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Two-Stage Fast Flotation of Coal Tailings using Reflux Flotation

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Abstract

Low pulp density and low grade slurries in the coal and minerals industries are discharged as waste to tailings dams, incurring significant losses of valuable particles. This paper investigates the rapid processing and cleaning of hydrocyclone overflow coal slurry using two laboratory scale Reflux Flotation Cells in series as a means to economically beneficiate low quality tailings streams. The Reflux Flotation Cell incorporates a novel arrangement of inclined channels to enhance bubble-liquid segregation, enabling extremely high gas rates and liquid rates per unit of vessel area. Hence, in the first stage, fast flotation is employed to rapidly recover fine coal particles using a feed flux of 11.4 ± 0.5 cm/s, up to an order of magnitude increase in the throughput rate over conventional flotation systems. First stage product was then sent to a second stage for counter-current washing using fluidisation wash water to produce a fully deslimed product, having ash percent in agreement with the minimum ash attainable using flotation as determined through tree flotation analysis. The results demonstrate the potential for two-stage Reflux Flotation to deliver high throughput at a high separation efficiency from low quality slurry, with a five-fold reduction in the required vessel footprint, thus overcoming the principal economic deterrent of having to install banks of large-scale flotation cells.

Keywords: fast flotation, desliming flotation, coal tailing, inclined channels, Reflux Flotation Cell

1 Introduction

In the coal and minerals processing industries, low quality streams of low pulp density or low grade particles are commonly discarded to tailings dams once thickened through sedimentation. In the coal industry, fine clays and mineral particles, or “slimes”, often accompany fine coal processing in high quantity, giving rise to high levels of unwanted “ash” content. Thus, prior to the 1970s, fine coal slurry was commonly treated as a waste due to the difficulty in separating coal from the slimes using available technology, resulting in major losses to tailing dams. However, improved system design, environmental standards and economical return have progressed contemporary coal preparation plants to routinely utilise flotation as the core technique for fine coal recovery. Yet, for similar reasons, losses of coal to tailing dams still occur, albeit at a lower particle size. For instance, a desliming hydrocyclone is routinely used prior to the fine coal gravity circuit to remove ultrafine slimes, below 100 microns, resulting in a loss of coal at around 50% ash.

Since the 1930's, the volume capacity of flotation cells has increased exponentially from 1 m³ to 300 m³ (Degner, 1988; Euston, 2010; Lynch et al., 2007, 2010; Wills and Finch, 2016), with cell volumes in the minerals industry now exceeding 600 m³ (Camomile et al., 2013). This trend, illustrated in Figure 1, has been driven by the need for improved economies of scale, and to process lower grade ores at a higher throughput, but is likely nearing an upper limit of scale up. While the coal industry has implemented multi-staged flotation operations (Jameson et al., 1991; Lambert and Revell, 2008; Wex

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et al., 2004), the industry has seldom found an economical argument to support the use of the larger cell capacities commonly utilised in minerals flotation (Wieniawski et al., 2010). Hence, alternative low cost options for fine particle recovery are required.

Figure 1 Growth in mechanical cell volume from 1970 to 2013, plotted using data sourced from Lynch et al. (2007) and Camomile et al. (2013).

This paper provides a way forward for industry to beneficiate low grade or low pulp density tailings, by gaining an economical advantage through a five-fold reduction in the required system footprint. A two-stage rapid flotation operation is proposed consisting of (1) first stage fast flotation and (2) second stage desliming flotation, using novel Reflux Flotation Cell (RFC) technology. This study brings these two independent directions of our recent work into one for the very first time.

Fast flotation is effectively a single stage rougher approach aimed at maximising throughput and particle recovery using up to a ten-fold increase in the conventional volumetric feed flux. A key challenge is in being able to reject the bulk of the liquid flow passing to the product when operating under flooding conditions induced by the extreme operating rates. The cell residence time during fast flotation is typically less than 30 s, about 1/10th the time of a conventional flotation cell treating an equivalent volumetric rate of feed (Dickinson et al., 2015).

Desliming flotation utilises an inverted fluidised bed approach to apply fluidisation wash water to deslime the flotation concentrate free of entrained gangue particles (Dickinson and Galvin, 2014). It is hypothesised that the sizable volumetric reduction of the flotation concentrate achieved during fast flotation should permit desliming flotation at a more conventional rate of feed using a single unit to obtain a high grade (low ash), high recovery concentrate .

Figure 2 Schematic representation of the first stage Reflux Flotation Cell rougher.

Figure 3 Schematic representation of the second stage Reflux Flotation Cell cleaner.

Our first investigation into fast flotation using the RFC involved a gas-liquid hydrodynamic study, absent of particles, and demonstrated desirable bubble-liquid segregation under flooding conditions using fine bubbles and feed fluxes of up to 15 cm/s, as well as achieving an order of magnitude increase in the bubble surface area flux, up to 600 m²/m²s, (Jiang et al., 2014). Conventional flotation systems comparatively operate with a maximum bubble surface area flux of 60 to 80 m²/m²s (Massinaei et al, 2009; Wace et al, 1968) in order to avoid the flooding condition and excessive gangue entrainment. Later work confirmed fast flotation capable of providing a sufficient rate of flotation of fine particles in a very short cell residence time of about 25 s using an extremely low feed pulp density of 0.35 wt% (Dickinson et al., 2015), about a tenth of the feed pulp density examined in this study. Overall combustible recoveries from 70 to 80% were obtained, with a near complete recovery of the +38 µm particles, up to 98.5%. High levels of slimes entrainment contributed to a notable rise in product ash content, from 18% to nearly 50% as the gas flux increased from 2 cm/s up to 6.1 cm/s. Hence, secondary processing was essential to reject the entrained slimes in the concentrate. In a separate investigation, fluidisation wash water has been shown to produce effective counter-current washing in flotation (Dickinson and Galvin, 2014; Galvin and Dickinson, 2014; Galvin et al., 2014). Separations achieved in these desliming flotation studies have often bettered the “ideal” recovery-ash response curve for flotation, as determined by the Tree Flotation Method (Australian Standard AS 4156.2.2).

