4 Reagents

The amines used in the flotation tests were those selected in Chapter 2; namely hexadecyl-pyridinium chloride (HPYC), hexadecyl-trimethyl-ammonium bromide (HTAB), and dodecyl-trimethyl-ammonium bromide (DTAB). All were obtained as 98% pure laboratory grade reagents from the Aldrich Chemical Company.

For the forward flotation tests, a commercial paraffin collector oil was used, namely "Shellsol A", which was obtained from the Shell Chemical Co (South Africa). This collector is a 165–185 °C distillate, and has an aromatic content of >95% (by volume). The frother used was MIBC (methyl isobutyl carbinol) which is very commonly used in conventional coal flotation.

In some of the experiments HCl was used to modify the pulp pH. The acid was the same as used in the adsorption experiments.

The dextrin sample used was the Type IV, 90% water soluble sample used in the adsorption experiments.

2.5 Experimental method

2.5.1 Sample preparation

The coal used to make up the artificial mixtures was milled in batches of four, 1 kg samples. After milling, the samples were homogenized and then re-divided into approximately 250 g sub-samples using a rotary splitter.

The quartz material was obtained pre-packed in 1 kg (dry) samples. As with the coal, sets of four samples were blended and then re-divided into 250 g sub–samples.

The kaolin was obtained as a dry bulk sample. For each batch of artificial mixtures, a 2 kg sub-sample was removed and divided in a splitter into approximately 100 g samples.

The total mass of solids used in each flotation run was 300 g (except were otherwise indicated), which gave a pulp density of approximately 10% when added to three litres of water. For each artificial feed, coal and gangue were combined in the correct proportion to give a sample with the required gangue fraction. For example, 25% quartz/coal feed samples were prepared by adding 75 g of quartz and 225 g coal (LAC) to small plastic bags which were then filled with (tap) water and sealed to prevent oxidation until the samples were used. Other quartz/coal feeds were made by combining different mass proportions of quartz and coal, depending on the ratio required.

The same procedure was followed for the kaolin/coal feeds (except that kaolin was used instead of the quartz).

2.5.2 Flotation procedure

Flotation experiments were carried out according to a strict procedure to ensure that reproducibility was maintained throughout the testwork.

2.5.2.1 Cell parameters

For all the runs, both reverse and forward flotation, the aeration rate was set at 4 l/min and the impeller speed at 1200 rpm. Unless otherwise indicated, the pH of all the flotation runs was that of the tap water (pH=7.6).

2.5.2.2 Reverse flotation runs

The flotation method was as follows:

Approximately 0.5 l of water was introduced into the flotation cell. The feed solids were added and the slurry agitated at 1200 rpm for two minutes. The cell was then filled to the desired pulp level height according to the setting on the constant head device. For these experiments, the pulp level was set to obtain a froth height of 2.5 cm.

The pulp was allowed to agitate for a further five minutes after which the amine was added. The required amount of (crystalline) amine reagent was weighed out on a four-figure Mettler electronic laboratory balance and dissolved in approximately 50 ml water. This was then added to the cell and a period of four minutes conditioning time was allowed before aeration commenced. The amines were also used as frothers, so no additional frother was added.

Aeration was commenced by opening the air valve to obtain the desired flow rate, and five seconds later concentrate collection was begun. Concentrates were collected over fixed time intervals in numbered, pre-weighed dishes. The concentrate in each dish thus corresponded to a specific time period and sequence in the flotation experiment. The froth concentrate was manually scraped into each dish and scraping was performed at fixed intervals within each concentrate collection period to ensure reproducibility.

2.5.2.3 Forward flotation runs

The procedure was the same as for the reverse flotation runs except in the following respects:

After the feed sample had been pre-conditioned in the flotation cell, the required volume of collector oil, Shellsol A, was added with a "Pipetteman" automatic pipette. A period of four minutes conditioning time was allowed, after which the frother, MIBC, was injected into the pulp with a micro-syringe. A further minute of conditioning time was allowed before aeration was commenced as before.

2.5.3 Analysis of samples

At the end of each flotation experiment, the sample from each dish was recovered by filtration and oven dried overnight. The mass of each individual concentrate sample was recorded and the ash content was determined according to the method outlined in Appendix V. The results were then entered into a "Quattro" program spreadsheet and the cumulative recoveries for gangue and coal were determined according to the calculations outlined in Appendix VI.

2.5.4 Reproducibility tests

Before any commencing reverse flotation test work, reproducibility runs were performed in order to test both operator and cell reproducibility. These reproducibility tests were conventional forward coal flotation experiments, performed according to the method described in section 2.5.2.3 above. The coal used was a South African r.o.m. coal, obtained as a –0.25 mm thickener underflow sample from the Rietspruit Colliery near Witbank (further details of this coal sample are given in Chapter 5, section 2). The coal collector used was Shellsol A at a dosage of 1000 g/t, and the frother was MIBC at a dosage of 12 μl/l.

The results are shown in Figure 3.2. As can be seen from the graph, the largest difference in coal recovery in four different runs (using identical conditions) is 1%. This result shows that the reproducibility is extremely good, in terms of both the repeatability of the method and the reliability of the flotation cell.

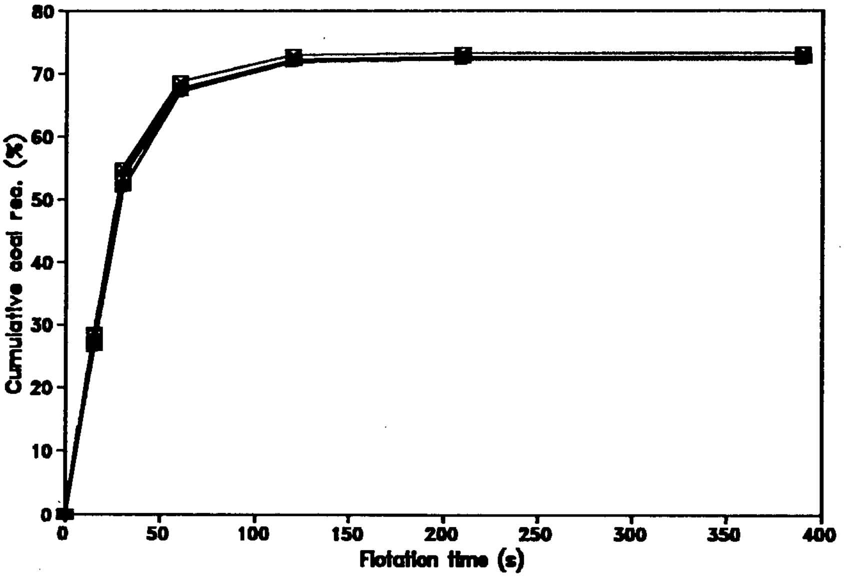

Figure 3.2: Results of reproducibility tests carried out on thickener underflow from the Rietspruit colliery (forward flotation).

3. Initial flotation tests

The aim of the batch flotation testwork in this chapter was to assess the suitability of the reagents selected for reverse flotation. The purpose of the initial experiments was to find the optimum concentration of each amine in terms of ash recovery, coal depression and frothing action. This value would probably differ from one amine to the next on account of differing solubilities, hydrocarbon chain length and other factors such as hemi-micelle formation and the CMC (see Chapter 2, section 4.1.3). Subsequent flotation tests would be aimed at investigating other factors influencing reverse flotation performance, such as pulp conditions, improved flotation methods and the use of additional reagents to improve reverse flotation performance.

3.1 Experimental details

In the initial flotation tests, the feed was a 50% quartz mixture, made up from quartz and the sample of LAC containing 7.2% ash. The coal and quartz were blended in an equal mass proportion and the resulting feed ash content was 54%. For each of the three amines, a number of reverse flotation runs were performed at different amine concentrations. The approximate ranges in which the concentration of each amine was varied are were follows:

DTAB — 150 to 800 g/t
HTAB — 250 to 1200 g/t
HPYC — 250 to 1200 g/t

For each run, the pulp density was 10%, the impeller speed 1200 rpm, and the aeration 4 l/min. The pH of the flotation pulp was that of tap water (pH=7.6) and was not modified. Details of each run and the results obtained are given in Appendix VII.

3.2 Results and discussion

The optimum flotation result for each of the three amines is reported in Table 3.1. Figure 3.3 shows the recovery/time trajectories for quartz and coal in the concentrates for each of the three reagents at the optimum concentration.

As can be seen, the amine DTAB gave the highest quartz recovery (98.1%) and so is the best collector. It also gave the best result in terms of grade; the ash content of the tails product was 11.1%. In terms of product (tails) coal recovery, however, HTAB gave the best result at 75.1%, and so is the best "depressant". The high quartz recoveries obtained with all three reagents indicate that the quaternary amines have excellent ash collecting ability.

Table 3.1: Initial reverse flotation results

Name	**Amine conc. (g/t)**	**Conc. quartz recov.(%)**	**Tails coal recovery (%)**	**Tails ash (%)**
DTAB	354	98.1	65.9	11.1
HTAB	595	94.0	75.1	14.0
HPYC	861	95.9	65.8	12.7

PULP DENSITY: 10% TAP WATER
FEED ASH: 54%

The frothing action of the amines was monitored visually during each run. Generally, too low a concentration did not produce a stable froth, while too much amine produced a froth which was too vigorous and gave rise to handling problems. At the optimum concentrations, however, all three amines were observed to produce very stable froths and so no additional frother was used.

In contrast to the collector and frother action, the coal depressant action of the quaternary amines was not found to be convincing. An ideal separation for the artificial mixtures would be 100% LAC recovery in the tails and 100% quartz recovery in the concentrates: however, at best, only 75.1% coal recovery was obtained in the tails which represents a substantial loss of product, i.e. too much coal reporting to the concentrate.

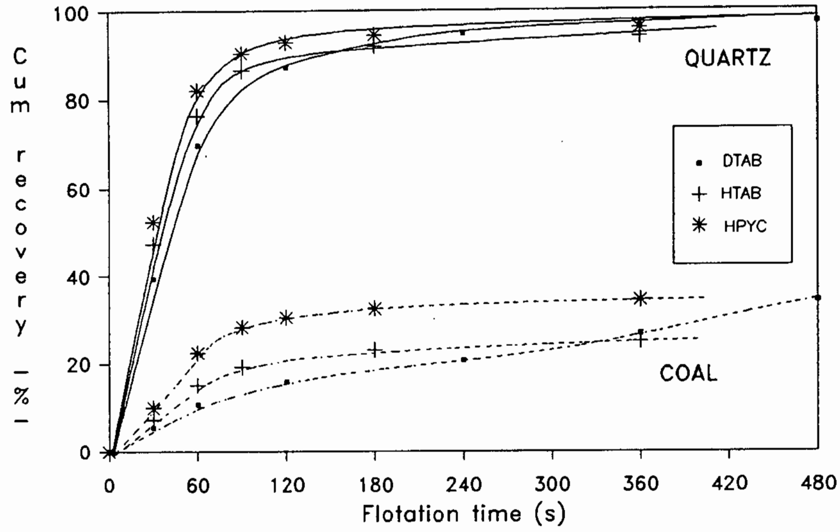

Figure 3.3: Initial reverse flotation results using HTAB, HPYC and DTAB.

The poor depression of coal could have been due to a number of causes. It is clear from the adsorption experiments (Chapter 2, section 5.3) that the amines do adsorb onto the coal but it is possible that this adsorption does not impart hydrophobicity as expected. A second possibility is that the coal was being entrained into the concentrates (in spite of amine adsorption), while a third possibility is that the loss was due to a combination of both of these factors. In an effort to improve the overall performance of the reverse coal flotation process, an investigation was carried out to determine which of these causes was responsible for the poor recovery of coal into the tailings.

4. Depression of coal studies

4.1 Flotation under acidic conditions

In Chapter 2 (section 5.3.1)it was seen that the amine adsorption onto "pure" coal (LAC) was a function of pH above pH=4.0, while below this pH, the adsorption was independent of pH. It was concluded from this that flotation at a pH below 4.0 would be the ideal condition in order to avoid any collector action by the amine on the coal and thus improve depression. A number of flotation experiments were carried out under acidic conditions to investigate whether this would improve the efficiency of beneficiation.

4.1.1 Experimental details

Batch reverse flotation experiments were carried out on the 50% quartz/coal mixtures at pH=3.4 and pH=7.6 (tap water). The HPYC amine was used in these tests, since this amine was used in the adsorption experiments in which the pH was varied (Chapter 2, section 5.3.1). Details of each experiment and the the results obtained can be found in Appendix VII.

For the flotation experiments at pH=3.4, the tap water supply to the cell was replaced by a tank supply. The tank water pH was modified to pH 3.4 by adding concentrated HCl and checking the resulting pH with a pH meter. A pH probe was fitted to the cell and the pH was constantly monitored during conditioning and flotation. It was found that the pH rose steadily as flotation progressed and dilute HCl was added dropwise into the cell (from a burette) when necessary to maintain the pH at 3.4.

4.1.2 Results and discussion

Figure 3.4 shows the recovery/time trajectories for two reverse flotation experiments using HPYC at a concentration of 810 g/t. The curves represent optimal flotation results at pH=3.4 and pH=7.6. It can be seen that although there was a slight improvement in coal depression, particularly in the first few concentrates, flotation under acidic pH conditions did not offer any significant benefit for reverse flotation.

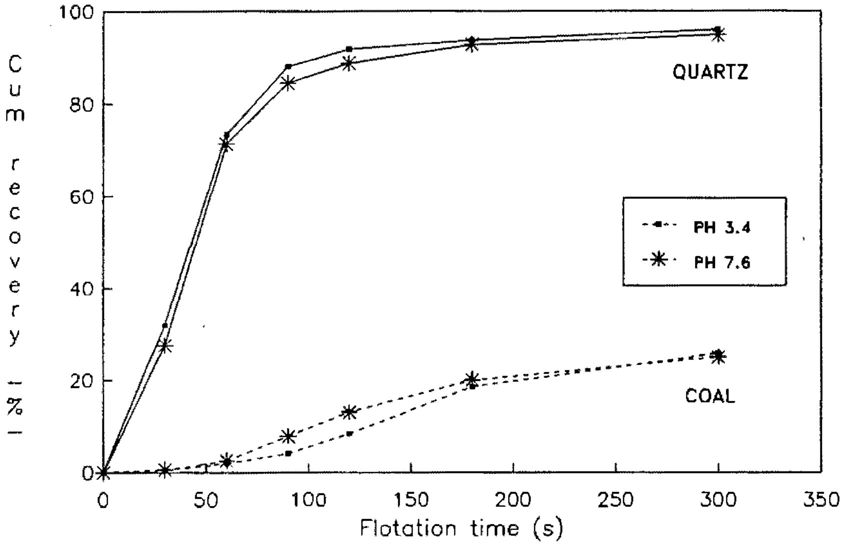

Figure 3.4: Effect of pH on coal and quartz recovery in reverse flotation.

This is contrary to the results of the adsorption experiments. It was seen in Chapter 2 that the pH of the pulp had a marked effect on the adsorption of HPYC on coal; but in these flotation tests, the pH of the pulp had virtually no effect on the coal recovery. Thus the depressant action was not improved by flotation at a pH below 4.0 as was indicated by the adsorption tests. There could be two reasons for this:

1. The amine might have a poor depressant action even though it has adsorbed onto the coal. The low pH would inhibit any collector action of the amine (onto the coal), but coal which was still partially hydrophobic (due to poor depressant action) might still become attached to air bubbles and be recovered in the concentrate.

2. The depressant action might be masked by coal entrainment. A a particular particle size, the low density coal particles might be more susceptible to entrainment than the mineral particles would be in forward flotation. The chemical depressant action of the amines might well be occurring as predicted, in the sense of the coal surface being made hydrophilic, but particles might still be entrained in spite of this.

To determine which one of these situations was actually occurring, further flotation experiments were performed.

4.2 Entrainment studies

The aim of these experiments was to distinguish coal entrainment from poor coal depression in reverse flotation. The strategy was as follows:

1. Separate samples of LAC and quartz were floated (using the three amine reagents at various concentrations) to investigate the specific flotation activity of the coal and the quartz without the one interfering with the other.

2. The "natural floatability" of the coal was determined by floating a separate sample of LAC using a "neutral" frother as the only flotation reagent, i.e. a frother with little or no collector ability. The difference between the coal's natural floatability and the recovery obtained with the amines was taken as an indication of whether or not the coal floatability was deactivated using the amines, i.e. whether the coal was being depressed.

3. The extent of coal entrainment in reverse flotation was investigated by carrying out reverse flotation experiments with artificial mixtures containing different proportions of coal and quartz, and examining the relationship between the coal recovery in the concentrates and the percentage quartz in the feed. The hypothesis here was that a higher proportion of quartz in the feed would mean a higher quartz mass flux into the concentrates and thus a higher water recovery which might entrain more coal particles into the concentrates.

4.2.1 Experimental

The first set of flotation experiments was performed under exactly the same conditions as before (sections 2.5.2.2 and 3.1), except that the pulp density was 5% solids, since only half the feed mass was used (i.e. either only coal or only quartz). The LAC coal used in the artificial mixtures was the 7.2% ash sample (the same as that used in the initial flotation tests). The pulp pH was 7.6 (tap water) for all the runs. Flotation experiments were performed at various concentrations for each of the three amine reagents DTAB, HPYC, and HTAB.

A second set of flotation experiments was performed on the coal only (no quartz added) using the frother MIBC instead of the amines as the flotation reagent. This was to provide a reference set of runs for the flotation of the coal, using a frother which is known to have little or no coal collector action. These runs would give some idea of the coal's "natural" floatability. Five different concentrations of MIBC were used, namely 8, 10, 12, 15 and 18 μl/l.

A third set of runs was performed on the quartz/coal artificial mixtures in which the percentage of quartz in the feed mixtures was varied. It was hoped that these tests would show any dependence of (undesirable) **coal** recovery in the concentrates on the amount of **quartz** recovered in the concentrates. Four feeds were prepared; containing 16.7%, 25%, 33% and 50% quartz. The LAC sample used in the mixtures was the same 7.2% ash sample used in the other tests. Reverse flotation experiments were performed on each feed type using the amine HTAB as the flotation reagent at a concentration of 550 g/t for each feed mixture.

4.2.2 Results and discussion

The detailed results for all the above experiments may be found in Appendix VII. Figure 3.5 shows the results of the separate flotation runs on coal and quartz. In the concentration range 200 to 500 g/t, the quartz recovery was a strong function of amine concentration. At 450 g/t 90% quartz recovery was obtained for all three reagents. All three amines were very similar in their performance. Coal recovery was extremely low in the same amine concentration range, the highest coal recovery being of the order of 4%. HTAB gave the lowest coal recovery (of the three reagents) at low concentrations and thus appears to be the best depressant, although the differences between the reagents were small.

Only at higher amine concentrations (700 g/t and above) did coal recovery become significant and the differences between the three reagents become apparent. The reason for the high coal recovery at the high amine concentrations is not known, but it is possibly

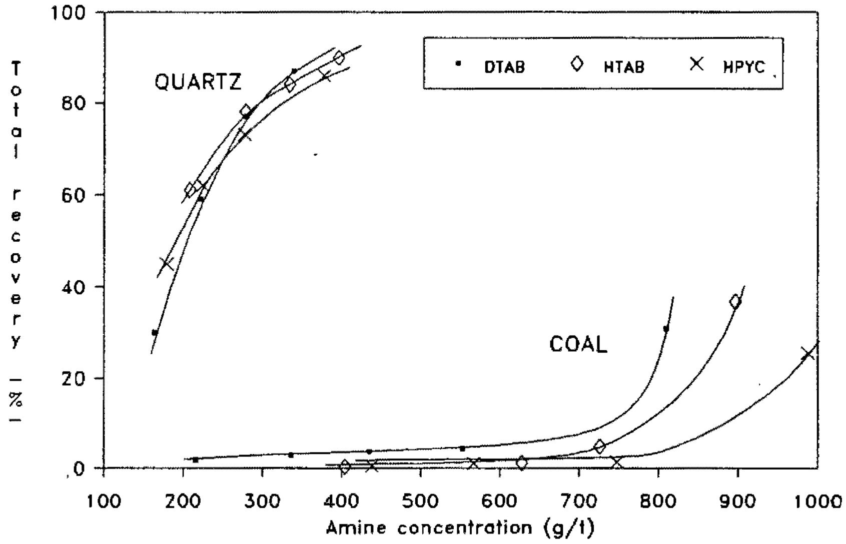

Figure 3.5: Separate flotation of LAC and quartz using HTAB, HPYB and DTAB.

due to double layer reagent adsorption on the coal surface. Another possibility is that the critical micelle concentrations (CMC) of the amines were exceeded, which would detract from their coal depressant action. Whatever the cause, however, at the amine concentrations required to recover the quartz, the coal recovery was minimal. It may be concluded that the amines are not good collectors of this coal, except at very high concentrations.

Figure 3.6 shows the results of the runs in which MIBC frother alone was used to float the coal only. Final cumulative recovery is plotted against frother dosage. The maximum coal recovery obtained was 60% at a dosage of 18 µl/l. By comparing the reverse flotation results shown in Figure 3.5 with these results, it is apparent that the coal recovery obtained with the amines (which are also frothers) was far below the natural floatability of the coal (provided the amine concentrations were less than about 700 g/t). The results indicate that the amines are, indeed, depressing the coal.

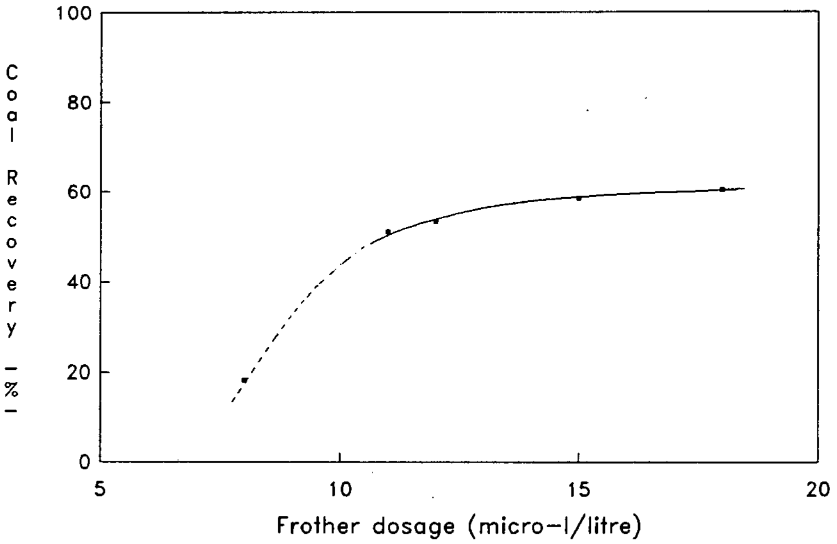

Figure 3.6: Flotation of LAC with MIBC frother only.

Figure 3.5 can thus be used to delineate a "safe" concentration region for each amine in which the flotation process should be operated in order to minimize coal loss to the concentrates and still obtain the desired quartz recovery: for DTAB it is below 600 g/t, for HTAB it is below 700 g/t and for HPYC it is below 800 g/t (these values are approximate). In the initial flotation tests (see Table 3.1) the DTAB and HTAB concentrations were, in fact, within these safe regions and yet significantly more coal was recovered in the concentrates than would be expected from Figure 3.5. Given the fact that the testwork described above indicates that the amines do depress the coal, some other mechanism must have been at work whereby coal was recovered in the concentrates.

In the initial flotation tests, using a 50% quartz/coal mixture, it was found that 24.9% coal recovery was obtained in the concentrates when using HTAB at a concentration of 595 g/t. With no quartz present, and using the same coal sample, the coal recovered was in the region of 4%. (see Figure 3.5). It would thus seem that the presence of quartz in the feed, and its recovery into the concentrates, increases the coal recovery significantly compared to when coal is floated alone. The same is true for DTAB and HPYC. This finding was investigated quantitatively in the next set of experiments.

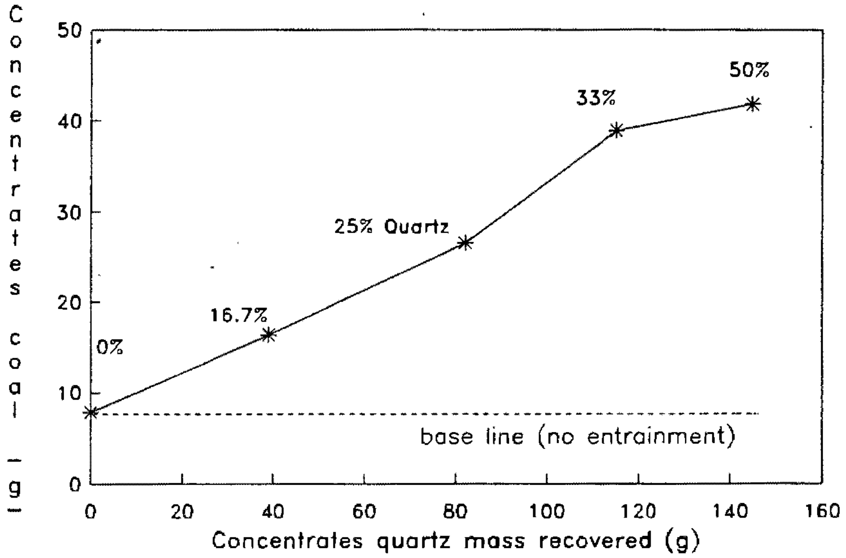

Figure 3.7: Dependence of concentrate coal recovery on the amount of quartz in the flotation feed.

Figure 3.7 shows the results of the reverse flotation tests carried out on four feed mixtures containing increasing proportions of quartz. The feed quartz percentage is indicated above each data point. The graph shows the mass of coal recovered as a function of the mass of quartz recovered in the concentrates. In addition, a horizontal (dotted) base-line indicates the coal recovered where no quartz was present in the feed. (This value is obtained from Figure 3.5 at an HTAB concentration of 550 g/t.)

As can be seen, the coal recovery showed a marked increase for each successive increase in the amount of quartz recovered (which corresponds to each increase in the feed quartz percentage). This occured in spite of the fact that the total mass of coal in the feed decreased as the proportion of quartz in the feed increased (the total feed mass was constant). If no entrainment of coal was occurring, then a constant mass of coal would be recovered as quartz proportion was increased, as indicated by the base-line. It can thus be concluded that the coal was recovered by mechanical means (i.e. by entrapment and/or entrainment) in addition to its natural floatability, and that this mechanical recovery is dependent on the amount of quartz recovered.

From the above results, it would seem that entrainment or entrapment and not poor depression was the cause of coal loss to the concentrates in all the reverse flotation experiments which have been reported thus far in this chapter.

4.3 Reduction of coal loss: staged addition

The coal loss that is a feature of the reverse flotation experiments reported thus far in this Chapter was unacceptably high. A method was required to reduce coal entrainment (and/or entrapment) while operating within the "safe" concentration regions discussed in the previous section. One possibility was staged addition of the flotation reagent(s). In this process, the collector is added in a number of small doses, say three. The mass recovery of concentrate is not as great in each stage as for a single large dose and so the tendency for hydrophilic material to be entrained is substantially reduced.[1] Reverse flotation tests using staged addition of amine reagents were performed in order to investigate this.

4.3.1 Experimental

Flotation runs were again performed on the 50% quartz/coal mixtures using the LAC sample containing 7.2% ash. The conditions were identical to all the previous reverse flotation runs except that for each of the three amines, the total quantity of each amine was added in one, two or three stages. The total (approximate) concentrations for each amine were as follows: DTAB 330 g/t, HTAB 550 g/t, and HPYC 750 g/t. The amount of amine added in each stage as a proportion of the total amount added was (approximately) as follows, for each of the three amines:

2–Stage: 1st; 0.8, 2nd; 0.2.
3–Stage: 1st; 0.7, 2nd; 0.2, 3rd; 0.1.

These concentrations were found to give optimal results and were all within the "safe" regions depicted in Figure 3.5.

For each stage of the multistage addition runs, concentrate was collected until recovery reached extinction. The aeration was then terminated and the next quantity of amine was added to the cell and allowed to condition with the pulp for a period of four minutes. The aeration was then recommenced and the run allowed to proceed. Details of the experiments and results obtained may be found in Appendix VII.

4.3.2 Discussion

Table 3.2 reports the optimum flotation results for 1–, 2– and 3–stage reverse flotation runs for each of the three amines. The table shows the following: using HTAB at a total dosage of (approximately) 550 g/t, 79.8% product coal recovery was obtained at 16.3% ash in one stage; 89.4% (cumulative coal) recovery was obtained at 14.2% ash in two stages, and 88.15% (cumulative) coal recovery was obtained at 11.8% ash in three stages. The HTAB concentrations in the first stage of the 2–stage and 3–stage runs were 408 g/t and 401 g/t, well within the "safe" region for this amine (see Figure 3.5). A

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[1] This is one of the methods used to reduce entrainment of slimes in forward flotation (see Chapter 2, section 3.2).

similar situation exists for HPYC. Thus, for HTAB and HPYC, the improvement in flotation efficiency using staged addition is obvious. For the DTAB reagent, no significant benefit was observed using a 2– or a 3–stage addition method (the coal recovery increased but do did the ash content) .

Table 3.2: Results of staged addition runs

Description	DTAB	HTAB	HPYC
1-stage: Flotation time	240 s	360 s	360 s
Ash recovery	94.68%	91.3%	92.74%
Coal recovery	80.16%	79.77%	69.44%
Product ash %	12.0%	16.3%	15.88%
reagent conc. (g/t)	365	550	745
2-stage: Flotation time	270 s	180 s	210 s
Ash recovery	92.70%	92.87%	93.30%
Coal recovery	89.32%	89.38%	79.85%
Product ash %	14.22%	14.2%	14.23%
reagent conc. (g/t)	264 + 53.3	408+148	538 + 203
3-stage: Flotation time	360 s	270 s	270 s
Ash recovery	91.54%	95.75%	92.90%
Coal recovery	87.10%	88.15%	79.49%
Product ash	15.3%	11.75%	15.72%
reagent conc. (g/t)	237+61.7+32	401+96+56	535+72+37

Figure 3.8 shows the recovery/time trajectories for quartz and coal using the HTAB amine (The graphs for HPYC and DTAB may be found in Appendix VII). It can be seen that the coal recovery was substantially reduced by performing reverse flotation in stages, especially in the first reagent addition stage. This confirms the previous conclusion that entrainment was the cause of coal recovery in the concentrates.

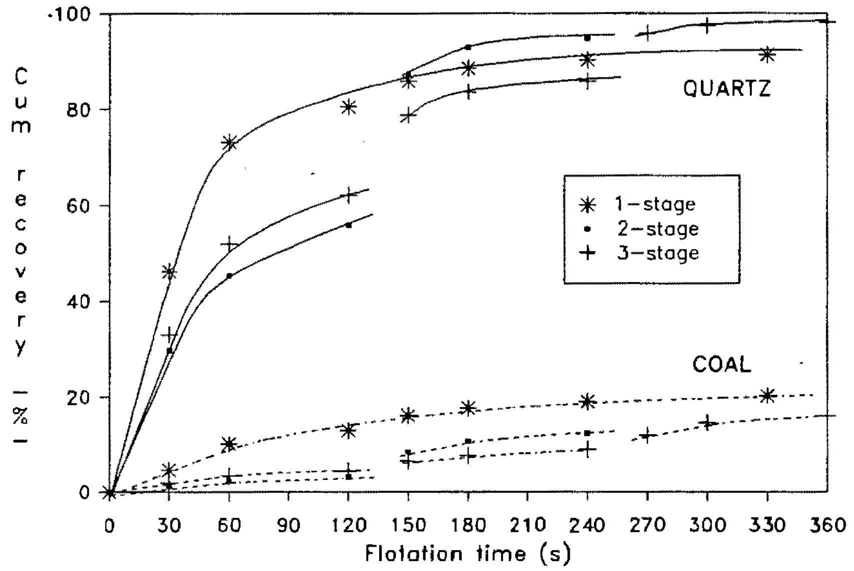

Figure 3.8: Reverse flotation of 50% quartz/coal mixtures using staged addition of HTAB.

The above results indicate that coal entrainment in reverse flotation can be reduced by a staged reagent addition method. HTAB gave the best overall separation of the three reagents, using a 3–stage addition process. As a consequence, this reagent was used exclusively in all subsequent reverse flotation tests described in this thesis.

5. Kaolin/coal mixtures

Until this point in the experimental program, kaolin was not used in the artificial coal/gangue mixtures on account of the small particle size of the sample available for experiment (50 % –0.002 mm). It was thought that such fine particles would cause difficulties in flotation; both in terms of poor flotation efficiency and also for practical reasons, such as high reagent consumption due to the large surface area which characterizes ultrafine particles.

After reverse flotation was found to be successful on artificial mixtures containing quartz, it was decided that the process should be tested on kaolin/coal mixtures as well, since kaolin is the dominant gangue mineral in South African coals.

Preliminary reverse flotation runs were performed on a 50% kaolin/coal feed. These experiments were unsuccessful because of an excessively large froth volume, caused (presumably) by the very high amine concentration (3000 g/t or more) required to obtain even a small mass recovery. These results are not reported. However, it was thought that a staged reagent addition method as well as a smaller proportion of kaolin in the feed mixture would allow this problem to be circumvented. These experiments were carried out, and are described below.

5.1 Experimental details

The 7.2% ash LAC sample used in all the preceeding experiments was, by this stage, depleted. A fresh LAC sample containing 4.4% ash was obtained (from the same colliery), and this was used in the kaolin/coal feeds. Feed mixtures were made by blending kaolin and LAC to make four feeds; containing 10%, 16.7%, 20% and 30% kaolin. The total feed mass for each run was 300 g and this was added to 3 litres of water to give a pulp density of (approximately) 10% by mass.

Reverse flotation runs were performed on each of the four feed mixture types. For all of the runs the impeller speed was 1200 rpm, the aeration was 4 l/min and the pulp pH that of tap water (pH=7.6). A 3–stage reagent addition method was employed, as described above (section 4.3.1). The total amine dosage was much higher than for the quartz/coal feeds and, furthermore, additional amounts of reagent were added for each successive increase in the kaolin feed percentage in order to compensate for the greater amount of reagent required by the kaolin particles. For the 10% kaolin feed, the optimum dosage was found to be 1267 g/t (based on total feed) and this increased up to 2367 g/t for the 30% feed.

In previous runs, flotation was continued until recovery ceased (usually after six minutes). For these tests, however, it was found more practical to perform a flotation run for a total period of 12 minutes (780 s), which served as a standard cut-off point. The flotation time increments for individual stages were as follows:

0 to 360 s for stage 1,
360 to 600 s for stage 2,
and 600 to 780 s for stage 3.

A second set of (1–stage) runs was performed on the four kaolin/coal mixtures in which the frother MIBC was used as the only flotation reagent. These represented "blank" runs which could be used to determine whether the kaolin particles were being floated or entrained (non-selective recovery) in the reverse flotation runs. The reasoning was as follows: If substantial recovery of kaolin occurred in both the frother–only and the reverse flotation experiments, then it is likely that kaolin was recovered by entrainment (in both cases). If, on the other hand, the kaolin recovery in the frother-only runs was insignificant in comparison to the reverse flotation runs, then it is likely that the amine was floating the kaolin and was thus acting as a kaolin collector.

Details of the experiments performed and the results obtained are given in Appendix VII.

5.2 Discussion of results

5.2.1 Reverse flotation runs

The results of the kaolin/coal runs are presented in Figure 3.9, which shows recovery/time trajectories for kaolin and coal for each feed mixture. The coal recoveries were almost identical and so are not identified in the Figure.

It is apparent from Figure 3.9 that the rate of kaolin recovery in reverse flotation was low: at best, only 60% kaolin was recovered after 780 seconds. This may be compared with the quartz/coal mixtures (Figures 3.8 and 3.9) where typically 90% quartz recovery was obtained after 360 s. It is likely that the low recovery rate is a direct consequence of very small size of the kaolin particles. It is generally accepted that fine particles float more slowly than coarse particles and very fine particles, as is the case here, are difficult to float simply because the air bubbles in the pulp are neither small enough nor present in sufficient quantity.[1]

Although the final kaolin recoveries are lower than achieved in the quartz/coal runs, this does not necessarily mean that the HTAB amine is a poor kaolin collector. In fact, the opposite appears to be true. It may be observed that the rate of kaolin recovery, although small initially, increased substantially by the end of each reverse flotation run. The rate of coal recovery, on the other hand, remained more or less constant. Furthermore, the

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[1] It is quite possible that the kaolin particles require micro-bubbles (0.5 mm diameter or less) for efficient flotation. Bubbles of this size are rare in the Leeds cell design.

total amount of coal recovered was very low; typically 5% or less. It is possible that, with longer flotation times, the separation would improve. What needs to be checked, however, is that the recovery of the kaolin particles is the result of flotation by HTAB and not simply the result of entrainment by the upward flow of water into the froth created by the addtion of HTAB. If this can be shown, then it may be deduced that HTAB is a good collector for kaolin. This was the object of the next set of flotation experiments.

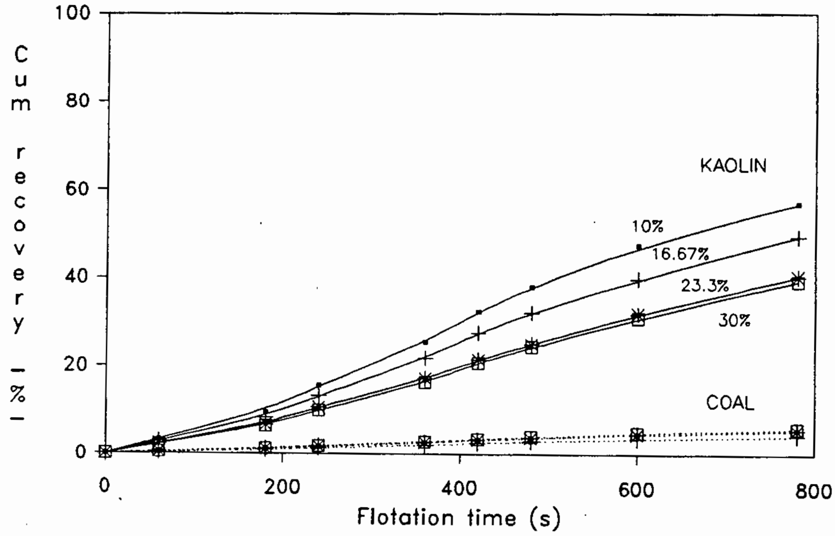

Figure 3.9: Reverse flotation of kaolin/coal mixtures using staged addition of HTAB.

5.2.2 Flotation using MIBC frother

Figure 3.10 shows the results of flotation runs on four kaolin/coal mixtures in which MIBC frother was used as the only flotation reagent. The graph shows coal, kaolin and ash recovery in the concentrates as well as the ash content of the concentrates, in bar format.

It can be seen that substantial coal recovery occurred, of the order of 62 to 70%, which is expected since the low–ash coal has a natural floatability. The coal recoveries obtained in these experiments were almost identical to those obtained with flotation of LAC only, using MIBC frother at 18 μl/l (see Figure 3.6). On the other hand, the kaolin recovery was very low—at most it was 3%, even though the coal recovery was as high as 60%. It is clear that entrainment of kaolin was very slight when using the frother only.

In the reverse flotation runs, exactly the opposite was found (cf. Figure 3.9)—the coal recovery was small (ca. 5%) and the kaolin recovery was of the order of 60%. If kaolin was being recovered by entrainment when using the HTAB reagent, then the same order of kaolin recovery would have been expected in the experiments with MIBC frother only. This did not occur and it may be concluded that the amine is, indeed, selective for the kaolin in the reverse flotation runs, as it is for quartz.

It can also be seen by comparing Figures 3.9 and 3.10 that the depressant action of HTAB on coal is effective, since the natural hydrophobicity of the coal displayed in the frother-only runs (Figure 3.10) was suppressed in the reverse flotation runs.

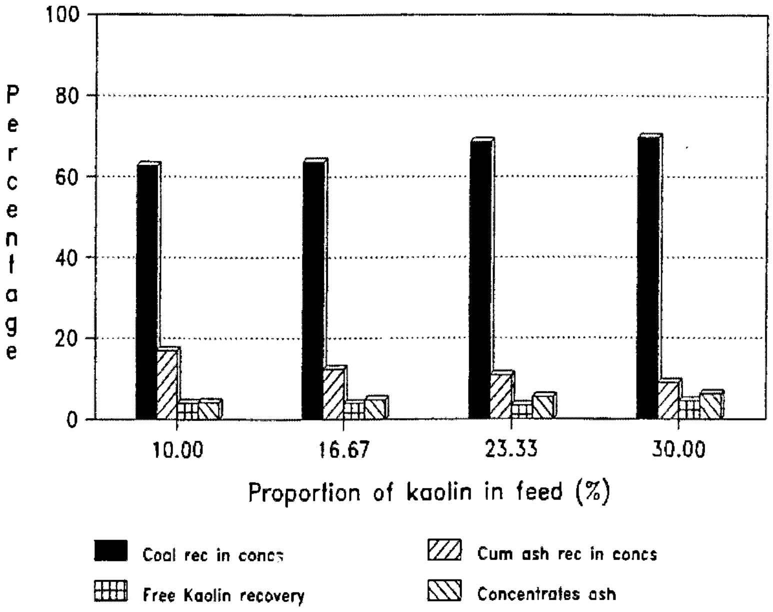

Figure 3.10: Reverse flotation of kaolin/coal mixtures using MIBC frother only.

6. Use of a coal depressant: dextrin

In the initial flotation tests using quartz/coal mixtures, coal depression was found to be unsuccessful; coal loss to the concentrates was of the order of 25 to 35 %. It has since been shown that a considerable amount of this coal loss was the result of entrainment, rather than poor depressant action. It was also found that this entrainment can be reduced by a stage-wise flotation technique. For example, for a 50% quartz/coal mixture using the amine HTAB, coal loss was reduced from 25 % to 13 % in a 3-stage reverse flotation run (see Table 3.2).

In the flotation of coal only using amine reagents (section 4.2.2) coal concentrates recovery (product loss) was of the order of 4 to 7% when using HTAB (see Figure 3.5). This suggested that, although entrainment can be reduced by stage-wise flotation, there was still room for improvement in terms of coal depression in reverse flotation of the artificial quartz/coal feeds. Accordingly, it was decided to investigate the use of dextrin as an additional coal depressant.

In Chapter 2, the adsorption behaviour of the quaternary amine HPYC onto samples of LAC and its interaction with dextrin was investigated. It was found that the HPYC and the dextrin compete in terms of adsorption; in order to obtain the maximum dextrin adsorption on the coal, the dextrin needed to be adsorbed first. For flotation, it was assumed that the feed would have to be conditioned with dextrin prior to adding the amine if the maximum dextrin adsorption and thus maximum depressant action was to be obtained. Reverse flotation experiments were performed using various methods of dextrin addition in order to test this assumption.

6.1 Experimental details

Quartz/coal mixtures were used in these flotation tests. Unfortunately, the 4.4% LAC sample used in the kaolin/coal feeds described in section 5.1 had been disposed of inadvertantly, and hence a new LAC sample was obtained from the colliery, which contained 9.2% ash [1]. Flotation runs were performed in the laboratory cell as before. In all the flotation runs, HTAB was added in two stages. (A 2–stage run is more convenient to perform than a 3–stage run and results are only slightly inferior.)

Flotation experiments were performed on four feeds, containing 16.7%, 25%, 33.3% and 50% quartz, in order to determine the effect (if any) of prior dextrin conditioning. The dextrin concentration was 800 g/t (based on the LAC mass) and the total HTAB concentration was 550 g/t (based on the total feed mass). The procedure was as follows: For each feed type, three reverse flotation runs were performed. One run was performed without adding dextrin, another was performed by adding the dextrin and the HTAB simultaneously and conditioning the pulp for four minutes, and the last run was performed by conditioning the dextrin in the pulp for four minutes before adding the amine.after which a further 3 minutes conditioning time was observed.

Two further sets of reverse flotation experiments were conducted in order to determine the optimum dextrin concentration to be used. One set was performed on 50% quartz/coal mixtures for comparison with previous flotation work and a second set was performed on 25% quartz/coal mixtures which more closely represent real coals in terms of ash content. The following dextrin addition rates were used: 0, 200, 400, 800, 1200 and 2400 g/t. These ratios were based on the amount of LAC in the feed and not the total feed mass. The dextrin was added to the agitated feed and conditioned for four minutes before the HTAB was added. Conditioning was then continued for a further three minutes. The total amine concentration was kept constant for all the runs, namely 550 g/t (based on total feed mass).

Details of each experiment and the results obtained may be found in Appendix VII.

6.2 Results and discussion

6.2.1 Effect of prior dextrin conditioning

The results of reverse flotation of the 25% quartz/coal feed is depicted in Figure 3.11 which shows quartz and coal recovery in the concentrates (with the latter referred to as "coal loss"), and the product (tails) grade. The results are grouped to represent three cases; one in which no dextrin was used, one in which dextrin was added simultaneously

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[1] Note, the 9.2% ash coal sample was also used in the dextrin adsorption experiments, thus it is expected that the results obtained in these flotation experiments can be related with confidence to the adsorption experiments

with the HTAB and one in which the feed was conditioned with dextrin prior to HTAB conditioning.

As can be seen from Figure 3.11, the coal loss was reduced from 18% to 12% when dextrin was added simultaneously with HTAB, compared to when no dextrin was added; this was further reduced to 9% when the feed was conditioned with dextrin prior to HTAB addition. Furthermore, the highest quartz recovery (98%) occurred with prior dextrin conditioning.

The results for the three other feed mixtures are not presented here; they show the same trends as the 25% quartz feed. Detailed results may be found in Appendix VII. The results are consistent with the findings of the adsorption studies, i.e. that maximum dextrin adsorption occurred when dextrin was added prior to the amine.

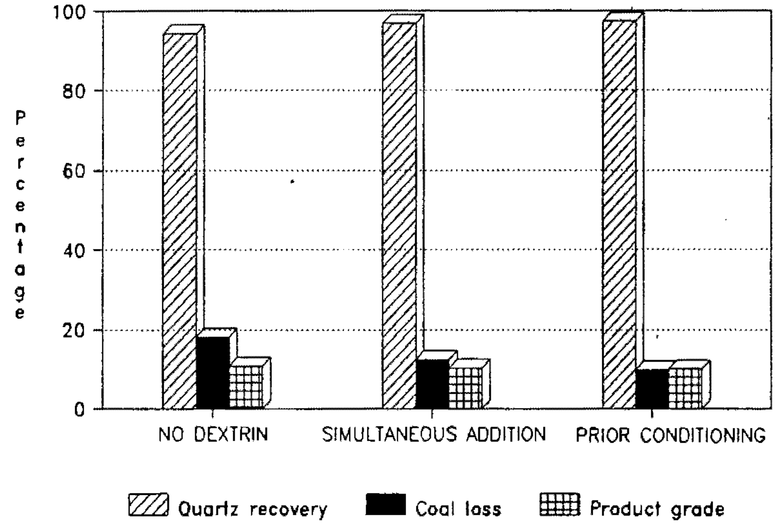

Figure 3.11: Effect of different methods of dextrin addition on reverse flotation of 25% quartz/coal mixtures.

6.2.2 Effect of dextrin: optimum concentration

Experiments were carried out on quartz/coal mixtures containing 25% and 50% quartz. The results of the flotation experiments on the 25% quartz/coal feed are presented in Figure 3.12. The graphs show quartz recovery, coal loss and product grade (as in Figure 3.11) for each of the dextrin dosage rates.

It can be seen from Figure 3.12 that the coal recovery (product loss) to the concentrates dropped from 18% to 7.6% as the dextrin concentration was increased to 1200 g/t. The increased recovery was, furthermore, accompanied by a slight improvement in grade (lower ash content) of the tails product. Usually the inevitable result of improved recovery is a poorer grade; therefore it may be concluded that the addition dextrin was having a highly beneficial effect on the separation efficiency.

The optimum dextrin concentration appears to be in the range of 800 g/t to 1200 g/t for both feed mixtures. In the case of the 25% quartz feed, the best grade (9.8% ash) was obtained at 800 g/t, while at 1200 g/t the lowest coal loss occurred (7.6%). In the case of the 50% mixture, the best result occurred at 800 g/t both in terms of grade (9.8% ash) and in terms of the smallest concentrate coal recovery (11.3%). In practice, a dextrin concentration of more than 400 g/t should be sufficient to obtain the benefit of using dextrin. The results for the 50% quartz feed were very similar; they can be found in Appendix VII.

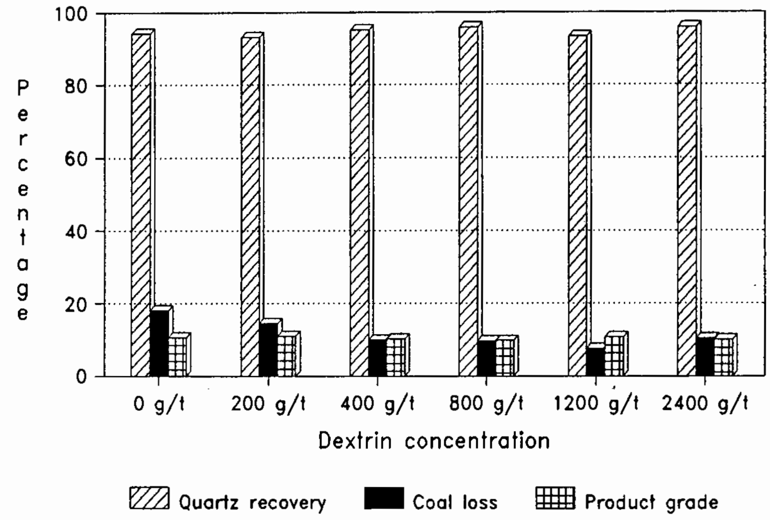

Figure 3.12: Determination of the optimum dextrin concentration in reverse flotation of 25% quartz/coal mixtures (HTAB concentration: 550 g/t).

6.2.3 Conclusion

It is clear that the addition of dextrin as a coal depressant has a highly beneficial effect on reverse flotation of the artificial quartz/coal feeds. The best results are obtained when the feed is conditioned with dextrin before the amine is added. This fact is consistent with conclusions made from the adsorption studies.

7. Comparison with forward flotation

The reverse flotation process has been developed to the point where it is highly successful on the artificial quartz/coal mixtures. A reagent system has been developed which achieves both coal depression and quartz flotation and thus a reversal of the "natural" surface properties has been accomplished: the naturally hydrophobic coal is rendered hydrophilic and the naturally hydrophilic quartz is rendered hydrophobic. Batch flotation experiments have shown the process to be technically feasible. At this point in the thesis it is necessary to ask the question, "How does reverse flotation compare with conventional forward flotation?"

In Chapter 1 (section 3), the anticipated advantages of reverse coal flotation were *reduced concentrate mass recovery* and *reduced entrainment*, which were expected to lead to an improvement in grade compared with that obtainable in forward flotation. In this section reverse flotation is compared with forward flotation of the same artificial quartz/coal feed mixtures, and the anticipated advantages of reverse coal flotation are re-evaluated in the light of the results obtained.

7.1 Experimental details

In order to compare the performance of reverse flotation with that of conventional forward flotation in the batch cell, experiments were again performed on the artificial quartz/coal feeds. Four feeds were used, containing 16.7%, 25%, 33% and 50% quartz (the LAC sample used contained 9.2% ash). Feed mixtures were prepared in exactly the same way as for the reverse flotation runs. Reverse and flotation runs were performed on each of the four feeds, making a total of eight flotation experiments.

In all the experiments the air rate was 4 l/min., the agitation rate was 1200 rpm, the pulp density was 10%, and the pulp pH was that of tap water (pH=7.6).

For forward flotation, the flotation procedure followed was the same as that for the reproducibility runs (see section 2.5.4). The coal collector used was Shellsol A and the frother MIBC. The addition rates were 1000 g/t and 12 μl/l respectively.

The reverse flotation experiments were 2–stage addition runs using HTAB and dextrin as before. The dosages were 550 g/t (basis: total solids mass) and 800 g/t (basis: LAC mass) respectively.

Details of each run and the results obtained may be found in Appendix VII.

7.2 Results and discussion

The flotation results are presented in Figure 3.13. The graph has two y-axes: the left one represents the coal recovery in the product (tailings of reverse flotation; concentrates of forward flotation), and the right axis represents the product ash percentage. The x-axis shows the percentage quartz in the total feed. As can be seen from the graph, the performance attained in each of the two methods was fairly similar; both methods resulted in an extremely good separation of coal and quartz. In some respects, however, the trends are opposite. For the feed with the lowest quartz content, 16.7% quartz, reverse flotation resulted in the greater coal recovery (94.12% vs 89.68%). As the proportion of quartz in the feed was increased the coal recovery in the product decreased, whereas in forward flotation it remained virtually constant. With 50% quartz in the feed, the situation was reversed: the greatest coal recovery was achieved in forward flotation (88.39% vs 84.96%).

In terms of product grade, the results for forward flotation show a clear trend: the ash content of the product increased as the feed quartz percentage increased; from 8.95%

ash at 16.7% feed quartz, to 10.3% at 50% feed quartz. In reverse flotation, the grades were mostly poorer than in forward flotation, but there was an inconsistency at 33% quartz, the ash content was lower for reverse flotation (9.48% vs 9.7%). However, allowing for possible experimental error (determination of ash content, feed variability etc), this deviation can be ignored. On an overall basis, it may be concluded that forward flotation gave the best product grade, while reverse flotation gave the higher overall product coal recovery for quartz levels up to ca. 33%.

