

**School of Civil and Mechanical Engineering
Department of Civil Engineering**

**Production and Utilisation of Bayer Process Red Sand™ for
Construction and its Beneficiation by Magnetic Separation**

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**This thesis is presented for the Degree
of
Master of Philosophy
Curtin University**

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Declaration

To the best of my knowledge and belief this thesis contains no material previously published by any other person except where due acknowledgment has been made.

This thesis contains no material which has been accepted for the award of any other degree or diploma in any university.

Note, this thesis contains a collection of company reports that have been written by myself on work conducted by myself or in collaboration with others - where acknowledged.

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Summary

The extraction of alumina from bauxite produces a high volume by-product which is stored in secure impoundments. In Western Australia, typically half of the by-product has a particle size in excess of 90 microns and is called Red Sand™ by Alcoa of Australia Limited (Alcoa). This coarse fraction is unique to Western Australia due to the high quartz content of bauxite from the Darling Range. Alcoa has three Western Australian refineries and this coarse fraction represents a potential resource of up to 20,000 tonnes per day.

Red Sand™ currently requires storage in large and expensive impoundment sites due to the entrained sodium hydroxide. This is both an expensive cost upon the production facility as well as a wasted resource to the community. A variety of uses for coarse bauxite residues have now been evaluated. Primarily, Red Sand™ has been deemed to have physical properties suitable for use as construction sand because it is little different from fine crushed rock (which it actually is). Some of the uses now evaluated include use as a general fill material, in concrete, use in geopolymers, for topdressing of turf, for drainage improvement of turf, as lower sub-grade base in road construction and as the valuable sub-base in road construction. The normal use of sand in Western Australia is about 9 million tonnes per annum. Significant infrastructure projects, however, could require several multiples of this; whereas the production of Red Sand™ could be as high as 20 million tonnes per annum. Clearly new markets for this material will be required to take advantage of this unutilised resource if it is to be fully commercialised.

One method for opening markets and improving product value is to apply magnetic separation techniques. Magnetic separation of Red Sand™ has yielded a material that may be suitable for iron production and another that may be suitable for concrete manufacture. Other fractions appear to have new opportunities but are not covered here. Initial separations tended to provide a band of sand that resists separation and totals about 30% of total volume. Operational parameters and investigations were required to reduce or remove this fraction.

This thesis looked at the assessment of sand sources and the production of clean sand for various applications. This was an iterative process as the production of clean sand (Red Sand™) ultimately required a process that would allow subsequent magnetic separation if required. The production of clean sand was tested in the laboratory and then on a 10t/hr pilot plant. Product from this plant was then used in the construction of a trial section of road.

Assessments were also made on the magnetic separation products to remove alumina, both for recovery to the alumina process, but also to improve product quality of the high iron fraction.

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1.0 Literature Review

1.1 Red Sand™

Alcoa of Australia Limited (Alcoa) has three Western Australian refineries processing Darling Range bauxite. The resulting coarse fraction of the residue represents a potential resource of up to 7 Million tonnes per annum (Mtpa).

Jamieson et al (2005) described the coarse fraction of Darling Range bauxite residue as being physically little different from crushed rock, which is what the sand is derived from.

Attempts to turn coarse residue into a by-product have been evaluated for some time, however recent activity has been more focused upon product requirements. See (Sri et al 2006), (Jitsangiam et al 2007a, 2007b), (Jamieson 2007). Hammond (2003) conducted a physical comparison between coarse residue and road base. Hammond reported...

“Physical testing undertaken on the treated residue sand demonstrated suitability for road sub-grade and general construction fill. However, with present processing techniques, residue sand has unsuitable particle size distribution and insufficient strength characteristics to be considered as road sub-base;”

Hammond (2003) went on to evaluate the leaching from coarse residue, which identified that only aluminium exceeded DEC Inert Waste Classifications and that all elements were below health investigation trigger levels. Two elements, chromium and arsenic, did trigger ecological investigation levels, however this sand was unwashed and untreated.

A report from Golder & Associates (2003) also looked at the market for coarse residue in application in general construction, fill, concrete etc. Their findings suggested that there was considerable market competition but that the sand could find various “higher value” applications.

Clearly there is a need for upstream processing of the coarse residue to improve its physical characteristics for construction applications and to improve its environmental suitability.

1.2 Magnetic Separation

There are many different types of magnetic separators; this section will discuss some of these.

Dry Magnetic Separation

The two main types of permanent magnetic separators that are widely used in industry are the rare earth roll (RER) and the rare earth drum (RED). They are used to separate weakly magnetic materials and magnetic impurities that may be present in low levels in silica sand.

- 1) Rare earth roll separators; feed is fed onto a thin roll that travels at desired velocity. The feed material is passed through the magnetic field and the magnetic particles are attracted to the roll and separated from the non-magnetic stream by a splitter.

- 2) Rare earth drum separators; weakly magnetic particles pinned to the drum are carried to the region of higher magnetic intensity and are released as magnetics. The centrifugal force of the rotating drum throws those particles not influenced by the magnetic field into the non-magnetic collection hopper.

- 3) The drum separators can handle coarse (0.075 to 12.5 mm) particles, whereas roll separators are better suited to separating fine particles (less than 1 mm). Rare earth roll separators generally have a lower capacity than drum separators (Fuerstenau 2003).

Induced roll separators are used to remove trace level paramagnetic impurities from non-magnetic minerals. Selectivity is obtained by varying roll speed and magnetic flux intensity. The main limitation is the low capacity of these mechanisms.

Wet Magnetic Separation

- 1) Low Intensity Magnetic Separator (LIMS); is used as a primary stage beneficiation to remove mainly ferromagnetic material to improve the performance of downstream electromagnetic separators, used to separate weakly magnetic materials. LIMS have field intensities of up to 2000 Gauss. LIMS have been used extensively in the iron ore industry for enriching ores high in magnetite.
- 2) Wet High Intensity Magnetic Separator (WHIMS); WHIMS are used for separating paramagnetic or weakly magnetic particles which require higher magnetic field densities. The feed slurry passes through the separator into plate boxes where the separation takes place. The separator can be supplied with a continuous feed. The non-magnetic slurry passes through the plate boxes whilst the weakly magnetic particles are held by the plates, any non-magnetic particles entrained are washed and collected as a fraction.
- 3) High Gradient Wet Magnetic Separator (HGMS); HGMS use the same separation techniques as WHIMS. However, it is a batched operation. HGMS are designed for the purification of clay and other “fine-grained” and weakly magnetic particles.

A large volume of work has been done in the area of magnetic separation of Bayer Residue Mud (Red Mud), (Peiwang 1995), (Mishra 2001), (Fofana 1995). Very little work however has been done looking at the magnetic separation of Bayer Residue Sand (Red Sand™), due to sand being only a minor component of the residue at

most Bayer process refineries, typically only 5%. However in Western Australia sand makes up approximately 40% of the residue.

The work by Peiwang (1995) looked at the magnetic separation of Red Mud resulting from the processing of Chinese P bauxite. Two samples of bauxite (one roasted at between 500 and 700°C, and the other unroasted) were digested for 1 hour at 260°C in recycled (spent) liquor from an alumina refinery. The composition of the Red Mud from the two bauxite samples was very similar.

The Red Mud from both samples was subjected to pulsating high gradient magnetic separation; the roasted sample produced an iron concentrate with a higher grade and recovery than the unroasted sample, and that at 54 to 56% Fe the iron concentrate would be marketable as a smelting charge for blast furnaces.

Peiwang (1995) believed that “all of the iron particles should be dissociated from the lode filling of Red Mud and be able to be recovered into the iron concentrate.”

The iron recovery achieved, however, was only about 50%. Peiwang (1995) suggested this could be improved by adjustments to the set up of the magnetic separator. A multiple stage separation process could also improve the recovery. Peiwang (1995) put the lower recovery of the unroasted sample to “the iron mineral particles in the Red Mud of the unroasted sample aggregated with newly formed alumino–silicate particles”. Although this is possible, a more likely explanation is that during the roasting process the goethite (which makes up 11% of the bauxite) is converted to the more magnetically susceptible haematite (which makes up 4.5% of the bauxite) and thus reports to the iron concentrate during magnetic separation.

Most of the literature on magnetic separation of Red Mud involves either roasting of the bauxite or the Red Mud itself prior to magnetic separation. The cost of the process of roasting either the bauxite or the Red Mud, however, is likely to be prohibitive for alumina producers.

The work by Tedder (1983, 1984) is one of the few studies that looked at the magnetic separation of Bayer residue sand along with residue mud and bauxite. The samples from Alcoa refineries, in the USA and Australia were subjected to both wet

and dry roller magnetic separation. A magnetic fraction was easily isolated from the sand using the dry separator. Wet magnetic separation proved to be more difficult due to problems with the filter plugging.

Tedder (1983, 1984) found sharp separations for sand using a dry magnetic separator. The magnetic and non-magnetic fractions exhibited differences in colour, with the particles in the magnetic fraction appearing dark brown/black, and the non-magnetic particles being primarily white quartz grains. The magnetic fraction of the sands contained 50 to 60 wt % Fe_2O_3 . The Australian sand had higher aluminium content in the magnetic fraction than in the original sand fraction. This was the opposite of the residue sands from the USA. A similar finding was made for the non-magnetic fraction. The differences between residue sands from the USA and Australia are probably due to differences in the original bauxite ores and the operations of the refineries.

Tedder (1983, 1984) found that the non-magnetic fraction of the Australian residue sand has a silica content of 95% with an iron oxide content of less than 1%, indicating that a high grade silica fraction was produced. The magnetic fraction of the Australian sand had high aluminium content (~23% as Al_2O_3). This is likely to be due to aluminium substitution in the goethite. The magnetic fraction had an iron content of ~ 52% as Fe_2O_3 . The author suggests that if the separations can be performed economically, the iron rich fractions may be of value as iron ore substitutes; however the iron content of the magnetic fraction is probably too low and the aluminium content too high for use as an iron ore substitute.

Kimber (2004) looked at dry magnetic separation of West Australian bauxite residue. She concluded that dry magnetic rollers were an effective process for recovering separate iron and silica products that can be further treated to obtain cleaner products. The highest grade iron product recovered was 45% Fe_2O_3 in the magnetic fraction. The recovery of alumina was fairly constant over each fraction (magnetic, middlings and non-magnetic), and so dry magnetic separation was not considered an effective process for greatly upgrading the alumina in the sand, but does give a

product (non-magnetics fraction) that may yield more alumina after further processing. Kimber suggests that magnetic separation is not an effective process for recovering alumina close to Bayer plant feed grade from Red Sand™, however Western Australian refineries are operated with lower grade bauxite than refineries in other countries. The silica content of the magnetic fraction was higher than expected, and it was suggested that this may be due to entrainment of non-magnetic particles, or a fine film of magnetic material coating the non-magnetic particles. This suggests that a multi-stage separation process may be needed to recover high grade iron oxide and silica products rather than a single stage separation.

Kimber found that the separation of residue sand by wet drum separation is a more effective process for recovering higher amounts of iron oxides and alumina than dry magnetic separation, and can recover a high silica fraction with a grade comparable to the results obtained from electrostatic separation.

There has been a lot of research done on evaluating bauxite red mud, but the evaluation of coarse bauxite residue has only just begun. The few initial investigations into preparation of Red Sand™ suggested that further evaluation and testing are required.

1.3 Background of the Bayer Process

Aluminium is the third most abundant element in the Earth's crust and constitutes approximately 7% by mass. In nature it exists only in very stable combinations with other materials, and it was not until 1808 that its existence was first established. It took many years of research to liberate the metal from its ore and many more years after to produce a viable commercial production process.

In 1889 Karl Josef Bayer (Austria), son of the founder of the Bayer chemical company, invented the Bayer Process for the large scale production of alumina from bauxite. This was patented in United States Patent Application 20050238571.

Today the aluminium industry still relies on the Bayer process to produce alumina from bauxite. It remains the most economic means of obtaining alumina of suitable quality for the production of aluminium metal. The Bayer Process is briefly described below and can be reviewed in more detail in (Hudson 1982).

The Bayer Process for Alumina Refining

1) Grinding of the Bauxite

The bauxite ore is ground to a size of less than 1.5 mm (this ensures that the surface area is large enough so that there is sufficient solid/liquid contact during the digestion stage). This achieved through the use of crushers at the mine site and either rod mills, ball mills, or Semi-Autogenous Grinding (SAG) mills.

Hot concentrated sodium hydroxide (caustic soda) recycled from the caustic circuit is added to the bauxite during the grinding stage to produce a slurry. This slurry is then pumped into a series of holding tanks prior to digestion. The slurry is held in these holding tanks for sufficient time as to allow the desilication process to occur. This process removes silica as desilication product (DSP).

2) Digestion

Bauxite slurry is pumped from the holding tanks to the digestion units where additional hot recycled liquor is added to the bauxite slurry. During the digestion stage the hydrated alumina is removed from other insoluble oxides in the bauxite by reacting it with sodium hydroxide as per the reaction below:



The slurry exits the digestion units and is pumped to clarification. The slurry contains alumina in solution (normally referred to as green liquor) as well as undigested solids from the bauxite ore.

3) Clarification

In the clarification stage, undissolved solids from the bauxite ore are separated from the green liquor. This is done by using large clarification tanks (mud thickeners) which allow the undigested solids to settle out. These solids are passed through a counter-current washing train (mud washers) using water to recover as much of the sodium hydroxide as possible to enable it to be returned to the recycled caustic liquor circuit.

The washed solids are called “Process Residue” and are pumped to the Residue Drying Area (RDA).

Approximately halfway through the mud washing process the washer overflow is heated and contacted with lime slurry. This process is referred to as causticisation, whereby a portion of the sodium carbonate that has formed in the liquor is converted back to sodium hydroxide.

Causticisation enables a refinery to remain productive without the requirement for large quantities of fresh sodium hydroxide to be added.

4) Organics Removal

Organic material is naturally present in the bauxite and in some of the specialised chemicals (e.g. flocculants) used in the refinery. The organic materials react to form various organic sodium compounds, and over time these compounds build up in the recycled liquor circuit, reducing the efficiency of the precipitation process. These organic compounds can also adversely affect the formation of alumina tri-hydrate crystals, resulting in poor quality alumina product.

This build up of organics can be controlled by the concentration of sodium oxalate (the most significant of the organic compounds) and removal by seeding, precipitating and washing to produce an oxalate cake. The oxalate cake is sent to kilns which convert oxalate to carbonate and CO₂ by thermal decomposition. If the refinery does not use oxalate kilns then the cake is stored in a secure part of the residue storage area.

Another process of controlling the build up of organics is achieved by taking a small side stream of liquor and processing it in a “liquor burning” plant. In the

liquor burning plant the liquor is concentrated by evaporation, slurried with fine alumina dust, and then combusted in a rotary kiln. The organic compounds are oxidised and the resulting sodium aluminate is returned to the liquor circuit.

A biological oxalate destruction process can also be used to control the build up of organics in the Bayer Process. Biological oxalate destruction uses naturally occurring bacteria to consume oxalate. The process uses a series of tanks containing warm liquid and bacteria growing on plastic carriers. Oxygen and nutrients are added, and the oxalate is introduced as a feed source, bacteria consume the oxalate.

5) Precipitation

Green liquor is pumped to precipitation after being cooled via a heat exchange process. The heat recovered from the green liquor is transferred to the cold spent (liquor from which the alumina has been removed) which is returned to the start of the digestion process.

The green liquor is seeded with small alumina tri-hydrate crystals, which act as nuclei for more alumina tri-hydrate to precipitate. This seeded liquor is passed through a series of large vessels (precipitator tanks) where the crystals agglomerate and grow.

After leaving the last precipitator tank the hydrate slurry is classified by size in cyclones. Coarser particles are sent to calcination, whilst the finer particles are thickened, filtered and then recycled back to the start of precipitation and are used as seed crystals.

Spent liquor from classification, thickening and filtering of the hydrate slurry is recycled back to the digestion process.

6) Calcination

Calcination involves the washing and drying of the alumina tri-hydrate and heating it to approximately 1000°C to drive off the water of crystallisation.



The final product alumina is a dry white material which is the feedstock for aluminium smelters. Alumina is also used in the manufacture of advanced ceramics and the production of fused alumina.

7) Bauxite Residue Storage Area

The Bayer alumina refining process produces a caustic-insoluble residue (consisting mainly of oxides of iron and silicon) which has passed through the refining process along with residual quantities of caustic soda that were not recovered in the mud washing process.

The residue is separated into different size fractions during the refining process; these fractions are recombined prior to pumping to the residue area, and are then re-separated by a sand separation plant within the residue area.

Cooled liquor and runoff water are collected from the residue area and pumped back to the refinery for re-use in the process.

Globally, one tonne of residue is produced for each tonne of alumina. However the lower grade (average available alumina content 32.5%) of Darling Range bauxite results in the production of approximately two tonnes of residue per tonne of alumina. The low grade of Western Australian bauxite (this low grade is offset by the alumina in the ore being easily leached) is due to the high quartz content which is difficult to process and typically reports to residue with a large particle size in excess of 90 microns.

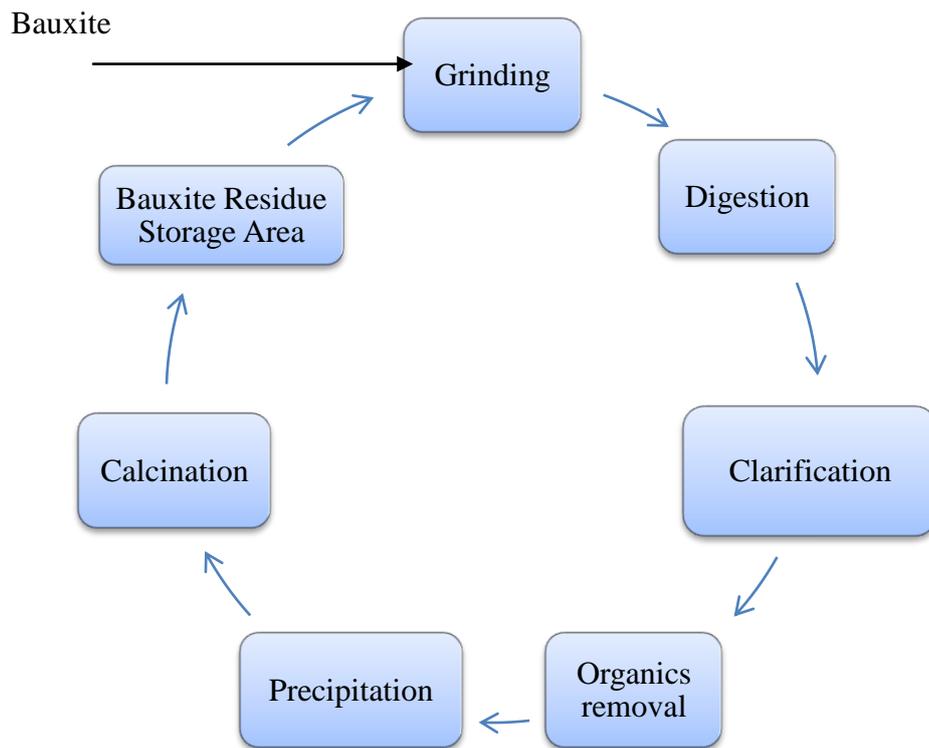


Figure 1.1 Basic Bayer Process Flow Diagram

2.0 Research Plan

The research program for this thesis is best summarised below in the plan diagram. The basic objective was to find out where the sand can be separated, derive its properties, assess beneficiation opportunities, and finally decide the best site for operational trials. Operational trials in this case resulted in a significant demonstration of the use of Red Sand™ in road construction.

