AN IMPROVED LABORATORY FLOTATION METHOD

N Lambert[1], M Campbell[2], K McLennan[1], A Coffey[1]
1 TUNRA Clean Coal noellambert@bigpond.com
2 ACIRL Pty Ltd michael.campbell@alsglobal.com.au

ABSTRACT

A new test method was developed which increases flotation yield across a range of coal types. It removes much of the inconsistency that occurs with the use of the current Australian Standard tree method and incorporates some improvements to equipment.

The Ultimate Flotation Test (UFT) involves floating a 400 g sample of coal sequentially to produce a number of concentrates and a tailings. All samples are then sized at 0.106 mm. The two tailings samples are sent for drying and analysis whilst the coarse and fine concentrate fractions are added back to the flotation cell in turn and sequentially refloated. Each new concentrate and tailings is then dried and analysed for mass, moisture and ash. Both yield and ash are reported on a dry basis. The advantage of sizing after flotation is that the ash values associated with the +0.106 mm fraction are guaranteed of being slimes free.

INTRODUCTION

The Australian Coal Association Research Program (ACARP) commissioned a study to develop a new test procedure that would determine the best possible flotation performance for a wide variety of coal samples, with varying characteristics and feed ash values. This requires the production of a cumulative mass yield versus ash curve that is as close as possible to the ultimate flotation response. To achieve this, the procedure needs firstly to be able to eliminate or at least minimize any slimes contamination within the concentrates. This material is currently recovered using standard laboratory test procedures.

Through successive trials of the various methods and subsequent analysis of the results achieved on the same flotation feed sample, it is possible to show how different methods perform in their prediction of flotation response. From the results of each of the froth flotation tests a cumulative mass versus cumulative ash curve was produced for each sample. The best response curve for each method was then charted for each separate sample on the same graph allowing the methods to be directly evaluated.

Four coal samples of different type and from various regions of Australia were tested, namely;

- Hunter Valley Coal (HV), very high ash (72% d), high slimes
- Bowen Basin Coal (BB), intermediate ash (37% d), high slimes
- Newcastle-Lake Macquarie Coal (NML), intermediate ash (35% d), intermediate slimes
- Illawarra Coal (I), low ash (15% d), low slimes

Various methods were assessed during this project. The samples above were analysed by each of the following flotation methods:

- AS4156.2.2(1998)
- ISO 8858-3 (2004)
- ACARP Project C6044
- Ultimate Flotation Test (UFT developed during ACARP project 14068)

PROPOSED METHODOLOGY

Equipment & Settings

- Flotation machine - mechanical impeller-type flotation machine with air flowmeter
- Flotation cell – 3.5 litres capacity, transparent
- Deflection block, minimum clearance of 3mm from cell
- Scrapers, for removing the froth from the entire surface of the pulp
- Calibrated micro-syringe or micro-pipette

- Containers (2L), marked to show 200mL and 1000mL volumes
- Calibrated timing device, accurate to ±1 second

Method Summary
The UFT method consisted of 4 discreet stages. The first stage is the initial flotation of the sample. The flotation process needs to be controlled very slowly to produce the low ash concentrates. This is achieved through the use of low dose rates of frother (laboratory grade MIBC at 5 µL/L) and collector (dodecane at 0.01 g/kg solids). The air rate is set at 4 L/min and the vacuum pressure recorded. This initial flotation produces up to seven separate concentrates and is continued until extinction, that is, no floatable material left in the tails.

The second stage is then a desliming step. The concentrates are sized at 0.106 mm to remove any slimes contamination from the + 0.106 mm material. This ensures that little if no slimes contamination is present in the coarse fraction.

The third stage is to refloat the sized concentrates so that each is separated further into a variety of high and low ash fractions. The number of concentrates produced from the individual refloat of each sample will depend on the mass of the individual sized fraction.

The fourth stage is the air drying and visual inspection of all concentrates for slimes contamination. If there is a significant amount of slimes contamination in the -0.106 mm concentrates, they should then be sized at 0.020 mm and given adequate sample mass, the subsequent coarse and fine fractions undergo further flotation.

Sample preparation is then able to proceed, ensuring all fractions are air dried and the mass, moisture content, and ash of each determined. The mass and ash are calculated to a dry basis, all concentrate and tailings fractions are sorted into ascending ash order and flotation response is shown graphically by plotting the cumulative ash Vs cumulative mass yield curve % (d). A detailed description of the method is given in ACARP 14068 report.

