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Single-stage recovery and concentration of mineral sands using a REFLUXTM Classifier

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Abstract

The REFLUXTM Classifier is a gravity separation device that consists of a system of inclined channels located above a fluidized bed. Here we report for the first time the results obtained processing a minerals sands feed using narrow 6 mm channels that promote a laminar-shear separation mechanism that enhances the separation based on density. The feed had a head grade of approximately 5 wt.% heavy minerals of density greater than 2800 kg/m³, with the majority in the size range from 50 to 150 µm. The overall recovery of the heavy minerals component was approximately constant at 85 % for solids throughputs of 10 to 18.5 t/(m² h). The unrecovered heavy mineral had an average density close to that of the gangue, hence was deemed unrecoverable by gravity separation. The recovery of the denser and more liberated zirconium mineral exceeded 95 %. So this work demonstrated the potential for the heavy mineral to be upgraded by a factor of 16 or more in a single stage operation.

Key Words:

REFLUXTM Classifier; Mineral sands; Beneficiation; High shear rates.

1.0 Introduction

Deposits of mineral sands are formed through alluvial deposition, with the relatively fine heavy minerals such as ilmenite and zircon being dispersed with silica sands covering a coarser size range. Gravity separation is the preferred approach for recovering and concentrating the heavy minerals. In a simple elutriator, however, the coarser sands will settle faster than the heavy minerals, significantly limiting the potential for upgrade, while ultrafine heavy minerals will report with the overflow tailings. Spirals have been the traditional method of separation due to the tendency for oversize particles to report with the lower density gangue due to drag and Bagnold forces (Wills & Finch, 2016). High recovery and upgrade have been possible, however this has required as many as three or even five spirals stages, incorporating rougher, cleaner, and scavenger streams. Generally desliming cyclones are used to prepare the feed, and the spirals product also requires a final upward current desliming stage to produce a clean concentrate (Marcos & Gilman, 2007). These additional processing steps lead to additional losses.

Increasingly today, the remaining deposits of mineral sands are lower in grade, and the heavy mineral particles are finer, hence the challenge for producers to recover and concentrate the

heavy minerals using spirals is more difficult than ever. More stages are required while the slurry throughput per stage must be decreased, with the recovery of the finest heavy minerals tending to decrease. While a conventional teetered bed separator may be capable of achieving an improved performance under these conditions, involving lower concentrations of the coarser sands, the throughput and heavy mineral recovery will also be unfavourable.

The REFLUX™ Classifier is a gravity separation device that consists of a vertical fluidised bed section with a set of inclined channels mounted above (Figure 1). The inclined channels provide a significant throughput advantage due to enhanced settling caused by the Boycott effect (Boycott, 1920). An overflow weir is set above the system of inclined channels to remove flow connectivity, thus driving consistent flows through each channel.

Figure 1: REFLUX™ Classifier a) schematic layout of the laboratory-scale system including the new underflow buffer system (Galvin, 2015) and b) cabinet projection view showing definition of key dimensions.

The REFLUX™ Classifier performance in recovering and concentrating mineral sands has been reported previously (Zhou et al., 2006). That work was conducted on a very low grade feed, nominally 1 wt. % heavy minerals, with relatively coarse sands. The results demonstrated high recovery, throughputs well in excess of 20 t/(m² h), and a satisfactory single stage upgrade.

However, in that early work, the inclined channel spacing (z) was 20 mm. Since then, significant advances have taken place. In 2008 we discovered a new “laminar-shear” separation mechanism, achieved by introducing more closely spaced inclined channels (Galvin et al., 2009). Fundamental analysis was later reported quantifying the role of the shear rate and the contribution of a highly selective inertial lift force (Galvin & Liu, 2011). In 2009 the REFLUXTM Classifier technology was re-designed to incorporate this discovery, with the inclined channel spacing reduced to 6 mm for all industrial units. Since then several studies have been published that demonstrate the ability of these narrow channels to efficiently beneficiate coal and iron ore (Galvin et al., 2010; Amariei et al., 2014), but none on mineral sands.

We have recently collaborated with a mineral sands producer who has permitted publication of the research findings. This is the first time this has been possible since 2006. The new laminar-shear mechanism has led to remarkable separations, with very significant single stage upgrades achieved. The de-slimed feed used in this work had a low heavy mineral grade of approximately 5 wt.%, and covered a relatively fine size range, with relatively little coarse sands, making the feed ideal for achieving a significant upgrade using a REFLUXTM Classifier. This paper examines the separation performance at solids throughputs covering the range 10 to 25 t/(m² h), focussing on the grade of the product, and the recovery of the heavy minerals as a function of the particle size. In this paper we present a complete, single-stage, separation of a mineral sands feed, demonstrating upgrade from 5 wt.% to about 80 wt.%.

