DEVELOPMENT OF A RAPID METHOD FOR PROGRESSIVE ATTRITION TESTING OF ALUMINA

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Abstract

High level of fines in alumina is a well known problem in aluminium smelters, negatively affecting conveying, segregation, dusting, fume treatment, cell feeding stability and sludge formation. The ultimate effect can be poor pot line performance and increased emissions.

Fines are present in the dispatched alumina from the refinery, and also formed along the alumina supply chain from calcination to cell feeding. The standard test for analysing fines forming potential, the Forsythe-Hertwig Attrition Index, has a number of disadvantages. There is a wider consensus that a new definition for how attrition is defined and measured would be of advantage to the industry. A new method should improve the ability to predict fines generation, as well as giving a better characterisation of particle strength, to be used as feedback to the ongoing development work aiming at producing stronger hydrate and alumina particles at the refinery.

A rapid method for progressive attrition testing of alumina has been developed, which characterises particle breakage properties at progressively increased forces. Main advantages of the method are: i) it is less time consuming than the Forsythe-Hertwig Attrition Index test; ii) particle breakage can be measured at forces more relevant to each individual smelter; iii) the measurement data output gives the complete particle size distribution (PSD) before and after exposure to attrition testing, which gives a number of alternative possibilities to how attrition can be defined.

1. Introduction

High level of fines in alumina is a well known problem in aluminium smelters, negatively affecting conveying, segregation, dusting, and fume treatment along the route from ship unloading to cell feeding. At the cell consequences can be cell feeding instability and poor solubility, which can cause sludge formation, anode effects and unstable thermal balance. The ultimate effect can be poor pot line performance and increased emissions.

Fines are present in the dispatched alumina from the refinery, and also formed along the alumina supply chain from calcination to cell feeding, due to particle breakage caused by handling. The shipment Certificate of Analysis (CoA) reports content of <45µm mass fraction ('fines'), and usually also <20µm mass fraction ('superfines'). Most alumina suppliers also report Forsythe-Hertwig Attrition Index (AI), in order to indicate to what extent the alumina will break down during handling. However, the Forsythe-Hertwig Attrition Index test has a number of disadvantages, as described in a subsequent section of this article, which means that the smelter will not get sufficient information from the CoA with regards to the expected content of fines and superfines at cell feeding.

There is a wider consensus that a new definition for how attrition is defined and measured would be of advantage to the industry (Perander 2011, Bemrose 1987, Bentham 2004, Petukhov 2003, Veesler 1993, Chandrashekar 2005). Targets for an improved method include:

- improved ability to predict fines generation during handling at smelters, i.e. testing under conditions more comparable to what is the case during handling at smelters
- obtain more information about the particle breakage product, e.g. do the particles break into larger fragments or into smaller pieces
- obtain a more comprehensive characterisation of particle strength to be used as feedback to the ongoing development work aiming at producing stronger hydrate and alumina particles at the refinery
- less time consuming than the Forsythe-Hertwig Attrition Index test

There are a number of alternative tests available for characterising breakage properties of particles, but so far none of these have been proven superior to the Forsythe-Hertwig Attrition Index test when it comes to alumina strength.

This paper describes a newly developed method for progressive attrition testing of alumina, which characterises particle breakage properties at progressively increased forces. Main advantages of the method are: i) it is it is less time consuming than the Forsythe-Hertwig Attrition Index test; ii) particle breakage can be measured at forces more relevant to each individual smelter; and iii) the measurement data output gives the complete PSD before and after exposure to attrition testing, which gives a number of alternative possibilities to how attrition can be defined.

The article will report on the Progressive Attrition Test setup and preliminary test method. Further development of the test method will be discussed.