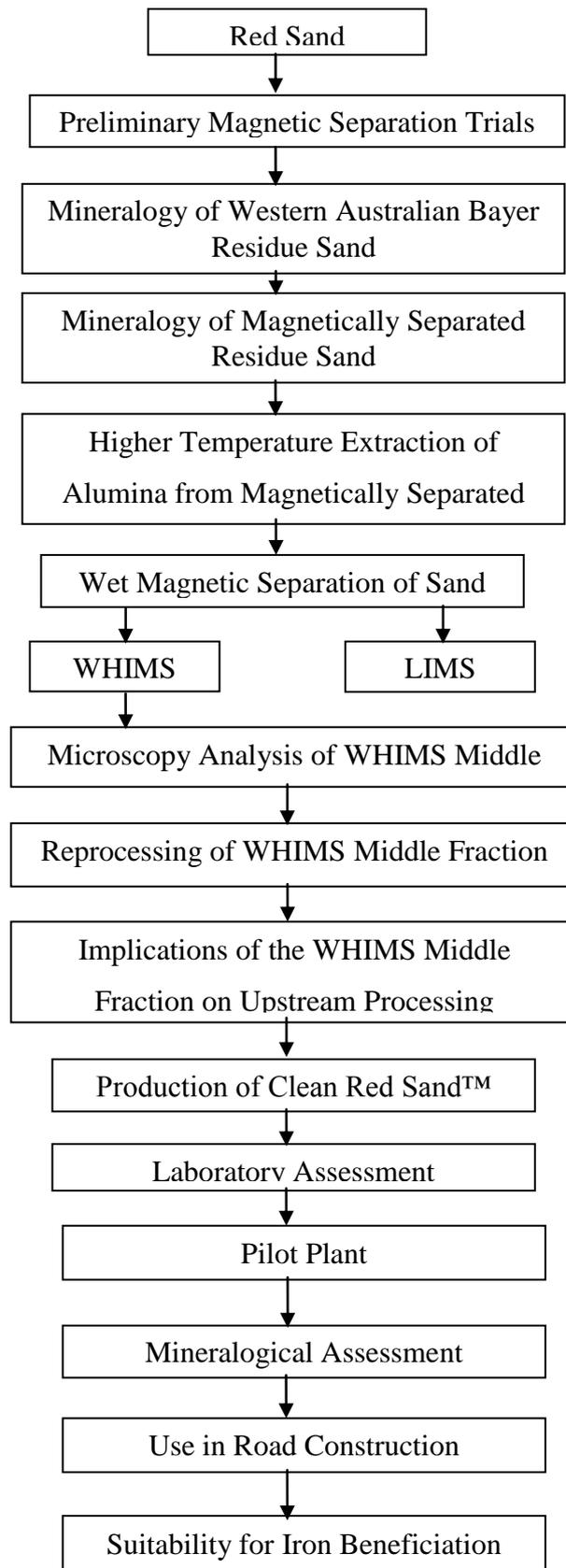


Figure 2.1 Outline of Research Program

3.0 Analytical and Equipment Methods

Methods of Analysis

Samples were prepared for elemental composition by X-Ray Fluorescence (XRF) using *Alcoa method 83379: Prepare solid samples for XRF Analysis*.

The samples were then analysed for elemental analysis by X-Ray Fluorescence (XRF) using the bauxite program on Alcoa's Phillip's model PW2400 XRF.

Total elemental composition analyses were performed by ICP at Ultra Trace Laboratories in Perth.

X-Ray Diffraction (XRD) mineralogical analyses were performed using the scan program on Alcoa's XRD model X'Pert MPD.

Samples prepared for mineralogical analyses by XRD firstly using *Alcoa method 4083: Micronise Samples for XRD Analysis (WAO)* followed by *Alcoa method 4079: Prepare Sample for XRD - Sample Pressing (WAO)*.

Samples prepared for viewing on the Scanning Electron Microscope (SEM) using *Alcoa method 4218: Perform Platinum Coating Using the Sputter Coater SDC-040 (WAO)*.

Electron Microscopy work performed using a FEI QUANTA 400F Scanning Electron Microscope.

Atmospheric digests were performed using *Alcoa method 4042: Available Alumina Analysis of Sand Residue From Blow Off Samples (WAO)*.

Bomb digestions were performed using *Alcoa Method 4302: Operate 100 mL Bomb Digest up to 145°C* (with an adjustment for the required digest temperature).

Lime Solids Analysis was performed using *Alcoa method 67533: Digestion of Residue Solids for Lime Solids Analysis (WAO)*.

Alian (Alcoa Liquor Analyser) liquor analysis is an automated titrimetric method based on the method published by H.L Watts and D.W. Utley in *Analytical Chemistry*, November 1956.

XRF Calibration for High Iron Matrix

Part of this project has been looking at magnetic separation of residue sand. The magnetic separation produces a fraction high in silica and another fraction that is high in iron. The samples from the magnetic separation process have been analysed on Alcoa's Kwinana Refinery laboratory's XRF using the bauxite program. When the bauxite program was originally set up it was not envisioned that it would be used to analyse samples high in iron and silica. It was not known if the calibration for iron and silica was valid at high levels. Initial investigations determined that the method was flawed for this sample matrix and would need to be modified appropriately. That work is described in Appendix 1 and is a summary of the Alcoa Report, (Jones 2006). Analysis of Samples high in Silica or Iron Oxide by XRF. Alcoa Report AWA 7.05 CAB011.

Method Assessment

Samples of Ferric oxide, ASCRM 005 (iron ore standard), and silicic acid ($\text{SiO}_2 \cdot x\text{H}_2\text{O}$) were heated at 1200°C for 2 hours. Samples were then placed in a desiccator to cool.

Ferric oxide was used as well as the iron ore standard so that the accuracy of the iron calibration could be checked across a wider range of concentrations.

The samples were weighed out with flux to give varying concentrations of iron or silica, samples were then fused and the beads were analysed using the bauxite program on the Alcoa Kwinana Refinery Laboratory's XRF.

Results

Table 3.1 XRF results

| SAMPLE NAME | XRF_BAUX | | | | | | | | | |
|-----------------------|--------------------------------|-------|--------------------------------|--------------------------------|------------------|-------------------|-------------------------------|------------------|------------------|--------|
| | Al ₂ O ₃ | CaO | Fe ₂ O ₃ | Ga ₂ O ₃ | K ₂ O | Na ₂ O | P ₂ O ₅ | SiO ₂ | TiO ₂ | Total |
| | % | % | % | % | % | % | % | % | % | % |
| 100% SiO ₂ | 0.38 | 0.090 | 0.41 | 0.00 | 0.000 | 0.00 | 0.001 | 100.11 | 0.046 | 101.01 |
| 75% SiO ₂ | 0.38 | 0.065 | 0.40 | 0.00 | 0.000 | 0.00 | 0.001 | 74.86 | 0.041 | 75.73 |
| 50% SiO ₂ | 0.37 | 0.039 | 0.41 | 0.00 | 0.000 | 0.00 | 0.001 | 49.76 | 0.037 | 50.58 |
| 25% SiO ₂ | 0.35 | 0.013 | 0.39 | 0.00 | 0.000 | 0.00 | 0.001 | 24.69 | 0.031 | 25.44 |
| 90% SiO ₂ | 0.38 | 0.079 | 0.45 | 0.00 | 0.000 | 0.00 | 0.000 | 90.13 | 0.045 | 91.06 |
| 95% SiO ₂ | 0.38 | 0.086 | 0.40 | 0.00 | 0.000 | 0.00 | 0.002 | 95.17 | 0.045 | 96.07 |
| ASCRM005 100% | 3.28 | 0.156 | 96.81 | 0.00 | 0.023 | 0.17 | 0.102 | 6.62 | 0.290 | 107.45 |
| ASCRM005 50% | 1.81 | 0.074 | 47.28 | 0.00 | 0.007 | 0.06 | 0.048 | 3.28 | 0.161 | 52.73 |
| ASCRM005 25% | 1.08 | 0.032 | 23.94 | 0.00 | 0.000 | 0.02 | 0.025 | 1.63 | 0.095 | 26.83 |
| Ferric oxide 100% | 0.36 | 0.000 | 109.80 | 0.00 | 0.000 | 0.16 | 0.000 | 0.05 | 0.050 | 110.42 |
| Ferric oxide 75% | 0.36 | 0.000 | 80.46 | 0.00 | 0.000 | 0.11 | 0.002 | 0.03 | 0.044 | 81.00 |
| Ferric oxide 50% | 0.35 | 0.000 | 53.04 | 0.00 | 0.000 | 0.07 | 0.000 | 0.02 | 0.038 | 53.51 |
| Ferric oxide 25% | 0.36 | 0.000 | 26.39 | 0.00 | 0.000 | 0.01 | 0.000 | 0.00 | 0.032 | 26.78 |
| Ferric oxide 65% | 0.37 | 0.000 | 69.62 | 0.00 | 0.000 | 0.10 | 0.000 | 0.05 | 0.043 | 70.17 |

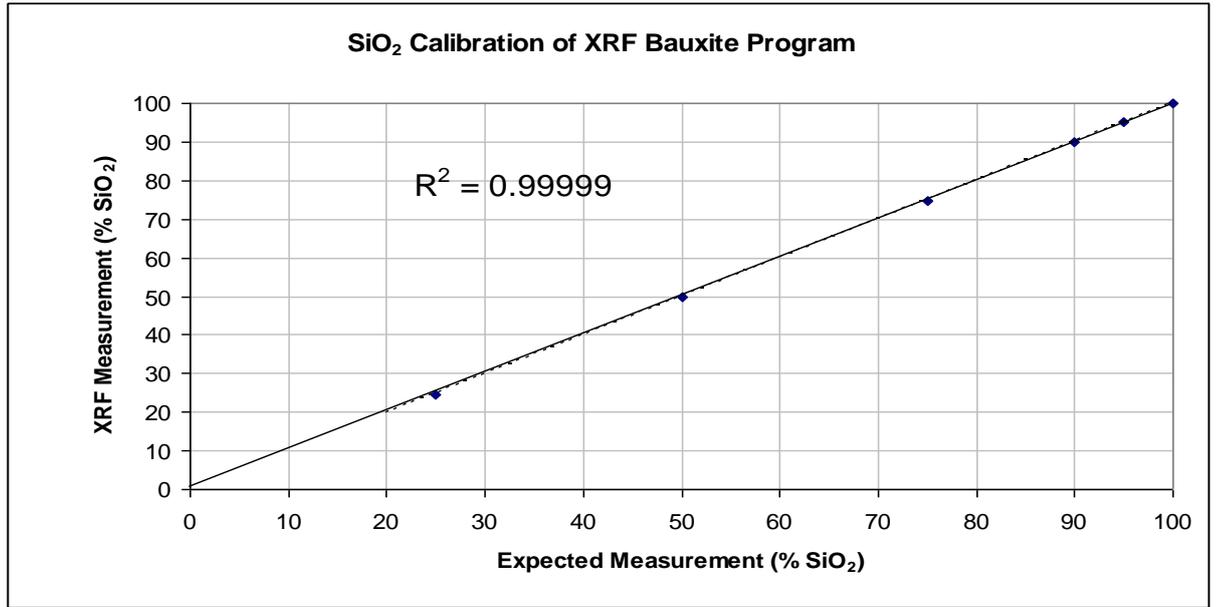


Figure 3.1 Silica Calibration

The results show that the silica calibration on the XRF bauxite program of Kwinana Laboratory's XRF is accurate and the differences between the measured and expected results are probably due to the normal error associated with the analysis.

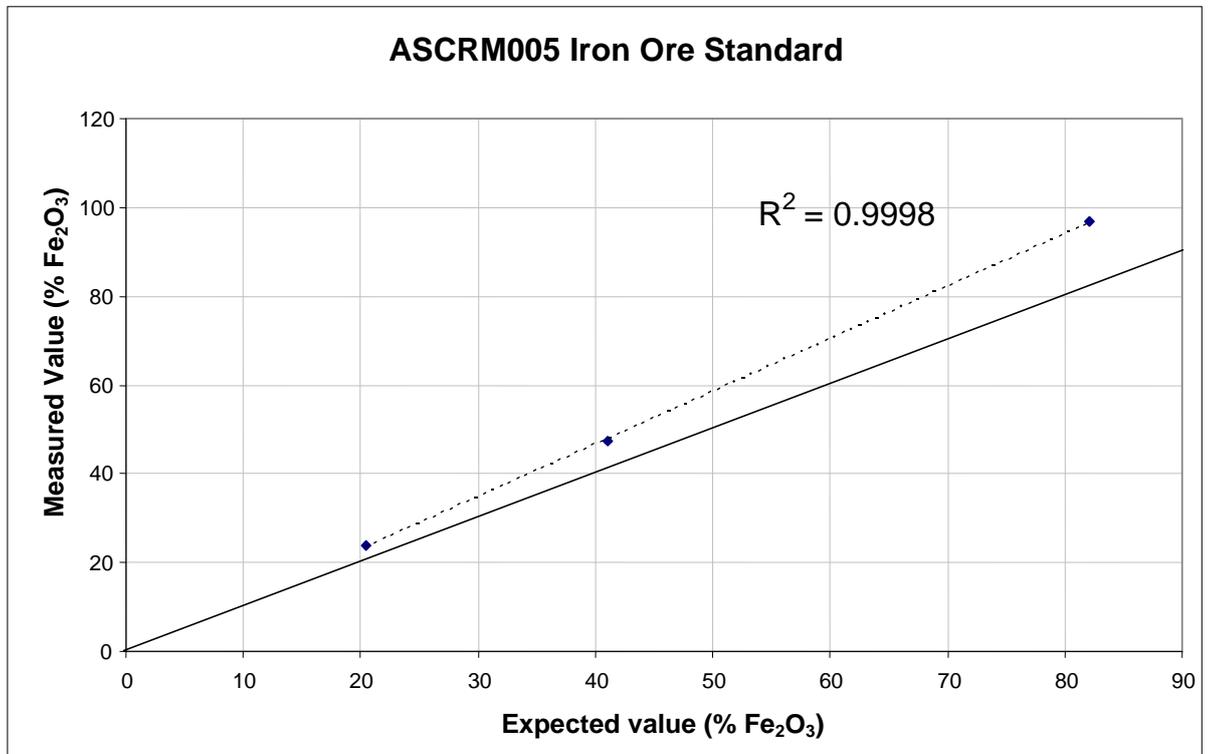


Figure 3.2 Results for ASCRM005 iron oxide standard

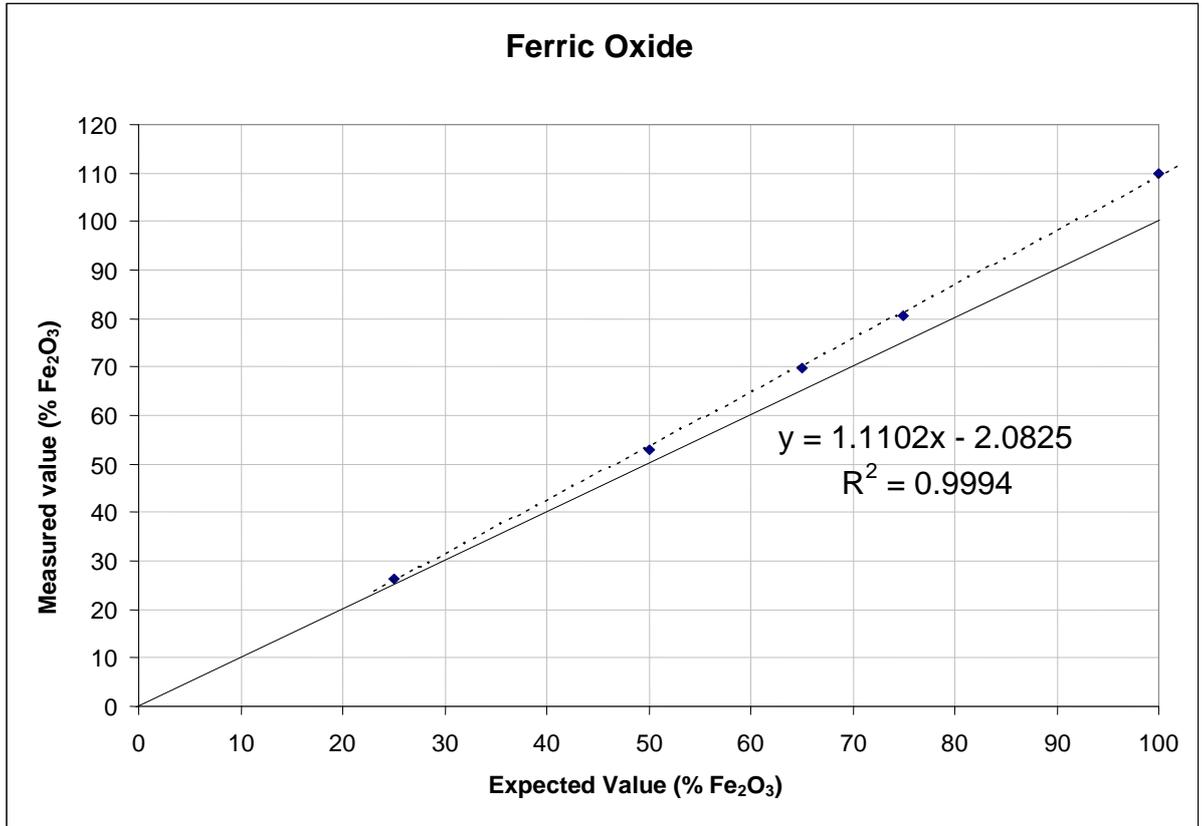


Figure 3.3 Results for Ferric Oxide

The ASCRM005 standard has an LOI (Loss on Ignition) of 8.84% factoring this in gives the following:

Table 3.2 Fe₂O₃ Results for ASCRM005 Adjusted for LOI

| | % Fe ₂ O ₃ Adjusted for LOI | Expected % Fe ₂ O ₃ |
|---------------|---|---|
| ASCRM005 100% | 88.25 | 82.09 |
| ASCRM005 50% | 43.10 | 41.05 |
| ASCRM005 25% | 21.82 | 20.52 |

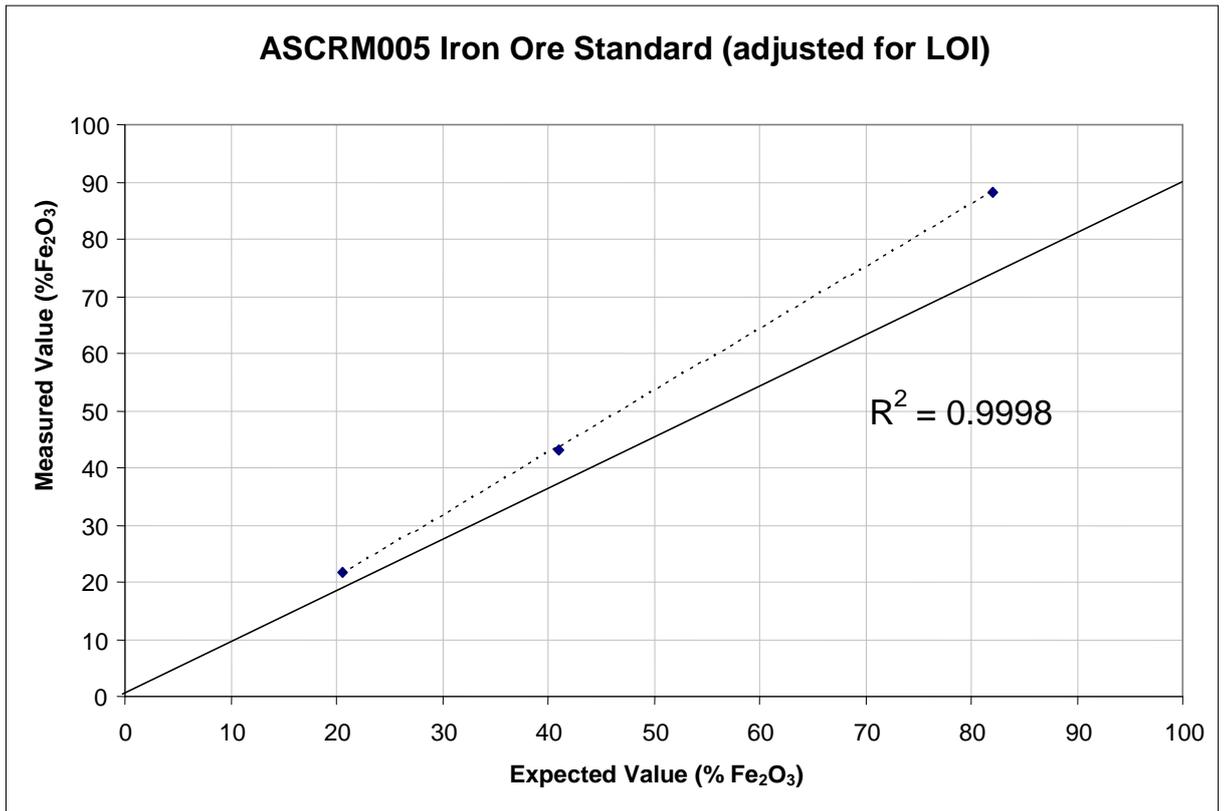


Figure 3.4 Results for ASCRM005 iron oxide standard adjusted for LOI

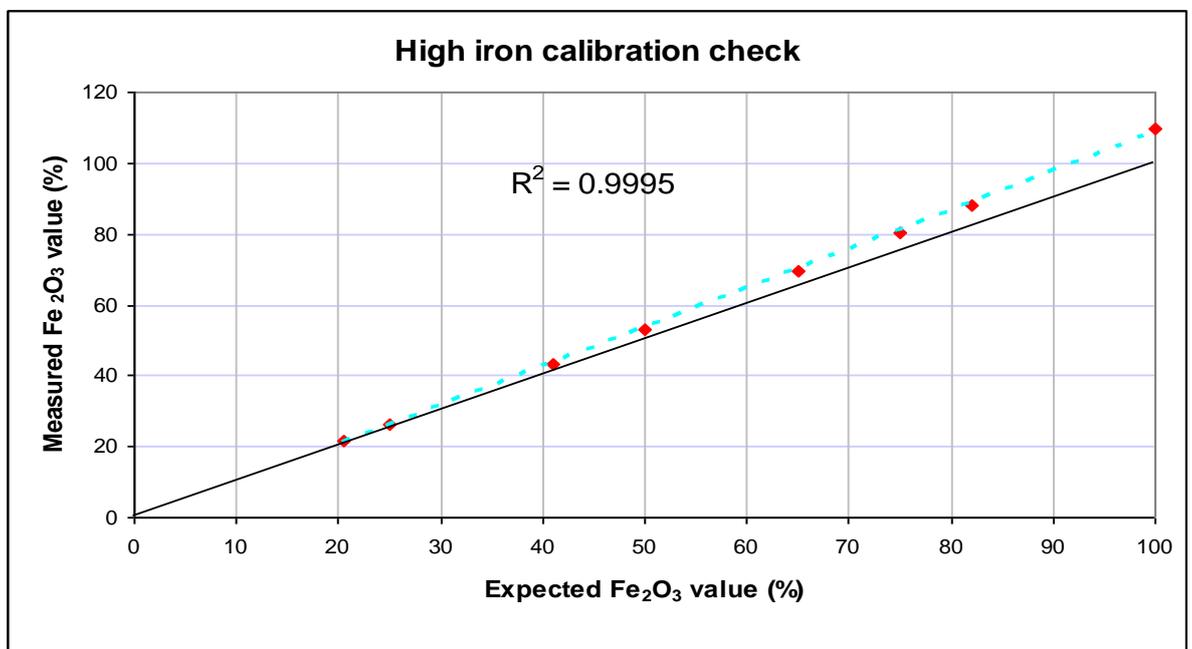


Figure 3.5 Combined Ferric Oxide and ASCRM005 Standard (adjusted for LOI)

The iron results of the ASCRM005 iron ore standard (figure 3.2) show a bias on the high side. The bias increased with increasing iron oxide concentration.