2. Theoretical

In REFLUXTM Classifiers, there is a strong synergy between the lower fluidised bed and the upper inclined channels. The lower fluidized bed operates at a fluidization velocity moderately higher than the so-called minimum. It is critical that the suspension be fluid-like rather than packed-bed like so that relatively large, low density, particles can freely move and be displaced upwards through the autogenous dense medium. Provided the fluidisation velocity is below the critical value for layer inversion, fine high-density particles can form a dense layer below the larger low-density particles, despite the fact that the relatively large low density particles may have a higher terminal settling velocity at low solids concentrations (Moritomi et al., 1982).

The fluid-like state permits the suspension density to be measured, in turn allowing the device to be controlled. When the measured suspension density, basically an average value between two pressure transducers, exceeds the set point the underflow valve opens permitting the discharge of high density suspension; conversely when the measured density is too low the valve will close; proportional-integral (PI) control is used to target a fixed set point density.

The feed slurry enters part-way up the vertical section of the REFLUXTM Classifier. A portion, especially the larger and faster settling particles, will immediately settle downwards towards the lower part of the vertical section. However, above the feed entrance the system's

superficial velocity is relatively high, and therefore most of the entering solids are initially conveyed upwards. Thus, much of the valuable, heavy mineral material is conveyed upwards towards the overflow tailings. These solids then enter the system of closely spaced inclined channels with a perpendicular spacing (z) of 6 mm. The channels have a length (L) of 1000 mm, and thus have a high aspect ratio (L/z) of 167. A significant shear rate develops between the inclined surfaces, promoting shear-induced lift, and shear-induced hydrodynamic diffusion. Thus, relatively coarse, low-density particles are re-suspended and continue to be conveyed upwards to the overflow, whereas the relatively fine high density particles settle on the surface and slide downwards eventually returning to the vertical section. Ultimately these fine dense particles must leave the separator, either via the overflow or the underflow. Their concentration builds to a level sufficient to cause their dispersion down through the vertical section towards the underflow. Once these particles transport to a position below the level of feed entry, they become exposed to a much lower superficial velocity, and hence their transport towards the underflow discharge is suddenly much easier. In fact, many of these particles are capable of settling downwards once located below the position of feed entry while much finer particles continue to rely on dispersion. Finally, a lower fluidized bed, consisting of a high suspension density, develops. This high suspension density in turn generates a dense medium effect that promotes the upwards displacement of the misplaced coarse low-density particles until they eventually can enter the channels and be conveyed out the overflow of the system. Thus, there is strong synergy between the two sections of the REFLUXTM Classifier (Galvin & Iveson, 2014).

As with any density separation method, when targeting a lower particle size limit for the high density particles, there is an increasing tendency for the larger low-density particles to also report to the underflow. The presence of these much coarser low density particles in the lower bed demands a higher fluidization rate just to maintain incipient fluidization. Increasing the fluidisation rate will then result in the loss of some of the finest high-density material from the underflow. In the REFLUXTM Classifier this tendency is delayed because of the ability of the inclined channels to selectively recapture the fine high density particles (Section 4.1). However, although low Ep values can be obtained over a broad size range, the density cut point reduces with increasing particle size. So the maximum particle size range ratio used is typically 8:1 (e.g. Mitchell et al., 2014).

3. Experimental

Experiments were performed in a RCTM100 unit. This is a laboratory-scale REFLUXTM Classifier with a cross-section in the vertical section of breadth (B) 100 mm and width (W) 100 mm. The height (H) of the vertical section was 1.0 m, the length (L) of the inclined section was 1.0 m, and this section had 15 channels with a channel spacing (z) of 5.3 mm. Experiments were run on a continuous steady-state basis, with the feed entering via a 20 mm diameter inlet located 700 mm above the distributor flange. The fluidising water distributor was an inverted pyramid shape of height 90 mm, with four 0.75 mm diameter holes in each triangular face (16 holes total). The underflow valve at the base was 9 mm diameter.

The feed was a mineral sands sample from a commercial Australian deposit of nominal size range -2 mm which was then deslimed using a Kason VIBROSCREEN®. In the size distributions shown in Figure 2 it can be seen that 95 % of the feed mass was in the narrow size range -150 +53 μm . The feed was prepared in an agitated 300 L feed tank at the required solids concentration for the experiment. Feed was then continuously pumped out of this tank and into the RCTM100 unit. As the experiment progressed, the tank was regularly kept topped up with buckets of representative feed solids and an appropriate mass of water.

Figure 2: Mass frequency size distribution of the feed, underflow product and overflow reject streams from the experiment at 10 t/(m² h) feed solids flux (raw data).

The RCTM100 unit was operated with an automatic PI controller set to maintain the average density in the column at a specified value. It did this by opening and closing the underflow valve. When the measured value exceeded the set point the underflow valve opened, lowering the so-called bed level and hence the value of the measured density. Conversely, the valve was closed when the density fell below the set point. The average suspension density was inferred from the difference in pressure measured between two pressure transducers located in the vertical section at heights 60 mm and 440 mm respectively above the distributor flange.

