Introduction

Coal is currently one of the most important commodities in South Africa; it is the main source of energy, providing about 88 per cent of the country’s total energy needs. South Africa is ranked sixth in the world in economically recoverable coal reserves (34 224 Mt in 2005) and fifth globally in annual production (245 Mt in 2005) (Van Niekerk et al., 2008).

Coal preparation or washing plays a significant role in the removal of the high inert mineral matter content from South African coals (Kershaw and Taylor, 1992). The beneficiation of South African coals usually generates large quantities of wastes in the form of discards and ultrafine slurries. A survey by the Department of Minerals and Energy (DME, 2001) indicated that the South African coal processing sector generated about 48 Mt of coal discards and 12 Mt of ultrafines per annum (this amount will have increased by 2013). This represents a neglected energy source, as noted by Lloyd (2000). However, while his paper was concerned only with the coarse discard material, in this paper we will focus attention on the ultrafines.

According to Bunt (1997), the mechanization and automation techniques used in modern coal mining are responsible for the large amount of fine and ultrafine coal being produced. Although ultrafine coals represent a potential resource for energy production – they are generally comparable in quality to the run-of-mine: the air-dry calorific value is typically between 20–27 MJ/kg, while the air-dry sulphur content ranges from 0.6–2.2 per cent – the cost of dewatering has limited the reclamation and treatment of these materials. This is despite the fact that they also represent a long-term environmental liability, particularly with regard to the generation of acid rock drainage (ARD) (Geldenhuis and Bell, 1998; Gray, 1997; Bell et al., 2001; Naicker et al., 2003; Akcil and Koldas, 2006). In keeping with the increased need to meet the demand for energy in South Africa, coal production is expected to increase in the coming years, and with it the amount of ultrafines generated. Efficient use will need to be made of valuable coal resources that are currently discarded, while at the same time the macerals tend to have a higher density than vitrinite, and are less floatable) (Falcon, 1977; Sanders and Brookes, 1986; Kershaw and Taylor, 1992). Consequently, the beneficiation of South African coals usually generates large quantities of wastes in the form of discards and ultrafine slurries. A survey by the Department of Minerals and Energy (DME, 2001) indicated that the South African coal processing sector generated about 48 Mt of coal discards and 12 Mt of ultrafines per annum (this amount will have increased by 2013). This represents a neglected energy source, as noted by Lloyd (2000). However, while his paper was concerned only with the coarse discard material, in this paper we will focus attention on the ultrafines.

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Flotation of coal and sulphur from South African ultrafine colliery wastes

Environmental footprint of coal mines will need to be reduced as far as ARD is concerned. In view of its proven nature, froth flotation should represent a viable method for coal ultrafine beneficiation in South Africa – according to Aplan and Arnold (1991), it is the only method used in the beneficiation of ultrafine coal at the commercial scale worldwide – and for removing sulphides, including pyrite (Miller, 1975; Miller and Deurbrouck, 1982; Laskowski, 2001) using xanthate collectors.

The recovery of coal by flotation is possible due primarily to the inherent separability of coal and secondly to the use of reagents to promote the flotation process. Investigations have shown that South African coal ultrafines can be beneficiated by flotation (Fickling, 1985; Horsfall et al., 1986; Anderson, 1988; Stonestreet, 1988, 1991, 1992; Bunt, 1997; Harris and Franzidis, 1995; Opperman et al., 2002). These flotation processes are aimed at beneficiating coal by removing ash-forming minerals. Desulphurizing flotation focuses on selective removal of sulphide minerals, especially pyrite, which are the major environmental problem in ARD generation (Kawatra and Eisele, 2001). Several investigations have suggested the flotation of sulphur-bearing minerals, in particular pyrite, from coal with simultaneous depression of coal (Kawatra and Eisele, 2001; Laskowski, 2001). Sulphide flotation, in which bulk coal is left behind in the tailing, is sometimes referred to as reverse flotation. Direct sulphide flotation might be useful in cases of coal processing wastes with little or no economic value, but which nevertheless represent a perpetual risk of environmental. The recovered sulphur stream might be used as raw material in the manufacture of products such as sulphuric acid; otherwise it might be amenable to bioleaching. Options also exist for the desulphurized stream in the area of ARD mitigation: it could either be used for co-disposal with high-sulphur waste to produce environmentally benign composites or used as cover to limit the diffusion of oxygen, one of the driving forces in the oxidation of sulphide minerals.