This bias is reduced once the LOI is taken into account (see figure 3.4), but is not eliminated and the increase in the bias with increasing iron oxide concentration is still present.

The ferric oxide (figure 3.3) results are also biased high and like the ASCRM005 iron ore standard the bias increases with increasing iron oxide concentration.

Combining the results for ferric oxide and the ASCRM005 iron ore standard (see figure 3.5) gives a wider range of concentrations with which to check the calibration. The combined results show the same high bias which increases with increasing iron oxide concentration.

RECOMMENDATIONS

The iron calibration for the bauxite program is checked with a standard with an iron oxide concentration of 17%. This is below the level of the iron oxide in the KH09 control sample (% Fe₂O₃ ~ 18.8%).

The XRF bauxite program should be recalibrated for iron or a separate program set up for analysis of samples with iron oxide concentrations greater than 20%.

4.0 Results and Discussion

Samples of coarse residue sand had been produced in the laboratory. While markets were being sought to determine if the production of sand could have an economic outcome, the details of the production process were being assessed in light of the potential products. The criteria for construction sand were easy to achieve while the specifications for concrete sand and plasterer's sand are very tight and thus more difficult to achieve.

Hence the market would decide the criteria and the criteria would determine the process.

It was during this iterative process that the sand was assessed for magnetic separation. It was found that earlier reports of a high iron fraction were indeed accurate and that separation appeared relatively easy, provided the criteria were met. Determining those criteria was a significant part of this project and had major implications for the ultimate production process. This was because it was deemed that the production of an iron rich material would potentially provide the economic driver for implementation. It was also determined that the iron rich fraction held a high content of alumina that would preferably be removed to achieve premium product, this process is described below.

4.1 Initial Magnetic Separation Trials

Magnetic Separation of Wagerup Residue Sand

Approximately half of the residue produced by the three WA refineries is a coarse fraction termed "Red Sand™". This represents about 20,000 tonnes per day and approximately 30% of Alcoa World Alumina (AWA) residue by volume. This "Red Sand™" can be processed through standard mineralogical processes, e.g. Low Intensity Magnetic Separator (LIMS) or a Wet High Intensity Magnetic Separator (WHIMS).

One of the fractions that can be separated from the Red Sand™ is high in iron oxides. This report looks at treatment options for improving the suitability of this fraction for use as an iron ore, specifically by removal of the alumina content. This

section is a summary of data presented in (Jones, A. 2005). Magnetic Separation of Wagerup Residue Sand. Alcoa Report AWA 7.05 CAB002.

Method

Approximately 60 kgs of Wagerup Residue Sand was passed through a Reading Induced Roll magnetic separator at Curtin University’s Bentley Campus. The sand was first passed through a sieve to remove any material greater than 2 mm.

The Red Sand was then passed through the magnetic separator with the magnetic field strength dial setting at 240 (the magnetic intensity vs. dial setting is shown in Figure B1 Appendix B), in order to pull any slightly magnetic material out of the sand to produce a high silica sand for possible use in manufacturing of concrete.

The fraction with the magnetic material in was then reprocessed through the magnetic separator but this time with the magnetic field strength dial setting at 20 to try to produce a high iron fraction which it is hoped could in future be used as a feedstock for pig iron production.

The non-magnetic material from the low magnetic field strength setting was labelled as “middlings”.

Results

Table 4.1 XRF Results of Magnetically Separated Wg Red Sand™

| | <i>XRF_BAUX</i> | | | | | | | | | |
|---------------------------------|------------------------------------|------------------------------------|------------------------|------------------------|------------|------------------------|------------------------------------|-----------------------------------|-----------------------|--------------|
| | Al₂O₃ | Fe₂O₃ | Na₂O | SiO₂ | CaO | TiO₂ | Ga₂O₃ | P₂O₅ | K₂O | Total |
| SAMPLE NAME | % | % | % | % | % | % | % | % | % | % |
| Non-Magnetics (Run at Mags 240) | 1.78 | 2.24 | 0.01 | 93.38 | 0.3032 | 0.1545 | 0.0004 | 0.0041 | 0.0388 | 97.91 |
| Middlings | 6.91 | 19.33 | 0.21 | 67.71 | 0.4019 | 0.8233 | 0.0040 | 0.0187 | 0.0649 | 95.47 |
| Magnetics (Run at Mags 20) | 15.38 | 54.77 | 0.43 | 17.72 | 0.3686 | 2.7658 | 0.0116 | 0.0408 | 0.0714 | 91.56 |

XRF results show that the non-magnetic fraction has a silica content of 93%. This fraction was subsequently washed to try to remove some of the fine red dust which was coating the sand particles in this fraction. It is thought that this fine red dust contains most of the iron present in this fraction of the sand. The washed, high silica portion of the sand was then given to BGC for testing for concrete use.

The “high” iron magnetic (labelled as Mags 20 magnetics) fraction of the sand contains ~ 55% iron as Fe₂O₃ (38% Fe). Pig iron requires a feedstock with a Fe₂O₃ content > 60%.

To determine if further processing could increase the % Fe₂O₃ the magnetic (Mags 20 magnetics) was then reprocessed through the magnetic separator with a lower magnetic field strength dial setting of 10. The magnetic fraction that was separated off was labelled as “Mags 10 magnetics”. The non-magnetic fraction is the fraction that remains after the highly magnetic material was separated off and was labelled as Mags 10 non-magnetic.

A portion of the Mags 10 magnetic fraction was then reprocessed through the magnetic separator with a field strength dial setting of 5 in an attempt to further enrich the iron content of the Red Sand™. The separated fractions were labelled as Mags 5 magnetics and Mags 5 non-magnetics.

Table 4.2 XRF Results for the Reprocessed Magnetics Fraction of Wg Red Sand™

| SAMPLE NAME | XRF_BAUX | | | | | | | | | Total |
|-----------------------|----------|-------|------|-------|--------|--------|--------|--------|--------|-------|
| | Al2O3 | Fe2O3 | Na2O | SiO2 | CaO | TiO2 | Ga2O3 | P2O5 | K2O | |
| | % | % | % | % | % | % | % | % | % | % |
| Mags 10 Magnetics | 21.32 | 59.70 | 0.34 | 8.95 | 0.2642 | 3.0337 | 0.0153 | 0.0443 | 0.0514 | 93.68 |
| Mags 10 Non-Magnetics | 11.50 | 50.83 | 0.50 | 24.45 | 0.4164 | 2.1566 | 0.0093 | 0.0375 | 0.0764 | 89.94 |
| Mags 5 Magnetics | 26.17 | 62.11 | 0.25 | 4.82 | 0.1626 | 2.3106 | 0.0179 | 0.0470 | 0.0330 | 95.88 |
| Mags 5 Non-Magnetics | 14.02 | 57.80 | 0.46 | 14.37 | 0.3865 | 3.9287 | 0.0108 | 0.0372 | 0.0716 | 91.04 |

The high iron samples Mags 5 and Mags 10 magnetics have 26% and 21% Al₂O₃ respectively, which is too high if it is to be used as an iron ore and further processing is therefore required.

The first step was to attempt an atmospheric digest using 8% NaOH to leach out the Al₂O₃.

Table 4.3 XRF Results for Atmospheric Digest Residues

| SAMPLE NAME | XRF_BAUX | | | | | | | | | Total |
|---------------------------------|--------------------------------|--------------------------------|-------------------|------------------|--------|------------------|--------------------------------|-------------------------------|------------------|-------|
| | Al ₂ O ₃ | Fe ₂ O ₃ | Na ₂ O | SiO ₂ | CaO | TiO ₂ | Ga ₂ O ₃ | P ₂ O ₅ | K ₂ O | |
| | % | % | % | % | % | % | % | % | % | |
| Mags 5 Magnetics digest residue | 26.44 | 63.09 | 0.39 | 4.19 | 0.1396 | 2.0026 | 0.0166 | 0.0459 | 0.0241 | 96.33 |
| Mags 5 Non-Mags digest residue | 13.18 | 59.01 | 0.82 | 16.25 | 0.5805 | 4.0186 | 0.0099 | 0.1553 | 0.0718 | 94.10 |

The XRF results for the atmospheric digest residues show that the Al₂O₃ is not readily extracted from the samples.

The next step was to do a 100 ml bomb digest at 145°C using 8% NaOH on ground and unground samples, the samples were digested at temperature for 15 minutes. Ground samples were used in the hope that this would make the alumina more accessible to the NaOH and thus increase the extraction of the Al₂O₃.

Table 4.4 XRF Results for 8% NaOH 100 ml Bomb Digest Residues

| SAMPLE NAME | XRF_BAUX | | | | | | | | | Total |
|---------------------------------|--------------------------------|--------------------------------|-------------------|------------------|--------|------------------|--------------------------------|-------------------------------|------------------|-------|
| | Al ₂ O ₃ | Fe ₂ O ₃ | Na ₂ O | SiO ₂ | CaO | TiO ₂ | Ga ₂ O ₃ | P ₂ O ₅ | K ₂ O | |
| | % | % | % | % | % | % | % | % | % | |
| Mag 5 Magnetics start | 25.84 | 62.78 | 0.31 | 5.57 | 0.2934 | 2.5631 | 0.0183 | 0.0549 | 0.0356 | 97.4 |
| Mag 5 Magnetics ground 8% NaOH | 23.46 | 67.21 | 0.41 | 5.21 | 0.269 | 2.6275 | 0.0154 | 0.0463 | 0.022 | 99.22 |
| Mag5 Magnetics Unground 8% NaOH | 23.49 | 65.97 | 0.35 | 5.83 | 0.2046 | 2.9244 | 0.0149 | 0.0428 | 0.0348 | 98.82 |

The XRF results for the 8% NaOH digest residues show that very little (~ 2.5%) of the Al₂O₃ was extracted.

The next step was to repeat the 100 ml bomb digests this time using 33% NaOH instead of 8%. Again ground and un-ground samples were used.

Table 4.5 XRF results for 33% NaOH 100 ml Bomb digest residues

| SAMPLE NAME | XRF_BAUX | | | | | | | | | |
|-----------------------------------|--------------------------------|--------------------------------|-------------------|------------------|--------|------------------|--------------------------------|-------------------------------|------------------|-------|
| | Al ₂ O ₃ | Fe ₂ O ₃ | Na ₂ O | SiO ₂ | CaO | TiO ₂ | Ga ₂ O ₃ | P ₂ O ₅ | K ₂ O | Total |
| | % | % | % | % | % | % | % | % | % | % |
| Mags 5 Magnetics start | 25.84 | 62.78 | 0.31 | 5.57 | 0.2934 | 2.5631 | 0.0183 | 0.0549 | 0.0356 | 97.40 |
| Mags 5 Magnetics ground 33%NaOH | 20.31 | 70.63 | 0.38 | 4.76 | 0.3250 | 2.8290 | 0.0139 | 0.0386 | 0.0174 | 99.27 |
| Mags 5 Magnetics unground 33%NaOH | 22.82 | 68.59 | 0.55 | 4.80 | 0.1279 | 2.2879 | 0.0143 | 0.0396 | 0.0229 | 99.22 |

The XRF results show that the higher strength NaOH used has leached slightly more Al₂O₃ than in the lower strength NaOH, with the ground sample having slightly higher extraction than the unground sample.

The fact that very little of the Al₂O₃ was extracted even when digested in a 100 ml bomb with 33% NaOH suggests that the alumina is locked up in the crystal lattice possibly as aluminous goethite and aluminous haematite.

XRD results showed that the major phase for the high iron sample is haematite, with minor phases being quartz, magnetite, and goethite, with a trace of ilmenite. For the high iron sample the mole percentage of aluminium in goethite is 26% and 9% in haematite.

The next step was to perform a high temperature and high caustic strength digests. The high iron sand was digested in 33% NaOH at 200°C for 40 minutes.

Table 4.6 XRF Results for 1 Litre Autoclave Digest

| SAMPLE NAME | XRF_BAUX | | | | | | | | | |
|----------------------------|--------------------------------|--------------------------------|-------------------|------------------|--------|------------------|--------------------------------|-------------------------------|------------------|--------|
| | Al ₂ O ₃ | Fe ₂ O ₃ | Na ₂ O | SiO ₂ | CaO | TiO ₂ | Ga ₂ O ₃ | P ₂ O ₅ | K ₂ O | Total |
| | % | % | % | % | % | % | % | % | % | % |
| Mags 5 Magnetics | 26.29 | 62.32 | 0.31 | 5.58 | 0.2944 | 2.5305 | 0.0182 | 0.0520 | 0.0389 | 97.44 |
| Mags 5 Mags 200°C 33% NaOH | 8.16 | 83.17 | 1.77 | 4.14 | 0.2103 | 2.8894 | 0.0067 | 0.0214 | 0.0062 | 100.38 |

Here 50 ml of the digest liquor was diluted with 50 ml of de-ionised water. The diluted liquor was then analysed on the Alian, with four replicates (see Table 4.7 below).

Start weight = 108.7 g

Volume NaOH used = 600 ml

% Al₂O₃ in start sample by XRF = 26.29%

Maximum alumina concentration = 600 mL x 26.29%
= 47.6 g/L

Table 4.7 Alian results for 200°C 33% NaOH Digest using 1 litre autoclave

| | g/L TC | g/L TA | g/L Al ₂ O ₃ | A/TC | TC/TA |
|---------------|--------|--------|------------------------------------|--------|--------|
| 33% OH 200°C | 298.63 | 298.60 | 17.54 | 0.0587 | 1.0001 |
| 33% OH 200°C | 298.68 | 298.60 | 17.74 | 0.0594 | 1.0003 |
| 33% OH 200°C | 296.97 | 300.05 | 17.98 | 0.0605 | 0.9925 |
| 33% OH 200°C | 297.20 | 299.45 | 18.05 | 0.0607 | 0.9925 |
| A v e r a g e | 297.87 | 299.18 | 17.83 | 0.0599 | 0.9956 |

TC of undiluted liquor = 297.87 x 2

= 595.7 g/L

Undiluted Al₂O₃ concentration = 17.83 x 2

= 35.7 g/L

% Extracted = 35.7 g/L ÷ 47.6 g/L × 100

= 74.9 %

Alian results suggest that about 75% of the total alumina present in the high iron fraction of the sand was extracted.

XRF results showed that approximately 69% of the total alumina in the start sample had been extracted through a high temperature digest. These results were in good agreement with the liquor analysis results.

XRD analysis of the digested and undigested samples shows the mole percentage of Al in goethite has reduced from 27 to 22 % in the digested sample and the mole percentage Al in haematite has reduced from 8 to 6 % in the digested sample.

This is consistent with high Al-substituted goethite preferentially dissolving and precipitating as low Al mole percentage haematite.

It is believed that the large particle size of the quartz grains and the moderately high temperature of 200°C, prevented greater dissolution of SiO₂.

Conclusion

A high temperature digest using high strength caustic gives the best alumina extraction of the three digest methods used.

XRD results indicate that the alumina is being extracted from the high Al substituted goethite and precipitating low Al substituted haematite.

Extraction of the alumina from the high iron fraction of Wagerup residue sand concentrates the Fe₂O₃ component from 62 (43% Fe) to 83 % (58% Fe). At the same time the alumina concentration decreases from 26% to 8% for this fraction. This will probably be more suitable as feed stock for the production of pig iron than the undigested high iron sand.

Recommendations

The high iron fraction “Mags 5” represents only a small percentage (~ 2%) of Red Sand™. A high temperature/caustic strength digest of the “Mags 20” magnetic fraction would allow for a greater proportion (~25%) of the Red Sand™ to be used as feed stock for the production of pig iron.

Re-use of the “Mags 20” magnetic fraction would reduce the total amount of AWA residue by approximately 10% (by volume).

To determine the best starting location for the production of Red Sand™, an investigation was made into the various locations and potential separation sites. Samples from these sites were used to determine the best site, location and process for separation. Further processing was assessed to refine the separation process that would give a product of suitable quality for construction, would be environmentally acceptable for use, and have the potential for beneficiation using magnetic separation. The process is outlined below.