Laboratory-scale fluidized bed separators suffer from a common problem, namely an over-size underflow valve relative to a full-scale unit. This is because there is a physical limit on how much a valve can be scaled down before the relatively coarse particles begin to arch and block the exit. This situation becomes acute when a small vessel of square cross-section 100 mm × 100 mm is used. The minimum valve size that can be used is thus over-sized with respect to the size of the bed, such that the entire contents of the vessel can be discharged within perhaps as little as one minute. Hence the underflow rate is very sensitive to valve position which makes it difficult for the control system to maintain steady conditions. Thus other pilot scale devices are often as large as 300 mm in diameter. However, given the REFLUXTM Classifier is a high throughput device a much smaller cross-section is essential under laboratory conditions.

In this paper we describe for the first time our novel “underflow buffer” introduced to address the above issue (Galvin, 2015). The arrangement permits the underflow valve to open to a high proportion, and remain open, while discharging relatively modest quantities of material. The underflow discharges directly into a small diameter column, fluidized through the base at a fixed buffer flow rate of 0.2 L/min, with material pumped from the top of this column via an upper side-arm using a positive displacement pump set at 0.4 L/min (Figure 1a). Thus, when the underflow valve of the REFLUXTM Classifier is closed, the pump produces a suction condition, only withdrawing water at the supplied buffer rate of 0.2 L/min. When the underflow valve is fully open, the pump only withdraws material at its set rate of 0.4 L/min, and hence the maximum discharge rate from the REFLUXTM is the difference between the two flows, $0.4 \text{ L/min} - 0.2 \text{ L/min} = 0.2 \text{ L/min}$. Clearly, the pulp density of the material discharged by the pump is diluted by the buffer and so an adjustment is required based on the known settings to obtain the actual underflow pulp density and flowrate from the REFLUXTM Classifier.

The small diameter fluidized column located below the underflow valve accumulated a very limited quantity of over-size (1 – 5 mm) particles that settled too fast to be conveyed by the upwards buffer flow. These collected in a trap and were discharged at the end of the experiment when the run was completed. The quantity of this oversize material was extremely small and was not included in the system mass balance which was focussed on much finer particles.

Once a satisfactory set of conditions is set there is no need for further adjustment of this arrangement. If it is observed that the underflow valve is open to nearly 100 % for a significant fraction of the time, then it is appropriate to operate the upper pump at a higher rate. Application of the underflow buffer at this small laboratory scale delivers very precise and uniform control. It is stressed, however, that this buffer arrangement is not required at larger process scales where the underflow valve is relatively small compared to the size of the unit.

Initially the controller keeps the underflow valve shut and the large/dense particles start to accumulate inside the unit, whilst fine/less dense material is elutriated in the overflow. The suspension density gradually increases until the set point is reached and the underflow valve starts to be opened by the controller. However, at this stage the system is not yet at steady state, because fine dense particles continue to accumulate inside the unit. They are captured by the enhanced settling area of the inclined channels and refluxed back into the vertical chamber. Gradually their concentration in the vertical section becomes high enough for them to force their way by dispersion down through the bed of particles that has formed at the base. It is not until after the pressure readings from both probes plateau that the system should be sampled.

Once steady state was reached, full stream samples of the overflow and underflow were collected simultaneously, and then after that a sample of the feed was collected. These flow samples were collected continuously for a period of the order 1 to 10 min, depending on the stream flow rate, to give samples with at least 900 g solids. All flow stream samples were

weighed and dried to calculate the suspension solids concentration. The dried solids were then screened at 45, 53, 63, 75, 90, 106, 125, 150, and 212 μm to obtain the size distribution, before being sent to a commercial laboratory for sink/float separation in TBE at 2800 kg/m^3 . The sinks fraction is referred to as the heavy minerals (HM). Selected sinks samples were then subjected to XRF analysis to measure their mineralogy. For the $10 \text{ t/(m}^2 \text{ h)}$ experiment, the density of each sinks sample size fraction was also measured using water pycnometry.

Figure 3 shows the distribution of the HM within the feed as a function of the particle size. The values shown are the percentage of the total HM in the feed that is resident within each particle size range divided by the width of that size range. It is clearly evident that the HM lies mostly within the size range 53 – 106 μm . Therefore any partition values calculated outside of this range will be subject to large uncertainties.

Figure 3: The significant concentration of the Heavy Mineral (HM) over a relatively narrow size range from 53 to 125 μm (mass-balanced data from $10.0 \text{ t/(m}^2 \text{ h)}$ run).