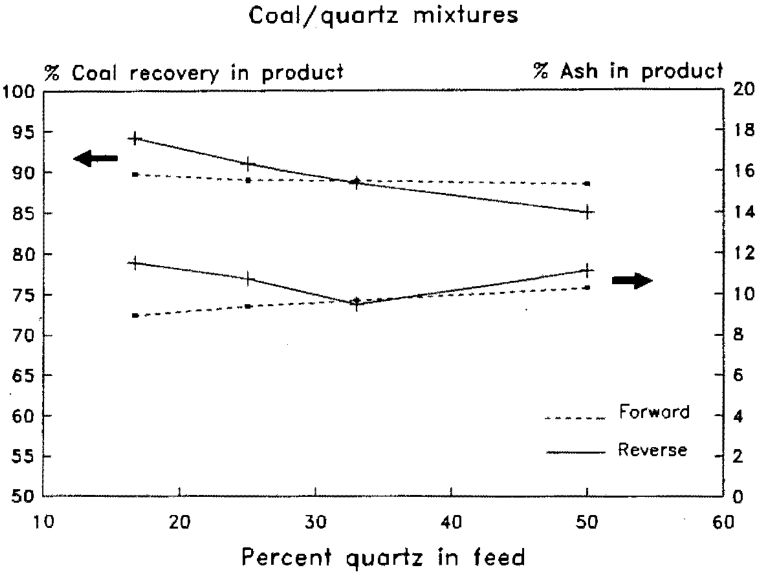

Figure 3.13: Comparison of reverse flotation and forward flotation (artificial mixtures).

The results are also shown in Figure 3.14, which plots the mass of material recovered in the concentrates of reverse and forward flotation against the quartz feed percentage, on a basis of 100 g feed (for each process). Three trajectories are shown, representing the two processes: the recovery of LAC in the reverse flotation concentrates, the recovery of quartz in the forward flotation concentrates, and the total ash material (i.e. quartz *plus* the ash in the LAC coal) in the forward flotation concentrates.

In the reverse flotation runs, it can be seen that the recovery of LAC increased with an increase in the feed quartz percentage; the mass of LAC in the concentrates was 4.9 g for the run in which the quartz percentage was 16.7%, and this increased to over 7 g for a feed quartz percentage of 33% (see indicated line). This suggests that entrainment of LAC occurred in the concentrates (discard) as a result of the increased quartz mass flux in the concentrates. This effect was shown in previous reverse flotation experiments (see section 4.2.2, and Figure 3.7).

In the forward flotation runs, a slight increase in the product ash percentage was observed (Figure 3.13) as the feed quartz percentage increased. This can be explained by means of Figure 3.14, which shows that the mass of quartz recovered in the concentrates (although small in absolute terms) increased as the proportion of quartz in the feed increased. At the same time, the mass of coal recovered decreased (there was less coal in the feed) and this would cause the ash content of the concentrate product to increase.

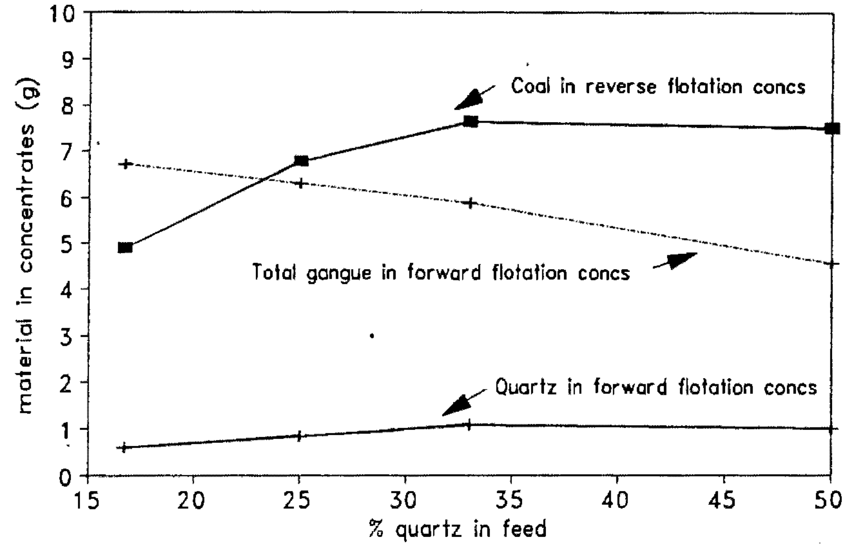

Figure 3.14: Comparison of entrainment occuring in the reverse and forward coal flotation process using artificial quartz/coal feeds (basis: 100g feed).

It may also be observed from Figure 3.14 that in the forward flotation runs, the total mass of gangue recovered in the concentrates (indicated by the dotted line) decreased as the quartz percentage increased. This occurred presumably because the mass of LAC recovered in the concentrate decreased, so less associated ash material was recovered (the LAC has an ash content of 9.2%). Since very little quartz was recovered in absolute terms, the sum of the LAC ash material and the quartz in the concentrate was smaller for each successive increase in the feed quartz percentage.

The total concentrate mass recoveries of the two processes can be compared in Figure 3.15, which shows the percentage yield of concentrates against feed quartz percentage for reverse and forward flotation. It can be seen that at feed quartz percentages in the range 16.7–33% (which would reflect a typical range for gangue material in r.o.m. coals) the concentrate yield was substantially smaller in reverse flotation. In fact, for a feed quartz percentage of 16.7%, the yield was almost three times lower (19.7% vs 75.2%). Thus, one of the two proposed advantages of reverse flotation (see Chapter 1) has been achieved.

For higher quartz percentages, the yields of the two processes approached each other, and with 50% quartz in the feed, reverse flotation had a slightly higher yield than forward flotation. This is not surprising, however: the LAC contains 9.2% ash and thus the theoretical yield of coal in a 50% quartz/coal mixture (forward flotation) would be ca. 46% and not 50%..

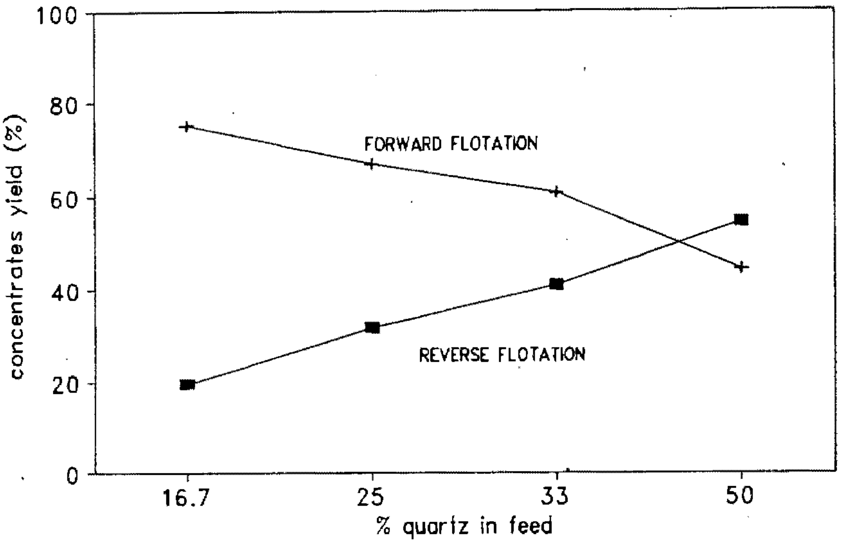

Figure 3.15: Comparison of the concentrate yields obtained in reverse and forward flotation.

7.3 General discussion

In absolute terms, entrainment was *worse* in reverse flotation than in forward flotation of the artificial feeds. It can be seen from Figure 3.14 that the mass of entrained coal (LAC) in reverse flotation was in the range 4.9–7.6 g, whereas in forward flotation the mass of entrained quartz was in the range 0.6–1.1 g (on a basis of 100 g of feed in each case). This is contrary to what was anticipated in Chapter 1 (see section 3): it was hypothesized that reverse flotation would result in less entrainment than forward flotation. This was based on the premise that coal particles generally have a larger average particle size than gangue particles and thus are less likely to be entrained. This does not take account of fact that coal particles are less dense than gangue particles (R.D.=1.3 vs 2.6) and thus, even though coal particles might be larger than gangue particles, they might be lighter and thus more prone to entrainment. Furthermore, the size distribution of the LAC particles in the artificial feeds was very similar to that of the quartz, thus entrainment would be *expected* to be worse for the LAC coal particles in reverse flotation than for the quartz particles in forward flotation.

It was proposed in Chapter 1 that entrainment would affect the two processes differently: it would cause a loss of coal recovery in reverse flotation, but a decrease in grade in forward flotation. This is consistent with what was observed (see Figures 3.13 and 3.14).

In general terms, it may be concluded from the results of the batch flotation testwork on the artificial mixtures that the reverse flotation method was highly successful at achieving the desired separation, and its performance compared well to forward flotation. However, coal entrainment was unexpectedly high. A staged addition process was employed to reduce entrainment but there is room for further improvement. This might be achieved by the use of column flotation cells, which offer improved cleaning over conventional cells. The addition of wash water in the froth phase of a column cell provides a mechanism for the drainage of entrained particles from froth concentrate (see Chapter 1, section 2.3, p 6–7).

It was seen that the concentrate mass recovery of reverse flotation was substantially less than forward flotation, this having been defined as one of the advantages of reverse flotation. This advantage could be exploited with the use of a column flotation cell: the high concentrate mass recovery in forward flotation would limit throughput as a result of the column carrying capacity which places an upper limit on the concentrate overflow rate (see Chapter 1, sec 2.3). In reverse flotation, the reduced concentrate recovery would be expected to allow operation of a column at increased feed rates and/or pulp densities. This advantage could well override any slight disadvantage of reverse flotation in terms of cleaning, and provides an additional motivation for testing the reverse flotation process in a column cell.

8. Conclusions

1. The purpose of the batch flotation tests was to evaluate the selected reagent suite in reverse flotation and to identify the optimum flotation conditions and procedures.

2. For artificial quartz/coal feeds, the quaternary amines were found to be convincing ash collectors and frothers.

3. With kaolin/coal mixtures, gangue selectivity was found to be good in spite of the small kaolin particle size which is unsuitable for flotation. This "worst case" suggests that reverse flotation of kaolin (the dominant coal mineral) in "real", r.o.m. coals should be successful.

4. In the initial flotation experiments, coal recovery in the tailings (product) was poor. Investigation showed that entrainment/entrapment was largely responsible for product coal loss in the flotation concentrates.

5. Staged reagent addition was found to reduce coal loss in to the (gangue) concentrates. The reagent HTAB was found to give the best overall results, using a three stage addition process. This reagent was used in all subsequent testwork.

6. Dextrin was found to be successful as an additional coal depressant in reverse flotation. It was also shown that the feed needed to be conditioned with dextrin prior to collector addition for maximum benefit.

7. Reverse flotation resulted in very similar beneficiation results to forward flotation. However, contrary to what was expected, entrainment occurred to a greater extent in reverse flotation and resulted in a loss of coal recovery from the product.

8. Reverse flotation resulted in a reduction of mass reovery of up to threefold less than in forward flotation, depending on the proportion of gangue in the feed. This was expected to be a major advantage for operation in a column flotation cell.

Following the successful result obtained with conventional batch flotation, further reverse flotation work using artificial mixtures will was extended to the laboratory column cell to investigate the possibility of improved cleaning, and to evaluate the proposed advantage of increased throughput in column cells. This is the subject of the next chapter.

CHAPTER 4
COLUMN CELL FLOTATION TESTWORK

1. Introduction

In Chapter 2, a reagent suite was identified for reverse flotation of coal. Adsorption tests gave a preliminary confirmation of its suitability. In Chapter 3, batch flotation tests using artificial mixtures showed that the reagent suite was successful for obtaining gangue flotation and coal depression; in this way the technical feasibility of the process was established. In this chapter, the investigation is extended to the evaluation of reverse coal flotation in a column cell in an attempt to improve the efficiency of the process still further.

Column cell technology, which has become increasingly popular since the early 1980s for mineral ore beneficiation, offers a number of advantages over conventional, sub-aeration cells. Column cells have been found to reduce entrainment almost completely, with consequent improvements in concentrate grade (Yianatos et al., 1988). Furthermore, the installation of column cells can result in simpler plant circuits and improved separability and recovery of ultrafine (–0.025 mm) material (Yianatos, 1989).

Reverse flotation experiments performed using the conventional (batch) cell (Chapter 3) revealed that entrainment of coal to the concentrate was a significant cause of clean coal (product) loss. This was reduced by a staged reagent addition method, but not eliminated completely: 6 to 13% coal loss was still occurring. Column cells can effectively eliminate entrainment by virtue of wash water addition to the froth; accordingly, coal entrainment in reverse flotation might be reduced by the use of a column cell, which would improve the overall separation that could be obtained by the process.

A second motivation for conducting reverse coal flotation experiments in a column cell is the possibility of improved performance over the process in the conventional cell (using the artificial mixtures). In contrast to most mineral ore flotation, coal flotation suffers from the problem of a high concentrate mass recovery which aggravates gangue entrainment into the froth. This results in poor concentrate grade (high ash content). In addition, the high mass recovery coupled with an inherently low carrying capacity results in severe throughput limitations in coal flotation column cells. Reverse coal flotation offers a means of substantially reducing the mass recovery and thus improving the column throughput capability.

For these two reasons, it was thus thought appropriate to this investigation to carry out the new reverse flotation process in a column cell to investigate whether these proposed advantages could be achieved.

The objectives of this chapter are twofold: 1) to examine reverse coal flotation in a laboratory column cell and compare the results with those of a) reverse coal flotation in the conventional batch cell and (b) forward coal flotation in the column cell; and 2) to investigate reverse coal flotation as a means of increasing the throughput of a column cell for beneficiating fine coal.

These objectives are divided into the following sections:

1. The determination of the optimum conditions for reverse coal flotation in a laboratory column cell, using ideal artificial quartz/coal mixtures.
2. An entrainment study, using artificial quartz/coal mixtures, to compare the cleaning achievable by reverse flotation in the column cell with that obtained previously in the batch cell (and reported in Chapter 3).
3. The comparison of reverse and forward coal flotation in a column cell in terms of cleaning, carrying capacity and throughput capability, using the artificial quartz/coal mixtures as feeds.

The laboratory column used in this work had already been constructed in the Department of Chemical Engineering at the University of Cape Town, and operated successfully for a number of coal beneficiation projects (forward flotation). In order to establish suitable column operating conditions for reverse coal flotation, a brief review of the relevant literature on column flotation was performed. This enabled the selection of conditions to be used as a starting point and served as a guide to assess the performance of reverse flotation in the column cell.

In this chapter, the literature review is presented first. The experimental equipment and procedure is then described. This is followed by the results of experimental work on the artificial mixtures. An overall discussion then follows in which results from the column are compared with those obtained in the conventional batch cell, and a prognosis is given for the technical feasibility and potential of the reverse coal flotation process in industry.

2. Literature review

Three distinct types of flotation cells are in commercial use for beneficiating a wide variety of ores, including coal. These are mechanical, or "sub-aeration" cells, pneumatic machines and column flotation cells. All are operated continuously.

The sub-aeration cells are the most commonly used devices in coal flotation. Typical sub-aeration cells, which are also self aerating, are the Denver and the Wemco cells. They consist of a tank or series of tanks in which the feed pulp is mechanically agitated. Air is drawn into the pulp by centrifugal pressure and is dispersed into bubbles by a specially designed impeller rotor or diffuser device. The impeller thus performs three functions; mixing, solids suspension and air dispersion. Other sub-aeration cells, such as the Galigher Agitair device, rely on pumped air for operation (Wills, 1985).

Pneumatic cells have no mechanical agitation devices and rely on turbulence created by the aeration for the pulp agitation. They use either air entrained by turbulent pulp addition or (more commonly) blown in by a compressor. The main advantages of this type of machine are: the absence of moving parts, lower capital cost, and the ability to operate at higher feed pulp densities and over a wider particle size range than sub-aeration cells. At present, only a few pneumatic cells are in use for coal flotation (Osborne, 1988).

In both the pneumatic and the sub-aeration cells, there is a net upward flow of water into the froth zone (usually 5 to 30 cm thick) at the top of the pulp. This net upward flow of water can drag or "entrain" undesirable, hydrophilic particles (e.g. the mineral particles in coal flotation) into the froth phase. There is no mechanism to assist these particles, once entrained, to drain back into the pulp. Some particles will naturally fall back into the pulp phase (possibly to be entrained again), while others remain in the froth and are collected in the concentrate, adversely affecting the grade of the concentrate product. Increasing the froth height is a means of reducing entrainment, but there is a limit to the height that can be achieved: generally, a height of greater than 30 cm is difficult to achieve with sufficient stability for flotation.

In the last decade or so, a new kind of flotation cell, the column cell, has become popular for beneficiating mineral ores (Yianatos, 1989). This cell consists of a tall column of square or circular cross section (see Figure 4.1). The lower part of the column contains a quiescent pulp zone in which ore particles move downwards, countercurrent to a swarm of rising air bubbles which are introduced at the base of the pulp. On top of the pulp zone is a deep froth zone, typically 0.5 to 3 m high. Wash water is added near the top of the froth at a flow rate which results in a net downward flow of water in the column, called positive bias. This provides the mechanism by which entrained particles are drained back into the froth. This results in better cleaning, particularly when operating on fine material (Yianatos, 1989; Wills, 1985).

Column cells occur in various sizes, depending on their use. Industrial columns are typically 8 m to 15 m in height and 0.5 m to 3 m in diameter. Pilot scale columns are usually 0.1 to 0.2 m in diameter and 3 to 6 m high, while laboratory scale columns are typically 0.05 to 0.1 m in diameter and 1 to 5 m high.

As well as improving separation efficiency, column cells have simplified flotation circuit designs. For instance; a single column may be used to upgrade a finely sized rougher concentrate in a single step, whereas conventional flotation cells would require several sequential stages (Yianatos, 1989). This is a direct result of the efficient cleaning action that is an inherent feature of column cells.

Although there was an initial reluctance to employ column cells, the situation has changed dramatically since 1981. Column flotation is now standard in Mo upgrading, and application on other circuits, such as Cu/Ni (Inco), Cu/Mo (Gibraltar, Magma Copper) and Cu cleaning (Los Bronces, Chile), are becoming widespread (Yianotos, 1989). Recently, column cell technology has been applied to coal flotation as well. An industrial scale column installation (four 2.4 m diameter columns) is in operation at the Mayflower plant of the Powell Mountain Coal Co in Lee County, VA., which is the first commercial application of a coal column circuit (Groppo and Parekh, 1990).

2.1 Description of column cell operation

As depicted schematically in Figure 4.1, a column cell consists of two basic regions; namely the collection zone (or pulp zone) and the cleaning zone (or froth zone). The collection zone is equivalent to the pulp phase in a conventional cell. Feed slurry enters this zone about one-third of the way from the top and the feed particles descend counter-current against a large assemblage of rising gas bubbles generated by the sparger at the base of the column. The particles collide with the bubbles and the hydrophobic particles attach to the bubbles and are transported into the cleaning zone. The hydrophilic particles and partially hydrophobic particles which do not attach to bubbles are transported down the column and are removed from the bottom. They can, however, also be entrained into the froth phase.

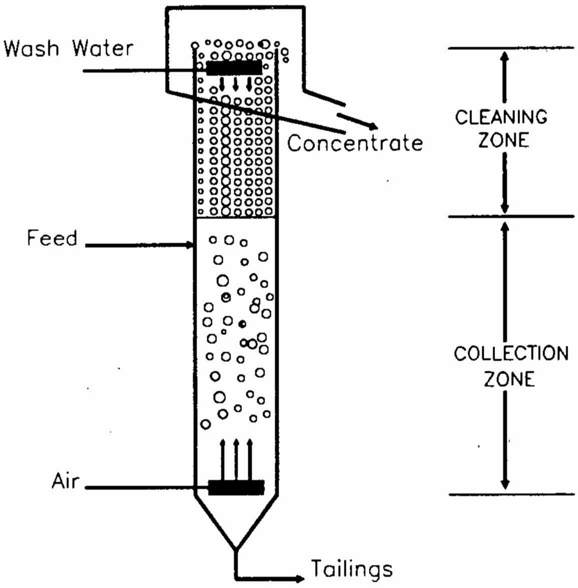

Figure 4.1: Schematic diagram of a column cell

In the cleaning zone, wash water is added near (or at) the top of the froth. The flow rate is set to provide a net downward liquid flow in the column, called a positive bias. In practice, this means that the tails water flow rate is higher than the feed water flow rate. This positive bias "cleans" the froth of entrained particles and also stabilizes the froth, promoting the formation of a deep froth (0.5 to 3 m).

The overall performance of a column is affected by the behaviour of both the collection zone and the cleaning zone. This behaviour is in turn affected by a number of variables (reagent dosage, gas and liquid flow rates, bubble size, feed particle size, froth depth, etc) some of which are under the control of the operator. In the next two sections, the behaviour of the collection zone and cleaning zone are examined, together with the variables that affect them.

2.2 Collection zone

2.2.1 Gas holdup and flow regimes

When gas is introduced into a column containing a flotation pulp, it displaces a certain volume of slurry. The volumetric fraction displaced is known as the gas holdup, ε_g. The gas holdup is an important variable; it gives an indication of the hydrodynamic condition of the collection zone. The relationship between gas holdup and gas flow rate in the column is often used to determine the hydrodynamic condition.

A useful way of defining gas and liquid flow rates in a column is by expressing them as volumetric flow rate per unit cross-section. This is called superficial velocity. Superficial velocities facilitate comparisons with columns of different cross-sections. For the gas, the superficial gas rate is given by the equation

$$J_g = Q_g / A_c \quad \text{..........} [4.1]$$

where A_c is the cross-sectional area of the column and Q_g is the volumetric gas rate. J_g represents the superficial gas rate at a specific point in the column where the volumetric rate is Q_g. To determine the average gas rate, reference must be made to standard conditions. The following relationship may be used to estimate the average J_g (Yianatos, 1989):

$$J_{g(ave)} = \frac{P_c J_g^* \ln (P_t / P_c)}{P_t - P_c} \quad \text{..........} [4.2]$$

In this equation, J_g^* is the superficial gas (air) rate at atmospheric conditions, P_t is the pressure (absolute) at the bottom of the column and P_c is the pressure at the concentrate overflow. For a tall industrial column of 12 m the value for $J_{g(ave)}$ will differ considerably from J_g^* because of the large head of water. For a laboratory scale column with a 1.0 m to 1.5 m head, $J_g(ave) \approx 0.95 J_g^*$, so J_g^* is usually a good enough estimate.

The relationship between gas holdup, ε_g, and superficial gas rate, J_g, is used to define the flow regime in the collection zone. Figure 4.2 shows a general relationship (Finch and Dobby, 1990, pp 9-25). As J_g increases, ε_g increases. The linear section of the relationship represents a flow condition that is characterized by a homogeneous distribution of bubbles of fairly uniform size rising at a uniform rate. This is called the "bubbly flow regime", which is the desirable flow condition in which to operate a column. As J_g increases, the gas holdup eventually becomes unstable and the flow is characterized by rapidly rising bubbles, which displace water and small bubbles downward. This is called the "churn-turbulent region" and is undesirable because it causes excessive mixing which adversely affects column performance.

2.2.2 Effect of variables on collection zone behaviour

In order to operate a column in the desired bubbly flow regime, it is important to know the influence that various parameters will have on the gas holdup and on J_{gmax}, the

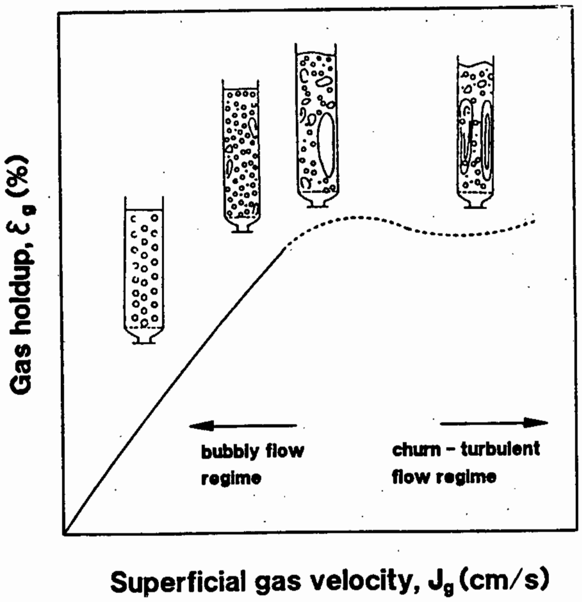

Figure 4.2: Relationship between holdup and gas rate (after Finch and Dobby, 1990)

maximum gas rate to maintain bubbly flow. The holdup is dependent on a number of factors, such as gas rate, liquid rates, frother concentration (which affects bubble size) and feed solids.

2.2.2.1 Gas rate and liquid rate

As can be seen from Figure 4.2, there is an upper limit on J_g for a given set of conditions if the churn turbulent flow regime is to be avoided. J_{gmax} is defined as the value of J_g at the onset of turbulence. In normal column operation (operating at positive bias) the superficial liquid rate, J_l, is counter-current to the bubbles. Increasing the liquid rate for a given J_g will increase the gas holdup because of a decrease in bubble rise velocity. This means that J_{gmax} will be lower if the liquid rate is increased while all the other factors are kept constant (Finch and Dobby, 1990, pp 9-25).

2.2.2.2 Frother dosage

The addition of a frother reduces the bubble size and increases the gas holdup for a given J_g because of the reduced bubble rise velocity. J_{gmax} will again be lower. Above a certain level of frother dosage (dependent on frother type), however, the bubble size change becomes very small and the ε_g dependence levels off.

2.2.2.3 Bubble size (d_b)

The bubble size in the collection zone is of fundamental importance because it determines the contact area for bubble-particle interactions which, in turn, will determine the recovery of mineral to be floated. Typically, the bubble size in a column cell is in the range 0.8 to 2 mm (Mills and O'Connor, 1990). The optimum size is usually 1.0 to 1.5 mm (Finch and Dobby, 1990, pp. 27-35), although this may be lower for smaller particle sizes that are floated—finer particles require smaller bubbles than coarse particles.

The bubble size is dependent on frother dosage (as indicated in the previous section), J_g, J_l, and the gas sparger design. Generally, reducing the bubble size for a given set of conditions will increase the gas holdup but decrease the bubble rise velocity. The effect of this will be to improve the quiescent behaviour of the collection zone, and to reduce the shear forces that might cause bubble-particle detachment, as well as to increase the overall bubble surface area.

2.2.2.4 Particle size (d_p)

Particle size is an important variable in the collection zone. One of the major problems in flotation is the relatively poor flotation response of both fine and coarse particles (Jowett, 1980).[1] For any given flotation system, the relationship between flotation recovery and particle size fraction is usually of an "inverse U" shape, i.e. the flotation recovery increases with increasing particle size and then drops off again after a certain maximum particle size. The variation in flotation response with size fraction is a combination of many factors, such as induction time, decreased opportunity for particle-bubble collision (fine particles) and disruption of particle-bubble aggregates in turbulent zones (coarse particles).

2.2.2.5 Solids

The nature of the solid particles can also have an effect on bubble size and bubble rise velocity. The bubble-particle aggregates cause the bubble rise velocity to decrease because of the reduced buoyancy of the loaded bubbles and this increases gas holdup which will, in turn, reduce J_{gmax}. The solids can either inhibit or promote coalescence (which increases bubble size), depending on their surface properties.

2.2.3 Bubble generation

The nature of the generation of air bubbles in the column cell is another factor which distinguishes column cells from other flotation equipment and which is also of fundamental importance in determining collection zone behaviour. The bubbles are generated by the use of spargers, of which there are two main types:

2.2.3.1 Internal porous spargers

An internal sparger is one in which bubble generation occurs in the pulp zone itself. The spargers are made of porous materials such as ceramic or fritted glass with pore sizes up to 300 μm or steel sieve plates into which holes (of more or less equal sizes) have

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[1] Note: the terms "fine" and "coarse" are relative to the flotation size fraction: "fine" particles usually mean 20 μm or less; "coarse" means 110 to 150 μm or more (depending on the flotation system).

been drilled. Other types are made of perforated rubber or filter cloth wrapped around a steel pipe into which holes have been drilled. The pressurized gas passes into the pulp through the pores and this causes the formation of the bubbles.

The bubble size that is generated using this type of sparger is a function of the gas flow rate, the sparger material and the frother content of the pulp. The bubble diameter increases with increasing gas rate: it is proportional to J_g^n, where n can be approximated as 0.25 (so the effect is moderate). Sparger material has a small effect on bubble size; filter cloth tends to give the largest bubbles (Finch and Dobby, 1990, pp 27-36).

The most important factor in determining bubble size (at a given gas rate) is frother concentration. Thus, the chemical conditioning of the pulp is an important factor in determining bubble size for internal spargers.

2.2.3.2 External spargers

An external sparger is one in which the gas bubbles are generated by contacting the gas with a separate water stream outside of the column. The combined stream is then fed into the column. This is the basis for a number of designs, including the U.S. Bureau of Mines sparger design. In this design, pressurized air and water streams are mixed in a contact chamber and the resulting mixture is injected into a column through small orifices (typically 1 mm diameter) (Ynchausti et al., 1988).

Factors which affect bubble size in this instance are air and water pressure, water flow rate, number of orifices and orifice size, and frother concentration (Finch and Dobby, 1990, pp 27-36). Because the sparger water contains the frother (which is fed from a separate tank) the bubble size can easily be altered by changing the frother concentration. This makes control over bubble size largely independent of the feed pulp condition.

Another advantage of the external sparger is that the bubble sizes are smaller. This is because the frother concentration is high at the point of bubble formation and there is a high degree of turbulence and shear because of the high velocities through the orifices. This will, in turn, have implications for J_{gmax}, since the smaller bubble sizes will increase holdup and this decreases J_{gmax}.

2.3 Cleaning zone

Conventional (sub-aeration) cells (and also pneumatic cells) suffer from entrainment of unwanted materials in the froth phase which adversely affects the cleaning action. Generally, it is the fine hydrophilic particles that are entrained but all particles, both hydrophilic and hydrophobic, can be entrained. Entrainment is inherent because water recovery to the froth cannot be avoided.

2.3.1 Entrainment in flotation cells

Entrainment occurs when particles enter the froth phase suspended in the water occupying the spaces between the bubbles (Warren, 1985). The finer the particle, the more likely it is to remain suspended and thus to be recovered in the concentrates by entrainment rather than by true flotation. This recovery by entrainment is a problem in flotation because it is non-selective—there is no discrimination between hydrophobic and hydrophilic particles; whereas with true flotation, only hydrophobic particles are attached to bubbles.

The nature of entrainment has been the subject of numerous studies. Jowett (1966) states that recovery of the non-floatable gangue mineral occurs by a mass transfer process initiated by water currents. Furthermore, he has shown that, for a simple two-component flotation system of coal/shale and added quartz, the free gangue mineral in the froth is proportional to the free gangue mineral in the pulp.

Other researchers (Warren, 1985; Trahar, 1981) have shown that recovery by particle entrainment is proportional to froth water recovery. In particular, Warren (1985) has investigated the contributions of true flotation and recovery by entrainment in various flotation systems. He has shown that overall flotation recovery, R_M, obeys the relationship:

$$R_M = F_M + e_M W \qquad [4.3]$$

where F_M is the true recovery by flotation, e_M is the degree of entrainment and W is the water recovery.

The e_M term in this formula is dependent on the flotation system (i.e. type of gangue minerals) and on the particle size. Thus, the finer the particle size of the gangue minerals in a flotation system, the greater will be the degree of entrainment and the less selective will be the flotation process. In conventional cells, the selectivity obtained in the finer size fractions is poor for precisely this reason.

It can be seen from equation 4.3 that, in addition to the e_M term, the magnitude of the entrainment term is determined by the degree of water recovery, W. It would seem logical then, that if the feed water recovery to the froth could be eliminated, then recovery by entrainment could be avoided. This is exactly the method by which the column cell operates.

The froth phase of a column cell differs fundamentally from a conventional cell because of the condition of "positive bias", or net downward flow of water in the column. This is maintained by the addition of wash water at the top of the froth. Positive bias achieves two things:

1) it prevents the hydraulic entrainment of hydrophilic particles in the froth which *improves selectivity*, particularly in the finer size fractions, and

2) it enhances froth stability, promoting the formation of a deep froth bed.

Bias is usually expressed as superficial bias rate which is expressed symbolically as J_b with the units cm/s.

2.3.2 The froth structure of a column cell

A two-phase study (Yianatos et al., 1986) has revealed the presence of 3 distinct regions in the froth zone: an expanded bubble bed situated just above the interface with the pulp, a packed bubble bed on top of this and a conventional drained froth above the wash water distributor. These regions are depicted in Figure 4.3 below.

As the bubbles rise in the froth zone, they coalesce because of bubble collision in the expanded and packed bubble beds (Finch and Dobby, 1990, pp 75-101). The greatest degree of coalescence occurs in the expanded bed region. Studies have shown (Finch and Dobby 1990, pp 75-101) that the mean bubble diameter can increase by as much as 67% in the expanded zone, while in the packed bubble bed the increase is a further 16% (approximate values).

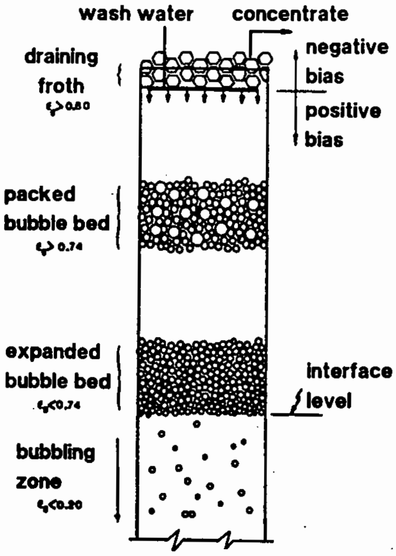

Figure 4.3: Regions in a 3-phase froth zone (Yianatos et al., 1986).

In the packed bubble bed, the bubbles remain fairly spherical but, due to coalescence, have an increasing variation in size as they move upwards. The movement of bubbles is near to plug flow in behaviour, provided that the wash water is well distributed and the bias rate is not excessive so as to cause mixing. Typical operating J_b (bias) rates are in the range 0.1 cm/s to 0.3 cm/s (Yianatos, 1989).

The conventional draining froth region that occurs above the wash water sprinkler is one which is characterized by negative bias. In this region, the bubbles are no longer spherical, but polyhedral. The vertical froth motion is transformed into a horizontal motion and in this way the solids are recovered in a launder.

2.3.3 Effect of variables on froth behaviour

In order to minimize feed water recovery to the froth and to obtain the best possible cleaning action, it is important to know the effects of various parameters on the froth zone behaviour.

2.3.3.1 Effect of gas rate, J_g

Increasing the gas rate results in an increase in the amount of feed water in the froth. This is detrimental to the cleaning action. It is even possible that, at a high enough gas rate, sufficient water will be entrained into the froth to bring about a condition of negative bias. Clearly there is an upper limit to the value of J_g both in terms of efficient cleaning and in maintaining the desirable bubbly flow regime in the pulp zone. In practice, the value of J_g is usually in the range 1.5 to 3 cm/s.

2.3.3.2 Effect of frother dosage

Increasing the frother dosage will reduce the bubble size. This means that for a given pulp/froth interface area, there will be a greater number of bubbles crossing the interface per unit time and consequently more water is entrained. The frother dosage will thus also affect the upper limit of J_g for the onset of negative bias in the froth zone. The higher the frother dosage, the lower will be the maximum J_g that can be tolerated to maintain positive bias.

2.3.3.3 Wash water rate

It has been shown that, as long as the wash water rate is sufficient to maintain a positive J_b value, the cleaning action is relatively insensitive to the actual value of J_b, provided that excessively high bias rates are avoided (Yianatos, 1989).

Excessive bias rates should be avoided. Tracer studies (Finch and Dobby, 1990) showed that, as J_b was increased from 0.1 cm/s to 0.3 cm/s, the concentration of feed water at the top of the froth zone was reduced from 8% to 5% (approximately). However, if the J_b value was increased further to 0.5 cm/s, the situation was reversed and the feed water concentration became 47% at the top of the froth. This is depicted in Figure 4.4.

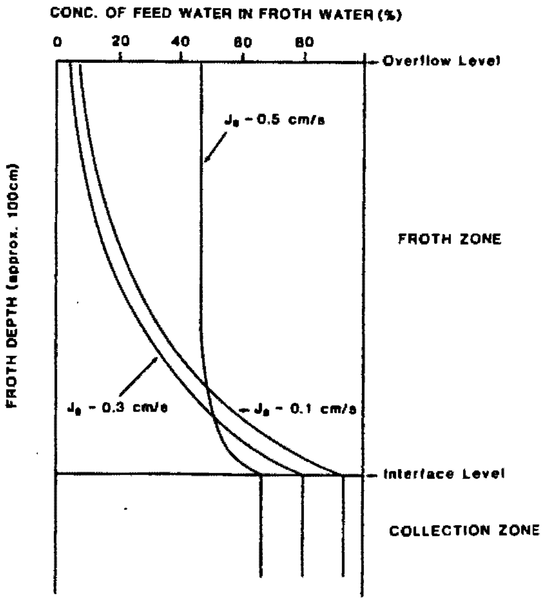

Figure 4.4: Effect of high bias rate (Yianatos, 1989).

A large bias rate increases mixing by altering the plug flow regime of the froth to a heterogeneous behaviour in which severe channeling and recirculation occur. The sprinkler design is thus an important factor in minimizing channeling and mixing. The design should be such so as to avoid a jetting behaviour for the whole range of water rates that is likely to be used.

A "rule of thumb" method has been proposed to estimate the wash water requirement in a column (Yianatos, 1989):

$$J_w = \frac{J_g{}^*\varepsilon_c + J_b}{1 - \varepsilon_c} \quad [4.4]$$

where ε_c represents the fractional holdup of concentrate at the top of the column and is given by the equation:

$$\varepsilon_c = \frac{J_c}{J_c + J_g{}^*} \quad [4.5]$$

J_c is the superficial liquid concentrate rate and $J_g{}^*$ is the superficial gas rate at standard conditions. Generally, ε_c values are in the range 0.1 to 0.2 in plant operation.

Another method is to estimate the concentrate liquid rate, J_c, from total concentrates overflow rate and pulp density of the concentrate and then calculate J_w by simple mass balance around the top of the column:

$$J_w = J_c + J_b \quad [4.6]$$

J_c may also be calculated from the following equations (Finch and Dobby, 1990, pp 75-101):

$$J_c = \frac{J_g{}^*(1 - \varepsilon_{g0})}{\varepsilon_{g0}} \quad [4.7]$$

where ε_{g0} is the gas holdup at the top of the froth, which can be taken as typically 0.8.

2.3.3.4 Froth depth

The function of the cleaning zone is to provide the opportunity for the rejection of entrained particles and so obtain the cleanest possible product. The froth depth must, therefore, be sufficient to allow for this rejection. It has been found that rejection of entrained particles occurs very close to the pulp/froth interface. In one particular study, it was determined that 95% of entrained particles were rejected in the first 50 cm of froth (Yianatos et al., 1988). Other than this, no general rule has emerged regarding froth depth. Typical froth depths in plant operation are 0.5 to 1.5 m and there is little effect on performance over this range (Yianatos, 1989).

There are, however, some cases where the froth depth does have some importance. In a study on the flotation of fluorite ore (Ynchausti et al., 1988), it was found that increasing the froth depth to 3.5 m improved the grades of the fluorite concentrates, but no link with recovery was found.

There are other cases where froth depth can affect cleaning action. For example, when particles differing in hydrophobicity need to be separated, the froth depth might have a bearing on selectivity. A study by Yianatos et al. (1988) on the selective flotation of Mo from a Mo/Cu/Fe/Si ore showed that a froth depth of over 1.0 m was required before selective recovery of Mo was obtained.

2.3.4 Froth carrying capacity

2.3.4.1 Definition

If the feed rate to a column flotation cell is increased, the concentrate recovery rate will increase until a certain point, after which it will level off, even though the feed rate is still increased. This occurs because, at a given superficial gas rate and bubble loading, there is a certain limiting mass rate of solids that can be accommodated in the froth. This is called the maximum carrying rate.

The practical measure of the maximum carrying rate for a given set of conditions is called the carrying capacity. An expression which relates carrying capacity, C_a, to particle size and gas rate is the following (Finch and Dobby, 1990, pp 90-97):

$$C_a = K_1 \rho_p d_p \, [\, J_g^{(1-q)} \,] \quad \text{[4.8]}$$

where K_1 is the fractional monolayer bubble loading and q is a constant. It is clear that the carrying capacity is directly proportional to particle density and particle diameter. This last proportionality might seem strange but it must be remembered that larger particles will have a larger mass for a given surface area than fine particles. Thus, for the same bubble coverage, a greater mass is recovered.

C_a is also proportional to a power moderated function of the gas rate. Studies with laboratory and pilot scale columns have revealed that C_a is largely independent of gas rate and the value of q can be taken as 1 for J_g in the range 1.5 to 3 cm/s (Finch and Dobby, 1990, pp 90-97).

An empirical model may be used for estimating the carrying capacity for a given set-up. One such empirical relationship is as follows (Espinosa-Gomez, 1988):

$$C_a = 0.041 d_{80} \rho_p \quad (\text{t/h/m}^2) \quad \text{[4.9]}$$

Because it is an empirical model, it only applies within the range of variables tested. In this case, the upper limit on the value of d_p is 40 μm.

Carrying capacity is also affected by the "lip capacity" of a cell, which results from geometric factors and physical characteristics of a concentrate in the froth zone. Lip capacity is normally determined empirically for a particular column installation (Amelunxen, 1990).

For design and scale-up purposes, the values for C_a must be determined experimentally. This would be done by measuring the concentrate solids rate as a function of the feed solids rate for a given column set-up and operating conditions.

2.3.4.2 Carrying capacity constraints

In order to maximize the product rate from a particular flotation column, it would seem logical to operate the column at (or close to) its carrying capacity. However, problems can arise with this strategy.

A column operated at its carrying capacity is sensitive to fluctuations in the feed. For example, an increase in feed grade or pulp density will increase the feed rate of the floatable component in the feed. If the column is already at its carrying capacity, this will mean a recovery loss as the extra solid material will exceed the carrying capacity and will not be recovered. The column will then be carrying capacity limited. This is an undesirable situation because correcting this condition is not straightforward. Decreasing the overall solids feed rate to maintain a constant feed rate of the floatable material is not a very practical solution because of the difficulty of measuring the grade fluctuation on-line to incorporate into a control strategy (Finch and Dobby, 1990, pp 90-97).

Generally speaking, a column should be operated close enough to the carrying capacity so as to obtain the best possible recovery but sufficiently far to allow for variations in feed grade.

2.4 Factors affecting column design

2.4.1 Column residence time

When designing a new column or optimizing an existing installation, it is necessary to know the kinetic behaviour of the ore to be floated in order to determine the required retention time, or residence time of the slurry in the column. A model to predict the rate of particle collection has been proposed (Finch and Dobby, 1990; Dobby and Finch, 1986). The basis of this model is an expression for the fractional recovery of the mineral, R_c, as a function of the residence time, based on a first order rate law. This recovery expression is dependent on two factors:

1) The rate constant, k_c
2) The degree of mixing.

The overall rate constant, k_c, is expressed in terms of E_k, the collection zone efficiency; the gas rate, J_g; and the mean bubble diameter, d_b:

$$k_c = \frac{1.5\, J_g\, E_k}{d_b} \qquad [4.10]$$

The E_k term contains the effects of factors such as particle size, particle hydrophobicity, and particle density.

The degree of mixing determines the form of the first order rate law for solids recovery. The collection zone can have two extremes of mixing: if it is assumed to be an ideal, plug flow, reactor then the rate law is of the form

$$R_c = 1 - \exp(-k_c \tau_p) \qquad [4.11]$$

where τ_p is the particle residence time in the collection zone which is the same for all particles.

If, on the other hand, the collection zone is assumed to be a perfectly mixed reactor, then the rate expression is as follows:

$$R_c = 1 - \frac{1}{(1+k_c\tau)} \qquad [4.12]$$

In this case, there is a distribution of residence times, so the parameter τ represents an average residence time for the particles.

Strictly speaking, these two equations represent the fractional recovery of a mineral of a particular particle size. Since different size particles will have different residence times, average values are used for τ_p, τ and k_c for a size distribution of particles being floated. In both these models, the residence time refers to the average particle residence time. In practice, it is easier to measure the liquid residence time, given by:

$$\tau_l = Q_t / V_c \qquad [4.14]$$

where Q_t is the tails liquid rate and V_c is the collection zone volume. The particle residence time can be related to τ_l by the following equation (Dobby and Finch, 1986):

$$\tau_p = \frac{\tau_l(u_l)}{u_l + u_p} \qquad [4.15]$$

where u_l is the liquid velocity and u_p is the average particle settling velocity.

In actual column operation, the flow will be between these two mixing extremes. In laboratory or pilot columns, where the column diameter is typically less than 10 cm, the flow is a close approximation to plug flow. In industrial scale columns, however, there will be a degree of mixing. In order to determine the actual rate of recovery, the degree of mixing has to be determined.

In theory, mixing has a detrimental effect on recovery. This is illustrated in Figure 4.5 which shows the relationship between R_c and residence time (τ) for plug flow (upper curve) and mixed flow (lower curve) using equations 4.11 and 4.12 respectively with the same value of k_c. It is clear that the closer the column approaches plug flow behaviour in the collection zone, the better the recovery for a given residence time.[1]

Because of the asymptotic behaviour of the recovery-residence time functions (see Figure 4.5), the residence time will be chosen on practical as well as performance criteria. For

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[1] This argument assumes that the rate constant does not change. However, in reality the value for the rate constant might be higher if the collection zone is more mixed, so the detrimental effect of mixing might be moderated.

a given column diameter, the larger the length of the collection zone, the larger will be the collection zone volume and thus the residence time for a given tailings rate. The residence time must be sufficient to obtain an acceptable recovery but constraints on column size must also be met.

For different ores, the value of k_c will be different. Some ores will be fast floating, while others will be slow. A smaller k_c value will mean a longer residence time is required to achieve the same recovery as for a faster floating ore with a large k_c. Coal is an example of a fast floating ore—typical residence times for coal flotation columns are 5 to 6 minutes. For a mineral ore like pyrite, residence times of 13 to 15 minutes are common and some other minerals may require even longer residence times for acceptable recovery.

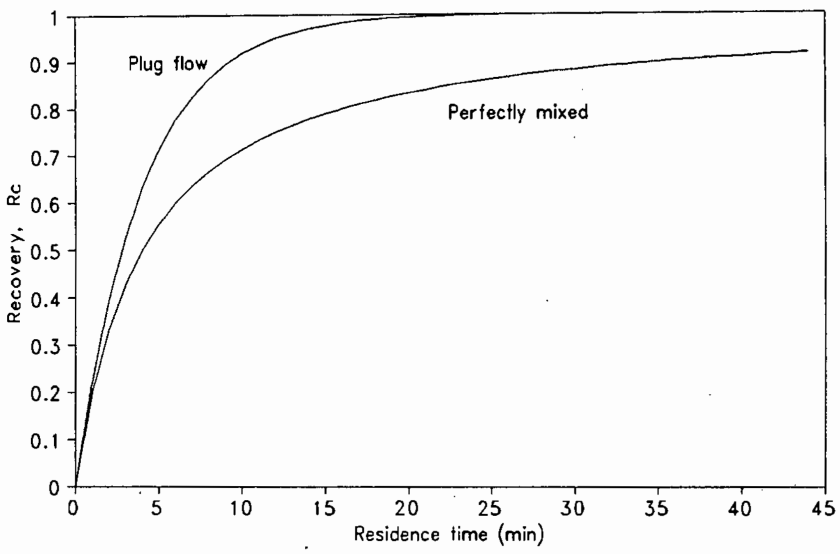

Figure 4.5: Effect of collection zone mixing on recovery.

2.4.2 Length to diameter ratio

The collection zone height and, in particular, the column length to diameter ratio (L/D) have a significant effect on column performance. Large L/D ratios result in less axial mixing which means a closer approach to plug flow behaviour. This will result in a greater recovery, as has already been discussed in section 2.4.1, and possibly a small improvement in grade. As L/D ratios increase above 10, the effect becomes progressively smaller (Finch and Dobby, 1990, pp 68-73).

For laboratory and pilot scale columns which have a diameter of 10 cm or less, the collection zone behaviour is close to plug flow. Large diameter columns may be baffled to obtain the desired L/D ratio without resorting to very tall columns (a 3 m diameter column would have to have a collection zone height of 30 m to obtain an L/D of 10). With baffles, the column effectively consists of many small diameter columns. This solves the problem of height restrictions as well as minimizing the detrimental effect of mixing.

2.5 Coal beneficiation in column cells

In the last five years, column cells have been investigated for coal beneficiation by a number of researchers in the U.S.A., India, Australia, and South Africa.

Reddy et al. (1988) compared the performance of 3 columns of different diameter with that of a conventional cell, using two samples of high ash coal (25.3 and 31.0% ash). The results for two columns are presented in Figure 4.6. As can be seen, the cleaning obtained in both column cells was superior to the conventional cell: a much better product grade (lower % ash) was obtained in the column cells at similar or greater yields.

In an investigation by Abdel-Khalek and Stachurski (1990), an comparison was made of column and conventional cells for the flotation coals of different rank and degrees of oxidation. It was found that, in all cases studied, the column cell gave consistently higher product yields at a particular grade (ash content) than the conventional cell. This improved cleaning was attributed to two factors; 1) the increased probability of particle-bubble collision in the collection zone of the column cell, and 2) the high selectivity and better cleaning action of the froth zone in the column cell.

The column cell has also been successful for producing a low-ash product at a good yield from plant refuse. In a study performed on refuse coal streams from four coal preparation plants in Kentucky (Parekh et al., 1988), it was found that refuse samples containing 30 to 50% ash could be upgraded to give a product containing 2 to 8% ash at up to 94% coal (combustible) recovery. Further work by these researchers has resulted in the design and installation of an industrial scale column plant at the Powell Mountain Coal Co in Lee County, VA., which is the first commercial application of a coal column circuit (Groppo and Parekh, 1990). A marketable product of 5 to 8% ash is produced from a -0.15 mm refuse stream of 40 to 60% ash. This circuit increases the overall plant yield by 2 to 3%.

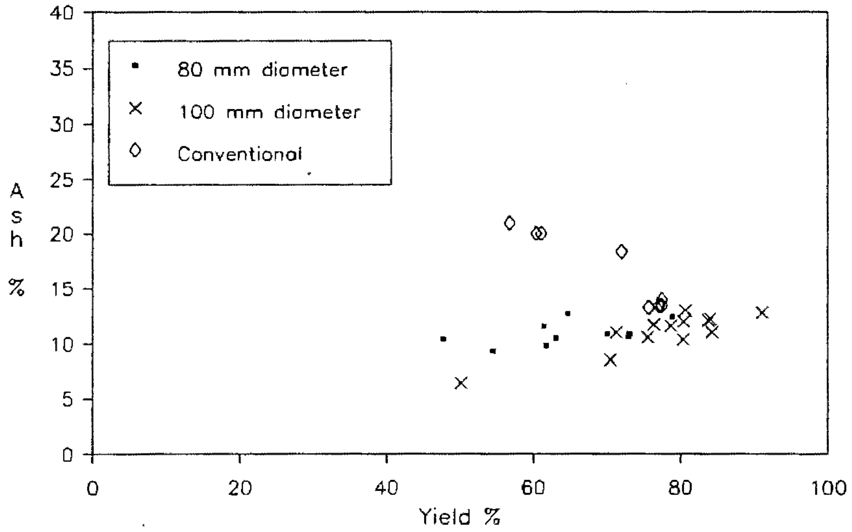

Figure 4.6: Coal flotation in 2 column cells of different size compared to a conventional flotation cell (Reddy et al., 1988).

Two general results emerge from these studies. The first is that the column cell consistently gives better cleaning than conventional cells. This is a direct result of the reduction of entrainment in the column cell. The second is that the product rate that is attainable in a coal column is much smaller compared to mineral ore flotation. For example, the industrial column at Powell Mountain Coal Co. (Groppo and Parekh, 1990) produces 0.3 to 0.45 $t/h/m^2$ (5.4 to 7.3 t/h), which is considerably less than, for example, a pyrite column where the product rate is in the range 4 to 5 $t/h/m^2$. This highlights a major problem with coal flotation in columns: a limitation of the product rate, or throughput, because of a high mass recovery in the concentrates and a low column carrying capacity.

2.6 Discussion

2.6.1 Summary of literature review

In the preceding sections, a number of parameters and factors have been identified as being important in determining the behaviour and metallurgical performance of a column flotation cell. These are listed below with typical operating values (where applicable):

- Gas rate, J_g: 1.5–3 cm/s
- Bias rate, J_b: 0.1–0.3 cm/s
- Frother dosage: affects bubble size
- Bubble size: typically 0.8–2 mm dia..
- Feed solids rate: 0.5–5 $t/h/m^2$
- Pulp density: 5–30%
- Froth height: 0.5–3 m (usually 0.5–1 m)
- L/D ratio: >10
- Sparger type and design
- Collection zone residence time/collection zone length
- Froth carrying capacity

The operating ranges for the parameters listed above will be used as a starting point for the experimental testwork. In addition, the effect of selected parameters on column performance will be investigated.

2.6.2 Reverse flotation: a means of improving throughput in column cells

The literature has revealed that column cells are effective in reducing entrainment. With coal flotation, however, there is still the problem of high mass recovery. The carrying capacity of a column cell places an upper limit on the concentrate overflow rate. It has been seen (section 2.3.4) that carrying capacity is a function of particle density, and because of the low density of clean coal particles, the carrying capacity of coal flotation equipment is very low: for a given bubble surface area (in the froth) that is covered by attached particles, the mass that this coverage represents is much less for coal particles than for mineral ores such as pyrite.[1]

The low carrying capacity, coupled with the high mass recovery, places a severe limit on the throughput of coal flotation equipment and in particular, the column cell. If the mass recovery in coal flotation could be reduced and/or the carrying capacity could be improved, then both these throughput limitations inherent to a coal flotation column would be reduced. This could be achieved by operating a reverse flotation column at the same (or higher) concentrate recovery rate as for forward flotation. Because the gangue material in a coal feed makes up the smaller proportion, the tails product rate in reverse flotation would be much higher than the concentrate product rate in forward flotation. This, in turn, means that the feed rate to the reverse flotation column could be higher and thus the throughput would be improved. The actual improvement would, of course, depend on the relative proportions of gangue and coal in a particular feed.