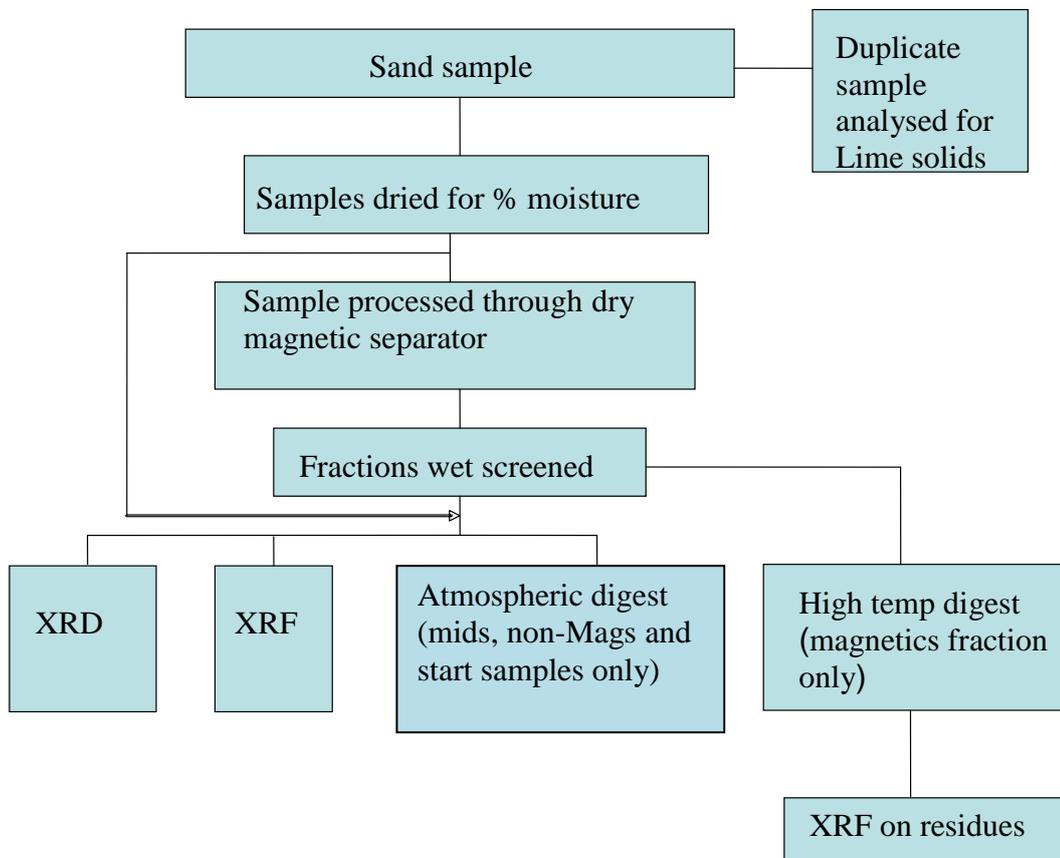


Figure 4.1 Mineralogical Investigation of Western Australian Red Sand™

4.2 Preliminary Mineralogical Investigation of Western Australian Operations Red Sand™

This section is a summary of work reported to Alcoa in Jones, A., Jamieson, E., Cooling, D., Dye, S. (2005). Atmospheric Digest of Pinjarra Residue Sand. Alcoa Report AWA 7.05 CAB001: and also in Jones, A. (2009). Mineralogical Investigation of WAOP's Sand. Alcoa Report AWA 7.05 CAB023.

Sand samples from the sand rake classifier and sand separation plant (SSP) were collected from Alcoa's three Western Australian refineries (Kwinana (Kw) Pinjarra (Pj) and Wagerup (Wg)) and a preliminary mineralogical investigation performed (a more detailed investigation of the Red Sand™ from the refineries is discussed later).

The XRF results shown below in Table 4.8 indicate that the main components of the sand samples were SiO₂, Fe₂O₃ and Al₂O₃ with no major variation between the sites.

Table 4.8 XRF Results for WA Sand snapshot samples

| | Al ₂ O ₃ | SiO ₂ | Fe ₂ O ₃ | TiO ₂ | CaO | Na ₂ O | K ₂ O | Ga ₂ O ₃ | P ₂ O ₅ | Total |
|--------------|--------------------------------|------------------|--------------------------------|------------------|--------|-------------------|------------------|--------------------------------|-------------------------------|-------|
| Sample | (%) | (%) | (%) | (%) | (%) | (%) | (%) | (%) | (%) | (%) |
| Kw rake sand | 12.71 | 59.56 | 19.18 | 0.7402 | 0.6593 | 0.23 | 0.0704 | 0.0054 | 0.0210 | 93.17 |
| Kw SSP sand | 11.13 | 60.59 | 22.70 | 0.9285 | 0.2740 | 0.40 | 0.0660 | 0.0062 | 0.0190 | 96.12 |
| Pj rake sand | 11.98 | 57.83 | 22.38 | 0.7745 | 0.0770 | 0.98 | 0.0667 | 0.0063 | 0.0180 | 94.11 |
| Pj SSP sand | 10.53 | 62.35 | 19.80 | 0.8007 | 0.3819 | 0.54 | 0.0934 | 0.0050 | 0.0186 | 94.51 |
| Wg rake sand | 11.42 | 56.76 | 24.92 | 0.5099 | 0.0926 | 1.08 | 0.0480 | 0.0058 | 0.0159 | 94.84 |

The XRD results are shown in Table 4.9. Results showed that the major mineral phase present in the sand samples was quartz (with the Pj rake sand being an exception with gibbsite also present as a major mineral phase), with haematite, gibbsite and goethite being the minor phases present. The XRD results were consistent with the XRF silica results, but not the XRF iron oxide results. This disagreement is because XRD analysis estimates if a particular phase is present as either a major, minor or trace amount based on peak heights alone. This often leads to an over-estimate of phases that are crystalline (e.g. quartz, gibbsite),

crystallographically highly symmetric or have high diffracting structure factors, and under-estimates phases that are finely or poorly crystalline (such as iron oxides in bauxites and bauxite residues), low crystal symmetry or low structure factor (Pearson 2008).

The Kw sand rake sample has the highest % Tricalcium Aluminate or TCA6 (as determined by bomb digest) of all the sand samples. This was a result of the digester unit which feeds the sand rake sampled receiving lime supercharging at that time. The XRF results show that the CaO results for the Kw rake sand were higher than for the other sand samples. The XRF CaO was in agreement with the TCA6 results.

The Pj rake classifier sand sample had lower CaO and TCA6 results than the Pj sand separation plant sample. This was due to the individual digester unit which feeds the rake classifier sampled not receiving lime supercharging, whereas the sand separation plant received the sand from all of the digester units and one or more of the other digesters would have had lime supercharging thus resulting in the sand separation plant having higher TCA6 and CaO levels than the individual sand rake that was sampled. The trend for the TCA6 results was in agreement with that for the XRF CaO results.

Table 4.9 XRD, Lime Solids, Percentage Moisture and Available Alumina Results of WA Sand Snapshot samples

| Sample | XRD analysis results | | | % Moisture | Lime Solids | |
|--------------|----------------------|-------------------------------|----------------------|------------|---------------------------|--|
| | Significant | Minor | Trace | | %TCA6 wt/wt in dry solids | % Available Al ₂ O ₃ |
| Kw rake sand | Quartz | haematite, gibbsite, goethite | Muscovite | 17.79 | 2.19 | 5.99 |
| Kw SSP sand | Quartz | haematite, gibbsite, goethite | Muscovite, boehmite | 20.08 | 0.36 | 5.41 |
| Pj rake sand | Quartz, gibbsite | haematite, goethite | Muscovite | 21.12 | 0.28 | 8.40 |
| Pj SSP sand | Quartz | haematite, gibbsite, goethite | Muscovite, calcite | 17.68 | 0.47 | 4.06 |
| Wg rake sand | Quartz | haematite, gibbsite, goethite | Muscovite, kaolinite | 21.2 | 0.31 | 6.76 |

4.3 Detailed Mineralogical Investigation of Magnetically Separated Western Australian Operations Red Sand™

After drying to determine the percentage moisture, the sand rake classifier and sand separation plant samples were passed over a Reading induced roll dry magnetic separator (figure 4.2) at Curtin University.

The objective of the magnetic separation was to produce a fraction that is high in iron oxides (magnetic fraction) and another fraction that was low in iron oxides and high in silica. With this objective in mind the magnetic separation was done in two stages. The first of these, with a low magnetic field strength of approximately four thousand Gauss (0.4 Tesla) produces two sand fractions: a magnetic fraction high in Fe₂O₃ and a “non-magnetic” fraction which is similar to the original Red Sand™.

In the second stage of the separation process the “non-magnetic” fraction was then re-passed over the magnetic separator with a field strength of approximately 15000 Gauss (1.5 Tesla), which then produced two sand fractions. The first fraction was similar to the original Red Sand™ and will be known as the Middlings (mids) fraction and a second fraction high in silica will be known as the non-magnetic fraction.

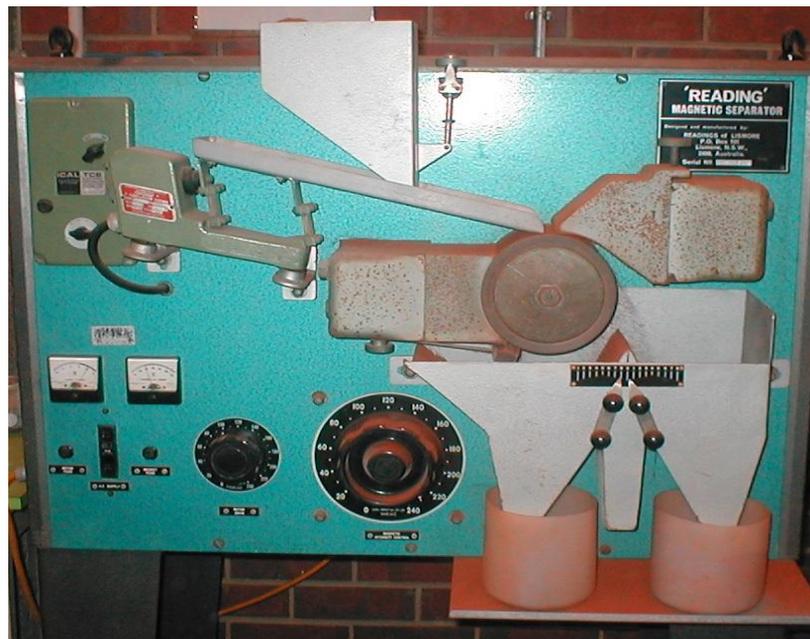


Figure 4.2 Reading Induced Roll Magnetic Separator

Table 4.10 Magnetic Separation of WA Sand Samples

| Sample | % Non Magnetics | % Middlings | % Magnetics |
|--------------|-----------------|-------------|-------------|
| Kw rake sand | 65.41 | 22.35 | 12.24 |
| Kw SSP sand | 61.20 | 23.13 | 15.67 |
| Pj rake sand | 60.78 | 34.60 | 4.62 |
| Pj SSP sand | 62.25 | 30.50 | 7.25 |
| Wg rake sand | 59.15 | 31.47 | 9.39 |

The non-magnetic fraction made up approximately 60% of the sand which was consistent with the XRF SiO₂ results for the original sand samples. The data in table 4.10 is also presented in figure 4.3.

The magnetic fraction made up approximately 4.5% to 15.5% of the original samples. This was lower than the XRF Fe₂O₃ result of approximately 19 to 25% Fe₂O₃ in the original samples. This would suggest that the Fe₂O₃ present in the samples was not all highly magnetically susceptible. The XRD analysis showed that the main iron oxide phases present were haematite and goethite. Due to the differences in the magnetic susceptibility (the magnetic susceptibility of haematite is higher than that of goethite); haematite is more likely to report to the magnetic fraction, whilst the goethite is more likely to report to the middlings fraction.

The presence of the middlings fraction was due to the objective of the magnetic separation process, being to produce a fraction high in iron oxides and a second fraction low in iron oxides and high in silica.

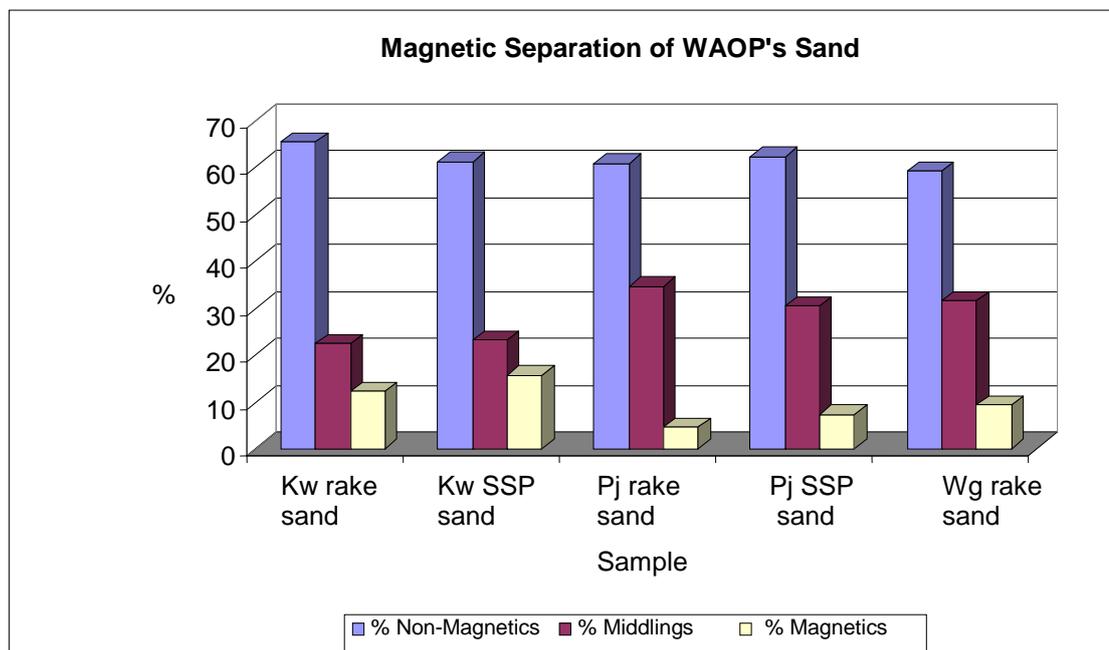


Figure 4.3 Magnetic Separation of Rake & Sand Separation Plant Samples

4.4 Refinery Sands Processing

This section outlines the mineralogical and processing survey of the key locations at Alcoa's three Western Australian refineries from which sand could be taken for beneficiation and reuse. These sites being:

- 1) Kwinana Sand Rake
- 2) Kwinana Sand Separation Plant
- 3) Pinjarra Sand Rake
- 4) Pinjarra Sand Separation Plant
- 5) Wagerup Sand Rake

The three fractions resulting from the magnetic separation process of each of the refinery rake classifier and sand separation plant samples were sized using test sieves of 850, 600, 425, 250, 150, and 75 micron apertures. Various analyses were then performed on these size fractions, the results of which are discussed in the sections that follow.

4.4.1 Kwinana Sand Rake

Magnetics Fraction:

From the XRF & XRD results of this fraction, the Al_2O_3 content of the magnetic fraction ranged from 20% to 30%. The trend was that the Al_2O_3 content also decreased with decreasing particle size.

The XRD analysis did not show the presence of any boehmite except for in the +850 μm fraction. The boehmite content of WA bauxite is low and most would report to the mud fraction after digestion. This would indicate that the Al was more likely to be present as aluminous goethite and aluminous haematite which does not show up using an id scan on the XRD (an id scan identifies is a qualitative analysis by XRD which identifies the major and minor peaks of the x-ray diffraction pattern through the use of search/match software with a database containing the x-ray diffraction pattern of minerals).

The Fe_2O_3 content of the magnetic fraction ranged from 46% to 58%, the coarsest size (+850 μm) fraction having the lowest Fe_2O_3 content and the finest size (-75 μm) fraction having the highest. The XRD results showed that haematite was present as a significant mineral phase in all of the size fractions; magnetite and goethite were also present as major mineral phases in all of the size fractions greater than 250 μm . The XRD results were in agreement with the XRF Fe_2O_3 results.

The SiO_2 content ranges from 5.5% in the + 850 μm fraction to 11% in the 150 to 250 μm fraction. This is likely to be due to entrained silica that was carried over with the magnetic fraction during the separation process.

Quartz is listed as a significant phase in all of the size fractions. This does not agree with the XRF results which showed the SiO_2 content ranging from 5 to 11%. The discrepancy is due to the previously mentioned error in the XRD analysis.

The Na_2O content increased with increasing particle size. This was probably due to liquor entrained in the crevices and pores of the particles (coarser particles have more pores and crevices than finer particles). The Na_2O content of the fractions below 150 μm were the exception, possibly due to desilication product (DSP) being present in the finer fractions.

High Temperature Digestion:

High temperature digestions were performed on the magnetics fraction with the objective of recovering the Al_2O_3 and upgrading the Fe_2O_3 content.

The high temperature digestion results showed a general trend that the coarsest fractions have the highest alumina extraction, with the exception of the -75 μm fraction which showed a higher Al_2O_3 extraction than in the 75 to 150 μm fraction.

The XRD analysis showed the presence of gibbsite as a major phase in the coarsest (+850 μm) fraction and as a minor phase in all of the other fractions except for the

150 to 250 μm fraction. This gibbsite is present in the sand due to incomplete extraction in the Bayer leach.

Comparison of the XRF analysis of the magnetic fraction and the residues from the high temperature (250°C) digestion (Figure 4.4) showed that approximately 30 to 80% of the Al_2O_3 present in the various size fractions was recovered, with the highest recovery being in the coarsest size fractions.

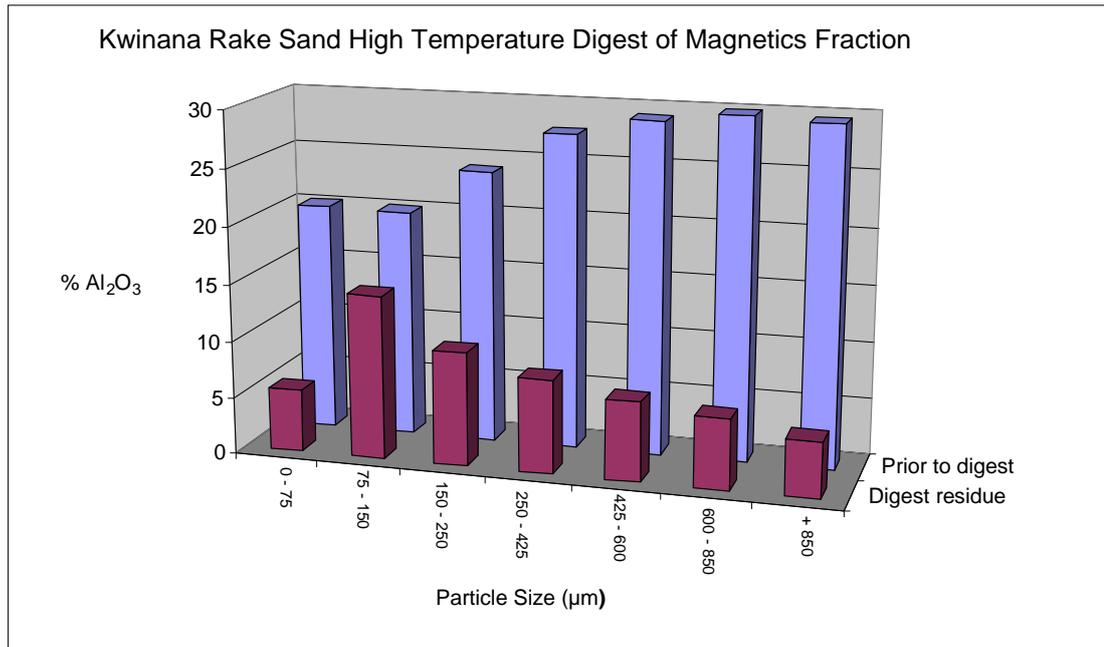


Figure 4.4 Kwinana Rake Sand High Temperature Digest of Magnetics Fraction Al_2O_3 Results

The Fe_2O_3 concentration of the digest residues was between 15 and 75% higher than in the start samples with the exception of the 75 to 150 μm fraction where the concentration decreased by ~ four percent, the reason for this decrease is not known.

The digest residues for the coarsest fractions showed the largest increase in Fe_2O_3 content. This corresponds with the digest residues of these fractions having the largest decrease in Al_2O_3 content.

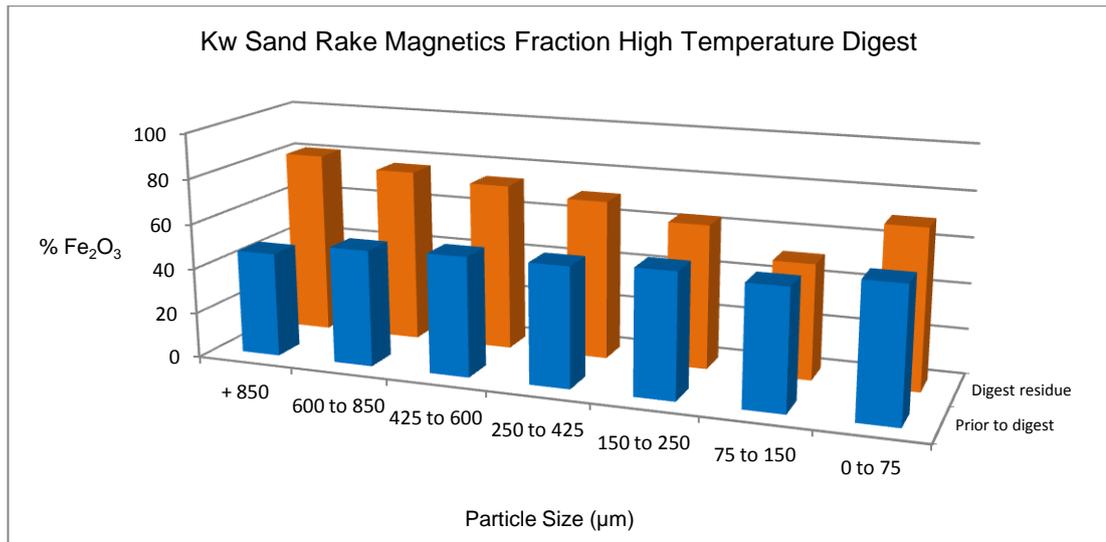


Figure 4.5 Kwinana Rake Sand High Temperature Digest of Magnetics Fraction Fe₂O₃ Results

The XRF results for the digest residues showed the Na₂O levels increasing as the particle size decreased (with the exception of the – 75 µm fraction. The increase in Na₂O indicates possible DSP formation as a result of the dissolution of some of the quartz during the high temperature digest.