In the low-throughput run at $10 \text{ t/(m}^2 \text{ h)}$, there were also an additional 10 samples of around 300 g each collected down the height of the vertical section via 6 mm sampling valves mounted in the wall of the vertical section at heights of 915, 715, 565, 415, 340, 265, 165, 115, and 65 mm above the distributor flange. A detailed analysis of the suspension at each elevation was then undertaken, by determining the mass of solids and water, then determining the size distribution of the solid particles, and then the density of the particles within each particle size range. These data were then used to calculate the volume fraction of the solids and the suspension density at each elevation.

During operation, the bulk of the underflow and overflow streams were collected in separate tanks. This material was allowed to settle and then the excess water added to the system as fluidisation water was decanted. These samples were then mixed back together and used as the feed for the next experiment. By sampling the underflow and overflow simultaneously for the same time period, the reconstitution of the remaining material had a negligible effect on the feed composition.

Estimates of the flow split to product (yield) and recovery of a component can be calculated from the concentration of that component in the three streams by the two-product formula (Wills & Finch, 2016):

$$Y = \frac{P}{F} = \left(\frac{x_F - x_R}{x_P - x_R} \right) \quad (1a)$$

$$R = \frac{x_P P}{x_F F} = \left(\frac{x_F - x_R}{x_P - x_R} \right) \left(\frac{x_P}{x_R} \right) \quad (1b)$$

where Y is the solids split of feed material F to the product P , R is the fraction of the component recovered, and x_F , x_P and x_R are the concentrations of the component of interest in the feed, product and reject streams respectively. When applied to the HM fraction in each stream, Eq. (1a) and (1b) give respectively the total solids split to product and the recovery of HM in the product. When applied to the ZrO_2 assay in the HM fraction of each stream, they give the split of HM to the product and the recovery of ZrO_2 respectively (see Appendix B for sample calculations). These formulae only apply when there is one feed stream and two product streams. Hence in this work they cannot be applied to the total stream flows or slurry solids concentration (pulp density) data because there is an additional fluidisation stream involved carrying water into the system.

The two-product formula is also based on the assumption of perfect steady state operation with no breakdown of the solids and no sampling or measurement errors. However, random sampling and measurement errors can create inconsistencies in the raw data such that the recoveries calculated by different routes can vary. In the mass balance reconciliation, standard adjustments were made to the experimental data to insure all the mass-balance constraints were satisfied, subject to the minimization of an objective function based on the sum of the squares of the relative errors of the adjustments (Galvin et al., 1995). This mass balance reconciliation provided a systematic approach for determining the HM recovery as a function of the particle size.

4. Results and Discussion

A couple of preliminary runs (Runs 1 and 2) were conducted to identify suitable operating conditions for this feed (no samples were collected for analysis from these runs). A feed solids concentration of 44 wt.%, fluidisation flux of $2.7 \text{ m}^3/(\text{m}^2 \text{ h})$, underflow buffer flow rate of 0.20 L/min, and controller density set point of 1725 kg/m^3 were selected. Then four experiments (Runs 3 to 6) were conducted to investigate the effect of feed rate on the system's performance. The chronological sequence of these experiments was 14.3, 18.5, 25.4 and then $10 \text{ t}/(\text{m}^2/\text{h})$, where the flux is based on the cross-sectional area of the vertical section, $0.10 \text{ m} \times 0.10 \text{ m}$. Run 6 at $10 \text{ t}/(\text{m}^2 \text{ h})$ was analysed in greater detail by collecting samples down the length of the vertical section. Table 1 summarises the key parameters from

Runs 3 to 6. The results of the analysis of Run 6 are presented first in Section 4.1. Then the effects of varying feed rate in the other three runs are presented and discussed in Section 4.2.

Table 1: Key parameters and raw data from the four experiments analysed in this paper. Fluxes are based on the cross sectional area in the vertical section. Sinks refers to the heavy mineral (HM) particles denser than 2800 kg/m³.

	Run 6	Run 3	Run 4	Run 5
Feed rate (L/min)	2.8	4.0	5.0	7.2
Feed solids flux (t/(m ² h))	10.0	14.3	18.5	25.4
Fluidisation rate (L/min)	0.45	0.45	0.45	0.45
Buffer water rate (L/min)	0.20	0.20	0.20	0.20
Controller bed density set point (kg/m ³)	1725	1725	1725	1725
Feed slurry solids concentration (wt.%)	45.0	43.9	44.5	44.2
Product ... (wt.%)	78.8	77.3	76.7	82.1
Tailings ... (wt.%)	39.3	39.5	40.9	41.2
Feed sinks (HM) solids density (kg/m ³)	2704.2	2701.0	2700.4	2703.6
Product ... (kg/m ³)	3796.5	3732.1	3661.9	4077.3
Tailings ... (kg/m ³)	2659.4	2658.7	2657.4	2660.9
Feed solids sinks (HM) fraction (wt.%)	5.14	4.78	5.59	5.31
Product ... (wt.%)	77.15	75.9	71.67	88.09
Tailings ... (wt.%)	0.82	0.72	1.03	1.41
Feed sinks (HM) ZrO ₂ XRF assay (wt.%)			17.67	18.68
Product ... (wt.%)			22.88	25.54
Tailings ... (wt.%)			1.67	3.00
Yield of all solids to product (wt.%) ¹	5.7	5.4	6.5	4.5
Recovery of sinks (HM) to product (wt.%) ²	84.9	85.8	82.8	74.6
Recovery of ZrO ₂ to product (wt.%) ³			97.7	95.1

¹ Calculated from Eq. (1a) based on the mass fraction sinks raw data.