A novel feature of the RFC is the system of parallel inclined channels located below the vertical column. The laboratory scale Reflux Flotation Cells used in this study are shown schematically in Figures 2 and 3. The inclined channels are utilised to enhance the segregation rate between the rising bubbles and

descending liquid. Analogous applications of the Boycott effect (Boycott, 1920) are found to increase the throughput advantage in particle-liquid segregation in a lamellar thickener (Yao, 1973), enhance particle-particle segregation in the Reflux Classifier (Galvin et al., 2009), and increase the drainage rate of liquid in pneumatic foam (Dickinson et al., 2010). In the RFC, bubbles entering the channels with the net downward flow of liquid rise to the top of the channels due to an effective increase in the vessel cross-sectional area, and return to the bubble suspension above. The refluxing of the bubbles prevent losses to tailings, and permits a controlled level of liquid to report with the product to the overflow, even while the system is flooded. Thus, extremely high gas fluxes and liquid fluxes, imposed by the liquid feed flux or the wash water flux, are permitted in the RFC.

This laboratory scale study investigates two-stage Reflux Flotation using as-received industrial coal slurry sampled from hydrocyclone overflow. The objective was to achieve a low ash, final coal product from low pulp density slurry that industry currently discards, using a very high rate of feed. Hence, fast flotation was applied in the first stage to rapidly recover the fine coal. Rougher product was then fed to a second RFC where fluidisation wash water was applied to obtain a low ash final coal product.

2 Experimental

The two laboratory scale RFC units used are illustrated in Figures 2 and 3. They differed in two ways. Firstly, the rougher unit shown in Figure 2 has a multi-channelled downcomer arrangement, while a sleeve and frit downcomer arrangement was used in the desliming RFC shown in Figure 3. Secondly, the top of the desliming RFC was enclosed by a fluidising chamber used to evenly disperse the wash water into the cell, while in the fast flotation experiments no wash water was applied, and an overflow launder surrounding the top of the rougher RFC sufficed.

The first stage RFC had five inclined plates, forming six parallel inclined channels, set to an angle of inclination of 70° to the horizontal. The inclined channels were 925 mm long, and had a perpendicular channel spacing of 20 mm. The vertical section above the channels was 960 mm in length, with cross sectional dimensions 146 mm x 146 mm. The internal cross-sectional area was 130.5 cm^2 , excluding the area occupied by the downcomer. Fluxes reported are calculated based on this area. A two channelled, rectangular downcomer was used. The dimensions of the downcomer were 1140 mm in length, 127 mm width, and 55 mm breadth. The spacing of each channel was 4.5 mm. An acrylic plate dividing the two channels had two porous sintered stainless steel plates of nominal pore size $2 \mu\text{m}$, fitted flush on either side. Feed slurry pumped to the head tank above the downcomer allowed for an even split of the feed between each channel. Compressed air was supplied to the downcomer, permitting fine bubbles to be sheared off the sintered sparger surface by the flow of feed. A high liquid shear rate generated by the slurry flowing through the narrow channels within the downcomer is ideal for enhancing particle-bubble collision, as well as producing very fine bubbles for improved flotation kinetics.

The second stage desliming RFC had dimensions $80 \text{ mm} \times 100 \text{ mm}$. In the bottom half of the system, eight channels, 1 m in length, were formed from 1 mm stainless steel plate, at an angle of inclination of 70° to the horizontal. The perpendicular spacing of the inclined channels was 9.3 mm. The vertical vessel above the channels was 1 m long, and the internal cross-sectional area 72 cm^2 , excluding the downcomer area. The top of the system was enclosed by the fluidisation water distributor that functioned as a manifold for the wash water to pass through a grid of 1.5 mm diameter holes. A sleeve and sparger downcomer was used that had an annulus width of 4.8 mm. The nominal pore size of

sintered steel sparger was 10 μm . Product was discharged from the RFC through an outlet above the fluidisation chamber.

Coal slurry collected from the hydrocyclone overflow of a coal preparation plant located in the Hunter Valley, Australia was used as the flotation feed. Figure 4 illustrates the experimental procedure used in the two-stage experiments. The flotation feed was prepared from a bulk supply of 4 m^3 stored in four 1.04 m^3 intermediate bulk containers (IBC). Each IBC was sequentially pumped to a 1.4 m^3 agitated tank for conditioning, and then into a 2 m^3 agitated tank which functioned as the main feed tank. Prior to the transfer, the feed in the IBC was recirculated in order to re-suspend settled particles. A minor amount of municipal water was used to hose any remaining solids out of the IBC. Collector was added and conditioned for 10 min. Frother addition then followed for a further 10 min of conditioning time. The reagents used were diesel at a concentration of 2.0 kg/t of solids and MIBC at 40 ppm. It is noted that there was no attempt made in this study to optimise the reagent doses. It is noted however that collector dose should be increased in direct proportion to the total surface area of the recoverable hydrophobic particles, so for exceedingly fine tailings the higher collector dose may be warranted. During the runs, the slurry level in the feed tank was maintained using the conditioned feed in the conditioning tank. A Mono-pump was used to pump feed to the RFC rougher, while compressed air was supplied to the downcomer and controlled using a rotameter.