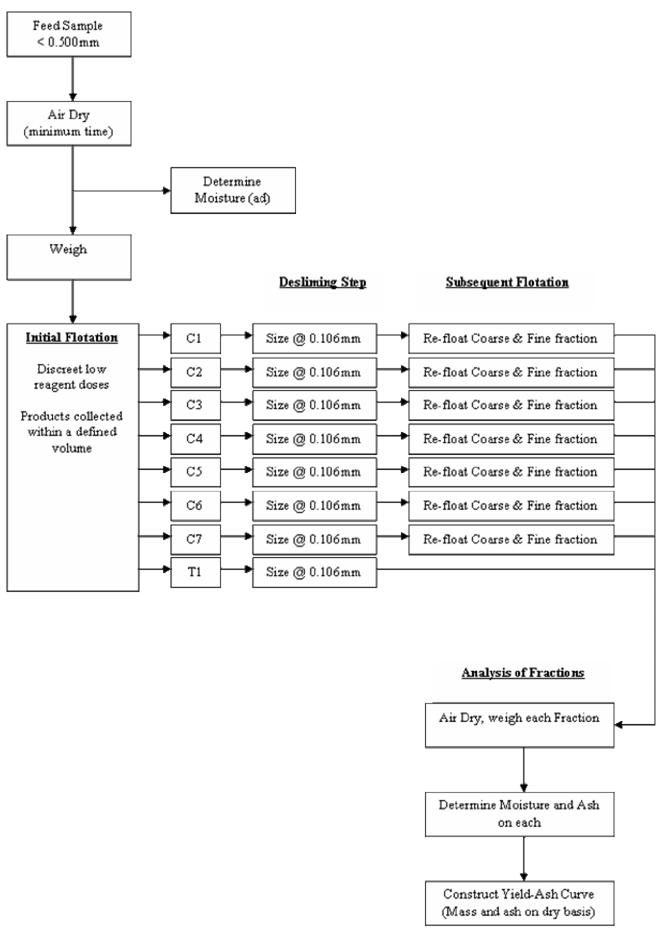

Figure 1 – Flowsheet

RESULTS

The different methods were compared to each other using yield-ash graphs produced by each coal type. The four samples were chosen to allow comparison of flotation responses over a large range of coal ash values and levels of slimes.