3. Flotation samples, equipment and procedures

Reverse flotation experiments were performed in a laboratory column cell using artificial mixtures of coal and quartz as feeds. Some forward flotation runs were performed in the same laboratory column cell, for comparison. The sections which follow describe the column cell and its ancillary equipment and give details of operating samples and procedures.

3.1 Flotation samples

The artificial mixtures used in the column cell experiments consisted of blends of washed low-ash coal and quartz samples as used in the batch flotation experiments (Chapter 3). The coal sample used was the 9.2% ash sample. This was milled to 98% –0.15 mm as for the batch flotation work (see Chapter 3), except that a larger, 2.5 kg capacity (rod) mill was used. The milling time to obtain the required size fraction is given in Appendix II. The quartz sample was the sample of Delmas Silica used in all the previous test work. (see Chapter 2, section 5.1.1.2).

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[1] The recovered coal particles typically have a relative density (R.D.) of about 1.3 to 1.35, whereas pyrite has an R.D. of 5.4. For a given size fraction of particles, the carrying capacity for coal will be approximately four times smaller than for pyrite.

Four feed mixtures were used, containing 17%, 25%, 33% and 50% quartz by mass. The respective feed ash contents were 24.4%, 31.9%, 39.4% and 54.6%.

3.2 Equipment

The equipment used in the column flotation testwork was already constructed in the Department of Chemical Engineering at the University of Cape Town, and had been used successfully in previous (forward) coal flotation projects. The equipment comprised the column, which could be fitted with either internal or external spargers, and ancillary equipment such as pumps, tanks and stirrers. Except where indicated, the same equipment was used in all the flotation tests reported below.

3.2.1 Column cell

The laboratory scale column was constructed of flanged 600 mm lengths (sections) of perspex pipe with an internal diameter of 53 mm. By removing or inserting sections, the length of the column could be varied as desired. Certain sections of the column were manufactured with threaded ports for the feed pipe attachment, sparger insertion and tailings removal.

Figure 4.7 is a schematic diagram of the column. As illustrated, the top section of the column consisted of a froth overflow weir and a concentric launder with a base angled at 18° to the horizontal to facilitate froth flow. The top section of the column was covered by a perspex lid. The lid had a diameter slightly larger than the froth launder and a center hole which allowed for the insertion of a wash water distributor into the froth zone below the weir level. The launder lid was also fitted with a concentric rinse sprinkler. This was fixed in such a way as to irrigate the froth only in the launder (and not in the froth zone of the column) in order to promote froth breakage and concentrate removal. A second concentrate launder placed just below the outlet of the first launder. This had a pipe attachment at the base and a length of hosing allowed the concentrate to flow into a sampling bucket.

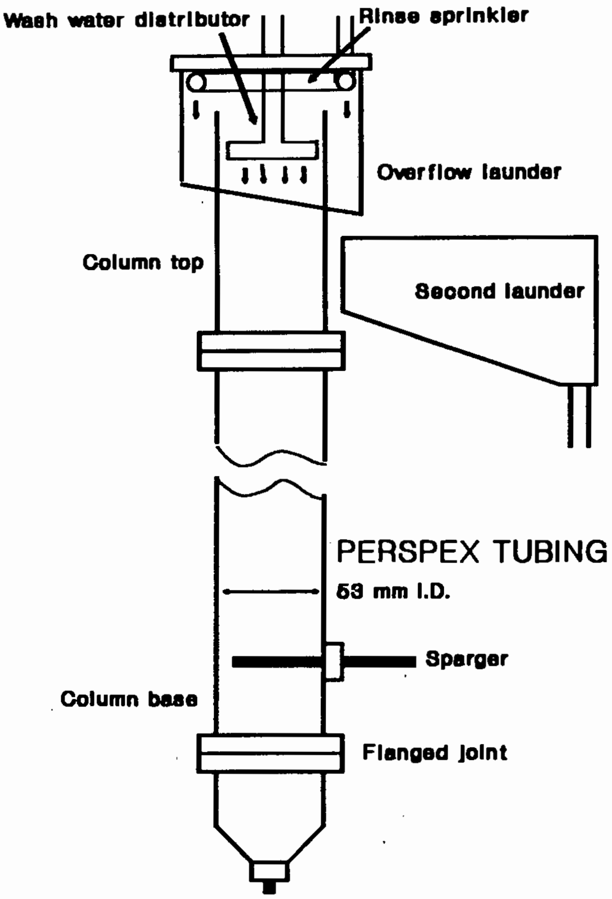

Figure 4.7: Schematic of the experimental column

The wash water distributor was constructed from 7 mm o.d. copper tubing, consisting of a 300 mm vertical length and 4 sprinkler arms attached to the base, projecting radially from the center pipe and spaced at 90 degrees from one another, i.e. in the form of a cross. Each arm was 22 mm in length and had five equidistant 1 mm holes (on the bottom) for water distribution. The penetration depth of the sprinkler arms into the froth bed was set by simply moving the vertical pipe up or down in the center hole of the column lid. Unless otherwise specified, the wash water distributor was set at a depth of 2 cm below the weir for all column runs.

The feed pipe insertion port was located approximately one-third of the way from the top of the column, the sparger port approximately 15 cm from the bottom of the column, and the tailings removal hose was fitted to the base of the column. This hose was connected to the tailings pump which was used to control the pulp/froth interface height. This was done by manually adjusting the pump flow rate to (visually) align the interface level with a mark on the outside of the column.

3.2.2 Air spargers

Two types of air spargers were used in the testwork: a filter cloth sparger and an external sparger based on the United States Bureau of Mines design (USBM). These are illustrated in Figure 4.8.

The filter cloth sparger consisted of a short length of 15 mm i.d. PVC pipe with a number of 4 mm holes drilled into it. Filter cloth was wrapped around the pipe and glued into place. The base of the sparger had a 20 mm o.d. threaded section which allowed it to be screwed into the column port just above the base of the column.

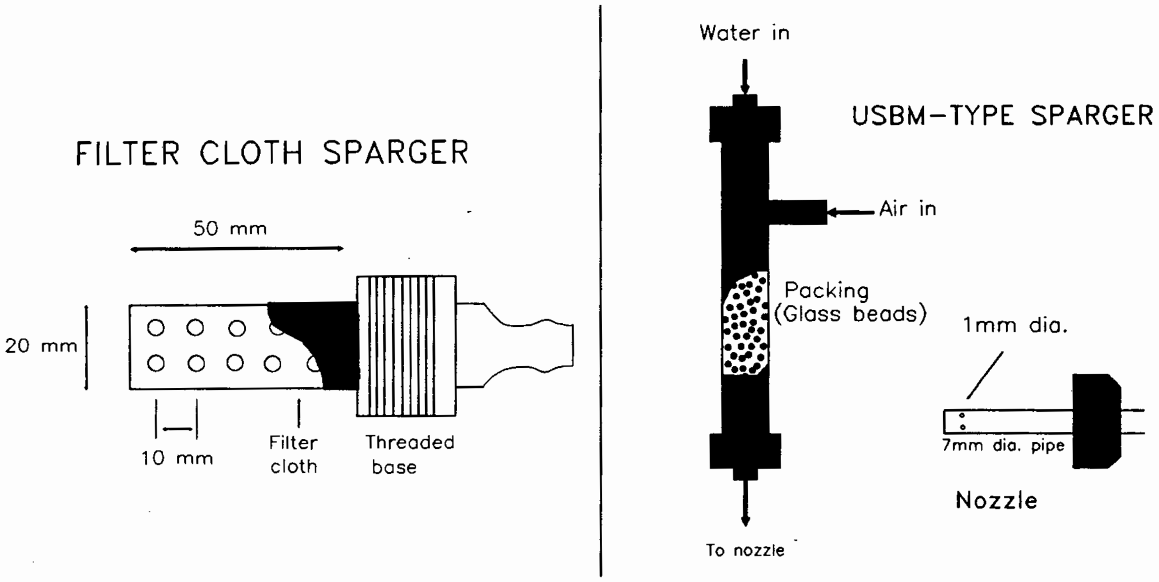

Figure 4.8: Air spargers used in the column flotation testwork.

The USBM sparger consisted of a T-piece joint in which the air and water supplies were joined at right angles to each other. The T-piece was packed with 2 mm glass beads to improve mixing but a separate contacting zone (as is usual with this type of sparger) was found to be unnecessary. A short section of 6 mm i.d. hosing fed the water/air mixture into the column. This hosing was sealed off at the end inside the column and three 0.8 mm nozzle openings were made in the base of the pipe so that the water/air stream was directed downwards as it entered the column.

The air rate to the spargers was controlled with a gas rotameter and two pressure regulators. A regulator upstream of the rotameter was used to reduce the line pressure from 6.5 bar to 4 bar. A second regulator was placed downstream of the rotameter, just before the sparger entry point. The desired flow rate was obtained by adjusting the rotameter valve and the downstream regulator valve so that the desired rotameter reading was obtained at the sparger operating pressure. The rotameter was calibrated at 3 bar using a soap film meter and a stopwatch.

3.2.3 Ancillary equipment

Figure 4.9 is a diagram of the entire rig showing the equipment layout. Also shown are the ranges in which certain column parameters were varied.

A 30-litre, square section, baffled plastic tank was used to contain and condition the feed pulp. The solids were suspended by agitating the pulp with the impeller from a laboratory Denver cell. The base of the tank was fitted with a valve and a detachable feed hose which enabled the tank to be cleaned and filled separately and then connected up to the column when a run commenced.

Variable speed peristaltic pumps were used for feed addition and tailings removal. The feed pump was a Watson Marlow 501R model. This had a maximum feed rate of 0.6 l/min using 6 mm (neoprene) pump tubing. For each run, the feed rate was set at the desired value and was subsequently not varied during the course of the run. Two tailings pumps were used, depending on the sparger type. For the filter cloth sparger, a Watson Marlow 503S pump was used. This had a maximum flow rate of 2 l/min, using 6 mm pump tubing. When using the USBM-type sparger, a higher capacity Masterflex peristaltic pump was used, with a maximum flow rate of 13 l/min. This was necessary because the addition of sparger water to the column increases the tailings rate to beyond 2 l/min.

The municipal (potable) water supply to the wash water distributor and rinse sprinkler were fed through 14K and 14S rotameters (metric) respectively. The water supply for the USBM-type sparger was obtained from a 200 l polyethylene tank containing a mixture of water and frother. A centrifugal pressure water pump (maximum pressure 5 bar) was used to deliver water to the sparger through a 14S rotameter. A water pressure regulator was placed upstream of the rotameter which enabled the water supply pressure to be set at the sparger operating pressure.

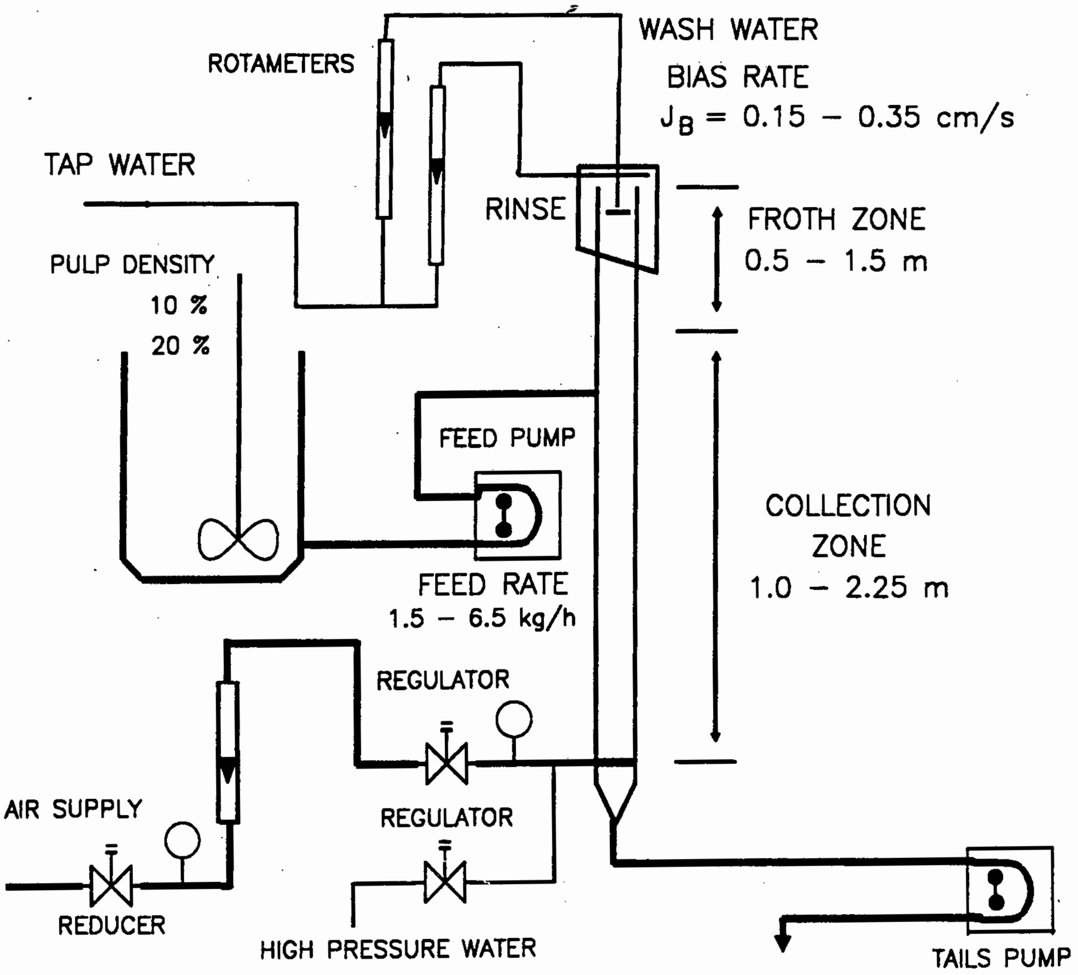

Figure 4.9: Layout of entire flotation rig

3.3 Reagents

3.3.1 Reverse flotation

The quaternary amine Hexadecyl-trimethyl ammonium bromide (HTAB) was used as the ash collector and frother for all the column flotation runs. This reagent was found in the batch flotation testwork (Chapter 3) to be the most effective ash collector and frother for reverse flotation.

The Type IV, 90% water soluble dextrin (described previously in Chapter 2, section 5.1.3) was used as the coal depressant in all the testwork, added at a concentration of 800 g/t, based on the amount of LAC in the feed

3.3.2 Forward flotation

The coal collector used in the forward flotation runs was "Shellsol A", an aromatic paraffin obtained from the Shell Chemical Company (S.A.). This reagent was the same as that used in the forward flotation experiments using the batch cell (Chapter 3). The frother that was used was DIBK (diisobutyl ketone, or 2,6-Dimethyl-4-heptanone), added at a concentration of 15 μl/l (based on the total water feed rate to the column).

3.4 Experimental procedure

Column flotation experiments were carried out according to a set procedure to try to ensure reproducibility throughout the testwork.

3.4.1 Feed preparation and conditioning

For a specific set of experiments, sufficient coal was milled (10 to 15 kg) and individual artificial mixtures were prepared by blending the required amounts of coal and quartz in large plastic bags with the aid of a top loading 8 kg laboratory balance. The total sample mass was either 2 kg for a run in which the pulp density was 10%, or 4 kg for 20% pulp density. In order to inhibit oxidation, as much air as possible was squeezed out of the bags, which were then sealed until required. A set of samples was usually consumed within two days.

At the start of each run, the feed tank was filled with the required volume of water: 18 l for runs in which the pulp density was set at 10%, or 16 l for the 20% pulp density runs (i.e. the total feed was 20 kg). The impeller was then placed in position and switched on. The coal sample was added from a plastic bag and the slurry was agitated for a period of 10 minutes to ensure that the coal slurry was well mixed.

3.4.2 Reagent addition

3.4.2.1 Reverse flotation

The required mass of dextrin and HTAB, respectively, were weighed on a Mettler four-figure balance and each was dissolved in a beaker of warm water. The dextrin was added to the feed tank after the initial 10 minute agitation period and allowed to condition with the feed for a period of 8 minutes. The amine solution was then added and the pulp conditioned for a further period of 8 minutes. At the start of this second conditioning period the column feed hose was connected to the tank outlet, the end of the hose was placed in the tank, the tank outlet valve was opened, and the feed pump was switched on to allow the feed to recirculate.

3.4.2.2 Forward flotation

After the initial 10 minute agitation period, the required volume of Shellsol A was added by means of a Pipetteman automatic pipette. The slurry was allowed to condition and recirculate for 8 minutes, after which the DIBK frother was added (automatic pipette). A further 2 minute conditioning time was allowed.

3.4.3 Column start-up

During the feed conditioning period, the column was partially filled with water (via the wash water distributor) in order to submerge the sparger; the air was turned on and the desired flow rate was set. At the end of the second conditioning period, the feed pump was temporarily switched off and the feed hose was connected to the column. The sparger water pump was switched on and the flow rate set on the rotameter (USBM sparger). The feed pump was then switched on, the wash water and rinse rates were set and the column was allowed to fill to the desired pulp level.

The pulp-zone length was measured from the feed inlet to the sparger position. The froth zone height was measured from the pulp interface to the froth weir. In all the experiments, a 200 mm section of pulp was maintained above the feed inlet to the pulp interface level. This was done in order to provide a zone whereby partially hydrophobic particles in the feed would be discouraged from entering the froth zone.

The froth height in the column was controlled by manually adjusting the tails pump rate to maintain a constant pulp interface level. Once the pulp had reached the required level in the column, the tails pump was switched on and the flow rate was adjusted so that the pulp/froth interface level remained constant. The column was allowed to reach steady state over a period of about 12 minutes, during which time fine adjustments to the tails pump rate were made to maintain a constant pulp level.

3.4.4 Operation and sampling

Unless otherwise specified, the feed rate was set at one of two values, either 1.5 or 3 kg per hour. This corresponded to 0.68 or 1.36 $t/h/m^2$ (based on a column cross-sectional area of 22.1 cm^2). The pulp density was 10% or 20% solids depending on which of the two flow rates was used. The wash water rate was varied in the range 0.35 to 0.6 l/min (which translated to a J_w value of 0.26 cm/s to 0.45 cm/s). The air rate was varied between 1.8 and 3.0 cm/s. The rinse rate was 0.7 to 1 l/min (this was kept constant during a specific column run).

A feed sample was taken during the conditioning period by alternately directing the flow of slurry into a small beaker and back into the tank for short periods over two minutes to ensure that the sample was representative of the slurry.

For each run or set of conditions, sampling was performed by taking simultaneous tailings and concentrate samples over a total period of ten minutes. Sampling was done in two minute intervals, with a two minute break in between, so the actual sampling time was six minutes. For some of the runs, the sampling time was shorter, namely five minutes total time with one minute sampling alternating with a one minute break to give an actual sampling time of 3 minutes. If required, the conditions such as air rate, pulp level and wash water rate were then reset to different values and, after a further 12 minute period to allow for steady operation, sampling was again performed.

At the end of a run, the column was shut down by turning off the feed pump, air supply and sparger water (if applicable) and setting the tails pump to full speed to drain the column. Any coal adhering to the column walls was flushed out by setting a very high wash water rate.

3.4.5 Sample analysis

The tailings and concentrate samples were weighed to obtain their respective slurry masses and then were filtered in a pressure filter and dried in an oven at 80° overnight, and the dry masses recorded. The samples were then ashed according to the method outlined in Appendix V. The recoveries were then calculated on a spreadsheet. The detailed calculations are shown in Appendix VIII.

3.4.6 Sieving

For some of the runs, size distributions of feed, tailings and concentrate samples were determined by sieving. This was done wet, using an electric sieve shaker.

Before each sieving operation, each sieve was cleaned of solid matter by placing it in an ultrasonic bath for 20 minutes. The sieves were then loaded into the shaker and 50 to 80 g of dry material was placed on the uppermost sieve. The lid of the shaker was clamped into place and the shaker activated. The shaker lid contained a water sprinkler to irrigate the solid material—the water flow rate was set to provide continuous wetting without the sieves overflowing.

The underflow from the sieving process was collected in a bucket and at the end of the sieving process the solids were recovered by means of a pressure filter. The solid material in each size fraction was recovered in a buchner funnel. The wet samples were placed in an oven to dry at 80 °C overnight and the dry masses were recorded. The ash content of each sample was determined (Appendix V) and the size distributions were calculated (see Appendix XI).

4. Column flotation testwork

Reverse and forward flotation experiments were performed on artificial feed mixtures consisting of blends of LAC and quartz. The column flotation work was performed in three sections: Firstly, the optimum conditions were determined for reverse flotation of the artificial quartz/coal mixtures. Secondly, an entrainment study was performed using the artificial quartz/coal feeds to compare the cleaning obtained with reverse flotation in the column cell with results achieved in the batch cell. Thirdly, reverse flotation was compared with forward flotation (in the column cell) in terms of separation efficiency and carrying capacity.

4.1 Initial work: determination of optimum conditions

These runs were performed in order to find the optimum column configuration and operating conditions for the artificial quartz/coal feeds and to make a preliminary assessment of reverse coal flotation in column cells.

4.1.1 Experimental details

The feed used was a 50% quartz/coal blend with an ash content of 54.6% as used previously in the batch cell experiments. For all the runs the feed pulp density was 10%, the feed rate was 1.5 kg/h, the dextrin addition was 800 g/t (based on the mass of LAC in the feed), and the wash water rate was set at 0.4 l/min. This gives an approximate J_b value of 0.2 cm/s, using equations 4.5 and 4.6 with J_g = 2 cm/s and ε_{g0} = 0.8 (calculation shown in Appendix IX).

Experiments were performed over a range of parameters to find the best results in terms of recovery and grade (of coal in the tailings). The following parameters were varied in the indicated ranges:

Froth height: 0.5 to 1.3 m
Collection zone height: 1.0 to 1.8 m
HTAB concentration: 1200 to 2500 g/t
Gas rate (J_g): 1.5 to 2.8 cm/s

Column operation was facilitated by a stable froth phase and clear distinction between pulp and froth zones. The HTAB amine was found to produce a closely packed bubble bed with very little observable coalescence over the entire length of the froth bed. Manual control of the froth height was simplified by the clear colour distinction between the grey, quartz laden froth and the black, coal rich pulp zone.

4.1.2 Results and discussion

The results of 8 runs are presented in Table 4.1 below. More runs were performed but the results were so poor that they are not reported (these were all at air rates of less than 1.9 cm/s). The Table shows the tails product coal recovery and ash content, the concentrates quartz recovery, the collection zone and froth zone heights, the calculated bias rate, the gas rate, the liquid residence time and the HTAB concentration for each run.

It would appear that the collection zone height and/or froth height is the major factor determining the quartz recovery and the metallurgical performance of the column. Comparing runs 3 and 4 it can be seen that with a 0.8 m froth height and 1.6 m collection zone the quartz recovery was 77.13%, whereas with a 0.5 m/1.0 m configuration the recovery increased to 98.4% (all other parameters were constant).

It was found that an HTAB concentration of 1200 g/t (runs 5 and 6) was sufficient to obtain optimum beneficiation results. (This value was found by trial and error to be the smallest concentration that could be used to maintain a stable froth zone.) It can be seen that very similar recoveries and grades were obtained at 1200 g/t (e.g. run 6) and at 1820 g/t (run 4). In the previous batch cell experiments, the optimum HTAB concentration was around 600 g/t. The increased amine concentration requirement in the column cell is, in all likelihood, due the much deeper froth which requires a higher level of the amine surfactant to maintain its stability.

Table 4.1: Initial column results using artificial quartz/coal mixtures.

No.	Tails coal rec (%)	Concs quartz rec (%)	Product ash %	Froth height (m)	Collection zone height (m)	Bias rate cm/s	Gas rate cm/s	Residence time (min)	HTAB conc g/t
1	98.25	56.18	26.46	1.30	1.80	0.150	1.95	7.50	2500
2	96.37	62.39	29.86	0.80	1.60	0.157	1.95	6.45	2260
3	95.95	77.13	21.50	0.80	1.60	0.112	2.32	7.36	1820
4	92.10	98.40	11.08	0.50	1.00	0.069	2.32	5.44	1820
5	90.08	97.78	11.63	0.50	1.00	0.118	2.32	4.40	1200
6	91.40	99.40	10.01	0.50	1.00	0.087	2.26	4.79	1200
7	92.87	97.46	11.12	0.50	1.00	0.147	2.26	3.98	1636
8	93.39	98.62	10.40	0.50	1.00	0.165	2.04	3.65	1636

The residence time for the runs varied from 3.7 to 5.44 minutes (depending on the tails rate). It is obvious that the flotation kinetics are very fast, since the residence time has no effect on the concentrates quartz recovery; even at 3.7 min, which is a relatively short residence time for a column cell.[1]

Overall, the metallurgical performance of the column was extremely good at the configuration of a 0.5 m froth height and a 1 m collection zone (runs 4 to 8). The ash content was reduced from the feed of 54% to around 11%, the product coal recovery was 90 to 93%, and the concentrates quartz recovery was generally above 98%. The optimum conditions can thus be summarized as follows:

- Froth zone height: 0.5 m
- Collection zone height: 1.0 m
- HTAB concentration: 1200 g/t
- Dextrin concentration: 800 g/t
- Air rate (J_g): 2 – 2.5 cm/s
- Bias rate (J_b): 0.1 – 0.2 cm/s

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[1] Note: these are liquid residence times. Strictly speaking, the particle residence time should be calculated using equation 4.15 and estimating an average particle settling velocity, U_p. However, for comparison purposes, the liquid residence time was considered adequate, since the average velocity would be a constant for all the experiments.

4.2 Reduction of entrainment: column vs batch flotation

The reverse flotation experiments conducted in the laboratory batch cell (Chapter 3) showed that coal recovery in the concentrates (i.e. loss) was the result of entrainment. This was reduced by staged addition of reagent. The column cell is designed to eliminate entrainment, so reverse flotation experiments on the artificial quartz/coal feeds were conducted to investigate the validity of this claim.

4.2.1 Experimental details

In the batch cell testwork the effect of entrainment was investigated by carrying out a series of experiments in which the percentage quartz in the quartz/coal feeds was varied (see Chapter 3, section 4.2). In this way, the relationship between the concentrates overflow rate and the (undesirable) coal recovery in the concentrates was determined. A similar series of reverse flotation experiments was conducted in the column to compare the results with those from the laboratory batch cell.

Four feed mixtures were used as before, containing 16.7%, 25%, 33.3% and 50% quartz (by mass). The LAC used to make up the remainder of each feed type was the 9.2% ash sample. The resulting feeds had ash contents of 24.4%, 32.0%, 39.4% and 54.6% ash respectively.

Reverse flotation runs were performed on each feed mixture. For each run, column parameters were kept constant at the following values: the wash water rate was set at 0.3 cm/s, the air rate 2.3 cm/s, the feed rate was 1.5 kg/h, the pulp density 10%, the HTAB concentration was 1500 g/t (based on total feed mass) and the dextrin concentration 800 g/t (based on LAC mass).

4.2.2 Discussion of results

The results for the column runs are shown in Table 4.2 below. The Table shows the feed quartz content, the tails product coal recovery and ash content, the concentrates quartz recovery, the calculated bias rate (cm/s), the air rate, the concentrates overflow rate (t/h/m^2), the product (tails) rate, the concentrate water recovery, and the HTAB concentration (g/t). As can be seen the separation achieved in all the runs was very good. The concentrates quartz recovery was above 94% for all the feed mixtures and the maximum coal loss to the concentrates was just over 7% .

In order to check the extent to which entrainment of coal occurred in the column cell, the mass of LAC reporting in the concentrates was calculated for each of the four feed mixtures (the data can be found in Appendix XII). The data is presented in Figure 4.10, which shows the results for the column cell, and the results obtained in the previous batch cell experiments on identical artificial mixtures (see Chapter 3, section 7; and Figure 3.14). In this figure, the Y-axis shows the mass of LAC in the concentrate expressed on the basis of 100 g feed in each case, so the two sets of data can be compared directly. It can be seen that in the batch (sub-aeration cell), the amount of coal recovered in the

Table 4.2: Effect of quartz feed content on Reverse flotation performance using artificial mixtures.

Quartz %	Coal rec (%)	Quartz rec (%)	Prod ash (%)	BIAS RT cm/s	Air RT cm/s	OVFLW t/h/sqm	PROD Rt $t/h/m^2$	Water rec. kg/h	HTAB g/t
16.7	93.34	98.08	9.58	0.210	2.30	0.158	0.557	6.71	1500
25.0	92.77	94.90	10.82	0.189	2.30	0.206	0.506	8.33	1500
33.0	95.17	96.02	10.95	0.171	2.30	0.242	0.472	9.80	1500
50.0	93.74	98.33	11.02	0.149	2.30	0.358	0.340	11.58	1500

concentrates increased as the proportion of quartz in the feed increased, indicating that the coal was recovered by a mechanism of entrainment or entrapment (due to the increased quartz recovery in the concentrates). In the column cell, the situation was different: the mass of LAC in the concentrates decreased from 5.35 g at 16.7% quartz feed, to 3.2 g at 50% quartz feed. Because less coal was recovered for each sucessive increase in the quartz recovery in the concentrate, it seems likely that coal entrainment had not occured in the column cell to any significant degree.[1]

It is of interest that entrainment of coal into the concentrate was not observed even with a steadily decreasing bias rate. From Table 4.2 it can be seen that the concentrate (solids) overflow rate increased from 0.158 to 0.358 $t/h/m^2$ as the feed quartz content increased from 16.7% to 50% and that this was accompanied by a decrease in bias rate (column 5, Table 4.2). This implies that there was an accompanying increase in the froth water recovery, since the wash water addition rate was kept constant and thus any increase in the concentrates water recovery would decrease the bias rate (which is defined as the net downward flow of water). This did, in fact, occur as can be seen from the water recovery data in Table 4.2 (column 9): the concentrate water recovery increased from 6.71 kg/h at 0.158 $t/h/m^2$ solids overflow, to 11.58 kg/h at 0.358 $t/h/m^2$. Entrainment is usually linked to water recovery—if any coal entrainment was occurring then one would expect the effect to be successively more severe as the water recovery increased. Since this did not happen, it can only be concluded that the column is very effective in eliminating entrainment.

It may be observed from Table 4.2 that the product grade declined as the feed quartz content increased. A possible reason for this is the following: Although the quartz recovery (concentrates) and the coal recovery (tails) for the 50% quartz feed were virtually the same (respectively) as for the 16.7% quartz feed, (and thus it would be expected that the grade of the tails product should be the same) it should be noted that the actual

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[1] In theory the LAC recovery in the concentrate should have been zero, since it is assumed that the LAC is perfectly depressed. In reality, however, some residual LAC recovery did occur in both the column and the batch cell, over and above that which was entrained.

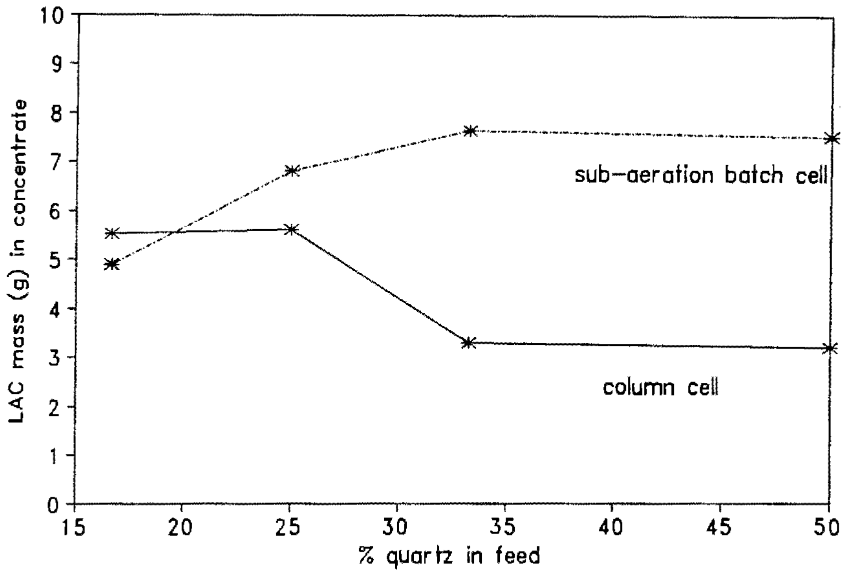

Figure 4.10: Reduction of entrainment: column cell vs conventional batch cell flotation (cf. Figure 3.13).

amounts of quartz and coal recovered differed greatly for the two feeds. For the 50% mixture, 2% quartz remaining in the tails is a larger mass than 2% remaining in the tails of the 16.7% quartz mixture (because the percentages are expressed on the feed amounts). Similarly, 93% coal reporting to the tails of the 50% mixture is a smaller amount (mass) than the same percentage of the 16.7% mixture. Thus, the mass ratio of quartz to LAC in the tails was different for the 50% feed than for the 16.7% feed. The product grade would be expected to decline as the amount of feed quartz increased because of the greater amount of quartz relative to LAC reporting to the tails.

On the basis of the above results it may be concluded that the column did result in a reduction or elimination of entrainment in comparison to the batch cell, as was claimed. This was most obvious where the percentage of quartz in the feed was high (33% to 50%), since entrainment was a significant factor in the batch cell in this region.

4.3 Comparison: forward vs reverse flotation.

In the previous section, reverse flotation in the column cell was compared with reverse flotation in the conventional cell using the artificial quartz/coal feeds. The claimed advantage of column flotation, i.e. the reduction or elimination of entrainment, was shown to be valid. Coal loss was low and constant, even with the feed quartz percentage at 50%.

In this section, reverse flotation in the column cell is compared with forward coal flotation in the column cell. Here the aim is to investigate the hypotheses that reverse flotation will result in increased solids throughput capability, leading to greater clean coal production rate, compared to forward flotation. The separation efficiencies of both processes are evaluated, the effect of increasing the feed pulp density in each process is compared, and the column carrying capacities for the two types of coal flotation are determined.

4.3.1 Experimental details

In order to compare forward and reverse flotation directly, forward flotation runs were performed on four feed mixtures identical to those used in the previous section (containing 16.7%, 25%, 33.3% and 50% quartz respectively). The coal collector used was Shellsol A at a dosage of 750 g/t (based on mass of coal in feed). The frother used was DIBK at a dosage of 15 μl/l (based on the total water feed rate to the column). As with the reverse flotation runs, the feed pulp density was 10%, the feed rate was 1.5 kg/h, the air rate was 2.3 cm/s, the wash water addition rate was 0.3 cm/s, the collection zone height was 1.0 m and the froth height was 0.5 m.

Separation efficiencies of the two processes were calculated for the runs in which the feed contained 50% quartz. This feed was used for the calculations because the theoretical yield of concentrate and tails is the same for both forward and reverse flotation and thus the two processes may be compared on an equal basis. Separation efficiencies were calculated according to the formula given in section 4.4.2.2 below.

To compare the effect of increased feed pulp density on reverse and forward coal flotation, a series of runs was performed at a feed pulp density of 20%. Both reverse flotation and forward flotation experiments were carried out on two feeds, containing 16.7% and 25% quartz, respectively. These feeds were used because they are fairly representative of real coals in terms of ash content. The feed pump setting was kept the same as for the previous work and thus the feed rate was 3 kg/h because of the increased pulp density. All other column settings were the same as before.

As outlined in section 4.3.4.1 above, column carrying capacity can be determined by investigating the relationship between concentrates overflow rate and feed rate. In order to compare the column carrying capacity of reverse flotation and forward flotation, experiments were carried out using a 25% quartz/coal feed. The feed rate was varied from 1.5 to 6.7 kg/h for both forward and reverse flotation runs, at a feed pulp density of 20%.

4.3.2 Discussion of results

4.3.2.1 General comparison of forward and reverse coal flotation

The results for the forward coal flotation runs are shown in Table 4.3 below (this table shows the data in the same format as in Table 4.2). If Table 4.3 is compared to Table 4.2, it can be seen that forward column flotation resulted in a higher product coal recovery and better grade than does reverse flotation. In fact, the grade in forward flotation (8.7% ash in all four runs) was better than the feed LAC ash percentage (9.2%), which would represent an ideal separation. This indicates that, as well as rejecting all the hydrophilic quartz, forward flotation in the column results in a beneficiation of the

Table 4.3: Variation of feed quartz percentage
Forward flotation

Quartz Percentage	Coal rec (%)	Quartz rec (%)	Prod ash (%)	Bias Rt cm/s	Air RT cm/s	OVFLW t/h/m^2	Water rec. kg/h	SSol A g/t
16.7	98.89	100.00	8.70	0.263	2.30	0.574	0.87	1000
25.0	96.42	100.00	8.70	0.277	2.30	0.456	1.37	1000
33.0	96.78	100.00	8.70	0.286	2.30	0.412	0.68	1000
50.0	96.42	100.00	8.72	0.293	2.30	0.313	0.13	1000

feed LAC . This is confirmed by the fact that the ash content of the concentrates was constant irrespective of the amount of quartz in the feed. This occured presumably because the LAC sample in the feed mixture was consistent for each run and thus the same quality of coal was floated in each run. At the same time, none of the quartz particles were recovered in the froth. (Note: it was assumed that no quartz was recovered in the concentrates, so the tails quartz recovery is reported as 100%.)

It may be observed that in forward flotation, the bias rate increased as the proportion of quartz in the feed increased. This is the opposite of reverse flotation (Table 4.2) but was to be expected. As the proportion of quartz increases, there is a corresponding decrease in the proportion of coal in the feed. The flux of coal to the concentrates decreases, recovery of feed water to the froth decreases, and thus the bias rate should increase (assuming constant wash water addition).

Further comparison of Tables 4.2 and 4.3 reveals a fundamental difference between reverse and forward coal flotation—the concentrates overflow rate for a given feed quartz content. For the 16.7% quartz/coal feed mixture, the concentrates (discard) overflow rate for reverse flotation was 0.158 t/h/m^2 in comparison to 0.574 t/h/m^2 for forward flotation (the clean coal product). In reverse flotation, the tailings overflow rate (product) was 0.557 t/h/m^2, which corresponds to the concentrates rate of forward flotation. This implies that if the concentrates overflow rate in reverse flotation (discard) was similar to that of forward flotation, then the product tails rate would be correspondingly higher. Using the 16.7% quartz feed as an example: if the reverse flotation overflow rate were identical to the forward flotation rate (0.574 t/h/m^2) then the tails rate would theoretically (by simple ratio) be 2.023 t/h/m^2. Since the concentrates rate is limited by carrying capacity, the reverse flotation process has an inherent advantage because the maximum attainable product rate would be considerably higher for the same carrying capacity as forward flotation (note: the 16.7% quartz feed is the best example—the effect becomes progressively less as the feed ash content increases). The carrying capacities for reverse and forward flotation are examined more closely in section 4.3.2.3 below.

The results in Tables 4.2 and 4.3 are compared graphically in Figure 4.11, which shows the product coal and ash content plotted against quartz percentage in the feed. The left Y-axis represents the coal recovery in the concentrates of forward flotation and the tailings of reverse flotation which, in both cases, is the product. As can be seen, forward flotation resulted in the greater coal recovery; recovery was over 96%, whereas in reverse flotation the recovery was in the range 92 to 96%. Forward flotation also produced a better product in terms of ash content: 8.7% compared to 9.6 to 11%. This was to be expected: coal is naturally hydrophobic and would prefer to report to a flotation concentrate, while quartz is naturally hydrophilic and would prefer to remain in the tails. Reverse flotation is contrary to Nature, whereas forward flotation conforms to Nature. Nevertheless, in absolute terms, the differences in performance between the two processes were relatively small; both process were extremely efficient at separating the quartz from the LAC.

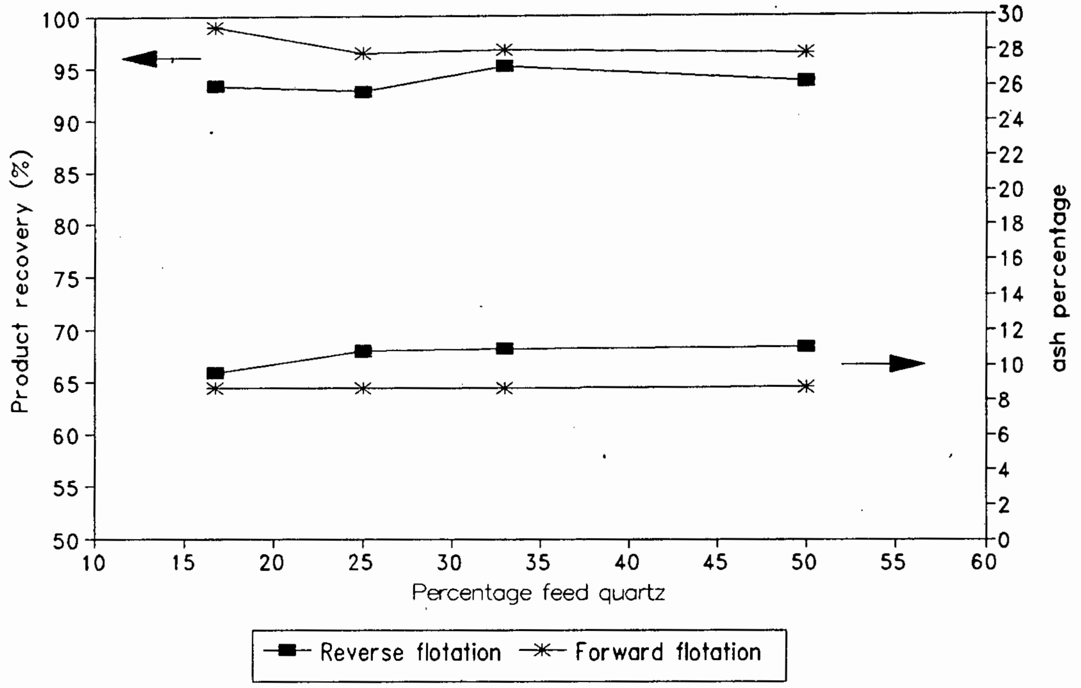

Figure 4.11: Comparison of reverse flotation and forward flotation performance in the column cell.

If the results in Figure 4.11 are compared to the equivalent results in Chapter 3 (Figure 3.13) for the batch cell, it can be seen that the efficiencies were better in both processes in the column cell. In reverse flotation, the coal recoveries in the tails products were greater (93%–95% vs 85–94% for batch), while the grades were very similar. In forward flotation, the product coal recoveries (concentrates) were much greater (96.4%–98.8% vs 88.4%–89.7% for batch) and the grades were better (8.7% ash vs 8.95%–10.3% ash). It may be concluded that the performance of both processes was improved in the column cell, with forward flotation giving the best overall separation.

4.3.2.2 Separation efficiency

In order to quantify the performance of reverse and forward flotation, separation efficiencies were calculated for the runs in which the 50% quartz/coal feeds were employed. These efficiencies were calculated as follows:

Size analyses were performed by wet sieving the LAC coal sample and the 50% quartz/LAC feed, and the tailings and concentrates of 50% quartz/coal reverse and forward column flotation runs. The size ranges chosen were:–0.025, +0.025 –0.038, +0.038 –0.052, +0.052 –0.075, +0.075 –0.125 and +0.125 mm. The ash content of each size fraction was determined. From the analyses of the feed, a theoretical "ideal" LAC yield was calculated on the basis that in an ideal separation, all of the LAC present in the feed and none of the quartz would report to the product (tailings of reverse flotation; concentrates for forward flotation). The actual product mass yield in each size fraction was then calculated from the size analyses of a specific flotation run. By expressing the ideal LAC yields and actual product yields as percentages of the mass of feed in each size fraction, the theoretical and "real" recoveries could be compared graphically. Furthermore, a flotation efficiency index for LAC was calculated for each size range using the following formula:

E = (product yield/ideal LAC yield) X (LAC feed ash/product ash) ....[4.16]

This index is similar to the flotation efficiency that is often quoted in the literature (Tsiperovich and Evtushenko, 1959)[1], but has a yield ratio term to account for the deviation from ideal separation in terms of yield as well as ash. This index can be used to compare quantitatively the separation efficiency of flotation in each size fraction. The size analyses, worksheets and sample calculations for this analysis may be found in Appendix XI.

The above analysis can be illustrated by using the following example: 100 g of an artificial quartz/LAC feed contains 20 g of material that is +0.125 mm: If 10 g of this is LAC with an ash content of 6%, then this 10 g should report to the product. The ideal LAC yield in this size fraction is 10/20 x 100 = 50%. Since zero quartz recovery would occur, the ideal ash content is the LAC ash content. i.e. 6%. In the actual experiment, if 8 g LAC is recovered, then the actual yield is 8/20 x 100 = 40%. If the actual product ash in this size range is 7%, then the efficiency is calculated as
(40%/50%) x (6%/7%) = 0.69.

The separations achieved for the reverse and forward flotation runs on the 50% quartz/coal mixture are shown in Figures 4.12 and 4.13 respectively. For each size fraction, the following information is shown, as a percentage in each case: the actual product yield, the ideal LAC yield (assuming ideal separation), the actual ash content,

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[1] Efficiency = Yield (%) x [feed ash (%) ÷ Concentrate ash (%)]

and the LAC ash content. The calculated separation efficiencies are given above each size fraction.

Taking the +0.125 mm size fraction for reverse flotation as an example (Figure 4.12), it can be seen that approximately 71% of the mass of material of this size in the feed was recovered in the product, whereas the ideal yield (based on the assumption of 100% LAC and 0% quartz recovery) is 69%. The actual recovery was then very close to the ideal. The ash content, however, was somewhat higher than ideal (8% vs 6%) and this explains why the efficiency index (as calculated by Equation 4.16) is calculated to be 0.73. For the +0.125 mm size fraction of the forward flotation concentrates, it can be seen (Figure 4.13) that approximately 74% mass yield occurred (leftmost bar) vs the 69% ideal recovery. The concentrates ash content was 7% (vs 6% for LAC) and the efficiency index is calculated to be 0.88. The two processes are thus similar in this size fraction but forward flotation provides the best separation.

In general, the separation efficiencies for reverse flotation were almost as good as for forward flotation except in the –0.025 mm fraction where a low tails mass yield occurred even though the tails ash content was very close to ideal. This indicates that some LAC was being lost to the concentrates. This might have been the result of entrainment of –0.025 mm LAC particles. This proposition is, however, in apparent conflict with the results obtained in section 4.2, where it was concluded that little or no entrainment occurred in the column cell. This is true on the basis of the whole feed, but it is quite possible for entrainment to have occurred in the –0.025 mm size fraction, and for the overall effect to have been minimised by the other size fractions in which entrainment did not occur.

Generally, however, it is assumed that entrained particles are washed out of a column flotation cell froth. It can be seen from a comparison of the water recovery data for the two processes (Tables 4.2 and 4.3) that a great deal more water was recovered in the reverse flotation concentrates (6.71–11.58 kg/h) than in forward flotation (0.13–1.37 kg/h) in forward flotation. On this basis alone, the opportunity for entrainment was greater in reverse coal flotation than in forward coal flotation. Furthermore, it was shown that the degree of entrainment is determined by both particle size and mineral type (section 2.3.1). This principle would apply as follows: –0.025 mm LAC particles would be very prone to entrainment as a result of their small size and their low density (R.D.=1.3–1.4). Once entrained, some of these –0.025 mm LAC particles would remain in the froth and thus eventually be recovered in the concentrate.

Examination of the graphs reveals that the selectivity of both processes improved in the finer size fractions, in the sense that the ash content approached the ideal LAC ash.

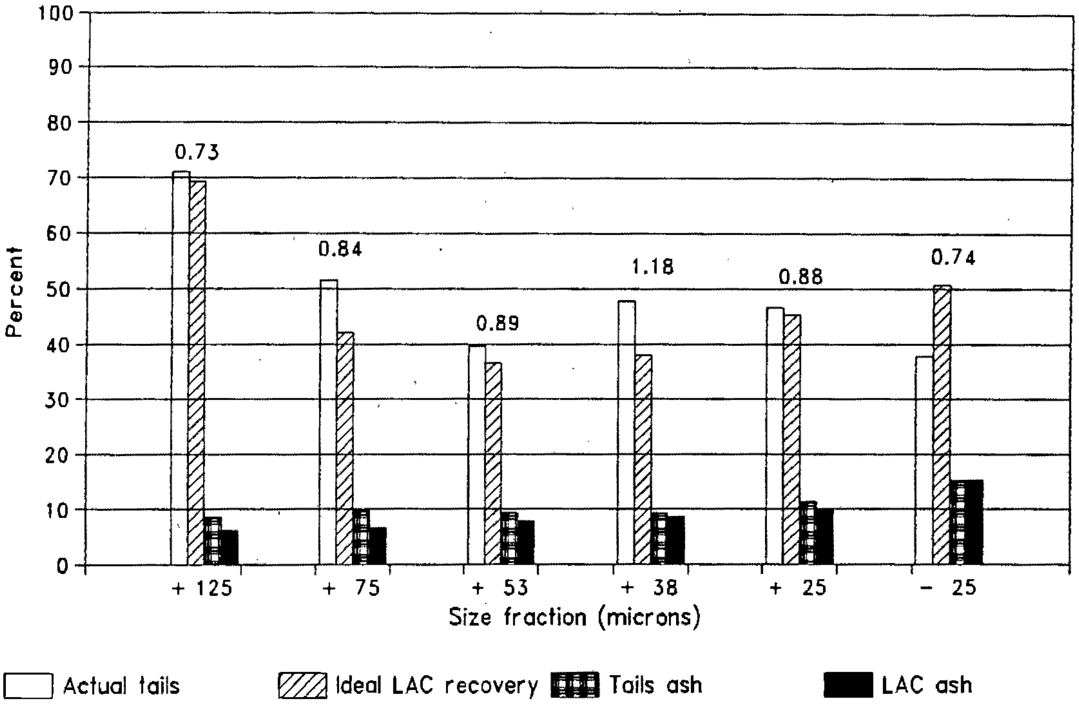

Figure 4.12: Separation effeciency by size fraction: Reverse flotation tailings sample (50% quartz/coal feed mixture).

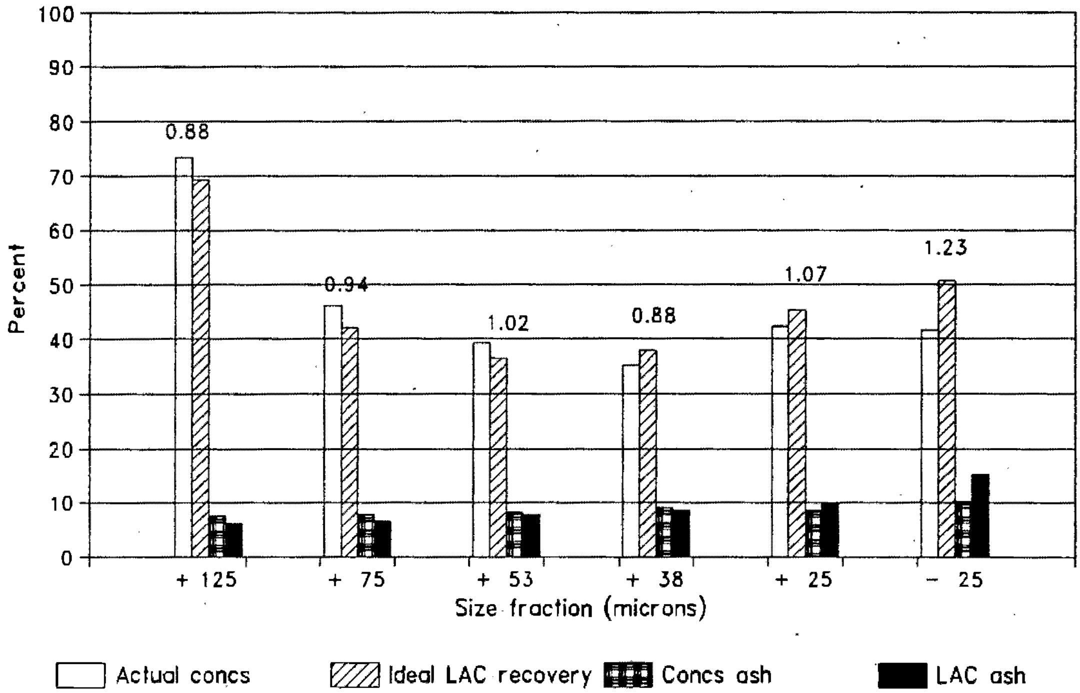

Figure 4.13: Separation efficiency by size fraction: Forward flotation concentrate sample (50% quartz/coal feed mixure).

Efficiency indexes of forward flotation are greater in all but one size range (−0.053 +0.038 mm). This is particularly marked in the finer size fractions (−0.038 mm), where the efficiency indexes are greater than 1.[1] It is clear that in the finer size fractions, forward flotation was extremely selective and this high selectivity in the very fine size fractions implies that entrainment (which is usually more prevalent for the finer fractions) was virtually nonexistent in the column cell. If entrainment of quartz was occurring, one would have expected the grade to be worse and the efficiency index to be consequently be lower.

These results for both reverse and forward flotation show the column cell to be extremely efficient in beneficiating the 50% quartz/coal feed mixture, with reverse flotation being the slightly inferior process. However, one of the main objectives in developing reverse flotation was to overcome the carrying capacity limitations of coal flotation. In the tests described above, it was assumed that, at the 1.5 kg/h feed rate, the column was being operated below the carrying capacity of both the forward and reverse flotation processes. The next series of experiments reflect reverse and forward column flotation at higher feed rates.

4.3.2.3 Effect of solids feed rate and determination of column carrying capacity

The effect of increasing the solids feed rate was investigated in two ways: by increasing the pulp density (at a constant volumetric feed rate), and by increasing the volumetric feed rate (at a constant pulp density).

Forward and reverse flotation runs at 20% pulp density were performed on the 16.7% and 25% quartz/coal mixtures. The feed pump setting was the same as before, thus the feed rate was 3 kg/h. All other parameters were the same. The results of these runs are presented in Table 4.4. The Table shows the data in the same format as in Tables 4.2 and 4.3; note that a distinction is made between the "product rate" and the "overflow rate". The former refers to the tailings of reverse flotation or the concentrates of forward flotation, whereas the latter refers specifically to the concentrate (for both processes).

