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A Water-Based Method for Measuring Density-Based Partition Curves of Separators used in Coal and Mineral Processing

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Abstract

Traditional sink-float methods for measuring the density distribution of particulate samples rely on expensive and toxic heavy liquids. An alternative method has been developed which uses aqueous glycerol solutions in a laboratory-scale Reflux Classifier run in semi-batch mode. The high viscosity of these solutions promotes laminar high-shear flow in the channels which suppresses the effect of particle size on separation performance. Thus this technique was able to accurately measure the yield-ash curve of coal samples, and from this their density distribution could be inferred. Applying this approach to feed, product and reject samples enabled calculation of the density partition separation performance. Samples were collected from two case studies: a laboratory-scale continuous Reflux Classifier and a single spiral start from a full-scale coal handling and preparation plant. In both cases the partition curve measured by the new method was within experimental uncertainty of the partition curve measured by the standard sink-float method.

Key Words:

Washability; partition; sink-float method; density separation; Reflux Classifier

Highlights:

- Feed, product and reject samples were collected around full-scale unit operations.
- Each sample was fractionated in a laboratory Reflux Classifier.
- From the yield-ash curves, the density distribution was inferred.
- The resultant partition curves were in close agreement with sink-float results.
- So the Reflux Classifier is a potential alternative to the sink-float method.

Graphical Abstract:

Density Partition Curve Measurements

Comparison of new fractionation method using aqueous glycerol solutions in a laboratory Reflux Classifier vs. standard sink-float method using hazardous heavy organic liquids.

1.0 Introduction

The coal and mineral processing industries use a variety of separation unit operations for beneficiation, including dense medium cyclones, jigs, Reflux Classifiers, spirals and flotation cells. Ideally the partition performance of these units should be regularly measured to ensure that they are operating efficiently and in order to be able to predict the likely product grade and recovery if the feed stock varies. For large particles, partition performance can be measured relatively easily in real time by using tracer particles of varying density (Davis et al., 1985; de Korte, 2003; Wood, 2013⁰⁷). However, for fine particles tracer particles are not an option. Instead, partition performance is determined by measuring the density distribution of samples from the feed, product and reject streams.

The sink-float method is the industry standard method for measuring the density distribution of particulate material. In the coal industry, this method uses mixtures of heavy organic liquids with densities ranging from 1200 up to 2400 kg/m³. However, these liquids are expensive, hazardous and can also adversely affect the carbonisation properties of clean coal composites (Callen et al., 2008; Iveson & Galvin, 2012). In separations involving denser minerals, an upper limit of around 5000 kg/m³ can be obtained using aqueous Clerici solution, but this is expensive, highly toxic and corrosive. Effective densities of up to 18 000 kg/m³ can be produced using paramagnetic salts or ferrofluids (suspensions of nano-sized magnetite particles) situated in a magnetic field gradient. This method is still being developed and is only useful for non-magnetic particles with a lower size limit of the order 0.1 mm (Galvin, 2006; Wills & Napier-Munn, 2006).

Hence at this stage many industries are “blind” to the partition performance of their beneficiation processes at high densities and particularly at fine particle sizes. Even at lower densities, the hazardous nature of the dense liquids used makes it desirable to find alternative methods to measure the density distribution.

The Reflux Classifier (RC) consists of a set of parallel inclined channels located above a vertical section with fluidising water added through the base (Figure 1). This technology has been in development for over a decade and is now being used extensively in coal preparation plants (Osborne, 2013) and increasingly in mineral processing plants, especially in South Africa. Inclined channels have a higher throughput capacity compared to a conventional fluidised (teetered) bed separator (TBS) due to their increased effective settling area, the so-called Boycott effect (Boycott, 1920). In addition, low-density particles that settle onto the inclined surface are more likely to be re-suspended than high-density particles. This improves the efficiency of the device as a density-based separation process (Laskovski et al., 2006). When narrow channels are used to promote laminar flow, the effects of the near linear velocity profile and the high-shear-rate induced lift force at the wall “conspire” to almost totally suppress the influence of particle size (Galvin et al., 2009, 2010; Galvin & Liu, 2011). As a result full-scale Reflux Classifiers are routinely able to achieve Ep values below 0.1 across a wide range of density cut points, even when there is a significant amount of near density material (Mitchell et al., 2014).

Figure 1: a) Side view of Reflux Classifier semi-batch configuration and b) cabinet-projection view showing definition of key dimensions.

In industrial processes the Reflux Classifier is operated continuously, with feed added centrally below the system of inclined channels. An underflow of dense material is continuously removed from a central discharge point in the base and the overflow of low density material exits via the launder at the top. During continuous processing, a bed of particles builds up inside the unit, producing an autogenous dense medium effect which further improves density separation performance (Galvin, 2012).

However, the Reflux Classifier can also be used in a semi-batch configuration as a laboratory tool for performing density-based fractionation. In this mode, shown in Figure 1a, an initial charge of particles is first placed in the vertical section. There is no further feed addition or underflow removal, but a flow of fluidization water commences through the base and the overflow is collected. The fluidisation rate is then increased in a number of steps. Initially low density particles (and also fine slimes) are preferentially conveyed out of the system. As the flowrate is increased, particles of higher density (and also larger size) are successively elutriated. The particles conveyed out at each flowrate are collected as a sample, weighed and analysed. These data can then be used to generate a cumulative yield-ash or grade-recovery curve.

For feed samples of 0.038 mm to 0.250 mm size coal, using water with 1.7 mm channel gaps gives near perfect agreement with the sink-float generated yield-ash curve. Coal samples in the 0.250 to 2.00 mm size range required larger 6 mm channels and so the flow Reynolds number, for a given fluid velocity and viscosity, was higher. Under batch processing conditions the solids concentration within the inclined channels is often so low that particles settle at velocities close to their dilute terminal values, hence the required fluid velocity needed to convey the particles is much higher than under continuous processing conditions. This means that under batch conditions in 6 mm channels, the flow is often no longer laminar. Hence the performance, whilst still good, was not in perfect agreement with the sink-float data (Walton et al., 2010; Iveson & Galvin, 2011).

However, recently it has been shown that the regime of near perfect separation performance can be extended using aqueous glycerol solutions. The higher viscosity of the glycerol solutions is beneficial as it maintains the channel flow in the laminar regime, even in wider channels involving particles up to 16 mm in size. The higher density of glycerol solution also aids the separation. Further, the lower conveying velocities required with glycerol solutions cause much less particle attrition compared with using water (Hunter et al., 2014).

Hence given the near perfect separation performance that can now be obtained in the laboratory-scale Reflux Classifier, it should be possible to use this method to infer the density distribution of material in feed, product and reject samples collected around a separation unit. From these data the partition performance of the unit could then be calculated, without needing to resort to the sink-float method. Glycerol also has the advantage of being cheap and non-toxic.

This paper reports work done to test the effectiveness of this proposed method for measuring partition performance. Two different case studies are reported, a laboratory-scale Reflux Classifier and a single spiral stage at a commercial coal handling and preparation plant. Both of these case studies involved coal particles in the nominal $-2.00 +0.125$ mm size range. The details of the experimental method are first presented in the materials and methods section. The method of analysing the data to produce a partition curve is then explained in the calculations section. This is followed by the results and discussion, and then some final conclusions.

2.0 Material and Methods

2.1 Samples

Two different case studies were conducted. Case Study 1 involved the testing of feed, product and reject samples taken around a steady-state laboratory-scale Reflux Classifier with a 6 mm channel spacing operated in continuous mode. The Reflux Classifier was running at steady state at the time of sampling with full-stream cuts collected of each stream. The feed used in this experiment was a Lower Hunter Valley coal taken from the feed stream of a fine gravity circuit.

The samples for Case Study 2 were obtained from full-stream cuts collected by hand around a single spiral start operating in a Coal Handling and Preparation Plant (CHPP) in the Hunter Valley, Australia. This spiral was one of multiple spirals in a circuit where the middling streams were recycled to the feed distributor at the head of the spiral circuit. The circuit configuration and sampling points are shown in Figure 2. It took over half an hour to collect these samples from all four streams.

For both case studies, the collected samples were wet screened at 0.130 mm, dried and then subdivided (by either a rotary sample divider or riffle splitter) into representative subsamples each weighing approximately 3 kg. Sink-float analysis of one sub-sample from each stream was performed by an external coal laboratory as per Australian Standard AS4156.1. For Case Study 1, twelve density intervals were used based on densities 1300, 1350, 1400, 1450, 1500, 1550, 1600, 1700, 1800, 1900 and 2000 kg/m³. In Case Study 2, the 1350 and 1450 kg/m³ liquids were omitted, but an additional sink-float was performed at 1650 kg/m³ in order to gain greater resolution near to the density cut-point. For Case Study 2, each density fraction was also screened into five size fractions at 1.00, 0.710, 0.500, 0.250 and 0.125 mm prior to

the ash measurements. These data were then subject to mass-balance reconciliation to obtain an unambiguous partition curve. A second sub-sample from each stream was fractionated in the laboratory Reflux Classifier, as described in Section 2.2.

Figure 2: Spiral circuit configuration for Case Study 2. Samples were collected from the feed, product, middlings and reject around a single spiral start. Stars (★) indicate locations of sampling points (Iveson et al., 2014).