2. Attrition Tests

The standard test in the alumina industry for quantifying alumina strength is the Forsythe-Hertwig Attrition Index (Benrose 1987), where strength is measured under fluidising process conditions. The method reports on the relative change in <45 µm mass fraction. The drawbacks of this method are listed below:

1. The method is relatively time consuming typically taking 1.5 hours for testing of one single sample, where roughly one third of the time involves manual labour.
2. The Attrition Index is highly sensitive towards the initial particle size distribution of the sample (Clerin 2001).
3. The method reports breakage at higher forces than what the alumina will typically experience in a smelter, as described in a subsequent section of this paper.
4. The interlaboratory repeatability of the method is relatively poor. The test is highly sensitive to the operating parameters of the fluidised bed unit. The impact on the particles is poorly controlled since there may be dead spaces in the fluidised bed where the particles receive a different level and type of breakage compared to particles more directly in the fluidised stream. Furthermore the breakage mechanism will depend on the orifice-to-particle diameter ratio.

A number of other attrition or breakage tests are available, e.g. single particle strength (Audet 2008), friability test, compressive strength, as well as variants of tests conducted in the modified Forsythe-Hertwig fluid bed (Ilievski 2005). However each of them seems to have some limitations, such as inability to differentiate between different alumina samples, and extensive time/labour requirement of the method. So far none of these tests have been able to out-compete the Forsythe-Hertwig Attrition Index test.

3. Apparatus and Method

A schematic of the Progressive Attrition Test setup is shown in Figure 1. It is a laser PSD analyzer from Malvern (Insitec analyzer with RTSizer), fitted with a vibrating tray, a hopper, and a 80 cm feeding tube. The rate at which the dry alumina sample is fed into the apparatus is controlled by the vibrating tray. The feeding tube provides dispersion of the powder, and the 90° eductor provides breakage of the particles at elevated flow rates. The optical head, consisting of the laser and the detectors, measures the PSD.

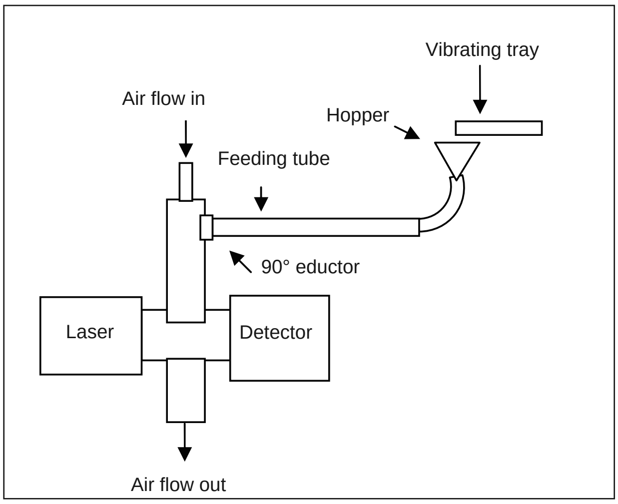


Figure 1. Schematic of Progressive Attrition Test setup

When the Insitec is used as an ordinary PSD analyser, a coaxial eductor is used instead of the 90° eductor since particle breakage would be undesirable. A flow titration is always run prior to the PSD analysis in order to find the optimal flow rate range, to ensure sufficient powder dispersion (will be a problem at low flow rates) and to avoid particle breakage (will be a problem at high flow rates). An example of a flow titration curve obtained with a coaxial eductor is shown in Figure 2. The optimal flow rate range is here 4 – 6 m^3/h.

For the Progressive Attrition Test setup, the coaxial eductor has been replaced with a 90° eductor in order to obtain particle breakage already at low flow rates. The test method uses the flow titration principle, where PSD is measured at progressively increased flow rates. An example is shown in Figure 3.