If Table 4.4 is compared to Table 4.3, it can be seen that the beneficiation of forward flotation deteriorated in going to the higher pulp density. There was a loss of coal recovery: whereas typically 98% of the LAC was recovered in the 10% pulp density runs (1.5 kg/h feed rate), the recovery at the 3 kg/h feed rate was around 87 to 90%. This suggests that the column was operating at, or exceeding its carrying capacity. In contrast, there was no significant decline in efficiency in the reverse flotation runs. The results are virtually identical to those at 10% pulp density (cf. Table 4.2).

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[1] This occurs because the product ash contents obtained in the −38 +25 μm and especially the −25 μm range were lower than the ideal ash contents—i.e. the LAC was beneficiated in these size fractions. In terms of equation 4.16, the ratio [LAC ash/product ash] is greater than 1 which accounts for the large efficiency values.

Table 4.4: Reverse and forward flotation of artificial quartz/coal mixtures 20% pulp density.

Run	Quartz %	Coal rec (%)	Product Ash	Ash rec (%)	Feed t/h/m²	Overflow t/h/m2	Product t/h/m²
Reverse							
1.	16.7	96.87	11.07	91.89	1.460	0.271	1.189
2.	25.0	93.92	10.43	95.95	1.380	0.383	0.997
Forward							
1.	16.7	91.33	7.82	100	1.338	1.021	1.021
2.	25.0	88.74	8.32	100	1.355	0.911	0.911

In order to determine the carrying capacity for reverse and forward flotation, the column feed rate was varied in the range 1.5 to 6.7 kg/h at a pulp density of 20%, using the 25% quartz/coal mixture. The results are shown in Figures 4.14 and 4.15 which show the concentrates overflow rate and tailings rate as a function of feed rate.

Figure 4.14 shows the result for reverse flotation. The upper line shows the tailings rate; the lower line the concentrate rate. As can be seen, the column did not reach its carrying capacity over the range of feed rates used: there was a continual increase in the concentrates rate with increasing feed rate. At the highest feed rate used, 3.05 t/h/m^2 (6.7 kg/h)[1], the tailings (product) rate was 2.17 t/h/m^2 (4.8 kg/h) which was more than double than the concentrates (product) rate for forward coal flotation (see below).

Figure 4.15 represents the situation for forward column flotation. As can be seen, the concentrates overflow (clean coal product) reached a maximum rate of 0.9 t/h/m^2 (2 kg/h) at a feed rate of 1.35 t/h/m^2 (2.9 kg/h). This is the carrying capacity of the column for the LAC used in the artificial mixture. This would explain the decline in separation efficiency observed in the previous experiments at 20% pulp density. At the 3 kg/h feed rate, the column was at its carrying capacity, a situation which usually results in a loss of recovery (see section 2.3.4.2).

Figures 4.16 and 4.17 show the relationship between both product grade and coal recovery with feed rate for reverse and forward flotation respectively. It can be seen that reverse flotation could be operated at a feed rate of up to 3.05 t/h/m^2 without sacrifice of grade or recovery. For forward flotation, the coal recovery declined as the carrying capacity was exceeded (1.35 t/h/m^2).

The above results bear out the theory that a higher duty can be attained with reverse flotation (for a given column size) than forward flotation. For a projected column plant installation, this could result in a considerable saving in terms of capital cost because

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[1] This was the practical upper limit of the experimental equipment.

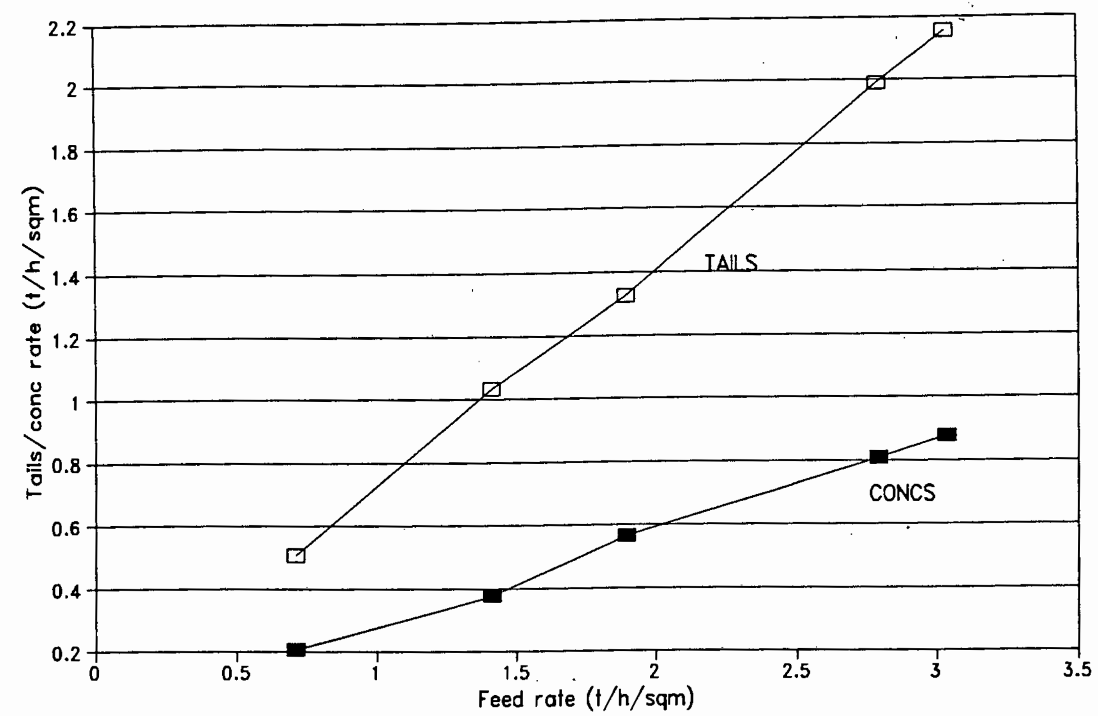

Figure 4.14: Determination of column cell carrying capacity: concentrates and tails rates vs feed rate in Reverse flotation.

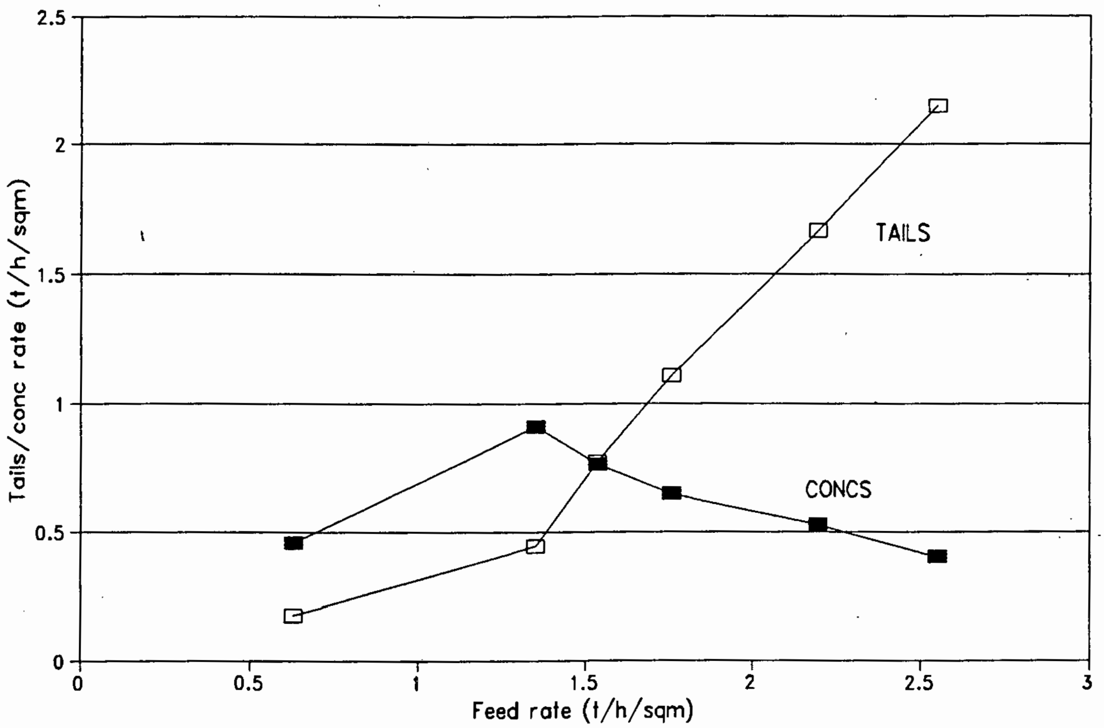

Figure 4.15: Determination of column cell carrying capacity: concentrates and tails rate vs feed rate in Forward flotation.

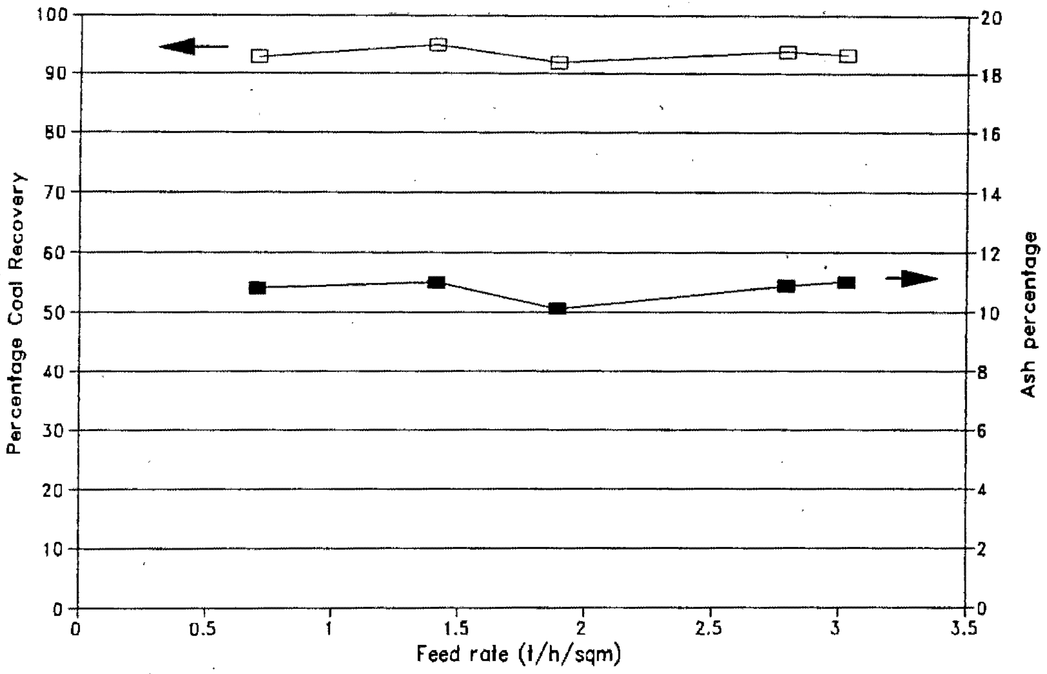

Figure 4.16: Reverse flotation: effect of feed rate on tailings recovery and grade.

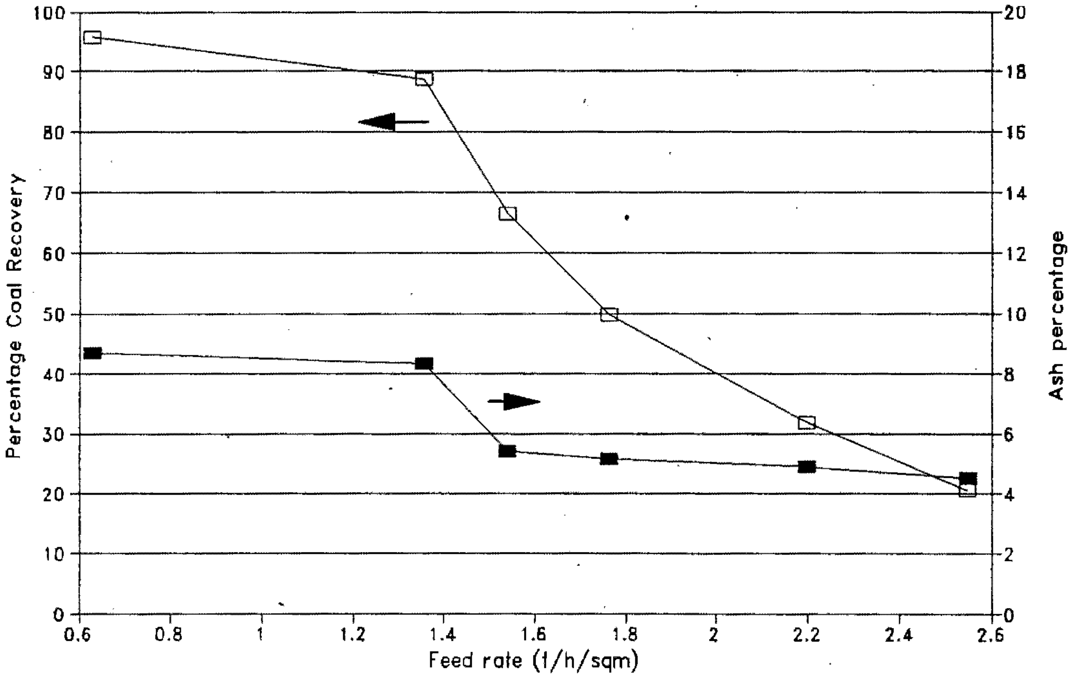

Figure 4.17: Forward flotation: effect of feed rate on concentrate recovery and grade.

less than half of the number of columns would be required for a given duty than for forward flotation.

Thus, the advantage of the concept of reverse coal flotation has been clearly demonstrated. Although reverse flotation results in slightly inferior beneficiation than forward flotation, this has to be weighed up against the advantages. Furthermore, the efficiency of reverse flotation might be improved further by optimizing column operating conditions and the reagent selection.

5. Conclusions

1. Reverse flotation of the artificial mixtures in the column cell was found to be highly successful. Effective quartz flotation, coal depression (in the tailings product) and frothing action was achieved by the reagent suite.

2. The optimum column configuration was found to be a 1 m collection zone and a 0.5 m froth height. An HTAB concentration of 1200 g/t was the minimum reagent dosage required, and the optimum air rate was $J_g>2$ cm/s.

3. Reverse flotation in the laboratory column cell resulted in better cleaning that in the conventional laboratory batch cell. This was due to the elimination of (coal) entrainment by wash water in the froth zone.

4. Forward flotation was found to give marginally better cleaning than reverse flotation, but the maximum throughput was much lower due to the carrying capacity constraint inherent to forward coal flotation.

5. The greatest throughput that was obtained in reverse flotation was approximately three times more than could be attained in forward flotation. Furthermore, this was attained without sacrifice of coal recovery or product grade. Thus, the proposed advantage of reverse flotation was proven.

At this point in the thesis, it is useful to reflect on what has been achieved. Reverse flotation has been proposed as a new process to address the problems of entrainment and a high concentrate mass recovery in coal flotation. A reagent suite for reverse flotation has been selected, and has been shown to be highly successful in the flotation of artificial mixtures. The use of a column cell was proposed as a means of obtaining improved reverse flotation performance and this has been shown to be the case. Furthermore, the proposed advantage of reverse flotation of improved throughput in column cells has been realized. The process has thus been shown to be technically viable. Up to this point, however, all the test work has been conducted on ideally liberated feeds containing pure coal and pure gangue. The process must now be evaluated for a real-life situation, i.e. reverse flotation of run-of-mine coals. This is the subject of the next chapter.

CHAPTER 5
REVERSE FLOTATION OF R.O.M. COALS

1. Introduction

Thus far in this thesis, the experimental testwork has been restricted to ideal, artificial mixtures consisting of blends of low ash coal (LAC) and quartz or kaolin in various proportions. The reason for this was to obtain results that were as unambiguous as possible, without being clouded by the additional factor of feed liberation. Experimental work was carried out in a small laboratory sub-aeration flotation cell and in a laboratory scale column cell. These results are described and discussed in Chapters 3 and 4. Results showed that reagents selected for reverse flotation were suitable, and good separations of coal and gangue were achieved. Optimum flotation conditions and procedures were identified.

In this chapter, reverse flotation testwork is extended to a number of South African run-of-mine (r.o.m.) coals. This was thought necessary because, as described in Chapter 2, most South African coals are poorly liberated and poorly floating (see Chapter 2, section 3.1); thus the separation would not be expected to be as clear cut as for the artificial mixtures. Tests on r.o.m. coals would also afford the opportunity to find out more about the nature of the reverse coal flotation process.

The objective of this chapter is to investigate the metallurgical performance and separation efficiency of the reverse coal flotation process in the beneficiation of a number of South African r.o.m. coals, and to compare the results with those obtained using the artificial mixtures and with forward flotation of the same r.o.m. coals.

Experimental work was performed first in the conventional laboratory batch cell to compare the performance of reverse flotation on r.o.m. coals with the performance obtained previously with the artificial mixtures in the batch cell (Chapter 3). Reverse flotation of the r.o.m. coals was then performed in the laboratory column cell; the process was compared to forward flotation of the same r.o.m. coals in the column cell, as well as to the results obtained previously (Chapter 4) using artificial mixtures.

2. Flotation samples

Four coals were selected for testing, each representing a major South African coalfield. These were: 1) a Waterberg coal from the Grootegeluk Colliery in the Northern Transvaal; 2) a Witbank coal from the Rietspruit Colliery; 3) an Eastern Transvaal coal from the Ermelo Colliery and 4) a Natal coal from the Durban Navigation Colliery (Durnacol) (The geographical location of the coal seams are described in Appendix I.).

All the samples were raw, bituminous coals which are beneficiated by the collieries to produce coking coal (Grootegeluk and Durnacol) or thermal coal (Grootegeluk, Rietspruit and Ermelo) products.

The Rietspruit coal had an ash content of 26.5%, the Grootegeluk coal contained 42% ash, and the Ermelo 23.4%. These three coals were chosen for practical reasons: they were available for use in the Department, having been obtained for previous coal flotation projects. Furthermore, extensive testwork had already been performed on these coals in the Department of Chemical Engineering, University of Cape Town. These tests included batch flotation tests and washability analyses, which would assist with the evaluation of the performance of reverse flotation on these coals. All were thickener underflow samples, already divided into sub-samples by the collieries.

The Durnacol coal was selected because of its good liberation characteristics (by South African standards), and high rank. It was considered an ideal (r.o.m.) feed to evaluate the reverse flotation process.

It should be noted at this point that the Grootegeluk, Ermelo and Durnacol collieries have flotation plants which produce coal products of 12–14% ash (DMEA, 1986), thus these coals are separable by conventional forward flotation.

3. Batch flotation experiments

3.1 Experimental procedure

Reverse flotation of two r.o.m. coals, from the Rietspruit and Grootegeluk Collieries, was investigated in the laboratory (sub-aeration) batch flotation cell. Some of the flotation experiments were single stage runs, but most were 2–stage runs performed according to the procedure described in Chapter 3, section 4.3.1.

The coal samples had been received packaged in sachets which contained approximately 300 g of wet coal. To ensure consistency for each individual flotation feed sample, the following procedure was adopted: For a set of flotation experiments, the contents of a number of sachets (e.g. 10) were combined and thoroughly mixed in a large bucket using the impeller of a laboratory Denver flotation cell. This bulk sample was then dewatered in a pressure filter and the resulting filter cake was divided into the same number of samples (i.e. 10) of approximately equal mass. Each of these samples was then placed in a plastic bag, which was filled with water and sealed.

For the flotation experiments, conditions were kept at the same values as when investigating reverse flotation of artificial mixtures: the air rate was 4 l/min, the impeller speed was 1200 rpm and the froth height was 2.5 cm. The pH of flotation was not adjusted. There was a small difference in the pulp density, since the dry mass of each sachet was approximately 200 g and this corresponded to a pulp density of 7% in the 3-litre cell as opposed to 10% used in all the previous testwork on artificial mixtures.

As before, the HTAB and dextrin were used as the reverse flotation reagents. The dextrin concentration was kept constant at 800 g/t and the HTAB concentration was varied in the range 1300 to 2800 g/t. In a few of the experiments on the Rietspruit sample, an anti-sliming agent, sodium silicate, was employed in an attempt to counteract the (possible) deleterious effect of sliming. This was added at a dosage of 290 to 950 g/t.

3.2 Results and discussion

The complete set of results for both coals can be found in Appendix VII. The results of selected runs are presented in Table 5.1. This shows the tails (product) yield and ash content, HTAB concentration, and Sodium Silicate ($NaSiO_2$) concentration for selected (optimal) runs. Also shown is the result of a forward flotation run (obtained from the reproducibility tests in Chapter 3, section 2.5.4) for comparison.

As can be seen, the (tailings) yields obtained with reverse flotation of the Grootegeluk coal were 80 to 90%, which are very high, but the grades obtained were extremely poor (the ash contents were high). A similar situation is true for the Rietspruit coal. The yields obtained with reverse flotation were in the range 63 to 75% but the product grades were in the range 21 to 22% ash, which represents only a marginal reduction from the feed ash content of 26%. In contrast, the yield obtained in the forward flotation run was 63% but the ash content of the feed was reduced to 14.25% (in the concentrates).

Table 5.1: Reverse flotation of two r.o.m. coals
in the batch cell

Run no.	Yield (%)	Ash (%)	HTAB (g/t)	$NaSiO_2$ (g/t)
Grootegeluk (feed ash 42%)				
1.	91.4	38.80	1090 + 670	0
2.	81.2	36.30	1900 + 671	0
Rietspruit (feed ash: 26.5)				
1.	63.4	21.87	2063 + 614	0
2.	75.4	22.15	2070 + 589	941
3.	69.7	22.06	2006 + 598	696
4.	65.2	21.27	1907 + 786	450
5.	63.0	14.25	n/a (forward flotation)	—

It should be noted (Table 5.1) that the HTAB concentrations required to obtain these modest beneficiation results were in the range 1700 to 2600 g/t which is considerably greater than was used in the previous experiments on the quartz/coal feeds (typically 500 to 600 g/t) even though the size range was similar (–0.25 mm). It should also be noted that the anti-sliming agent had a small but advantageous effect on flotation performance.

Figure 5.1 shows the concentrates recovery/time data for Rietspruit coal for the run in which the best beneficiation results were achieved (no. 4, Table 5.1) as well as the corresponding data for a forward flotation run (cf. Figure 3.2) for comparison purposes.

It can be seen that, in reverse flotation, approximately 50% ash recovery and 30% coal recovery occurred in the concentrate, whereas in the forward flotation run 70% coal recovery and 33% ash recovery occurred (also in the concentrates). For reverse flotation to have achieved the same (or better) separation as forward flotation, at least 70% ash recovery should have occurred in the concentrates and 30% (or less) coal recovery. Thus, the problem with the reverse flotation of this r.o.m. coal is that the flotation response of the gangue was poor, i.e. the process was not selective for the gangue. In the absence of further information, one can only speculate on the reasons for this. One possibility is that the gangue particles were much finer in size than the coal particles and consequently had a poor floatability. Whatever the reason(s), however, it may be deduced that the problem lay with the nature of the feed, rather than with the process itself, since reverse flotation has been found to be highly successful on the artificial feeds.

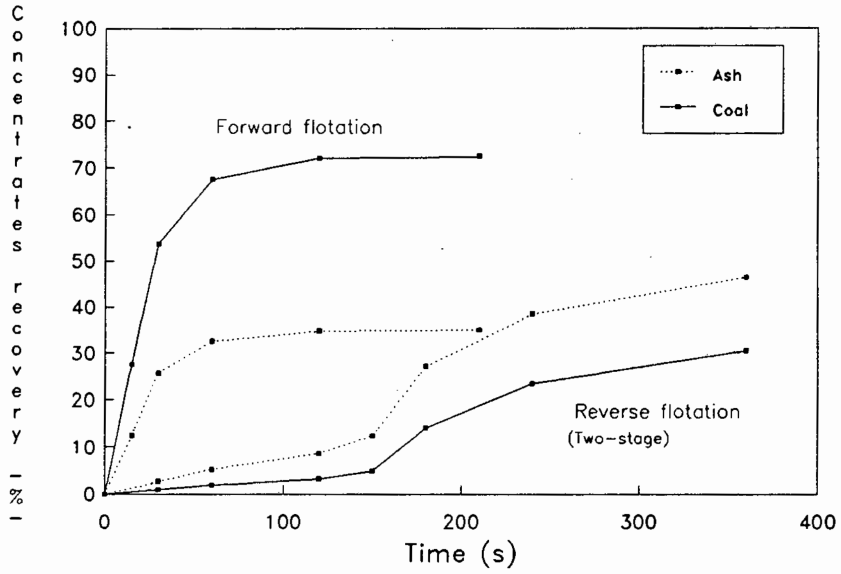

Figure 5.1: Comparison of reverse flotation with forward flotation: Rietspruit coal.

3.3 Conclusion

It is clear from the results that reverse flotation was not successful in beneficiating the two r.o.m. coals tested in the batch cell. The fundamental problem appeared to be one of selectivity towards the gangue particles. Clearly, for the process to be comparable with forward flotation, improved gangue selectivity needed to be obtained.

It was thought that a possible solution to poor gangue selectivity might be found in the use of column cells, which offer better cleaning and better selectivity due to the deep drained froth zone. This had been found to be the case with artificial mixtures (Chapter 4). Thus, the reverse flotation testwork on r.o.m. coals was extended to the laboratory column cell. The aim of this work was to examine reverse flotation performance more thoroughly, to compare the separation efficiencies of reverse and forward flotation, and to identify reasons for any differences. The column cell flotation testwork is described in the next section.

4. Column flotation experiments

As reported in Chapter 4, reverse flotation of the artificial mixtures in the column cell resulted in improved performance over the results obtained in the batch cell. In this section, the testwork on r.o.m. coals is extended to column cells in an attempt to obtain improved cleaning for r.o.m. coals than was found in the conventional batch cell and to find reasons for differences between the reverse flotation performance of r.o.m. coals and artificial mixtures. Reverse flotation is also compared to forward flotation on the same coal.

4.1 Description of experimental work

The same equipment was used as for the testwork on the artificial mixtures (Chapter 4, see section 3). The reagents used were also the same, namely: HTAB collector, dextrin depressant and DIBK frother for reverse flotation experiments, and Shellsol A collector and DIBK for forward flotation experiments. The procedures were also identical to those outlined in Chapter 4, section 3.

Experimental work was performed first on the samples from the Grootegeluk and Ermelo Collieries (the Rietspruit sample was depleted by this stage; the Ermelo sample was used as a substitute). Reverse flotation tests were carried out at various conditions in order to obtain the best possible result in terms of recovery and grade.

For the sample from the Durnacol Colliery, the testwork was more extensive. Both reverse and forward flotation tests were performed using the column cell at various operating conditions; size analyses were carried out on feed, concentrate and tailings samples of typical reverse and forward flotation runs; and washability characteristics of the feed, and of the same concentrates and tailings samples selected for size analyses, were determined in order to assess and compare reverse flotation and forward flotation in absolute terms. Finally, some sulphur analyses were performed on selected reverse and forward flotation runs.

4.2 Grootegeluk and Ermelo coals

4.2.1 Experimental procedure

As described in section 3.1, the Grootegeluk and Ermelo samples were obtained pre-packaged in 300 g sachets. For each column experiment, a number of sachets were combined to give a larger sample size (2–4 kg), oven dried at 80 °C overnight, and milled to 90% passing 0.075 mm. (The milling was performed in an effort to improve the liberation characteristics of the coals.).

In all of the runs, the pulp density of the feed was 10% and feed rate was 1.5 kg/h which corresponds to 0.68 t/h/m^2. The following parameters were varied in the ranges indicated:

Froth height: 0.5 to 1.2 m
Collection zone height: 1.0 to 1.5 m
HTAB concentration: 2500 to 4500 g/t
Dextrin concentration: 500 to 1000 g/t
Gas Rate (J_g): 1.8 to 2.5 cm/s

4.2.2 Results and discussion: Grootegeluk coal

The results of the reverse flotation experiments on the Grootegeluk coal are shown in Table 5.2 below, which lists the mass yield and ash content of the tails product, and the coal and ash recovery in the tails and concentrates, respectively, for different collection zone and froth zone heights, HTAB and dextrin concentrations, and air rates. The best beneficiation results were achieved at the shortest column configuration, namely 1 m collection zone and 0.5 m froth height (as was the case for the artificial mixtures). The best beneficiation result was obtained in run 11, in which a product containing 34.1% ash (feed ash 42.0%) at 89.6% organic coal recovery was achieved. The HTAB concentration that was required to achieve this result was 3500 g/t, which is almost three times more than the amount required for the artificial mixtures.

If the results in Table 5.2 are compared with those of the (optimal) batch results in Table 5.1, it may be observed that cleaning was marginally better in the column cell: the best grade obtained in the batch cell was 36.3% ash, while the best obtained in the column cell was 34.1% ash.

Of the parameters investigated, the column configuration had the greatest effect on the metallurgical performance of the column. A comparison of runs 8 and 9 illustrates this: the flotation of ash (i.e. ash recovery) increased from 12.72% to 30.72% when the collection zone height was reduced from 1.5 to 1.0 m, and the froth height was reduced from 1 to 0.5 m. The only difference between these runs (apart from the collection zone height) was the air rate. This difference was small and, in any case, the effect of air rate was not significant at the 1 m collection zone, as can be seen by comparing runs 9 to 12. For some conditions, however, the air rate was seen to affect flotation. For run 5 where the air rate was 1.88 cm/s, the ash recovery was 12.72%. For run 6 in which the air rate was increased to 2.33 cm/s (all other conditions were identical), the ash recovery was 22.24%. Increasing the air rate was thus beneficial to reverse flotation performance but this is only true below J_g=2 cm/s.

Table 5.2: Reverse flotation of Grootegeluk coal in the column cell.

No.	Yield (%)	Product ash (%)	Product coal rec (%)	Discard Ash rec(%)	Froth ht (m)	Collec zone ht (m)	HTAB g/t	Dextrin g/t	Air rate cm/s
1	97.47	40.94	98.53	4.02	0.5	1.5	2500	0	1.88
2	93.12	39.45	95.76	10.67	0.5	1.5	4500	0	1.88
3	89.09	41.00	90.70	13.34	0.5	1.5	3500	0	1.88
4	98.14	40.50	98.70	2.67	0.5	1.5	3500	1000	1.88
5	90.55	40.40	92.91	12.72	0.5	1.5	4000	500	1.88
6	83.62	38.43	87.74	22.24	0.5	1.5	4000	500	2.33
7	89.71	40.32	91.27	12.50	1	1.5	3000	800	2.30
8	89.81	40.60	91.63	12.72	1	1.5	3000	800	2.11
9	81.00	34.39	89.07	30.72	0.5	1	3000	800	2.33
10	81.68	34.52	90.01	30.51	0.5	1	3000	800	1.92
11	81.06	34.10	89.59	31.54	0.5	1	3500	800	2.33
12	82.40	34.80	90.30	29.21	0.5	1	3500	800	2.46

In runs 3 and 4, the only difference was the addition of 1000 g/t of dextrin in run 4. This resulted in an increase in the tailings coal recovery from 89% to 98%. This was, however, at the expense of ash recovery, which decreased from 13.34% to 2.67%. What this suggests is that the gangue in the Grootegeluk coal is poorly liberated. In a flotation study by Fickling (1986) on a number of South African coals, a link between gangue recovery rate and coal flotation rate was attributed to insufficient liberation of coal and gangue particles (this has already been discussed in Chapter 1, see section 2.3, p 5). In these reverse flotation experiments the same phenomenon is being observed, except in reverse—an increase in depression of coal to the tailings was accompanied by a decrease in gangue flotation to the concentrate. It was expected that the addition of dextrin would

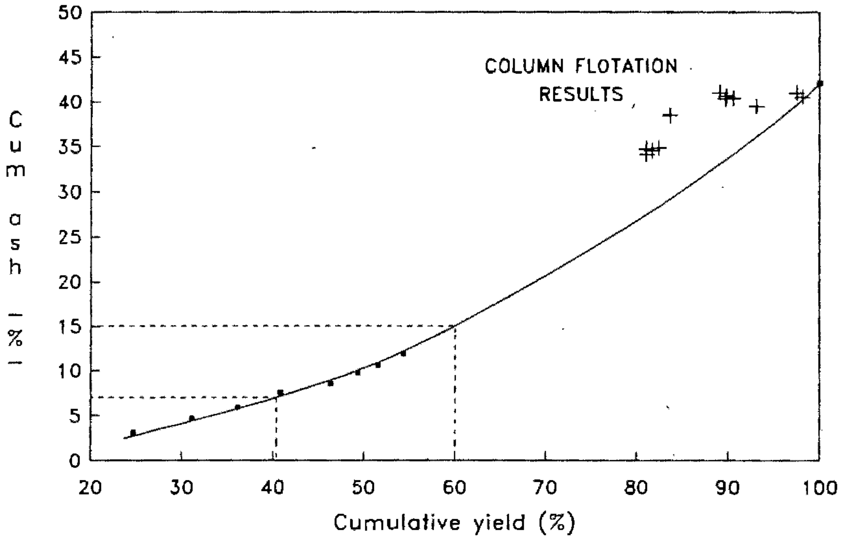

Figure 5.2: Washability data (solid curve) and reverse flotation results for Grootegeluk coal.

improve the recovery of coal particles in the tails and also increase the availability of the HTAB collector for gangue particles. This should have increased the flotation yield of the gangue particles, and thus improved the overall separation. This did not happen; in fact the opposite occurred. This suggests that the coal and gangue particles were still associated with each other, which would account for the poor flotation recovery of gangue in the reverse flotation of Grootegeluk coal.

The results of the reverse flotation experiments on the Grootegeluk coal are also shown in Figure 5.2, in which they are compared with the float-sinks washability data (yield vs ash curve) for the coal (Buys, 1989). As can be seen, the beneficiation results for reverse flotation were poor: the runs lie in the high yield, high ash region indicating poor selectivity. Ideally, flotation results should be in the 40 to 60% yield region to produce a saleable product with an ash content of 15% or lower. Clearly, reverse flotation does not result in a worthwhile beneficiation of the Grootegeluk coal sample.

4.2.3 Results and discussion: Ermelo coal

The results for the Ermelo sample are shown in Table 5.3 below. Once again, the beneficiation was poor. The best beneficiation result was a product containing 17.6% ash at 95.8% coal recovery (feed ash 23.4%). This was obtained using the 0.5 m froth, 1.0 m collection zone height configuration (run 6). The amount of HTAB added was 3000 g/t .

A comparison of runs 4 and 5, in which the conditions were virtually identical, indicates that good reproducibility was obtained in the laboratory column: the product grade, coal recovery and gangue recovery differed (individually) by less than 1.5 percentage points.

A comparison of runs 1 and 2 show that the addition of dextrin was necessary to improve coal depression and cleaning. For run 1, in which no dextrin was added, the tails coal recovery was 84.97%, and the tails ash content was 20.35%. For run 2, in which 500 g/t dextrin was added, while all other conditions remained the same, these values were 89.8% and 19.31% respectively.

Table 5.3: Reverse flotation of Ermelo coal
in the column cell.

No.	Yield (%)	Product ash (%)	Product coal rec (%)	Ash rec (%)	Froth ht (m)	Collec zone HT	HTAB g/t	Dextrin g/t	Air rate cm/s
1	82.17	20.35	84.97	27.24	1.2	1.3	4000	0	1.90
2	86.02	19.31	89.80	26.85	1.2	1.3	4000	500	1.90
3	91.81	19.60	95.83	21.57	0.5	1	2500	800	2.05
4	87.54	19.07	91.59	26.42	0.5	1	2500	800	2.30
5	88.09	18.77	92.86	27.91	0.5	1	2500	800	2.35
6	89.99	17.60	95.83	30.08	0.5	1	3000	800	2.30

The effect of increasing the HTAB addition rate can be seen by comparing runs 4 and 6, which differed only with respect to their HTAB concentration. For run 4 (2500 g/t) the gangue recovery was 26.42%, the product ash was 19.07%, and the coal recovery was 91.59%. For run 6 (3000 g/t), the values were 30.08%, 17.6% and 95.83% respectively. Increasing the HTAB addition rate beyond 3000 g/t would, in all likelihood, have improved the performance still further. However, since the dosage was already much greater than the 1200 g/t which was required for the artificial mixtures (Chapter 4, section 4.2.2) it was decided that increasing the dosage beyond 3000 g/t would be impractical, whatever the benefit that might be achieved.

Also note that the increase in coal recovery (tails) is accompanied by a decrease in concentrate ash recovery (cf. runs 1&3), which, like the Grootegeluk coal, indicates a lack of liberation.

4.2.4 Conclusion

It is apparent that the beneficiation achieved by reverse flotation of the r.o.m. coals in the column cell, although an improvement over the results obtained in the sub-aeration cell, was not sufficient to obtain a marketable product for either Grootegeluk or Ermelo coal. At best, the feed ash content was reduced by 8% (from 42% to 34% ash in the case of Grootegeluk coal). Since reverse flotation was highly successful on artificial mixtures, a reason for the poor results was sought in the nature of the coals themselves. The next section describes flotation tests carried out with a different r.o.m., chosen on account of its good liberation characteristics, to determine whether an improvement in beneficiation could be obtained.

4.3 Durnacol coal

The Durnacol coal sample was chosen specifically because of its good liberation characteristics; it was thought that it would be a good coal with which to evaluate reverse flotation. Because of the sharp contrast between the success of the testwork with the artificial mixtures and the poor results which had been obtained up to this point with the r.o.m. coals, much more extensive tests were carried out with the Durnacol coal than with the Rietspruit, Grootegeluk, or Ermelo coals. These included the investigation of flotation performance by size fraction and a comparison with forward flotation runs of the same coal in the same laboratory column cell. Some float and sinks analyses were also carried out on concentrates and tailings samples of typical reverse and forward flotation runs.

4.3.1 Feed characterization

The coal was obtained from the Durnacol Colliery as a 200 kg thickener underflow sample, stored under water. The particle size was nominally –0.2 mm. The coal was prepared for use as follows: excess water was drained off and the (moist) bulk sample was separated into smaller, 12 kg sub-samples by the method of coning and quartering described by Gy (1981). These sub-samples were transferred to plastic buckets, sealed and stored until required.

The washability of the coal was determined by float and sink analysis. The raw data can be found in Appendix XIII. The mass and ash distributions according to density are shown in Figure 5.3. As can be seen, there was little near density material in the 1.5—1.7 R.D. range (floats) which indicates fairly good liberation of gangue and coal. Figure 5.4 shows the cumulative ash vs cumulative yield curve (washability); it can be seen that a theoretical yield of approximately 60% is indicated at 7% ash, or 74% at 15% ash (thermal quality). This coal has a superior washability to the Grootegeluk coal (see Figure 5.2) where the theoretical yield is 42% at 7% ash, or 61% at 15% ash.

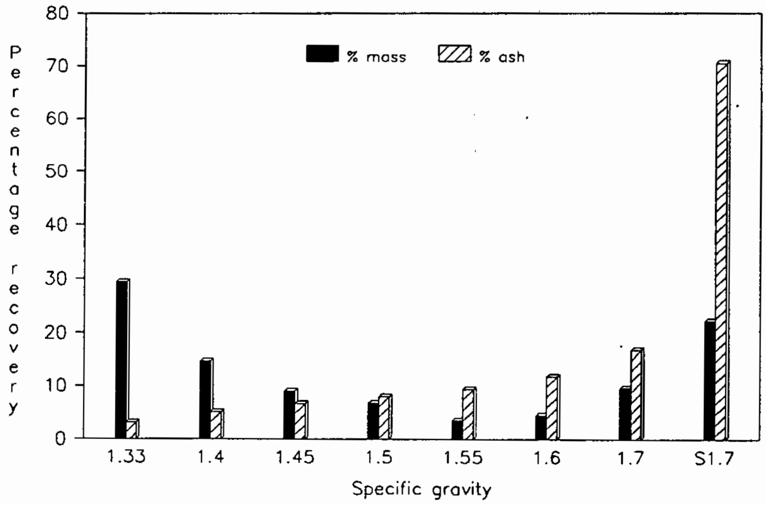

Figure 5.3: Distribution of the Durnacol sample by relative density.

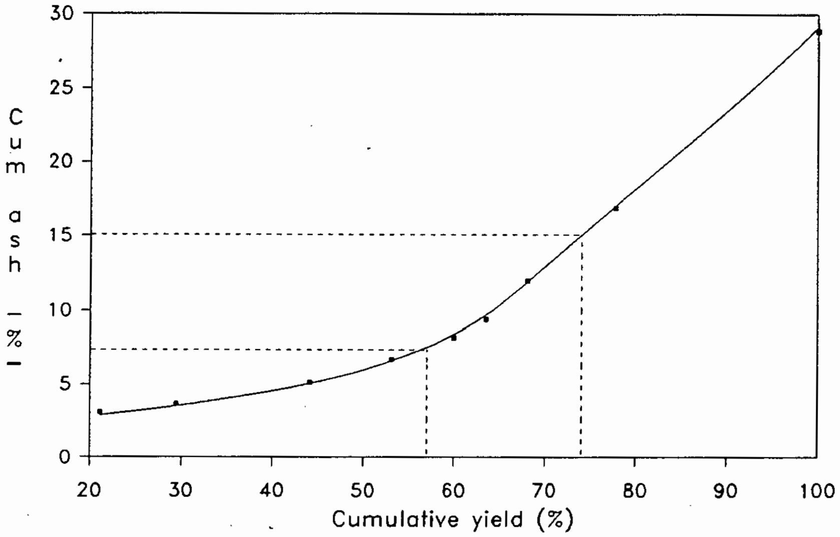

Figure 5.4: Durnacol washability curve

A size analysis was also performed on the feed, by a wet sieving technique. These results are depicted in Figure 5.5 (the raw data may be found in Appendix XIII). As can be seen, there was a significant proportion of lower ash material in the coarser fractions. For example, the +0.125 mm fraction contained 17% of the feed with an ash content of 13%. At the same time, there was a predominance of higher ash material in the finer size fractions; the –0.053 +0.028 mm size fraction contained 12% of the feed at 31% ash, the –0.038 +0.025 mm fractions contained 8% of the feed at 36% ash, and the –0.025 mm fraction contained 37% of the feed at 41% ash.

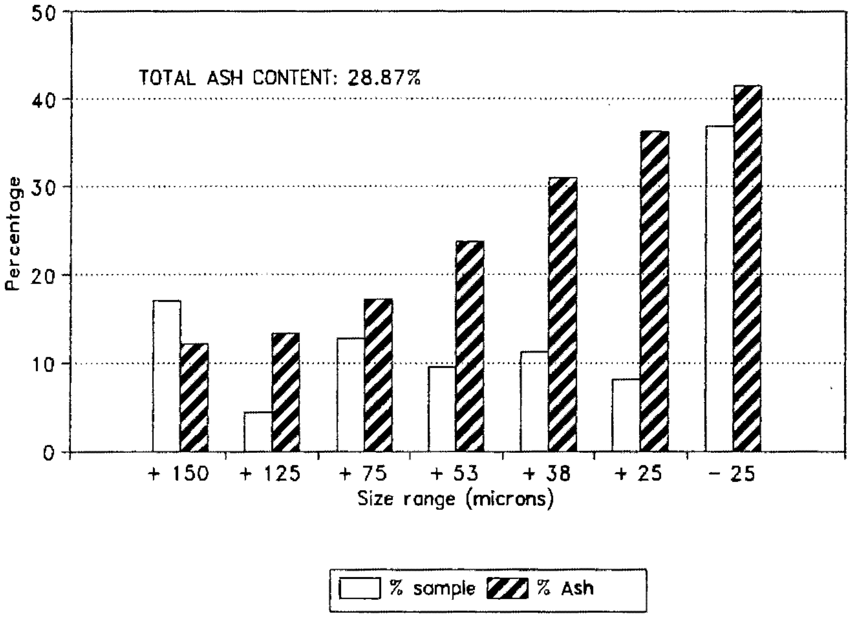

Figure 5.5: Durnacol feed size analysis

4.3.2 Experimental

Reverse flotation of the Durnacol coal was performed in the column only, using both the filter cloth sparger used in the previous tests, and a USBM-type external sparger (described in Chapter 4, section 3.1.2). For all the experiments, HTAB was used as the ash collector with dextrin as the coal depressant. With the runs using the filter cloth sparger, the frother DIBK was used as an additional frother. For the runs using the external sparger, HTAB was used as the only frothing agent, and was added to the sparger water to give a concentration of (approximately) 200 g/t of solids (over and above the HTAB addition in the feed pulp).

In addition to the reverse flotation runs, a number of forward flotation runs were performed in the column on the same coal for comparison purposes. For these tests, a filter cloth sparger was used. The coal collector was Shellsol A and the frother was DIBK.

The following parameters were varied in the indicated ranges:

Froth height: 0.5 m
Collection zone height: 1.0 or 2.25 m
HTAB concentration: 1750 to 4000 g/t
Dextrin concentration: 800 g/t
Shellsol A collector (forward flotation): 500 to 1000 g/t
Gas Rate (J_g): 1.2 to 2.50 cm/s
DIBK Frother (reverse flotation): 8 or 15 μl/l
(forward flotation): 15 μl/l
Pulp density: 10 or 20%

A feed rate of 1.5 or 3.0 kg/h was used for the reverse and the forward flotation runs depending on the feed pulp density (10 or 20% solids, respectively).

In order to compare the performance of reverse and forward flotation by size fraction, samples of concentrates and tailings of a selected reverse and a selected forward flotation run were subjected to size analysis by wet sieving, as well as by a Malvern particle size analyzer to obtain more detailed analyses of the slimes size fraction (i.e. –0.025 mm) in the samples than is possible with wet sieving. All the size analyses data are shown in Appendix XIII. Float/sink washability tests were performed on the same concentrates and tailings samples subjected to size analysis, using a density of separation of 1.7.

Determination of total sulphur content of the feed, concentrates and tailings was made for a number of reverse and forward flotation runs. The analyses were performed on a LECO SC32 Sulphur Determinator. The analyses are shown in Appendix XIII.

4.3.3 Reverse flotation results

The reverse flotation results for the Durnacol coal are shown in Table 5.4 The Table shows the yield, ash content, and organic coal recovery of the product (in the tailings); the ash recovery and ash content in the discards; as well as the HTAB concentration, pulp density, and the air rate in each run. For some of the runs, the total sulphur content of the product is also shown. Note: the froth height was 0.5 m and the collection zone height was 1.0 m in all runs, except for runs 14 to 17, for which it was 2.25 (this is also indicated on the table). A more detailed table of results may be found in Appendix XII.

In general terms, it can be seen that the beneficiation achieved by reverse flotation of the Durnacol coal was poor, though slightly better than that achieved with the other two coals. Quite a number of results were obtained with a product ash in the range 20 to 22% and a coal recovery in the range 80 to 90% (runs 2, 4, 8, 12, 13, 15, 16, and 17). The feed ash was 29%. The best reverse flotation result achieved was a (tails) product of 17.9% ash and 81.34% coal recovery (run 4), obtained with the filter cloth sparger. None of the results represent a saleable product of even thermal quality (15% ash); it should be noted that the mine produces a coking coal product of approximately 12% ash from the flotation plant.

Table 5.4: Reverse flotation of Durnacol in the column cell.

No.	Yield (%)	Product ash (%)	Coal rec (%)	Ash rec (%)	Discards ash (%)	Product sulphur %	HTAB g/t	Frother g/t	Pulp Density	Air rate cm/s
FILTER CLOTH SPARGER								**DIBK**		
1	94.06	25.30	97.92	15.75	74.89	1.3	4000	-	10	2.11
2	71.55	18.50	81.25	53.11	52.71		2000	15 μl/l	10	2.11
3	89.41	23.40	95.14	25.32	66.96		1500	8 μl/l	10	2.11
4	70.84	17.90	81.34	55.50	54.23	1.35	2500	8 μl/l	10	2.11
5	88.68	23.66	94.32	25.66	63.97		2000	8 μl/l	20	1.96
USBM								**HTAB**		
6	75.81	22.87	83.63	42.37	47.56		2250	200	20	2.11
7	76.90	22.98	83.02	38.34	52.69		1750	200	20	1.89
8	70.75	21.23	79.07	49.12	49.57		2750	200	20	2.04
9	82.55	22.25	90.14	36.22	59.76		2500	200	20	1.83
10	85.86	23.92	93.14	31.75	66.02		2500	200	20	1.43
11	89.71	24.92	95.14	23.45	66.58		2750	200	20	1.19
12	76.62	20.95	84.88	43.96	53.86		2750	200	20	1.92
13	79.93	20.93	88.08	40.77	57.38		2750	200	20	1.55
14*	86.20	22.02	94.30	33.80	70.72		2500	200	20	1.89
15*	83.01	20.92	92.53	40.22	68.78	1.35	2500	200	20	2.41
16*	80.60	20.99	90.40	42.84	65.23	1.33	2750	200	20	2.41
17*	80.00	19.80	90.40	45.50	65.98		3000	200	20	2.41

*** 2.25 m collection zone**

FEED ASH: 28.8%

FEED SULf:1.21%

Examining the results more closely, it may be observed that the air rate had a significant effect on the metallurgical performance of the column. This can be seen from a comparison of runs 11, 12 and 13 (highlighted on the table): As the air rate was increased from 1.2 cm/s to 1.55 cm/s to 1.9 cm/s in runs 11, 13 and 12 respectively, the ash recovery increased from 23.5% through 40.8% to 44.0%. At the same time, the coal recovery in the tailings decreased from 95.1% through 88.1% to 84.98%. The product grade improved from 24.9% at 1.2 cm/s, through 20.9% at 1.55 cm/s and was 21.0% for 1.9 cm/s. The discards ash dropped from 66.6% through 57.4% to 53.9%. This indicates that, as the ash recovery improved (i.e. more ash was floated), more coal was recovered in the concentrates (causing the tails coal recovery to decrease). Also, the amount of coal recovered in the discards increased more than the increase in ash recovery, which is indicated by the decline in the ash content of the discards. This can be demonstrated by the following mass balances, in which the basis is 100 g reconstituted feed:

Run no.	Air rate	Conc Yield (g)	Ash %	Ash(g)	Coal(g)
11	1.19	10.29	66.58	6.85	3.44
13	1.55	20.07	57.38	11.16	8.55
12	1.92	23.38	53.86	12.59	10.79

As more ash was recovered, the separation was less selective, since the amount of undesired material in the concentrates (i.e. coal) increased at the expense of the desired material (ash).

The effect of increasing the HTAB concentration can be seen by comparing runs 3 and 4, in which the air rate was constant (2.11 cm/s) but in which the HTAB concentration increased from 1500 g/t to 2500 g/t. The ash recovery increased from 25.32% to 55.5%, the coal recovery decreased from 95.14% to 81.34%, the product grade improved from 23.4% ash to 17.9% ash, and the concentrate (discard) ash decreased from 66.96% to 54.23%. Thus, the same phenomenon is observed as in the previous discussion—increasingly more coal was recovered in the concentrates as more ash was floated.

It can be seen from the data for runs 1 and 2 that the use of DIBK as an additional frother in the runs using the filter cloth sparger had a marked effect on the both metallurgical performance and the amount of HTAB required. In run 1, where the HTAB concentration was 4000 g/t and no DIBK was added, the coal recovery was 97.92%, the product ash was 25.3%, and the ash recovery in the discards was 15.75%. In run 2, in which 15 μl/l DIBK was added, the coal recovery decreased to 71.55%, the product grade improved to 18.5% ash, and the ash recovery in the discards increased to 53.11%. Thus, much better performance in terms of ash recovery and product grade was obtained by the addition of DIBK as a frother in reverse flotation, using half the HTAB dosage. In interpreting this, it should be noted that a substantial proportion of the Durnacol feed was present as –0.025 mm slimes was present (see Figure 5.5). This could have resulted in a high consumption of the HTAB which would, in turn, have had negative impact on the frothing action of HTAB, since less "free" HTAB would be in solution to act as a frother; thus a large amount would have to have been added to try and counteract this effect. The addition of DIBK frother could alleviate this situation since it would stabilize the froth without interacting with the feed, thus allowing the HTAB to perform the single role of ash collector.

Runs 14 to 17 reflect the situation where the collection zone height was increased from 1 m to 2.25 m. This would cause a corresponding increase in collection zone residence time, since the feed rate was kept constant. In general terms, it can be seen that ash recoveries in the discard were not affected by this change, but the coal recoveries in the product improved slightly in comparison to runs in which similar ash recoveries were obtained. In all the runs at 2.25 m in which 40% or more ash recovery occurred, the product (tails) coal recovery was 90% or more, whereas in all the other runs (6 to 13), the coal recovery was below 90%. Also, the concentrate ash content was in the range

65% to 69% in runs 14 to 17 (2.25 m collection zone height), but only 47% to 59% in runs 6 to 13. It may be concluded that increasing the collection zone height (which increased residence the residence time correspondingly) led to a decrease in the recovery of coal to the concentrate and thus an improvement in selectivity.

Of the two sparger types tested, the filter cloth sparger resulted in marginally better flotation results, but most of these runs were conducted at a pulp density of 10%, whereas the USBM runs were conducted at 20% pulp density. If run 5 (the only 20% pulp density filter cloth sparger run) is compared with run 7, which had similar settings of air rate and HTAB concentration, it can be seen that the USBM gave a product of higher grade but lower coal recovery (22.9% ash vs 23.66%; 83.02% recovery vs 94.2%). This is a classic trade-off between recovery and grade, thus no inherent advantage to using the either the USBM or the filter cloth sparger can be deduced.

A final observation can be made that the HTAB requirement for the reverse flotation of Durnacol coal was less than for the Grootegeluk and Ermelo experiments. The best beneficiation results were obtained at a HTAB dosage of between 2000 and 3000 g/t. For the Grootegeluk and Ermelo experiments (see Tables 5.2 and 5.3 respectively) the best results were achieved at an HTAB dosage of between 3000 and 3500 g/t.

4.3.4 Comparison with forward flotation

The results of six forward flotation runs using a filter cloth sparger are presented in Table 5.5. The data are shown in exactly the same format as in Table 5.4, except that frother (DIBK) dosage is not shown in a separate column, since it was a constant at 15 μl/l for all the runs.