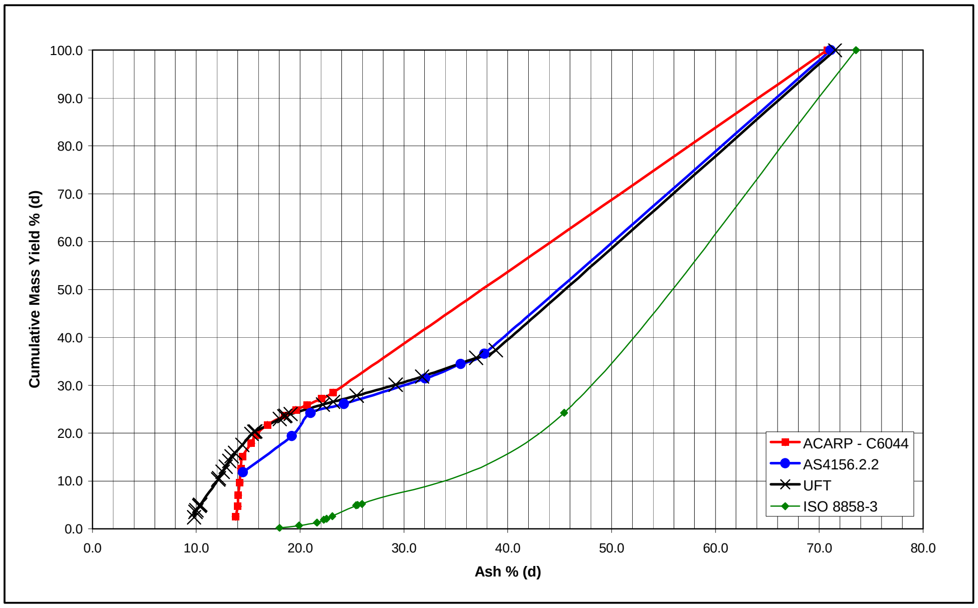

Figure 2 Hunter Valley Sample – Comparison of Methods

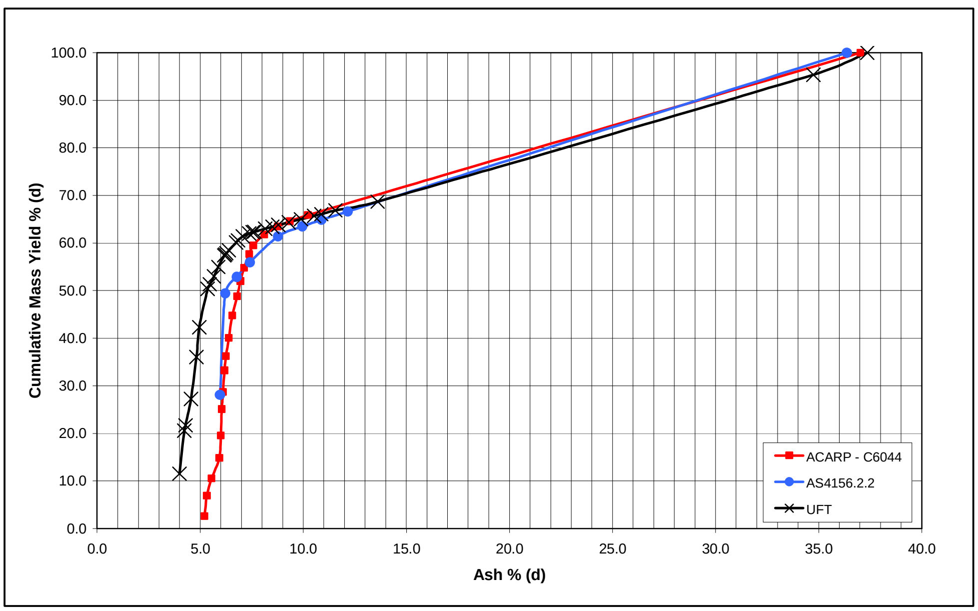

Figure 3 Bowen Basin Sample – Comparison of Methods

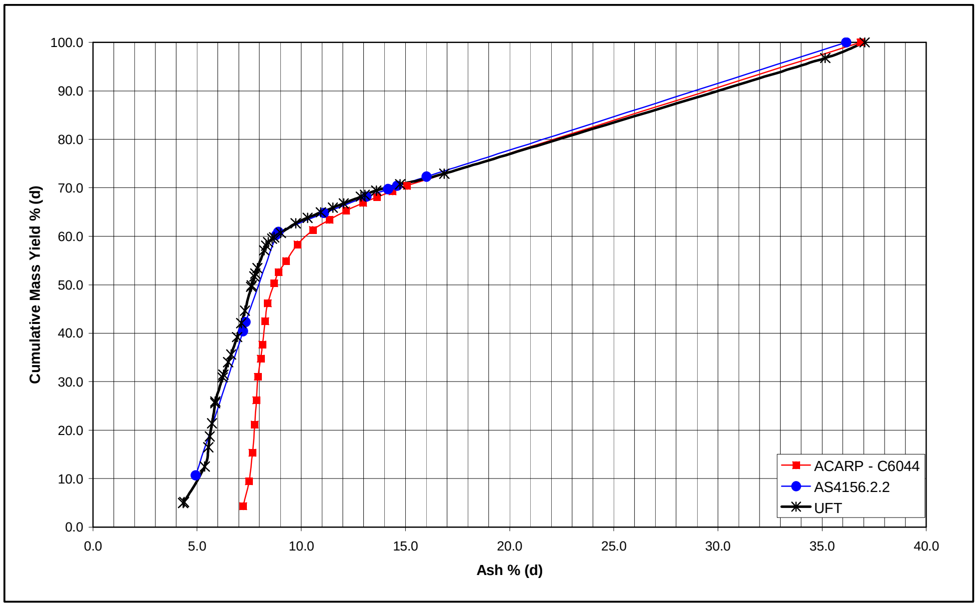

Figure 4 Newcastle Lake Macquarie Sample – Comparison of Methods

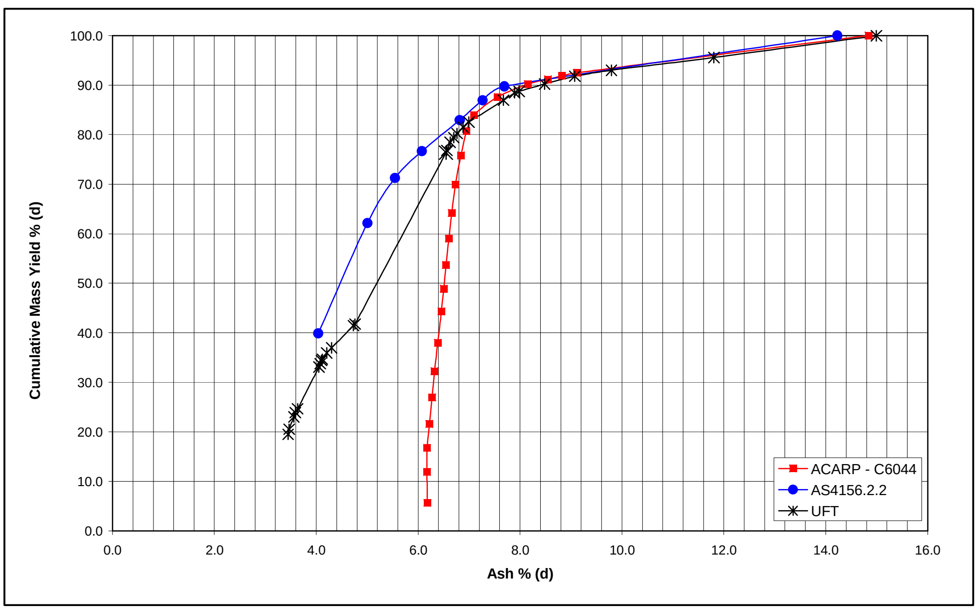

Figure 5 Illawara Sample – Comparison of Methods

	Test Method			
	AS4156.2.2	**C6044**	**UFT**	**ISO**
	Yield %	Yield %	Yield %	Yield %
HV Sample (Refer Figure 2)				
Product Ash - 12%	0.0	0.0	10.0	0.0
Product Ash - 18%	18.0	22.9	22.9	0.2
BB Sample (Refer Figure 3)				
Product Ash - 6%	0.0	24.4	55.8	-
Product Ash - 7.5%	55.5	58.5	62.2	-
NLM Sample (Refer Figure 4)				
Product Ash - 6%	25.0	0.0	27.5	-
Product Ash - 8%	50.6	34.0	54.6	-
I Sample (Refer Figure 5)				
Product Ash - 6%	75.7	0.0	65.8	-
Product Ash - 7.5%	88.3	86.7	85.8	-

Table 2 – Estimated yield for a specific product ash

		Relative Flotation Response				**Reference**
Sample	Ash % (d)	AS4156.2.2	C6044	UFT	ISO 8858-3	
Hunter Valley (HV)	72	Poor	Good	Very Good	Very Poor	Figure 2
Bowen Basin (BB)	37	Good	Good	Very Good	-	Figure 3
Newcastle, Lake Macquarie (NLM)	35	Very Good	Poor	Very Good	-	Figure 4
Illawarra (I)	15	Very Good	Poor	Very Good	-	Figure 5

Table 3 – Summary of flotation response

Table 2 and Table 3 endeavour to summarise the flotation response for each of the methods and each of the samples. This summarised data was interpreted from each of the cumulative yield versus cumulative ash graphs that were produced.

The International standard ISO 8858-3 (2004) froth flotation test for coal was only trialled on the Hunter Valley sample. The performance of this test was extremely poor when compared to the other froth flotation test methods. The method does not use desliming steps so the concentrates produced by this method will inevitably include slimes and hence produce a yield-ash curve away from the theoretical optimum response for the coal sample. Due to this poor performance, no further tests using ISO 8858-3 were conducted.

DISCUSSION

Flotation Response

A number of the methods tested had their own benefits and range over which they seem to give the closest yield-ash curve to the theoretical response curve for the four samples tested. The method developed in ACARP project C6044 uses wash water to remove entrained slimes from the products and as expected it performed well on the high ash Hunter Valley sample and the Bowen Basin samples. The C6044 froth flotation test however does have limitations which are clearly observed in the results of the low feed ash Illawarra sample (refer Figure 5). The resultant yield-ash curve understated substantially the possible flotation response for this coal type. Also, method C6044 requires specialised laboratory equipment as well as a much larger sample mass (800g) when compared to the Australian Standard or UFT method (400g).