Middlings Fraction:

The XRF & XRD results showed the Al_2O_3 content ranged from 17 to 30% and decreased with decreasing particle size down to 150 μm , below which the Al_2O_3 content increased.

The XRD results showed that gibbsite was present as a significant mineral phase in all of the size fractions above 425 μm , and as a minor phase in the other size fractions with the exception of the 150 to 250 μm fraction where no gibbsite was detected.

The Fe_2O_3 content increased with decreasing particle size down to 250 μm below which the level decreased. The Fe_2O_3 content ranged from 37% (<75 μm fraction) to 48% (250 to 425 μm fraction).

Goethite and haematite were detected as significant mineral phases in all of the size fractions. This is in agreement with the Fe_2O_3 XRF results. The iron oxides present in the middlings fraction are mainly due to the way the sand was separated.

The SiO_2 content increased down to 150 μm , below which the level starts to decrease. The SiO_2 content ranged from 7% in the +850 μm size fraction to 19% in the 150 to 250 μm size fraction. Quartz was listed as a significant mineral phase in all of the size fractions below 850 μm . This is in general agreement with the XRF SiO_2 results.

The Na_2O content increased with increasing particle size. This was likely to be due to liquor entrained in the crevices and pores of the larger particles.

Atmospheric Digestion:

The atmospheric digest results followed a similar trend to the XRF total Al_2O_3 results with the Al_2O_3 level decreasing with particle size down to the 150 to 250 μm fraction before increasing again.

The increase in the 75 to 150 μm fraction was possibly due to the presence of DSP in this size fraction. There was insufficient sample to be able to do an atmospheric digest on the -75 μm fraction.

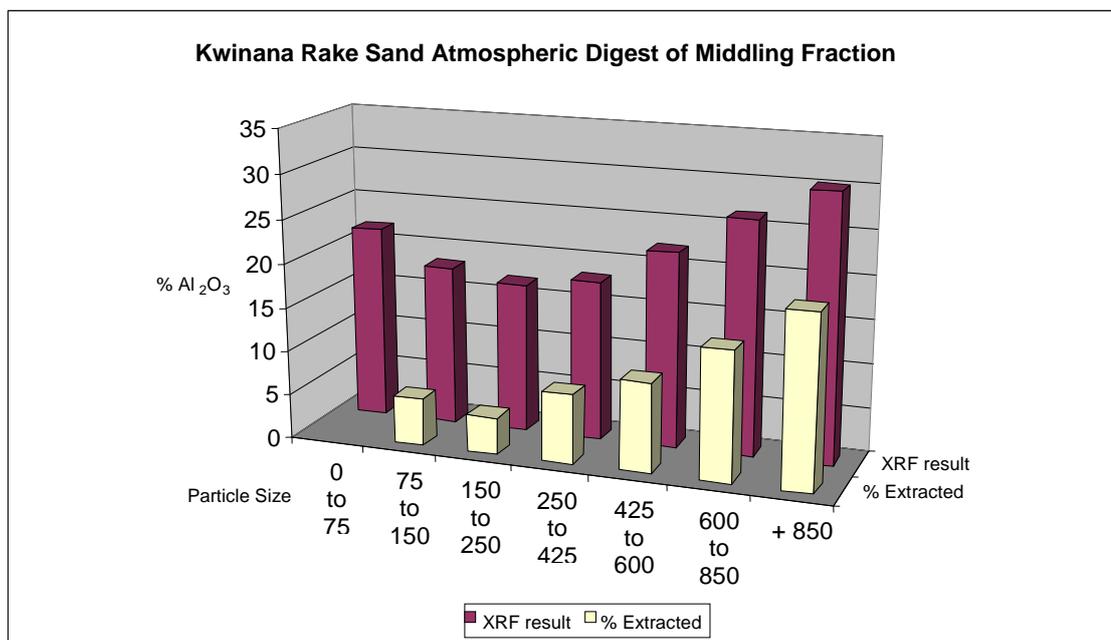


Figure 4.6 Kwinana Rake Sand Atmospheric Digest of Middlings Fraction

Figure 4.6 shows that the recovery from the atmospheric digest of the middlings fraction is less than 100% total Al₂O₃ and varies depending on the size fraction, with the highest recovery being in the +850 µm fraction and the lowest being in the 150 to 250 µm fraction.

The atmospheric digest results suggest that not all of the Al₂O₃ present was gibbsite. XRD results showed boehmite present as a trace mineral phase in the +850 µm fraction. Boehmite is unlikely to be digested at atmospheric digest temperatures especially in the presence of gibbsite which has dissolution kinetics that are much faster than that of boehmite.

However boehmite is unlikely to account for the discrepancy between the atmospheric digest results and the XRF Al₂O₃ results as the level of boehmite in Western Australian bauxite is low and most is likely to report to the mud fraction after digestion. This indicates the Al₂O₃ was present as another mineral phase that does not digest at the low temperatures of an atmospheric digest, and is not easily detectable on an XRD scan. Aluminous goethite and aluminous haematite are the likely phases.

Non-Magnetics Fraction

The XRF & XRD results showed that the Al_2O_3 content decreased with decreasing particle size down to 150 μm , below which the level increased. The increase in Al_2O_3 content in the fractions below 150 μm may be the result of the breakdown of larger alumina particles or conglomerate alumina/gangue particles (Dye & Collett Sept 2005) or that DSP may be present.

The Al_2O_3 is likely to be due to the presence of undigested gibbsite, which is a function of the extraction efficiency of the individual digester. Coarse particles dramatically short circuit relative to the liquor residence time in vertical digesters which are used in the WA refineries [Dye & Collett Aug 2005].

The coarsest fraction had the highest Fe_2O_3 level. The Fe_2O_3 level increased again below 150 μm . This along with the increase in Al_2O_3 and Na_2O for these fractions indicates the presence of DSP; or as a result of the breakdown of conglomerate particles.

The SiO_2 content increased with decreasing particle size down to 150 μm , below which the SiO_2 level decreased. The size fractions 150 to 425 μm had the highest SiO_2 level with the coarsest fractions having the lowest SiO_2 level.

The XRD results showed the major mineral phase present in all of the size fractions was quartz, with the + 850 μm also having gibbsite listed as a major phase and goethite, muscovite and haematite being present in trace amounts. All of the non-magnetics fractions below 850 μm showed gibbsite present in trace amounts. The fractions between 425 μm and 850 μm also showed muscovite and haematite present in trace amounts.

Atmospheric Digestion:

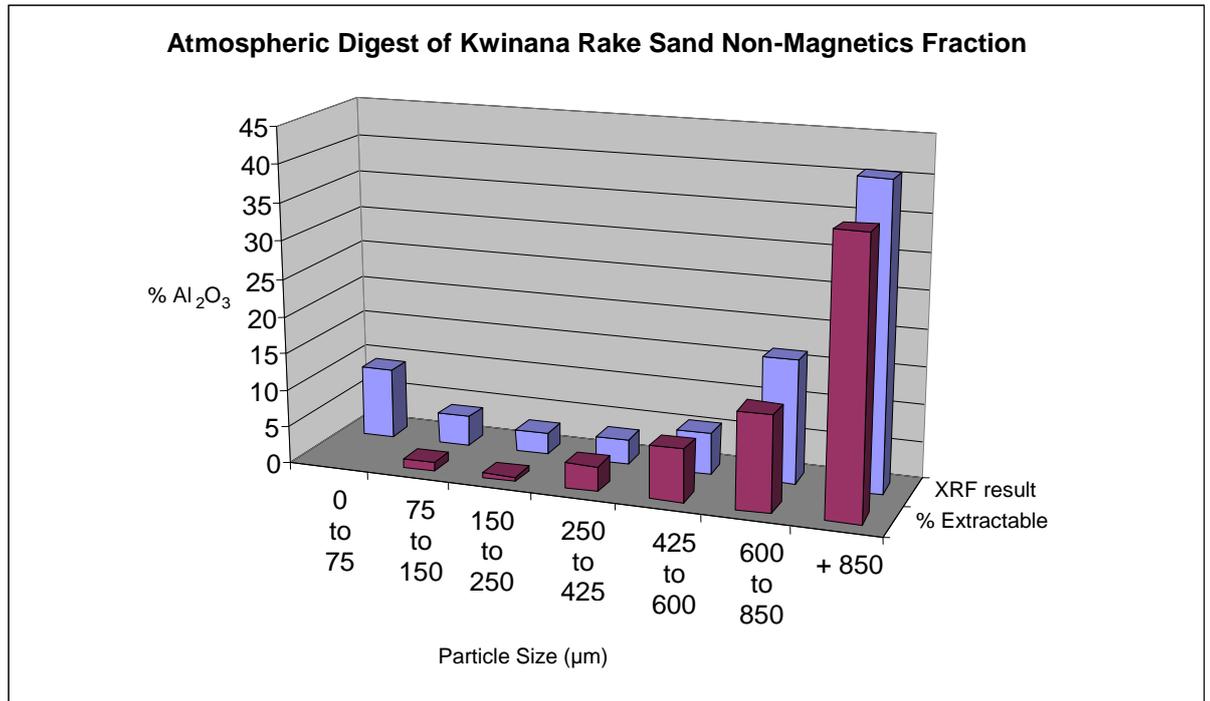


Figure 4.7 Kwinana Rake Sand Atmospheric Digest of Non Magnetics Fraction

The atmospheric digest results showed that the level of extractable Al₂O₃ decreased with decreasing particle size down to 150 µm and that the 75 to 150 µm had a higher extractable Al₂O₃ than the 150 to 250 µm fraction. The increase in extractable Al₂O₃ content in the fraction below 150 µm is likely to be the result of the breakdown of larger alumina particles or conglomerate alumina/gangue particles [Dye & Collett Sept 2005]. There was insufficient sample to be able to do an atmospheric digest on the -75 µm fraction.

The results from the atmospheric digest were generally a little lower than the XRF Al₂O₃ results. This difference may be due to different methods of analysis used – the atmospheric digest liquors were analysed using the Alian titrator (using the fluoride inflection method).

4.4.2 Kwinana Sand Separation Plant

Non-Magnetics Fraction

The XRF & XRD results showed the SiO₂ content increased with decreasing particle size down to 150 µm; below this size the SiO₂ level decreased with decreasing particle size.

The size fractions between 75 and 600 µm had a silica content over 90% indicating that magnetic separation can be used to produce a fraction with a high silica content. These fractions may be suitable for use as a substitute building material.

The trend for iron oxide is that the Fe₂O₃ content decreased with decreasing particle size, with the exception being the -75 µm fraction. The higher Fe₂O₃ content in the -75 µm fraction may be an indicator of the presence of DSP.

The Al₂O₃ content decreased with decreasing particle size down to 150 µm; below this size the Al₂O₃ content increases. The increase in Al₂O₃ content in the fractions below 150 µm is likely to be the result of the breakdown of larger alumina particles or conglomerate alumina/gangue particles (Dye & Collett Sept 2005) but it could also indicate the presence of DSP.

The XRD results showed that in the non-magnetics fraction; gibbsite was present as a major mineral phase in the + 850 µm, and as a minor phase in the fractions below 850 µm. The gibbsite is likely to be undigested gibbsite which is due to the extraction efficiency of the individual digester.

The XRD results are in agreement with the study by Dye & Collett (Aug 2005) which found that coarse particles dramatically short circuit the digesters relative to the liquor residence time in vertical digesters which are used in the WA refineries.

Atmospheric Digestion:

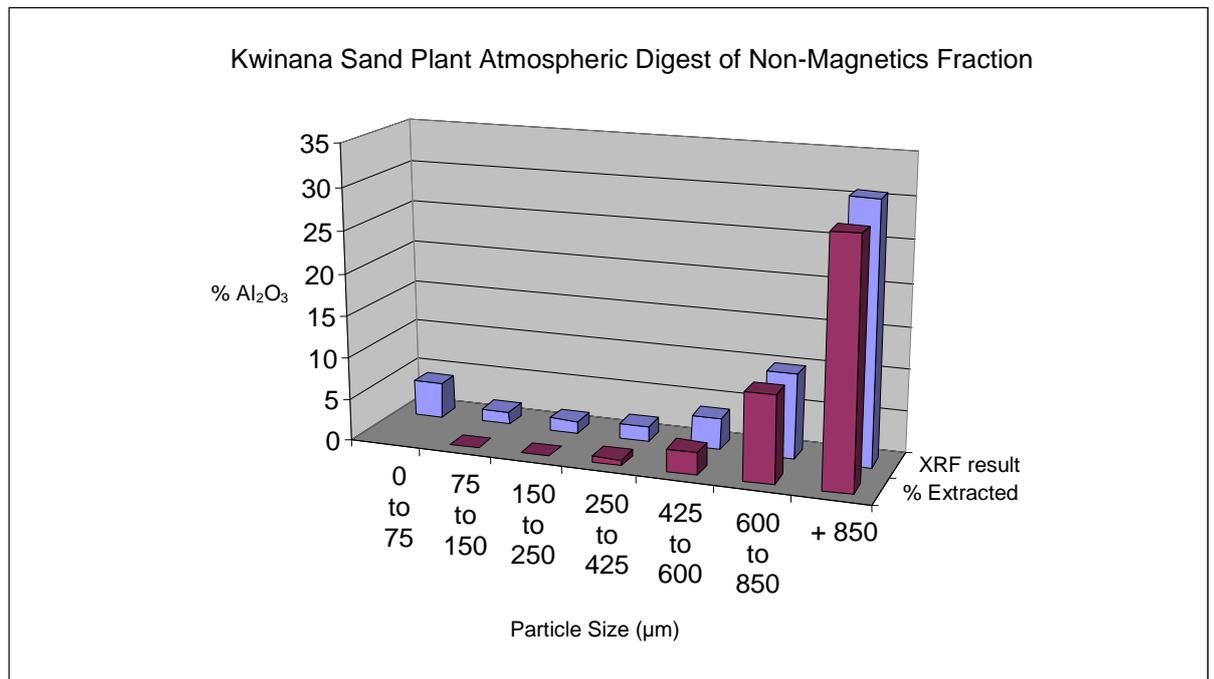


Figure 4.8 Kwinana Sand Separation Plant Atmospheric Digest of Non-Magnetics Fraction

The atmospheric digest results followed the same trend as the Al_2O_3 XRF results with the coarsest fractions having the highest extractable alumina content and the finer fractions having the lowest. There was insufficient sample to do an atmospheric digest on the $-75 \mu\text{m}$ fraction. The Al_2O_3 level of the size fractions below $150 \mu\text{m}$ was too low to give a sufficiently large Al_2O_3 titre for the atmospheric digest to give a result.

Middlings Fraction

The XRF & XRD results for Al_2O_3 results show that the level decreases with decreasing particle size down to $150 \mu\text{m}$, below this size the level increases. The higher level in the finer fractions may be due to the breakdown of larger alumina particles or conglomerate alumina/gangue particles (Dye & Collett Sept 2005) but it could also indicate the presence of DSP.

XRD results show that gibbsite is present as a major phase in the size fractions down to $250 \mu\text{m}$ and as a minor phase below $150 \mu\text{m}$. Boehmite is present as a trace phase in all size fractions below $850 \mu\text{m}$.

The Al_2O_3 is likely to be undigested gibbsite; which is due to the extraction efficiency of the individual digester. Coarse particles dramatically short circuit relative to the liquor residence time in vertical digesters which are used in the WA refineries (Dye & Collett Aug 2005).

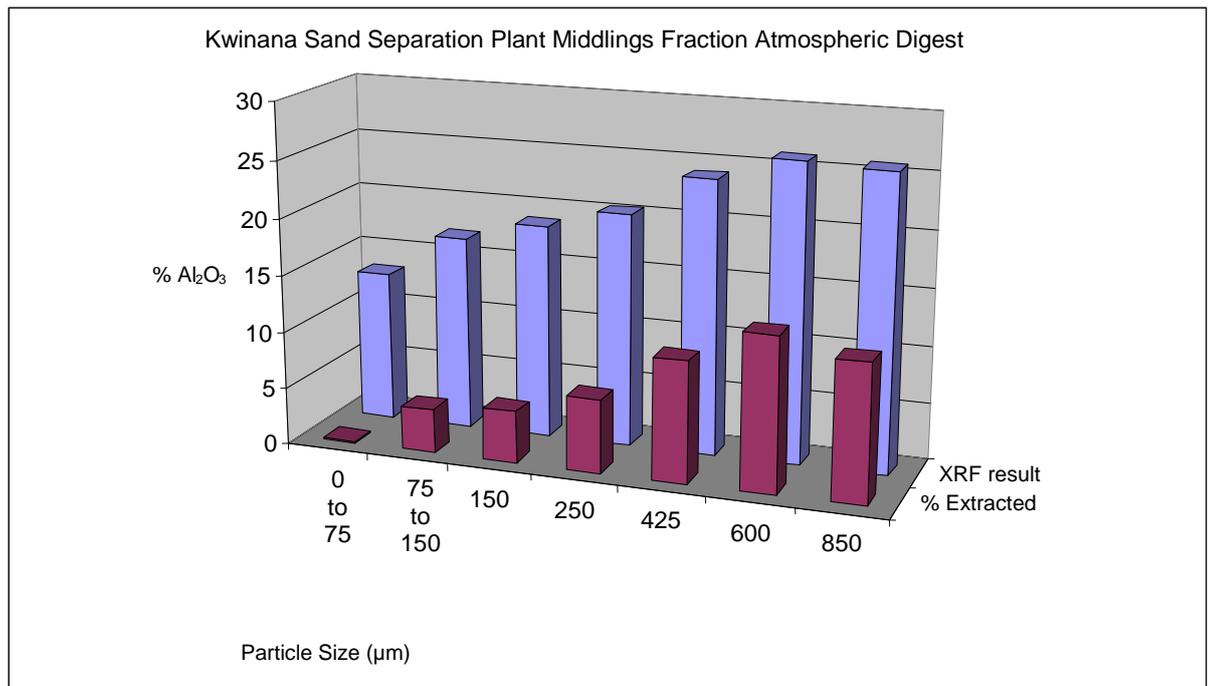


Figure 4.9 Kwinana Sand Separation Plant Atmospheric Digest of Middlings Fraction

The atmospheric digest results of the middlings fraction of the Kwinana sand separation plant show the same trend as the XRF Al_2O_3 results with the coarser size fractions having the highest Al_2O_3 recovery. The atmospheric digest results show lower alumina results than the XRF results. This is due to some of the Al_2O_3 in the middlings fraction being present as boehmite.

Boehmite however, is unlikely to account for all of the discrepancy between the atmospheric digest results and the XRF total Al_2O_3 results as the level of boehmite in Western Australian bauxite is low and most reports to the mud fraction after digestion. This indicates that the Al_2O_3 is present as another mineral phase such as aluminous goethite or aluminous haematite that does not digest at the low

temperatures of an atmospheric digest, and is not easily detectable on an XRD scan and thus is not extractable at the low temperature of an atmospheric digest.

The iron oxide level is similar for all of the size fractions, with the -75 μm fraction having the lowest concentration. The XRD results show haematite and goethite present as major phases of iron oxide in all of the size fractions. The XRD scan only identifies the major, minor and trace mineral phases present and cannot determine the proportion of different phases (such as haematite and goethite) present. This requires analysing under a different program and requires a longer scan time.