As can be seen, beneficiation was generally good: the product grades were all below 15% ash content and coal recoveries were greater than 80%. The best result obtained was a product containing 9.9% ash at 84.3% coal recovery, with a tailings ash content of 65.3% at 76.8% ash recovery. If this is compared to the best reverse flotation result (run 4, Table 5.4), it is obvious that the forward flotation process resulted in far better

Table 5.5: Forward flotation of Durnacol in the column cell.

No.	Yield (%)	Product ash (%)	Coal rec (%)	Ash rec (%)	Discards ash (%)	Shellsol g/t	Product sulphur %	Pulp density	Air rate cm/s
FILTER CLOTH SPARGER									
1	76.50	12.44	90.76	63.58	77.31	1000	1.37	20	2.11
2	77.57	12.80	93.53	64.13	79.14	1000	1.39	10	2.11
3	70.79	11.23	87.87	72.09	72.09	750		10	2.11
4	65.79	10.20	83.27	76.90	76.77	500		10	2.11
5	66.01	9.90	84.25	76.77	65.30	500		10	2.11
6	76.50	12.75	92.88	65.40	78.23	1000	1.42	10	2.11

FEED SULf:1.21%

FD ASH: 28.9%

separation of the Durnacol coal than did reverse flotation. In all six forward flotation runs, a marketable grade was obtained in the product, similar to that obtained in the flotation plant in the colliery (see section 2).

In terms of sulphur distribution, it may be seen from a comparison of Tables 5.4 and 5.5 that there is little to choose between the two processes. Both resulted in an increase in the sulphur content of the product, whether it was the tailings of reverse flotation or the concentrates of forward flotation. In the case where a high sulphur coal is to be floated, additional measures would have to be taken to reduce the sulphur content in the product. A possible solution for reverse flotation is that a pyrite collector be added to the reagent suite. This would exploit the fact that the coal is already depressed for the ash removal stage.

The two processes are compared graphically in Figure 5.6, which depicts the results of the reverse and forward flotation runs in relation to the washability and floatability data. Additional forward flotation runs from a column flotation study by von Holt (1991), using the USBM sparger, are shown for comparison purposes. As can be seen from Figure 5.6, some of the reverse flotation data points (indicated by hollow squares or stars) are close to (but above) the washability curve; they represent good results in terms of organic efficiency, but they lie in the high ash, high yield region of the washability curve which is undesirable. None of the results obtained represent a saleable product of thermal quality coal (15% ash or less).

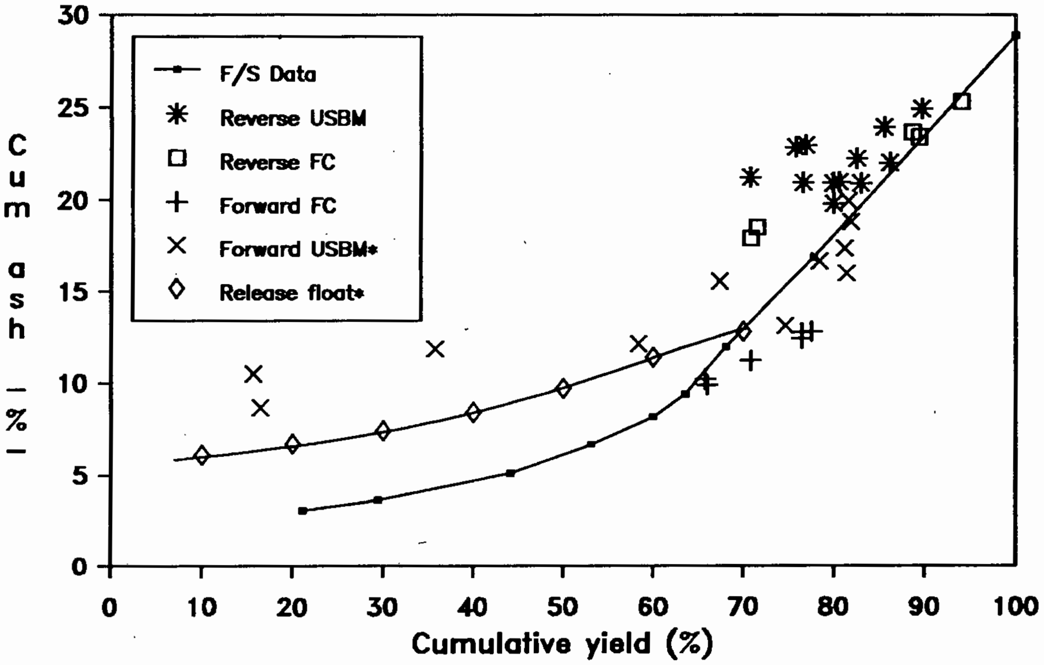

Figure 5.6: Durnacol flotation results in relation to washability and floatability (*data of van Holt, 1991).

For forward flotation, the situation is different. Many of the data points in Figure 5.6 lie below 15% ash, some representing a yield in excess of 70%. Some points are even situated below the washability curve indicating excellent organic efficiency [1]. Clearly, the flotation performance of forward flotation is superior to reverse flotation in terms of obtaining a product of marketable grade.

4.3.4.1 Discussion

It is clear from a comparison of the two processes that reverse flotation is a far less efficient process for beneficiating the Durnacol r.o.m. sample than is forward flotation. The problem is one of poor selectivity—ash recovery was poor, and a substantial amount of coal reported to the discard. This may have been as a result of coal associated with ash (i.e. not liberated), and/or as a result of liberated, clean coal that was misplaced in the discards.

Although reverse flotation of Durnacol gave inferior results when compared with forward flotation, the results were better than for the Grootegeluk and Ermelo coals. The ash recovery in the concentrates was greater and better grades relative to the feed grades were obtained in the product. Thus Durnacol coal enjoys the best beneficiation by reverse flotation of the three coals, which could possibly be due to better liberation and/or better floatability.

It was seen that the Durnacol feed contained 37% –0.025 mm material with an ash content of 41% (the feed ash was 29%), indicating that a substantial proportion of the ash occurred in the finer size fractions. This could be a detrimental factor in reverse flotation, since it is the ash that is floated, and ultrafine ash might be difficult to float; in the forward flotation process the coarser, low ash coal particles are floated. In order to investigate possible inefficiencies in reverse flotation in relation to the particle size, the two processes were investigated on the basis of size fraction. This is described in the next section.

4.3.5 Comparison of performance by size analysis

In the previous section it was shown that the poor selectivity of reverse flotation in comparison to forward flotation of r.o.m. coals was a result of deficient ash flotation and undesired recovery of coal in the concentrate discards. Possible reasons for the poor selectivity might be sought in the manner in which feed particles are distributed by size fraction, and how these particles differ in their response to flotation.

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[1] This also suggests that the washability data is under-estimating the liberation in these particular regions depicted on the graph

Size analyses were performed by the wet sieving technique on the tailings and concentrates of reverse flotation experiment 2 (Table 5.4) and forward flotation experiment 1 (Table 5.5). The reverse flotation run was chosen because it represents a good result in terms of ash flotation in the concentrate (53.15% ash recovery). The forward flotation run was chosen because it represents a good result in terms of coal recovery (90.76%) in the concentrate. Each is thus a good result (but not the best) in terms of the material that is to be floated in each process. In addition to the wet sieving, the same samples were sized by a Malvern particle size analyzer. This provides greater resolution of the very fine size fractions than is possible with a wet sieving technique. [1]

In addition to the size analyses, washability analyses were performed on the same concentrates and tailings samples of reverse and forward flotation in order to investigate differences in the liberation characteristics of the particles recovered in the product and the discard of each process. This analysis was performed by a centrifugal float and sink method, using a zinc chloride solution of R.D.=1.7 to determine separation.

The data for the wet sieving, Malvern and float and sink analyses are tabulated in Appendix XIII.

The wet sieving size analyses are depicted in Figures 5.7 to 5.10. Each graph shows: 1) the percentage yield in each size fraction based on the total (reconstituted) feed, 2) the percentage yield in each size fraction based on the feed occurring in that size fraction, and 3) the ash content (%) of the material in each size fraction. To clarify this, the +0.15 mm size fraction of the tailings sample represented in Figure 5.7 is taken as an example: the fraction of the total (reconstituted) feed that reported to the +0.15 mm fraction of this tailings sample was 15.05% (indicated by the left bar); this represented 94.2% of the feed material in this size fraction (indicated by the middle bar); and the ash content of the +0.15 mm fraction in the tailings sample was 6.98% (right bar).

If the size analysis of the reverse flotation tailings of (Figure 5.7) is compared with the size analysis of the forward flotation concentrates (Figure 5.9), which are both product, it can be seen that, in all the size fractions below 0.15 mm, forward flotation resulted in a greater yield (middle bars) with a lower ash content.[2] The differences between the two processes became more marked in the finer size fractions, particularly in the –0.025 mm fraction, where separation efficiency of reverse flotation was very poor: a 44% yield (of the –0.025 mm size fraction in the feed) at 36% ash was obtained, whereas forward flotation resulted in 50% yield at 13% ash.

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[1] Note: with the Malvern apparatus, the sample size used is very small and thus the results cannot be taken as absolute values. The feed cannot be reconstituted as in a wet sieving test, and this means that these results are not directly comparable to each other.

[2] In the +0.15 mm fraction the ash content was lower for reverse flotation.

Calculation shows that the amount of higher ash (unwanted) material reporting to the –0.025 mm fraction of the product was much greater for reverse flotation than for forward flotation. This can be demonstrated by the following mass balance, in which the basis is 100 g of reconstituted feed. In each case, the data refer to the same runs depicted in Figures 5.7 to 5.10:

–0.025 mm	**product Yield (g)**	**Ash %**	**Ash(g)**
Reverse (tails) :	16.8	36.0	6.05
Forward (concs):	19.1	13.2	2.52

It can be seen from this data that 6.05 g of ash reported to the tails of reverse flotation, while only 2.52 g of ash reported to the concentrates of forward flotation. The difference (3.53 g) should have been floated in reverse flotation, since it was rejected from the concentrates in forward flotation. In addition, much less coal was recovered in the tails of reverse flotation (10.75 g) than in the case of forward flotation (16.58 g).

If the size distribution of the reverse flotation concentrates (Figure 5.8) is compared to the size distribution of the forward flotation tailings (Figure 5.10), which are both discards, then the following can be observed: in each size fraction below 0.15 mm reverse, flotation resulted in a greater yield of material at a lower ash content. In the +0.15 mm fraction, the ash content was higher, and this was the only size fraction in reverse flotation which displayed better flotation efficiency than forward flotation. The difference in ash content in each size fraction was the greatest in the –0.025 mm fraction: in reverse flotation, the ash content was 55% for a 56% yield, whereas the ash content for the forward flotation material in this size fraction was 71% for a 49% yield. Thus, less ash was recovered in reverse flotation but, at the same time more coal was recovered. This can be shown in the following calculation:

–0.025 mm	**Discard Yield (g)**	**Ash %**	**Ash(g)**	**Coal (g)**
Reverse (concs)	21.2	54.0	11.45	9.75
Forward (tails)	18.3	70.9	12.97	5.33

As can be seen, 1.52 g more ash was rejected to the tails in forward flotation than was floated in reverse flotation. At the same time, 4.42 g more coal was recovered in the reverse flotation concentrate than in the forward flotation tailings. This undesired coal recovery is another indication of the fact that reverse flotation was far less selective than forward flotation, particularly in the –0.025 mm ultrafines.

It is interesting to note that this effect was also seen, although to a lesser extent, in the testwork on artificial mixtures. It can be seen from Figure 4.12 (Chapter 4), which represents the tailings of a reverse flotation run on a 50% quartz/coal feed, that approximately 25% of low ash coal which should have reported to the –0.025 mm size

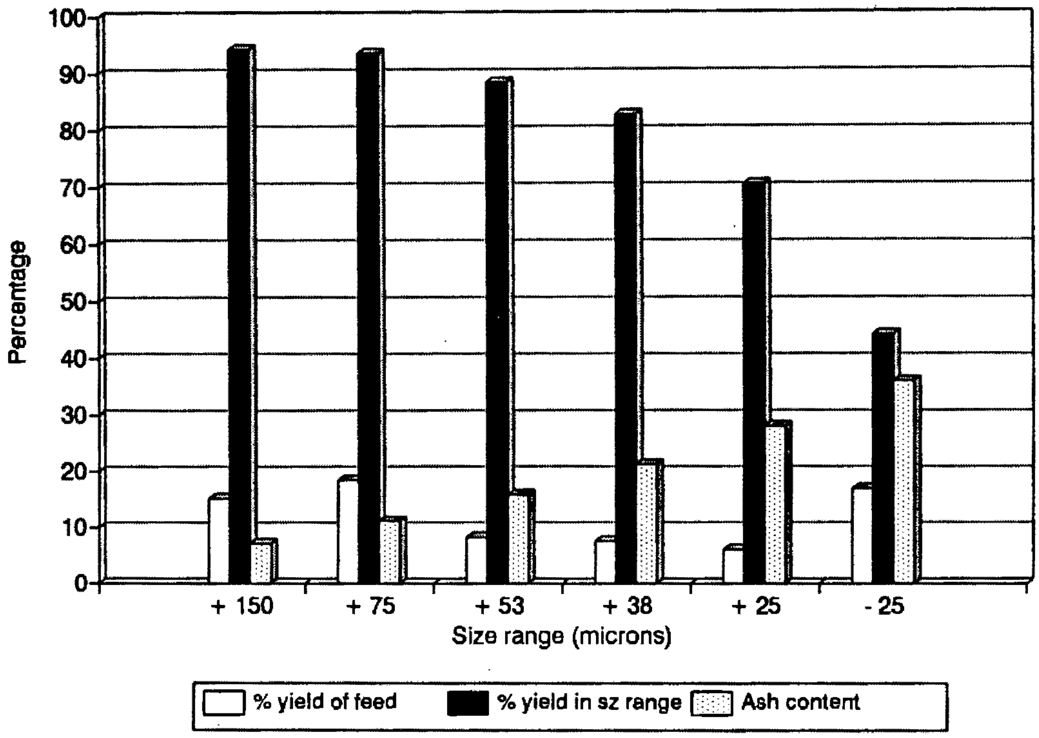

Figure 5.7: Particle size and ash distribution in reverse flotation tailings (run 2, Table 5.4).

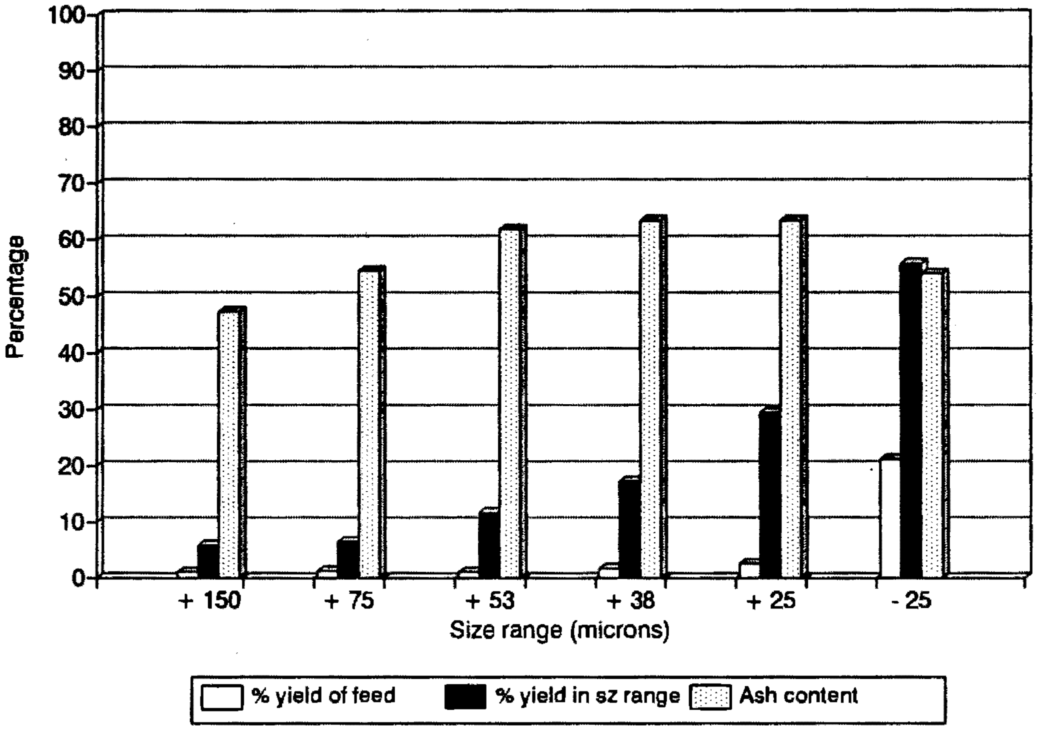

Figure 5.8: Particle size and ash distribution in reverse flotation concentrates (run 2, Table 5.4).

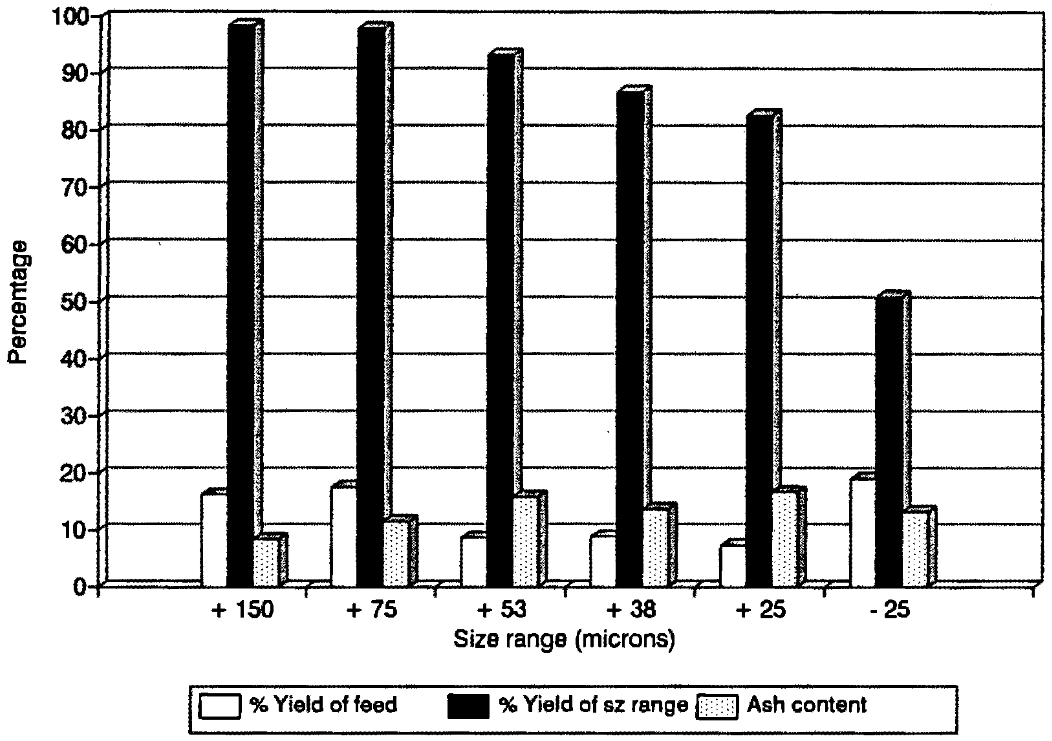

Figure 5.9: Particle size and ash distribution in forward flotation concentrates (run 1, Table 5.5).

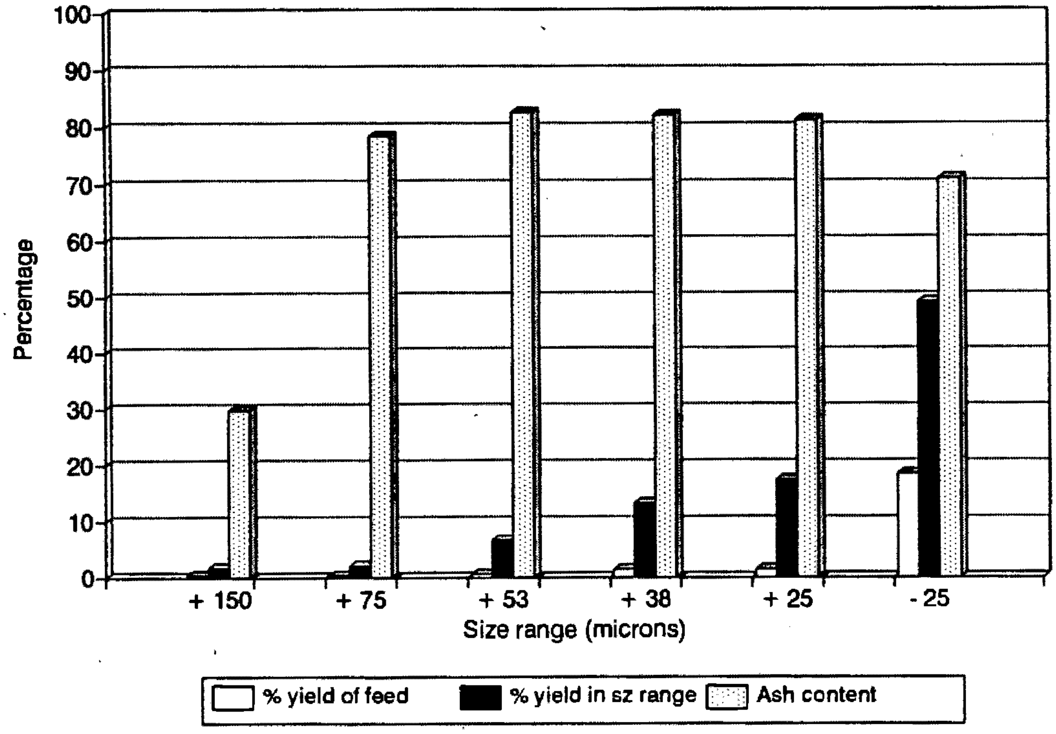

Figure 5.10: Particle size and ash distribution in forward flotation tailings (run 1, Table 5.5)

fraction of the product was lost to the discard. In contrast, the efficiency of separation was highest in the slimes for forward flotation of the artificial mixtures (see Figure 4.13). This indicates that the poor reverse flotation efficiency occurring in the –0.025 mm size fraction of both the artificial mixtures and the Durnacol coal was inherent to the process. The effect was, however, far worse with the r.o.m. coal than with the ideal coal.

Reverse and forward flotation are compared in Table 5.6 below. The two cases depicted are the same reverse and forward flotation runs discussed above (run 2 from Table 5.4 and run 1 from Table 5.4). The table shows the results of calculated mass balances (on the basis of 100 g reconstituted feed in each case), as well as measured concentrate water recoveries, size analysis, and the results of the float and sink analyses done on the samples at R.D.=1.7. The results of the Malvern size analyses are shown in the table as d_{50} data, i.e. the particle size below which 50% of the sample occurs. The full size distributions are shown in Appendix XIII.

First of all, it can be seen that the mass of ash recovered by reverse flotation in the discards was less than the mass of ash rejected in the tailings of forward flotation (15.0 g vs 18.2 g), and the "quality" of the material was worse: the reverse flotation concentrate contained only 56% material with a R.D. greater than 1.7 at 79.8% ash, whereas the tailings of forward flotation contained 87.24% at 84.9% ash.

At the same time, the amount of coal recovered in the reverse flotation concentrate was 13.45 g compared with only 5.33 g in the forward flotation tailings, which is a substantial difference. It may be observed from the float/sink data that 44% of the reverse flotation concentrate consisted of material which had a density less than R.D.=1.7, with an ash content of 14.8%, indicating that a substantial amount of clean coal had been recovered. The high coal recovery in the reverse flotation concentrate was thus more likely the result of liberated, clean coal particles reporting to the concentrate, rather than coal associated with recovered ash particles.

Turning to concentrate water recoveries, it is interesting to note in Table 5.6 that water was recovered at a rate of 7.21 kg/h in the reverse flotation run, whereas in forward flotation the rate was 1.26 kg/h. Thus, the froth of reverse flotation contained far more water than that of forward flotation.

Also of interest are the differences in the d_{50} values for the product and discards of each process. In reverse flotation, the d_{50} of the discard was 12.5 μm, whereas in forward flotation it was 10.1 μm. In the reverse flotation product the d_{50} was 64.0 μm, and in forward flotation it was 54.3 μm. This indicates that, in both process, the product was much coarser than the discard. This was to be expected, since the feed contained a significant proportion of high ash, ultrafine material.

Table 5.6: Column flotation of Durnacol: comparison of reverse and florward flotation

	REVERSE FLOTATION	**FORWARD FLOTATION**
Concentrates	**discard**	**product**
Coal mass (g)	13.45	66.98
Ash mass (g)	15.00	9.52
Water recovery (kg/h)	7.21	1.26
bias rate (cm/s)	0.21	0.29
ash content (%)	52.71	12.44
Yield (g)	28.45	76.50
Coal recovery (%)		90.76
Ash recovery (%)	53.11	
Malvern d_{50} (μm)	12.5	54.3
Float/sink data (R.D.=1.7)	44% floats at 14.8% ash 56% sinks at 78.9% ash	90.5% floats at 8.55% ash 9.5% sinks at 54.9% ash
	product	**discard**
Coal mass (g)	58.31	5.33
Ash mass (g)	13.23	18.17
Water recovery (kg/h)	29.54	36.72
ash content (%)	18.50	77.31
Yield (g)	71.55	23.50
Coal recovery (%)	81.25	
Ash recovery (%)		63.58
Malvern d_{50} (μm)	64.0	10.1
Float/sink data (R.D.=1.7)	81.6% floats at 9.6% ash 18.4% sinks at 64.9% ash	12.8% floats at 19.9% ash 87.24% sinks at 84.9% ash

4.3.6 Discussion

It has been shown that the poor efficiency of reverse flotation of the Durnacol coal was a result of a) the recovery of good quality coal in the discards, and b) the poor flotation of high ash material. These two effects were more pronounced in the finer size fractions of the feed, particularly in the –0.025 mm ultrafines. Reasons for these two effects are now proposed and discussed individually.

4.3.6.1 Possible reasons for misplaced coal in reverse flotation

In the reverse flotation experiments on the Durnacol coal, it was assumed that the clean coal particles in the feed would be depressed, since the depressant action of dextrin was clearly demonstrated in the artificial feed testwork (Chapters 3 and 4), and also in other studies (Miller et al., 1984). Depressed particles are, by definition, non-floating, and should not attach to air bubbles in the pulp, and should not be floated. Thus, coal particles in the Durnacol feed arising in the reverse flotation concentrates could only have been recovered in the froth by entrainment or entrapment.

It was noted in the discussion above (section 4.3.5) that the recovery of clean coal in the reverse flotation concentrates was worse in the finer sizes, particularly in the –0.025 mm size fraction. In fact, the mass of coal recovered in the –0.025 mm fraction of the concentrates of reverse flotation (9.75 g from the table on p 139) is 72% of the total mass of coal recovered in the concentrates (13.45 g from Table 5.6). This is consistent with an entrainment mechanism, since particle settling velocities decrease as the particle size becomes smaller, making particles more susceptible to entrainment in the froth water. This effect was also observed in the testwork on the artificial quartz/coal mixtures (see Chapter 4, Figure 4.12), but it was much less pronounced. However, the Durnacol sample has a much larger proportion of coal occurring in the –0.025 mm size fraction in feed. This can be seen in Figures 5.11 and 5.12, which show the size distributions of coal and quartz (or ash) in the 50% quartz/coal feed and the Durnacol feed, respectively.

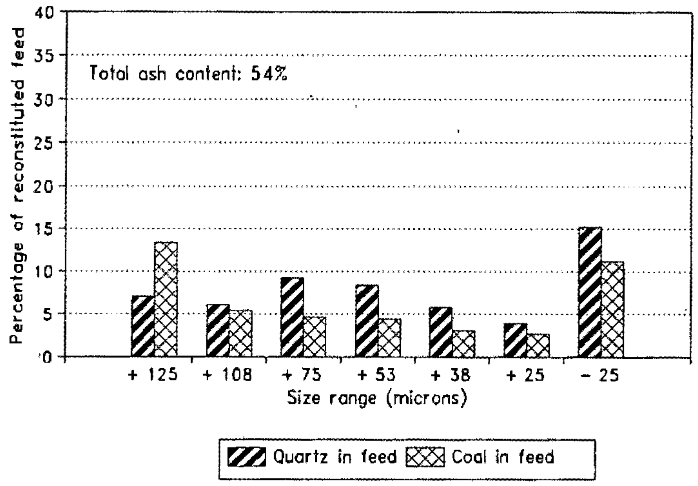

Figure 5.11: Distribution of coal and quartz by size fraction in the 50% quartz/coal feed mixture.

As can be seen in Figure 5.12, the Durnacol sample had 22% of the coal in the feed occurring in the –0.025 mm size fraction, whereas the artificial feed only had 11% coal in the –0.025 mm size fraction. It is thus not surprising that entrainment was more pronounced in the Durnacol sample.

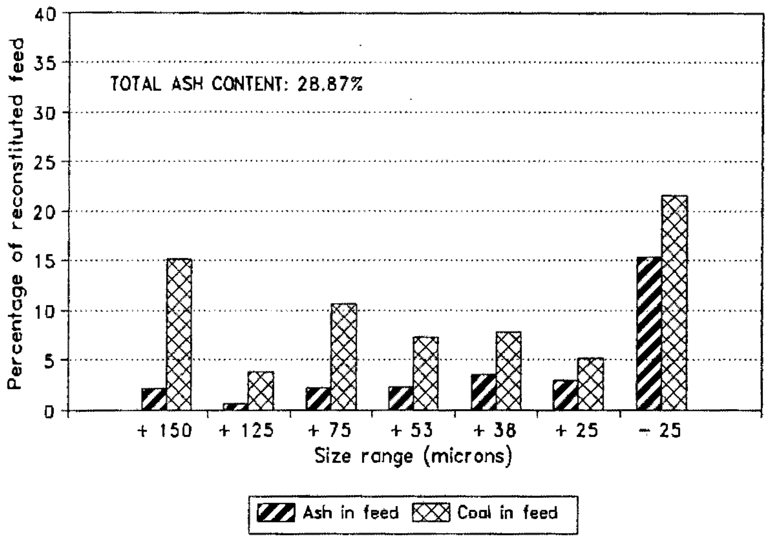

Figure 5.12: Distribution of coal and ash by size fraction — the Durnacol feed.

Normally it is assumed that entrainment is eliminated in a column flotation froth by the wash water providing a positive bias. However, it is possible that in the reverse flotation experiments the ultrafine low density coal particles had such low settling velocities that they were easily entrained into the froth and were then recovered in the concentrate by the upward transport of air and solids (i.e. ash), even though there was positive bias. It was seen in Table 5.6 that the water recovery in the concentrate of reverse flotation was very high in comparison to forward flotation. This could indicate a high froth velocity, which would "assist" the transport of entrained coal particles into the concentrate. By contrast, in the forward flotation process, entrainment would be less likely to occur, since ash particles are heavier than coal particles of the same size, and the water recovery was very low. In fact, recovery of quartz in the concentrates of forward flotation runs on the artificial mixture tests was found to be nonexistent (see Figure 4.13).

Studies of the nature of entrainment in flotation equipment have shown that entrainment is proportional to froth water recovery (Warren, 1985; see Chapter 4, section 2.3.1). These studies were performed in conventional, sub-aeration cells but it is most probable that the same phenomenon occurs in the column cell. Evidence for this can be found in Figure 5.13. This depicts the mass of coal recovered in the concentrates (assumed to be entrained) plotted against concentrate water recovery, for all the Durnacol reverse flotation runs, on a basis of 100 g of reconstituted feed (the water recovery data can be found in Appendix XII). The data points lie in two distinct regions; those representing the runs in which the filter cloth sparger was used, and those representing the USBM sparger results. Two observations can be made from the graph:

1) coal recovery is proportional to concentrate water recovery for both sparger types, and
2) the water recoveries for the USBM are much higher than for the filter cloth sparger.

The first observation is consistent with the theory that coal recovery is a result of entrainment in the reverse flotation froth. In terms of the second observation, it is interesting to note that HTAB was used as the frother and the collector in the USBM sparger runs, whereas DIBK was used as a frother (in conjunction with HTAB as the collector) in the filter cloth runs (see Table 5.4). It is thus likely that the water recoveries are linked to the frother *type*. HTAB (hexadecyl-trimethyl ammonium bromide) is a soluble ionic surfactant with strong frothing properties, and would be expected to produce a stable, wet froth. In contrast, DIBK (2,6-Dimethyl-4-heptanone) is a poorly soluble, polar surfactant with a relatively weak frothing action, and thus would be expected to produce a much drier, less stable froth than HTAB.

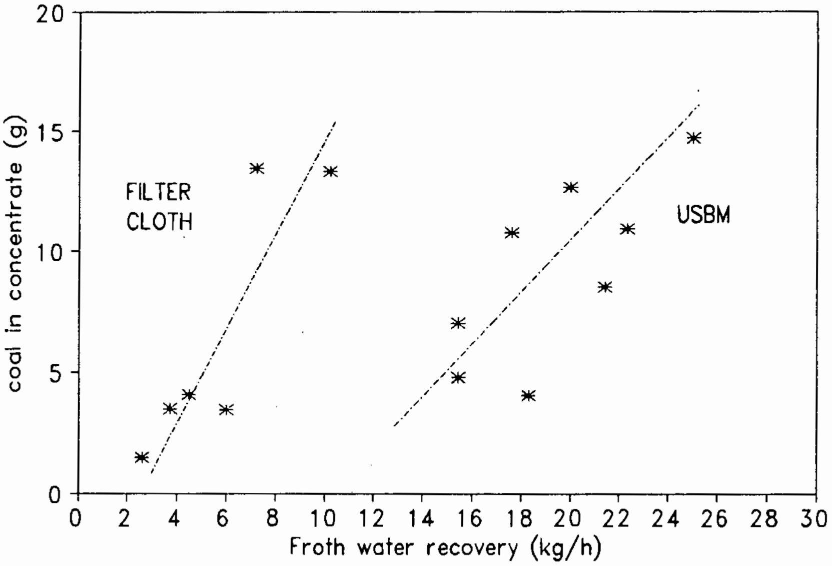

Figure 5.13: Concentrate coal recovery vs froth water recovery for the Durnacol reverse flotation experiments: USBM and filter cloth spargers

This has implications as regards the choice of the reagent suite for reverse flotation. It was initially proposed in Chapter 2 that HTAB could be used as the collector, depressant *and* frother. While this is a simple and arguably elegant solution, it might not be ideal in terms of limiting coal entrainment. Better metallurgical performance might be obtained in reverse coal flotation by reducing the HTAB concentration so as to minimize its frothing action, but retain its ash collector action, and employing DIBK (or a similar frother) in order to reduce froth water recovery and thus reduce the tendency for coal entrainment.

Another possible mechanism by which clean coal particles might have been recovered in the concentrate of reverse flotation is by the effect of sliming. By this it is meant that the ultrafine ash material coats the surface of clean coal particles, thus preventing the depressant from adsorbing onto them. Because the ash material is floated in reverse flotation, these slime coated coal particles would have attached to air bubbles and thus been floated. However, in forward flotation, these slimed coal particles would not be collected because of the hydrophilic ash coating; so this does not account for the greater inefficiencies observed with reverse flotation compared to forward flotation of the Durnacol coal. It is interesting to note, nonetheless, that the addition of an anti-sliming agent in the batch flotation tests on Rietspruit coal (see section 3) improved reverse flotation performance, although the effect was small. It may be concluded that if sliming did occur in the flotation of Durnacol, the effect was small enough to be masked by the greater effect of entrainment/entrapment.

4.3.6.2 Reasons for poor ash flotation in reverse flotation

It was seen from the Malvern particle size analyses performed on the tails and concentrates of reverse and forward flotation, that the material to be floated in the reverse flotation process had a high proportion of very fine high ash material. Any flotation process becomes less efficient as the particle size becomes very small; this effect was seen clearly in Chapter 3 with the flotation of ultrafine kaolin material (see Chapter 3, section 5.2). This would suggest that poor recovery of ash by reverse flotation might have been due to poor flotation response of these (high ash) slimes. This would be consistent with the results of the wet sieving analysis of the reverse flotation concentrate (Figure 5.8), which showed that the recovery of ash declined in the finer size (below –0.053 mm) fractions and was worst in the –0.025 mm size fraction. The forward flotation process (on the same feed) would be expected to be more efficient than reverse flotation, because the very fine ash would be rejected to the tailings and the coarser clean coal floated.

Poor ash flotation, although worse for the very fine size fractions, was not limited to these size fractions (see Figure 5.8). Thus, an additional explanation must be sought to account for poor ash flotation. A likely explanation is related to differing densities of ash and coal in the Durnacol sample, and how the degree of liberation of a particle will affect its floatability in both forward and reverse flotation:

Consider the forward coal flotation experiment depicted in Table 5.6 above. If an "average" particle that was floated is taken as having an ash content of 8.55% (the ash content of the floats at R.D.=1.7), it is a simple calculation to show that the ash content on a volume basis is only 4.4% [1], and this relates directly to surface area. For this average particle, 95.6% of the volume is coal and thus naturally hydrophobic, and the

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[1] taking the relative density of ash as 2.65, and that of carbonaceous coal as 1.3

hydrophobicity is enhanced by the addition of a collector oil to achieve flotation. For reverse flotation to achieve the same separation, the opposite would have to occur, i.e. an "average" particle of 84.9% ash (tailings sinks at R.D.=1.7) would have to be made hydrophobic in order to be floated. This particle would have a volume fraction of ash of only 73 % and hence such a particle might suffer from competing mechanisms; hydrophobicity imparted by the collector on 73% of the particle would be counteracted to a certain extent by the dextrin depressant making 27% of the particle hydrophilic. Depending on the relative strengths of these opposing effects, the particle might not have a strong enough hydrophobicity to be floated.[1]

In terms of this explanation, the floatability of the ash particles in reverse flotation (of the Durnacol coal) would be determined by the amount of associated coal, which is a function of the liberation characteristics of the feed. A coal with even a modest amount of middlings material might pose a problem, since these middlings would have to be floated (and the clean coal rejected to the tailings). The associated coal in these middlings particles, depending on the exact proportions, could make them poorly floatable in terms of reagent competition as described above. In contrast, in forward flotation clean coal is floated and middlings and high ash particles are rejected to the tailings. The clean coal is naturally hydrophobic and is easily floated by adding the collector oil. Thus, reverse flotation could suffer from an *inherent* disadvantage due to the liberation characteristics of the coal.

As noted above, it was seen that the Durnacol gave better reverse flotation performance than any of the other r.o.m. coals tested; at the same time it was better liberated. Excellent results were obtained with the artificial mixtures, which were "completely" liberated. This suggests that the liberation of the coal is indeed an important factor affecting the difference between reverse flotation and forward flotation performance for a particular coal.

4.3.7 Conclusion

Reverse flotation of Durnacol coal gave inferior beneficiation results than forward flotation did. The poor performance was explained in terms of insufficient ash flotation and undesired recovery of clean coal in the concentrate of the reverse flotation process.

The inefficiencies of reverse flotation relative to forward flotation occurred predominantly in the ultrafine size fractions. Size analyses revealed the presence of a significant amount of –0.025mm ultrafine material in the feed which contained a high proportion of ash.

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[1] One would expect then, that only very high ash particles would stand any chance of being floated. In fact, Table 5.6 shows that 56% of the material floated had an ash content of 78.9%, which is lower than the 84.9% "average" particle. However, it should be pointed out that an average particle was chosen merely to illustrate the phenomenon.

Coal entrainment or entrapment in the concentrate was thought to be the main cause of clean coal loss. Entrainment was proportional to the amount of froth water recovery. This, in turn, was related to the frother type and it was proposed that DIBK should be used as a frother to minimize water recovery and thus coal entrainment.

Poor flotation of ash was thought to be the result of two factors: 1) poor flotation recovery of the ultrafine ash particles, and 2) poor floatability of middlings particles. In spite of these inefficiencies, the reverse coal flotation process did succeed in obtaining a separation in the desired manner. This is encouraging, but there is considerable scope for improving the process and obtaining selectivities similar to those obtained with the reverse flotation tests on the artificial mixtures.

5. Conclusions

1. Reverse flotation of four different South African r.o.m coals did not result in an acceptable degree of beneficiation in either the batch cell or the laboratory column cell. This was in direct contrast to the excellent results obtain with artificial mixtures.

2. The column cell resulted in better cleaning than the batch cell, but the results were poor when compared with forward flotation of the same feed(s) in the column cell..

3. Of the four coals tested, the Durnacol coal gave the best beneficiation results by reverse flotation. This was also the coal with the best liberation and floatability.

4. The poor reverse flotation results on r.o.m. coals were attributed to coal entrainment, poor flotation recovery of ultrafine ash particles, and poor floatability of middlings.

5. In spite of its poor performance relative to forward flotation, the reverse flotation process did work for the r.o.m coals. This is encouraging and future studies might well be able to improve the efficiency of the process. The incentive for these studies would be to realize the advantage of reverse flotation in column cells, namely increased throughput due to a reduction in concentrate mass recovery. This advantage was demonstrated in the testwork on the artificial quartz/coal feeds

Clearly the revere flotation process is not viable for the four South African r.o.m. coals tested in this study in terms of obtaining a product of marketable yield. However, it was clearly demonstrated in the reverse flotation testwork on artificial feeds that the process is highly successful and that the process has a potential advantage over forward flotation of greatly increased throughput in column flotation cells. It would thus be a pity if the process were rejected because the feeds employed in this study were simply unsuitable for the process. It is recommended that the process be investigated further and applied to coals that are more amenable to reverse flotation than the r.o.m. coals used in this study.

6. Recommendations for obtaining improved reverse flotation performance

In order for reverse flotation to give beneficiation results similar to forward flotation, coals should be obtained which are similar to the artificial mixtures. This implies that 1) the coal feed should not contain a large amount of ultrafine material, so as to reduce the effects of coal entrainment and poor flotation of ultrafine ash particles, and 2) it should be well liberated, so as to have as little middlings material as possible.

Both these criteria might be met by a Northern Hemisphere (Carboniferous) coal. In contrast to South African (Gondwanaland) coals, these coals generally are lower in ash content and have better liberation characteristics because the mineral matter is predominantly epiginetic in origin. This also means that such coals do not have to be ground down to very fine sizes to achieve liberation. An example of a North American coal is illustrated in Figure 5.14, which shows the washability data of a Pennsylvanian coal from the "Sewickly" seam (EPRI, 1988), together with that of the Durnacol sample used in this study, and the 25% quartz/coal mixture used in the artificial mixture experiments. A striking feature of the diagram is that the Pennsylvanian coal has a remarkably similar washability to the quartz/coal mixture (which is assumed to have perfect liberation) but is actually better!

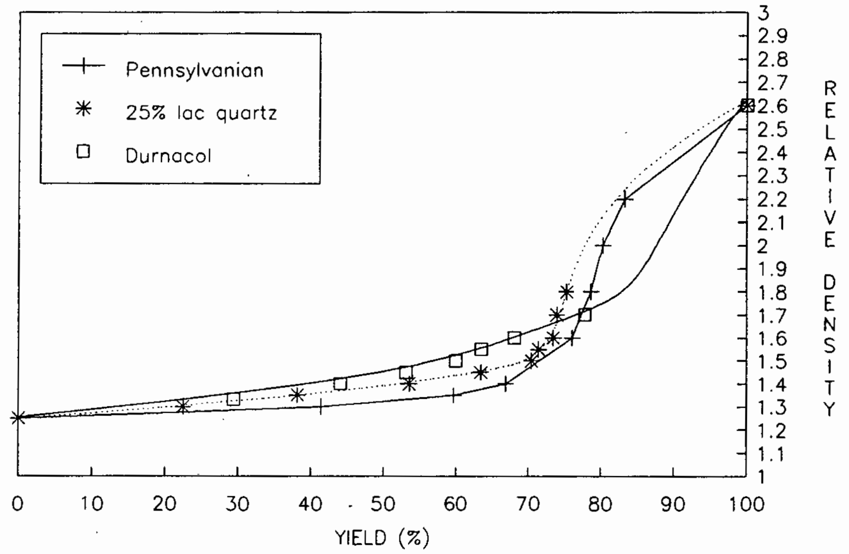

Figure 5.14: Comparison of the washabilities of a Pennsylvanian coal (EPRI, 1988), Durnacol, and an artificial mixture.

The Durnacol coal, in stark contrast, has a poor washability. Even though it is good by South African standards, it is clearly different from either the artificial mixtures or the Pennsylvanian coal. This implies two things: 1) the artificial mixtures were not a good approximation of South African r.o.m. coals, and 2) reverse flotation might well be highly successful on the Pennsylvanian coal given the similarity of the liberation to the artificial mixtures. It is thus recommended that reverse coal flotation should be tried out on a

Northern Hemisphere coal, such as the Pennsylvanian coal given as an example above. If the process can be made to work, then a major advantage of the process over forward flotation, could be realized: increasing throughput and/or reducing the equipment requirement of a coal column flotation plant.

Another recommendation is that the reagent suite for reverse flotation be re-evaluated in the light of the discovery that high water recoveries occur when using the quaternary amines as frothers. Alternative frothers should be sought to reduce water recovery and thus reduce the possibility of entrainment of clean coal in the concentrates (and the consequent loss of product coal recovery).

CHAPTER 6
CONCLUSIONS AND RECOMMENDATIONS

A reverse flotation process has been developed for the beneficiation of fine coal. In this process, the ash forming minerals (a small fraction of the feed) are rendered hydrophobic, and are floated as a concentrate, whereas the bulk of the feed, namely the coal, is rendered hydrophilic and reports to the tailings. The flotation concentrate is discarded and the tailings recovered as product.

The process was developed to reduce both the high mass recovery which is inherent in conventional coal flotation, and the associated gangue entrainment which adversely affects the grade of the product.

The reagents required for the process are: an <u>ash collector</u>, a <u>coal depressant</u>, and a <u>frother</u>. An investigation of the surface chemical structure of coal, and of the gangue minerals usually associated with coal, led to the selection of long chain cationic quaternary amine salts to fulfill all of the above requirements. Three amines were selected for investigation, namely: hexadecyl-trimethyl ammonium bromide (HTAB), dodecyl-trimethyl ammonium bromide (DTAB), and hexadecyl-pyridinium chloride (HYPC). Dextrin was selected as an additional coal depressant, and MIBC or DIBK as additional/alternative frothers.

Adsorption studies with HPYC on "pure" coal and quartz indicated that the quaternary amines would be appropriate for use both as a gangue collectors and coal depressants. Dextrin adsorption on coal was found to compete with that of the amine; it was deduced that if dextrin is to be used as an additional depressant, it should be conditioned with coal prior to amine addition to obtain the maximum effect.

Reverse batch flotation tests using artificial feed mixtures consisting of blends of low ash, washed coal and pure quartz or kaolin showed that the three amines tested are good ash collectors and frothers. Coal entrainment was a problem, but this was reduced by means of a staged reagent addition procedure and the use of dextrin as an additional depressant. The amine HTAB, in conjunction with dextrin, gave the best separation of the three amines. Maximum coal depression was achieved when dextrin was added to the feed prior to amine addition. Using a 25% quartz/coal feed, with a feed ash content of 31%, coal recovery of approximately 90% was obtained at an ash content of between 10 and 11%. Conventional forward flotation of the same artificial mixtures in the batch cell gave similar metallurgical results; however, reverse flotation demonstrated a clear advantage in terms of low concentrate mass recovery.

Reverse flotation in a laboratory column cell showed the process to be highly successful. Improved froth cleaning was obtained (relative to the batch cell) which resulted in increased recovery of clean coal in the tailings. Using the same 25% quartz/coal feed as above, coal recovery increased to 93 to 95%, with a sightly better product quality. The kinetics of reverse flotation were found to be fast; these results were produced in a relatively short column (1.0 m collection zone and 0.5 m froth zone) which corresponds to a residence time of approximately 5 minutes. Aeration rate was an important parameter at values less than 2 cm/s; for air rates greater than 2 cm/s, there was very little effect on performance. An HTAB (frother and collector) dosage of 1200 g/t was found to be adequate.

Forward flotation of the artificial mixtures in the column cell gave slightly better metallurgical results to reverse flotation, with the product ash content even lower than that of the low ash, washed coal used to make up the artificial mixtures. However, forward flotation was severely constrained by the low carrying capacity of the column: when operating in this mode, the coal recovery dropped steadily as the feed rate was increased beyond 1.3 t/h/m^2. In contrast, in reverse flotation, the carrying capacity of the column had not yet been reached at three times this feed rate, with no sacrifice in either coal recovery or product quality. Thus, reverse flotation in column cells appears to offer an attractive alternative to conventional coal flotation in terms of good metallurgical performance at high feed rates.

However, reverse flotation of four South African run-of-mine coals gave disappointing results. A product of marketable grade was not obtained for any of the coals evaluated. In contrast, forward flotation tests did produce acceptable grades. The poor performance of reverse flotation on the run-of-mine coals may be explained by 1) entrainment of clean coal particles in the discard, and 2) poor floatability of ash material.

The entrainment of clean coal particles in the concentrate discard may be attributed to their ultrafine size (much finer than in the coal used in the artificial mixtures, and in greater proportion), and to the very high froth water recoveries observed in reverse flotation (seven or more times greater than those found in forward flotation). This latter phenomenon is likely to have been caused by the use of the quaternary amines, which are vigorous frothers.

Poor ash floatability may be ascribed to the fact that the ash material in the run-of-mine coal was extremely fine. Flotation efficiency is known to become poorer with decreasing particle size. An additional factor is thought to be lack of liberation; the best liberated run-of-mine coal gave better results than the others, while the artificial mixtures, which were "perfectly" liberated, gave excellent results.

It is recommended, in order to reduce entrainment, that the amine dosage be decreased to only that required for collector action, and that frothers which result in less water recovery, such as DIBK, be employed in reverse flotation. Additionally, in view of the success of reverse flotation on artificial mixtures, especially in column cells, it is recommended that the process be applied to run-of-mine coals which more closely resemble the artificial mixtures, i.e. having similar liberation characteristics and feed size distribution. Northern Hemisphere coals are generally very well liberated, and liberation is usually achieved at much coarser sizes than South African coals.

SUGGESTIONS FOR FUTURE WORK

The following are suggestions for specific areas of focus for future studies of the reverse coal flotation process:

1. Clarification of gangue selectivity and amine depressant action on clay minerals, with fundamental studies such as electrokinetic (zeta potential) experiments, contact angle determination and micro-flotation experiments. This might involve the selection of additional/different types of reagents for reverse flotation.

2. Investigation of the effect of gangue particle size on recovery in reverse flotation by determining response of different (milled) size fractions of quartz: e.g. a range from −150 μm to −25 μm.

3. A coal entrainment study in a column cell using a salt tracer to investigate the extent of coal entrainment in the froth at different bias rates using both artificial mixtures and r.o.m. coals. This would include the investigation of the use of different frother types as a means of reducing water recovery and thus reducing entrainment.

4. Investigatation of the use of a combined reagent suite to improve gangue selectivity of r.o.m. coals; for example the addition of pyrite collectors to remove sulphur.

5. A test of the reverse flotation process on well-liberated North American coal and/or European coal types.

6. The identification and evaluation of commercially available amine reagents, and an economic feasibility study of the process.

REFERENCES

Abdel-Khalek, N.A. and Stachurski, J.. "The effects of surface oxidation and grain size on the flotation of coal with column flotation and conventional flotation". Paper Presented at SME Annual Meeting, Salt Lake City, Utah. February 26–March 1, 1990.

Adamson, A.W. Physical chemistry of surfaces. 2nd Edition. John Wiley, New York. 1967.

Aplan, F. F. "How the nature of raw coal infuences its cleaning". from Industrial Practice of Fine Coal Processing. Klimpel, R.R. and Luckie, P.T., editors. Society of Mining Engineers, Inc. Littleton, Colorado, 1988. pp 99–112.

Amelunxen, R.L. "Column Flotation: New carrying capacity considerations for scale-up". Cominco Engineering Services Ltd. Paper presented at EXPOMINERIA '90, Santiago, Chile, May 14–18, 1990.

Arnold, B.J. and Aplan, F.F. "The effect of water carry-over in a bank of industrial coal froth flotation cells with a comparison to laboratory test results". Paper Presented at SME Annual Meeting, Salt Lake City, Utah. February 26–March 1, 1990.

BP Statistical Review of World Energy. BP pubication, pp 26–28, June 1990.

Bustamante, H. and Warren, L.J. "Relation between the relative density fo composite coaly grains and their flotation recovery". *Int. J. Min. Proc.* Volume 10, 1983. pp95–111.

Buys, I.E. A liberation study on ultrafine South African coals. M.Sc. Thesis. University of Cape Town, 1989.

Crawford, R and Ralston, J. "The influence of particle size and contact angle in mineral flotation". *Int. J. Min. Proc.* Volume 23, 1988. pp 1–25.

Davies, J.T. and Rideal, E.K. Interfacial phenomena. Academic Press. London. 1961.

Deetlefs, C. and Johnson, A. Reverse flotation of coal. Undergraduate project. University of Cape Town. 1986.

Dell, C.C. "The analysis of flotation test data". *Colorado School of Mines Q.* Volume 56, 1961. pp 115–127.

Dell, C.C., and Bunyard, M.J. "Development of an automatic flotation cell for the laboratory". *Trans. Inst. Min. Met.* Volume 81. 1972. pp C246–C248.

DMEA (South African depatment of Mineral and Energy Affairs). South African Discard and Duff Coal National Inventory 1985. Government Printer, Pretoria, 1987.

DMEA. Operating and Developing Coal Mines in the Republic of South Africa. 1986. Government Printer, Pretoria. July, 1986.

DMEA. South Africa's Mineral Industry 1988. Government Printer, Pretoria, 1989

Dobby G. S. and Finch J. A."Mixing characteristics of industrial flotation columns". *Chem. Eng. Sci.* Vol 40, no 7, pp1061–1068, 1985

Dobby, G.S. and Finch, J.A. "Flotation column scale-up and modelling". CIM Bulletin, May 1986.