² Calculated from Eq. (1b) based on the mass fraction sinks raw data.

³ Calculated from Eq. (1b) based on the sinks ZrO₂ assay raw data.

Following the commencement of an experiment the measured density increased in a first order fashion, but then slowed to a more gradual, almost linear, trend towards the set point. The larger, faster-settling, particles accumulated at the base first. The relatively fine, high-density particles were initially conveyed upwards before being captured by the inclined channels and refluxed back into the vertical section. These fine high density particles, which in a conventional fluidized bed would normally report to the system overflow, instead gradually accumulate immediately below the entrance to the inclined channels. From there they are then transported down through the vertical section by dispersion, thus allowing the suspension density near the base to rise further towards the set point. A detailed analysis of the distribution of particles in the vertical section is presented in Section 4.1.

4.1 Analysis of the low solids throughput at 10 t/(m² h)

The concentration profile of the bed is shown in terms of the suspension density in Figure 4a and in terms of the volume fraction of the solids in Figure 4b. Above a height of about 400 mm, the bed density was almost constant at around 1500 kg/m³. Between 400 mm and 100 mm there was a rapid increase in bed density up to 1900 kg/m³, and then the bed density appeared to stabilise at this value below 100 mm. This lower region of high density visually coincided with a bed of coarser particles that could be observed at the base of the vertical section. This density profile is qualitatively similar to recent results observed for an ultrafine iron ore sample (Hunter et al., 2015).

Figure 4: a) Suspension density profile and b) volume fraction of solids profile up the height of the vertical section during the 10 t/(m² h) experiment.

Samples withdrawn from a fluidised bed via valves in the side wall are not perfectly isokinetic, so there is the possibility that sample bias will occur. However, integration of the bed profile shown in Figure 4a, between the heights of the two pressure probes at 60 mm and 440 mm, yields an average suspension density of 1718 kg/m³, very close to the set point of 1725 kg/m³. Thus there is no evidence of any bias in the profile samples. This agrees with previous work where samples withdrawn at the wall of relatively narrow laboratory-scale fluidised beds were found to give very close agreement with bed pressure profiles (e.g. Callen et al., 2002).

The density profile provides valuable insights into the operation and control of the REFLUXTM Classifier, beyond simply the reporting of the average density between the probes. It is evident that near the base a suspension density as high as 1900 kg/m³ was achieved, however, above the main bed the density was relatively constant at 1500 kg/m³. Thus by changing the positions of the pressure transducers, different density values would be measured for the same bed profile, and hence the same separation. In other words the same separation could be achieved using a set point as large as 1900 kg/m³ or as low as 1600

kg/m^3 , depending on the location of the pressure transducers. However, presumably the control of the system would be less stable if the transducers were located too close together. Conversely if the transducers were mounted too far apart, then the existence of a large region of constant density would reduce the control sensitivity. The optimum location of the two pressure transducers is being investigated in separate work. A low set point, as required in this work requires the bottom probe to be as low as possible.

Figures 5a and 5b show the average solids density and volume fraction, respectively, of particles in selected size ranges within the vertical section. There was significant scatter in the volume fractions measured over narrow size ranges, so volume fraction data are only shown over three relatively broad size bands in Figure 5b to minimise this noise. It is evident in Figure 5a that the coarsest particles had a low solids density at all elevations, whereas the finest particles exhibited significantly higher densities at the lower elevations. This reflects the fact that in the high solids fraction (dense) bed near the base of the column, there is a higher interstitial fluid velocity, and so only the very densest of the fine particles can penetrate by dispersion into that region. Coarse particles sink rapidly down the length of the column and accumulate at the base (Figure 5b). The presence of the low density coarse particles in the lower section limits the maximum suspension density obtainable. The average density across all particles existing near the base only reached 3200 kg/m^3 (with volume fraction of ≈ 0.4), giving a suspension density just above the base of $0.4 \times 3200 + 0.6 \times 1000 \approx 1900 \text{ kg/m}^3$ (Figure 4a).

Figure 5: Bed profile from the 10 t/(m² h) run showing (a) the average particle solid density and (b) the volume fraction of particles from selected size ranges as a function of elevation. The finest particles have the highest densities in the lower bed, reflecting their significant upgrade. Lines are drawn only to guide the eye.