2.2 Experimental Method – Laboratory Batch Reflux Classifier Fractionation

The experimental method used to operate the laboratory Reflux Classifier in semi-batch mode was the same as in previous published research (Walton et al., 2010; Hunter et al., 2014). The unit had a vertical section with a nominal height of $H = 1$ m and cross section width $W = 100$ mm and breadth $B = 80$ mm. The inclined section was $L = 1$ m long and inclined at $\theta = 70^\circ$ to the horizontal. A total of 11 stainless steel plates of thickness $t = 0.6$ mm were used in the inclined section to create 12 parallel flow channels of $z = 6$ mm perpendicular gap spacing (refer to Figure 1b for definition of the different dimensions).

The charge of approximately 3.0 kg of feed particles was first loaded into the vertical chamber. Then an initial very low flowrate was used to fluidise the particles to flush out any trapped air bubbles and allow the particles to start to stratify. After that, the fluidising rate was increased in steps. Each flowrate was allowed to run for a period of time until the rate of particle elutriation decreased to negligible levels. During this time, the elutriated particles were collected by passing the overflow through a 55 μm filter sock. The duration of each flow step varied depending on the sample size and the stage of the experiment, but was typically 10 to 60 min. Longer periods were used for initial flows and briefer periods were used for later flows.

Samples corresponding to different flowrates were labelled "Flow 1", "Flow 2", etc. Typically five to eight such flow increments were collected and the experiment was concluded when approximately 500 g of material was left inside the unit. However, as discussed later, for *product* stream samples it was found necessary to continue to collect flow increments until only the order of 50 g remained inside the unit. This was done in order to accurately measure the distribution of the small amount of high density material present in product samples. At the end of each experiment, the Reflux Classifier was rinsed out and the material remaining in the unit was collected and labelled "Remains". Each flow increment was rinsed in the filter sock using tap water and then soaked in 20 L buckets of tap water for a period no less than 12 h. Each sample was then dried in an oven overnight.

Two different fluidising mediums were used. A solution of 70 wt.% glycerol was used to process the samples for Case Studies 1 and 2. The glycerol solution was prepared in a 300 L tank. After passing through the 55 μm filter sock the overflow filtrate was returned to the tank so that the solution could be recycled (for more details see Hunter et al., 2014). For Case Study 2, one set of experiments was also conducted using tap water as the fluidising medium.

Each flow and remains sample was dry-screened at 0.125 mm. The undersize was not in the size range of interest, so these fractions were combined and analysed as one sample referred to as the "Undersize", purely for the purposes of mass-balancing. The +0.125 mm fractions from each flow were sent away to an external coal laboratory for screening. Case Study 1 samples were screened at 0.125, 0.180, 0.250, 0.355, 0.500, 0.710, 1.00 and 1.40 mm. For Case Study 2 the 0.180 and 0.355 mm sieves were omitted. This was followed by measurement of the ash value of each size interval on an air-dried basis as per Australian Standard AS3881.

3.0 Calculation

This section explains how the laboratory-scale semi-batch Reflux Classifier flow fractionation results from samples collected around a separator were analysed to determine the unit's density partition curve. Unlike the sink-float method, this test cannot fractionate samples at pre-determined density cut points. This is because each sample has a unique particle size and density distribution which produces varying hindered settling factors as the test progresses. So different samples will have different density cut points, even at the same fluidisation flow rate. However, in order to calculate partition values, we must know the amount of material in equivalent density fractions across all streams. Hence for each stream, the ash values must first be converted into densities (Section 3.1). This information is then used to determine the density distribution of the material in each stream (Section 3.2). After that the density distribution data are subject to conventional mass-balance reconciliation and then used to calculate the partition values (Section 3.3).

3.1 Establishing the Ash-Density Relationship

The dependency of particle density on the ash data will vary from coal to coal. Given that coals consist of a variety of macerals and may contain more than one type of gangue material, arguably there is no reason to expect a unique relationship to exist. However, in order to proceed, it is necessary to assume that on average there is a robust correlation between the particle density D (kg/m^3) and ash value A (wt.% a.d.). Previous workers have found that a simple linear model gives a good fit (Galvin, 2009). Here we use a linear relationship of the form:

$$D = \rho_c + (\rho_{mm} - \rho_c)(A/100) \quad (1)$$

where ρ_c is the density of “pure” coal particles and ρ_{mm} is the density of the “pure” mineral matter. Clearly these values will vary from seam to seam, but Iveson and Galvin (2011) found that the relationship remained fairly consistent across all size ranges between 50 mm and 0.038 mm for a given coal.

Figure 3 illustrates the density-ash relationship data obtained from sink-float tests of all three streams in Case Study 1. It shows that the linear model gives a very good fit to the data, and that there is very little variation between the three streams for the same coal sample. A similar result was found for Case Study 2 and the fitted parameters for both coals are shown in Table 1. These fitted parameters were used in Equation 1 to infer the density of each flow increment based on its ash value.

Figure 3: Sink-float interval average density versus interval ash value for the feed, product and reject samples from Case Study 1. Lines show minimum sum square error best fits of Eq. 1 and 2 to the feed data.

As noted in previous literature (Luttrell et al., 2003), if coal consisted of a binary mixture of two immiscible solid phases, then the average solid density should in theory be given by Equation 2:

$$D = \frac{100}{\frac{A}{\rho_{mm}} + \frac{100 - A}{\rho_c}} \quad (2)$$

This is a non-linear relationship which leads to data sometimes being plotted as inverse density versus ash value. However, Iveson and Galvin (2011) found that this model often implied unrealistically large values for the density of the mineral matter. For Case Study 1, the best fit of Eq. 2 (shown in Figure 3) gave values of $\rho_c = 1310 \text{ kg/m}^3$ and $\rho_{mm} = 2678 \text{ kg/m}^3$. Not only are these values higher than expected, but the fit of Eq. 2 to the data is clearly not as accurate as the simpler linear model. This indicates that the assumption of a pure binary mixture is overly simplistic.

Ultimately the form and number of parameters used in the density versus ash value correlation is unimportant. What matters is whether it reliably predicts a particle's density based on its ash value. Clearly the simple linear model allows this to be done quite accurately, particularly in the range of interest around the density cut point.

It might be argued that if the aim of this research is to develop an alternative to the sink-float method, then relying on density-ash relationships measured by the sink-float method defeats the purpose. However, for a given coal type, once the density-ash relationship has been established, it is unlikely to vary much within that seam. So in most cases there would already be existing historical data on the ash-density characteristics for that coal type.

Table 1: Best-fit parameters of Equation 1 to the ash-density sink-float data for the two case studies, where ρ_c = density of pure combustible matter kg/m³, ρ_{mm} = density of pure mineral matter and R^2 = correlation coefficient.

Case Study	Linear Fitted Parameters (kg/m ³)		R^2
	ρ_c	ρ_{mm}	
1	1219	2401	0.9964
2	1224	2351	0.9871

If the only purpose of the elutriation experiments was to measure density-partition performance (i.e. the yield-ash curves were not required), then it would be possible to measure the sample densities directly using liquid or gas pycnometry. This would potentially be more accurate since it would give the exact density of each sample, rather than using a correlation based on assuming the density of a sample was the average of the upper and lower density bounds of each sink-float interval. However, given the porosity of coal, some correction might be required in order to maintain consistency with the existing standards. Direct measurement of sample density would also be the best way to apply this elutriation method for measuring density-partition curves to non-coal minerals.

3.2 Extraction of the Density Distribution from Laboratory Reflux Classifier

The objective is to use the yield-ash information to generate a plot of cumulative mass versus density for each flow stream, from which partition values can then be calculated. This analysis assumes that there is a correlation between ash value and particle density and that the Reflux Classifier separates perfectly on the basis of particle density. The former assumption was discussed in Section 3.1, and the latter assumption is justified based on the close agreement between the yield-ash curves measured by the laboratory Reflux Classifier and the sink-float methods (Section 4).

This section presents a brief outline of the steps involved. Appendix A provides a full set of raw data and sample calculations for Case Study 1 to clearly illustrate the procedure used. Full details of the sink-float and Reflux Classifier raw data and analysis for Case Study 2 can be found in Iveson et al. (2014).

Starting with the ash by size data for a set of flow samples from a laboratory Reflux Classifier fractionation experiment, the first step was to sort the data within each size interval into order of increasing ash value. In cases where two or more flow increments had identical ash, they were combined and treated as one.

The measured ash value is the average of all material in that flow and size increment. It was assumed that this ash value applies to material at the mid-point of the increment. So for each size fraction the cumulative mass of material to the mid-point of each flow fraction was calculated by adding half the mass of that interval to the total cumulative mass of the previous increments.

The next step was to use Equation 1 to convert each measured ash value into a density. The densities at zero cumulative mass and at the total cumulative mass were estimated by linear extrapolation from the two nearest adjacent data points. This then gave a set of cumulative mass versus incremental density data. By interpolation, the mass of material in a chosen density interval was then found and from this the mass fraction was calculated. In Case Study 1 the density intervals were around the cut points 1300, 1350, 1400, 1500, 1600, 1700, 1800, 2000, and 2200 kg/m³. Case Study 2 used 2100 kg/m³ instead of 2200 kg/m³. The above steps were repeated for each size fraction of all the input and output streams.