EPRI (Electric Power Research Institute). Laboratory Guidelines and Procedures for Coal Analysis. Volume 1: Assessing the Cleanability of fine coal. EPRI CS-5644 Interim Report. May, 1988.

Falcon, R.M.S. "Coal in South Africa, Part I: The Quality of South African Coal in Relation to its Uses and World Energy Recources". *Minerals Science Engineering*. Volume 9, No 4, 1977.

Falcon, R.M.S. "Coal in South Africa, Part II: The application of Petrography to the Characterization of Coal". *Minerals Science Engineering*. Volume 10, No 1, 1978.

Espinosa-Gomez, R., Finch, J.A., Yianatos, J.B. and Dobby, G.S. "Column carrying capacity: particle size and density effects". *Minerals Engineering*, 1(1), pp77–79, 1988.

Fickling, R.S. An investigation into the froth flotation of four South African coals. M.Sc. Thesis, University of Cape Town, 1986.

Finch, J.A. and Dobby, G.S. Column Flotation. Pergamon Press. 1990.

Franzidis, J-P. Developments in fine coal beneficiation in South Africa. University of Cape Town, 1990.

Groppo, J.G. and Parekh, B.K. Continuous pilot-scale testing of column flotation for recovery of fine coal. Mining Engineering. October, 1990. pp 1189–1192.

Gy, P.M. Sampling of particulate materials: theory and practice. Elsevier Science Publishers. 1982.

Hanning R. N. and Rutter P. R. "A simple method of determining contact angles on particles and their relevance to flotation". *Int. J. Min. Proc*, Vol 27, pp133–146, 1989.

Harris, M.C. Liberation characteristics of Greenside Coal. M.Sc. Thesis. University of Cape Town. 1987.

Harris, M.C., Franzidis, J-P., Moody, D. and Buys, I.E. Flotation and dewatering of fine coal. Part 1: the chraracterization and flotation of coal fines from the Rietspruit Colliery. Report prepared for Shell Coal. University of Cape Town, August 1986.

Horsfall, D.W. "Cost effective routes in fine coal beneficiation". in Industrial practice of fine coal processing, Klimpel, R.R. and Luckie, P.T., eds. S.M.E. Littleton, Colorado, U.S.A. Chapter 34, pp 365–374, 1988.

Horsfall, D.W. (ed.) Coal Preparation for Plant Operators. 3rd edition. Coal Processing Society. Johannesburg. 1980. pp 6–10.

Horsfall, D.W. and Franzidis, J-P. "Beneficiation of South African coal fines". in Proceedings of the 5th Annual International Pittsburg Coal Conference. Pittsburg, PA. September 12–16, 1988, pp 311–325.

Ives, K. The scientific basis of flotation. Nato ASI Series E. Martinus Nijhoff. The Hague. 1984.

Jowett, A. "Formation and distribution of particle-bubble aggregates in flotation". Chapter 37. Fine Particles Processing. International Symposium. 1980

Jowett, A."Gangue mineral contamination of froth". *British Chemical Engineering*. Vol.II, No. 5. May, 1966. pp330–333.

King R. P. (ed). Principles of flotation. South African Institute of Mining and Metallurgy Monograph Series No. 3. Johannesburg. 1982. pp144–147.

Klimpel, R.R. and Hansen, R.D. "Chemistry of fine coal flotation". in Fine coal processsing. Mishra, J.D. and Klimpel, R.R. eds. Noyes Publications. New Jersey. 1987. pp. 78–106.

Laplante A. R., Yianatos J. and Finch J. A. "On the mixing characteristics of the collection zone in flotation columns". in Column flotation '88. Proceedings, International Column Flotation Symposium, Phoenix, AZ. ed. Sastri. pp. 69–79.

Laskowski, J. "Flotation of difficult-to-float coals". Proceedings of the 10th International Coal Preparation Congress. Canadian Institute of Mining and Metallurgy. Edmonton, 1986. Volume 1, pp. 122– 142.

Levenspiel, O. The Chemical Reactor Omnibook. Corvallis, Oregon, U.S.A., O.S.U. Book Stores, 670, 1979.

Lin, I.J. and Metzer, A. "Co-adsorption of paraffinic gases in the system Quartz Dodecyl-ammonium-chloride and its effect on froth flotation". *Int. J.Min.Proc.* No. 1, pp. 319-334. 1974.

Lin, K.F. and Burdick, C.L. "Polymeric depressants". in Reagents in Mineral Techology. Somasundaran, P. and Moudgil, B.M. (editors). Marcel Dekker, New York. 1988. pp. 471–484.

Miller, F.G. "The Effect of Froth Sprinkling on Coal Flotation Efficiency". *Soc. of Min. Engineers. AIME Transactions*, Vol. 244. pp. 158–167. June, 1969.

Miller, J.D., Lin, C.L. and Chang, S.S. "Co-adsorption phenomena in the separation of pyrite from coal by reverse flotation". *Coal Preparation*. Volume 1. pp. 21–38. 1984.

Miller, J.D., Laskowski, J.S. and Chang, S.S. "Dextrin adsorption by oxidised coal". *Colloids and Surfaces*. Volume 8. 1983. pp. 137–151.

Mills, P.J.T. and O'Connor, C.T. The modelling of liquid and solids mixing in a flotation column. Minerals Engineering. Volume 3, No. 6, 1990. pp. 567–576.

Mishra S.K. "Improved recovery of fine coal by flotation process". in Fine coal processsing. Mishra, J.D. and Klimpel, R.R. eds. Noyes Publications. New Jersey. 1987. pp. 110–135.

Mougdil, B.M. "Effect of polyacrilamide and polyethylene oxide polymers on coal flotation". *Colloids and surfaces*. Vol. 8, p 227. 1983.

Nicol, S.K., Roberts, T., Bensley, C.N., Kidd, G.W. and Lamb, R. "Column of ultrafine coal: experience at BHP-UTAH Coal Limited's Riverside Mine". Proceedings, in Column flotation '88. Proceedings, International Column Flotation Symposium, Phoenix, AZ. ed Sastri.

Osborne, D.G. Coal preparation technology. Graham and Trotman. London. 1988. pp. 1–19.

Parekh B.K., Groppo J.G., Stotts W.F. and Bland A.E. "Recovery of fine coal from preparation plant refuse using column flotation". in Column Flotation 88, Proceedings, International Column Flotation Symposium, Phoenix, AZ. ,ed. Sastri. pp. 227–233.

Perry, R.H. and Chiltern, C.H. Chemical Engineer's Handbook. 5th Edition. McGraw-Hill. International Student Edition. New York. 1973. p B–26.

Petrick, A.J., van Rensburg W.C.J. and Vos, D. Commission of enquiry in the convervation of South Africa's coal reserves. Government Printer, Pretoria, 1975.

Picard, J.L. Coal Preparation Washing Process: A Technology Review. CANMET Report 85-2E. March, 1985.

Reddy, P.S.R., Kumar, S.G., Bhattacharyya, K.K., Sastri, S.R.S. and Nărasimhan, K.S. "Flotation Column for Fine Coal Beneficiation". *Int.J.Min.Proc.*, Vol 24, 1988, pp. 161–172.

Sanders, G.J. and Brookes, G.F. "Preparation of the Gondwana coals. I. Washability characteristics". *Coal Preparation*, Vol 3, No. 3, pp. 105–132, 1986.

Shaw, D.J. Introduction to colloids and surface chemistry. 3rd Edition. Butterworths. London. 1980.

Smith, J.M. Chemical reaction kinetics. 3rd Edition. McGraw-Hill. International Student Edition. 1984. pp. 275–278.

Smith, R.W. "Cationic and Amphoteric collectors". from Reagents in Mineral Techology. Somasundaran, P. and Moudgil, B.M. (editors). Marcel Dekker, New York. 1988.

Smith, R.W. "Co-adsorption of dodecylamine ion and molecule on quartz". *Trans. AIME* 226. pp. 427–433. 1963.

Stach, E. Stach's Textbook of Coal Petrology. Second Edition. Gebrüder Borntraeger. Stuttgart. 1975.

Tsai, S.C. Fundamentals of coal beneficiation and utilization. Elsevier, Amsterdam. 1982.

Tsiperovich, M.V. and Evtushenko, V.Ya. "Preparation and carbonisation of coals". Volume 1, Sverdlovsk, *Metallurgizdat*, 72, 1959.

Trahar, W.J. "A rational interpretation of the role of particle size in flotation". *Int. J. Min. Proc.*, 1981, pp. 289–327.

Von Holt, S.T. Unpublished work. University of Cape Town, 1991.

Voges, H.C. "Tests on the beneficiation of coal fines". *J. S. Afr. Inst. Min. Metall.* Vol. 91. No. 2. 1991. pp. 41–51

Warren, L.J. "Determination of the contributions of true flotation and entrainment in batch flotation tests". *Int. J. of Min Proc.* 14, 1985, pp. 33–44.

Weiss, N. L. SME Mineral Processing handbook. Volume 1. Society of Mining Engineers of AIME. New York. 1985. p 5.49.

Ward, C.R. Coal Geology and Technology. Blackwell Scientific Publications, 1984. pp. 34–39.

Wills, B.A. Minerals Processing Technology. 3rd Edition. Pergamon Press, Oxford, 1985.

Yianatos, J.B. "Column flotation, Modelling and technology". Paper presented at SAIMM conference Developments in Froth Flotation. Held at Gordons Bay, South Africa, August 3–4, 1989.

Yianatos, J.B., Finch, J.A. and Laplante, A.R. "Holdup profile and bubble size distribution of flotation column froths". *Canadian Metallurgical Quarterly*, 25 (1), pp. 23–29, 1986.

Yianatos, J.B., Finch, J.A. and Laplante, A.R. "Selectivity in column flotation froths". *Int. J. Min. Proc.* 15, 1988, pp. 11–14.

Ynchausti R.A., McKay J.D. and Foot D.G. Jr. "Column flotation parameters — their effects". in Column flotation '88, Proceedings, International Column Flotation Symposium, Phoenix, AZ. ed. Sastri. pp. 157–172.

APPENDIX I

SOUTH AFRICAN COALS

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1. Gondwana and Carboniferous coals

South African coals fall under the Gondwana coal group of the Southern Hemisphere originating from the Permian era and differ from the Carboniferous and Tertiary coals of the northern hemisphere in a number of ways:

1.1 Maceral structure

Carboniferous coals contain mostly Vitrinite, while Gondwana coals contain up to 70% Inertinite. There is a much wider seam variation from coal to coal and Exinite is rather insignificant when compared with Carboniferous coals. Figure I.1 illustrates the these differences in the form of a bar graph.

On account of their low Vitrinite content, Gondwana coals are comparatively low in Hydrogen while high in Nitrogen content. The substantial proportions of Fusinite and Semi-Fusinite in Gondwana coals result in a higher moisture content (due to their higher porosity) and lower heating values than those of Carboniferous coals of the same maturity. The volatile matter of South African coals (for a given rank) is comparatively low and is directly linked to their low Vitrinite content.

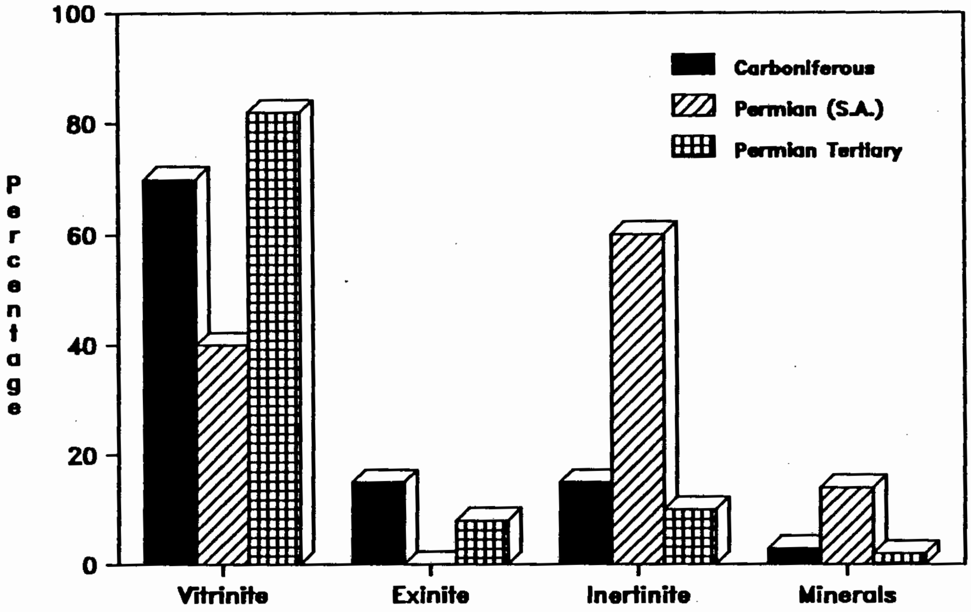

Figure I.1: Relative maceral proportions of S.A. and major world coal types (after Falcon, 1977)

1.2 Mineral matter

In addition to the purely organic substances, both the macerals and the microlithotypes of coal contain amounts of inorganic components. These can be classified into two groups according to their origin:

- **Syngenetic Minerals**
  This refers to inorganic matter from the original plants , minerals introduced to exposed decaying debris by water or wind during the early phase of the coalification process; and transport of debris from its original site to another. In this case silts were often intimately mixed into the debris so as to become colloidally dispersed. Syngenetic minerals are finely intergrown with the organic matter in the coal macerals.

- **Epigenetic minerals**
  This refers to minerals deposited during the metamorphic stage of the coalification process (after consolidation of the coal), by depositions in cracks, fissures or cavities and by deposition from percolating mineral waters.

In South African coals the mineral matter is mainly syngenetic and is often intergrown in the Inertinite maceral which makes mineral liberation and thus beneficiation difficult. The inferior washability characteristics of S.A. coals has already been shown in Chapter 2 (see Figure 2.6). Also, South African coals have generally high ash levels (Falcon, 1977).

1.2.1 Clay minerals

The most important Syngenetic minerals are the various clays which occur as microscopic particles intimately intermixed with the coal macerals. Clay represents up to 70% of the syngenetic mineral matter in Gondwana coals. The major clay type is Kaolinite (china clay). Kaolinite is one of the most common sheet-structure aluminosilicates minerals. Other clay types commonly found in S. A. coals are Illite (Muscovite) and Chlorite, which are both three-layered sheet structures.

1.2.2 Silica (Quartz)

Quartz is mainly an epigenetic mineral and makes up between 10 to 20% of the mineral matter in S.A. coals. It occurs as a characteristically coarse material blown or washed into the coal swamps and is often found in discreet layers, which can be useful for determining coal seams. Quartz may also be found intimately associated with clays.

1.2.3 Sulphur minerals

The main form of mineral sulphur is pyrite, which occurs in a disseminated form. The sulphur content is generally a lot lower than in Carboniferous coals: the sulphur levels are usually in the range 0.5% to 1.3% (air dry basis). Table I.1 shows a few S.A. coals as regards their ash content and relative amounts of mineral and organic sulphur. These are quite variable and there is no significant relationship of the total sulphur content with the ash content for different coal types.

Table I.1: Sulphur levels of selected S.A. coals (DMEA, 1986)

Colliery	Coal type	Ash %	Mineral S %	Organic S %	Pyrite FeS_2 %
New Denmark	Crushed coal	22.2	0.35	0.65	0.65
Old crown Douglas	Duff	30.3	1.08	0.27	2.02
New Vaal	Crushed coal	36.2	0.42	0.15	0.79
Zululand Anthracite	Premium 8 X 0 mm	8.9	0.04	0.73	0.07
	Middling	19.6	0.43	0.58	0.80

1.2.4 Other minerals

Siderites occur in nodules up to a few millimeters in diameter and is preferentially associated with Vitrinite.

The phosphorous content of Gondwana coals is higher than Carboniferous coals, the source being Apatite.

2. Coal types and coalfields of South Africa

The coal seams which are mined in S.A. today were formed about 200 million years ago and occur in the so-called Eca beds of the Karoo system. The coalfields were laid down from fresh water deposits in horizontal beds. The coals have been formed from a wide variety of plants varying from the simplest forms to ferns and large trees. The dominant plants contributing to the formation of S.A. coal were rather different from those of the Northern Hemisphere.

Figure I.2 is a map showing the principal coal seams of South Africa The major coalfields are described below as regards their formation, seam type and coal quality.

The coal deposits in South Africa were formed in three basic types of deposits (taken from Falcon, 1977; Horsfall, 1980)

2.1 OFS and SW Transvaal

Deposits in this region were fan-type in pre-existing glacial valleys. Mineable seams are less than 3 m thick and dull in type. The ash contents vary from very high percentages laterally to lower percentages centrally in the pre-existing valleys

The main coalfields in this region come from the seams occurring south of Vereeniging. There are three main seams; numbered 1,2 and 3 from the bottom. The no.1 seam is variable in thickness and quality. In the north the thickness is greatest at 15 m and quality is best; ash content is as low as 15%. Generally, however, the ash content is over 20%.

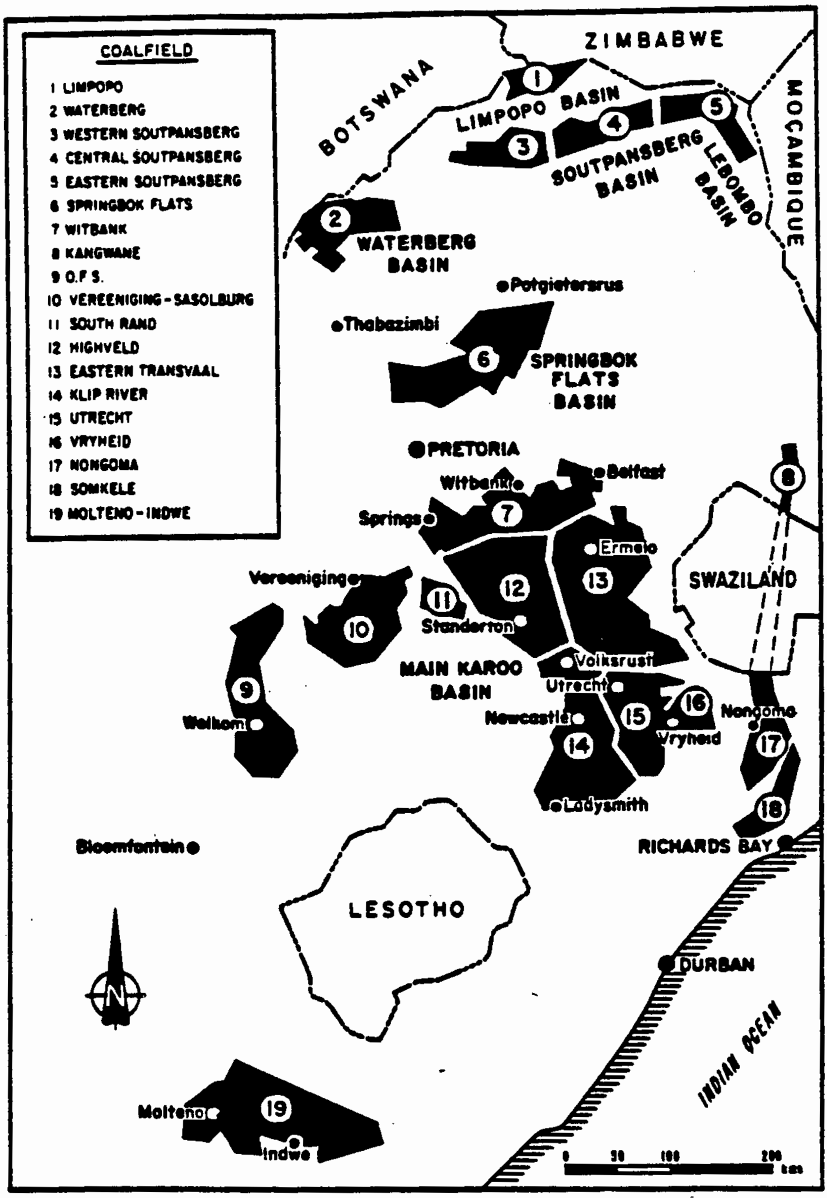

Figure I.2: South African coalfield locations
(DMEA, 1989)

2.2 E. Transvaal and N. Natal

Deposits were stable shelf fluvial plain deposits. There are five or more thin seams of bright or dull coal less than 2 m thick. In the Witbank-Middelburg region of the Eastern Transvaal, five seams are mined, numbered no.1 to no.5 and identified by these numbers. No.1 seam contains mainly dull coal, seam thickness is 2 m at most and the ash content is 15% or less. The no.2 seam is the main economic seam and has been the most extensively worked. It is normally over 6 m thick, contains dull coal with bright bands at the bottom and the ash content varies from 13% to 18%. Another important seam is the no.4 seam. Seam thickness is 3 m or more, the ash content is generally over 15% and exceeds 20% at places. The coal is mainly dull in type.

In Natal, there are two major coalfields; the Klip River Area and the Vryheid/Newcastle area. In the Klip River, two main seams are utilized. The seam thickness varies between 1 m and 2 m but in places can be up to 4 m. The seams consist mainly of bright coal and coking properties are often present. The ash content varies from about 12% to 20%.

In the Vryheid/Newcastle area, four major seams are mined. They are called (from the bottom) Coking, Dundas, Gus and Alfred. The Coking seam is about 1 m thick, is a bright coal and has an ash content of around 10%. The three other seams are also bright in appearance, normally between 1 m and 2 m thick and ash contents vary from around 12% to 20%.

The Vryheid coalfield extends into the Transvaal where it is known as the Ermelo coalfield. It has the same seam sequence although they are named B, C, D and E corresponding to the Vryheid seams. The main seam is the C seam which is 1.5 m thick, consists mainly of bright coal and has an ash content of 13% to 18%.

2.3 N. Transvaal and Lebombo

Coals in this region are from the so-called Lacustrine deposits in fault bounded troughs. Two main coalfields are mined, the Waterberg and the Soutpansberg basin.

The Waterberg coalfield is characterized by coal bearing zones. These zones are found up to 30 m thick and consist of narrow coal bands, interspersed with dirt bands. The raw ash content of these zones can be up to 50% and the coal contains closely intergrown mineral matter, making beneficiation difficult. For example, in order to obtain a blend coking coal of 10% ash, the yield is only 12% with an additional 18% yield of 35% ash middlings.

The Soutpansberg field is currently not utilized. It has been extensively investigated, though, and found to contain high quality coking coal. The field has similar geological characteristics as the Waterberg field and presents similar beneficiation problems.

The table below depicts the specific qualities of some of the coals from major collieries with respect to their uses, properties, ash content and sulphur content. A few northern hemisphere coals are included for comparison.

P.T.O

Table I.2: S.A. coal types and characteristics (DMEA, 1986), and three Northern Hemisphere Coals (Ward, 1984)

Coalfield	Colliery	Type of coal	V.M. %	Ash %	Total S %	C.V. Mj/kg
Witbank-Mdlbg	Greenside no.2 seam	Duff coal	23.7	18.4	0.68	25.9
Witbank-Mdlbg	Landau	Low ash	31.8	7.5	0.59	30.9
Northern Tvl	Grootegeluk	Middlings	28.6	32.6	0.98	20.9
Vereeniging, OFS	Sigma	minus 6 mm	20.9	29.1	0.53	18.7
Vierfontein OFS	Vierfontein	crushed	22.9	23.5	1.50	21.0
Klip river	Durban Navigation	Bit. coking	28.8	11.3	1.4	30.8
Natal	Zululand Anthracite	Premium 8 X 0 mm	5.9	8.9	0.77	31.3
Witbank-Mdlbg	Secunda	Liquefaction crshd	23.1	21.1	0.94	23.2
Lancashire, UK	Crombouke seam	Carboniferous	32 - 39	3 - 8	1.0 - 3.6	29 - 33
Pennsylvania, USA	Lwr Kittaning seam	Carboniferous	21.3	8.6	1.5	32.26
Wilcox, Alabama	Wilcox group	Tertiary	23.6	8.4	2.7	13.35

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now $A = La_L$

assume that a_{Lc} = ash of sample with lowest ash content, then a_{LC} used in euations (8) and (9) in place of a_L for concentrate cumulative recoveries.

For the tails sample: $A_T = A - a_{Lc}L_c$

$$\text{Actual tails ash} = \frac{(G - G_c) + A_T}{M_T} \qquad (10)$$

and

$$L_c = M_c - \frac{M_c(a_m - a_{Lc})}{(a_g - a_{Lc})} \quad \text{(from eqns 3\&4)}$$

The LAC ash content of the tails is calculated as follows

$$a_{LT} = \frac{A_T}{L - L_c} \qquad (11)$$

Example: See sample spreadsheet 3

M_c = 211.25 g, M_T = 87.07 g, so T = 298.32 g

a_L = 0.072, a_{Lc} = 0.068, a_m = 0.0688 (6.88% ash)

q = 0.25 (25% added Kaolin) a_m (tails) = 0.757

G = 74.58 g, G_c = 0.18, L = 223.74 g, A = 16.11 g

$$L_C = 211.25(1- \frac{(0.0688-0.068)}{(0.86 - 0.068)}$$

$$= 211.04 \text{ g}$$

and $A_T = 16.11 - 0.068*211.04 = 1.760$ g

using equation (11), $a_{LT} = \dfrac{1.76}{223.74 - 211.04} = 0.1386$ (13.86%)

tails ash (10) = $\dfrac{(74.58 - 0.18) + 1.760}{87.07} = 0.8746$ (87.46%)

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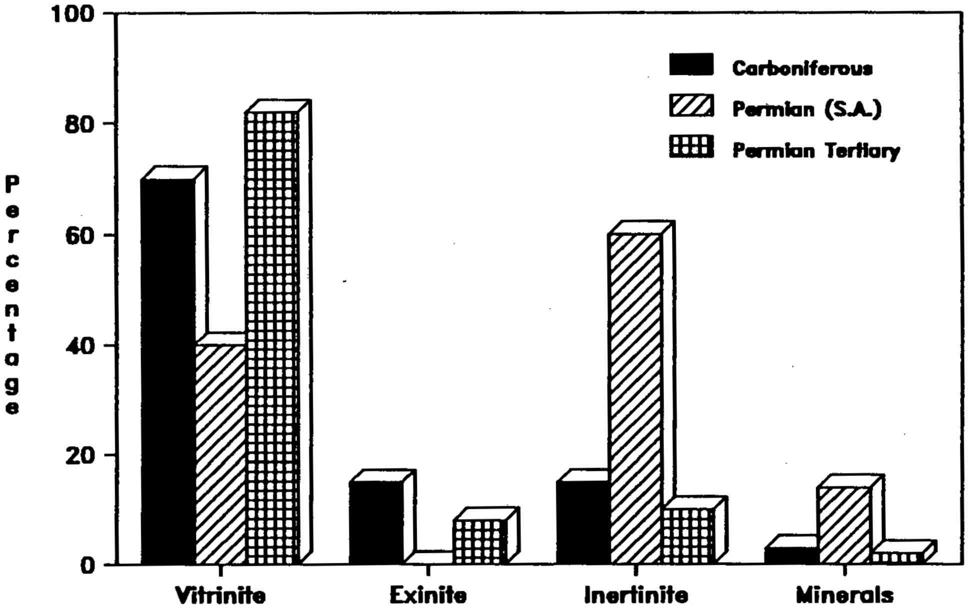

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  This refers to inorganic matter from the original plants , minerals introduced to exposed decaying debris by water or wind during the early phase of the coalification process; and transport of debris from its original site to another. In this case silts were often intimately mixed into the debris so as to become colloidally dispersed. Syngenetic minerals are finely intergrown with the organic matter in the coal macerals.

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$$= \frac{M_{ci} - \dfrac{M_{Ci}(a_{mi} - a_L)}{(a_g - a_L)} + \sum_{j=1}^{i-1} M_{Cj} - \dfrac{M_{cj}(a_{mj} - a_L)}{(a_g - a_L)}}{M_c + M_T - G_c - G_T} \qquad (8)$$

(adapted from equations (3) and (4))

b)Concentrates cumulative gangue recovery for each sample is given by:

$$= \frac{\dfrac{M_{ci}(a_{mi} - a_L)}{(a_g - a_L)} + \sum_{j=1}^{i-1} \dfrac{M_{cj}(a_{mj} - a_L)}{(a_g - a_L)}}{G_c + G_T} \qquad (9)$$

c)Concentrates cumulative ash calculation:

$$\text{cumulative ash} = \frac{a_{mi}M_{ci} + \sum_{j=1}^{i-1} (a_{mj}M_{cj})}{\Sigma M_{ci}}$$

d) Recostituted feed

feed mass: $T = M_C + M_T$

ash content: $\dfrac{G_T + G_C + (L_c + L_T)a_L}{T}$

4. Case where LAC is beneficiated. (section 1 assumption invalid)

In some cases in forward flotation, it has been found that the ash content of the concentrates is less than the LAC ash content. This means that the LAC is actually beneficiated. To calculate recoveries, it is assumed that no gangue is recovered in the concentrate sample wich has the lowest ash content and this ash content is taken as the LAC ash content of the concentrates.

Specify a_{Lc} = LAC ash fraction of concentrates
a_{LT} = LAC ash fraction of tailings
q = mass fraction added gangue of original feed
G = Total gangue mass in original feed
L = Total L.A.C. mass in original feed
L_c = LAC in the concentrates
A = Total ash in L.A.C. in original feed
A_T = mass LAC ash in tails

Then $T = M_c + M_T$ and $G = qT$ so $L = T - G$

now $A = La_L$

assume that a_{Lc} = ash of sample with lowest ash content, then a_{Lc} used in euations (8) and (9) in place of a_L for concentrate cumulative recoveries.

For the tails sample: $A_T = A - a_{Lc}L_c$

$$\text{Actual tails ash} = \frac{(G - G_c) + A_T}{M_T} \qquad (10)$$

and

$$L_c = M_c - \frac{M_c(a_m - a_{Lc})}{(a_g - a_{Lc})} \qquad \text{(from eqns 3\&4)}$$

The LAC ash content of the tails is calculated as follows

$$a_{LT} = \frac{A_T}{L - L_c} \qquad (11)$$

Example: See sample spreadsheet 3

$M_c = 211.25$ g, $M_T = 87.07$ g, so $T = 298.32$ g

$a_L = 0.072$, $a_{Lc} = 0.068$, $a_m = 0.0688$ (6.88% ash)

$q = 0.25$ (25% added Kaolin) a_m (tails) $= 0.757$

$G = 74.58$ g, $G_c = 0.18$, $L = 223.74$ g, $A = 16.11$ g

$$L_c = 211.25(1- \frac{(0.0688-0.068)}{(0.86 - 0.068)}$$

$$= 211.04 \text{ g}$$

and $A_T = 16.11 - 0.068*211.04 = 1.760$ g

using equation (11), $a_{LT} = \dfrac{1.76}{223.74 - 211.04} = 0.1386$ (13.86%)

$$\text{tails ash (10)} = \frac{(74.58 - 0.18) + 1.760}{87.07} = 0.8746 \text{ (87.46\%)}$$

SPREADSHEET CALCULATIONS - R.O.M COALS

A sample spreadsheet is given below (page 9) for flotation runs (both forward and reverse) where R.O.M. coals are used instead of artificial mixtures. The various calculations are as follows:

A_c = Ash mass in concentrate
a_c = ash fraction in concentrate
C = Total concentrate mass
i = denontes concentrate sample i.

C_c = combustible coal in conc
a_t = ash fraction in tails
T = total tails mass.
n = number of conc. samples

1. Cumulative recoveries

For each concentrate sample i, the ash and coal recoveries are given by

$$A_{ci} = C_i a_{ci}, \qquad C_{ci} = C_i - A_{ci}$$

The cumulative ash recovery for each sample i is given by:

$$\frac{\underline{\text{Ash mass in sample i + sum of previous ash masses}}}{\text{Total ash in concentrates and tails}}$$

$$= \frac{A_{ci} + \sum_{j=1}^{i-1} A_{cj}}{Ta_t + \sum_{j=1}^{n} A_{ci}}$$

The cumulative coal recovery is given by:

$$\frac{\underline{\text{Coal mass in sample i + sum of previous coal masses}}}{\text{Total coal in concentrates and tails}}$$

$$= \frac{C_{ci} + \sum_{j=1}^{i-1} C_{cj}}{T(1-a_t) + \sum_{j=1}^{n} C_{ci}}$$

2. Cumulative concentrate ash

The cumulative ash percentage of the concentrate samples is given by:

$$\frac{\underline{\text{ash in sample i + sum of previous ash masses}}}{\text{total concentrates mass}} \times 100$$

$$= \frac{A_{ci} + \sum_{j=1}^{i-1} A_{cj}}{C} \times 100$$

LAC ash	7.20
Gng Ash	100.00

SAMPLE WORK-SHEET FOR QUARTZ

CONC NO.	MASS (g)	ASH %	Cum. LAC REC. (%)	Cum. Quartz REC. (%)	Cum. ASH (%)	Time s	Quartz	LAC	Org coal	Ash
1	40.81	94.21	1.64	27.70	94.21	30	38.26	2.55	2.36	38.45
2	22.18	91.07	3.01	42.20	93.10	60	20.05	2.13	1.98	20.20
3	13.70	89.12	4.05	50.96	92.39	120	12.09	1.61	1.49	12.21
4	40.98	88.78	7.24	77.03	91.13	150	36.03	4.85	4.60	36.38
5	18.33	87.70	8.80	88.54	90.67	160	15.90	2.43	2.25	16.08
6	9.60	67.05	11.00	93.02	89.11	240	6.19	3.41	3.16	6.44
	145.60 g						128.52	17.08	15.85	129.75

Tails mass:	147.87	g	Ash:	13.25	%	Org. Coal mas	128.3	g	Coal Rec:	89.00%
Concentrates: mass:	145.60	g	Ash:	69.11	%	Ash mass:	129.8	g	Ash rec:	86.88%
Feed mas	293.47	g	fd ash:	50.89	%					

Tails ash:	19.59	LAC in Tails:	138.23	Total LAC:	155.31
Total ash:	149.34	Qtz in Tails:	9.64	Total quartz:	138.16
Tot coal:	144.13				

LAC ash	7.20
Gng Ash	86.00

SAMPLE WORK-SHEET FOR KAOLIN

CONC NO.	MASS (g)	ASH %	Cum. LAC REC. (%)	Cum. KAOLIN REC. (%)	Cum. ASH (%)	Time s	Kaolin	LAC	Org coal	Ash
1	20.25	82.00	0.67	13.85	82.00	30	19.22	1.03	0.95	16.61
2	22.18	80.12	1.76	28.65	81.02	60	20.52	1.66	1.54	17.77
3	13.70	79.85	2.46	37.75	60.73	120	12.63	1.07	0.99	10.94
4	12.56	78.23	3.27	45.91	60.27	150	11.32	1.24	1.15	9.83
5	10.87	75.34	4.23	52.68	79.60	160	9.40	1.47	1.36	8.19
6	9.60	72.68	5.29	58.43	78.86	240	7.98	1.62	1.51	6.98
	89.16 g =========						81.08	8.08	7.50	70.31

Tails									
mass:	202.36 g	Ash:	29.66 %	Org. Coal mas	142.3 g	Coal Re	88.30%		
		Crctd:	33.85 %						
Concentrates:									
mass:	89.16 g	Ash :	78.86 %	Ash mass:	70.3 g	Ash rec:	53.95%		
		Crctd:	91.01 %						
Feed mas	291.52 g								
		fd ash:	44.71 %						
		crctd :	51.37 %						

Tails ash:	60.02	LAC in Tails:	144.68	Total LAC:	152.77
Total ash:	130.33	Kao in Tails:	57.68	Total kaolin:	136.75
Tot coal:	161.19				

FLOTATION WORKSHEET

Run id:	KF1	Coal:	225.00	g	Cell id:	top denver
		Kaolin	75.00	g		

Collector:	_	Coll. Concentration:	_	g/t	st 1
		:	_	g/t	st 2
Molecular Wt.:	-	:	_	g/t	st 3

Float PH:	7.60	Air Rate :	4.00	litres/min	FEED LA	7.20
Frother	MIBC	Froth.Concentration	18	micro-litre/l	GANGUE:	86.00
Anti Sliming:	none	AS concentration:	0.00		CONC LA	8.80
Pulp Density:	9.94%	Froth height :	2.50	cm		

SAMPLE WORKSHEET FOR KAOLIN

CONC NO.	MASS (g)	ASH % Measured	Cum. LAC REC. (%)	Cum. Kaolin REC. (%)	Cum. Crctd Ash %	Time s	LAC	Kaolin	Tot ash	False ash
1	40.81	8.80	18.24	0.00	6.80	60	40.81	0.00	2.78	2.78
2	55.23	6.89	42.90	0.08	6.86	180	55.17	0.06	3.81	3.81
3	48.56	6.92	64.57	0.18	6.89	300	48.49	0.07	3.37	3.36
4	32.87	6.87	79.25	0.22	8.89	480	32.84	0.03	2.26	2.26
5	19.99	8.81	88.18	0.23	6.88	720	19.99	0.00	1.36	1.36
6	13.79	6.88	94.34	0.24	6.88	720	13.78	0.01	0.95	0.95
	211.25 g						211.07	0.18	14.53	14.51

Tails mass:	87.07	g	Meas. Ash:	75.70	%	Org coal:	10.9	g	Coal Rec:	5.26%
			Corrected:	87.48	%					
Concentrates: mass:	211.25	g	Crctd Ash:	6.88	%	Ash mass:	14.5	g	Ash rec:	16.03%
Feed ma	298.32	g	Calc feed: ash	30.40	%					

Tot Kaolin	74.58	Tails Kaolin	74.40	
Tot LAC:	223.74	Tails LAC	12.67	
Tot LAC ash:	16.11	Tails LAC ash	1.76	
Total Ash:	90.69	Tails LAC ash	13.86	%
Tot Org. coal	207.63			

Run id: PST02 Coal: Grootegeluk Cell id: Denver bottom imp.

Collect.:	Pegasol 3745	Coll. Conc:	3000.00 g/t
Frother :	MIBC	Froth Conc:	12.00 ul/l
Float PH:	not set	Pulp Dens :	6.00 %
		Air Rate :	4.00 l/min
Depress :	none	Depr. conc:	0.00 g/t
Anti Slm:	none	AS conc :	0.00 g/t
		Froth ht :	1.50 cm

MASS (g)	ASH %	Cum. COAL REC. (%)	Cum. ASH REC. (%)	Cum. ASH (%)	Time s	Coal	Ash
20.62	12.67	15.33	3.19	12.67	15.00	18.01	2.61
18.30	12.65	28.94	6.01	12.66	30.00	15.99	2.31
5.32	12.82	32.89	6.85	12.68	60.00	4.64	0.68
2.92	13.39	35.05	7.32	12.72	120.00	2.53	0.39
1.68	13.84	36.28	7.61	12.76	210.00	1.45	0.23
1.01	14.05	37.02	7.78	12.79	390.00	0.87	0.14
49.85 g						43.48	6.37

149.52 g	Tails Ash 50.53 %	Coal in Tails:	73.97 g
25.00 %	Conc Ash 12.79 %	Ash in Tails:	75.55 g
	Calc. feed ash: 41.09 %		

APPENDIX VII

BATCH FLOTATION RESULTS

a) Artificial mixtures: Initial flotation tests

INITIAL STUDY - REVERSE FLOTATION USING 3 AMINES

	Cumulative recoveries (percent)																							
Name:	**R1Q1**		**R1Q2**		**R1Q3**		**R1Q4**		**R1Q5**		**R2Q1**		**R2Q2**		**R2Q3**		**R2Q4**		**R3Q1**		**R3Q2**		**R3Q3**	
Time (s)	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz
30	1.25	2.83	5.37	39.33			1.95	11.23	4.69	25.30	0.49	2.50	7.16	47.31					0.35	0.44	2.19	20.06	9.82	52.38
60	2.68	5.76	10.63	69.65	3.39	29.97	10.05	37.60			1.13	5.69	15.06	76.27	8.48	33.95	11.91	25.55	0.89	1.12	4.56	39.67	22.44	81.89
90									9.15	38.64			19.20	86.51	23.71	50.48	30.49	37.04			7.26	56.35	28.21	90.37
120	4.99	9.91	15.83	87.09	9.26	48.20	16.78	51.72	15.33	45.30					38.16	58.10	44.84	54.52	1.44	1.60	9.22	68.52	30.39	92.93
150															54.13	71.25								
180									21.54	56.03	3.65	16.74	23.02	91.90			60.07	77.65	2.15	2.68	10.24	72.52	32.38	94.55
210															68.17	84.45								
240	8.05	14.70	20.62	94.71	12.55	56.27	40.85	61.00			4.67	20.54												
300									25.79	63.76							65.01	82.14	3.54	4.34				
330															73.68	90.11								
360	9.92	17.27	26.75	96.42	12.79	57.08	63.10	75.57			5.26	22.60	24.91	93.97									34.31	95.96
420									26.97	87.03														
480	11.24	18.93	34.14	97.28			81.49	90.43									65.37	82.8						
540											5.93	24.76												
720	12.45	20.22	49.42	98.1																				
Product ash	52.00		10.78		38.30		38.77		35.22		47.52		13.97		33.03		38.85		50.56		28.99		12.85	
Coal Tails re	87.55		50.56		87.21		16.51		73.03		94.07		75.09		26.32		34.63		96.46		89.78		65.69	
Conc ash %	65.18		70.28		83.34		55.96		72.56		81.49		80.24		58.65		59.68		55.47		88.54		75.8	
AMINE TYPE	DTAB		DTAB		DTAB		DTAB		DTAB		HTAB		HTAB		HTAB		HTAB		HPYC		HPYC		HPYC	
Conc (g/t)	198		354.3		870		875.6		1018.7		483.3		890.3		1338.7		1000		391.67		828		881	

b) PH experiments

PH STUDY : RECOVERY-TIME DATA

Name:	PHQ3R1		PHQ3R2		PHQ3R3		PHQ4R4	
Time	Cumulative recs		Cumulative recs		Cumulative recs		Cumulative recs	
(s)	LAC	Quartz	LAC	Quartz	LAC	Quartz	LAC	Quartz
30	0.51	31.93	0.57	27.59	0.63	38.99	0.73	34.26
60	1.99	73.44	2.53	71.35	2.47	79.94	4.05	71.08
90	4.06	87.98	7.81	84.47	5.65	87.59	9.52	78.58
120	8.41	91.74	13.02	88.71	10.95	89.39	14.5	84.27
180	18.54	93.59	20.03	92.73	19.7	93.31	20.09	91.14
240								
300	25.83	95.85	24.99	94.66	23.19	95.18	22.46	94.86
360								
Product ash %	12.22		13.25		12.81		12.98	
LAC Tails rec	74.17		75.01		76.81		77.54	
conc ash %	80.65		80.31		82.17		82.26	
Amine	HPYC: 810 g/t		HPYC: 812 g/t		HPYC: 625 g/t		HPYC: 625 g/t	
pH:	3.4		7.6		7.6		3.4	

c) Entrainment study: separate flotation of LAC and quartz

SEPARATE FLOTATION TESTS: LAC ONLY

Name:	1C1	1C2	1C3	1C4	1C5	2C1	2C2	2C3	2C4	3C1	3C2	3C3	3C4
Time (s)	Cumulative coal recovery												
20													4.53
30	0.08	0.24	0.26	0.32	1.11			5.27					
40													13.38
60	0.31	0.66	0.76	0.93	3.54	0.03	0.51	17.01	2.12	0.17	0.35	0.21	18.81
90		1.12	1.30	1.54	6.38			20.26					22.02
120					9.22	0.11	0.84	21.7	3.89	0.35	0.63	0.73	
150	1.73	2.13	2.52	2.97									
180					15.36			28.7					24.34
240						0.32	1.12		4.79				
270	1.73	2.84	3.64	4.24									
300								36.76					25.47
360					30.86					0.51	1.01	1.35	
Product ash %	6.8	6.66	6.54	6.43	6.66	6.78	6.74	6.58	6.7	6.84	6.88	6.81	6.58
LAC Tails rec	98.27	97.16	96.36	95.76	69.15	99.68	98.88	63.24	95.21	99.49	98.99	98.65	74.53
Amine TYPE	DTAB	DTAB	DTAB	DTAB	DTAB	HTAB	HTAB	HTAB	HTAB	HYPC	HYPC	HPYC	HYPC
CONC (g/t)	216.6	335.2	435.2	553.2	810	404	628.6	896.6	726	438.6	567.2	748	988

c) Entrainment study: separate flotation of LAC and quartz (cont.)

SEPARATE FLOTATION TESTS: QUARTZ ONLY

Name:	1Q1	1Q2	1Q3	1Q4	2Q1	2Q2	2Q3	2Q4	3Q1	3Q2	3Q3	3Q4
Time (s)	**Cumulative quartz recovery**											
20								13.82			15.39	13.81
30	3.62	14.06	10.10	21.39	32.16	40.86	33.83		20.27	28.88		
40								65.4			52.81	59.4
60	10.35	41.84	27.15	55.76	70.16	75.54	56.52	81.60	37.20	54.90	67.79	75.58
90				70.69				88.18				
120	20.24	66.24	47.94	78.26	75.15	81.03	60.09		42.58	60.55	71.72	82.2
180		76.63		85.48				90.58				
240	32.41		64.11	90.29	76.871				45.11	62.3	73.78	85.95
360		86.01				82.93	61.58					
420			70.51									
Amine TYPE	DTAB	DTAB	DTAB	DTAB	HTAB	HTAB	HTAB	HTAB	HPYC	HYPC	HYPC	HYPC
CONC (g/t)	164	239	221	340	278	335	209	405	179	224	277	380

c) Entrainment study: flotation of LAC with MIBC frother only quartz

	CUMULATIVE RECOVERIES (PERCENTAGE)									
Name:	F2R1		F2R2		F2R3		F2R4		F2R5	
Time (s)	COAL	ASH	COAL	ASH	COAL	ASH	COAL	ASH	COAL	ASH
30	5.19	2.82	2.28	1.13	6.86	3.72	8.89	5.80	8.97	5.79
60	18.77	10.83	6.52	3.17	24.54	14.44	27.72	20.81	28.23	21.12
120	39.84	24.92	11.70	5.71	43.93	26.65	49.70	38.59	50.89	39.75
180	47.92	30.58	14.60	7.20	48.15	29.29	54.92	43.00	56.29	44.31
360	53.37	34.40	18.27	9.20	51.09	31.15	58.58	45.98	60.41	47.71
Product ash %	9.25		7.50		9.65		8.97		9.14	
LAC Tails rec	46.63		81.73		48.91		41.42		39.59	
conc ash %	4.46		3.55		4.42		5.6		5.67	
MIBC ul/l	12.00		8.00		11.00		15.00		18.00	

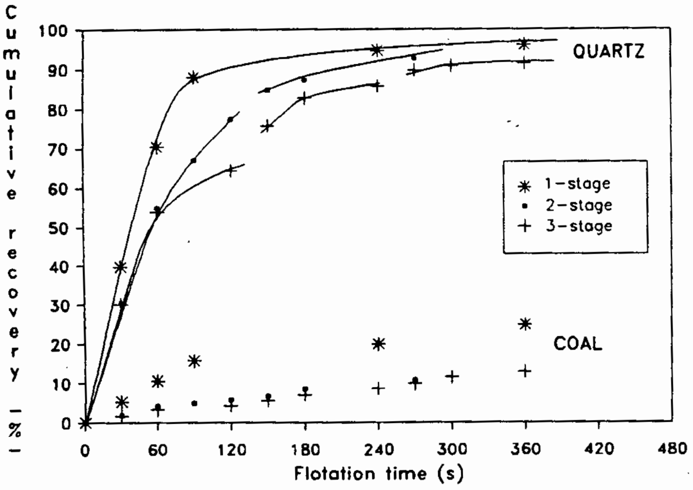
c) Entrainment study: variation of feed quartz content

Run no.	Quartz percent	PROD coal Recovery	PROD as (%)	Conc ash %	CONC coal mass (g)	HTAB Conc
RQ150_01	50	70.94	9.8	79.2	41.81	584
RQ120_01	40	77.71	10.18	76.55	38.92	584
RQ90_01	30	87.04	11.29	77.57	26.55	584
RQ50_01	16.7	93.18	11.6	72.52	16.36	584
2C4	0	95.21	6.74	6.74	7.18	726

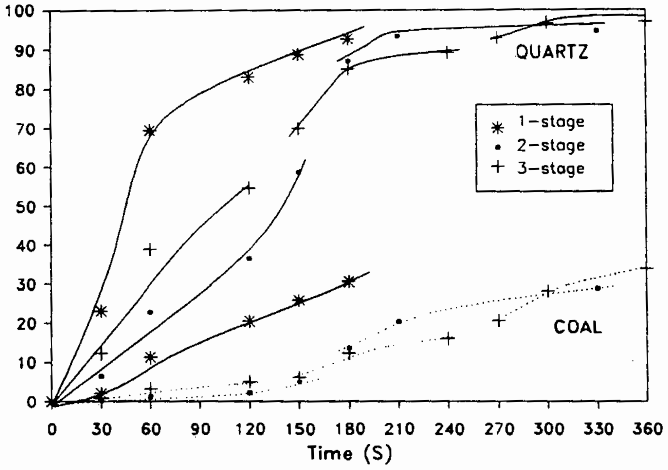
d) Staged addition of reagent

Description	DTAB	HTAB	HPYC
1-STAGE:			
Run id	SA1–3	SA2–3	SA3–3
Flotation time	240 s	360 s	360 s
Ash recovery	94.68%	91.3%	92.74%
Coal recovery	80.16%	79.77%	69.44%
Product ash %	12.0%	16.3%	15.88%
reagent conc. (g/t)	365	550	745
2-STAGE:			
Run id	SA1–1	SA2–4	SA3–1
Flotation time	270 s	180 s	210 s
Ash recovery	92.70%	92.87%	93.30%
Coal recovery	89.32%	89.38%	79.85%
Product ash %	14.22%	14.2%	14.23%
Flotation time	—	max (240s)	360 s (max)
Ash recovery	—	94.7	94.63%
Coal recovery	—	87.70	71.18%
Product ash %	—	12.6	13.55%
reagent conc. (g/t)	264 + 53.3	408+148	538 + 203
3-STAGE:			
Run id	SA1–2	SA2–2	SA3–2
Flotation time	360 s	270 s	270 s
Ash recovery	91.54%	95.75%	92.90%
Coal recovery	87.10%	88.15%	79.49%
Product ash	15.3%	11.75%	15.72%
Flotation time	—	360 s (max)	360 s (max)
Ash recovery	—	98.05%	97.02%
Coal recovery	—	83.94%	66.2%
Product ash %	—	9.45%	11.7%
reagent conc. (g/t)	237+61.7+32	401+96+56	535+72+37

Staged Addition: DTAB



Staged addition: HPYC



e) Kaolin/coal mixtures: Reverse flotation

	CUMULATIVE RECOVERIES (PERCENTAGE)							
Name:	RK30-3		RK50-3		RK70-3		RK90-3	
Time (s)	**LAC**	**KAOLIN**	**LAC**	**KAOLIN**	**LAC**	**KAOLIN**	**LAC**	**KAOLIN**
60	0.36	3.25	0.29	2.80	0.40	2.25	0.42	2.12
180	1.07	9.43	0.84	8.27	1.16	6.69	1.22	6.31
240	1.65	15.50	1.29	13.14	1.74	10.54	1.82	9.85
360	2.55	25.36	2.03	21.78	2.67	17.15	2.78	16.25
420	3.17	32.29	2.53	27.5	3.22	21.4	3.42	20.73
480	3.67	37.91	2.89	32.08	3.66	24.97	3.92	24.31
600	4.54	47.24	3.5	39.71	4.51	31.76	4.8	30.79
720	5.5	56.89	4.26	49.31	5.56	40.3	5.9	39.18
Product ash %	11.29		15.29		21.5		26.81	
LAC Tails rec	94.5		95.74		94.44		94.1	
conca ash %	58.51		73.92		72.63		76.83	
KAOLIN %	10		17		23.3		30	
HTAB g/t	833+300+133		1067+350+150		1333+333+166		1667+400+300	

e) Kaolin/coal mixtures: Flotation with MIBC only

	CUMULATIVE RECOVERIES (PERCENTAGE)							
Name:	KF1		KF2		KF3		KF4	
Time (s)	LAC	KAOLIN	LAC	KAOLIN	LAC	KAOLIN	LAC	KAOLIN
60	20.12	0.89	20.33	1.11	27.47	2.08	27.32	2.28
180	48.54	0.95	48.55	1.11	56.71	2.54	55.13	2.90
300	54.75	1.13	54.72	1.20	60.98	2.61	59.72	2.99
480	59.03	1.26	59.62	1.25	65.02	2.72	63.36	3.05
720	62.56	1.37	63.72	1.34	68.44	2.80	67.24	3.16
Product ash %	26.68		38.29		50.86		58	
LAC Tails rec	37.11		36.15		31.42		32.62	
conc ash %	4.14		4.62		5.4		6.11	
KAOLIN %	10		17		25		30	
MIBC ul/l	18		18		18		18	

f) Use of dextrin: Effect of prior conditioning

Run no.	Remark	Quartz percent	Tails coal Recovery	Tails ash (%)	Concs ash %	Conc Quartz Recovery	Dextrin conc	HTAB Conc
bqdex_01	SA	16.7	87.15	11.62	60.2	87.34	800	400+250
bqdex_02	PC	16.7	94.12	11.57	77.45	88.94	800	400+250
bqdex_03	ND	16.7	84.2	12.13	56.3	85.85	-	400+250
bqdex_06	PC	33	76.4	9.93	84.23	98.81	800	430+220
bqdex_05	SA	33	76.8	11.42	71.85	96.41	800	430+220
bqdex_04	ND	33	47.05	12.54	54.02	96.64	-	430+220
bqdex_08	SA	50	70.57	11.3	79.96	98.63	800	450+216
bqdex_09	PC	50	70.84	9.73	80.23	99.6	800	450+216
bqdex_07	ND	50	45.23	12.27	67.23	98.54	-	450+216
dex25_01	SA	25	87.83	10.59	68.63	93.79	800	560+173
dex25_04	PC	25	90.17	10.05	78.63	97.4	800	560+173
dex25_09	ND	25	81.78	10.73	66.63	94.72	-	560+173

PC = Prior conditioning

SA = simultaneous conditioning

ND = no dextrin

g) Use of dextrin: optimum concentration

Optimum concentration: Dextrin

25% quartz

Run no.	Tails coal Recovery	Tails ash (%)	Concs ash %	Conc Quartz Recovery	Dextrin conc	HTAB Conc
dex25_02	85.42	11	70.9	93.28	200	560+173
dex25_03	89.93	10.3	78.23	88.94	400	560+173
dex25_04	90.17	10.05	78.63	97.4	800	560+173
dex25_05	92.38	10.8	81.95	93.37	1200	560+173
dex25_06	89.5	10.1	78.1	95.93	2400	560+173
dex25_09	70.57	11.3	66.63	98.63	0	560+173

g) Use of dextrin: optimum concentration (continued)

EFFECT OF DEXTRIN: 50% COAL/QUARTZ FEED

	CUMULATIVE RECOVERIES (PERCENTAGE)													
Name:	DQ50_01		DQ50_02		DQ50_03		DQ50_04		DQ50_05		DQ50_06		DQ50_07	
Time (s)	**LAC**	**Quartz**	**LAC**	**Quartz**	**LAC**	**Quartz**	**LAC**	**Quartz**	**LAC**	**Quartz**	**LAC**	**Quartz**	**LAC**	**Quartz**
30	0.88	24.25	1.24	39.32	1.14	52.15	1.39	57.68	0.98	56.87	0.76	50.48	1.83	62.83
60	1.55	39.09	1.66	49.5	1.57	66.2	1.93	74.45	1.32	70.09	1.04	64.85	3.08	79.13
120	2.1	48	2.06	56.71	1.98	74.24	2.45	81.69	1.68	76.54	1.35	72.61	4.41	85.38
150	12.19	89.45	6.91	84.88	5.49	88.28	5.07	90.82	4.72	89.8	4.3	89.13	6.72	91.48
180	20.81	93.49	10.78	90.53	8.68	91.75	7.74	93.07	7.57	92.31	7.1	92.32	9.27	93.31
240	25.48	94.89	13.65	92.57	11.39	93.72	10.58	94.46	10.23	93.94	9.57	94	11.97	94.34
360	26.4	95.67	14.48	93.51	12.37	94.73	12.3	95.48	11.32	94.99	10.61	94.99	14.38	95.26
Product ash %	10.19		11.56		10.35		9.5		9.78		9.86		9.85	
LAC Tails rec	73.6		85.52		87.63		87.7		88.68		89.39		85.62	
conc ash %	80.54		87.74		89.73		89.76		90.13		90.91		88.21	
DEXTRIN g/t	0		200		400		1200		800		600		2400	
HTAB g/t	431 + 154		431 + 154		431 + 154		431 + 154		431 + 154		431 + 154		431 + 154	

h) R.o.m. coals

REVERSE FLOTATION - ROM COALS ("GG" = Grootegeluk, "RIET" = Rietspruit)

	CUMULATIVE RECOVERIES (PERCENTAGE)																							
Name:	GG1		GG2		GG3		RIET01		RIET02		RIET03		RIET04		RIETAS1		RIETAS2		RIETAS3		RIETAS4		RIETAS5	
Time (s)	Coal	Ash	Coal	Ash	Coal	Ash	Coal	Ash	Coal	Ash	Coal	Ash	Coal	Ash	Coal	Ash	Coal	Ash	Coal	Ash	Coal	Ash	Coal	Ash
30	0.18	0.42	0.27	0.63	0.47	1.26	0.50	1.07	0.34	0.66	0.57	0.97	0.43	0.69	0.92	2.69	1.81	5.41	1.13	3.24	0.72	1.89	1.99	4.95
60	0.38	0.68	0.58	1.34	0.89	2.44	0.96	2.02	0.74	1.43	1.21	2.00	0.86	1.41	1.86	5.22	3.81	9.89	2.27	6.22	1.52	3.90	3.81	9.10
90	0.77	1.78	1.18	2.70	1.63	4.51	1.38	2.91			2.38	3.86	1.78	2.80										
120									1.44	2.79	3.54	6.04	2.47	4.12	3.24	8.59	5.56	14.34	3.68	9.59	2.65	6.56	8.17	14.06
150	1.08	2.81	1.70	4.12	4.44	9.69	2.20	4.76	2.31	5.12			3.25	5.53	4.89	12.36	13.85	26.25	10.94	20.74	8.20	18.60	14.86	26.36
180	1.40	3.51	2.31	5.83	8.81	14.90	3.49	7.67	3.22	7.41	4.79	8.31	4.78	8.19	14.07	27.15	22.20	36.07	18.20	30.31	13.83	25.59	23.73	37.02
240	1.98	5.09	3.42	8.86	10.12	21.41			3.99	9.25			7.23	12.48	23.48	38.59	26.36	42.88	24.60	38.77	18.37	33.13	28.75	42.72
300							4.81	11.87	6.25	11.54	7.03	12.28											32.78	47.38
360	2.98	7.78	5.17	13.55	12.69	27.81									30.88	46.81	29.91	44.80	26.47	40.95	20.39	38.31		
420							5.78	13.98			10.56	18.42												
540							7.14	16.99																
Product ash	39.80		38.80		38.30		22.88		23.95		26.30		26.90		21.27		22.06		22.50		22.15		21.87	
Coal Tails re	97.01		94.83		87.31		92.88		93.75		89.44		92.77		69.34		70.09		73.53		79.61		67.22	
conc ash %	64.44		64.56		60.17		44.13		38.14		40.55		40.26		34.78		34.81		35.87		88.78		34.06	
AS CONC															0.0678		0.1417		0.2092		0.2822		0.0000	
AMINE TYP	HTAB		HTAB		HTAB		HTAB		HTAB		HTAB		HTAB		HTAB		HTAB		HTAB		HTAB		HTAB	
Conc (g/t)	1090+670		1628+670		1900+671		1536		1380		2160		2160		1907+788		2037+597		2006+598		2070+589		2063+614	

i) Comparison: Reverse flotation vs Forward flotation

Comparison between Forward and Reverse flotation

Run no.	Quartz percent	PROD coal Recovery	PROD ash (%)	DISC Quartz Recovery	DISC ash %	Shellsol conc g/t	HTAB Conc
Forward flotation							
BNQ0_01	0	83.04	8.2	0		1000	
BNQ17_01	16.7	89.68	8.95	96.36	69.63	1000	
BNQ25_01	25	88.96	9.4	96.61	78.21	1000	
BNQ33_01	33	88.88	9.7	96.99	83.31	1000	
BNQ50_01	50	88.39	10.3	98.01	91.00	1000	
Reverse flotation							
BQ17_01	16.7	94.12	11.57	88.94	77.45		400+250
BQ25_01	25	90.94	10.76	95.46	80.63		560+173
BQ33_01	33	88.59	9.48	98.49	83.12		450+216
BQ50_01	50	84.96	11.15	97.4	86.72		450+216

Basis: 100 g feed	REVERSE FLOTATION				
quartz in fd (g)	Yield (g) concs	% ash concs	% quartz recov.	% Coal rec Tails	Coal (g) Conc
16.7	19.67	77.45	87.58	94.12	4.90
25	31.8	80.63	95.46	90.94	6.80
33	40.86	83.12	98.49	88.59	7.64
50	54.23	86.72	97.4	84.96	7.52

Basis: 100 g feed	FORWARD FLOTATION					
quartz in fd (g)	Yield (conc)	% ash concs	% Coal rec concs	% quartz rec Tails	total ash conc (g)	Quartz (g) concentrate
16.7	75.15	8.95	89.68	96.36	6.73	0.61
25	66.91	9.4	88.96	96.61	6.29	0.85
33	60.64	9.7	88.8	96.69	5.88	1.09
50	44.35	10.3	88.39	98.01	4.57	0.99

APPENDIX VIII

COLUMN FLOTATION SPREADSHEET CALCULATIONS

SPREADSHEET CALCULATIONS: COLUMN FLOTATION EXPERIMENTS

Two sample spreadsheets are presented below on pages 3 and 4. The first is for an artificial coal/quartz feed using a filter cloth sparger; the second is for the artificial coal/quartz feeds using a USBM type sparger.