Four fast flotation were conducted (Runs 1, 2, 3A and 3B), two of which are repeats using a different tailings rate (3A and 3B). A manual ball valve at the bottom of the RFC was used to set the tailings discharge rate and, in effect, the liquid rate reporting to the overflow. Opening the valve allowed a greater flow rate of liquid to the tailings and entrainment of bubbles into the inclined channels. Due to the limited quantity of feed available and the high throughput rate used, the ball valve was quickly adjusted at the start of each run such that the bubbles entered ideally to a depth of between one-quarter to one-third the way into the inclined channels. After 2 to 3 min of steady operation, full stream samples of the overflow product were collected into 20 L buckets for later processing using desliming flotation. Following this, timed samples of the product and tailings were simultaneously collected into 20 L buckets, immediately followed by a feed sample. The tailings solids mass rate was given by the average of the two tailings samples, and the fractional ashes determined from a combined sample. Replication of each run using specific operating conditions, such as gas flux and liquid split, was not examined considering that the processing of 1 m^3 of slurry at laboratory scale permitted only about 10 minutes run time using fast flotation. Therefore collecting a sufficient volume of first stage concentrate for second stage processing for three two-stage trials required a significant volume of feed despite being laboratory scale experiments. Thus, the experimental conditions were chosen to evaluate the response of the system over a wide range of gas fluxes rather than examining repeatability at a single operating condition.

Figure 4 Schematic representation of the laboratory two-stage fast flotation process. Fast flotation product was collected in buckets to be later processed using desliming flotation.

Three desliming flotation runs were conducted (Runs 4, 5 and 6) using fast flotation product added to a 0.12 m³ mixing feed tank for second stage processing. No further collector or frother was added to the second stage feed. Wash water at 40 ppm MIBC was prepared in a 0.9 m³ tank using municipal water. Product and tailings samples were simultaneously collected after about 20 min of steady operation, followed by a feed sample.

Mass balance reconciliation was performed using the generalized least squares minimization method for each flotation stage to minimize the presence of random measurement errors. The method assumes the measured values to be unbiased, uncorrelated to the other measured values and of normal distribution. A weighting is specified for the random error associated with each measurement, then the sum of the squares of the relative errors is minimized, subject to the constraint that all mass balance equations are satisfied to a convergence criterion. Four measured values were used in the mass balance reconciliation; fractional size distribution, fractional ash, stream flow rates (including fluidisation water) and pulp density. The weighting on the relative error associated with the flow rate, pulp density and mass fraction was taken as 10% standard deviation of the measured value. A higher precision of 1% standard deviation was placed on the fractional ash values which were reported with a repeatability of $\pm 0.1\%$ of the absolute value, according to the Australian Standard AS 2434.8 – 2002. These values changed little from their reported values, apart from the few noted below having a relaxed weighting. The reconciled data are reported, with the yield, cumulative yield, combustible recovery and cumulative combustible recovery for the first stage, second stage and the overall two-stage separation were all calculated on an ash basis using the n-product formula. A sensitivity analyses on the n-product formula involved determining the propagation of error using a 0.1% standard deviation on the ash assays.

Samples were wet screened at 38 μm , dried and sieved in our laboratory, before being sent for external ash analysis. The process of wet screening must be undertaken with considerable care to insure quality results. A feed sample was sent for tree flotation analysis as per the Australian Standard 4156.2.2 - 1998(1). Unfortunately, for Runs 1, 2, 3B and 5 the mass of tailings sampled in the size fractions -125 + 90 μm and +125 μm were too small to perform repeated assays. Hence, these samples were combined

to obtain a single but precise ash value across the wider size fraction. Similarly, for Runs 4 and 6, less than 5% of the particles in the tailings sample were above 45 μm . Hence, the five size fractions spanning from +38 μm up to the upper nominal fractional size range of +125 μm were combined to a single ash assay. For these combined tailings size fractions, the weighting given to the precision of the ash values in the mass balance reconciliation were relaxed to 10% standard deviation of the experimental value.

3 Results and Discussion

3.1 First Stage Fast Flotation

Fast flotation runs were conducted using three gas fluxes, 2.0 cm/s, 4.1 cm/s and 6.1 cm/s, and an extremely high liquid feed flux of 11.4 ± 0.5 cm/s, around ten times higher than the standard flux to conventional flotation systems. These high gas and liquid fluxes induced the system to flood, forcing the need for the bubbly mixture to segregate within the inclined channels. The cell residence time was approximately 20 to 25 s. Figure 5 summarises the feed, showing the majority of the particles were well below 125 μm , and the finest size fraction, -38 μm , accounting for $81.5 \pm 1.3\%$ of the mass. This fraction also contained the highest ash content at 58%, and is responsible for the high head ash of around 49% shown in Figure 6. Particles above 38 μm had a cumulative ash of $8.7 \pm 0.7\%$. The mean feed pulp density was 3.1 ± 0.8 wt%.

Figure 5 Cumulative size distribution of the feed plotted versus increasing particle fractional size.

Figure 6 Cumulative ash in the feed plotted versus decreasing particle fractional size.

A summary of the fast flotation experimental conditions and separations is given in Table 1. Considering the F80 (the size at which 80% of the feed is finer) is shown in Figure 5 to be below 38 μm , and that flotation recovery is known to rapidly decline when floating very fine particle sizes, reasonable recoveries spanning 64 to 87% were obtained in a very short residence time. When starting each experiment, the high feed rate granted little time to be too pedantic in manually setting the underflow rate without squandering too much feed. This in turn set the overflow liquid flux for each run. The ratios of the overflow liquid flux to feed liquid flux, referred to as the liquid split in Table 1, are therefore not necessarily reflective of optimal run conditions, but do provide a range of results for interpretation.