For a steady state system with no losses, reactions, breakage or agglomeration, the total mass flow rate of material in each density and size fraction in the feed should equal the total rate out in the combined product streams. Invariably the data did not appear to satisfy this requirement and this prevents direct calculation of an unambiguous partition value at each density. In order to overcome this ambiguity, the data was then mass-balance reconciled, in the same manner as would be used for any other set of experimental mass distribution data (Galvin et al., 1995). All results presented in Section 4 are based on the reconciled data unless specified otherwise.

The above procedure uses the full information obtained from the screening of each flow increment into a number of size fractions. We refer to this as a “double fractionation” (DF) i.e. the batch elutriation into flow increments is the first fractionation, followed by the screening into multiple size fractions as the second fractionation. However, it was of interest to test how necessary it is to perform the size fractionation step prior to performing the ash measurements. Thus the above analysis was also repeated using only the total mass and average ash value of the +0.250 mm combined size fraction from each flow increment. In effect, this simulates what would have happened if only one ash value was measured per flow increment, and thus represents a significant saving in the cost and time of sample screening and analysis. We refer to this as a “single fractionation” (SF).

Once mass balance reconciliation had been performed, the density partition performance of the unit operation could be determined. Here we define the partition number as the probability of a particle in a given size and density interval of the feed exiting in the specified product stream. It was sometimes convenient to fit a standard partition curve to the data to enable determination of the density cut point D_{50} (kg/m³) and Ecart probable Ep values. The so-called Whiten equation used was (Wood, 1990):

$$P = \frac{1}{1 + \exp\left(1.098 \frac{D - D_{50}}{1000Ep}\right)} \quad (3)$$

where P is the partition to floats of particles of density D (kg/m³). The best fit was found by varying D_{50} and Ep to minimise the sum of the square of the errors between the model and experimental partition values.

4.0 Results and Discussion

The results of 12 laboratory scale Reflux Classifier experiments are presented and analysed in this paper. These are summarised in Table 2. For Case Study 1 (a laboratory-scale continuous Reflux Classifier), the feed, product and reject streams were each fractionated using 70 wt.% glycerol solution. A repeat run was performed on the product sample to collect multiple small flow fractions at the end of the run in order to obtain greater accuracy in measuring the distribution of high density material (see Figure 4b). All calculations presented for Case Study 1 are based on the repeat product run, rather than the original. For Case Study 2 (an industrial single spiral stage), the feed, product, middlings and reject streams were all fractionated in the batch Reflux Classifier using both water and 70 wt.% glycerol solution.

In Table 2 it can be seen that for Case Study 1, the mass of material lost during the batch Reflux Classifier fractionation experiment was less than 3.3 wt.% in all cases. The greatest loss was from the reject stream. The head ash was always slightly lower after the Reflux Classifier test, compared with the sink-float method. Hence, the lost material is most likely to be clay slimes that escaped through the 55 μm filter sock. Overall the rates of attrition in Case Study 1 were low.

Table 2: Summary of batch laboratory Reflux Classifier fractionation experiments. The head ash values of the +0.250 mm material recovered after the sink-float and laboratory Reflux Classifier (RC) fractionation are shown.

Case Study	Fluidisation Medium	Stream	Run Time (h)	Mass Lost in RC Test (wt.%)	Sink- Float Head Ash (wt.%)	Post-RC Head Ash (wt.%)
Case Study 1 (RC)	70 wt.% Glycerol	Feed	8	1.1	22.7	20.3
		Product	11	1.6	8.4	7.8
		Product (Repeat)	3	2.1	8.4	7.7
		Reject	6	3.3	45.5	42.6
Case Study 2 (Spiral)	70 wt.% Glycerol	Feed	10	3.6	40.7	37.0
		Product	7	0.5	8.2	7.9
		Middling	11	5.7	56.1	55.3
		Reject	4	3.1	79.9	80.1
	Water	Feed	4.5	19.0	40.7	24.3
		Product	3.5	1.7	8.2	7.5
		Middling	4	23.8	56.1	47.8
		Reject	3	23.2	79.9	77.4

In contrast, the coal collected in Case Study 2 was known at the coal preparation plant to be one that suffered significant slimes problems due to clay breakdown. When the samples were initially wet screened at 0.130 mm, a high level of slimes was observed. During the batch Reflux Classifier experiments, slimes were also observed continually exiting the unit throughout the run, even at the higher flow rates when all clay fines initially present in the sample should have already been elutriated. This is evidence of progressive clays breakdown occurring during the runs. When water was used as the fluidising medium, the mass loss often exceeded 20 wt.% (Table 2). Hence no further analysis was performed on the flow samples generated using water as the fluidising medium.

However, when 70 wt.% glycerol solution was used in Case Study 2, the mass of material lost during the batch fractionation experiment was reduced to 5.7 wt.% or less, even though the duration of these tests was often twice as long as the water-only tests. This is consistent with previous work which indicates that glycerol reduces attrition and breakage (Hunter et al., 2014). This is likely because of the lower fluidisation rates required to convey particles into the channels when a viscous liquid is used, and also because of the cushioning effect of the viscous liquid during inter-particle impacts. This dramatic reduction in attrition is another benefit of using more viscous solutions to perform these batch elutriation tests.

A small amount of break down does not necessarily compromise the mass-balance data reconciliation technique. Provided that all material in a given density fraction experiences the same level of break-down regardless of which stream sample it was in, then the material remaining should still satisfy the assumption that input equals output. In reality, levels of breakage during the batch laboratory tests may vary due to variations in the run times, range of flow rates, and levels of collision with neighbouring particles. However, mass-balance reconciliation is the only basis on which to proceed to analyse the data, as otherwise it is impossible to determine unambiguous values of the partition coefficients.

Each flow fraction was screened and the ash value of each size interval measured. From this data the cumulative yield-ash curves for each sample were calculated. Figures 4 and 5 show

the comparison of these results with the sink-float data for Case Studies 1 and 2 respectively. As found previously for +0.250 mm coal particles in $z = 6$ mm channels, using 70 wt.% glycerol solution gave very close agreement with the sink-float data (Hunter et al., 2014). It is this close agreement which provides the foundation on which to accurately determine the partition curves. The only consistent discrepancy was that the head ash was usually slightly lower than the value obtained by the sink-float method (see also Table 1). This is attributed to the loss of a small amount of high mineral matter clay fines, as discussed above.

Figure 4: Cumulative yield versus cumulative ash for the +0.250 mm size fraction of a) Feed, b) Product and c) Reject streams from Case Study 1. Results using 70 wt.% glycerol to perform batch fractionation in the laboratory Reflux Classifier (Lab. RC) are compared with the sink-float method.

Figure 5: Cumulative yield versus cumulative ash for the +0.250 mm size fraction of a) Feed, b) Product, c) Middling and d) Reject streams from Case Study 2. Results using 70 wt.% glycerol solution in the laboratory Reflux Classifier (Lab. RC) are compared with the sink-float method.

The close agreement between the original and duplicate yield ash curve for the product from Case Study 1 (Figure 4b) demonstrates the reproducibility of the Reflux Classifier elutriation method. Note also that the repeat experiment was performed in 3 h, compared to the original elutriation experiment that took 11 h (Table 2). This demonstrates that these batch elutriation experiments can be performed much more quickly than in the cautious approach adopted in most of these runs (see also Hunter et al., 2014). This is an important consideration if the method is to become a viable alternative to the sink-float method.

4.1 Case Study 1 Results

The fractionation data from the batch Reflux Classifier were analysed in accordance with the method outlined in Section 3. Each Reflux Classifier flow fraction was screened into a number of fractions which were then weighed and their ash value measured. The +0.125 mm data were all analysed to obtain a set of cumulative mass versus density curves. From these the mass fraction was interpolated in each of 10 density intervals. This “raw” density distribution information for the three streams was then mass-balance reconciled to obtain a consistent set of feed, product and reject mass flow rates, and size by density distributions. From this, the partition to floats in each density interval was calculated for all size fractions, as well as the overall partition for the combined +0.250 mm material. Appendix A shows the raw data and results of these calculations.

In size and density intervals where there was very little material, the numerical routine would sometimes infer small amounts of solids in order to satisfy the material balances. Very small errors in these calculations can cause spurious partition values. These spurious results were filtered out by not calculating partition numbers for any size/density interval which contained less than 0.05 wt.% of the solids in the feed stream. The final results are shown in Figure 6.

Figure 6: Partition curves for each size fraction from Case Study 1 based on the laboratory Reflux Classifier method. The overall partition of the +0.250 mm size fraction based on combining the double fractionation (DF) data is also shown. Lines show the best fit of Equation 3 to each set of data.

Table 3 shows how the fitted density cut point D_{50} and E_p values from Equation 3 varied. The Reflux Classifier being operated in Case Study 1 had a density cut point that decreased steadily from 1712 down to 1338 kg/m³ with an increase in particle size from 0.125 up to greater than 1.40 mm. This is consistent with previously observed performance of the narrow channel Reflux Classifiers (Galvin, 2012). Previous work has also found that E_p decreases steadily from 0.07 down to 0.03 with an increase in particle size from 0.250 up to 2.00 mm (Galvin, 2012).