1. Column variables

The following nomenclature is used:

F = Feed slurry rate (kg/h)
T = Tails slurry rate
T_s = Tails solid rate
T_w = Tails water rate
R_w = rinse water
F_w = feed water rate
C = Concentrates slurry rate
F_s = Feed solids rate
C_s = concentrates solid rate
C_w = concentrates water rate
J_w = sprinkler water
S_w = Sparger water rate
C_T = Total concentrates mass (including rinse water)
t = sampling time (minutes)

Inputs to the spreadsheet are C_T, T, F, J_w, S_w and R_w.

C_T, T, F represent the masses, in grams, for the sampling time, t, and have to be converted to the required kg/h by multiplying by the ratio 60/(t*1000). Similarly S_w, J_w and R_w are input in the units l/min and are multiplied by 60 (assuming the density of water is 1kg/litre).

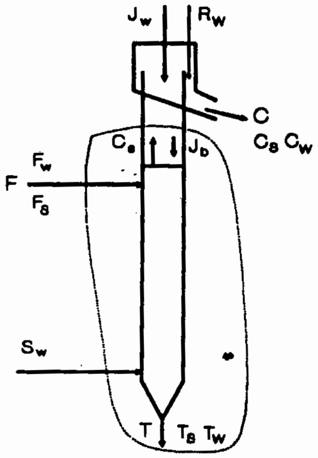
now:

$$C = C_T - R_w$$

$$C_w = C - C_s$$

$$T_w = T - T_s$$

Refer to the diagram below



The feed rate, F, is calculated by simple mass balance:

$$F = C + T - S_w - J_w$$

and

$$F_w = F - F_c$$

The bias rate, J_b is calculated by a water balance over the collection zone of the column:

$$T_w = J_b + F_w + S_w, \text{ hence}$$

$$J_b = T_w - (F_w + S_w) \dots\dots\dots\dots 1$$

For the Filter cloth sparger, the equation is simply

$$J_b = T_w - F_w \dots\dots\dots\dots 2$$

The value of J_b has the units Kg/h. To convert to cm/s, equations 1 and 2 are multiplied by the following:

$$1/60 * 1000\text{cm}^3/\text{kg} * 1/22.1\text{cm}^2$$

Where the value 22.1cm^2 is the cross-sectional area of the column.

Similarly, the air rate, J_g, is obtained by multiplying the l/min value by the ratio $1/60 * 1/22.1\text{cm}^2$.

2. Recoveries

a) Artificial mixtures

The following addtional nomenclature is used:

G_T = Mass added gangue in tails
G_c = Mass added gangue in concentrates
a_m = measured ash content (concentrates or tails)
a_L = ash content of LAC
L_c = Mass L.A.C. in concentrates L_T = Mass L.A.C. in tails

Assumption: the LAC is not beneficiated, i.e. LAC recovered in concentrates has the same ash content as the LAC in the tails which is the same as the feed

For a concentrate mass, C_s, the calculation is:

$$a_m = a_L L_c + G_c \quad \text{(Definition)}$$

$$G_c + L_c = C_s \quad \text{(mass balance)}$$

Combining these equations, we obtain:

$$G_c = \frac{C_s(a_m - a_L)}{(1 - a_L)} \dots\dots\dots\dots (3)$$

and $L_c = C_s - G_c$

Similarly, for the tails

$$G_T = \frac{T_s(a_m - a_L)}{(1 - a_L)} \dots\dots\dots\dots (4)$$

```
  COLUMN REVERSE FLOTATION
                Artificial Mixtures

Run number : CQ50-01          Date:      13/7/90
Coal type  :  Landau LAC      Size:      80% -75 um

COLUMN SET-UP

Collection zone ht :    1.8 m       Bias rate :       0.184  cm/s
Froth zone height  :    1.3 m       drainage          1.150  m

Reagent:     HTAB     addition:  2500 g/t
Depressant:  DEXTRIN  addition:  800 g/t
                                           CONCENTRATES

                                           Slurry mass:      5.11  Kg/h
                                           Solids mass:     0.283  Kg/h
                                 U U ====> Water recv :      4.83  Kg/h     QUARTZ    LAC      ASH     org. COAL
RECONSTITUTED                    I I       Ash %     :      94.94
FEED                             I I       Gangue rec :    56.18%           22.275             22.40
                                 I I       LAC recovry:     1.75%                     1.315              1.19
slurry rate:       14.87         I I
Solids rate:       1.380   ===>  I I
Reconst ash:      40.51%         I I
Meas.  Ash :      54.50%         I I
Plp density:       9.28%         I I
                                 I I       TAILINGS (PRODUCT)
                                 I I
                                 I I       Slurry mass:     26.56  Kg/h
Air (l/min):        2.58   ---->  I I      Solids mass:     1.097  Kg/h
Jg (cm/s)  :        2.38         I I       Water recv :     25.46  Kg/h
                                 U  ====>  Ash %     :      26.46
                                           Gangue rec :    43.82%           17.372             24.18
                                           LAC  recov :    98.25%                     74.018             67.21

INPUTS:
                                           FEED TEST
Conc mass:        23.6  Tail mass:    91.4 Feed mass  :       0.0        ______________________________________
Conc ash :       94.94  Tail ash :   26.46 Feed ash   :     54.50          39.65    75.33    46.58     68.40
slurry   :      4426.0  slurry   :  2213.0 Slurry mass:       0.0
Rinse rte:       0.800  l/min              Sample time:       0.0

Smpl time:         5.0  W Water l/m:  0.280 Air rte l/m:     2.580
```

```
   COLUMN REVERSE FLOTATION
                     USBM Sparger

Run number : usbm08                  Date:        07/01/1991
Coal type  :  Durnalcol t/u          Size:        -200 um

COLUMN SET-UP

Collection zone ht :      1.0 m      Bias rate :        0.187  cm/s
Froth zone height  :      0.5 m      drainage           0.480  m

Reagent:       HTAB     addition:   2750 g/t
Depressant:    DEXTRIN  addition:   800 g/t
Frother:       HTAB                 200 g/t     CONCENTRATES

                                                Slurry mass:     22.03  Kg/h
                                                Solids mass:     0.610  Kg/h
                                    U U ====> Water recv :      21.42  Kg/h          ASH     org. COAL
RECONSTITUTED                       I I         Ash %     :      57.38
FEED                                I I         Ash recovry:     40.77%               17.50
                                    I I         Coal recov :     11.92%                           13.00
slurry rate:        16.16           I I
Solids rate:        3.040  kg/h ===> I I
Reconst ash:       28.24%           I I
Meas.  Ash :       29.89%           I I
Plp density:       18.82%           I I
                                    I I         TAILINGS (PRODUCT)
                                    I I
                                    I I         Slurry mass:     89.52  Kg/h
Air (l/min):         2.05    ---->  I I         Solids mass:     2.430  Kg/h
Jg (cm/s)  :         1.89           I I         Water recv :     87.09  Kg/h
                                    U   ====> Ash %     :      20.93
                                                Ash recovry:     59.23%               25.43
Sparg. rate:         1.03  l/min                Coal recov :     88.08%                           96.07
                                                Yield (%)  :     79.93
                                                Product rt :     1.100  t/h/sqm
INPUTS:
                                                                                    __________________
Conc mass:          30.5  Tail mass:       121.5                                   42.931    109.069
Conc ash :         57.38  Tail ash :       20.93
slurry  :         4071.7  slurry   :      4476.1
Rinse rte:          0.99  l/min

Smpl time:           3.0  W Water l/m:     0.560  Air rte l/m:      2.050
(min)


Bias rate:         0.203  l/min
```

and $L_T = T_s - G_T$ (5)

Feed mass: $F = T_s + C_s$ or

$F = L_T + G_T + L_c + G_c$

Then

Feed ash content: (reconstituted) $$\frac{G_T + G_c + (L_c + L_T)a_L}{F_s} \qquad (6)$$

Tails Gangue recovery (%) = $G_T/(G_T + G_c)*100$ (7)

Tails LAC recovery = $L_T/(L_c + L_T)*100$ (8)

Concs Gangue recovery = $G_c/(G_T + G_c)*100$ (9)

Concs LAC recovery = $L_c/(L_c + L_T)*100$ (10)

Case where LAC is beneficiated.

In some cases in forward flotation, it has been found that the ash content of the concentrates is less than the LAC ash content. This means that the LAC is actually beneficiated. To calculate recoveries, it is assumed that no gangue is recovered in the concentrate sample which has the lowest ash content and this ash content is taken as the LAC ash content of the concentrates.

Specify a_{Lc} = LAC ash fraction of concentrates
a_{LT} = LAC ash fraction of tailings
q = mass fraction added gangue of original feed
G = Total gangue mass in original feed
L = Total LAC. mass in original feed
A = Total ash in LAC. in original feed
A_T = mass LAC ash in tails

Then $F_s = C_s + T_s$ and $G = qF_s$ so $L = F_s - G$

now $A = La_L$

assume that a_{Lc} = ash of sample with lowest ash content, For the tails sample: $A_T = A - a_{Lc}L_c$ and

$$L_c = C_s - \frac{C_s(a_m - a_{Lc})}{(1 - a_{Lc})} \quad \text{(from equations 3 \& 4)}$$

The LAC ash content of the tails is calculated as follows

$$a_{LT} = \frac{A_T}{L - L_c} \qquad (11)$$

Then the value a_{LT} is used in equation 4 instead of a_L

b) r.o.m coals

The following additional nomenclature is used:

A_C = Ash mass in concentrate
a_c = ash fraction in concentrate
A_T = Ash mass in tails

C_C = combustible coal in conc
a_T = ash fraction in tails
C_T = combustible coal in tails

Then $$A_C = a_c C_s,\ C_c = C_s - A_c$$

$$A_T = a_T T_s,\ C_T = T_s - A_T$$

and:

Tails coal recovery (%): $C_T/(C_c + C_T)*100$ (12)

Tails ash recovery: $A_T/(A_c + A_T)*100$ (13)

Concs coal recovery: $C_c/(C_c + C_T)*100$ (14)

Concs ash recovery: $A_c/(A_c + A_T)*100$ (15)

APPENDIX IX

COLUMN CELL CALCULATIONS

Calculation of cross sectional area:

Diameter = 5.3 cm

XS area $= \pi d^2/4 = 22.062\ \text{cm}^2$

Conversion factors:

to convert cm/s to l/min, multiply by

$$1\,\text{cm/s} \times 60\ \text{s/min} \times 22.06\ \text{cm}^2 \times 1\ \text{dm}^3/1000\ \text{cm}^3 = 1.324$$

to convert l/min to cm/s, divide by 1.324

Estimate wash water requirement:

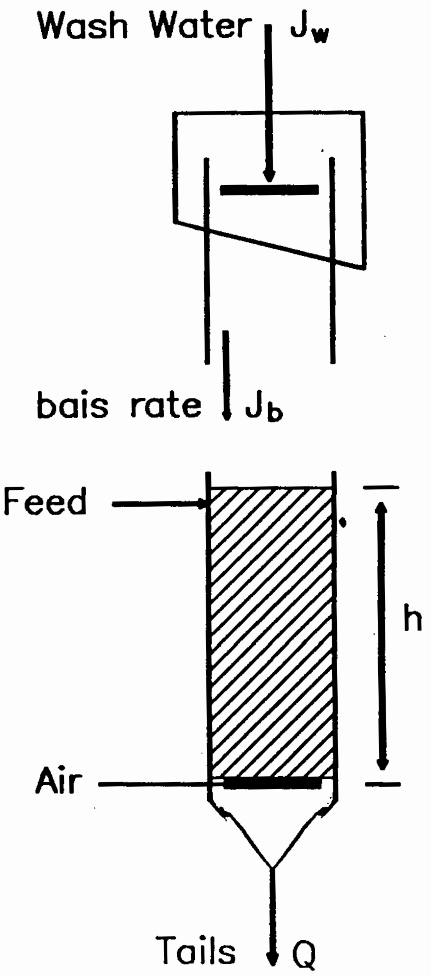
(for column set-up runs)

Assume holdup, $\varepsilon_{go} = 0.8$, take $J_b = 0.2$ cm/s, $J_g{}^* = 2.0$ cm/s

then, using equations 4.6 and 4.7 (Chapter 4)

$$J_w = \frac{2.0 \times (1 - 0.8)}{0.8} - 0.2 = 0.300\ \text{cm/s}$$

Calculation of residence time:



Residence time $= \tau = V / Q$

need to correct V for holdup:

$V_{actual} = V \times \varepsilon_{go} = \pi\, d^2/4 \times h \times 0.8$

e.g. for a 1 m collection zone height (see diagram)

$V = \pi \times (0.53\ \text{dm})^2 /4 \times 10\ \text{dm} \times 0.8 = 1.765\ \text{dm}^3$

APPENDIX X

COLUMN FLOTATION DATA: ARTIFICIAL MIXTURES

a) Column setup runs

COLUMN SET UP RUNS - REVERSE FLOTATION
Quartz/coal mixtures

No.	TAILS Coal rec.	CONCS Ash rec.	Product Ash %	Froth height m	Col zon height	Bias rate cm/s	Gas rat cm/s	Residence Time (min)	HTAB conc g/
C3Q50_1	98.25	56.18	26.46	1.30	1.80	0.150	1.95	7.50	2500
C3Q50_2	96.37	62.39	29.86	0.80	1.60	0.157	1.95	6.45	2260
C3Q50_3	95.95	77.13	21.50	0.80	1.60	0.112	2.32	7.36	1820
C3Q50_4	92.10	98.40	11.08	0.40	1.00	0.069	2.32	5.44	1820
C3Q50_5	90.08	97.78	11.63	0.40	1.00	0.118	2.32	4.40	1200
C3Q50_6	91.40	99.40	10.01	0.40	1.00	0.087	2.26	4.79	1200
C3Q50_7	92.87	97.46	11.12	0.40	1.00	0.147	2.26	3.98	1636
C3Q50_8	93.39	98.62	10.40	0.40	1.00	0.165	2.04	3.65	1636

b) Entrainment study

COAL/QUARTZ MIXTURES: FORWARD FLOTATION

RUN ID	Coal rec (%)	Ash rec (%)	Prod ash (%)	Bais Rt cm/s	Air RT cm/s	OVFLW t/h/sqm	water rec kg/h	SSol A g/t
NC1610_1	98.89	100.00	8.70	0.307	2.81	574.00	0.87	1000
NC2510_1	96.42	100.00	8.70	0.300	2.81	456.00	1.37	1000
NC3310_1	96.78	100.00	8.70	0.309	2.81	412.00	0.68	1000
NC5010_1	96.42	100.00	8.72	0.317	2.81	313.00	0.13	1000

COAL/QUARTZ MIXTURES: REVERSE FLOTATION

RUN ID	Coal rec (%)	Ash rec (%)	Prod ash (%)	BIAS RT cm/s	Air RT cm/s	OVFLW t/h/sqm	Water rec kg/h	PROD RT t/h/sqm	HTAB g/t	Mass LAC in conce t/h/sqm	Basis: 100 g feed
CQ317_01	93.34	98.08	9.58	0.227	2.81	158	6.71	557	1500	39.54	5.53
CQ325_01	92.77	94.90	10.82	0.205	2.81	206	8.33	506	1500	40.01	5.62
C3Q33_01	95.17	96.02	10.95	0.185	2.81	242	9.8	472	1500	23.56	3.30
C3Q50_01	93.74	98.33	11.02	0.161	2.81	358	11.58	340	1500	22.34	3.20

c) Increased pulp density and column carrying capacity

COAL/QUARTZ MIXTURES: 20% Pulp Density
Reverse and normal flotation

RUN ID	COAL REC (%)	ASH REC (%)	PROD ASH (%)	OVFLW t/h/sqm	PROD RT t/h/sqm	FEED RT t/h/sqm
NC 25% Q	89.24	100.00	8.32	911	911	1355
RC 25% Q	93.92	95.95	10.43	383	997	1381
NC 17% Q	91.33	100.00	7.82	1021	1021	1338
RC 17% Q	96.87	91.89	11.07	271	1189	1460

Column Carrying capacity

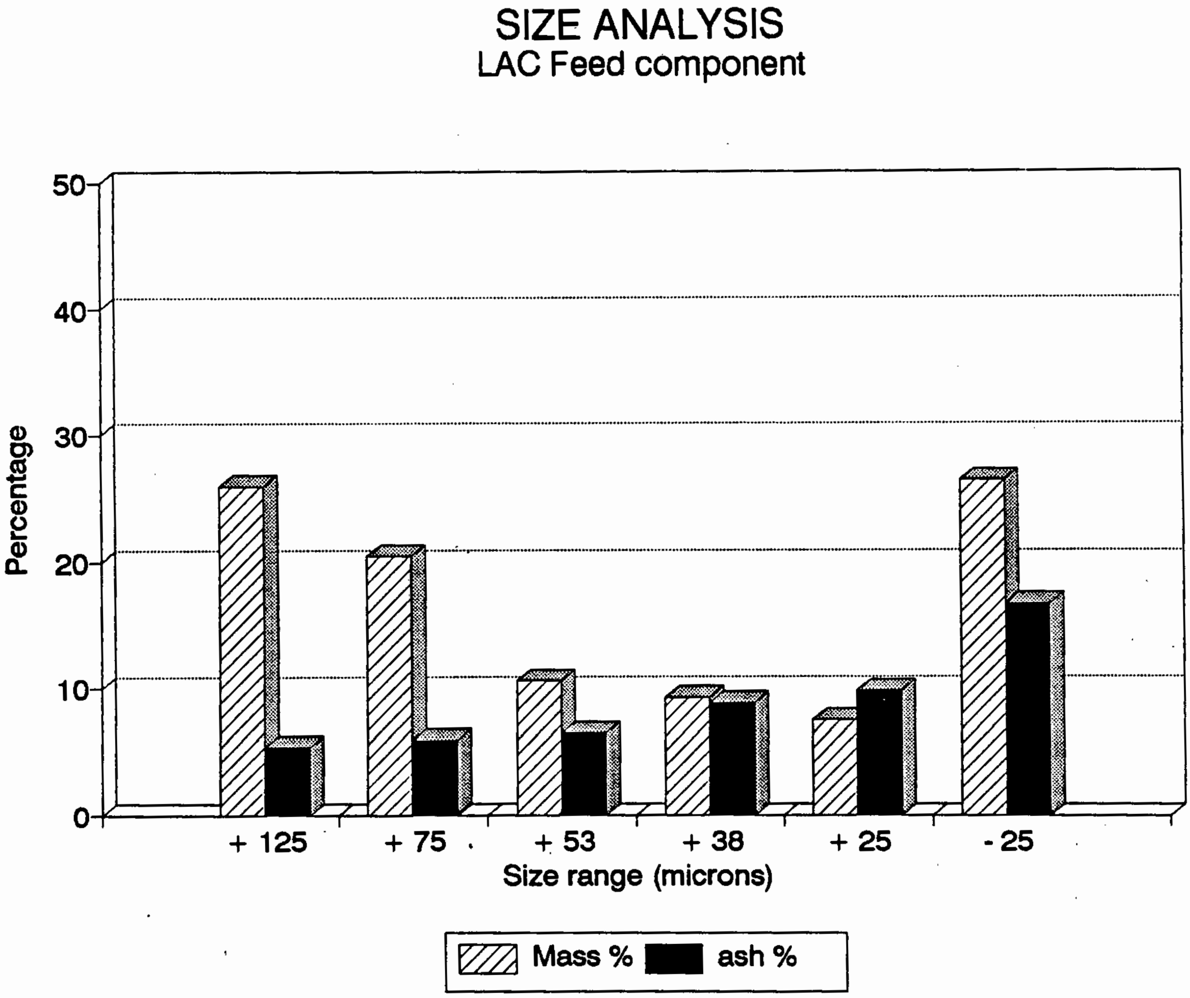
Run ID	Coal rec (%)	Product Ash %	Ash rec (%)	Feed t/h/sqm	Overflow t/h/sqm	Tails Rt t/h/sqm
Reverse						
C3Q25-01	92.77	10.82	94.88	0.707	0.206	0.507
RC2520_2	94.91	11.02	93.99	1.411	0.378	1.033
RC25CP1	91.94	10.1	97.18	1.892	0.567	1.325
RC25CP2	93.87	10.9	94.79	2.796	0.805	1.991
RC25CP3	93.26	11	94.45	3.033	0.879	2.154
Forward						
NC25	95.79	8.7	100	0.63	0.456	0.174
NC2520_01	88.74	8.32	100	1.355	0.911	0.444
NC25CP3	66.49	5.41	100	1.539	0.765	0.774
NC25CP1	49.87	5.15	100	1.762	0.649	1.113
NC25CP4	31.84	4.87	100	2.197	0.528	1.669
NC25CP2	20.42	4.5	100	2.549	0.401	2.148

APPENDIX XI

COLUMN FLOTATION OF ARTIFICIAL MIXTURES SIZE ANALYSES AND EFFICIENCIES

a) size analysis: LAC feed

```
Landau LAC     Date:  3/10/1990
Remark: lac feed component

Size fraction  Mass     Mass %    Ash %  Ash mass
-------------------------------------------------

+ 125          12.56    25.91     5.40      0.68
+ 75            9.87    20.36     5.90      0.58
+ 53            5.13    10.58     6.50      0.33
+ 38            4.47     9.22     8.80      0.39
+ 25            3.64     7.51     9.80      0.36
- 25           12.81    26.42    16.60      2.13
-------------------------------------------------
               48.48      100               4.47

Ash content     9.22 %
```

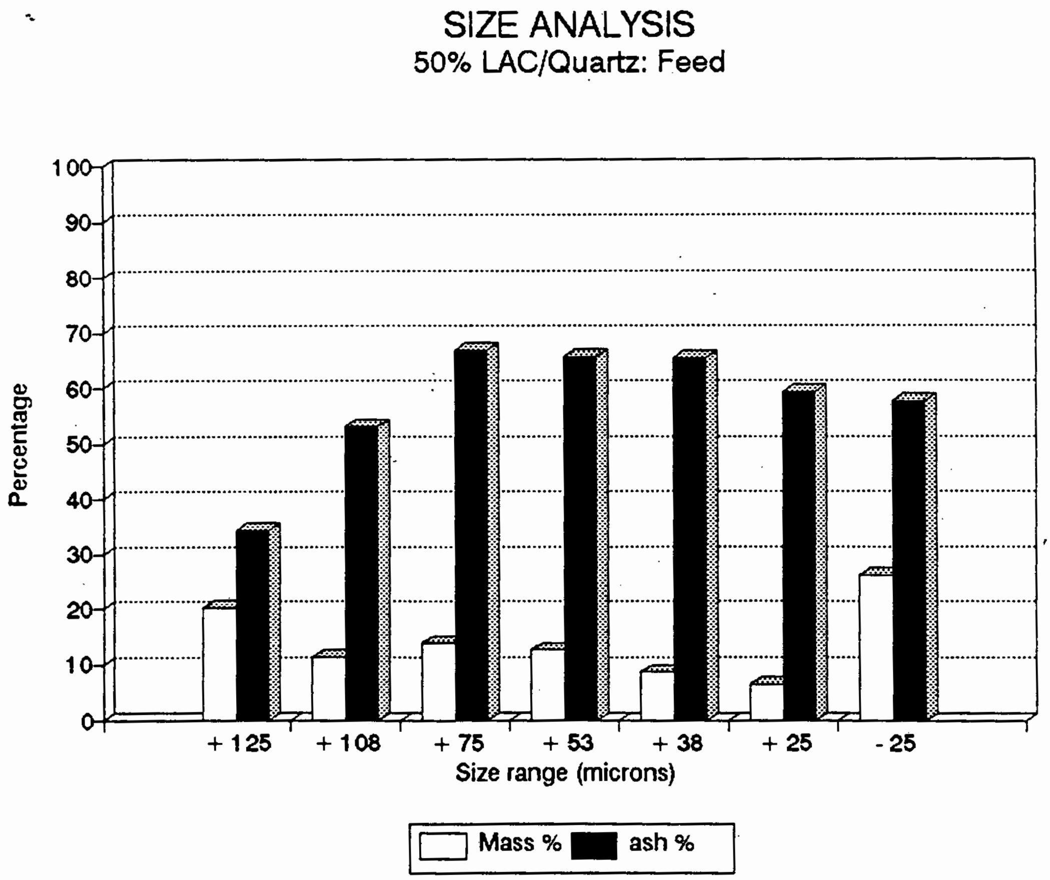


b) size analysis: 50/50% Feed mixture

Coal:50% MIX Date: 3/10/1990
Remark: Feed sample

Size frac	Mass	Mass %	Ash %	Ash mass (g)	Qtz rec (g)	Ideal rec %	LAC ash % in size rng
+ 125	11.33	20.30	34.46	3.90	3.48	30.72	5.4
+ 75	14.07	25.21	60.55	8.52	8.17	58.08	5.9
+ 53	7.14	12.79	65.8	4.70	4.53	63.42	6.5
+ 38	4.95	8.87	65.5	3.24	3.08	62.17	8.8
+ 25	3.64	6.52	59.21	2.16	1.99	54.78	9.8
- 25	14.69	26.32	57.7	8.48	7.24	49.28	16.6
	55.82	100		31.00			

Ash content 55.53 %

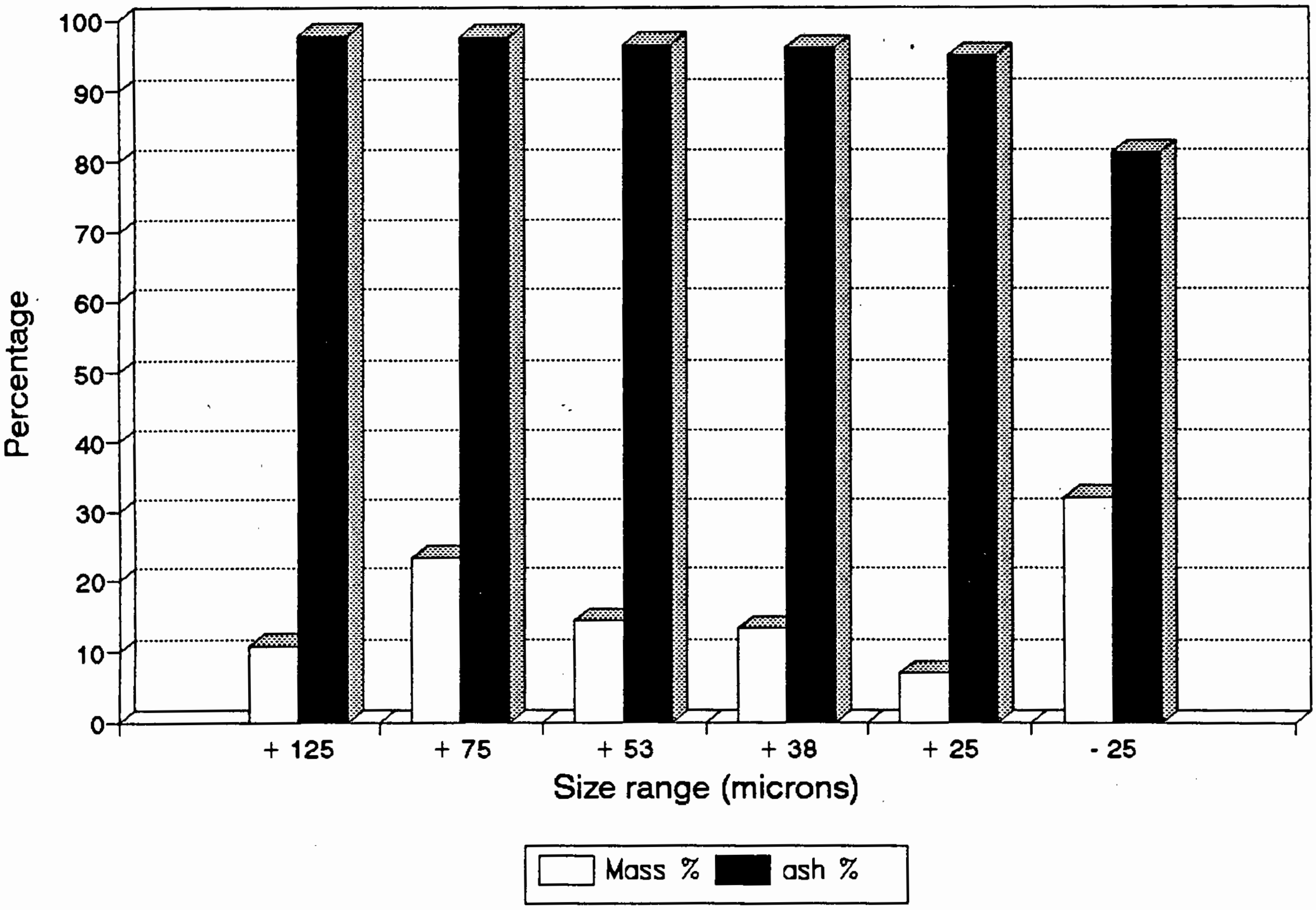


c) size analysis: Concentrates from run C3 Q50_06

```
Coal:50% MIX   Date:  3/10/1990
Concentrates from reverse flotation run

Size fraction  Mass    Mass %    Ash %  Ash mass
------------------------------------------------

+ 125            5.34    10.77    97.64     5.21
+ 75            11.51    23.21    97.52    11.22
+ 53             7.08    14.27    96.33     6.82
+ 38              6.5    13.10    95.96     6.24
+ 25             3.41     6.88    94.83     3.23
- 25            15.76    31.77     81.3    12.81
------------------------------------------------
                 49.6      100             45.54

Ash content     91.82 %
```

SIZE ANALYSIS: REVERSE FLOTATION

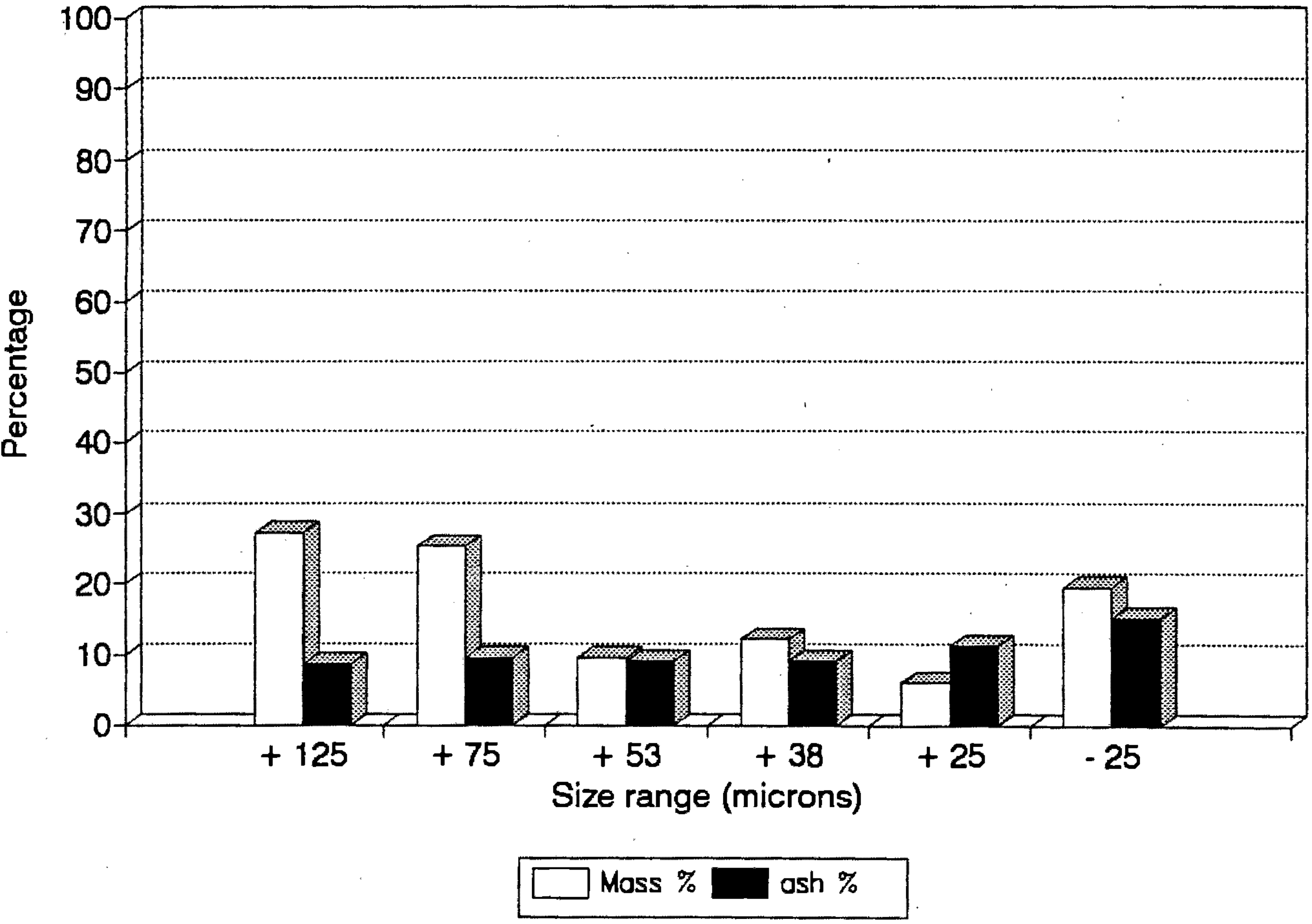
Column concentrates: 50% LAC/Quartz



d) size analysis: Tails from run C3Q50_06

```
Coal:50% MIX   Date:              3/10/1990
Remark: Tailings from 50% quartz column run

Size fraction  Mass   Percent    Ash %
-------------------------------------------------

+ 125               9.47    27.28      8.63      0.82
+ 75                8.76    25.23      9.62      0.84
+ 53                3.32     9.56      9.17      0.30
+ 38                4.23    12.18      9.14      0.39
+ 25                2.12     6.11     11.31      0.24
- 25                6.82    19.64     15.26      1.04
-------------------------------------------------
                   34.72      100                3.63

Ash content:       10.46
```

SIZE ANALYSIS: REVERSE FLOTATION
Column Tailings: 50% LAC/Quartz



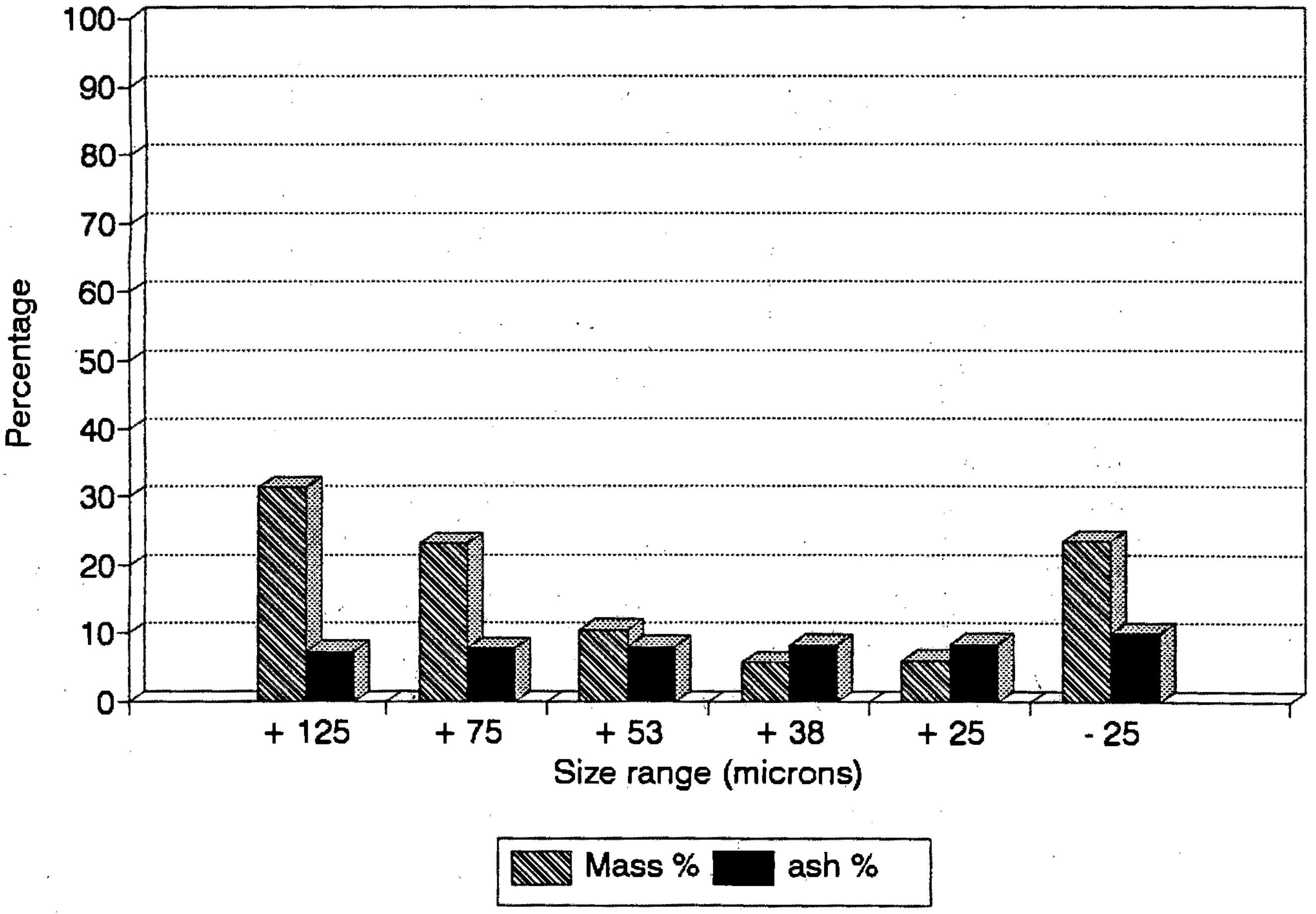
e) size analysis: Concentrates from run NCQ50_01

Coal:50% MIX Date: 3/10/1990
Remark: CONCS from forward column run

Size fraction	Mass	Percent	Ash %	
+ 125	12.37	31.38	7.34	0.91
+ 75	9.07	23.02	7.71	0.70
+ 53	4.09	10.38	8.01	0.33
+ 38	2.27	5.75	8.11	0.18
+ 25	2.33	5.91	8.44	0.20
- 25	9.28	23.55	10.1	0.94
	39.42	100		3.25

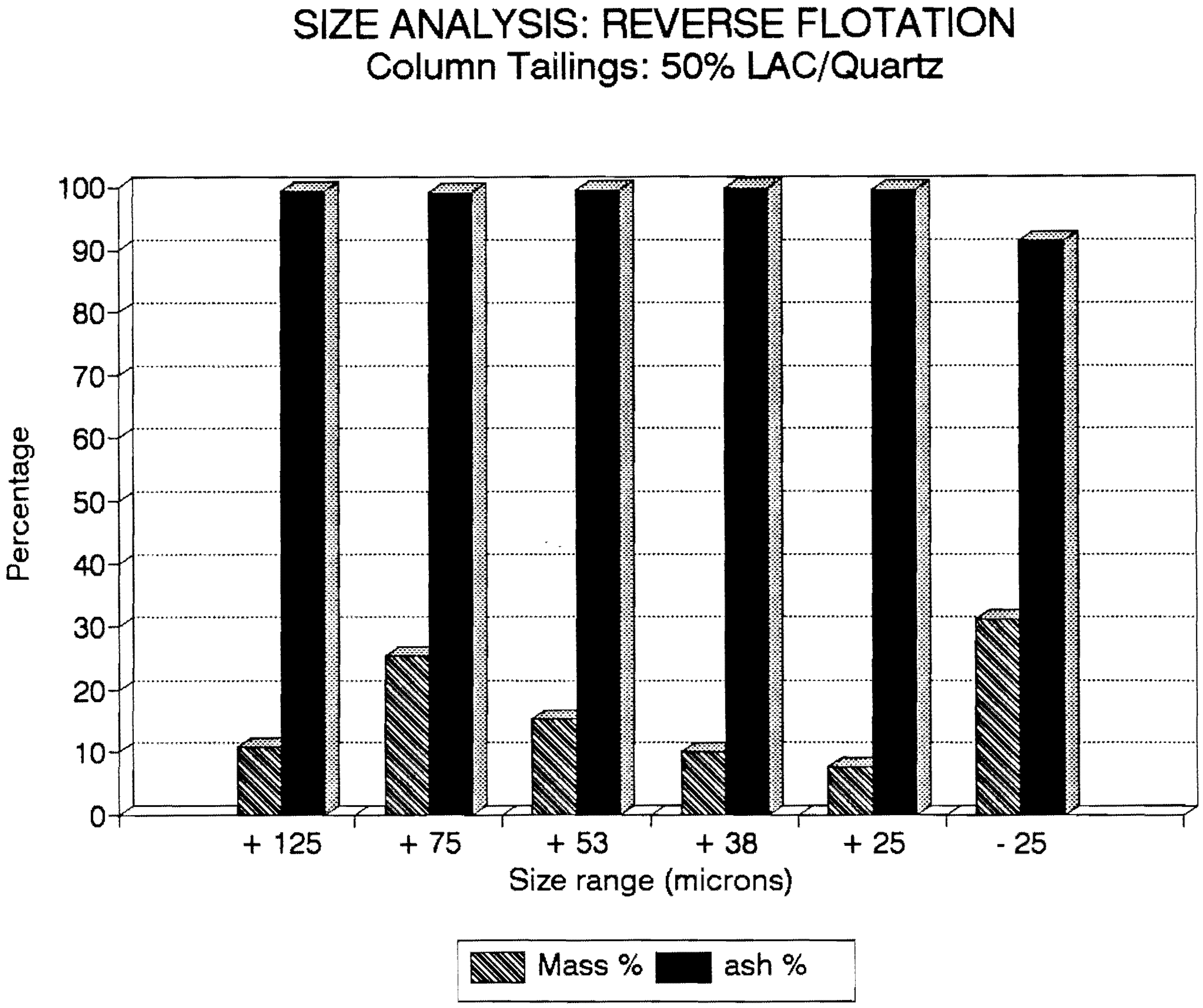
Ash content: 8.25

SIZE ANALYSIS: REVERSE FLOTATION
Column Tailings: 50% LAC/Quartz



f) size analysis: Tailings from run NCQ50_01

```
Coal:50% MIX   Date:          3/10/1990
Remark: tailings from forward column run

Size fraction  Mass   Percent    Ash %  Ash mass
------------------------------------------------

+ 125            6.52   10.76     99.2     6.46
+ 75            15.35   25.35    99.01    15.20
+ 53             9.14   15.09    99.54     9.10
+ 38             6.06   10.01    99.55     6.03
+ 25             4.62    7.63    99.34     4.59
- 25            18.87   31.16    91.36    17.24
------------------------------------------------
                60.56     100             58.62

Ash content:    96.81
```



g) Calculation method: separation efficiencies

For the 50% artificial coal/quartz mixture, a size analysis was performed on the flotation feed sample, samples of concentrates and tailings from a reverse flotation and on the LAC sample itself. These analysis are shown on pages 1 to 6 (this appendix).

In order to calculate the ideal concentrate recovery for reverse flotation, it is assumed that no LAC is recovered in the concentrates and all the quartz in the feed is recovered in the concentrates.

The quartz mass recovered in each size fraction i is calculated by means of a modified version of equation (3) given in Appendix VI:

$$G_{ci} = \frac{S_i(a_i - a_{Li})}{(1 - a_{Li})}$$

Where G_{ci} is the mass of quartz in the concentrates in size fraction i, S_i is the mass recovered in each size fraction i and a_{Li} is the LAC ash fraction in the same size range as determined from the LAC size analysis. The ideal recovery percentage in each size fraction is expressed as

$$R_i = \frac{G_{ci}}{S_i} \times 100$$

so R_i represents the percentage of the feed in the particular size fraction, i, which should report to the concentrates of reverse flotation. For forward flotation, the same calculation is used, except that the equations apply to the tailings. The ideal recoveries are shown in the feed size analysis on page 2.