The liquid split represents the effectiveness of the system to function without excessive liquid carryover when the system is flooded, which occurred in all runs. In a flooded system, a stable froth zone no longer remains, however the inclined channels in the Reflux Flotation Cell provide a further degree of freedom to control the liquid flux reporting to the overflow with the bubbles. For Run 2 a very moderate liquid split of 16% was obtained using a high gas flux of 4.1 cm/s, while contrary to conventional practice, the use of a lower gas flux of 2.0 cm/s in Run 1 obtained a higher liquid split of 33%. This result illustrates the departure from the fundamental relationship between the gas flux and the square of the bubble radius governing the interstitial liquid holdup in the rising froth of a conventional flotation system (Grassia et al., 2001; Leonard and Lemlich, 1965; Miles et al., 1945). A higher underflow rate could have been applied in Run 1 to lower the liquid split to perhaps 10%. Product from Run 1 was not sent to desliming flotation.

Table 1 Fast flotation experimental conditions and separations.

Run	Gas Flux (cm/s)	Feed Flux (kg/m ² s)	Feed Pulp Density (wt%)	Product Pulp Density (wt%)	Feed Ash (%)	Product Ash (%)	Reject Ash (%)	Liquid Split (%)	Yield (%)	Combustible Recovery (%)
1	2.0	120	2.6	3.8	49.6	28.2	70.7	33	49.8	70.9
2	4.1	119	2.8	7.3	49.5	18.2	75.5	16	45.4	73.5
3A	6.1	117	3.3	6.8	48.4	26.6	66.4	21	45.1	64.2
3B	6.1	113	3.6	5.8	45.2	29.5	78.4	42	68.0	87.4

Run 3A was repeated because of the relatively low 66% recovery obtained despite the high gas flux of 6.1 cm/s. Here, extremely fine bubbles, perhaps less than 100 μm , were suspected to have escaped with the high underflow liquid flux, therefore resulting in a loss in recovery. Thus, Run 3B is a repeat of Run 3A using a higher liquid overflow flux, which doubled the liquid split to 42% and increased the recovery to 87%.

The consequence of a higher liquid split is a higher entrainment of slimes. Yet, the rejection of fine slimes appears to be well defined with variation in liquid split. Previous work by Dickinson et al (2015) found that the partitioning of particles less than 1.65 μm to product followed one-to-one with liquid split values of 17% and 33%, and the 50% cut point for both liquid splits was 8 μm for particles less than 38 μm . Figure 7 examines the ash rejection across the entire feed particle size range as a function of the liquid split. Ash rejection represents the percentage of the ash present in the feed that reports to the reject, and is calculated on an ash% basis. Included in this figure is data sourced from Dickinson et al (2015), obtained from processing hydrocyclone overflow at a diluted pulp density of 0.35wt% using similar conditions. For the processing of fine, well liberated particles, the two data sets in Figure 7 illustrate how the amount of ash rejection can be generalised in terms of the liquid split, again reflecting a near one-to-one relationship driven by the hydrodynamics in the system. Thus, every 1% increase in the liquid split approximately delivers a 1% reduction in the ash rejection while flooded.

Figure 7 Ash rejection as a function of liquid split. Data for the 0.35 wt% feed pulp density was sourced from Dickinson et al (2015).

The recovery by particle size for Runs 2, 3A and 3B are plotted in Figure 8 with 95% confidence intervals. In general, recovery tended to increase with particle size for each run. For a gas flux of 4.1 cm/s in Run 2, recovery of the intermediate sized particles remained steady at 83%, increased to over 92% for particles greater than 90 μm , and decreased to 68% for particles below 38 μm . Table 2 provides a closer examination of the separation obtain for Run 2, showing a cumulative 75% ash in the reject, up from 49% in the original feed. With an acceptable liquid split of 16% and high recovery, Run 2 presents the best example from the set of fast flotation runs of promoting high bubble collection efficiency through a high bubble concentration, while achieving efficient bubble-liquid segregation under flooding conditions. Increasing the gas flux to 6.1 cm/s in Run 3B resulted in notably higher recoveries by particle size, outside the limits of uncertainty, but incurred a poor liquid split of 42%. Conversely, Run 3A shows how a too extreme reduction in the liquid split relative to the (same) gas flux, can lead to lower recoveries as bubbles become entrained to the reject.

Figure 8 Recovery by fractional particle size using fast flotation. Error bars indicating 95% confidence intervals are obscured by the symbols.

Table 2 Fast flotation separation data for Run 2.

Fractional Size (μm)	Fast Flotation Product				Fast Flotation Reject				Fast Flotation Separation			
	Fractional (%)		Cumulative (%)		Fractional (%)		Cumulative (%)		Fractional (%)		Cumulative (%)	
	Mass	Ash	Mass	Ash	Mass	Ash	Mass	Ash	Yield	Recovery	Yield	Recovery
+125	1.9	3.6	1.9	3.6	0.1	6.4	0.1	6.4	94.6	94.8	94.6	94.8
-125 +90	5.8	3.9	7.8	3.8	0.4	8.1	0.5	7.8	92.0	92.3	92.6	92.9
-90 +63	13.0	6.4	20.8	5.4	2.4	13.9	2.9	12.8	82.1	83.3	85.7	86.6
-63 +45	9.1	8.2	29.9	6.3	2.2	27.8	5.1	19.2	77.6	81.5	83.0	85.0
-45 +38	3.1	8.6	33.0	6.5	0.7	40.4	5.7	21.7	79.2	85.4	82.7	85.1
0 -38	67.0	23.9	100.0	18.2	94.3	78.8	100.0	75.5	37.1	68.0	45.4	73.5

The results above demonstrate the important role of process control in undertaking fast flotation. Regarding possible process control options for a full scale operation, it is anticipated that the freedom to control the liquid overflow rate relatively independently of the gas flux may provide a unique control variable for effective regulatory control to enable a steady operation of the Reflux Flotation Cell. Traditionally, to ensure stable and efficient flotation performance, the key control variable is to control

the pulp level in the cell in order to control the froth depth (Wills and Finch, 2016). The liquid flux reporting to the overflow is then governed by complex hydrodynamics within the froth. However, in the case of the Reflux Flotation Cell, the overflow liquid rate can simply be controlled by controlling the differential in the feed flow rate and the underflow flow rate in order to operate with a specified liquid split. Fluidisation water rate can also be included as a control variable in controlling the overflow liquid rate. Fluctuations in the net downward liquid flux or adjustments in the gas flux should readily be received by the inclined channels without a loss of separation performance provided the channels, and therefore the system, are not operating at full capacity. Such tolerance has been observed at a laboratory scale with the bubbly front in the inclined channels showing only slight fluctuations from a mean location. Similar observations have been made during a recent pilot scale trial involving desliming flotation. Pressure probes may too prove useful in monitoring the depth of the bubbly suspension in the system.