Consideration of the above suggests that there are environmental and economic benefits in processing coal ultrafines further because of their large content of liberated and high-grade coal in comparison to the coarse discards. The reclamation of these otherwise waste products provides potentially cost-effective and environmentally attractive solutions for the management of ARD. In doing so, it may be feasible to reduce the environmental footprint of coal mines as far as ARD is concerned. Furthermore, it will result in the recovery of substantial amounts of energy that are currently wasted and a marked reduction in the amount of ultrafine wastes to be handled and disposed of.

The present study was undertaken in an attempt to provide a technical basis for recovering valuable coal from current ultrafine wastes, or removing sulphide minerals, through the application of froth flotation techniques. In essence the project seeks to investigate whether flotation techniques can be successfully used to recover a saleable coal product from the coal ultrafines arising from mining and processing operations, or to remove acid-generating materials prior to the disposal of coal flotation residues. To this end, the recovery and desulphurization of coal through the application of froth flotation techniques was investigated systematically through laboratory batch flotation tests. The effects of key parameters, such as the type and dosage of reagents, the extent of both coal recovery in a direct coal float, and sulphide sulphur removal using one, two, and three stages of collector addition, were investigated. This paper presents the results of the study.

Experimental Work

Ultrafine coal sample

Details of the coal sample used in the experimental work have been presented elsewhere (Kazadi Mbamba et al., 2012). Briefly, the sample was a medium-rank bituminous waste coal, obtained as a naturally arising thicker underflow from a processing plant in the Middleburg area.

The sample was inertinite-rich (76 per cent by volume, mineral matter-free basis) and low in vitrinite (approximately 21 per cent), and contained 1.08 per cent total sulphur, of which 48 per cent was in the form of sulphide, 30 per cent in the form of sulphates, and 22 per cent in the form of organic sulphur. The ash content of the sample was 54.4 per cent.

The sample was red-milled to 75 per cent passing 150 μm prior to flotation.

Reagents

The collectors used in the coal flotation tests were laboratory-grade dodecane, supplied by Merck, as well as kerosene and oleic acid, which were supplied by Sigma-Aldrich and May & Baker Ltd, Dagenham England, respectively. Sodium ethyl xanthate (SEX), sodium isobutyl xanthate (SIBX), and potassium amyl xanthate (PAX), all supplied by Senmin, were used as sulphide mineral collectors. All sulphide flotation tests were performed using yellow dextrin, supplied by Africa Products (Pty) Limited, as coal depressant. Fresh depressant was prepared every second day.

The frother used in all the flotation experiments was methyl iso-butyl carbinitol (MIBC), supplied by Sigma-Aldrich.

Flotation procedures

All the flotation tests were carried out at room temperature in a 3-litre bottom-driven modified Leeds batch flotation cell. The procedures for coal and sulphide flotation are described in a previous paper (Kazadi Mbamba et al., 2012). The procedures for staged addition of xanthate collector are described in a later section. For all of the runs the impeller speed was 1200 r/min, the aeration rate was 0 r/min, and the pulp pH that of Cape Town tap water (pH 7.6).