Magnetic fraction

In the magnetic fraction, the Al_2O_3 content decreases with decreasing particle size. Most of the Al_2O_3 in the magnetic fraction is likely to be present as aluminous goethite and aluminous haematite, with some gibbsite and boehmite present. XRD analysis shows gibbsite is present as a trace phase in the magnetic fraction in all size ranges greater than 250 μm .

The iron oxide levels are similar for all fractions with the range being 52 to 58%. This is higher than both the middlings and non-magnetic fractions. The XRD results show that haematite is present as a significant phase in all of the size fractions, and that magnetite is also present as a significant phase in all of the size fractions except for the 425 to 600 μm . Goethite is also listed as a trace phase in the magnetic fraction. This may be an under-estimation of the amount of goethite present due to the way that the XRD analyses fine or poorly crystalline phases such as iron oxides. The iron oxide XRD results are in general agreement with the Fe_2O_3 XRF results.

The general trend for SiO_2 is that the coarser the size fraction the lower the SiO_2 content. XRD analysis shows quartz present as a major phase in all of the size fractions except for the 425 to 600 μm size fraction. This does not agree with the XRF results. The disagreement is because the XRD analysis often over-estimates phases that are crystalline (e.g. quartz, gibbsite), crystallographically highly symmetric or having high diffracting structure factors (Pearson 2008).

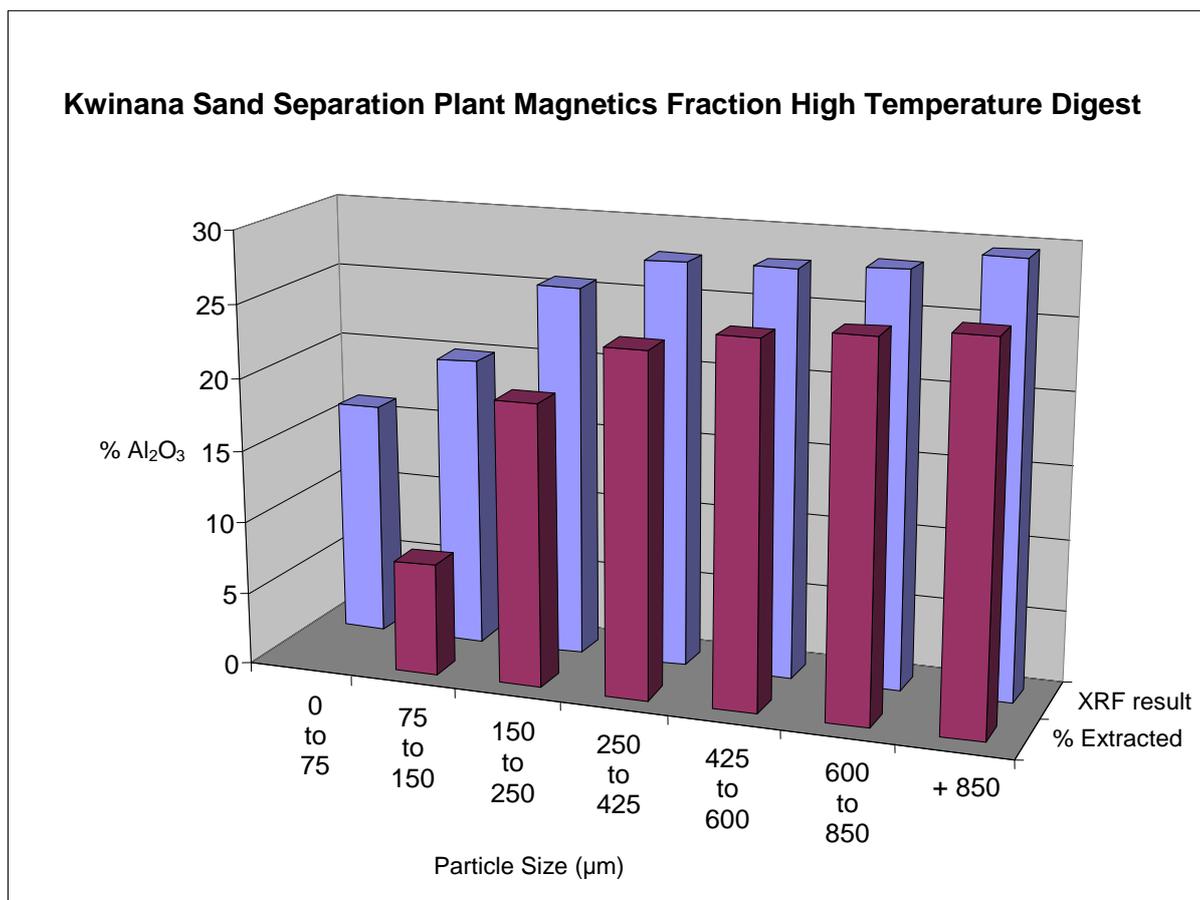


Figure 4.10 Kwinana Sand Separation Plant High Temperature Digest of Magnetics Fraction Al₂O₃ Results

High temperature digestions were also performed with the aim of recovering Al₂O₃ and also increasing the level of Fe₂O₃ in the digest residue.

High temperature digestion results showed that the coarser size fractions have higher Al₂O₃ recovery than the finer size fractions. The extraction from the finer fractions is considerably less than in the coarser fractions (Figure 4.10). This is possibly due the fine quartz being highly soluble in caustic at higher temperature and reacting to form DSP.

In a high temperature digest (250°C) the SiO₂ (present as quartz) in the samples will dissolve and result in DSP precipitation, which not only consumes caustic but will also remove much of the alumina recovered from the aluminous goethite.

Another possibility is that more of the Al₂O₃ in the finer fractions is present as

aluminous haematite and is more tightly bound into the crystal lattice than aluminous goethite. In a high temperature (250°C) digest the goethite is dehydroxylated and recrystallises as haematite and the alumina is taken into solution.

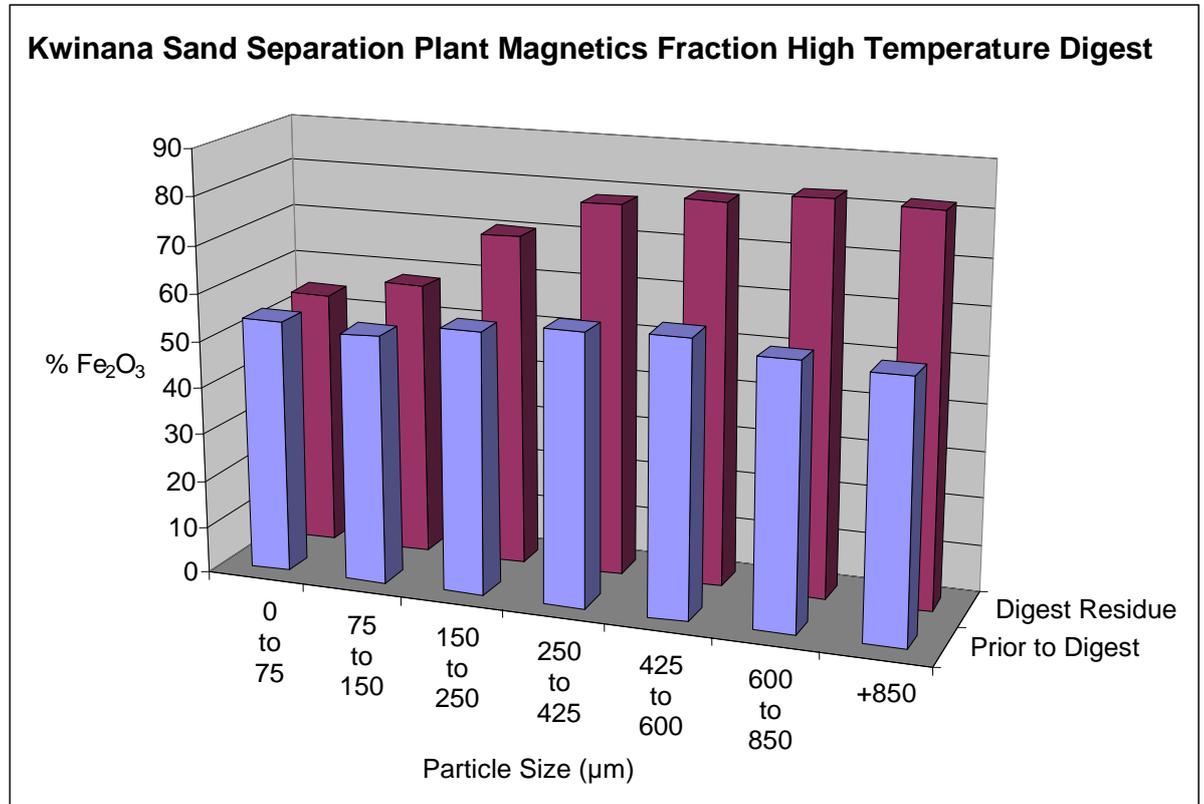


Figure 4.11 High Temperature Digest of Kwinana Sand Separation Plant Magnetics Fraction Fe₂O₃ Results

The high temperature digest residues for the coarsest fractions showed the largest increase in Fe₂O₃ content. This corresponds with these fractions having the largest decrease in Al₂O₃ content. The increase in Fe₂O₃ content is a concentration effect due to the digestion of the Al₂O₃.

The residues from the high temperature digestion (in particular the coarse fractions) may be suitable as a low grade iron ore.

4.4.3 Pinjarra Sand Rake

Non-Magnetics Fraction

The XRF results show that the Al_2O_3 content decreases with decreasing particle size, down to 150 μm . The Al_2O_3 content of the 75 to 150 μm fraction is slightly higher than the 150 to 250 μm fraction which may be an indication of the presence of DSP in the finest fraction. The XRD results show gibbsite present as a minor phase in the size fractions greater than 425 μm and as a trace phase in the other size fractions. The XRD results for the coarser size fraction do not agree with the XRF Al_2O_3 results, due to the way the XRD analysis is done.

The Fe_2O_3 content follows the same trend as Al_2O_3 - decreasing with decreasing particle size, with the 75 to 150 μm fraction being the exception. The increase in iron oxide in the 75 to 150 μm fraction is likely due to the presence of DSP. The XRD results show haematite and goethite are listed as trace phases in the non-magnetics fraction with the exception of the fractions in the size range of 250 to 600 μm . This is in general agreement with the XRF results. It would have been expected from the XRF results that the XRD results would show haematite and or goethite would be present as minor rather than trace phases. This is due to the XRD id scan underestimating iron oxide phases in bauxite residues.

The trend for SiO_2 is that the level increases with decreasing particle size. The size fractions below 425 μm have SiO_2 levels of 90% or higher. The XRD analysis results show quartz present as a major mineral phase for all size fractions. The XRD results are in agreement with the XRF silica results.

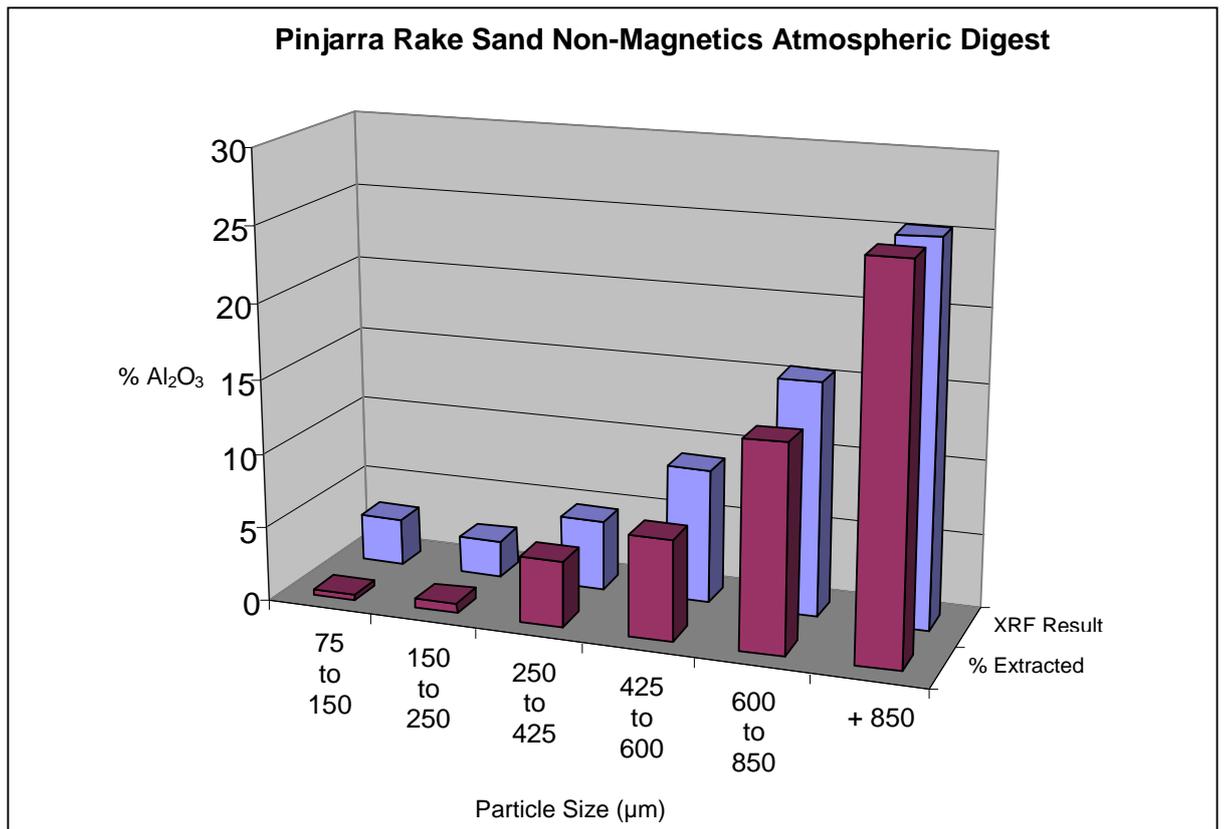


Figure 4.12 Pinjarra Rake Sand Atmospheric Digest of Non-Magnetics Fraction

The atmospheric digest results for the non-magnetics fraction follow the same trend as the XRF Al₂O₃ results, with the Al₂O₃ recovery increasing with increasing particle size.

The results of the atmospheric digest are in fairly good agreement with the XRF results down to 250 µm. The recovery in the fractions below 250 µm is low, and the Al₂O₃ level in these fractions is likely to be at the lower end of the detection capability of the Alian method used for the analysis of the atmospheric digest liquors.

Middlings Fraction

The Al₂O₃ content decreases with decreasing particle size, down to 75 µm where the level increased. The higher Al₂O₃ level in the – 75 µm fraction may indicate the presence of DSP. The XRD analysis results show gibbsite present as a minor phase in all fractions above 425 µm and as a trace phase in all size fractions. The XRD results are in agreement with the XRF Al₂O₃ results.

With the exception of the 600 to 850 μm and -75 μm fractions the Fe_2O_3 level increases with decreasing particle size. The XRD analysis shows goethite and haematite present as major phases in all size fractions. The XRD results are in agreement with the iron oxide XRF results.

The SiO_2 level increased with decreasing particle size down to 75 μm where the SiO_2 level increased. The SiO_2 level in this fraction is similar to the level in the 425 to 600 μm fraction. XRD shows quartz as a major phase in all of the fractions below 250 μm and as a minor phase in the fractions coarser than 250 μm . The XRD results are in general agreement with the XRF SiO_2 results.

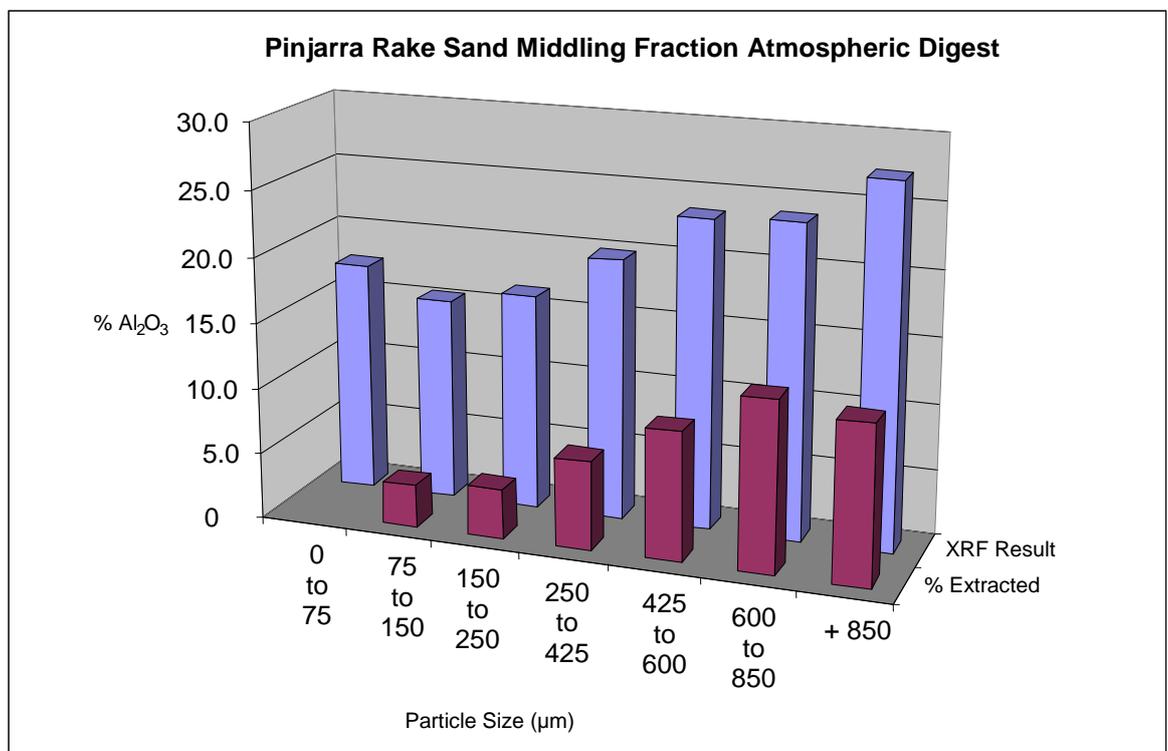


Figure 4.13 Pinjarra Rake Sand Atmospheric Digest of Middlings Fraction

The atmospheric digest results for the middlings fraction of Pinjarra rake sand show that the coarser fractions have higher levels of extractable Al_2O_3 than the finer fractions. There was insufficient sample for an atmospheric digest to be done on the -75 μm fraction. Comparison of the atmospheric digest results with the XRF total Al_2O_3 results show poor recovery (recoveries range from 20 to 54%).

Some of the Al_2O_3 in the middlings fraction is undigested gibbsite, whilst some of the Al_2O_3 is present as a mineral phase that does not digest at atmospheric digest

temperatures and does not show up on an XRD id scan, such as aluminous goethite and aluminous haematite. The trend for the atmospheric digest results agrees with the trend for the XRF Al₂O₃ results.

Magnetics Fraction

The XRF results show that the Al₂O₃ content is similar in all of the size fractions with the exception of the – 75 µm fraction which is lower. The XRD results show gibbsite present as a trace phase. This does not agree with the iron oxide XRF results. The difference between the XRD and XRF Al₂O₃ results is probably due to the fact that most of the Al₂O₃ is likely to be present as aluminous goethite and aluminous haematite.

The Fe₂O₃ content is similar (~ 60%) with the 150 to 250 µm size fraction having the lowest levels. There does appear to be a slight trend of the Fe₂O₃ content decreasing as the particle size decreases and then increasing again below 150 µm. XRD analysis shows haematite as the major mineral phase, maghemite as a minor phase and goethite as a trace mineral phase. The XRD results are in general agreement with the Fe₂O₃ XRF results.

The SiO₂ level increases with decreasing particle size, with the fractions below 150 µm being the exception. This may be the result of fine quartz particles being entrained in some of the larger iron oxide particles during the magnetic separation process. XRD results list quartz as a trace mineral phase; which does not agree with the XRF SiO₂ results ranging from 4 to 11%.

The high temperature digestion results show that the coarsest fraction and the finest fractions have the highest extractable alumina content. The amount of alumina extracted during the high temperature digest (measured by analysing liquors on the Alian) for the +850 µm is higher than the total Al₂O₃ by XRF. XRF analysis of the residues from the high temperature digest showed, however, that not all of the Al₂O₃ was extracted during the digest.