3.2 Second Stage Desliming Flotation

Fast flotation product collected from Runs 2, 3A and 3B was deslimed using the second RFC shown schematically in Figure 3. Table 3 summarises the run parameters, the run number corresponding to the first stage product in Table 2 from which the feed was sourced, and the separations. A gas flux of 2.6 cm/s was used in Run 4, and 3.2 cm/s in Runs 5 and 6.

Table 3 Summary of the desliming flotation runs.

Run	Feed Source	Gas flux (cm/s)	Feed Flux (kg/m ² s)	Wash Flux (cm/s)	Product Pulp Density (wt%)	Product Ash (%)	Reject Ash (%)	Liquid Split (%)	Yield (%)	Combustible Recovery (%)
4	2	2.6	4.8	0.7	9.7	7.7	79.3	49	79.1	94.4
5	3A	3.2	4.1	0.5	10.3	7.9	73.1	38	69.5	88.7
6	3B	3.2	5.1	0.5	7.6	7.7	80.7	43	69.8	91.7

The results in Table 3 show desliming flotation to be very effective. Uniform product ashes of $7.8 \pm 0.4\%$ were achieved from the three runs, and high combustible recoveries of between 89 and 94%. Previous desliming flotation investigations have demonstrated how fluidisation water imposes a strong positive bias flux permeating through a bed of bubbles of high bubble volume fraction (Dickinson and Galvin, 2014). The fluidisation wash water flows downward against the rising bubbles to impart a one-to-one translation in the bias flux to a positive value (net downward flow of liquid). Hence, the fluidisation wash water flux of 0.7 cm/s in Run 4, and 0.5 cm/s in Runs 5 and 6 imposed a positive bias flux of 0.5 cm/s for Run 4, and 0.3 cm/s for Runs 5 and 6.

The challenge of rejecting the slimes from low grade hydrocyclone overflow was partially accomplished in the first stage, as seen in Figure 7. The particle size distribution of the first stage concentrate therefore differed from the original feed. Figure 9 is a plot of the second stage feed (first stage concentrate), showing a F80 of 63 μm , suitably higher than the F80 in Figure 5. The cumulative ash corresponding to Figure 9 is presented in Figure 10, and shows 1) an approximate halving in the total cumulative ash, 2) the variation in the total cumulative ash value dependent on the -38 μm size fraction, and 3) a significant lowering of the ash % of the +38 μm size fraction over the original feed, from $8.7 \pm 0.7\%$ down to $6.6 \pm 0.7\%$.

Figure 9 Cumulative size distribution of the second stage feed.

Figure 10 Cumulative ash in the second stage feed plotted versus decreasing particle fractional size.

The principal objective of single-stage desliming flotation is clearly to accomplish a high rejection of gangue material. However, when using a once through approach, it is equally important to maintain high recoveries across the particle size range. The recoveries by particle size from the three desliming flotation runs are shown in Figure 11. Near identical performances were obtained from Runs 4 and 6, with recoveries greater than 90%. The lower recoveries for Run 5 are thought to have resulted from using a lower feed rate, about 20% below the rate used in Runs 4 and 6. When the feed rate is too low, less effective bubble generation may occur, producing some over-size bubbles which may lead to lower bubble-particle collision rates.

A closer examination of the separation for Run 4 is given in Table 4. The feed for this run corresponds to the fast flotation product in Table 2. Table 4 shows recovery increasing from 93% up to 99% with increasing particle fractional size, up to the nominal top size of 125 μm . Coinciding with this trend is a decrease in the fractional product ash, from a low 8.8% for particles below 38 μm to a very low 3.1% ash at the nominal size of +125 μm . Importantly, only the -38 μm size fraction experienced a significant but necessary drop in yield as a result of fluidisation wash water, reducing the fractional ash from 23.9% down to a low 8.8%, resulting in an overall product ash of 7.7%. Only 6% of the +38 micron particles exited the systems via the reject with a cumulative ash of 24.8%, comparing well with the 5.5% product ash over the same size range. Hence, high selectivity and recovery has been successfully demonstrated using single stage desliming flotation.

Figure 11 Recovery by fractional particle size using desliming flotation. Error bars indicating 95% confidence intervals are obscured by the symbols.

Table 4 Desliming flotation separation data for Run 4.