Analysis of flotation samples

Feed, concentrate, and tailings samples from each flotation experiment were filtered and oven-dried overnight and weighed before analysis. Sulphur analysis was carried out using the LECO sulphur analyser in the Department of Chemical Engineering at the University of Cape Town. Ash analysis was performed according to standard method SANS 131:1997. The results of the coal flotation tests were evaluated in terms of the yield (mass product / mass feed), ash content of the concentrate, and sulphur content of the clean coal and tailings. The results of the sulphide flotation tests were evaluated in terms of recovery and sulphur content of the concentrate.
Results and discussion

Coal flotation

Effect of frother and collector dosage

As reported previously (Kazadi Mbamba et al., 2012), the initial laboratory batch flotation tests were carried out using dodecane (which is used in the Australian Standard method for coal flotation) as collector. The dosage was varied from 0.70 to 3.72 kg/t, while the MIBC frother dosage was kept constant at 0.11 kg/t. After 5 minutes of flotation time, the yield of clean coal was only 20.5 per cent using 3.72 kg/t of dodecane.

In the present study, an attempt was made to increase the yield of clean coal by increasing the frother dosage from 0.11 to 0.28 kg/t MIBC. Figure 1 shows the results obtained compared to the previous results. The data is also presented in Table I to indicate the grades of clean coal and tailing obtained in the tests under the new conditions. Table I also shows the results of a test carried out using no dodecane collector, i.e. in the presence of 0.28 kg/t MIBC frother only.

For each collector dosage, the yield of clean coal increased with the increased dosage of MIBC frother, compared to the previous results. However, it is again interesting to observe that doubling the dosage of dodecane from 1.4 kg/t to 2.79 kg/t increased the yield of clean coal by no more than 6.2 per cent at the elevated MIBC level. Visually, low addition of dodecane was found to result in poor froth development during the flotation test: increasing dodecane dosage improved the froth structure and stability, resulting in increased coal recoveries.

As noted previously (Kazadi Mbamba et al., 2012), the coal sample under investigation may be regarded as difficult to float. The poor recovery of combustibles, even in the presence of high concentrations of dodecane and MIBC, was attributed to the petrographic composition of the coal (low-rank, inertinite-rich), oxidation of the coal surfaces (petrographic analysis indicated that at least 11 per cent of the coal was oxidized), and the high ash content of the coal sample.

Nevertheless, in spite of the poor yield, the ash content of the clean coal was good, ranging from 15.5 per cent to 18.1 per cent. This indicates that dodecane was able to disperse through the pulp and selectively adsorb onto the surfaces of the ultrafine coal particles. However, the addition of dodecane did not stop the sulphur-bearing minerals from floating at the same time as the coal. Table I shows that the sulphur content of the clean coal was about 0.46 per cent compared to 1.08 per cent in the feed. It may be assumed that the sulphur that reported to the concentrate was in the form of organic and sulphide sulphur (sulphate sulphur would have dissolved, hence the poor mass balance). This unintentional flotation of sulphide minerals may have been caused by locked particles, or by excessive collector and conditioning promoting the natural floatability of pyrite particles (Aplan, 1993). It is important to note that in this study no attempt was made to depress pyrite during coal flotation, because in almost every case a good pyrite depressant is also a good coal depressant at the same or higher reagent concentration (Purcell and Aplan, 1991). Perry and Aplan (1985) indicated that pyrite depression during coal flotation may involve a trade-off between coal yield and the sulphur content of the floated coal.

Effect of different collectors

In an attempt to improve the yield and recovery of clean coal, two other laboratory-grade reagents, namely kerosene and oleic acid, were tested in addition to dodecane. The flotation runs were conducted under similar conditions. The results are presented in Figure 2 and Table II. The clean coal yield and ash and sulphur contents provide the basis for comparing the three collectors (Table III).

Although kerosene has been used extensively in the flotation of coal at the industrial scale because it has low enough viscosity to disperse in the slurry and spreads over the coal particles easily, the results indicate that oleic acid was much more efficient in increasing the yield of clean coal.