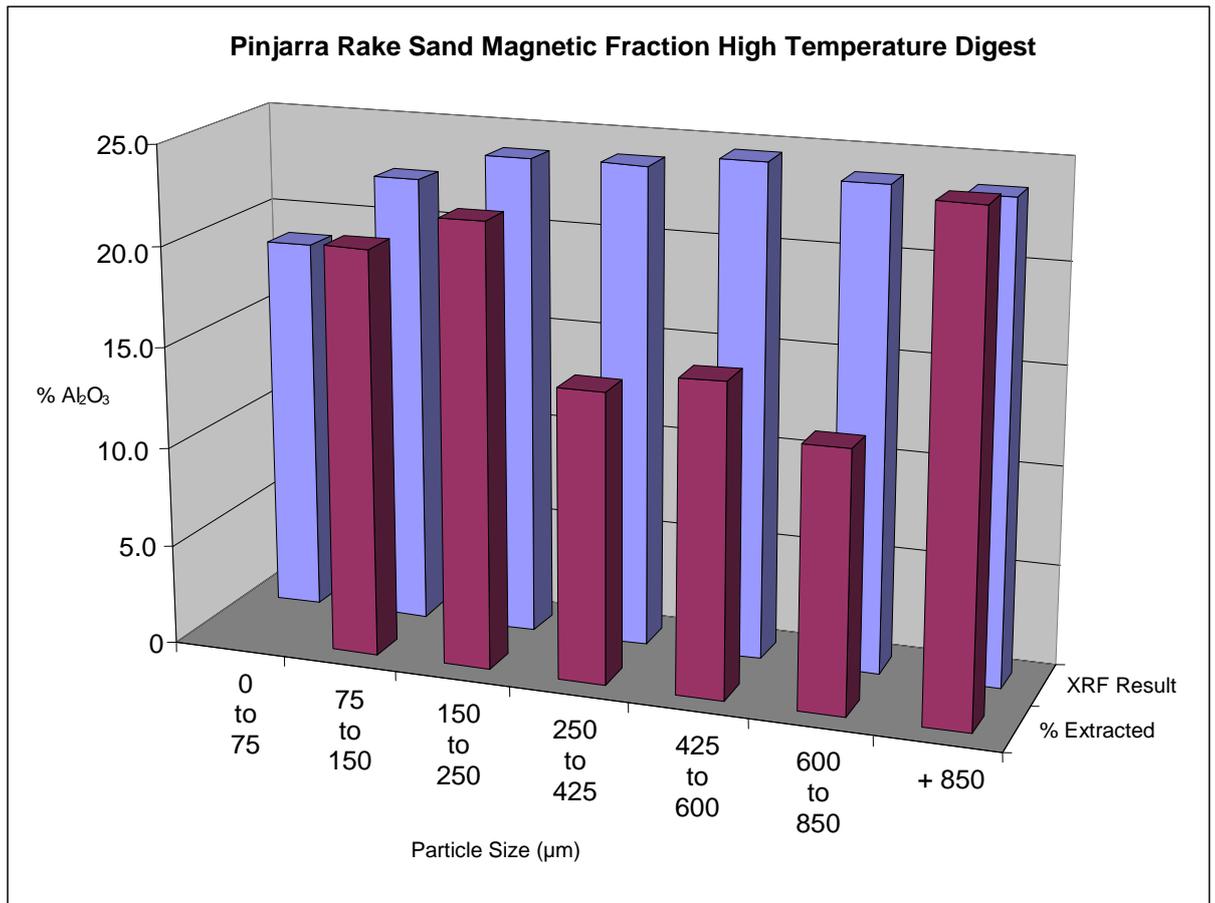


Figure 4.14 Rake Sand High Temperature Digest of Pinjarra Magnetics Fraction

XRF analysis of the residues from the high temperature digest of the magnetics fraction shows an increase in the iron oxide content across all size ranges. The largest increase in iron oxide level was in the coarsest fractions. This is a result of the decrease in Al₂O₃ (coarsest fractions had the largest decrease in Al₂O₃) level. There was insufficient sample to do a high temperature digest on the -75 µm fraction.

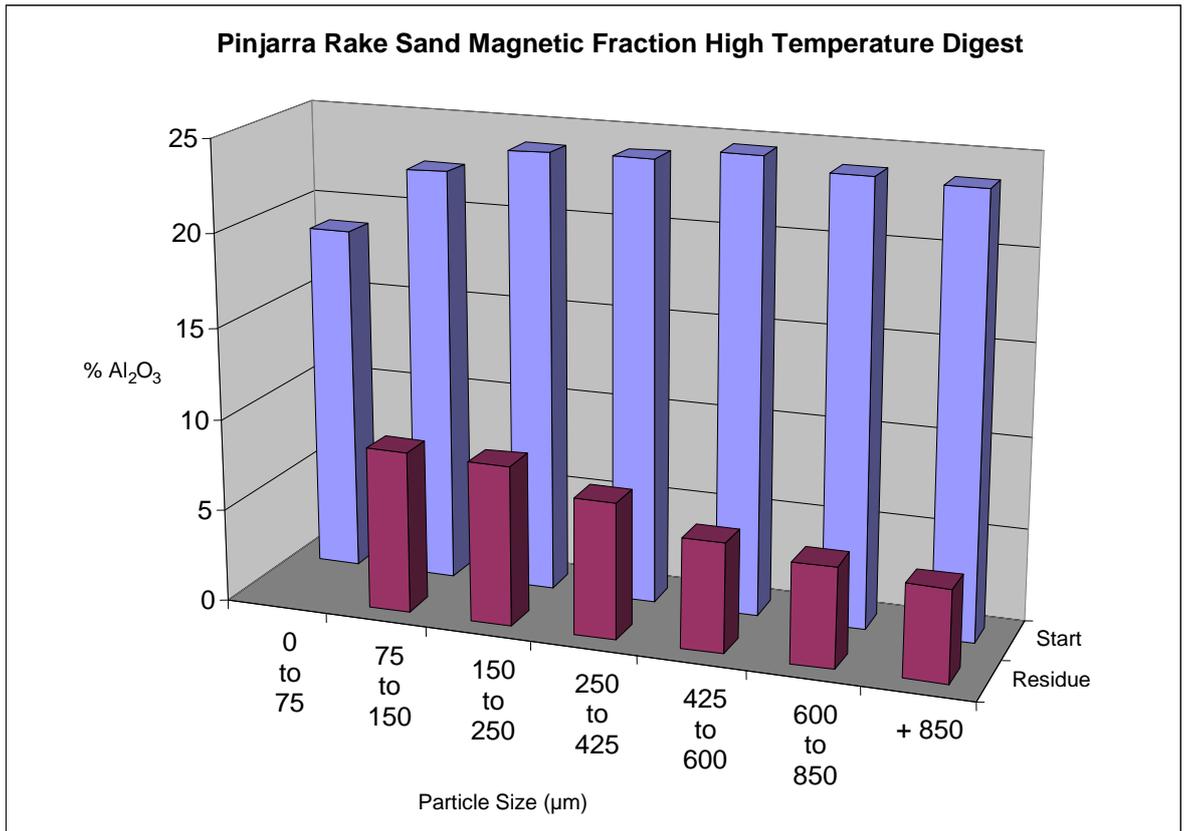


Figure 4.15 Pinjarra Rake Sand Magnetics Fraction High Temperature Digest Residues XRF Al₂O₃ Results

The Al₂O₃ in the digest residues is likely to be present as aluminous haematite. The Al₂O₃ in the digest residues is less than 10 % for all size fractions.

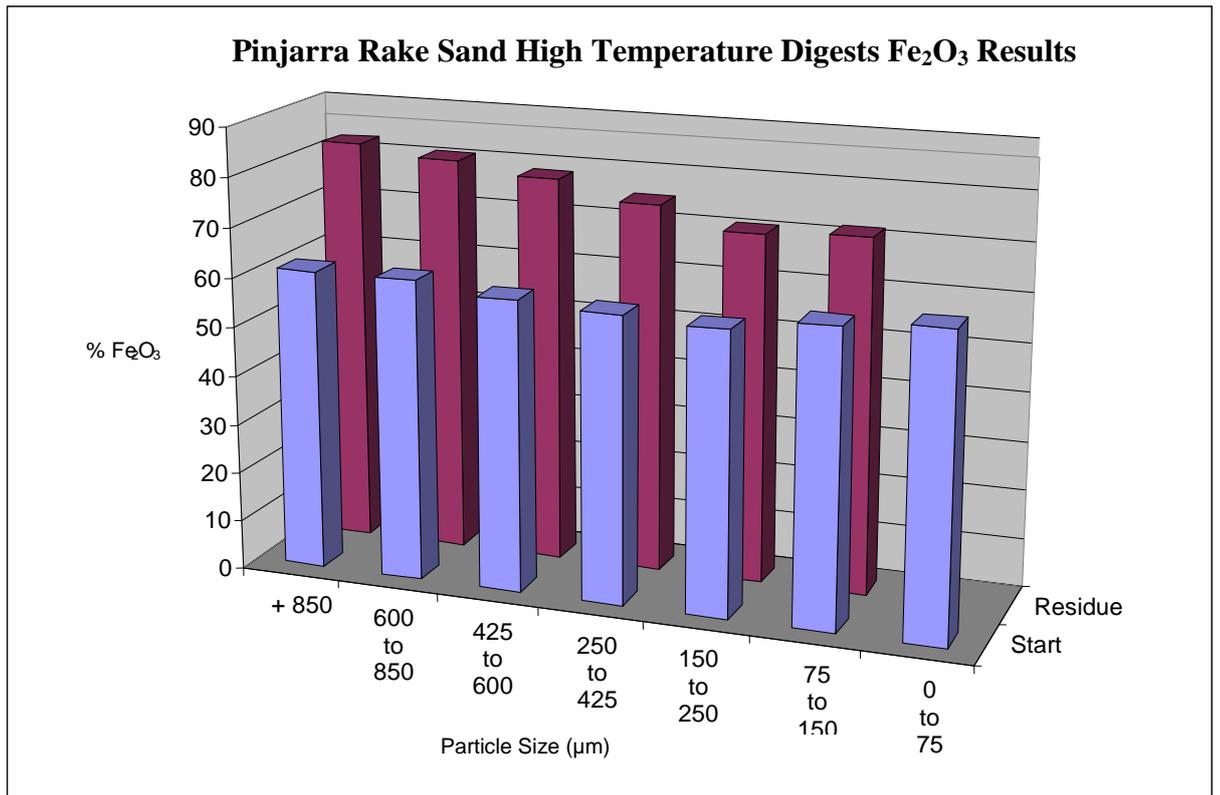


Figure 4.16 Pinjarra Rake Sand Magnetics Fraction High Temperature Digest Residues XRF Fe₂O₃ Results

The XRF results for the high temperature digest residues show the Fe₂O₃ content is 70% or higher for all of the size fractions. Hence the residues from high temperature digestion of the magnetics fraction may be suitable as a low grade iron ore.

The Na₂O level in the high temperature digest residues is at least twice the level in the start samples. The trend in the residues is the opposite to that of the start samples. In the start samples the Na₂O level is highest in the coarsest fractions indicative of entrained soda. The soda level in the residues is highest in the finest fractions; this indicates the formation of DSP due the dissolution of the silica in the start samples under the conditions of the high temperature digestions.

4.4.4 Pinjarra Sand Separation Plant

Non-Magnetics Fraction

The XRF results shows that as the particle size decreases the Al_2O_3 content decreases, with the exception being the finest fraction ($-75\ \mu\text{m}$). The increase in Al_2O_3 content in the $-75\ \mu\text{m}$ fraction is an indicator of the presence of DSP. XRD analysis shows gibbsite present as a minor phase in the fractions larger than $425\ \mu\text{m}$ and as a trace phase in the fractions below this size.

The gibbsite is likely to be undigested gibbsite, the amount of which is a function of the extraction efficiency of the individual digester units. The XRD results are in general agreement with the XRF Al_2O_3 results with the exception of the size fractions above $600\ \mu\text{m}$.

The silica content increased as the particle size decreased with the exception of the $-75\ \mu\text{m}$ fraction. The SiO_2 content of the fractions below $600\ \mu\text{m}$ was 90% or greater with the exception being the $-75\ \mu\text{m}$ fraction. XRD analysis showed quartz present as a major phase. This is in agreement with the XRF results for SiO_2 .

The iron oxide content decreases with decreasing particle size, with the exception of the $-75\ \mu\text{m}$ fraction. The increase in the $-75\ \mu\text{m}$ fraction indicates the possible presence of DSP.

XRD analysis showed that the iron oxide was present as goethite and haematite as minor phases in the $+850\ \mu\text{m}$ fraction, with goethite as a trace phase down to $425\ \mu\text{m}$, and haematite in the $-75\ \mu\text{m}$ fraction.

The iron oxide levels are less than 6% for all of the size fractions. This shows magnetic separation can successfully produce a non-magnetic fraction low in iron oxides and high in silica, which may be suitable as a building material.

The atmospheric digest results showed the amount of Al_2O_3 extracted decreased with decreasing particle size, with the exception being the 75 to $150\ \mu\text{m}$ fraction (this is possibly due to DSP being present). There was insufficient sample to do an atmospheric digest on the $-75\ \mu\text{m}$ size fraction.

The Al_2O_3 titre of the Alian analysis on the liquor for the size fractions 150 to 425 μm was small which is likely to be the cause of the low recovery for these fractions. The Al_2O_3 recovery compared to the XRF total Al_2O_3 results decreases with decreasing particle size.

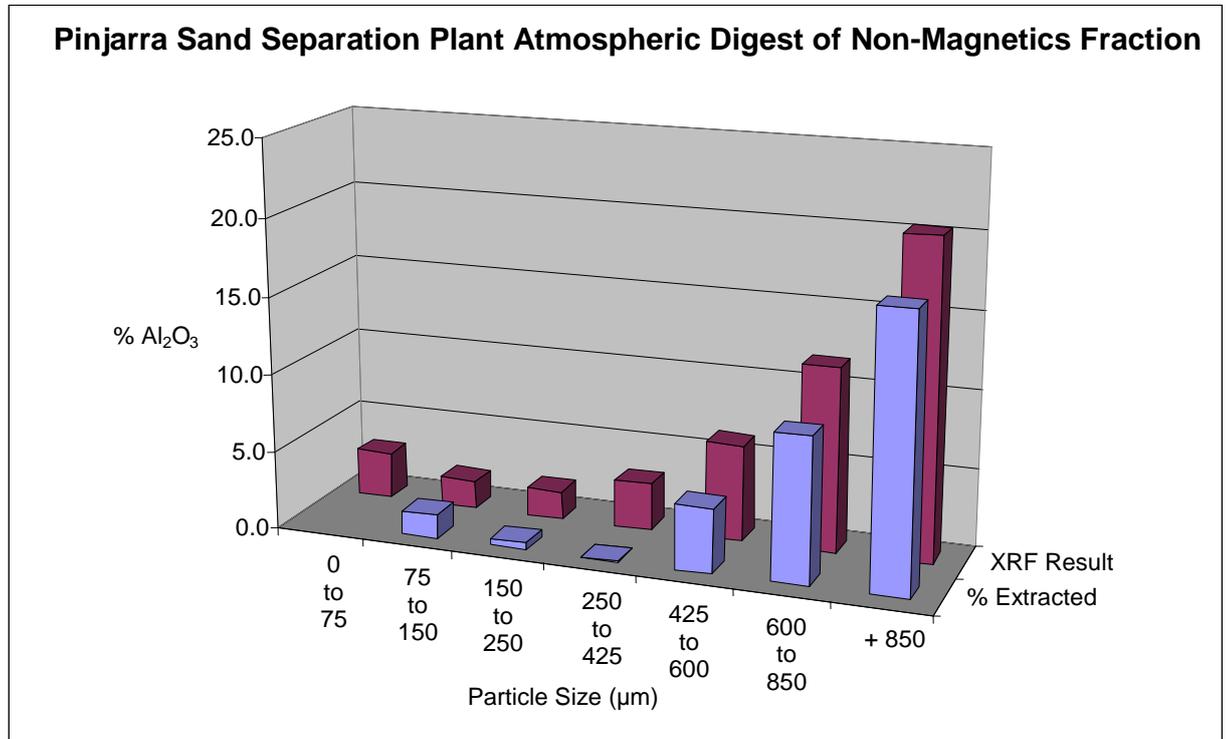


Figure 4.17 Pinjarra Sand Separation Plant Atmospheric Digest of Non-Magnetics Fraction

The atmospheric digest results are lower than the XRF results. This may be due to the atmospheric digest temperature and also the difference in the methods of analysis used (XRF vs. titration of digest liquors).

Middlings Fraction

The Al_2O_3 content decreases with decreasing particle size. The Al_2O_3 content ranged from 11% in the -75 μm fraction to 26% in the +850 μm fraction.

XRD analysis shows gibbsite as a major phase in the + 850 μm , a minor phase in the size range 425 to 850 μm and as a trace phase in the fractions below 425 μm .

The XRF results suggest that gibbsite should be present as a major phase. The XRD results do not agree with the XRF results suggesting that some of the Al_2O_3 content is present as other phases (boehmite is listed as a trace phase in the 600 to 850 μm fraction). This, however, is insufficient to account for the discrepancy between XRD and XRF results, as the boehmite content of WA bauxite is low and most will report to the mud fraction after digestion.

The SiO_2 content increases with decreasing particle size, the presence of SiO_2 in the middlings fraction is likely to be due to the entrainment of quartz particles. The results of the XRD analysis listed quartz as a minor phase about 250 μm and as a major phase in the fractions below 250 μm .

The Fe_2O_3 content increases with decreasing particle down to 250 μm and then decreases below this size. The iron oxide content is highest in the 250 to 425 μm size fraction. Haematite and goethite are listed as major phases in the size fractions above 150 μm and as minor phases in the size fractions below this size.

The Na_2O content increased with increasing particle size. This is possibly the result of entrained soda in the pores and crevices of the coarser particles, as the sand was dried and not washed prior to the magnetic separation.

The atmospheric digestion results follow the same trend as the XRF Al_2O_3 results with the coarsest size fractions having the highest extractable Al_2O_3 . Figure 4.18 shows that the amount of Al_2O_3 extracted in the atmospheric digest was considerably less than the XRF total Al_2O_3 . This suggests that not all of the Al_2O_3 is present as gibbsite. XRD analysis did not show the presence of any boehmite (except as a trace phase in the 600 to 850 μm fraction) – this indicates that it is more likely to be

present as aluminous goethite and aluminous haematite which does not show up using an id scan on the XRD.

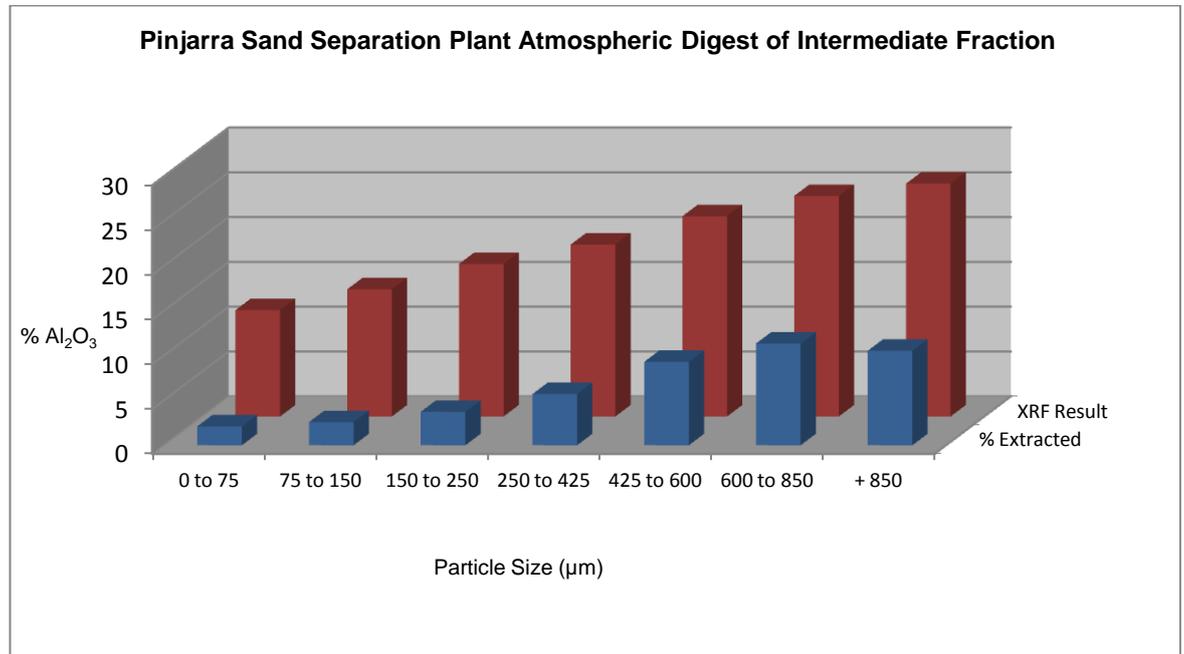


Figure 4.18 Pinjarra Sand Separation Plant Atmospheric Digest of Middlings Fraction

Magnetics Fraction:

The trend for Al₂O₃ is that the level decreases with decreasing particle size and ranges from 12% in the -75 µm fraction to 26% in the 850 µm fraction. XRD results show gibbsite present as a major phase in the + 850 µm fraction and as a trace phase in the size fractions ranging from 150 to 850 µm. Boehmite is also listed as a trace phase below 150 µm. The XRF Al₂O₃ results would suggest that gibbsite and or boehmite should be present as a major phase in all size fractions rather than just the + 850 µm fraction. The difference between the XRD and XRF results indicated that Al₂O₃ is likely to be present in a phase that does not show up on the XRD id scan, such as aluminous goethite and aluminous haematite.