Fractional Size (μm)	Desliming Flotation Product				Desliming Flotation Reject				Desliming Flotation Separation			
	Fractional (%)		Cumulative (%)		Fractional (%)		Cumulative (%)		Fractional (%)		Cumulative (%)	
	Mass	Ash	Mass	Ash	Mass	Ash	Mass	Ash	Yield	Recovery	Yield	Recovery
+125	2.0	3.1	2.0	3.1	0.1	22.3	0.1	22.3	98.6	98.9	98.6	98.9
-125 +90	5.9	3.5	7.9	3.4	0.5	17.0	0.6	18.0	98.0	98.3	98.2	98.4
-90 +63	14.3	5.3	22.2	4.6	1.6	22.3	2.2	21.2	97.1	97.6	97.5	97.9
-63 +45	9.2	6.8	31.4	5.2	2.6	26.7	4.8	24.2	93.1	94.5	96.2	96.9
-45 +38	3.3	7.6	34.7	5.5	1.4	26.8	6.2	24.8	89.8	91.7	95.5	96.4
0 -38	65.3	8.8	100.0	7.7	93.8	82.9	100.0	79.3	72.5	93.4	79.1	94.4

3.3 Separation Performance of Two-Stage Reflux Flotation

This next section provides an overview of the combined separation performance of fast flotation and desliming flotation in processing hydrocyclone waste. A column graph of the mean cumulative ash of

the feed, first stage product and second stage product are given in Figure 12, showing the significant reduction in ash percent, well beyond the uncertainty indicated by the 95% confidence intervals. The high uncertainty in the first stage product ash is due to the hydraulic entrainment of slimes, shown in Figure 7 to be highly dependent on the liquid split that varied greatly in this set of experiments. Yet, the effective application of fluidisation wash water in the second stage ensured that the final cumulative product ash of each run remained insensitive to the first stage product ash. Thus, this series of experiments has demonstrated excellent robustness to reject gauge material, particularly the very fine slimes. A similar trend in the comparative uncertainty between fast flotation recovery and desliming flotation recovery is evident in Figure 13, and again is reflective of the influence of the hydrodynamics on performance. Here, the uncertainty in the two-stage recovery is magnified by the uncertainty of each stage. Thus, there is a real need for precise process automation to maintain a constant recovery.

Figure 12 Mean cumulative ash of the initial feed, first stage product from Runs 2, 3A and 3B, and second stage product from Runs 4, 5 and 6. The error bars indicate 95% confidence intervals.

Figure 13 Mean cumulative combustible recoveries from first stage fast flotation (Runs 2, 3A and 3B) and second stage desliming flotation (Runs 4, 5 and 6), and the two-stage separation. The error bars indicate 95% confidence intervals.

Ideally, one would expect the hydrophobic particles floated in the first stage to be readily recovered in the second stage provided the hydrodynamics underpinning the kinetics and the selectivity are preserved across each system. A slight loss in the overall recovery is therefore expected in the second stage due to the low probability of floating ultrafine hydrophobic particles, a portion of which would have been entrained with first stage product. The two-stage separation for the sequence Run 2 and Run 4 is given in Table 5, and shows very respectable recoveries across all size fractions, with the lowest recovery at 65% for the finest size fraction containing difficult to float, ultrafine coal particles.

Table 5 Two-stage separation from Run sequence 2 and 4.

Fractional Size (μm)	Two-Stage Separation					
	Fractional (%)			Cumulative (%)		
	Ash	Yield	Recovery	Ash	Yield	Recovery
+125	3.1	92.3	93.0	3.1	92.3	93.0
-125 +90	3.6	88.1	88.7	3.4	89.0	89.7
-90 +63	5.3	75.6	77.6	4.6	81.0	82.5
-63 +45	6.8	72.3	77.0	5.3	78.7	81.4
-45 +38	7.9	75.8	82.4	5.6	78.7	81.8
0 -38	8.8	29.9	65.3	7.7	39.2	71.3

The recoveries by size for the three sequences of runs are compared in Figure 14, showing even higher recoveries from the Run 3B and Run 6 sequence across most size fractions. Inspecting the relative contribution of the sources of error, it was found the tailings samples to be the major contributor to the high uncertainty for the two coarser size fractions, contributing over 90% to the uncertainty. Hence, the magnitude of the uncertainty in two-stage fast flotation recovery could be made smaller by taking repeated samples of the fast flotation reject.

Figure 14 Two-stage recoveries by fractional particle size. Error bars indicate 95% confidence intervals.

In order to benchmark the two-stage separation, a feed sample was sent to an external laboratory for tree flotation analysis to determine the “ideal” separation obtainable using flotation for this particular feed. The resultant tree curve is presented in Figure 15, together with the first and second stage recoveries and the overall two-stage recovery, as a function of product ash percent. These are colour coded to match each sequence of runs. The two-stage recoveries are shown clearly placed above the tree curve, however, one would expect a smoother interpolation of the tree curve given additional tree data, with the knee of the tree curve likely to align in the vicinity of the two-stage Reflux Flotation separation, arguably around 70% recovery, 10% ash.

The three two-stage separations are in excellent agreement with the minimum product ash of 7.9% identified by the tree flotation analysis, demonstrating good reproducibility for two-stage Reflux Flotation to achieve complete rejection of hydrophilic slimes over a range of operating conditions. Confidence in these results is drawn by the agreement in the minimum ash between the two independent experimental methods, and indicates negligible systematic error or bias occurring in the fractional and cumulative ash data. Such high slimes rejection has been observed in previous laboratory desliming flotation investigations involving a single-stage RFC (Dickinson and Galvin, 2014; Galvin et al., 2014), and more recently in single-stage pilot trials of desliming flotation.

Flotation of hydrocyclone overflow in past investigations using conventional flotation columns has tended to produce a separation below and to the right of the tree curve due to inadequate slimes rejection and full collection of the combustible particles, while application of wash water has shown only limited success in desliming (Bensley et al, 1988). This inefficiency, combined with the low processing rate of conventional flotation systems relative to the large volumetric rate required, have prohibited industry from investing into processing such waste streams. Two-stage Reflux Flotation however, offers a solution that addresses these shortcomings.

Figure 15 First stage fast flotation separations (squares) and the overall two-stage separations (circles) compared to the “ideal” tree flotation curve. The three shades of symbols align each first stage result to its corresponding two-stage separation. Uncertainty is given as 95% confidence intervals.