Table I

<table>
<thead>
<tr>
<th>Dodecane dosage (kg/t)</th>
<th>Clean coal</th>
<th>Tailing</th>
<th>Ash of raw coal (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Yield (%)</td>
<td>Ash (%)</td>
<td>Sulphur (%)</td>
</tr>
<tr>
<td>0.0</td>
<td>7.6</td>
<td>18.1</td>
<td>0.48</td>
</tr>
<tr>
<td>0.7</td>
<td>17.2</td>
<td>15.6</td>
<td>0.46</td>
</tr>
<tr>
<td>1.4</td>
<td>21.2</td>
<td>15.1</td>
<td>0.45</td>
</tr>
<tr>
<td>1.86</td>
<td>24.4</td>
<td>14.0</td>
<td>0.45</td>
</tr>
<tr>
<td>2.79</td>
<td>27.4</td>
<td>15.5</td>
<td>0.47</td>
</tr>
</tbody>
</table>
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As can be seen from Figure 2, the coal ultrafine waste sample responded well to oleic acid as the collecting agent; significant yield was achieved compared to what was obtained in the presence of the aliphatic hydrocarbons. As shown in Table II, a yield of 29.7 per cent was obtained at a dosage of only 0.7 kg/t oleic acid collector. When the oleic acid dosage was increased to 2.79 kg/t, the yield increased further to 56.0 per cent (much more than was obtained with dodecane and kerosene). The results also indicate that when the oleic acid dosage was 2.79 kg/t the ash content of the clean coal was 18.1 per cent. At same dosage of kerosene and dodecane collectors, the yields were 31.4 per cent and 27.4 per cent, respectively, with ash contents of 15.4 per cent and 15.5 per cent, respectively (Table III).

These results suggest that there was a stronger interaction between the oleic acid molecule and the aromatic sites on the coal surface than between an aliphatic hydrocarbon chain and the coal surface. This can be explained by the strong π-bonding that occurs between the hydrophobic component of the coal surface and the double bond of oleic acid. The results also show that the oleic acid did not affect the clean coal grade beyond the increase that would be expected as a result of the greater mass of coal floated. The sulphur content of the clean coal remained at about 0.50 per cent.

In summary, a comparison of three collectors has shown that oleic acid was able to achieve a high yield of clean coal from the coal ultrafine waste, although the grade of clean coal was somewhat poorer compared to what was obtained with dodecane and kerosene. It is of particular interest that oleic acid use is environmentally safe and preferable because of environmental concerns over the use of traditional reagents in coal flotation, given that it is biodegradable and can be of vegetable origin.

Sulphide flotation results

Effect of collector type

Preliminary tests were carried out to determine the most effective xanthate collector for floating pyrite from the coal ultrafine waste under investigation. Potassium amyl xanthate (PAX), sodium isobutyl xanthate (SIBX), and sodium ethyl xanthate (SEX) were tested under the same conditions. These xanthates, most commonly used in mineral flotation, are highly selective for sulphide minerals, as they react chemically with the sulphide surfaces and do not have any affinity for the hydrophilic non-sulphide minerals.

Figure 3 shows the kinetic results for the three xanthates at dosages of 2.33 kg/t. The MIBC frother and yellow dextrin (coal depressant) dosages were kept constant at 0.11 kg/t and 0.93 kg/t, respectively. It is clear that PAX produced the best recovery of 26.3 per cent after 20 minutes of collection time, while SIBX and SEX recovered 20.8 per cent and 1.44 per cent respectively. This is as expected, as PAX is a stronger collector than the other xanthate homologues. As a consequence, PAX was used exclusively in the subsequent sulphide flotation tests.

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It is interesting to observe the large collector dosage required for the flotation of sulphide from coal, compared to that used to recover sulphide minerals from metallic ores (0.10 to 0.25 kg/t). This agrees with the literature (Miller and Deurbrouck, 1982) reports that coal-sourced pyrite differs significantly from ore-sourced pyrite due to surface heterogeneities in the sulphide component, such as clay inclusions, which contribute to the hydrophilic character of the coal-pyrite; as a result, xanthate consumption is about one order of magnitude greater for coal-pyrite than for ore-pyrite.