The Australian Standards AS 4156.2.2 (1998) tree method showed some limitations, in that it performed very well on the low ash Illawarra flotation feed sample and the NLM sample. However, on the high ash Hunter

Valley sample, it missed the "knee" of the response curve (refer figure 2). AS 4156.2.2 (1998) froth flotation method produced the poorest (but only slightly) resultant yield-ash curve for the Bowen Basin sample, again appearing to miss the "knee" of the curve. This is most likely due to the collection of slime material in the concentrates, which is not being removed through the multiple refloating of the concentrates. In general however, this method was shown to perform well.

The UFT froth flotation test method was the best performing test and in particular on the high ash Hunter Valley sample and also the Bowen basin sample. This is likely due to the desliming/sizing step which removes the slimes contamination from the + 0.106 mm material and the subsequent refloating of the -0.106 mm and the +0.106 mm material to further remove slimes from the concentrates. The UFT test also performed very well on the Newcastle Lake Macquarie sample giving a resultant yield-ash curve equivalent to the Australian Standard method. For the low ash Illawarra sample the UFT method was able to show a flotation response at lower ash values than the other comparable methods. A product ash of 3 % (d) with a yield of 20 % (d) was produced from the UFT method whereas the AS4156.2.2 (1998) method did not show any response until a 4 % (d) ash was produced and method C6044 indicated that there was no flotation response at all below a 6 % (d) ash.

Table 2 and Table 3 endeavour to summarise the flotation responses for all of the methods

Sizing of Flotation Fractions

To achieve the closest ultimate flotation response curve to the theoretical flotation response curve for any coal sample, the entrainment of slimes into the concentrates has to be minimised. There are various ways of achieving this, such as using wash water as in the C6044 froth flotation method. Using wash water however does not eliminate all slimes from the concentrates as is clearly seen in the test results. The use of multiple refloats as a desliming process works well on a low ash coal sample but does not remove the slimes from the concentrates for a high ash coal sample. The desliming process for the UFT froth flotation test is a 3 stage process. After the initial flotation, the concentrates are sized to totally remove the slimes from the +0.106 mm material which then is refloated to further split the sample into higher and lower ash concentrates. The -0.106 mm is also refloated to remove slimes, however after a visual inspection, if slimes are still present, then they may undergo another sizing step at 0.020 mm. The use of these desliming steps followed by refloating, dramatically improved the yield-ash curve for the coal sample, bringing it closer to the optimum response.

The sizing step helps show a more accurate flotation response for the coarser particles. The sizing is only performed on concentrates, ie particles that have floated. Observation shows that some of these coarser particles tend to remain in the flotation cell on refloating. This appears to be due to the hydrodynamics of the cell. The coarse particles are observably caught in eddies within the centre of the cell. Consequently, numerous refloats of concentrates are often required to remove the entrained slimes. Each time that this refloat is performed, it appears that a proportion of these coarser particles are lost, due to them being caught in these eddies, not because they are unwilling to attach to bubbles. Sizing the sample at 0.106 mm completely removes the entrained slimes from the plus 0.106 mm concentrate, meaning that only a single refloat is required. This reduces the loss of the coarser material to tails.

To size the flotation products for the purpose of slimes removal may at first be considered inappropriate, however it is merely a tool to help maximise the yield of low ash coal. This test is to find the theoretical yield from a sample, not to replicate exact plant practice. Sizing guarantees the removal of slimes from the flotation products. The choice of 0.106mm aperture was based on the ease and speed of sizing at this screen size and the requirement that a minimum (< 4L) of underflow be produced. In some circumstances, plant sizing is performed to maximise flotation yield. Hydrocyclones may be used prior to flotation and either the underflow floated only (this has a reduced slimes loading) or both the underflow and overflow floated separately. Cycloning is an imperfect plant practice, whilst laboratory sieve sizing ensures perfect desliming.

Sizing the coal sample into a coarse and fine fraction before the flotation test, could adversely affect the flotation results. The small fine coal particles would be removed. These fine coal particles can be very hydrophobic and may help the less hydrophobic larger coal particles to float. The fine coal particles also increase the froth stability. Another detrimental affect of sizing before the initial flotation process is that particles could break down into slimes during the flotation and contaminate the concentrates. Also, this method would not allow for subsequent mathematical recombination of the individual concentrates to show what affect if any, of the sizing step. As a result, the UFT method involves the sizing of each concentrate after the initial flotation.