The steady state feed, product, and reject samples were sieved into narrow size fractions, and each size fraction subjected to heavy liquid separation using TBE at a density of 2800 kg/m^3 . The percentage of the particles reporting to the sinks fraction was defined as the % heavy

minerals (HM). Values were also obtained for the sinks fraction in the overall feed, product, and reject. The REFLUXTM Classifier succeeded in upgrading the feed from 5 wt.% HM to 77 wt.% HM in a single step with a tailings containing 0.82 wt.% (Table 1). Application of the two-product formula gives a solids yield of 5.7 wt.% and HM recovery of 84.9 wt.%. With significant levels of HM as fine as 53 μm , this separation would normally require many stages of spirals to achieve the same upgrade.

Table A2 shows the size distribution and HM values by size for the feed, product, and reject. What is remarkable is the very high HM content of the particles in the product stream below 106 μm , with HM values greater than 90 wt.% and approaching 100 wt.%. Thus the product does not need to undergo a further de-sliming stage as is normally required following conventional spirals processing, risking losses of valuable HM when using a standard elutriator. With the REFLUXTM Classifier, the finer particles are always very high in grade, with the contamination instead at the larger particle sizes due to the presence of the coarser sands. Given the final product rate is much lower than the original feed rate, with the yield around 5 wt.%, final upgrading through either screening or application of spirals would involve relatively low tonnages.

Based on the mass-balance reconciled data (Table A3-A4), the partition to underflow of the 2.8 specific gravity sinks and floats fractions was calculated and is shown in Figure 6. This highlights the high recoveries achieved of the dense minerals to the underflow product. The reason the partition levels off below 100 % is believed to be due to the existence of composite particles of lower density. This hypothesis is supported by the data in Figure 7 where the density of the product sinks fraction quickly drops from above 4500 kg/m^3 down to below 2700 kg/m^3 as particle size increases from 70 to 150 μm . These values suggest that the unrecovered HM was not only low in density but may also have been porous, given that the densities were on average below the 2800 kg/m^3 value of the TBE liquid. Organic liquids presumably displace air from pores more effectively during the sink-float separation than water does during the pycnometry density measurement. So the pycnometry measurements reported in Figure 7 are lower than the true skeletal density of the particles. It is concluded therefore that up to 15 % of the HM was unrecoverable by gravity separation, and that the material may be of quite marginal value.

Figure 6: Partition to underflow of the 2.8 specific gravity sinks and floats fractions from the 10 t/(m² h) experiment (based on reconciled data in Tables A3 and A4). Lines drawn only to guide the eye.

Figure 7: Density, as measured by water pycnometry, of the “+2800 kg/m³” sinks material versus particle size from each stream in the 10.0 t/(m² h) run.

4.2 Operation at Higher Solids Throughputs

Additional experiments were conducted at feed solids fluxes of 14.3, 18.5, and 25.4 t/(m² h) to examine the effect of increasing the solids throughput. Table 1 reports the overall fractions of HM in the feed, product and reject streams of each run. Appendix B reports more details of the HM grades by size. Figure 8 shows the HM recovery as a function of particle size for the four different feed rates, as calculated using Eq.(1b) based on the raw sink-float data. Sample calculations are presented in Appendix B.

Initially the HM recovery increases with increasing particle size, approaching as high as 92 % for the lowest throughput experiment. Above 100 μm the HM recovery decreases and then appears to level off, albeit with considerable scatter which reflects the uncertainty in calculating recoveries when there is so little HM material in the feed above 120 μm . This decrease is unusual for this technology, as large particles are much easier to recover in the underflow product. However, as discussed above, this reduction in HM recovery most likely reflects changes in the mineralogy with particle size, with more of the larger HM matter consisting of composite particles of much lower density as shown in Figure 7. This is further confirmed in detailed XRF analysis which shows that, within the HM fraction above 106 μm , the proportion of the high density zirconium components declines to very low levels (Appendix B).

Figure 8: HM recovery as a function of particle size for the four different feed rates calculated using Eq. (1b) based on the raw sink-float data. Also shown is the distribution of HM in the feed (repeated from Figure 3).

XRF assays were conducted on a limited number of samples (see Appendix B). In cases where assays were measured on identical size fractions across all three streams, the recovery of zirconium was calculated by Eq. (1b). Figure 9 shows this calculated recovery as a function of particle size (note that these values have been calculated from the raw unbalanced XRF data, so some calculated recoveries lie just outside the range of 0 to 100 %). Recoveries of the high-density zirconium component were much higher than the recovery of the +2800 kg/m^3 heavy mineral fraction shown in Figure 8. In fact, in the region of interest between 50 μm and 100 μm where most of the HM is found (Figure 3), the recovery of ZrO_2 was

consistently of order 100 %. The calculated recoveries less than 90 % are all in size intervals where there was practically no material present, and so as well as being of less relevance these values will also have large uncertainties.