The variation in the fitted E_p values in Table 3 was not systematic. The -0.180 +0.125 mm had a relatively large E_p of 0.095, but the rest of the size fractions hovered around $E_p = 0.05$, with the exception of the -0.355 +0.250 mm size fraction which had a surprisingly low E_p of only 0.023. The reason for this low E_p value is because, as seen in Figure 6, some of the +0.250 mm partition data appears to have prematurely dropped to zero at a density of 1650 kg/m³. This sudden drop can be explained by examining the average ash values of the size increments collected from the Product stream (Table A.5). It can be seen that across all sizes above 0.250 mm, no sample had an ash value greater than 30 wt.%. Hence the average density as calculated by Equation 1 was always less than 1581 kg/m³. Hence, these data appear to imply that there were no particles denser than 1600 kg/m³ in any of the +0.250 mm

product flow increments, which explains the zero partition values. However, in reality we know that the Flow 8 samples with an *average* ash value in the range 25 – 30 %, must have included some higher density particles with *individual* ash values higher than the average. From the raw sink-float data (Table A.1), only 0.5 wt.% of the product was in the 1600 – 1700 kg/m³ density interval. The laboratory Reflux Classifier method can only distinguish this small number of dense particles if they are collected as a separate sub-sample by using extra flow steps towards the end of the product fractionation experiment. This is the reason a repeat fractionation experiment was performed on the product sample. This gave some improvement, but did not eliminate the problem. However, given that even the feed had only 5 wt.% of particles in this density range, there were extremely few of these particles in the system, and so the partition value calculated by either method will have a large uncertainty.

Table 3: Best fit of Equation 3 D_{50} and Ep parameters to the sink-float and laboratory Reflux Classifier (RC) fractionation data for Case Study 1. Table shows both single fractionation (SF) and double fractionation (DF) results.

Method	Size (mm)		D_{50} (kg/m ³)	Ep
	Upper	Lower		
RC DF (individual size fractions)		1.40	1338	0.050
	1.40	1.00	1386	0.051
	1.00	0.710	1437	0.046
	0.710	0.500	1494	0.052
	0.500	0.355	1534	0.044
	0.355	0.250	1563	0.023
	0.250	0.180	1620	0.049
	0.180	0.125	1712	0.095
RC DF		0.250	1465	0.065
RC SF		0.250	1463	0.059
Sink-Float		0.250	1473	0.070

Sink-float fractionation was performed on the +0.250 mm size fraction of the three streams. Mass-balance data reconciliation was performed on this sink-float data and the partition values calculated (shown in Figure 7). Equation 3 was best fitted to this data and gave $D_{50} = 1473$ kg/m³ and $Ep = 0.070$ (Table 3). The best fit to the laboratory Reflux Classifier +0.250 mm data gave $D_{50} = 1465$ kg/m³ and $Ep = 0.065$, so very close agreement. Looking at the composite +0.250 mm partition data in Figure 8, it can be seen that the only point that deviates significantly is the value at a density of 1650 kg/m³ which again shows the premature drop to zero.

Figure 7: Case Study 1 partition curves for the combined +0.250 mm size fraction. Comparison of laboratory Reflux Classifier (RC) double fractionation (DF) and single fractionation (SF) method with the sink-float result. Curves show best fit of Equation 3. Solid and dashed error bars show the span in the partition values as determined by applying Equations 4a-c to the sink-float and Reflux Classifier double fractionation data respectively.

The single fractionation result, shown in Table 3 and Figure 7, was satisfactory but not quite as good as the double-fractionation result. The fitted D_{50} value was slightly lower (1463 kg/m³ compared to 1465 kg/m³ for the double fractionation) and the Ep value sharpened from 0.065 to 0.059. However, overall this result is still quite close to the sink-float values of $D_{50} = 1473$ kg/m³ and $Ep = 0.070$. The fact that there is only a slight improvement going from single to double fractionation is unsurprising given the very close agreement of the single-fractionation yield-ash curves with the sink-float results (Figure 4).

In order to put these results into context, it is important to consider how much certainty there is in the two sets of partition values that are being compared. For the sink-float samples, the head ashes of the +0.250 mm material in the feed, product and reject streams were 22.7, 8.4 and 45.5 wt.% respectively (Table 2). Hence based on the two-product rule (Wills & Napier-Munn, 2006), the yield to product is estimated as $Y = (45.5 - 22.7)/(45.5 - 8.4) = 0.615$ i.e. 61.5 kg of product per 100 kg of feed. Using this flow split, there are three different routes that can then be used to calculate the partition values from the raw sink-float data:

$$\text{Partition} = \text{Product/Feed} \quad (4a)$$

$$\text{Partition} = \text{Product}/(\text{Product} + \text{Reject}) \quad (4b)$$

$$\text{Partition} = (\text{Feed} - \text{Reject})/\text{Feed} \quad (4c)$$

The span of these three results is shown in Figure 7 (solid error bars) as a rough indication of the uncertainty in each of the sink-float partition values. The average spread was ± 0.10 partition units. Note that this deviation represents the inconsistency in the mass balancing of the raw sink-float data from the three streams, rather than the actual error bar of the partition curve. The application of the mass balancing routine leads to a contraction of the error in the partition number (Galvin et al., 1995) hence the final partition curve is satisfactory. The errors could be due to random flow variations and sampling errors.

Similarly, for the samples fractionated in the Reflux Classifier, the average ash contents of the +0.25 mm material in the feed, product and reject samples were 20.3, 7.7 and 42.6 wt.% respectively (Table 2), giving a two-product predicted yield of 0.638. Based on this and using Equations 4a to 4c, the spread in partition values based on the “raw” Reflux Classifier density distributions was also calculated and included in Figure 7 (dashed error bars). The average spread of this data was ± 0.15 partition units. However, most of the large spreads occurred at very high densities, due to the large error in partition calculations that can occur when comparing the small amount of high density solids in the feed and product samples. In the density range of interest between 1200 and 1800 kg/m³, the average span in the partition values was only ± 0.10 partition units, similar to the sink-float uncertainty. As can be seen in Figure 7, the two sets of partition results agree with each other within the span of their mutual uncertainty.

4.2 Case Study 2 Results

Case Study 2 involved a set of feed, product, middling and reject samples collected from around a single spiral start at a coal preparation plant in the Hunter Valley. As described in Section 2, sink-float fractionation was performed on a representative sub-sample of each stream and the density intervals were then screened into five size fractions prior to ash measurements. From the mass-balanced results, the partition performance of the spiral was then calculated.

Figure 8 shows the partition to product of the different size fractions. The lines show the best fit of Equation 3 to the sink-float only data. The fitted density cut-point and Ecart probable values are summarised in Table 4. Equation 3 gives a reasonably good fit to the data, although there is considerable scatter in the -0.250 +0.125 mm size range. Both Figure 8 and Table 4 clearly show the deterioration in the sharpness of partition to product with decreasing particle size. This is consistent with typical published results for spirals (Sanders et al., 2007). The density cut point also shifts to lower values with decreasing particle size.

Figure 8: Partition to product performance of the spiral in Case Study 2 based on mass-balance reconciled data. Solid symbols (■, ▲, ●, ✕, ◆) show the sink-float data. Open symbols (□, △, ◇, +, ○) show the laboratory Reflux Classifier data. Lines show the best fits of Equation 3 to only the sink-float data. Fitted parameters are reported in Table 4.

Table 4: Density cut-point (D_{50}) and Ecart probable (Ep) values from the best fits of Equation 3 to the Case Study 2 mass-balance reconciled data.

Size Range		Sink-Float		Reflux Classifier Double Fractionation		Reflux Classifier Single Fractionation	
Lower (mm)	Upper (mm)	D_{50} (kg/m ³)	Ep	D_{50} (kg/m ³)	Ep	D_{50} (kg/m ³)	Ep
Partition to Product							
1.00		1764	0.08	1783	0.06		
0.700	1.00	1721	0.09	1684	0.13		
0.500	0.700	1663	0.16	1600	0.18		
0.250	0.500	1539	0.19	1505	0.21		
0.125	0.250	1486	0.25	1467	0.30		
0.250		1631	0.17	1596	0.18	1563	0.17

Figure 9 shows the partition to the combined product plus middlings. This shows similar trends of poorer sharpness and decreasing density cut point with decreasing particle size. The Equation 3 fitted values of D_{50} and Ep were found to be very sensitive to the arbitrarily assumed average density of the sinks +2000 kg/m³ material. Hence no analysis of the curve fit parameters was undertaken for the partition to combined product plus middlings, although the curves are still shown in Figures 9 and 10 to guide the eye.

Figure 9: Partition to combined product plus middlings in Case Study 2 based on mass-balance reconciled data. Solid symbols (■, ▲, ●, ✕, ◆) show the sink-float data. Open symbols (□, △, ◇, +, ○) show the laboratory Reflux Classifier data. Lines show the best fits of Equation 3 to only the sink-float data, with an assumed average density of 2150 kg/m³ for the 2000 kg/m³ sinks fraction.