**Effect of collector dosage**

On the basis of the above result, a series of tests was undertaken in which PAX collector dosage was varied from 0.93 kg/t to 2.33 kg/t. The MIBC frother and yellow dextrin (coal depressant) dosages were kept constant at 0.11 kg/t and 0.93 kg/t, respectively. As can be seen in Figure 4, flotation recovery increased with an increase in the xanthate collector concentration. At 0.93 kg/t of PAX, the lowest dosage, the recovery of the total sulphur mass was only 5.9 per cent. An increase of collector dosage to 2.33 kg/t increased the sulphur recovery to 27.26 per cent. Furthermore, the grade of total sulphur increased at high dosage of collector. The grade at low xanthate collector dosage is an indication that coal was floating as well, due to its natural hydrophobicity (Kazadi Mbamba et al., 2012).

**Stage-wise collector addition**

In order to make more efficient use of the high xanthate dosage, sulphide flotation tests were performed with stage-wise addition of collector. This has been shown in the past to increase flotation yields and recoveries (Stonestreet, 1991; Wills, 1997). The aim was to improve the sulphur recovery from the coal ultrafine waste, and thereby achieve a final tailing with as low sulphur content as possible. The conditions were similar to the previous single-stage addition runs, except that the total quantity of PAX was added in two or three stages. In addition, for the 3-stage addition test, six concentrates were collected over 30 minutes, instead of four.

In practice, the collector was added incrementally and the pulp conditioned for 5 minutes after each addition. MIBC frother and dextrin depressant were introduced into the pulp at the beginning in a single addition. Table IV summarizes the flotation results for 1-, 2- and 3-stage sulphide flotation runs in the presence of PAX as collector, dextrin as coal depressant, and MIBC as frother.

Figure 5 shows the recovery/time profile for sulphur: it can be seen that adding the collector in stages substantially increased the total sulphur recovery, especially in the 3-step addition test. However, the results indicate that 2-step collector addition gave the best overall separation in terms of residual total sulphur in the tailings, low yield, and shorter collection time as compared to the 1- and 3-stage flotation runs.

As shown in Figure 5, addition of 2.35 kg/t PAX in one step resulted in 27.3 per cent sulphur recovery at 1.8 per cent S in one step. With a 2-step addition, 38.5 per cent (cumulative) sulphur recovery was obtained at 1.5 per cent S, and with 3-step addition, 42.1 per cent (cumulative) sulphur recovery was obtained at 1.4 per cent S. The mass yields obtained were 14.1 per cent, 18.5 per cent, and 22.7 per cent, respectively. The sulphur grade in the tailings was 0.56 per cent S for both the 2-step and 3-step flotation, and 0.77 per cent S for the 1-step addition test. The corresponding coal recoveries were 16.0 per cent at 23.7 per cent ash in one stage, 21.7 per cent at 22.6 per cent ash with 2 stages, and 26.7 per cent at 11.3 per cent ash in 3 steps. This indicates the reason for the low sulphur content in the 3-step addition test: the higher the coal content of the sulphide concentrate, the lower the sulphur grade in the tailings.

**Table IV**

<table>
<thead>
<tr>
<th>Sulphide flotation results of stage-wise addition tests</th>
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<tbody>
<tr>
<td>1-stage</td>
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<tr>
<td>---</td>
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<tr>
<td>Flotation time (min)</td>
</tr>
<tr>
<td>PAX dosage (kg/t)</td>
</tr>
<tr>
<td>Yield (%)</td>
</tr>
<tr>
<td>Sulphur grade (%)</td>
</tr>
<tr>
<td>Sulphur recovery (%)</td>
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<tr>
<td>Coal recovery (%)</td>
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<tr>
<td>Ash recovery (%)</td>
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<td>Ash product (%)</td>
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<tr>
<td>Sulphur tailings</td>
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<tr>
<td>Recovery ratio (Sulphur/coal)</td>
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</tbody>
</table>

**Figure 4**—Kinetic flotation results for different dosages of potassium amyl xanthate (PAX) collector. MIBC frother and dextrin (coal depressant) were kept constant at 0.11 kg/t and 0.93 kg/t, respectively.