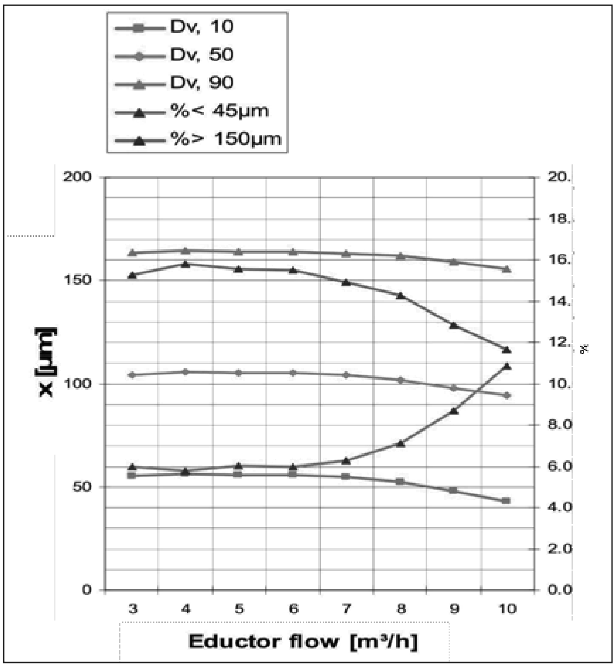


Figure 2. Example of flow titration curve obtained with a coaxial eductor (i.e. ordinary PSD analysis).

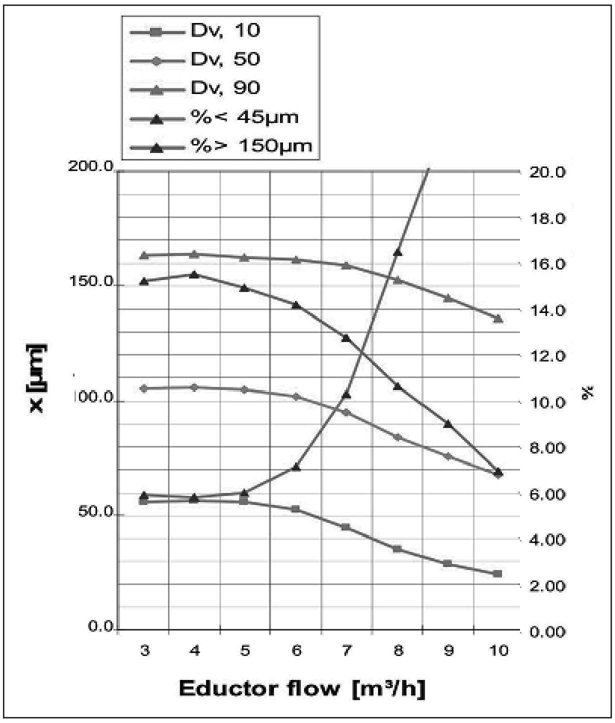


Figure 3. Example of flow titration curve obtained with a 90° eductor (i.e. progressive attrition testing)

Since the measurement data output gives the entire PSD curve (each sample will have one PSD curve for each flow rate), there is a number of possibilities to how attrition can be defined and displayed. Examples are:

- Attrition Index (Breakage Index) as described in a section further below.
- 'Total <45µm' (original fines + produced fines), as shown in Figure 3. Corresponding graphs can be shown for other sizes as well, e.g. 20µm.
- Particle size percentiles, e.g. D_5 and D_{10}, as shown in Figure 3.
- Span, i.e. PSD width

An Attrition Index [1] can be calculated from the change in <45 µm mass fraction when going from the baseline flow rate, e.g. 4 m^3/h to each of the higher flow rates (6 m^3/h, 7 m^3/h etc.), resulting in a progressive Attrition Index curve (as shown in Figure 5). To avoid any confusion, the Attrition Index obtained by the method described in this paper will be termed Breakage Index (BI). BI can also be reported for changes in the <20 µm mass fractions (BI_{20}), and for a number of other sizes.

The test setup (Figure 1) has two possibilities with regards to the 90° eductor; it can be used with a steel eductor or with a ceramic eductor, where the latter provides a higher particle breakage.

Required sample size is 50 g - 100 g for a single breakage index test (two different flow rates), and 400 g -500 g when running a progressive breakage test (~10 different flow rates). Time requirement is 10 minutes and 30 minutes, for single breakage index test and progressive breakage test, respectively.