Another major advantage of using the UFT method is that it allows for the mathematical recombination of the results so that the affect of the desliming/sizing step and the affect of the refloating of the size fractions can be easily identified. This is shown in Figure 6, where it is clearly seen that without the desliming/sizing step and the refloat step, the resultant yield-ash curve would be dramatically to the right and further away from the theoretical yield-ash curve for the sample being tested.

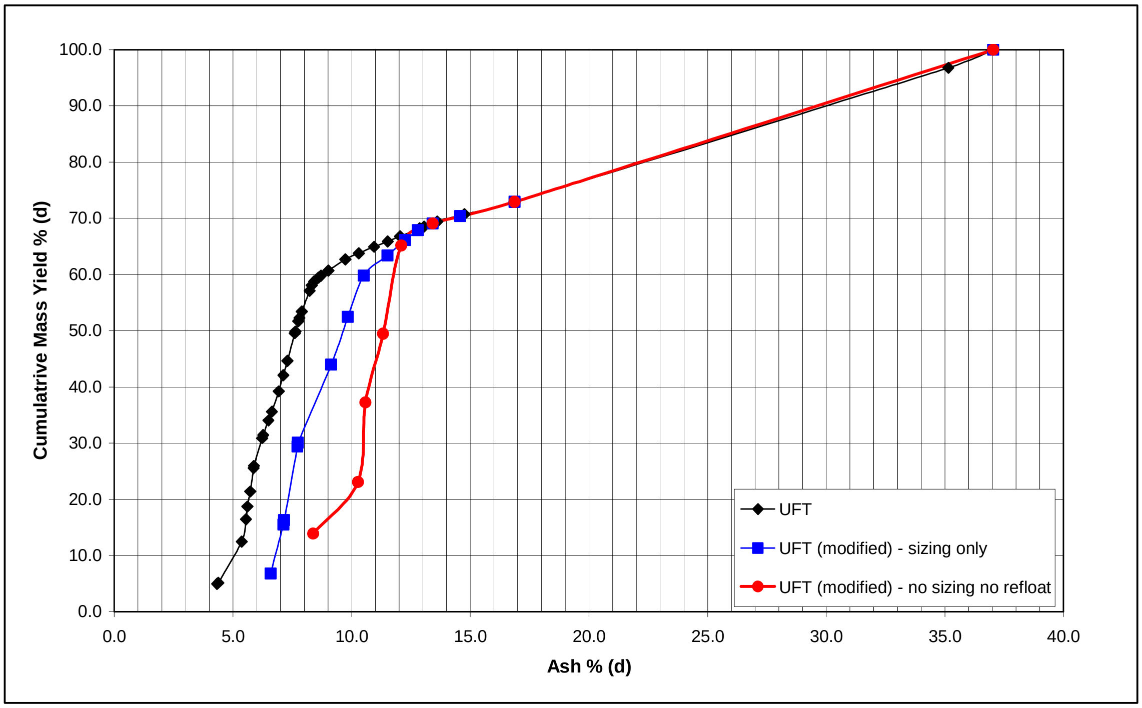

Figure 6 UFT Method – Effect of sizing and refloat

Equipment Recommendations

The use of a clear plastic flotation cell allowed the operator greater visual control over the UFT test. The operator can now visually see what is happening in the flotation cell throughout the UFT test as well as for the first time, the ability to continually monitor the froth depth. The disadvantage of a stainless steel cell is that only the surface of the froth can be seen.

The placement of the air control valve (refer Figure 7) after the rotameter ensures a true reading of the airflow and also the measurement of the vacuum pressure. If the vacuum pressure is not stable then there is a problem with the flotation system.

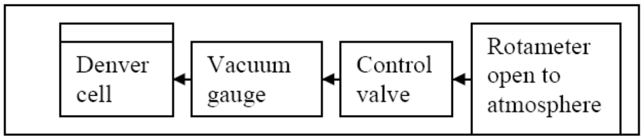

Figure 7 – Placement of air control valve

Cell level is adjusted by the operator. The transparent cell allows the level to be clearly seen and the level is adjusted by manual additions of water. The operator should use this water to wash the material that adheres to the deflector block and the cell walls adjacent to the deflector block, back into the slurry. It was found that having a 3mm gap between the deflector block and cell allowed the particles to be easily washed down and was the reason for increasing the gap from less than 1mm which is currently used.