Figure 9: ZrO₂ recovery calculated by Eq. (1b) as a function of particle size for the two different feed rates at which detailed assays were measured (raw data). Values over 100 % are due to mass balance errors in the raw data. Also shown is the distribution of HM in the feed (repeated from Figure 3).

A further observation in Figure 8 is that the location of the initial peak in recovery appears to shift from about 50 μm at 10 t/(m² h) up to around 80 μm at the three higher solids feed rates. This deterioration in performance can be explained by the increased volumetric flowrate through the system. This increases the velocity in the inclined channels, which reduces their ability to capture high density material that has been misplaced to the overflow reject stream.

Figure 10 shows the overall HM recovery and product HM grade as a function of the solids feed rate per unit area. Performance was remarkably robust with recoveries almost constant in the range up to 18.5 t/(m² h), and then with a modest deterioration in recovery above that level. However, the high product grade achieved at the highest solids throughput suggests that by using a lower set point the recovery could have been increased.

Figure 10: The overall HM recovery and product grade achieved versus the solids throughput at a fixed feed pulp density of 44 wt.%. Recoveries calculated by Eq. (1b) using raw data (Table 1). The unrecovered HM had a density close to that of the sands (Figure 7). The recovery only declined significantly once the throughput exceeded 18.5 t/(m² h).

5. Conclusions

The REFLUX™ Classifier, with closely spaced inclined channels, operates via a novel laminar-shear mechanism transporting low-density particles over a wide size range to the overflow, while segregating the denser particles onto the inclined surfaces from where they slide back and eventually exit via the underflow. This arrangement delivers efficient single-stage separation. The overall recovery of heavy minerals (particles greater than 2800 kg/m³ density) was approximately 85 wt.% for feed solids throughputs from 10.0 up to 14.3 t/(m² h), with an upgrade from 5 wt.% to about 80 wt.% heavy minerals in the product. The recovery remained close to this level at 18.5 t/(m² h), declining to 75 % at 25.4 t/(m² h). Interestingly, at the higher throughput the grade was significantly higher due to increased conveying of the low-density coarse sands upwards through the inclined channels. Hence a lower density set point could have delivered an equivalent heavy mineral recovery even at 25.4 t/(m² h) feed rate. The unrecovered heavy mineral was found to be relatively low in density, close to that of the gangue, and hence unrecoverable by gravity separation. The recovery of zirconium, however, exceeded 95 %. Hence this technology is well suited for single-stage application in the processing of minerals sands.

6. Acknowledgments & Disclaimers

The University of Newcastle holds international patents on the REFLUX™ Classifier and has a Research and Development Agreement with FLSmidth Pty Ltd. The inventor of the

REFLUX™ Classifier, and the first author of the publication, K.P. Galvin, is a beneficiary of the University's intellectual property policy.

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Appendix A: Detailed Data Mass Balancing for the 10 t/(m² h) Solids Feed Rate Experiment (Run 6)

A1. Raw Experimental Data

Table A1: Total flows and fraction sinks from 10 t/(m² h) experiment (raw data).

	Feed	Product	Reject
Solids Rate (t/(m ² h))	10.0	0.55	9.45
Heavy Mineral (wt.%)	5.14	77.15	0.82

Table A2: Size distribution and HM fraction from 10 t/(m² h) experiment (raw data).

Size	-212	-150	-125	-106	-90	-75	-63	-53	-45	
(μm)	+212	+150	+125	+106	+90	+75	+63	+53	+45	
Size Distribution (wt.%):										
Feed	0.42	1.92	8.31	22.41	23.92	32.41	6.35	1.48	0.52	2.26
Product	5.53	7.31	6.68	6.31	12.44	38.01	19.15	2.57	1.30	0.71
Reject	0.06	1.36	7.93	26.83	27.45	27.63	4.71	1.02	0.51	2.49
Heavy Mineral Concentration (wt.%) in each size interval:										
Feed	2.67	0.88	0.67	0.88	2.21	7.86	20.41	15.44	10.66	2.00
Product	3.06	3.87	15.13	55.35	92.59	98.34	99.38	97.86	97.09	91.79
Reject	1.60	0.13	0.09	0.11	0.30	1.61	2.50	1.54	1.33	0.40

A2. Mass-balance Reconciled Data

Table A3: Total flows and HM fraction from 10 t/(m² h) experiment (reconciled data).

	Feed	Product	Reject
Solids Rate (t/(m ² h))	9.99	0.56	9.43
Heavy Mineral (wt.%)	5.13	77.30	0.82

Table A4: Size Distribution and HM fraction from 10 t/(m² h) experiment (reconciled data).