The ash-density relationship was established based on the sink-float data (Table 1). Using this correlation, all the fractionation data from the laboratory Reflux Classifier experiments using 70 wt.% glycerol solution were converted into density distribution data, as per the method explained in Section 3.2. Mass-balance data reconciliation across the four streams was then attempted. There were initial numerical instability problems in performing the mass balancing. This was because the upper (+1.40 mm) size interval contained less than 2 % of the mass of material in the feed. Hence small errors in these measurements created large relative errors that dominated the mass balancing. This problem was overcome by merging together the +1.40 mm and -1.40 mm +1.00 mm data into a single +1.00 mm size fraction, which then accounted for 8.2 % of the feed. Once a consistent set of density distribution data were generated, the partition values were calculated.

Figure 8 shows the partition to product values obtained from the laboratory Reflux Classifier method compared with the sink-float values. To avoid cluttering the plot, the best fits of Equation 3 to the Reflux Classifier data are not included in Figure 8, but the parameters are reported in Table 4. The laboratory Reflux Classifier data shows similar trends and approximately matches the sink-float data at densities below 1800 kg/m³, but with a tendency to under predict the partition number by 10 to 20 % (hence the lower fitted D_{50} values in Table 4). With the exception of the -0.250 +0.125 mm size fraction, all of the laboratory Reflux Classifier partition values dropped to nearly zero at densities greater than 1800 kg/m³, whereas the sink-float data had distinct non-zero values for the partition coefficient in both the 1800 – 1900 and 1900 – 2000 kg/m³ density intervals.

Similarly Figure 9 also shows the partition to combined product plus middlings obtained from the laboratory Reflux Classifier method compared with the sink-float data. Again there

is approximate agreement between the two sets of results, but with a tendency for the laboratory Reflux Classifier results to lie below the sink-float values.

Due to the considerable scatter in the $-0.250 +0.125$ mm results in both the sink-float and laboratory Reflux Classifier data, it was decided to focus on the partition curves for the combined $+0.250$ mm size fraction. Figure 10 compares the laboratory Reflux Classifier double fractionation (DF) and single fractionation (SF) results with the sink-float method. Figure 10 also shows the results of fitting Equation 3 through each set of combined $+0.250$ mm partition to product data; the curve fit parameters are reported in Table 4.

Because this spiral had three product streams, the two-product formula could not be used. Their relative flowrates as determined by mass-balance reconciliation of the sink-float data were 46.1 % yield to product, 24.5 % yield to middlings and 29.3 % yield to reject. To gain some indication of the uncertainty of the raw sink-float partition values, these relative flowrates were used in Equations 4a-c, with the middlings flow lumped together with either the product or reject flow, depending on which partition value was being calculated. The average span was ± 0.03 partition units based on the sink-float data. Similarly using the Reflux Classifier “raw” density distribution data, mass balancing gave 42.7 % yield to product, 25.1 % yield to middlings and 32.2 % yield to reject. Using this in Equations 4a-c gave an average span of ± 0.11 partition units. These spans are shown as error bars on Figure 10.

In Case Study 2, going from a single to double fractionation has not significantly improved the quality of the laboratory Reflux Classifier data. Whilst the fitted curve through the double fractionation data lies closer to the fitted curve through the sink-float data, many of the individual data points are further from the sink-float data than some of the single-fractionation data points. However, the curve fit through the double fractionation data lies within 0.05 partition units of the sink-float curve fit result across most of the density range. This is comparable to the span of ± 0.03 partition units variability in the raw sink-float data and within the span of ± 0.11 partition units variability in the “raw” Reflux Classifier fractionation data itself. The D_{50} values on individual size fractions had a maximum deviation of -64 kg/m^3 from the sink-float results, with an average deviation of only -27 kg/m^3 (Table 4). For the combined $+0.250$ mm size fraction, the D_{50} was within 35 kg/m^3 of the sink-float value and the Ep was within 0.01 of the sink-float value. This is excellent agreement, particularly when it is recalled that this coal suffered significant water break down, particularly of composite particles which are the critical to determining partition performance at the intermediate densities of most interest (Table 2).

Figure 10: Combined +0.250 mm partition curves for Case Study 2. Plot compares sink-float results with laboratory Reflux Classifier (RC) double fractionation (DF) and single fractionation (SF) methods. Lines show best fits of Equation 3. Solid and dashed error bars show span of results from applying Equations 4a-c to the sink-float and Reflux Classifier double fractionation data respectively.

It should also be noted that the 1500 – 1600 kg/m³, 1600 – 1700 kg/m³, 1700 – 1800 kg/m³ and 1800 – 1900 kg/m³ density intervals each contained less than 3 wt.% of the feed mass. When there is so little near-density material in the feed, all methods will struggle for accuracy since small sampling and measurement errors cause a large uncertainty in the calculated partition values. Figure 11 compares the mass frequency distributions measured by the two methods for the product, middling and reject samples (feed is not shown to avoid overcrowding the plot). The laboratory Reflux Classifier results were in close agreement with the sink-float data. The only noteworthy discrepancy was the failure of the laboratory Reflux Classifier method to measure the small amounts of high density material in the product sample (the same problem also occurred in Case Study 1). Hence, when the purpose of the laboratory Reflux Classifier fractionation is to measure partition performance, it is very important to collect additional flow increments of the small amounts of high density material that exit towards the end of the fractionation of the product sample. This may involve collecting flow samples as small as 20 g from an original 3000 g charge. Using a curve-fit also helps to minimise this problem as it smooths out the premature drop to zero partition value which can occur at high densities (Figure 10). A similar improvement was seen using the curve fit in Case Study 1 (Figure 7).

Figure 11: Mass frequency distribution (weight percentage of material in an interval divided by the width of the density interval) for the product, middling and reject samples from Case Study 2. Comparison of results from the raw sink-float and Reflux Classifier (RC) data.

5.0 Conclusions

This work has confirmed the benefits of using viscous glycerol solutions to fractionate fine coal particles in a laboratory Reflux Classifier. The laminar flow and high shear rate suppress the effects of particle size and thus promote elutriation on the basis of density. The resulting fractionations closely match the yield-ash curves obtained by the sink-float method.

By performing these measurements on samples of the feed, product and reject streams around a unit, it is then possible to determine the partition performance. However, unlike the sink-float method, the Reflux Classifier cannot fractionate each sample at identical density cut points. Hence, a methodology has been developed for inferring the sample density distribution from the cumulative yield-ash data. This method has been proven to work very well under ideal conditions where the samples are representative and there is a reasonable amount of “near-density” material in the sample from which to accurately determine the partition curve (Case Study 1). Even with samples that suffered significant water breakdown of particles due to the presence of clays, use of the glycerol solutions reduced the level of breakdown sufficiently to still enable a reasonable determination of the partition curve (Case Study 2).

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Appendix A: Case Study 1 - Raw Data and Sample Calculations

This appendix presents the raw data and a full set of sample calculations for Case Study 1. For Case Study 2 refer to Iveson et al. (2014). Table A.1 reports the raw sink-float data for the +0.250 mm size fraction of the feed, product and reject samples in Case Study 1. This data was subject to mass-balance reconciliation using the methodology and software described in Galvin et al. (1995). This gave relative stream masses of Feed = 100, Product = 64.1 and Reject = 35.9 mass units.¹ Table A.1 also reports the mass fraction in each density interval after mass-balance reconciliation. It can be seen that most adjustments were less than 1 wt.%. The partition to product values were calculated based on this mass-balanced data, and the results are shown in the final column of Table A.1 e.g. for the Floats 1.3 Sp Gr fraction, partition to product = $x_P P / (x_F F) = 53.7(64.1) / 35.1 / 100 = 0.98$.

¹ For comparison, as noted in Section 4.1, based on the head ash of the +0.25 mm material in each stream, the two-product rule predicts relative flows of Feed = 100, Product = 61.5 and Reject = 38.5 mass units.

Table A.1: Sink-float data for +0.250 mm size fraction in Case Study 1. Comparison of raw incremental mass fraction and ash value with mass-balance reconciled mass fraction. Partition value based on reconciled mass flowrates of Feed = 100, Product = 64.1 and Reject = 35.9 mass units.