**Figure 5**—Sulphide flotation results for staged addition of collector PAX. MIBC frother and dextrin coal depressant were kept constant at 0.11 kg/t and 0.93 kg/t, respectively.
the lower the sulphur content. This suggests that the depression of coal could not be maintained over the longer collection time (30 minutes), resulting in a greater recovery of coal into the concentrate.

Effect of depressant
In an attempt to reduce the deportment of coal into the sulphide concentrate and improve the recovery of sulphur, a set of flotation tests was carried out with different dosages of coal depressant. A depressant inhibits the flotation of a given mineral; its function is opposite to that of a collector. Any reagent that will oxidize the surface of coal, thus destroying its natural hydrophobicity, may be considered as a potential coal depressant. In this study, yellow dextrin was used as coal depressant, and its dosage was varied from 0.7 kg/t to 1.4 kg/t while keeping the collector PAX and frother MIBC dosages constant at 1.4 kg/t and 0.11 kg/t, respectively. Figure 6 shows that the addition of dextrin reduced the recovery of total sulphur reporting to the concentrate. Visually, the increased level of dextrin had a negative impact on the froth structure and froth stability. It was noted that each increase in the dosage of the depressant decreased the stability of the froth and reduced the water recovery.

The flotation performance clearly declined on increasing the dextrin dosage from 0.70 to 1.4 kg/t. This supports earlier discussion that the coal under investigation may be oxidized. Fresh, unoxidized, and highly hydrophobic coal strongly adsorbs dextrin; its adsorption by the coal surface decreases as the coal becomes more oxidized. Most coals are susceptible to oxidation by weathering, which begins as soon as the coal is mined and continues during transportation and storage. Oxidation processes result in the formation of oxygen functional groups, most commonly carboxyl, phenolic, and carbonyl functionalities, on the coal surface, which reduce the hydrophobicity of the coal surface by increasing the number of sites that can hydrogen-bond with water molecules. Increasing the dosage of dextrin affected not only the recovery of coal to the concentrate, but also inhibited the floatability of sulphide to some extent. This confirms the finding of Purcell and Aplan (1991) that most pyrite depressants are also coal depressants at a similar, though usually at higher, dosage. For example, many common starches are pyrite depressants at ≈0.1 kg/t, but are coal depressants at 0.3–1.0 kg/t (Purcell and Aplan, 1991).

Conclusions
This study has presented a basis for recovering valuable coal from ultrafine wastes and selectively removing sulphide minerals through the application of froth flotation. Coal flotation experiments using aliphatic (dodecane and kerosene) collectors and MIBC frother indicated that the coal was difficult to float, probably as a result of its petrographic composition (high inertinite content) and oxidation due to weathering. The use of oleic acid as collector with MIBC frother improved the yield of coal to 55.96 per cent at an ash content 18 per cent, from a feed ash of 34.4 per cent.

Sulphide flotation tests were carried out to investigate the possibility of removing sulphur-bearing minerals from coal in one or more stages. Staged addition of PAX collector resulted in enhanced total sulphur recovery and low sulphur content in the tailing, but a substantial amount of coal also reported to the concentrate, indicating inadequate depression of the coal.

Thus, although the feasibility of using flotation to recover coal and remove sulphur from ultrafine coal wastes has been established, further experimental work is required to optimize the reagent addition. It should also not be forgotten that all of the research work described in this paper was conducted on only one thickener underflow coal sample. Owing to the variable nature of coal, the results may apply only to the specific sample tested. Consequently, the study is being extended to coal ultrafine wastes from other South African coalfields.

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