It should be noted that the results shown in Figure 2 and Figure 3 were obtained using a different Insitec instrument and different method, compared to what is described in this paper.

4. Mechanism of Particle Breakage

A number of material properties and process conditions control particle breakage. Material properties include particle size, shape, porosity, hardness, surface roughness and the presence of cracks. Process conditions include time, velocity, pressure, shear and temperature (Boerefijn 2000).

A number of particle breakage mechanisms are described in the literature, including wear, attrition or abrasion, cleavage, chipping, fracture and fragmentation (Bemrose & Bridgewater 1987). They can be broadly divided into processes that occur to particles exposed to small and to large external forces.

The rate of external energy application to a particle can result in different breakage behaviour. However, according to Schönert 1979 and Kelly 1990, generally for brittle materials it is the magnitude of the applied energy and not so much the power that is important.

Alumina will undergo different types of impacts during handling at refineries and smelters, causing a range of different mechanisms of breakage. The mechanism and extent of particle breakage will vary from smelter to smelter even if the alumina is the same, due to different alumina transport and handling systems (Taylor 2005). These systems can for example involve the following facilities:

- pneumatic lifter and vacuum unloader during shipment unloading
- trucks
- belt conveyor, pneumatic lifter, and multiple feeding points to alumina silos
- anti-segregation reclaiming devices from the silos to the dry scrubbers
- airslides to the dry scrubbers
- hyperdense phase systems

As seen from Figure 2 and Figure 3, the 90º eductor is essential for obtaining extensive particle breakage in the Progressive Attrition Tester. This indicates that most of the breakage occurs when particles hit the wall of the 90º eductor. No further investigations have been carried out to determine the relevance of the breakage mechanism in the Progressive Attrition Tester to refinery and smelter conditions. However, this is a limitation that applies for most breakage tests, including the Forsythe-Hertwig Attrition Index test.

1 Calculated the same way as the Forsythe-Hertwig Attrition Index, i.e. as described in 'Australian Standard - Determination of Attrition index AS 2879.10'.

5. Progressive Attrition Testing of SGA Samples

5.1 Measurement Data Output

Figure 4 shows the Progressive Attrition Test raw data for one SGA sample. These raw data can be processed in order to display particle breakage in a number of different ways, for example BI_{45}, 'total <45µm' and 'total <20µm' (as shown in Figure 5, Figure 6 and Figure 7). Other examples are BI_{20}, particle size percentiles and span, as mentioned in the section 'Apparatus and Method'. This means that the Progressive Attrition Test provides much more information about the particle breakage product compared to the Forsythe-Hertwig Attrition Index test.