Collector Rates

Slimes entrainment is inevitably collected with the concentrate due to the incredibly fine clays (less than 0.002 mm) being entrained in the froth water. The use of low reagent dosage rates allows for greater control, with low ash material floating with the least amount of reagent possible. Dependent on coal type, this very low ash material may even float with just the addition of air to the cell.

Reporting of Results
It is very important that yield and ash results from flotation studies are reported on a dry basis. This removes variations due to moisture when comparing the different fractions or when comparing one sample to another. As well, some understanding of the mineral matter would be a significant advantage when interpreting flotation results.

To calculate to a dry basis the following equations were used:

(1) Ash % (d) = 100 * Ash % (ad) / (100 – Moisture % (ad))

(2) Mass g (d) = Mass g (ad) * (100 – Moisture % (ad))/100

After converting all mass to a dry basis, percentage mass was then calculated.

Australian Standard AS2418 defines ash as "the inorganic residue after the incineration of coal to constant mass under standard conditions". Ash as determined in laboratory testing of coal samples, can sometimes be misleading if it is interpreted as being representative of the total inorganic portion of coal. The mineral matter present in coal undergoes many chemical changes during high temperature heating which will directly affect the ash Vs mineral matter relationship. For example, consider Table 4 which shows the tailings ash from a UFT sample.

Size Fraction	Moisture % (ad)	Ash % (ad)	Ash % (d)
+0.106 mm	1.4	92.4	93.7
-0.106 mm	7.0	84.7	91.1

Table 4 - Comparison of ash results at different moisture basis

On viewing the air-dry results only, it would be incorrect to assume that the flotation process has been more efficient with the coarse coal in separating the organic and inorganic components as it could be wrongly interpreted that there is 15.3 % organic matter remaining in the fines and only 7.6 % organic matter remaining in the coarse. The flotation results on a dry basis now show the two ash values being similar and in fact if mineral matter was to be determined then, the two values may even be identical and perhaps very close to 100.0 %.

ASSESSMENT OF PLANT PERFORMANCE

There has been much anecdotal evidence that the current Australian Standard AS 4156.2.2 (1998) froth flotation method underperforms actual industrial practice. Whilst this is a perception a number of points need to be considered.

Sampling of flotation streams
Sampling of flotation streams, particularly the feed, is often difficult and can be subject to bias. As an example, sampling slurry from the bottom of horizontal pipes may result in samples which are over representative of the coarse particles. As well, sampling fast flowing vertical slurry streams may over represent the coarse particles if the "boiling effect" results in the loss of mainly fine particles from hand held ladles. The feed can be the most difficult of the streams to sample due to the greater variability in ash of each of its particles. Flotation circuits are challenging due to their high volume, they are generally sampled manually and the sampling points themselves do not normally comply with the requirements for correct sampling location. The accurate sampling and testing of these streams is critical as the ash values of the feed, concentrate and tailings are then used to estimate flotation yield.

Consider the following:

Feed Ash = 20.0 % (d)
Concentrate Ash = 10.0 % (d)
Tailings Ash = 50.0 % (d)

} Calculated Yield = 75.0 %

If the feed ash had been biased low due to difficulties in sampling, then the actual recovery would have been:

Feed Ash =	22.0 % (d)	
Concentrate Ash =	10.0 % (d)	Calculated Yield = 70.0 %
Tailings Ash =	50.0 % (d)	

A 2.0 % (absolute) change in just the feed ash only has resulted in a 5.0 % change in yield.

If the laboratory flotation test and a plant audit are to be compared accurately, then the feed ash for both needs to be identical and all results reported on a dry basis. For accurate results, the feed sample should be the same as that used for flotation testing and any sub-division performed to ensure that all samples are representative.

Australian Standard AS 4156.2.2-1998 Method
The current Australian Standard AS 4156.2.2 (1998) method does suffer from a number of issues such as operator dependency (eg selection of reagent dosage rates). As such operators require a very high level of training and experience as various stages of the test necessitate operator judgement which will impact directly on the results obtained. As well, the lack of froth washing leads to the entrainment of slimes in the concentrates. These issues may not only contribute to poor reproducibility of the test but also accuracy as it may underestimate the flotation yield as well as perhaps overestimating product ash. The method also relies on a high number of sample transfer steps, each one running the risk of incomplete sample recovery. Any loss of sample may effect sample integrity and impact not only yield but also ash.