A method for assessing separation efficiency, E , is to calculate the recovery of the valuable material in a given product minus the recovery of the invaluable material in the same product (Schulz, 1970). Thus,

in assessing coal separation efficiency, $E = R_c - (100 - A)$, where R_c is the percent recovery of combustibles in the product, and A is the percent ash rejection to reject, equal to 100 minus the percent recovery of gangue in the product, R_g . The separation efficiencies for each flotation stage, and for the two-stage separation, are plotted in Figure 16. The diagonal lines defined by this efficiency equation represent a continuum of possible separations for each efficiency value, with $E = 0\%$ indicating no selective separation. The exception is the single point for $E = 100\%$ located in the top right hand corner that represents perfect separation in terms of both recovery and selectivity of a completely liberated feed. Included in the figure is the “ideal” separation efficiency curve given by tree flotation analysis, and indicates the best efficiency obtainable in a single flotation stage from the coal feed used is 62% at the knee of the curve, with a recovery of 75%. Higher recoveries then trended towards lower E values.

The fast flotation performance data plotted in Figure 16 indicates that Run 2 achieved the superior efficiency, not only in having the highest separation efficiency of 57%, but also in obtaining close proximity to the knee of the tree flotation E curve. The excellent performance of this result is not surprising given Run 2 favourably balanced a high gas flux of 4.1 cm/s and a low liquid split of 16%. The other three fast flotation runs achieved a lower efficiency of about 42%, yet the high recovery in Run 3B achieved a similar high precision as Run 2 relative to the tree flotation E curve. Therefore, better-quality process control of fast flotation should foresee separation efficiencies clustering in closer proximity to the characteristic curve produced by tree flotation analysis. Figure 16 also reveals comparable levels of ash rejection by the two flotation processes over the range of 72 to 83%, signifying how the rapid concentration of fine values from poor quality feeds using fast flotation significantly diminished the burden placed on second stage desliming flotation to produce a clean final product. Overall, the two-stage separation efficiencies, shown in Figure 16, ranked superior to the tree flotation E curve. The highest overall separation efficiency was obtained for the sequence Run 3B and Run 6, suggesting processes that preference higher recovery over process efficiency may benefit from using extreme operating conditions in the first stage of separation.

Figure 16 Separation efficiency, E , as a function of recovery and ash rejection. The shaded symbols align each result to its corresponding two-stage separation.

Two-stage Reflux Flotation has successfully recovered valuable particles from low pulp density waste streams. It is noted however, in the experiment the second stage feed flux was lower than first stage overflow flux, and ideally should have been equal in value to enact continuous flow from one stage to the next. For instance, the liquid overflow flux in Run 2 was 1.9 cm/s, four times higher than the second stage feed flux in Run 4, while for Runs 3B and 6 the liquid overflow flux was 5 times higher at 2.4 cm/s. On balance, the RFC has demonstrated considerable scope to increase the liquid flux considerably during desliming flotation without suffering a loss in separation efficiency.

3.4 Benefits Arising from Two-Stage Reflux Flotation of Coal Tailings

Prior to this study, our investigations involving fast flotation and desliming flotation have followed two independent directions; however, our intention has always been to couple these into a two stage operation in order to deliver an economical solution to processing low quality industrial waste streams.

Across New South Wales (NSW), Australia, operating coal mines produced 196.7 Mt of saleable coal at an average yield of 77.7% by end of financial year 2015 (DIRE, 2015). The industry therefore disposes of approximately 56.5 Mtpa of waste material, including more than 16.1 Mtpa of fine tailings containing up to 8 Mtpa of well liberated ultrafine coal. The recovery of this coal is currently deemed uneconomical. Hence, about 4% of the mined coal is discarded to waste in NSW, worth about \$400M pa, assuming AUD \$50/tonne.

Further benefits of coal beneficiation from tailings include a potential halving in the volume of fine coal tailings, a reduction in water consumption due to a reduction in the solids discharge rate to tailings, and a higher overall plant performance. The challenge in beneficiating values from waste streams however, has long been that traditional technology requires significant capital investment, high energy consumption, and carries significant risk with achieving the necessary level of cleaning to produce a low ash product.

The beneficiation of fine coal from hydrocyclone overflow at a typical coal handling and preparation plant would require processing 2000 m³/h of slurry containing about 5% solids, at 50 to 70% ash. With a residence time of less than 30 s, a single 3 m diameter, 3 m tall Reflux Flotation Cell is expected to process the 2000 m³/h of slurry using fast flotation. The first stage rougher product would then be deslimed using a second cell of similar dimensions. Desliming flotation using a Reflux Flotation Cell RFC300 has recently undergone successful pilot scale trials, while fast flotation pilot scale trials are underway. The RFC300 pilot unit had a height of 3 m and cross sectional dimensions of 0.3 m by 0.3 m. Further scaling up to an industrial sized unit requires projection of the system's footprint to accommodate the higher volumetric rates of throughput, and no further scaling of the system's height. Findings from these pilot trials are to be published in due course.

4 Conclusions

This study has demonstrated significant potential for Reflux Flotation to recover low ash, fine coal particles from low pulp density feeds, currently discarded as waste by industry, using a two-stage combination of fast flotation and desliming flotation. An overall superior separation relative to the tree flotation curve was obtained by the two-stage process achieving a fully deslimed product of 7.8% ash, and combustible recoveries of up to 80%. Fast flotation permitted a throughput rate an order of magnitude higher than the typical conventional flotation rate. Thus, a single full scale Reflux Flotation Cell is anticipated to provide an economical solution for industry to effectively replace ten flotation cells with a single unit, to rapidly recover valuable particles into a slurry concentrate. One additional

unit should then be sufficient to fully clean the concentrate free of unwanted hydrophilic gangue particles.