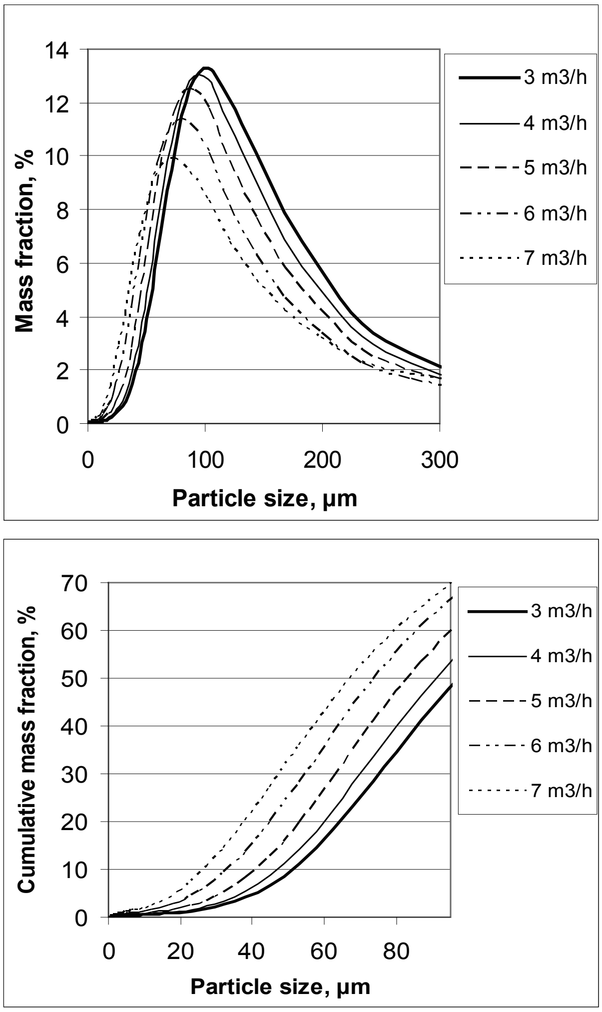


Figure 4. PSD for a SGA sample at progressively increased flow rate.

5.2 Repeatability Testing

Replicate analyses of five smelter grade alumina (SGA) samples were performed in order to investigate the repeatability of the method, and the ability to differentiate the samples with regards to breakage properties. Three replicate samples were tested for each SGA.

As previously explained, results can be displayed a number of different ways. Examples are BI_{45}, 'total <45µm' and 'total <20µm', as shown in Figure 5, Figure 6 and Figure 7. Each curve represents the average of the 3 replicate samples. Error bars displaying 1 standard deviation is included. 3 m^3/h was used as baseline flow rate for calculating BI_{45}. The 90º steel eductor was used in all these tests.

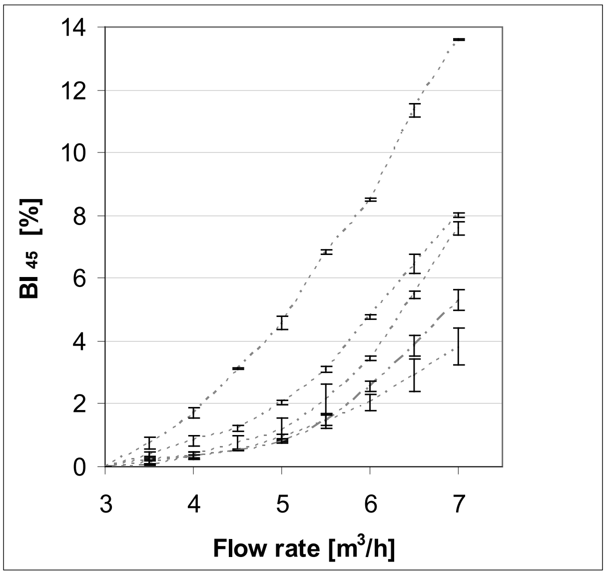


Figure 5. BI_{45} **for five SGA samples at progressively increased flow rate.**

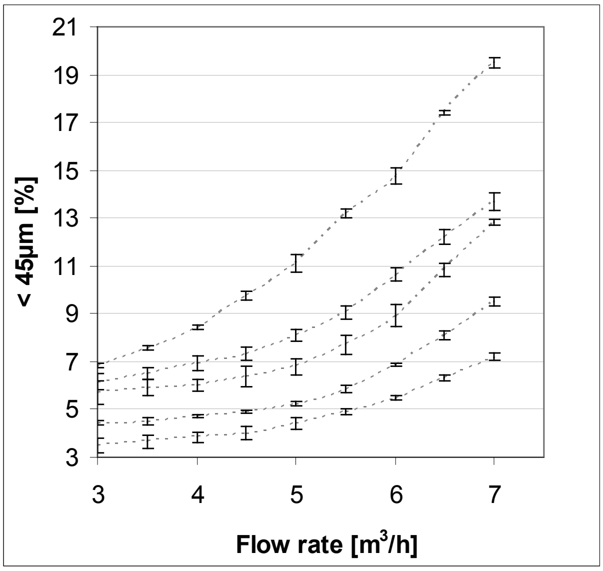


Figure 6. '<45µm mass fraction' for five SGA samples at progressively increased flow rate.