Another factor is a myriad of client defined "modified froth flotation methods" have become common, whereby a very small number of fractions only are requested, often as low as six. The curves produced from such methods may still be used for plant comparisons but in all probability would show an inferior flotation response which may in turn contribute to a poor reputation for AS4156.2.2.

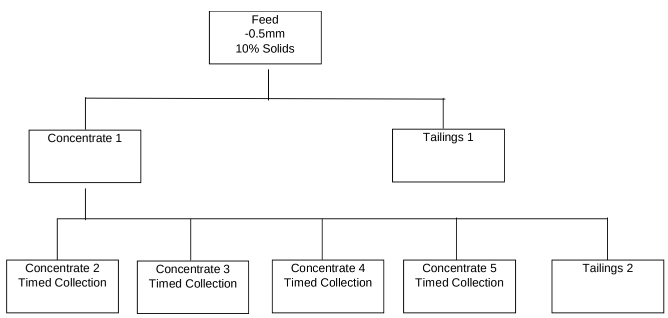

Figure 8 – Example of modified tree flotation method

This method recommends that mass and ash for each of the flotation fractions be reported on an air-dried basis. The influence of moisture variations between samples will distort the flotation response. Dry basis reporting should be mandatory.

In general however the method did perform relatively well. This indicates that with cautious application of reagents and a high degree of diligence being maintained throughout the test, then adequate flotation response curves can be produced. In fact the Australian Standard method performed as well as the UFT method for two of the samples tested (NLM & I). The other interesting point to note was that the repeatability of the method appeared to be quite good.

Tailings Ash
The tailings ash (dry basis), should be a very good indicator of plant performance and yield. If the tailings ash is lower for actual plant circuit samples than what was attained for the laboratory sequential tests, then plant performance is likely to be giving lower yields than the laboratory tests showed. If this is not the case, then the feed ash's may be different and as such no direct comparison between the two could be made with any confidence.

Sample Integrity
Samples collected for laboratory flotation testing may suffer a deterioration in flotation properties due to storage time and/or sample preparation procedures. This may in itself lead to discrepancies, if compared directly to the

flotation response from "fresh" flotation feed that is constantly delivered to industrial plant equipment. Essentially, regardless of the similarity in feed ash values, the laboratory sample may have quite different physical properties.

This may be especially true for borecore type samples that undergo the effects of drilling, lengthy pre-treatment stages followed by numerous air-drying steps.

CONCLUSIONS AND RECOMMENDATIONS

The new test method gave the best overall yield/ash response when compared against other known methods and using a variety of coal types. Analytical results indicate the most marked improvement in flotation response curves was for coals high in clay. The UFT method is less operator dependent than the current Australian Standard method AS 4156.2.2 (1998), with tight control over reagent dose rates, flotation cell design and setup, control of air rates and desliming procedures.

In summary, the UFT froth flotation method performed better than the current industry standard procedures. In particular, the major advantages with this method were considered to be:

- Improved flotation response curves
- Total elimination of slimes entrainment from coarse concentrate fractions
- Detailed operator instructions which define reagent additions
- Improvements to flotation test equipment and setup
- Requirement that mass yield and ash be reported to a dry basis
- No new specialised equipment needed
- Small (400g) sample mass required

The recommendation is that the UFT froth flotation method undergoes further testing to quantify precision. This testing is required to determine the repeatability, reproducibility and robustness of the test procedure.

ACKNOWLEDGEMENTS

The author's would like to thank the support of Fuch's Australia and the various coal preparation plants for providing samples. As well, ACARP, TUNRA and ACIRL for their support and permission to publish.

REFERENCES

AS 2418 (1995), '**Coal and Coke Glossary of terms**', Australian Standards

AS 4156.2.1 (2004), '**Coal preparation Part 2.2: Higher rank coal – Froth flotation – Basic Test**', Australian Standards

AS 4156.2.2 (1998) '**Coal preparation Part 2.2: Higher rank coal – Froth flotation – Sequential procedur**e', Australian Standards

Atkinson B, Blanshard S, ACARP C6044 (1999), '**Improved measure of ultimate flotation performance'**

Campbell M, Lambert N, ACARP 14058 (2008), '**Ultimate Froth Flotation Performance Test**'

ISO 8858-3 (2004), '**Hard coal – Froth flotation testing – part 3: Release evaluation**', International Standards Organisation