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6 References

Bensley, C.N., Roberts, T., Nicol, S.K. and Lamb, R. 1988. Column flotation: the development of the tower flotation cell. Proc. in Fourth Australian Coal Preparation Conference, Ed. Holtham, P., Paper 4A, 87-103.

Boycott, A. E., 1920. Sedimentation of blood corpuscles. *Nature* 104: 532.

Degner, R.V. 1988. Mechanical flotation machine design, proc in Industrial Practice of Fine Coal Processing, conference at Hidden Valley, Somerset, PA, September 25-28, Chapter 16, 135-146.

Dickinson, J.E., Galvin, K.P., 2014. Fluidized bed desliming in fine particle flotation – Part I. *Chemical Engineering Science* 108: 283-298.

Dickinson, J.E. Jiang, K., Galvin, K.P. 2015. Fast flotation of coal at low pulp density using the Reflux Flotation Cell, *Chemical Engineering Research and Design*, 101, 74-81.

Dickinson, J.E., Laskovski, D., Stevenson, P., Galvin, K.P. 2010. Enhanced foam drainage using parallel inclined channels in a single-stage foam fractionation column, *Chemical Engineering Science*, 65(8), 2481-2490.

DIRE 2015. New South Wales coalfields. NSW Department of Industry Resources and Energy website <http://www.resourcesandenergy.nsw.gov.au/investors/investment-opportunities/coal/coal> 08/03/16.

Euston, J., 2010. Two stage coal flotation using a mechanical cell. International Coal Preparation Congress 2010. R. Q. Honaker. Lexington, Kentucky, USA 382-390.

Galvin, K.P., Dickinson, J.E., 2014. Fluidized bed desliming in fine particle flotation – Part II: Flotation of a model feed. *Chemical Engineering Science* 108: 299-309.

Galvin, K.P., Harvey, N.G., Dickinson, J.E., 2014. Fluidized bed desliming in fine particle flotation - Part III flotation of difficult to clean coal. *Minerals Engineering* 66–68: 94-101.

Galvin, K.P., Walton, K., Zhou, J., 2009. How to elutriate particles according to their density. *Chemical Engineering Science* 64: 2003-2010.

Grassie, P., Cilliers, J.J., Neethling, S.J. Ventura-Medina, E. 2001. Quasi-one-dimensional foam drainage. *The European Physical Journal E: Soft Matter and Biological Physics* 6: 325-348.

Jameson, G.J., Goffinet, M., Hughes, D. 1991. Operating experiences with Jameson Cells at Newlands Coal Pty Ltd, Queensland. 5th Australian Coal Preparation Conference, 146-158.

Jiang, K., Dickinson, J.E., Galvin, K.P., 2014. Maximizing bubble segregation at high liquid fluxes. *Advanced Powder Technology* 25: 1205-1211.

Lambert, N. and Revell, P., 2008. Tripling flotation yield at Warkworth CHPP, 12th Australian Coal Preparation Conference and Exhibition and Proceedings, Sydney, NSW, 424-435.

Camomile, A., Weber, A., Lelinski, D., Foreman, D., Traczyk, F., Baker, T. 2013. Development of the largest flotation machine: 600 series SuperCell™ from FLSmidth. Proceedings of 10th International Mineral Processing Conference (Procemin 2013), Santiago, Chile, pp. 233-233.

Leonard, R.A., Lemlich, R. 1965. A study of interstitial liquid flow in foam. Part I. Theoretical model and application to foam fractionation. The American Institute of Chemical Engineers Journal 11: 18-25.

Lynch, A.J., Watt, J.S., Finch, J.A., Harbort, G.E. 2007. History of flotation technology, in: Fuerstenau, M.C., Jameson, G.J., Yoon, R.-H. (Eds.), Froth flotation: a century of innovation. Society for Mining Metallurgy and Exploration, pp. 65-91.

Lynch, A.J., Harbot, G.J., Nelson, M.G. 2010. History of flotation, Australasian Institute of Mining and Metallurgy, Carlton, Victoria (Spectrum series: issue 18).

Massinaei, M., Kolahdoozan, M., Noaparast, M., Olliazadeh, M., Yianatos, J., Shamsadini, R., Yarahmadi, M. 2009. Hydrodynamic and kinetic characterization of industrial columns in rougher circuit. Minerals Engineering 22: 357–365.

Miles, G.D., Shedlovsky L. & Ross, J. 1945. Foam drainage, Journal of Physical Chemistry, 49, pp 93-107.

Schulz, N.F. 1970. Separation efficiency, Transactions of the Metallurgical Society of American Institute of Mining, Metallurgical, and Petroleum Engineers, 247 (Mar.), 81-87.

Wace, P.F., Alder, P.J., Banfield, D.L. 1968. Foam separation process design. Internal Report. Harwell HL68/5699. Atomic Energy Research Establishment, Harwell, Oxfordshire.

Wex, T., Hill, B., Myers, A., Clark, L., 2004. Success achieved through incorporating recent developments in Jameson Cell technology into an existing Jameson Cell circuit. Proceedings of the 10th Australian Coal Preparation Conference, Pokolbin, New South Wales, Australia

Wieniawski, A., Mycfkowski, Z., Bfftoniek, W., 2010. Implementation of large-capacity flotation machines in coal flotation. International Coal Preparation Congress 2010. R. Q. Honaker. Lexington, Kentucky, USA 382-390.

Wills, B.A., Finch, J.A. (2016) Wills' Mineral Processing Technology, 8th Ed., An Introduction to the Practical Aspects of Ore Treatment and Mineral Recovery: Published by Butterworth-Heinemann, Elsevier, Oxford, ISBN 978-0-08-097053-0, pp. 329, 347.

Yao, K.M., 1973. Design of high-rate settlers. Journal of Environmental Engineering Division (Oct), 621–637.