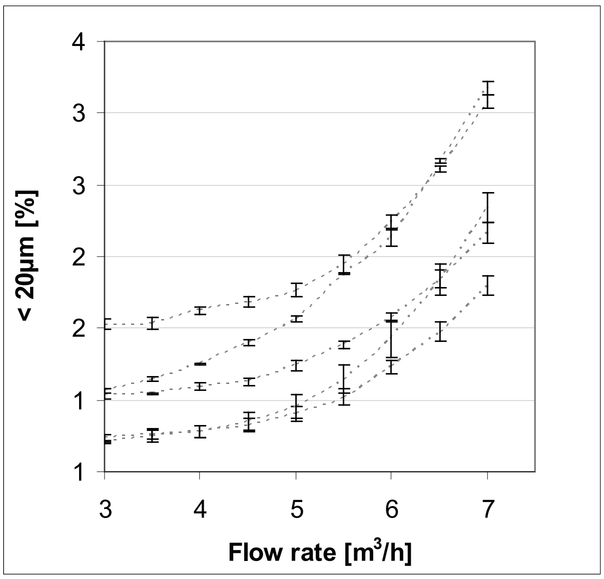


Figure 7. '< 20µm mass fraction' for five SGA samples at progressively increased flow rate.

The results indicate that the method has a sufficiently good repeatability in order to differentiate between alumina samples with different breakage characteristics. Nevertheless, the repeatability is expected to be improved further as more experience is gained and the method is optimised.

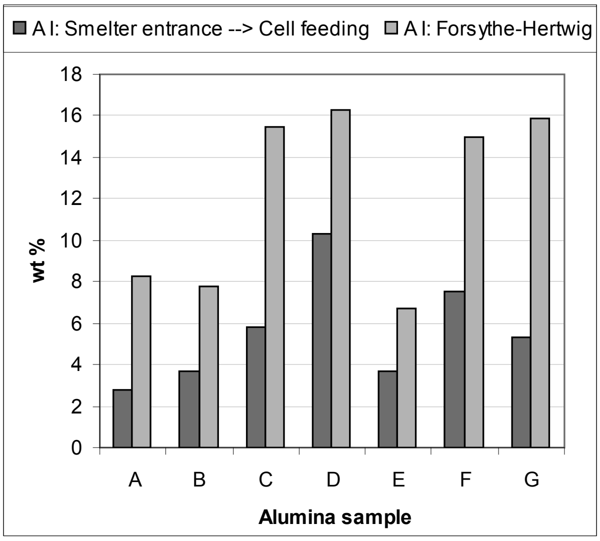
5.3 Further Development of the Method

PSD analysis by laser instruments is known to deviate from sieve analysis. Comparison of PSD measured at 3 m^3/h with PSD obtained by sieve analysis shows that this is also the case here. The deviation is more significant for the coarsest fractions. A number of adjustments for the Insitec PSD measurement has been planned in order to obtain an improved agreement with sieve analysis.

When using the 90º steel eductor it was not possible to obtain BI_{45} as high as the corresponding Forsythe-Hertwig Attrition Index for each of the samples. When the flow rate was increased beyond 7m^3/h, it was observed an increase in the >150µm mass fraction, indicating that 7m^3/h is the upper limit for this setup. However it was found that higher breakage can be obtained by replacing the steel eductor with a ceramic eductor, making it possible to obtain BI_{45} equal to corresponding Forsythe-Hertwig Attrition Index. Preliminary testing of the ceramic eductor has indicated that repeatability is at the same level as for the steel eductor, but more tests are required.