Density Fraction		Feed			Product			Reject			Partition
		Raw-Data		Mass-Balanced	Raw-Data		Mass-Balanced	Raw-Data		Mass-Balanced	Mass-Balanced
Sink (Sp Gr)	Float (Sp Gr)	Mass (wt.%)	Ash (wt.%)	Mass (wt.%)	Mass (wt.%)	Ash (wt.%)	Mass (wt.%)	Mass (wt.%)	Ash (wt.%)	Mass (wt.%)	
	1.30	32.9	2.7	35.1	53.7	3.5	53.7	1.9	4.3	1.9	0.980
1.30	1.35	12.5	7.6	13.5	20.5	8.3	19.3	3.4	9.0	3.3	0.911
1.35	1.40	8.6	12.8	8.2	9.8	13.9	10.3	4.3	13.9	4.4	0.809
1.40	1.45	8.7	17.4	7.8	7.6	17.8	8.1	7.0	18.1	7.2	0.668
1.45	1.50	6.9	22.0	6.2	4.5	21.9	4.7	8.4	22.2	8.8	0.488
1.50	1.55	4.9	26.5	5.2	2.5	26.0	2.4	11.0	27.0	10.2	0.301
1.55	1.60	3.1	31.5	3.2	0.8	30.0	0.8	7.8	31.4	7.5	0.156
1.60	1.70	5.2	38.5	4.8	0.5	35.2	0.5	11.7	37.9	12.4	0.069
1.70	1.80	2.7	46.2	2.5	0.1	45.5	0.1	6.6	45.2	6.9	0.022
1.80	1.90	1.0	53.7	1.2	0.0	47.2	0.0	6.0	52.5	3.3	0
1.90	2.00	2.0	58.5	1.6	0.0	0.0	0.0	4.0	59.7	4.6	0
2.00		11.4	82.9	10.6	0.0	0.0	0.0	27.8	82.5	29.4	0
Head Ash (wt.%)		22.7			8.4			45.5			

Tables A.2-A.4 shows the raw data from the laboratory Reflux Classifier double fractionation of the Feed, Product and Reject samples respectively. To transform this data into a density distribution, the first step is to sort the flow increments within each size fraction in order of increasing ash value. In cases where two flow increments had identical ash, they were combined and treated as one.

Table A.2: Raw data from Case Study 1 Feed sample as measured by laboratory Reflux Classifier using 70 wt.% glycerol solution.

Size (mm)	FEED	Flow 1	Flow 2	Flow 3	Flow 4	Flow 5	Flow 6	Flow 7	Remains
+1.40	Mass (g)	0.8	0.9	2.3	25.0	9.4	10.6	26.4	18.0
	Ash (%)	9.6	5.2	3.2	4.7	10.1	16.0	25.0	55.4
-1.40	Mass (g)	5.4	6.0	13.1	159.9	67.0	76.9	116.2	66.9
+1.00	Ash (%)	4.7	4.0	2.6	4.2	9.1	16.9	27.8	58.4
-1.00	Mass (g)	11.8	9.8	16.3	252.8	151.0	116.1	155.2	105.6
+0.710	Ash (%)	4.2	4.0	2.2	3.2	9.0	17.8	30.6	66.4
-0.710	Mass (g)	15.5	8.1	13.4	234.7	179.0	99.2	132.6	104.0
+0.500	Ash (%)	4.0	4.1	1.8	2.8	8.6	18.5	34.2	74.8
-0.500	Mass (g)	14.5	5.4	14.5	177.5	140.9	68.0	73.4	67.3
+0.355	Ash (%)	3.9	4.1	1.6	2.4	9.2	20.4	39.7	82.1
-0.355	Mass (g)	11.1	4.3	32.7	151.0	109.6	45.7	38.6	48.5
+0.250	Ash (%)	3.9	4.5	1.7	3.3	12.1	26.0	51.1	85.7
-0.250	Mass (g)	11.1	3.4	95.7	124.0	74.6	20.8	25.8	29.2
+0.180	Ash (%)	4.4	5.3	2.9	6.6	19.4	38.2	75.6	85.1
-0.180	Mass (g)	6.6	3.1	49.1	37.8	27.0	11.1	20.5	15.3
+0.125	Ash (%)	5.9	5.3	4.8	13.7	32.7	64.2	88.5	82.3
Total Mass (g):		76.8	41.0	237.1	1163.4	758.5	448.4	588.7	454.8
Average Ash (wt%):		4.3	4.3	2.9	3.9	11.3	21.1	37.1	72.8
Overall Feed Ash (wt.):									20.9

The +1.40 mm size fraction of the Feed sample has been chosen for the purpose of sample calculations. The initial sorting of this data in order of increasing ash value is shown in the second and third rows of Table A.5.

Table A.5: Initial sorting of the +1.40 mm Feed data in order of increasing ash values.

Sorted	Lower	1	2	3	4	5	6	7	8	Upper
Ash (wt.% a.d.)		3.2	4.7	5.2	9.6	10.1	16.0	25.0	55.4	
Mass (g)		2.3	25.7	0.9	0.8	9.4	10.6	26.4	18.0	
Cumulative Mass to Interval Midpoint (g)	0	1.2	15.2	28.5	29.3	34.4	44.4	62.9	85.1	94.1
Density at Interval Midpoint (kg/m ³)	1255	1257	1275	1280	1332	1338	1408	1515	1874	2020

For each size fraction the cumulative mass of material to the mid-point of each flow fraction was calculated by adding half the mass of that interval to the total cumulative mass of the previous increments. Row 4 of Table A.5 shows the results. For example, the cumulative mass up to the midpoint of the 3rd increment was calculated by:

$$2.3 \text{ g} + 25.7 \text{ g} + (0.9 \text{ g})/2 = 28.5 \text{ g}$$

Row 4 also includes a lower bound of zero mass and an upper bound which is the total mass of +1.40 mm material in the sample (94.1 g).

Row 5 in Table A.5 shows the densities of particles at each interval midpoint calculated from Equation 1 using the increment ash. So for example, at the midpoint of the 3rd increment in Table A.5 the density was:

$$D = 1219 + (2401 - 1219)(5.2/100) = 1280 \text{ kg/m}^3$$

The densities at zero cumulative mass and at the total cumulative mass were estimated by linear extrapolation from the adjacent two data points. For example, Row 5 in Table A.5 shows that at a cumulative mass of 1.2 g the density is 1257 kg/m³ and at 15.2 g the density is 1275 kg/m³. So it can be extrapolated that the very first material at zero cumulative mass had a density:

$$1257 \text{ kg/m}^3 - (1.2 \text{ g} - 0 \text{ g})(1275 \text{ kg/m}^3 - 1257 \text{ kg/m}^3)/(15.2 \text{ g} - 1.2 \text{ g}) = 1255 \text{ kg/m}^3.$$

Similarly the density of material at 94.1 g (total mass of +1.4 mm material in the Feed) is extrapolated to be:

$$1874 \text{ kg/m}^3 + (94.1 \text{ g} - 85.1 \text{ g})(1874 \text{ kg/m}^3 - 1515 \text{ kg/m}^3)/(85.1 \text{ g} - 62.9 \text{ g}) = 2020 \text{ kg/m}^3$$

Figure A.1 shows the resultant plot of cumulative mass versus density.

Figure A.1: Cumulative mass versus density for the +1.40 mm size fraction of Case Study 1 Feed. Crosses show the interpolated cumulative mass at the chosen density cut points.

By interpolation on Figure A.1, the cumulative mass of material up to an arbitrary density cut point could be determined. The chosen density intervals used in Case Study 1 started from 1219 kg/m³ (the lowest possible density according to Equation 1), and then stepped up to 1300, 1350, 1400, 1500, 1600, 1700, 1800, 2200 and finished at 2400 kg/m³. The interpolated cumulative masses at these values are shown on Figure A.1 and in Row 4 of Table A.6. From this, the mass of material in each of the desired density intervals was calculated, as shown in Row 5.

Table A.6: Calculated masses of material in each specified density interval of the +1.40 mm fraction of the Feed from Case Study 1.

Interval	1	2	3	4	5	6	7	8	9	10
Lower Density (kg/m ³)	1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
Upper Density (kg/m ³)	1300	1350	1400	1500	1600	1700	1800	2000	2200	2400
Interpolated Cumulative Mass to Upper Density (g)	28.8	36.1	43.2	60.4	68.2	74.4	80.5	94.1	94.1	94.1
Mass in Interval (g)	28.8	7.3	7.2	17.1	7.8	6.2	6.2	13.6	0.0	0.0

The above steps were repeated for each size fraction of all the input and output streams. Each increment mass was then divided by the total mass of the sample to give the mass fraction. The resulting mass percentages in each interval are shown in Tables A.7-A.9 for the Feed, Product and Reject respectively.

Table A.7: Mass percentage in each size by density interval for the Case Study 1 Feed as inferred from the laboratory Reflux Classifier fractionation experiments.

FEED	Density Range (kg/m ³)									
Size Range (mm)	1219 1300	1300 1350	1350 1400	1400 1500	1500 1600	1600 1700	1700 1800	1800 2000	2000 2200	2200 2500
+1.40	0.76	0.19	0.19	0.45	0.21	0.16	0.16	0.33	0.03	0.00
-1.40 +1.00	5.29	0.98	1.04	2.00	1.30	0.67	0.67	1.34	0.28	0.00
-1.00 +0.710	8.75	1.80	1.70	2.68	2.08	0.82	0.82	1.64	1.43	0.00
-0.710 +0.500	8.62	1.89	1.58	2.22	1.66	0.89	0.65	1.31	1.31	0.75
-0.500 +0.355	6.60	1.36	1.05	1.59	0.82	0.77	0.37	0.74	0.74	0.85
-0.355 +0.250	5.69	0.84	0.68	1.25	0.61	0.38	0.38	0.59	0.57	0.73
-0.250 +0.180	4.62	0.87	0.87	1.14	0.57	0.44	0.14	0.28	0.73	0.55
-0.180 +0.125	1.54	0.32	0.27	0.38	0.38	0.15	0.14	0.28	0.37	0.69
+0.125	41.88	8.25	7.37	11.72	7.62	4.28	3.33	6.50	5.45	3.58

Table A.8: Mass percentage in each size by density interval for the Case Study 1 Product as inferred from the laboratory Reflux Classifier fractionation experiments.