Size	-212	-150	-125	-106	-90	-75	-63	-53	-45	
(μm)	+212	+150	+125	+106	+90	+75	+63	+53	+45	
Size Distribution (wt.%):										
Feed	0.40	1.92	7.71	20.64	26.55	32.73	5.99	1.16	0.58	2.32
Product	6.14	7.42	5.37	5.29	11.57	40.03	19.51	2.90	1.06	0.72
Reject	0.06	1.59	7.85	21.55	27.45	32.30	5.19	1.06	0.55	2.41
Heavy Mineral Concentration (wt.%) in each size interval:										
Feed	2.77	0.90	0.68	0.90	2.38	8.33	20.27	15.12	11.09	2.00
Product	2.96	3.69	15.19	54.76	85.15	98.33	99.42	97.84	97.02	91.84
Reject	1.60	0.13	0.09	0.11	0.30	1.68	2.49	1.59	1.31	0.40

Appendix B: Sample Calculations and Sink-float and XRF Raw Data from Runs 3-5

Tables B1-B3 report the raw sink-float and XRF data (where measured) for Runs 3-5 respectively. The heavy mineral recoveries in Figure 8 were calculated from the raw sink-float data using Eq. (1b). For instance, the recovery of HM in the -90 +75 μm size fraction in Run 5 (Table B3) was: $R = (99.35/12./10)(12.10 - 3.06)/(99.35 - 3.06) = 0.771$ i.e. 77.1 %. The zirconium recoveries in Figure 9 were calculated from the raw XRF ZrO_2 assay data using Eq. (1b). For instance, in the -90 +75 μm size fraction from Run 5 (Table B3), the zirconium recovery was $R = (27.05/25.56)(25.56 - 3.95)/(27.05 - 3.95) = 0.990$ i.e. 99 %.

Table B1: Sink-float raw data from Run 3, the 14.3 t/(m² h) experiment.

Size (μm)	212	-212 150	-150 125	-125 106	-106 90	-90 75	-75 63	-63 53	-53 45	-45	Head Grade
Concentration of HM (wt.%) in each size fraction:											
Feed	2.19	0.71	0.68	2.01	8.59	12.21	6.47	5.48	4.74	11.56	4.78
Product	2.78	3.26	14.15	59.71	97.34	99.12	99.44	98.94	98.37	98.64	75.90
Reject	1.35	0.15	0.49	0.57	1.57	1.80	1.36	2.00	1.83	6.62	0.72

Table B2: Sink-float and XRF assays of from Run 4, the 18.5 t/(m² h) experiment. XRF data not available for all size fractions.

Size (μm)	212	-212 150	-150 125	-125 106	-106 90	-90 75	-75 63	-63 53	-53 45	-45	Head Grade
Concentration of HM (wt.%) in each size fraction:											
Feed	2.52	0.85	0.79	2.11	10.35	19.09	16.60	13.28	12.82	4.43	5.59
Product	2.78	3.48	14.63	48.06	93.47	98.72	98.89	98.77	95.62	97.15	71.67
Reject	1.64	0.24	0.28	0.50	1.93	2.95	3.15	3.85	4.90	5.60	1.03
Concentration of ZrO_2 (wt.%) in the HM:											
Feed	2.30	1.37	3.57	8.54	18.49	27.78	28.50			16.15	17.76
Product	3.29	2.22	3.79	5.77	17.97	27.85	32.42	35.76	38.47	39.88	22.88
Reject	0.86	0.98	0.60	1.01	1.49	2.80	2.78	3.47			1.67

Table B3: Sink-float and XRF assays from Run 5, the 25.4 t/(m² h) experiment. XRF data not available for all size fractions.

Size (μm)	212	-212 150	-150 125	-125 106	-106 90	-90 75	-75 63	-63 53	-53 45	-45	Head Grade
Concentration of HM (wt.%) in each size fraction:											
Feed	3.78	0.85	0.57	1.66	9.53	12.10	13.69	10.24	10.37	8.42	5.31
Product	5.51	5.59	41.10	84.13	97.56	99.35	99.05	99.61	96.57	99.55	88.09
Reject	0.86	0.15	0.22	0.54	2.43	3.06	3.53	3.25	4.23	7.51	1.41
Concentration of ZrO_2 (wt.%) in the HM:											
Feed	2.26	1.38		7.41	19.24	25.56	30.30	34.56		15.07	18.68
Product	3.42	1.83	4.14	6.70	16.20	27.05	35.81	40.15	42.96	46.32	25.54
Reject	0.69				2.63	3.95	5.17			8.48	3.00

Highlights:

- 0.053 – 0.15 mm mineral sand beneficiated in single stage REFLUX™ Classifier.
- A range of feed rates were used from 10 to 25 t/(m² h).
- Recovery of heavy mineral (+2800 kg/m³ material) was 85 % at lowest feed rate.
- Unrecovered heavy mineral had low density close to the gangue value.
- Recovery of zirconium component was greater than 95 %.

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