The actual mass recoveries in each size fraction are determined by multiplying the size distribution mass percentages obtained in the size analysis of the concentrates and tails by the total concentrates or tails mass for the particular column run which the size analysis applies to. The feed is then reconstituted according to size fraction. The concentrate mass recovery in each size fraction is then calculated as a percentage of the reconstituted feed (in each size fraction). The concentrate recoveries are also be compared on the basis of ash content: the ideal recovery implies 100% ash content in each size fraction. This is shown in the tables on page 8. For example, in the reverse flotation +125μm fraction, the actual concentrate recovery is 28.76% at 97.64% ash while the ideal is 30.72% quartz recovery (100% ash), which makes the ideal tails LAC recovery 100-30.72, i.e. 69.28%. The actual gangue collection in this size fraction was thus close to ideal.

h) Separation efficiencies: calculations

ARTIFICIAL MIXTURE: 50% QUARTZ-COAL

Ideal LAC recovery vs actual yield in each size fraction
for column run rcq_06

	TAILS				CONCENTRATES				RECONST FEED		E	Tails ideal rec
Size Fr	Mass%	% of feed	Mass	Ash %	Mass %	% of feed	Mass	Ash%	mass	mass %		
+ 125	27.28	71.24	252.1	8.63	10.77	28.76	101.8	97.64	353.8	18.93	0.7268	69.28
+ 75	25.23	51.52	233.1	9.62	23.21	48.48	219.3	97.52	452.5	24.21	0.8433	41.92
+ 53	9.56	39.58	88.3	9.17	14.27	60.42	134.9	96.33	223.2	11.94	0.892	36.58
+ 38	12.18	47.62	112.5	9.14	13.1	52.38	123.8	95.96	236.3	12.65	1.1844	37.83
+ 25	6.11	46.48	56.5	11.31	6.88	53.52	65.0	94.83	121.5	6.50	0.8815	45.22
- 25	19.64	37.67	181.5	15.26	31.77	62.33	300.2	81.3	481.7	25.77	0.7364	50.72

T. mass : 924.0 C. mass : 945.0 1869 100

ARTIFICIAL MIXTURE: 50% QUARTZ-COAL

Ideal coal recovery vs actual yield in each size fraction
for column run nc50

	TAILS				CONCENTRATES				RECONST FEED			E	Concs ideal rec
Size Fr	Mass%	% of feed	Mass	Ash %	Mass %	% of feed	Mass	Ash%	mass	mass %	Ash %		
+ 125	10.76	26.62	78.9	99.20	31.38	73.38	217.5	7.34	296.3	20.78	31.79	0.8803	69.28
+ 75	25.35	53.81	185.8	99.01	23.02	46.19	159.5	7.71	345.3	24.22	56.83	0.9433	41.92
+ 53	15.09	60.59	110.6	99.54	10.38	39.41	71.9	8.01	182.5	12.80	63.47	1.0167	36.58
+ 38	10.01	64.81	73.4	99.55	5.75	35.19	39.8	9.11	113.2	7.94	67.72	0.8783	37.83
+ 25	7.63	57.73	55.9	99.34	5.91	42.27	41.0	8.44	96.9	6.79	60.91	1.0744	45.22
- 25	31.17	58.33	228.5	91.36	23.55	41.67	163.2	10.1	391.7	27.47	57.50	1.2306	50.72

T. mass : 733.0 C. mass : 693.0 1426 100

i) Float and sinks analysis: 25% quartz/coal feed

25% Q + LAC				SPECIFIC GRAVITY
% FLOATS	% MASS LOSS	% ASH FLOATS	% ASH SINKS	
22.524	0.54	2.6	28.3	1.3
25.168	0.24	2.6	34.3	1.3
38.184	0.09	3.4	56	1.35
38.548	1.63	3.6	45.1	1.35
53.594	1.21	4.3	63.5	1.4
52.893	2.22	4.8	60.3	1.4
63.452	2.06	4.7	76.6	1.45
61.970	-0.18	4.8	75.7	1.45
70.422	2.33	6.5	86.8	1.5
68.709	0.62	6	86.6	1.5
71.376	0.43	6.2	91.9	1.55
72.051	0.59	6.4	91.7	1.55
72.476	1.11	6.5	95.1	1.6
73.400	0.77	6.9	95.1	1.6
74.152	1.13	7.5	97.3	1.7
74.160	1.20	6.9	97.3	1.7

APPENDIX XII

COLUMN FLOTATION OF R.O.M. COALS
FLOTATION RESULTS

a) Grootegeluk

REVERSE FLOTATION: COLUMN CELL
GROOTEGELUK COAL

No.	Yield %	Product grade	Conc Ash (%)	Coal rec (%)	Ash rec (%)	Froth ht (m)	Collec zone HT	HTAB g/t	DEXTRIN g/t	Bias rt cm/s	Air rt cm/s
COLGG1	97.47	40.94	66.05	98.53	4.02	0.5	1.5	2500	0	-	1.88
COLGG2	93.12	39.45	63.72	95.76	10.67	0.5	1.5	4500	0	-	1.88
COLGG03	89.09	41.00	48.9	90.70	13.34	0.5	1.5	3500	0	0.268	1.88
COLGG04	98.14	40.50	58.7	98.70	2.67	0.5	1.5	3500	1000	0.258	1.88
COLGG05	90.55	40.40	56.41	92.91	12.72	0.5	1.5	4000	500	0.265	1.88
COLGG06	83.62	38.43	56.1	87.74	22.24	0.5	1.5	4000	500	0.283	2.34
COLGG07	89.71	40.32	50.23	91.27	12.50	1	1.5	3000	800	0.289	2.30
COLGG08	89.81	40.60	52.14	91.63	12.72	1	1.5	3000	800	0.301	2.11
COLGG09	81.00	34.39	65.44	89.07	30.72	0.5	1	3000	800	0.227	2.30
COLGG10	81.68	34.52	67.58	90.01	30.51	0.5	1	3000	800	0.195	1.92
COLGG11	81.06	34.10	67.25	89.59	31.54	0.5	1	3500	800	0.19	2.30
COLGG12	82.40	34.80	67.23	90.30	29.21	0.5	1	3500	800	0.215	2.30

b) Ermelo

REVERSE FLOTATION: COLUMN CELL
ERMELO COAL

Run I.D.	Product grade	Conc. Ash	Coal rec (%)	Ash rec (%)	Froth HT (m)	Collec zone HT	HTAB g/t	DEXTRI g/t	Bias rate cm/s	Air rate cm/s
COLERM01	20.35	35.1	84.97	27.24	1.2	1.3	4000	0	-	1.90
COLERM02	19.31	43.6	89.80	26.85	1.2	1.3	4000	500	-	1.90
COLERM03	19.60	60.56	95.83	21.57	0.5	1	2500	800	0.250	2.04
COLERM04	19.07	47.95	91.59	26.42	0.5	1	2500	800	0.245	2.30
COLERM05	18.77	53.77	82.86	27.91	0.5	1	2500	800	0.208	2.30
COLERM06	17.60	67.86	95.83	30.08	0.5	1	3000	800	0.222	2.30

c) Durban Navigation (Durnacol): reverse flotation

REVERSE FLOTATION OF DURNACOL: COLUMN CELL

Name	Yield %	Product grade	Coal rec (%)	Ash rec (%)	DISC ASH (%)	HTAB g/t	PROD SULF %	COL. ZONE Ht (m)	Frother g/t	Pulp Density	Bias rt cm/s	Froth Water kg/h	Air rate cm/s
		-- FILTER CLOTH SPARGER --							DIBK				
RCDURN01	94.06	25.30	97.92	15.75	74.89	4000	1.3	1.0	-	10	0.262	2.59	2.11
RCDURN02	71.55	18.50	81.25	53.11	52.71	2000		1.0	15 l/l	10	0.207	7.21	2.11
RCDURN03	89.41	23.40	95.14	25.32	66.96	1500		1.0	8 l/l	10	0.15	3.74	2.11
RCDURN04	70.84	17.90	81.34	55.50	54.23	2500	1.35	1.0	8 l/l	10	0.226	10.22	2.11
RCD20_01	88.68	23.66	94.32	25.66	63.97	2000		1.0	8 ul/l	20	0.245	4.48	1.96
		-- USBM SPARGER --							HTAB				
USBM01	75.81	22.87	83.63	42.37	47.56	2250		1.0	200	20	0.072	20.03	2.11
USBM02	76.90	22.98	83.02	38.34	52.69	1750		1.0	200	20	0.142	22.33	1.89
USBM03	70.75	21.23	79.07	49.12	49.57	2750		1.0	200	20	0.009	25.03	2.04
USBM04	82.55	22.25	90.14	36.22	59.76	2500		1.0	200	20	0.171	15.42	1.83
USBM05	85.86	23.92	93.14	31.75	66.02	2500		1.0	200	20	0.236	15.42	1.43
USBM06	89.71	24.92	95.14	23.45	66.58	2750		1.0	200	20	0.249	6	1.19
USBM07	76.62	20.95	84.88	43.96	53.86	2750		1.0	200	20	0.201	17.62	1.92
USBM08	79.93	20.93	88.08	40.77	57.38	2750		1.0	200	20	0.153	21.42	1.55
USBM09	86.20	22.02	94.30	33.80	70.72	2500		2.25	200	20	0.147	18.32	1.89
USBM10	83.01	20.92	92.53	40.22	68.78	2500	1.35	2.25	200	20	0.072	24.27	2.41
USBM11	80.60	20.99	90.40	42.84	65.23	2750	1.33	2.25	200	20	0.076	23.93	2.41
USBM12	80.00	19.80	90.40	45.50	65.98	3000		2.25	200	20	0.092	25.69	2.41

FEED ASH: 28.9 %

FEED SULPHUR: 1.21%

APPENDIX II

MILLING TIMES AND ARTIFICIAL MIXTURE SIZE ANALYSES

a) Landau LAC milling times

Three samples of Landau Low ash coal were used during the course of the project, containing 7.2%, 4.4% and 9.2% ash respectively. They were obtained as -6mm samples but actually contained approximately 12% +6mm material. Each sample was thus screened at 5.6mm to remove this material.

For the 7.2% and the 4.4% ash sample, a 1-kg capacity, laboratory rod mill was used. A milling test was performed in order to determine the milling time to obtain a sample of 98% -150 µm.: 1 Kg coal was dry milled (20 rods) for the specified amount of time and a 100g sample was sieved (dry) on a sieve shaker for 20 minutes. The +150 and -150 µm masses were recorded and milling was then continued for the next time increment.

Milling time (minutes)	**% passing 150 µm**	
	4.4% Ash	**7.2% Ash**
60	99.8	99.8
40	99.8	99.7
25	99.5	99.4
20	97.92	97.87
18	97.67	97.56
17.5	97.38	97.08
17	95.45	94.89
16.5	93.15	92.37
16	89.42	88.35

Milling speed: 67 Rpm (90% of critical)

As can be seen from the above table, a milling time of 20 minutes is sufficient to obtain a 98% passing 150 µm.

For the 9.2% coal, a 2.5 kg-capacity laboratory mill was used. A milling test was again performed: 2.5 kg coal was milled dry with 20 rods. The results are shown below..

Milling time (minutes)	**% passing 150 µm**
75	98.2
60	97.0
50	96.1
45	95.6
42.5	94.9
40	93.1
37.5	90.5

Milling speed: 60 rpm (90% of critical)

As can be seen, a milling time of 75 minutes is required to obtain a 98% -150 µm sample. It was decided for practical reasons to mill for 45 minutes which gives a sample of 95% -150 µm.

b) Quartz and Kaolin size distributions

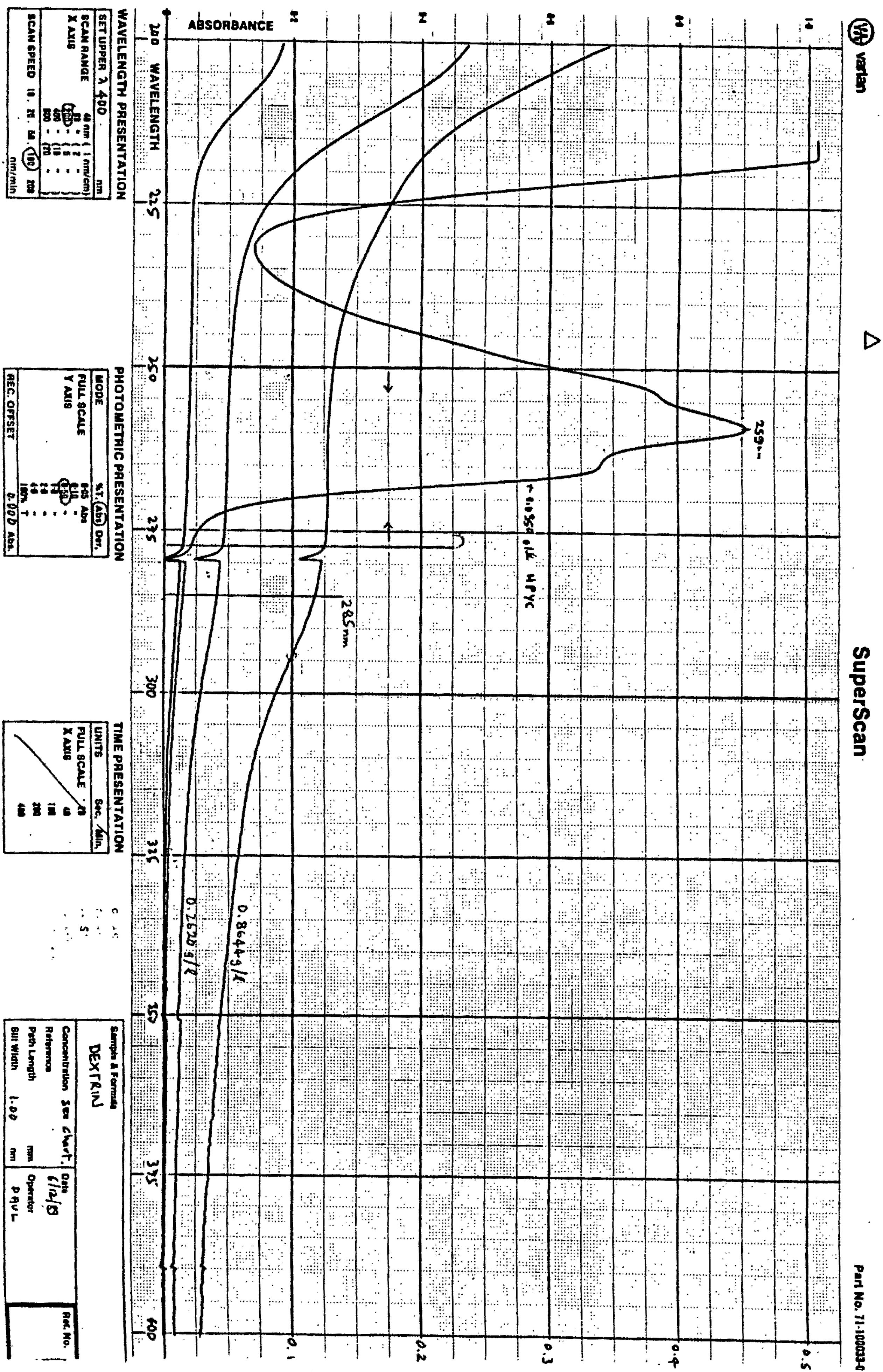
Size analysis: quartz and kaolin

Particle size microns	Percent passing	
	quartz	Kaolin
118.4	100.0	100.0
54.9	62.5	99.9
33.7	50.1	99.8
23.7	40.7	99.7
17.7	33.1	95.0
13.6	26.2	87.3
10.5	20.6	79.2
8.2	15.8	70.0
6.4	11.2	59.4
5	7.4	48.8
3.9	4.8	38.3
3	2.6	27.2
2.4	1.3	18.3
1.9	0.7	12.1
1.5	0.5	6.8
1.2	0.2	3.4
d50 (microns):	23.64	8.46

APPENDIX III

ADSORPTION EXPERIMENTS: UV DATA AND RTD CALCULATIONS

a) UV scan: HPYC amine and dextrin



b) Adsorption vessel step input data and RTD calculations

Adsorption equipment
Step input test

Time (s)	Absorbance 1	2	3	4
0	0	0	0	0
2.5	0.057	0.056	0.057	0.054
7.5	0.198	0.199	0.2	0.197
12.5	0.204	0.205	0.204	0.203
17.5	0.207	0.207	0.208	0.207
22.5	0.209	0.209	0.21	0.209
27.5	0.209	0.209	0.21	0.209

No	Absorb	C (calc)	measured t value	ti	ti^2	t/tbar	F function	J(theta) function
1	0.010	0.00080	7.5	1.311	1.72	0.29		0.0476
2	0.020	0.00161	10	1.748	3.06	0.39		0.0952
3	0.030	0.00241	11.8	2.063	4.25	0.46		0.1429
4	0.040	0.00322	13	2.272	5.16	0.51	0.0284	0.1905
5	0.050	0.00402	13.7	2.395	5.73	0.53	0.1251	0.2381
6	0.060	0.00482	14.4	2.517	6.34	0.56	0.2081	0.2857
7	0.070	0.00563	15.2	2.657	7.06	0.59	0.2893	0.3333
8	0.080	0.00643	16	2.797	7.82	0.62	0.3586	0.3810
9	0.090	0.00723	17	2.972	8.83	0.66	0.4318	0.4286
10	0.100	0.00804	17.9	3.129	9.79	0.70	0.4875	0.4762
11	0.110	0.00884	18.9	3.304	10.91	0.74	0.5403	0.5238
12	0.120	0.00965	19.8	3.461	11.98	0.77	0.5812	0.5714
13	0.130	0.01045	20.9	3.653	13.35	0.82	0.6241	0.6190
14	0.140	0.01125	22.2	3.881	15.06	0.87	0.6668	0.6667
15	0.150	0.01206	24.3	4.248	18.04	0.95	0.7219	0.7143
16	0.160	0.01286	26.5	4.632	21.46	1.03	0.7662	0.7619
17	0.170	0.01367	28.5	4.982	24.82	1.11	0.7978	0.8095
18	0.180	0.01447	31	5.419	29.36	1.21	0.8291	0.8571
19	0.190	0.01527	35	6.118	37.43	1.37	0.8660	0.9048
20	0.200	0.01608	46	8.041	64.65	1.79	0.9224	0.9524
21	0.210	0.01688	128.6	22.479	505.32	5.02	0.9901	1.0000
				94.077	812.148			

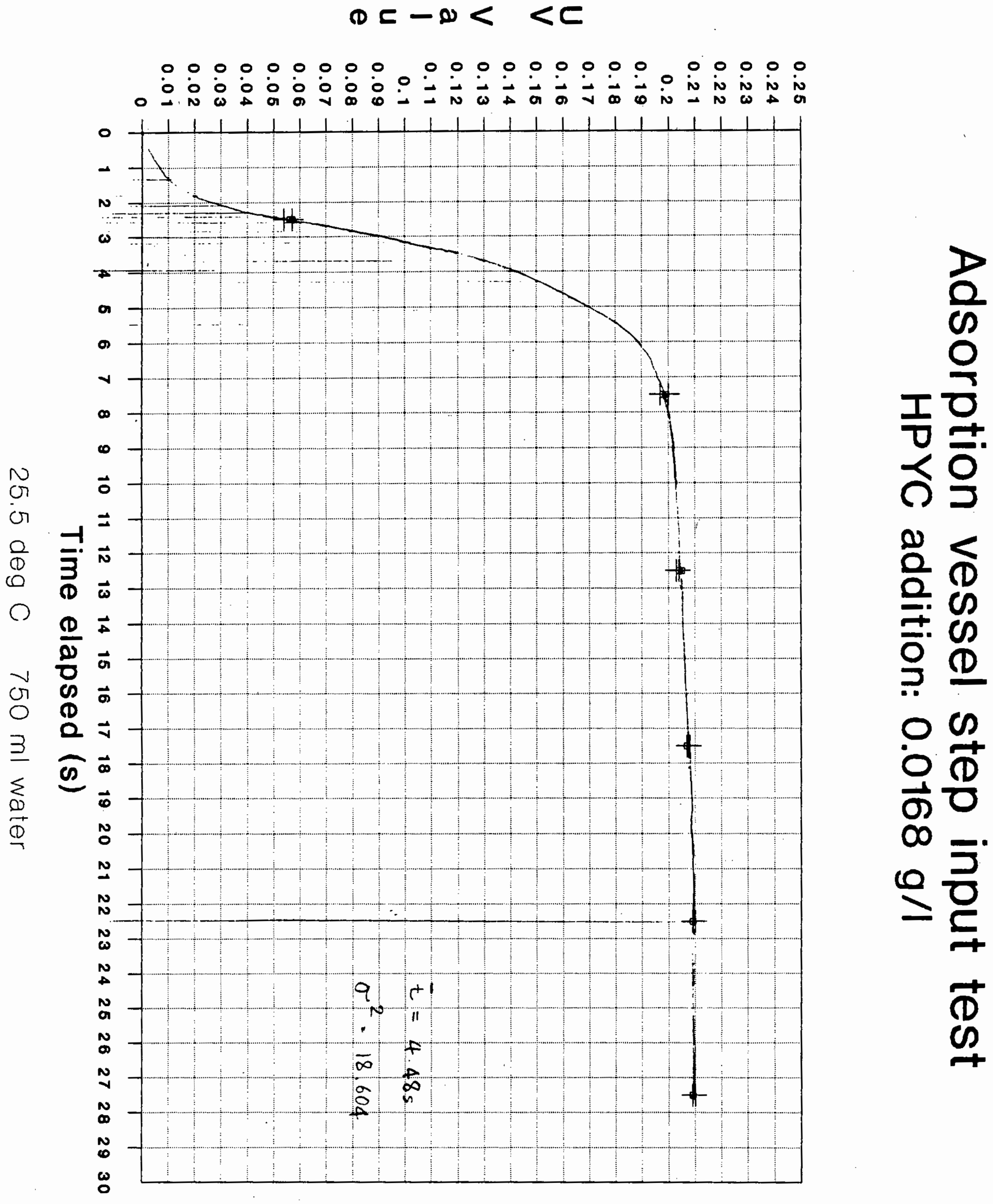
C factor: 0.08038

t bar = 4.48

var^2 = 18.604

D/uL = 0.464

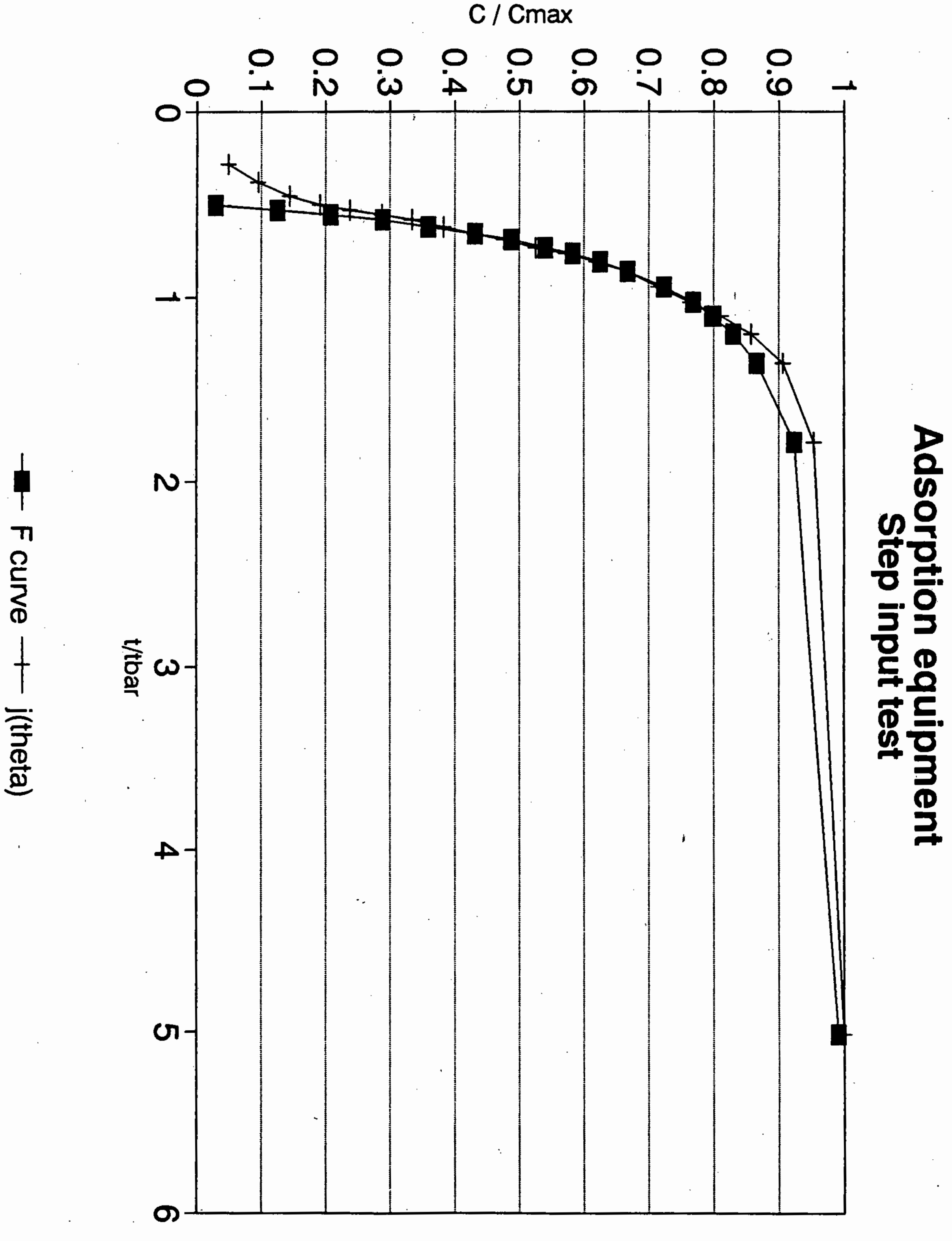
c) Graph for RTD calculations



$\bar{t}$ = 4.48s

σ^2 = 18.604

c) F-curve (segregated flow) and J(theta) vs t/tbar



APPENDIX IV

ADSORPTION TESTWORK DATA

a) Adsorption on Coal and quartz:

1) Coal

ADSORPTION WORK

COAL

Date: Coal type: Landau LAC
Run no.: BCPH02 Size frac: -150 +75 um
Agitation 500 rpm
UV (nm): 258.8 Temp (C) : 25.3 - 25.5

Amine adsorption on coal HPYC (g/l): 0.0253
as a function of pH Pulp Dens : 10 %

			UV VALUES				
Time(s)	pH 2.5	pH 3.0	pH 3.4	pH 4.0	pH 4.8	pH 5.95	pH 7.45
0	0.0255	0.0252	0.0253	0.0253	0.0254	0.0253	0.0253
5	0.0255	0.0252	0.0253	0.0253	0.0251	0.0252	0.0253
10	0.0252	0.0247	0.0250	0.0245	0.0249	0.0243	0.0251
15	0.0244	0.0235	0.0238	0.0234		0.0226	0.0224
20	0.0230	0.0226		0.0220	0.0229	0.0217	0.0198
25	0.0221	0.0214	0.0216	0.0215	0.0219	0.0205	0.0180
30	0.0203	0.0204	0.0202	0.0195	0.0202	0.0187	0.0159
35	0.0190	0.0187	0.0185	0.0178	0.0185	0.0165	0.0149
40	0.0173	0.0172		0.0175		0.0159	0.0135
45		0.0162	0.0166			0.0147	0.0126
50	0.0154			0.0161	0.0170	0.0141	0.0113
55		0.0150	0.0148			0.0126	0.0102
60	0.0135			0.0135	0.0161		0.0091
65		0.0132	0.0136			0.0110	0.0085
70	0.0121			0.0123	0.0139		0.0082
75		0.0120	0.0123			0.0095	0.0073
80	0.0112			0.0117	0.0132		
85							
90				0.0111		0.0085	
95		0.0118	0.0114				
100	0.0106				0.0125		0.0065

2) Quartz

ADSORPTION WORK

QUARTZ

Date:
Run no.: BQPH02
Agitation 500 rpm
UV (nm) : 258.8

Feed type: Delmas silica
Size frac: -150 +75 um

Temp (C) : 25.3 - 25.5

Adsorption of amine on quartz as a fuction of pH

HPYC (g/l): 0.0191
Pulp Dens : 10 %

	UV Values				Conc. values			
Time(s)	pH 6.00	pH 3.0	pH 2.5	pH 3.9	pH 6.00	pH 3.0	pH 2.5	pH 3.9
0	0.226	0.224	0.226	0.227	0.0191	0.0189	0.0191	0.0192
5	0.226	0.224	0.226	0.227	0.0191	0.0189	0.0191	0.0192
10	0.226	0.224	0.225	0.219	0.0191	0.0189	0.0190	0.0185
15	0.203	0.224	0.224	0.215	0.0172	0.0189	0.0189	0.0182
20	0.180	0.217	0.218	0.211	0.0152	0.0183	0.0184	0.0178
25	0.163	0.213	0.217	0.193	0.0138	0.0180	0.0183	0.0163
30	0.149	0.203	0.205		0.0126	0.0172	0.0173	
35		0.198	0.202	0.175		0.0167	0.0171	0.0148
40	0.146	0.194	0.198	0.165	0.0123	0.0164	0.0167	0.0139
45	0.142	0.188	0.195	0.162	0.0120	0.0159	0.0165	0.0137
50	0.141	0.187	0.195	0.161	0.0119	0.0158	0.0165	0.0136
55								

b) Effect of dextrin conditioning: Simultaneous addition of dextrin and HPYC

ADSORPTION WORK
COAL

Date: Feb 1990
Run no.: Dexads2
Agitation 500 rpm
UV (nm): 259.00

Simultaneous adsorption
of Dextrin and Amine

Coal type: Landau LAC
Size frac: -150 +75 um

Temp°(C) :25.7
DEXTRIN : 500 g/t
HPYC (g/l): 0.0185
Pulp Dens : 10 %

	UV Values				Concentration values (g/l)			
Time(s)	No dex 1	No dex 2	Dex 1	Dex 2	No dex 1	No dex 2	Dex 1	Dex 2
0.0	0.197	0.197	0.197	0.197	0.0185	0.0185	0.0185	0.0185
2.5	0.193		0.193		0.0181	0.0000	0.0181	0.0000
5.0		0.181		0.191	0.0000	0.0170	0.0000	0.0179
7.5	0.167		0.162	0.161	0.0157	0.0000	0.0152	0.0151
10.0					0.0000	0.0000	0.0000	0.0000
12.5	0.145		0.142	0.143	0.0136	0.0000	0.0133	0.0134
15.0		0.143			0.0000	0.0134	0.0000	0.0000
17.5	0.130		0.132	0.133	0.0122	0.0000	0.0124	0.0125
20.0					0.0000	0.0000	0.0000	0.0000
22.5	0.127		0.127	0.127	0.0119	0.0000	0.0119	0.0119
25.0		0.120			0.0000	0.0113	0.0000	0.0000
27.5	0.119		0.122	0.122	0.0112	0.0000	0.0115	0.0115
30.0					0.0000	0.0000	0.0000	0.0000
32.5	0.115		0.119	0.118	0.0108	0.0000	0.0112	0.0111
35.0		0.112			0.0000	0.0105	0.0000	0.0000
37.5	0.111				0.0104	0.0000	0.0000	0.0000
40.0			0.115	0.114	0.0000	0.0000	0.0108	0.0107
42.5					0.0000	0.0000	0.0000	0.0000
45.0		0.109			0.0000	0.0102	0.0000	0.0000
47.5	0.107				0.0100	0.0000	0.0000	0.0000
50.0			0.111	0.110	0.0000	0.0000	0.0104	0.0103
52.5	0.104				0.0098	0.0000	0.0000	0.0000
55.0		0.101			0.0000	0.0095	0.0000	0.0000
57.5	0.102				0.0096	0.0000	0.0000	0.0000
60.0			0.106	0.106	0.0000	0.0000	0.0100	0.0100
62.5	0.100				0.0094	0.0000	0.0000	0.0000
65.0		0.100			0.0000	0.0094	0.0000	0.0000
67.5	0.096				0.0090	0.0000	0.0000	0.0000
70.0			0.102	0.101	0.0000	0.0000	0.0096	0.0095
72.5	0.098				0.0092	0.0000	0.0000	0.0000
75.0		0.099			0.0000	0.0093	0.0000	0.0000
77.5	0.098				0.0092	0.0000	0.0000	0.0000
80.0			0.099	0.098	0.0000	0.0000	0.0093	0.0092
82.5	0.096				0.0090	0.0000	0.0000	0.0000
85.0		0.095			0.0000	0.0089	0.0000	0.0000
87.5					0.0000	0.0000	0.0000	0.0000
90.0			0.097	0.095	0.0000	0.0000	0.0091	0.0089
92.5	0.089				0.0084	0.0000	0.0000	0.0000
95.0		0.089			0.0000	0.0084	0.0000	0.0000
97.5					0.0000	0.0000	0.0000	0.0000
100.0					0.0000	0.0000	0.0000	0.0000

c) Effect of dextrin conditioning: prior conditioning with dextrin

ADSORPTION WORK
COAL

Date: Feb 1990
Run no.: Stepads3
Agitation 500 rpm
UV (nm): 259.00

Coal type: Landau LAC
Size frac: -150 +75 um

Temp (C) : 25.7

Step imput of amine -
effect of prior Dex cond

HPYC (g/l): 0.0185
Pulp Dens : 10 %

Time(s)	Dex 1	Dex 2	No Dex 1	No dex 2	Dex 3	No dex 3	No dex 3	Dex 4 no cond
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
2.5	0.090	0.081	0.041	0.030	0.081	0.032	0.031	0.033
7.5	0.149	0.156	0.106	0.091	0.138	0.089	0.082	0.089
12.5	0.184	0.196	0.133	0.122	0.177	0.121	0.115	0.128
17.5	0.198	0.205	0.138	0.129	0.198	0.129	0.128	0.143
22.5	0.202	0.207	0.137	0.129	0.202	0.130	0.133	0.152
27.5	0.203	0.207	0.134	0.126	0.198	0.129	0.133	0.154
32.5	0.203	0.205	0.132	0.124	0.203	0.127	0.132	0.156
37.5	0.202	0.204	0.128	0.121	0.203	0.123	0.129	0.154
42.5	0.200	0.204	0.125	0.118	0.202	0.120	0.127	0.152
47.5	0.198	0.201	0.120	0.115	0.199	0.117	0.123	0.151
52.5	0.196				0.195			
55.0		0.198		0.112		0.114	0.116	0.140
57.5	0.194							
60.0			0.113		0.190			
62.5	0.191							
65.0		0.183		0.106		0.103	0.111	0.129
67.5	0.189							
70.0			0.106		0.180			
72.5	0.186							
75.0		0.176		0.100		0.101	0.106	0.123
77.5								
80.0			0.102		0.175			
82.5								
85.0		0.172		0.097		0.096	0.102	0.119
87.5								
90.0	0.170		0.097		0.168			
92.5								
95.0		0.166		0.093		0.092	0.099	0.116
97.5								
100.0	0.159		0.093		0.162			
102.5								
105.0		0.161		0.089		0.088	0.095	0.113
107.5								
110.0	0.153		0.088		0.159			
112.5								
115.0		0.156		0.086		0.085	0.092	0.111
117.5								
120.0	0.148		0.085		0.155			
122.5								
125.0		0.151		0.083		0.082	0.089	0.107
127.5								
130.0	0.142		0.082		0.152			
132.5								
135.0		0.149		0.089		0.080	0.085	0.102
137.5								
140.0	0.138		0.079					

APPENDIX V

CALCULATION OF SAMPLE ASH CONTENT

Ash analysis procedure

The ash analysis on each sample is performed according to the following procedure:

1. The coal sample is oven dried at 80°C overnight and then removed from the oven and allowed to reach room temperature.
2. Sample pulverized and well mix - 20 g (approximate) sample taken and stored sealed in plastic sample bottle
3. Crucibles for ashing stored in desiccator for at least 6 hours before use.
4. Crucible mass removed from desiccator and empty mass recorded on four figure Mettler laboratory balance - C_e.
5. 0.8 to 1.2 g coal is placed in crucible and the full crucible mass is recorded - C_f.
6. Crucible is placed in muffle furnace and when fully loaded, furnace controller is set to 820°C and the samples ashed for 4 hours. The furnace is then set to 80°C and allowed to cool overnight.
7. Ashed samples are removed from the furnace and placed in a desiccator for 2 hours to cool to room temperature.
8. The mass of each crucible is then recorded - C_a.

The ash fraction for each sample is calculated as follows:

$$a_m = \frac{C_a - C_e}{C_f - C_e}$$

APPENDIX VI

BATCH FLOTATION SPREADSHEET CALCULATIONS

SPREADSHEET CALCULATIONS ARTIFICIAL MIXTURES:

Three sample spreadsheets are presented below (see pages 6-8).

The first spreadsheet is a Reverse flotation example with 50 % quartz as the added gangue material and a 7.2% LAC coal sample. The second shows the situation for Kaolin using the same spreadsheet. Corrections for ash content are shown.The third is an example of the case for forward flotation where the LAC is beneficiated and so various adjustments are made to the spreadsheet calculations.

1. Calculation of gangue and LAC recovery

The following nomenclature is used:

M_c = Concentrates mass	G_T = Mass added gangue in tails
M_T = Tails mass	G_c = Mass added gangue in concentrates
a_m = measured ash content	L_T = Mass L.A.C. in tails
a_L = ash content of LAC	L_c = Mass L.A.C. in concentrates
a_g = "ash" content of gangue	T = Total feed mass
(gangue may contain volatiles)	i = subscript for sample no. i

For Quartz: a_g=1.0, for Kaolin: a_g=0.86

Assumption: the LAC is not beneficiated, i.e. LAC recovered in concentrates has the same ash content as the LAC in the tails which is the same as the feed

Gangue recovery:

For a concentrate mass, M_C, the calculation is:

$$a_m = a_L L_c + a_g G_c \qquad (1)$$

$$G_c + L_c = M_c \qquad (2)$$

Combining these equations, we obtain:

$$G_c = \frac{M_c(a_m - a_L)}{(a_g - a_L)} \qquad (3)$$

and

$$L_c = M_c - G_c \qquad (4)$$

Similarly, for the tails

$$G_T = \frac{M_T(a_m - a_L)}{(a_g - a_L)} \qquad (5)$$

and

$$L_T = M_T - G_T \qquad (6)$$

Equations 3 to 6 are used to calculate the cumulative gangue and LAC concentrate recoveries of the concentrates as well as the final recoveries for both concentrates and tails.

Examples: For Quartz, $a_g = 1.0$, take $a_L = 0.072$ (7.2% ash)

equation 3 becomes:

$$G_c = \frac{M_c(a_m - 0.072)}{(0.928)}$$

If $M_c = 145.6$ g, $a_m = 0.891$ (89.1% ash), then $G_c = 128.5$ g and $L_c = 17.1$g (see sample spreadsheet 1)

For Kaolin, $a_g = 0.86$, take $a_L = 0.072$ (7.2% ash)

equation 3 becomes:

$$G_c = \frac{M_c(a_m - 0.072)}{(0.788)}$$

If $M_c = 89.16$ g, $a_m = 0.7886$, then $G_c = 81.08$ g and $L_c = 8.08$ g (see sample spreadsheet 2)

2. Corrected Ash

To calculate the actual ash content of a concentrate or tailings sample, the following calculation is used:

$$a_{actual} = \frac{G_c + a_L L_c}{M_c} \text{ (concentrate)} \qquad (7)$$

For Kaolin, where $a_g = 0.86$ and using previous example's data

$$a_{actual} = \frac{81.08 + 0.072(8.08)}{89.16}$$

$$= 0.9101 \text{ (91.01\% ash)}$$

Thus the actual value is considerable higher than the measured value (78.86%)

For quartz, ($a_g = 1.0$) the measured value is the actual value.

All the spreadsheets display additional calculated data which is not printed on the spreadsheets for the actual experimental runs

a)Concentrates cumulative LAC recovery for each sample (i) is given by:

$$\frac{\text{LAC mass in sample i + sum of previous LAC masses}}{\text{Total LAC in feed}}$$

c) Durban Navigation (Durnacol): forward flotation

FORWARD FLOTATION OF DURNACOL: COLUMN CELL

No.	Yield %	Product grade	Coal rec (%)	Ash rec (%)	DISC ASH (%)	Shellsol g/t	PROD SULF %	COL. ZONE Ht (m)	Frother g/t DIBK	Pulp Density	Bias rt cm/s	Froth Water kg/h	Air rate cm/s
			-- NORMAL FLOTATION --										
NCDURN	76.50	12.75	92.88	65.40	78.23	1000	1.42	1.0	15 ul/l	10	0.26	2.75	2.11
NCDURN	77.57	12.80	93.53	64.13	79.14	1000	1.39	1.0	15 ul/l	10	0.322	2.43	2.11
NCDURN	70.79	11.23	87.87	72.09	72.09	750		1.0	15 ul/l	10	0.271	2.46	2.11
NCDURN	65.79	10.20	83.27	76.90	76.77	500		1.0	15 ul/l	10	0.258	3.49	2.11
NCDURN	66.01	9.90	84.25	76.77	65.3	500		1.0	15 ul/l	10	0.251	4.02	2.11
NCD20_0	76.50	12.44	90.76	63.58	77.31	1000	1.37	1.0	15 ul/l	20	0.285	1.26	2.11

FEED ASH: 28.9 %

FEED SULPHUR: 1.21%

c) Durban Navigation (Durnacol): data of van Holt

Release flotation tests

Yield (%)	Ash (%)	
2.49	5.63	
5.52	5.66	
8.85	5.90	
11.21	5.96	
17.32	7.27	
25.89	7.50	
33.18	7.67	
36.92	7.92	
37.93	7.69	
40.97	8.58	
48.02	9.74	
51.39	9.83	
56.61	12.51	
58.41	11.76	
64.03	12.22	
68.03	12.06	
72.71	13.36	
76.01	15.34	
3.99		5.91
7.27		6.52
15.69		6.71
25.24		7.18
31.18		7.50
33.90		7.19
34.51		7.33
38.26		8.64
39.56		8.45
41.53		8.36
47.83		10.67
52.68		11.16
57.57		11.59
61.83		11.89
63.76		12.26
65.79		13.96
67.88		13.07
74.05		13.43

Forward flotation - USBM sparger

Yield	Rec	Ash
6.52	8.90	8.52
21.40	28.51	8.07
81.11	91.73	20.41
58.34	70.94	12.14
16.42	22.24	8.65
38.23	45.30	11.83
79.01	92.60	15.15
74.63	90.31	13.14
81.01	93.97	17.27
78.21	92.11	16.70
36.34	46.57	10.08
23.52	29.77	10.27
80.33	93.93	18.63
71.83	84.99	16.45
18.04	22.86	7.41
15.66	20.24	10.05
5.27	6.83	8.38
24.53	32.66	8.03
82.15	93.04	19.51
33.32	40.05	11.88
83.82	94.45	16.78
81.40	94.10	17.44
78.67	92.17	16.62
28.80	38.91	8.65
24.92	32.31	10.27
83.43	95.53	19.03
62.95	78.65	14.65
17.94	22.96	6.52

APPENDIX XIII

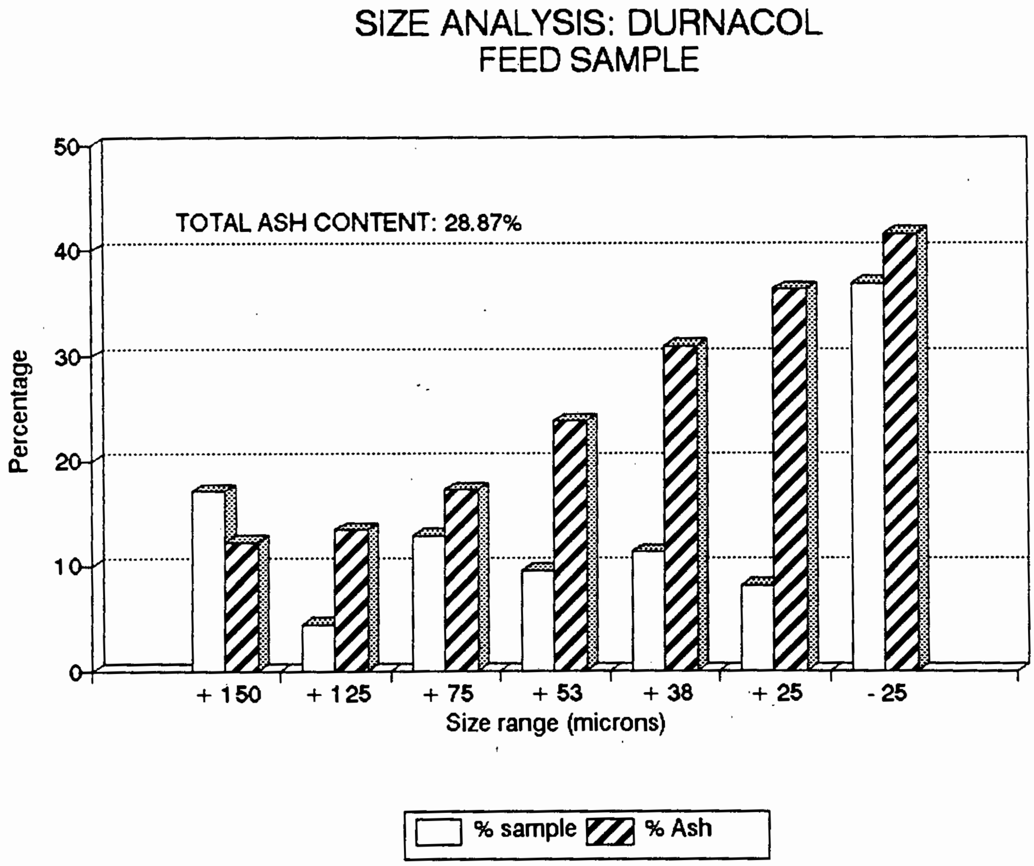
COLUMN FLOTATION DATA: DURNACOL SAMPLE ANALYSIS

a) Feed size analysis

Coal:DURNACOL Date: 10/10/90
Remark: FEED

Size fraction	**Mass**	**Mass %**	**Ash %**	**Ash mass**	**Ash in sz frac**	**Coal in sz frac**
+ 150	12.18	17.15	12.16	1.48	2.09	15.07
+ 125	3.09	4.35	13.4	0.41	0.58	3.77
+ 75	9.08	12.79	17.24	1.57	2.20	10.58
+ 53	6.76	9.52	23.81	1.61	2.27	7.25
+ 38	8.02	11.29	30.92	2.48	3.49	7.80
+ 25	5.74	8.08	36.32	2.08	2.94	5.15
- 25	26.14	36.81	41.57	10.87	15.30	21.51
	71.01	100		20.50		

Ash content: 28.87



b) Reverse flotation run RCDURN02: Concentrates and tailings

Coal:DURNACOL Date: 11/11/90
Reverse flotation concentrate run RCDURN

Size fraction	Mass	Mass %	Ash %
+ 150	0.69	3.27	47.2
+ 75	0.93	4.41	54.4
+ 53	0.78	3.70	61.9
+ 38	1.15	5.45	63.4
+ 25	1.85	8.77	63.4
- 25	15.69	74.40	54.03
	21.09	100	

Filter cloth sparger

Coal:DURNACOL Date: 11/11/90
Reverse flotation tailings RCDURN02

Size fraction	Mass	Mass %	Ash %
+ 150	6.44	21.03	6.98
+ 75	7.83	25.57	11.02
+ 53	3.45	11.27	15.76
+ 38	3.17	10.35	21.26
+ 25	2.55	8.33	28
- 25	7.18	23.45	36.01
	30.62	100	

SPARGER: filter cloth

c) Forward flotation run NCDURN02: Concentrates and tailings

Coal:DURNACOL Date: 11/11/90
Forward flotation concentrates

Size fraction	Mass	Mass %	Ash %
+ 150	6.47	21.00	8.3
+ 75	6.99	22.69	11.5
+ 53	3.39	11.00	15.7
+ 38	3.5	11.36	13.6
+ 25	2.88	9.35	16.7
- 25	7.58	24.60	13.2
	30.81	100	

SPARGER: filter cloth

Coal:DURNACOL Date: 11/11/90
Forward flotation tailings

Size fraction	Mass	Mass %	Ash %
+ 150	0.29	1.16	29.59
+ 75	0.39	1.57	78.4
+ 53	0.67	2.69	82.48
+ 38	1.49	5.98	82.02
+ 25	1.69	6.78	81.1
- 25	20.39	81.82	70.9
	24.92	100	

SPARGER: filter cloth

d) Size analysis calculations

DURNACOL: Reverse flotation run RCDURN02
Size Anaysis

	CONCENTRATES		% of total	% of	TAILINGS		% of total	% of	recons
Size fraction	Mass %	Ash %	in size fr	total fd	Mass %	Ash %	in size fr	total fd	feed
+ 150	3.27	47.2	5.82	0.93	21.03	6.98	94.18	15.05	22.03
+ 75	4.41	54.4	6.42	1.25	25.57	11.02	93.58	18.30	26.96
+ 53	3.70	61.9	11.55	1.05	11.27	15.76	88.45	8.06	12.57
+ 38	5.45	63.4	17.31	1.55	10.35	21.26	82.69	7.41	12.35
+ 25	8.77	63.4	29.51	2.49	8.33	28	70.49	5.96	11.66
- 25	74.40	54.03	55.78	21.17	23.45	36.01	44.22	16.78	52.33
									137.9

Concs	39.23
Tails	98.67
	137.90

DURNACOL Coal: Forward float Run: NCDURN02
Size Analysis

	CONCENTRATES		% of fd	% of	TAILINGS		% of fd	% of	Recons
Size fraction	Mass %	Ash %	in size fr	tot feed	Mass %	Ash %	in size fr	tot feed	feed
+ 150	21.00	8.30	98.43	16.29	1.16	29.59	1.57	0.26	25.03
+ 75	22.69	11.50	98.04	17.60	1.57	78.40	1.96	0.35	27.15
+ 53	11.00	15.70	93.40	8.53	2.69	82.48	6.60	0.60	13.81
+ 38	11.36	13.60	86.80	8.81	5.98	82.02	13.20	1.34	15.35
+ 25	9.35	16.70	82.67	7.25	6.78	81.10	17.33	1.52	13.27
- 25	24.60	13.20	50.99	19.08	81.82	70.90	49.01	18.34	56.59
									151.2

Concs	117.30
Tails	33.90
	151.20

e) Malvern size analysis: Reverse run RCDURN02

```
Coal:DURNACOL         5/01/91
Remark: reverse column concentrates
Reverse flotation     Filter cloth sparger
rcdurn02
Size fraction Mass %
-------------------------------------------------

+87              0.10
+53.5            4.90
+37.6            7.80
+28.1            8.00
+21.5            8.90
+16.7            9.80
+13.0            9.20
+10.1            8.90
+7.9             8.90
+6.2             7.20
+4.8             5.30
+3.8             6.20
+3.0             6.10
+2.4             3.00
+1.9             2.10
-1.9             3.60
-------------------------------------------------
               100.00

Ash content:    52.71      D50     12.5 um
```

```
Coal:DURNACOL         5/01/91
Remark: reverse column Tails
Reverse flotation
rcdurn02
Size fraction Mass %
----------------------------------------

+87             37.40
+53.5           18.40
+37.6            8.80
+28.1            5.70
+21.5            4.80
+16.7            4.30
+13.0            3.50
+10.1            3.50
+7.9             3.40
+6.2             2.50
+4.8             1.80
+3.8             2.10
+3.0             1.90
+2.4             0.80
+1.9             0.50
-1.9             0.70
----------------------------------------
               100.10

Ash content:     18.5      D50        64
```

f) Malvern size analysis: Forward run NC20_01

```
Coal:DURNACOL          5/01/91
Remark: column concentrates (20% PD)
Forward flotation - FCloth sparger
NDC20_01
Size fraction Mass %
----------------------------------------

+87             27.70
+53.5           22.80
+37.6            8.30
+28.1            8.50
+21.5            5.70
+16.7            4.20
+13.0            4.70
+10.1            3.80
+7.9             3.20
+6.2             2.60
+4.8             1.80
+3.8             2.20
+3.0             2.20
+2.4             0.80
+1.9             0.60
-1.9             0.90
----------------------------------------
               100.00

Ash content:    12.44       D50      54.3
```

```
Coal:DURNACOL          5/01/91
Remark: column tails (20% PD)
Forward flotation: NDC20_1

Size fraction Mass %
-------------------------------------------------

+87              0.20
+53.5            6.80
+37.6            6.40
+28.1            5.90
+21.5            8.10
+16.7            7.90
+13.0            7.10
+10.1            7.90
+7.9             8.50
+6.2             7.40
+4.8             6.10
+3.8             6.70
+3.0             6.80
+2.4             4.30
+1.9             3.40
-1.9             6.50
-------------------------------------------------
               100.00

Ash content: 63.97          D50      10.1 um
```

g) Float–sinks analysis

DURNACOL FLOAT AND SINKS ANALYSIS

RESULTS						SPECIFIC GRAVITY
COAL MASS	MASS OF FLOATS	MASS OF SINKS	RECONS MASS	% FLOATS	% MASS LOSS	
2.0498	0.4244	1.5870	2.0114	21.100	1.87	1.3
2.1284	0.6131	1.4680	2.0811	29.460	2.22	1.33
1.7425	0.7630	0.9665	1.7295	44.117	0.75	1.4
2.0065	1.0830	0.9531	2.0361	53.190	-1.48	1.45
1.9485	1.1315	0.7551	1.8866	59.976	3.18	1.5
1.8720	1.1800	0.6775	1.8575	63.526	0.77	1.55
1.7536	1.2044	0.5649	1.7693	68.072	-0.90	1.6
2.0240	1.5709	0.4493	2.0202	77.760	0.19	1.7

COLUMN FLOTATION: FLOAT AND SINKS ANALYSIS
OF REVERSE AND FORWARD FLOTATION.
DURNACOL COAL

	ND/RCD		FEED ASH			
	% FLOATS	% MASS LOSS	% ASH FLOATS	% ASH SINKS	SPECIFIC GRAVITY	CALC. FEED ASH %
C	90.754	0.98	8.56	54.89	1.7	12.84
	90.475	1.02	8.54	54.89	1.7	12.95
FORWARD T	10.195	1.91	19.86	85.90	1.7	79.17
	15.330	1.23	19.86	84.00	1.7	74.17
C	44.051	1.74	14.19	79.12	1.7	50.52
	44.056	0.82	15.37	78.75	1.7	50.83
REVERSE T	81.520	1.56	9.6	64.92	1.7	19.82
	81.855	1.94	9.6	64.92	1.7	19.64

h) Sulphur analysis

SULPHUR ASSAYS
LECO SC32
Durnacol coal

No. In table	Type	Sample name	Sulphur % 1	Sulphur % 2	Sulphur % average
1	F	NCDURN01 Feed	1.19	1.23	1.210
	F	NCDURN01 Tails	0.997	0.986	0.992
	F	NCDURN01 Concs	1.4	1.43	1.415
1	R	RCDURN01 Feed	1.22	1.25	1.235
	R	RCDURN01 Tails	1.31	1.28	1.295
	R	RCDURN01 Concs	1.01	1.01	1.010
2	F	NCDURN02 Feed	1.24	1.21	1.225
	F	NCDURN02 Tails	0.772	0.767	0.770
	F	NCDURN02 Concs	1.37	1.4	1.385
4	R	RCDURN04 Feed	1.22	1.25	1.235
	R	RCDURN04 Tails	1.32	1.38	1.350
	R	RCDURN04 Concs	0.889	0.902	0.910
6	F	NCD25 Feed	1.17	1.22	1.195
	F	NCD25 Tails	0.821	0.815	0.818
	F	NCD25 Concs	1.35	1.39	1.370
15	R	USBM10 Feed	1.23	1.19	1.210
	R	USBM10 Tails	1.35	1.35	1.350
	R	USBM10 Concs	0.665	0.666	0.666
16	R	USBM11 Feed	1.23	1.18	1.205
	R	USBM11 Tails	1.31	1.34	1.325
	R	USBM11 Concs	0.696	0.692	0.694

F=Forward
R=Reverse

Average of two readings