PRODUCT	Density Range (kg/m ³)									
Size Range (mm)	1219 1300	1300 1350	1350 1400	1400 1500	1500 1600	1600 1700	1700 1800	1800 2000	2000 2200	2200 2500
+1.40	1.16	0.17	0.14	0.08	0.00	0.00	0.00	0.00	0.00	0.00
-1.40 +1.00	5.48	2.10	1.13	0.63	0.18	0.00	0.00	0.00	0.00	0.00
-1.00 +0.710	9.33	3.83	2.82	1.65	0.33	0.00	0.00	0.00	0.00	0.00
-0.710 +0.500	13.01	2.01	3.01	2.86	0.64	0.00	0.00	0.00	0.00	0.00
-0.500 +0.355	10.29	1.88	1.91	2.23	0.66	0.00	0.00	0.00	0.00	0.00
-0.355 +0.25	7.27	2.16	1.31	1.90	0.66	0.00	0.00	0.00	0.00	0.00
-0.250 +0.180	7.04	2.15	1.28	1.64	0.69	0.10	0.00	0.00	0.00	0.00
-0.180 +0.125	2.71	1.17	0.62	0.94	0.61	0.14	0.07	0.00	0.00	0.00
+0.125	56.30	15.47	12.23	11.93	3.77	0.24	0.07	0.00	0.00	0.00

Table A.9: Mass percentage in each size by density interval for the Case Study 1 Reject as inferred from the laboratory Reflux Classifier fractionation experiments.

REJECT	Density Range (kg/m ³)									
Size Range (mm)	1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
	1300	1350	1400	1500	1600	1700	1800	2000	2200	2500
+1.40	0.23	0.31	0.69	1.64	0.90	0.49	0.34	0.37	0.31	0.00
-1.40 +1.00	0.56	0.81	1.65	5.88	4.45	2.20	1.50	1.82	1.31	0.26
-1.00 +0.710	0.45	0.63	0.81	4.96	5.55	2.91	2.26	2.83	2.24	1.86
-0.710 +0.500	0.30	0.31	0.31	2.14	3.97	2.62	2.08	2.42	2.58	2.79
-0.500 +0.355	0.16	0.13	0.13	0.61	1.88	1.83	1.10	1.56	1.92	2.35
-0.355 +0.250	0.09	0.11	0.11	0.23	0.66	1.28	0.96	0.97	0.96	2.87
-0.250 +0.180	0.00	0.07	0.14	0.28	0.28	0.32	0.77	0.86	0.71	2.79
-0.180 +0.125	0.00	0.01	0.08	0.15	0.15	0.15	0.15	0.42	0.78	2.27
+0.125	1.80	2.37	3.91	15.88	17.84	11.79	9.17	11.24	10.81	15.19

The average ash values of the +0.125 mm fractions of the Feed, Product and Reject streams were 20.9 %, 8.0 % and 45.5 % respectively (Tables A.2-A.4). Based on these values, the two-product formula (Wills & Napier-Munn, 2006) predicts an overall yield to product of:

$$\text{Yield} = (20.9 - 45.5)/(8.0 - 45.5) = 0.657$$

Using this flow split, the mass of material in and out of each size by density interval was compared. Table A.10 shows the results. For instance, in the first density interval of the +1.40 mm size fraction, the ratio is:

$$0.76/[1.16(0.657) + 0.23(1 - 0.657)] = 0.91$$

Table A.10: Ratio of mass in Feed divided by combined mass out in Product and Reject for each size by density interval based on the size by density mass distributions in Tables A.7, A.8 and A.9 and using a yield of 65.7 %.

IN/OUT	Density Range (kg/m ³)									
Size Range (mm)	1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
	1300	1350	1400	1500	1600	1700	1800	2000	2200	2500
+1.40	0.91	0.89	0.58	0.74	0.67	0.98	1.42	2.60	0.30	1.00
-1.40 +1.00	1.40	0.59	0.79	0.83	0.79	0.89	1.31	2.16	0.62	0.00
-1.00 +0.710	1.39	0.66	0.80	0.96	0.98	0.82	1.05	1.69	1.86	0.00
-0.710 +0.500	1.00	1.32	0.76	0.85	0.93	0.99	0.92	1.57	1.48	0.78
-0.500 +0.355	0.97	1.06	0.81	0.95	0.76	1.23	0.99	1.39	1.13	1.06
-0.355 +0.250	1.18	0.58	0.75	0.94	0.92	0.86	1.14	1.77	1.72	0.74
-0.250 +0.180	1.00	0.61	0.98	0.97	1.04	2.58	0.53	0.95	3.00	0.57
-0.180 +0.125	0.87	0.41	0.62	0.57	0.85	1.02	1.40	1.93	1.37	0.89

Clearly the ratio of mass in over mass out was often far from unity. This deviation is a normal occurrence due to random sampling and measurement errors. This prevents calculation of an unambiguous partition value at each density. For example, in the first density interval of the +1.40 mm size fraction, Equations (4a-c) give three different values for the partition to product:

$$\text{Product/Feed} = 1.16(0.657)/0.76 = 1.003 \quad (4a)$$

$$\text{Product}/(\text{Product} + \text{Reject}) = 1.16(0.657)/[1.16(0.657) + 0.23(0.343)] = 0.906 \quad (4b)$$

$$(\text{Feed} - \text{Reject})/\text{Feed} = [0.76(1) - 0.23(1 - 0.657)]/0.76 = 0.896 \quad (4c)$$

In order to overcome this ambiguity, the data was mass-balance reconciled, in the same manner as would be used for any other set of experimental size by density mass distribution data. Undersized material in the -0.125 mm size fraction was not included in this mass balance reconciliation process, as all the streams were wet sieved at this size prior to analysis. The experimentally measured flowrates and pulp densities were also not used in the mass balance reconciliation i.e. the only data used was the mass fraction of solids in each size and density interval shown in Tables A.7-A.9.

Tables A.11-A.13 show the mass-balance reconciled size by density mass distributions of the Feed, Product and Reject streams based on the “raw” data in Tables A.7-A.9. Mass-balancing of the full double-fractionation +0.125 mm data gave relative stream flowrates of Feed = 100, Product = 66.1 and Reject = 33.9 mass units in each stream.

Table A.11: Case Study 1 Feed size by density mass distribution (wt.%) based on mass-balance reconciliation of the laboratory Reflux Classifier “raw” data in Tables A.7-A.9.

FEED		Density Range (kg/m ³)									
Size Range (mm)		1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
		1300	1350	1400	1500	1600	1700	1800	2000	2200	2500
+1.40		0.84	0.22	0.25	0.54	0.25	0.17	0.14	0.16	0.04	0.00
-1.40 +1.00		4.46	1.25	1.23	2.29	1.50	0.73	0.59	0.77	0.34	0.00
-1.00 +0.710		7.42	2.21	1.98	2.85	2.18	0.92	0.82	1.18	0.94	0.00
-0.710 +0.500		9.01	1.65	1.85	2.54	1.79	0.92	0.71	1.00	1.05	0.86
-0.500 +0.355		7.00	1.37	1.20	1.70	1.00	0.71	0.39	0.63	0.72	0.86
-0.355 +0.250		5.41	1.04	0.80	1.35	0.67	0.42	0.36	0.40	0.40	0.85
-0.250 +0.180		4.83	1.07	0.92	1.20	0.58	0.20	0.17	0.30	0.30	0.68
-0.180 +0.125		1.72	0.40	0.33	0.48	0.43	0.15	0.11	0.18	0.31	0.76
+0.125		40.67	9.21	8.56	12.95	8.39	4.23	3.29	4.61	4.10	4.00

Table A.12: Case Study 1 Product size by density mass distribution (wt.%) based on mass-balance reconciliation of the laboratory Reflux Classifier “raw” data in Tables A.7-A.9.

PRODUCT	Density Range (kg/m ³)									
Size Range (mm)	1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
	1300	1350	1400	1500	1600	1700	1800	2000	2200	2500
+1.40	1.14	0.17	0.12	0.08	0.00	0.00	0.00	0.00	0.00	0.00
-1.40 +1.00	6.44	1.48	1.05	0.64	0.18	0.00	0.00	0.00	0.00	0.00
-1.00 +0.710	10.97	3.00	2.56	1.70	0.35	0.00	0.00	0.00	0.00	0.00
-0.710 +0.500	13.46	2.33	2.63	2.73	0.66	0.00	0.00	0.00	0.00	0.00
-0.500 +0.355	10.49	2.01	1.74	2.25	0.63	0.00	0.00	0.00	0.00	0.00
-0.355 +0.250	8.12	1.52	1.14	1.92	0.66	0.00	0.00	0.00	0.00	0.00
-0.250 +0.180	7.30	1.58	1.32	1.67	0.73	0.11	0.00	0.00	0.00	0.00
-0.180 +0.125	2.59	0.60	0.46	0.65	0.57	0.15	0.08	0.00	0.00	0.00
+0.125	60.52	12.69	11.04	11.63	3.78	0.26	0.08	0.00	0.00	0.00

Table A.13: Case Study 1 Reject size by density mass distribution (wt.%) based on mass-balance reconciliation of the laboratory Reflux Classifier “raw” data in Tables A.7-A.9.