6. Fines Generation between Calcination and Cell Feeding

The Forsythe-Hertwig Attrition Index reports breakage at forces higher than what the alumina will typically experience along the alumina supply chain from calcination to cell feeding. Figure 8 compares Forsythe-Hertwig Attrition Index with corresponding attrition index that is calculated based on the actual breakage during alumina transport from smelter entrance to cell feeding. The numbers are based on investigations in two of our smelters involving SGA from 5 different refineries. The $AI_{\text{smelter entrance – cell feeding}}$ is calculated the same way as the Forsythe-Hertwig Attrition Index, but is based on '<45µm mass fraction at smelter entrance' and '<45µm mass fraction at cell feeding'.



Alumina sample	<45µm		Attrition Index	
	Smelter entrance	Cell feeding	Smelter entrance --> Cell feeding	Forsythe-Hertwig
A	9.7	12.2	2.8	8.3
B	8.4	11.8	3.7	7.8
C	7.4	12.8	5.8	15.5
D	7.9	17.4	10.3	16.3
E	4.8	8.3	3.7	6.7
F	9.7	16.5	7.5	15.0
G	6.6	11.6	5.3	15.9

Figure 8. Forsythe-Hertwig Attrition Index and corresponding Attrition Index calculated based on the actual breakage during alumina transport from smelter entrance to cell feeding.

It should be noted that the above results should be interpreted with care due to a number of limitations:

- - Sizing changes are not solely due to particle breakage. Recycling of fines from the scrubber gives an overestimation of the AI $_{\text{smelter entrance – cell feeding}}$.
- - Six of the seven sets of result are from a smelter that has a relatively rough alumina handling system.
- - PSD analyses have been carried out by different laboratories and different methods
- - Obtaining representative samples is challenging and requires large resources. Sample-to-sample deviation is often significant, and experience has shown large deviations between different sections of a smelter.

The Progressive Attrition Test makes it possible to test particle breakage at forces more relevant to each individual smelter. Testing of alumina to be used in a smelter with gentle alumina handling should apply lower flow rate (force) compared to smelters with rough alumina handling. The appropriate flow rate (force) for each smelter may be found by running the Progressive Attrition Test on a range of samples (primary alumina) where the AI $_{\text{smelter entrance – cell feeding}}$ is known.

7. Future Aspects of the Progressive Attrition Test

Results shown in this article is based on the initial development of the Progressive Attrition Tester. Further work should include:

- improve the PSD measurement to obtain a better agreement with sieve analysis, especially for the coarsest fractions
- use the coaxial eductor to produce the baseline numbers (instead of the '90º eductor at 3 m^3/h) in order to eliminate particle breakage at the baseline.
- further testing of repeatability for the ceramic eductor
- investigating if it is possible to find a Progressive Attrition Test flow rate (force) that corresponds to the forces at each smelter

This article has focused on fines generation after the alumina has left the refinery. However, it should be noted that hydrate and alumina strength is important also for the refineries. Fines generation in the refinery leads to counter productive measures to produce in-spec material (Coghill 2008). Hydrate particle strength is an area of continuous R&D focus, and future development work for the Progressive Attrition Tester will include hydrate samples.

It can also be mentioned that the Insitec analyzer is suitable for on-line measurements, meaning that the Progressive Attrition Tester could be used for on-line alumina attrition testing.

Although the Insitec instrument and other larger scale PSD analyzers are not typically available in alumina laboratories, the principle of the described method could possibly be applied to a table model laser (dry powder PSD analyzer with adjustable suction). The authors have previously observed alumina breakage is such instruments, however the attrition test application was not considered at that time.

8. Conclusion

A rapid method for progressive attrition testing of alumina has been developed, which characterises particle breakage properties at progressively increased forces. Main advantages of the method are: i) it is less time consuming than the Forsythe-Hertwig Attrition Index test; ii) particle breakage can be measured at forces more relevant to each individual smelter; and iii) the measurement data output gives the complete PSD before and after exposure to attrition testing, which gives a number of alternative possibilities to how attrition can be defined.

The initial investigations of the repeatability of five SGA samples indicate that the method is able to differentiate between samples with different breakage properties.

Further development of the method, as well as extended areas of application have been discussed.

9. Acknowledgements

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