REJECT	Density Interval (kg/m ³)									
Size Range (mm)	1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
	1300	1350	1400	1500	1600	1700	1800	2000	2200	2500
+1.40	0.24	0.31	0.49	1.43	0.73	0.51	0.40	0.46	0.12	0.00
-1.40 +1.00	0.59	0.79	1.58	5.53	4.06	2.17	1.75	2.28	1.00	0.00
-1.00 +0.710	0.47	0.65	0.83	5.10	5.75	2.72	2.43	3.48	2.79	0.00
-0.710 +0.500	0.31	0.33	0.32	2.16	4.01	2.73	2.09	2.95	3.10	2.53
-0.500 +0.355	0.16	0.13	0.13	0.63	1.71	2.08	1.14	1.85	2.13	2.53
-0.355 +0.250	0.09	0.12	0.12	0.24	0.68	1.23	1.07	1.20	1.18	2.52
-0.250 +0.180	0.00	0.07	0.14	0.29	0.29	0.38	0.51	0.88	0.88	2.00
-0.180 +0.125	0.00	0.01	0.08	0.15	0.16	0.16	0.17	0.52	0.92	2.24
+0.125	1.88	2.40	3.70	15.52	17.39	11.98	9.57	13.63	12.12	11.82

Tables A.14-A.16 show the size by density distribution of material based on 100 mass units of feed, as calculated from the mass-balanced data in Tables A.11-A.13. Table A.17 shows the partition values calculated from this distribution of mass.

Table A.14: Feed distribution of mass units based on $F = 100$ units using distribution data in Table A.11.

FEED	Density Range (kg/m ³)									
Size Range (mm)	1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
	1300	1350	1400	1500	1600	1700	1800	2000	2200	2500
+1.40	0.84	0.22	0.25	0.54	0.25	0.17	0.14	0.16	0.04	0.00
-1.40 +1.00	4.46	1.25	1.23	2.29	1.50	0.73	0.59	0.77	0.34	0.00
-1.00 +0.710	7.42	2.21	1.98	2.85	2.18	0.92	0.82	1.18	0.94	0.00
-0.710 +0.500	9.01	1.65	1.85	2.54	1.79	0.92	0.71	1.00	1.05	0.86
-0.500 +0.355	7.00	1.37	1.20	1.70	1.00	0.71	0.39	0.63	0.72	0.86
-0.355 +0.250	5.41	1.04	0.80	1.35	0.67	0.42	0.36	0.40	0.40	0.85
-0.250 +0.180	4.83	1.07	0.92	1.20	0.58	0.20	0.17	0.30	0.30	0.68
-0.180 +0.125	1.72	0.40	0.33	0.48	0.43	0.15	0.11	0.18	0.31	0.76
+0.125	40.67	9.21	8.56	12.95	8.39	4.23	3.29	4.61	4.10	4.00

Table A.15: Product distribution of mass units based on $P = 66.1$ units using distribution data in Table A.12.

PRODUCT	Density Range (kg/m ³)									
Size Range (mm)	1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
	1300	1350	1400	1500	1600	1700	1800	2000	2200	2500
+1.40	0.76	0.11	0.08	0.05	0.00	0.00	0.00	0.00	0.00	0.00
-1.40 +1.00	4.26	0.98	0.70	0.42	0.12	0.00	0.00	0.00	0.00	0.00
-1.00 +0.710	7.26	1.99	1.70	1.12	0.23	0.00	0.00	0.00	0.00	0.00
-0.710 +0.500	8.91	1.54	1.74	1.81	0.44	0.00	0.00	0.00	0.00	0.00
-0.500 +0.355	6.94	1.33	1.15	1.49	0.42	0.00	0.00	0.00	0.00	0.00
-0.355 +0.250	5.38	1.00	0.76	1.27	0.44	0.00	0.00	0.00	0.00	0.00
-0.250 +0.180	4.83	1.05	0.87	1.11	0.48	0.07	0.00	0.00	0.00	0.00
-0.180 +0.125	1.72	0.40	0.31	0.43	0.38	0.10	0.05	0.00	0.00	0.00
+0.125	40.04	8.39	7.30	7.70	2.50	0.17	0.05	0.00	0.00	0.00

Table A.16: Reject distribution of mass units based on $R = 33.9$ units using distribution data in Table A.13.

REJECT	Density Interval (kg/m ³)									
Size Range (mm)	1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
	1300	1350	1400	1500	1600	1700	1800	2000	2200	2500
+1.40	0.08	0.10	0.17	0.48	0.25	0.17	0.14	0.16	0.04	0.00
-1.40 +1.00	0.20	0.27	0.54	1.87	1.37	0.73	0.59	0.77	0.34	0.00
-1.00 +0.710	0.16	0.22	0.28	1.73	1.95	0.92	0.82	1.18	0.94	0.00
-0.710 +0.500	0.11	0.11	0.11	0.73	1.36	0.92	0.71	1.00	1.05	0.86
-0.500 +0.355	0.06	0.04	0.04	0.21	0.58	0.71	0.39	0.63	0.72	0.86
-0.355 +0.250	0.03	0.04	0.04	0.08	0.23	0.42	0.36	0.40	0.40	0.85
-0.250 +0.180	0.00	0.02	0.05	0.10	0.10	0.13	0.17	0.30	0.30	0.68
-0.180 +0.125	0.00	0.00	0.03	0.05	0.05	0.05	0.06	0.18	0.31	0.76
+0.125	0.63	0.81	1.25	5.25	5.88	4.06	3.24	4.61	4.10	4.00

Table A.17: Partition to product for each size by density interval, based on distribution of mass units in Tables A.14-A.15.

PARTITION	Interval Average Density (kg/m ³)									
Size Range (mm)	1260	1325	1375	1450	1550	1650	1750	1900	2100	2350
-2.00 +1.40	0.90	0.52	0.33	0.10	0.01	0.00	0.00	0.00	0.00	
-1.40 +1.00	0.96	0.79	0.57	0.18	0.08	0.00	0.00	0.00	0.00	
-1.00 +0.710	0.98	0.90	0.86	0.39	0.11	0.00	0.00	0.00	0.00	
-0.710 +0.500	0.99	0.93	0.94	0.71	0.24	0.00	0.00	0.00	0.00	0.00
-0.500 +0.355	0.99	0.97	0.96	0.87	0.42	0.00	0.00	0.00	0.00	0.00
-0.355 +0.250	0.99	0.96	0.95	0.94	0.66	0.00	0.00	0.00	0.00	0.00
-0.250 +0.180	1.00	0.98	0.95	0.92	0.83	0.36	0.00	0.00	0.00	0.00
-0.180 +0.125	1.00	0.99	0.92	0.89	0.88	0.65	0.46	0.00	0.00	0.00
+0.250	0.98	0.90	0.84	0.55	0.22	0.00	0.00	0.00	0.00	0.00

The laboratory Reflux Classifier data were also analysed on the basis of a “single-fractionation” which assumes that there was no sizing performed on the flow samples (other than to remove any -0.250 mm material). Hence the total mass and average ash of the +0.250 mm material in each flow sample (see Tables A.2-A.4) were used as the experimental data. Table A.18 shows the resultant inferred mass percentage density distribution of each stream, the mass-balanced distribution, and the calculated partition values based on the mass-balanced distribution. Mass balancing gave a mass flow split of Feed = 100, Product = 63.1 and Reject = 36.9 mass units in each stream.

**Table A.18: Case Study 1 single fractionation analysis of the +0.250 mm laboratory
Reflux Classifier data.**

Density Interval (kg/m ³)										
Lower	1219	1300	1350	1400	1500	1600	1700	1800	2000	2200
Upper	1300	1350	1400	1500	1600	1700	1800	2000	2200	2500
Inferred "raw" density distribution (wt.%):										
Feed	41.70	8.27	7.36	11.48	8.88	3.98	3.27	6.55	6.55	1.97
Product	52.94	19.95	12.43	11.67	3.02	0.00	0.00	0.00	0.00	0.00
Reject	1.64	2.58	2.63	17.71	21.02	12.25	9.46	10.47	12.20	10.04
Mass-balanced density distribution (wt.%):										
Feed	38.25	10.18	8.30	13.31	9.63	4.36	3.49	4.71	5.34	2.44
Product	59.62	14.61	11.58	11.11	3.08	0.00	0.00	0.00	0.00	0.00
Reject	1.70	2.61	2.69	17.05	20.82	11.82	9.46	12.78	14.47	6.60
Mass units in each interval, based on F = 100, P = 63.1 and R = 36.9:										
Feed	38.25	10.18	8.30	13.31	9.63	4.36	3.49	4.71	5.34	2.44
Product	37.62	9.22	7.31	7.01	1.94	0.00	0.00	0.00	0.00	0.00
Reject	0.63	0.96	0.99	6.29	7.68	4.36	3.49	4.71	5.34	2.44
Partition to Product:										
	0.984	0.906	0.880	0.527	0.202	0.000	0.000	0.000	0.000	0.000