The Fe₂O₃ content decreases with decreasing particle size, the exception being the - 75 µm fraction which has a higher Fe₂O₃ content than the 75 to 150 µm fraction. XRD analysis shows haematite and magnetite as major iron oxide mineral phases above 150 µm, and haematite below 150 µm. Goethite is listed as a minor phase in

all size fractions, with magnetite as a minor mineral phase below 150 μm . The XRD results were in general agreement with the total XRF Fe_2O_3 results.

The SiO_2 level increased with decreasing particle size, with the -75 μm fraction being the exception. This may be the result of fine quartz particles being entrained in some of the larger iron oxide particles during the magnetic separation process. XRD results list quartz as a major mineral phase in all size fractions with the exception of the 600 to 850 μm fraction which has quartz as a minor mineral phase. This is in general agreement with the XRF SiO_2 results ranging from 6% in the + 850 μm fraction to 47% in the 75 to 150 μm fraction. Due to insufficient sample in most of the size fractions, high temperature digest was not performed on the various size fractions of the magnetic fractions.

4.4.5 Wagerup Sand Rake

Non-Magnetics Fraction:

XRF results show that as the particle size decreases the Al_2O_3 content decreases, with the exception being the fractions below 150 μm , where the Al_2O_3 content increases again. The increase in Al_2O_3 content in the fractions below 150 μm is likely to be the result of the breakdown of larger alumina particles or conglomerate alumina/gangue particles [Dye Sept 2005] or that DSP may be present. The Al_2O_3 content ranges from 1.5% in the 150 to 250 μm to 15% in the +850 μm fraction.

XRD analysis shows gibbsite present as a minor phase in the fractions below 600 μm , with the exception of the 75 to 150 μm fraction which shows no gibbsite to be present. XRD analysis shows gibbsite to be present as a major mineral phase in the fractions above 600 μm . The gibbsite is likely to be undigested gibbsite which is a function of the extraction efficiency of the individual digester units. Coarse particles dramatically short circuit relative to liquor residence time in vertical digesters which are used in the WA refineries (Dye and Collett Aug 2005).

The XRD results are in agreement with the XRF results with the exception of the 75 to 150 μm size fractions.

The SiO_2 content increases as the particle size decreases, the exception to this is the -75 μm fraction, the SiO_2 content in this fraction is similar to that in the +850 μm fraction. The SiO_2 content of the fractions below 600 μm is 90% or greater with the exception being the -75 μm fraction. XRD analysis shows quartz present as a major phase which is in agreement with the XRF results for SiO_2 .

The iron oxide content decreased with decreasing particle size, with the exception being the fractions below 150 μm . The increase in the fractions below 150 μm indicated the possible presence of DSP.

XRD analysis did not show the presence of any iron oxide phases in any of the size fractions.

The iron oxide levels were less than 4% for all of the size fractions. This showed

magnetic separation could successfully produce a non-magnetic fraction low in iron oxides and high in silica.

The Na_2O content decreased with decreasing particle size with the exception of the size fractions below 150 μm . The higher Na_2O content in the coarser fractions was likely to be the result of entrained soda in the pores and crevices of the particles. The higher soda levels in the fractions below 150 μm could indicate the presence of DSP.

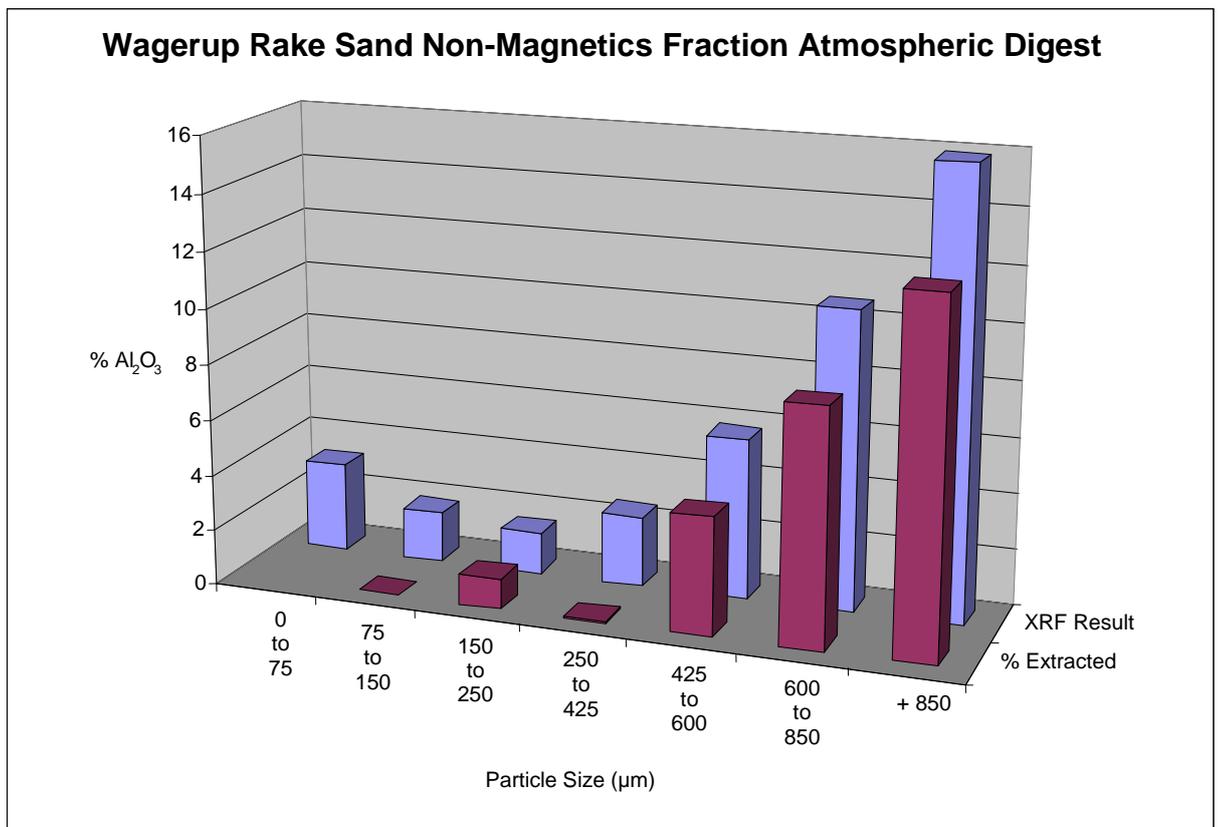


Figure 4.19 Wagerup Rake Sand Atmospheric Digest of Non-Magnetics Fraction

The atmospheric digest results (figure 4.19) showed the amount of Al_2O_3 extracted decreased with decreasing particle size, with the exception being the 75 to 150 μm fraction (this was possibly due to DSP being present). There was insufficient sample to do an atmospheric digest on the -75 μm size fraction.

The Al_2O_3 titre of the Alian analysis on the liquor for the size fractions 250 to 425

μm and the 75 to 150 μm fraction was small which is likely to be the cause of the low recovery for these fractions.

The Al_2O_3 recovery in the atmospheric digest is similar in the remaining fractions, ranging from 72 to 78% of the XRF total Al_2O_3 results.

Middlings Fraction:

The Al_2O_3 content decreases with decreasing particle size, with the exception of the -75 μm fraction. The Al_2O_3 content ranges from 14% in the 75 to 150 μm fraction to 24% in the +850 μm fraction.

XRD analysis showed gibbsite as a major phase in the size fractions above 425 μm , and as a trace phase in the size fractions below 425 μm .

The XRF results suggested that a phase of Al_2O_3 such as gibbsite would be expected to be present as a major phase in all of the size fractions. The XRD results do not agree with the XRF results suggesting that some of the Al_2O_3 content was present as a phase that does not show up on an XRD id scan.

The SiO_2 content increases with decreasing particle size. The presence of SiO_2 in the middlings fraction is likely to be due to the entrainment of quartz particles. The results of the XRD analysis lists quartz as a trace phase in all of the size fractions.

The Fe_2O_3 level increases with decreasing particle size, with the exception of the 600 to 850 μm fraction and the - 75 μm fraction. The Fe_2O_3 content ranges from 45% in the 600 to 850 μm fraction to 54% in the 75 to 150 μm fraction. The XRD analysis shows haematite and goethite as the only iron oxide phases present. The XRD and XRF Fe_2O_3 results are in general agreement.

The Na_2O content increases with increasing particle size, with the exception of the - 75 μm fraction. The higher Na_2O content in the coarser particles is likely to be the result of entrained soda in the pores and crevices of the coarser particles. The increase in Na_2O content in the - 75 μm fraction is an indication of the presence of DSP.

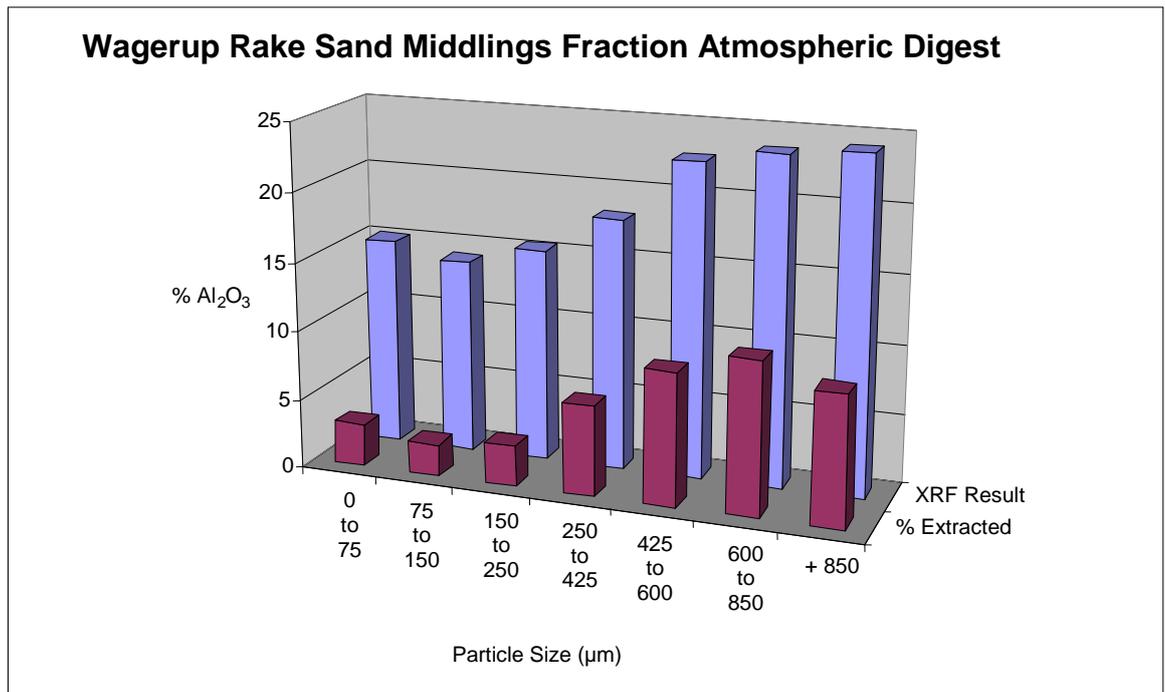


Figure 4.20 Wagerup Rake Sand Atmospheric Digest of Middlings Fraction

The atmospheric digest results follow the same trend as the XRF Al₂O₃ results with the coarsest size fractions having the highest extractable Al₂O₃. Figure 4.20 shows that the amount of Al₂O₃ extracted in the atmospheric digest is considerably less than the XRF total Al₂O₃; this suggests that not all of the Al₂O₃ is present as gibbsite. XRD analysis does not show the presence of any boehmite. This indicates that it is more likely to be present as aluminous goethite and aluminous haematite which does not show up using an id scan on the XRD.

Magnetics Fraction:

The trend for Al₂O₃ is that the level decreases with decreasing particle size and ranges from 16% in the -75 µm fraction to 29% in the 850 µm fraction. The XRD results do not show gibbsite or boehmite present in any of the size fractions.

The difference between the XRD and XRF results indicate that Al₂O₃ is likely to be present in a phase that does not show up on the XRD id scan, most likely as aluminous goethite and aluminous haematite.

The Fe_2O_3 content increases with decreasing particle size, the exception being the 250 to 425 μm fraction which has a lower Fe_2O_3 content than the 150 to 250 μm fraction.

XRD analysis shows haematite as a major mineral phase in all the size fractions. Goethite is listed as a minor phase in the +850 μm fraction and as a trace phase in the size fractions below 850 μm . Magnetite is listed a minor mineral phase above 425 μm and as a trace phase in the fractions below 425 μm . The XRD results are in general agreement with the total XRF Fe_2O_3 results.

The trend for SiO_2 is that the finer fractions have a higher SiO_2 content than the coarse fractions. The SiO_2 content in the magnetic fraction is likely to be the result of fine quartz particles being entrained with the iron oxide particles during the magnetic separation. XRD results list quartz as a minor mineral phase in all the size fractions. This is general agreement with the XRF SiO_2 results.

The results of the high temperature digestions follow the same trend as the XRF total Al_2O_3 results with the Al_2O_3 extraction increasing with increasing particle size. The Al_2O_3 recovery ranges from 73% in the 75 to 150 μm fraction to 88% in the 425 to 600 μm fraction.

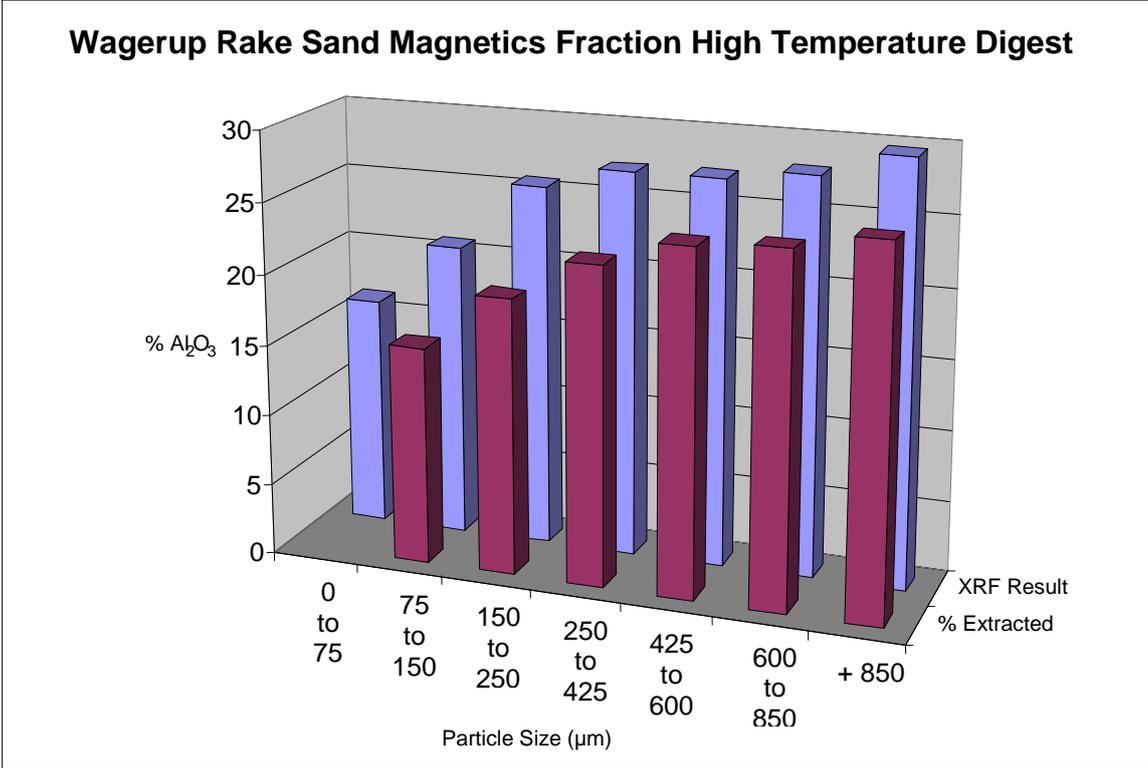


Figure 4.21 Wagerup Rake Sand High Temperature Digest of Magnetics Fraction Al₂O₃ Extraction

XRF analysis of the residues from the high temperature digest shows the Al₂O₃ content increases with decreasing particle size. The Al₂O₃ content of the digest residues is less than 8% in all of the size fractions. The Al₂O₃ recovery ranges from 65% to 82%.

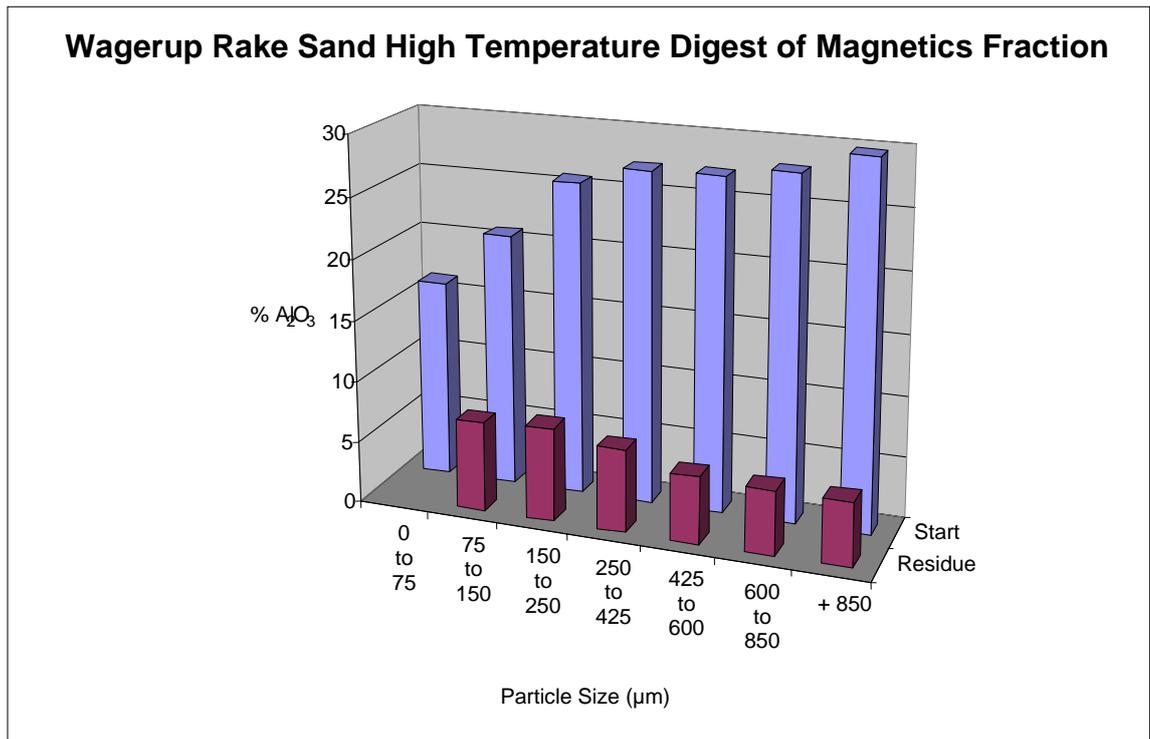


Figure 4.22 Wagerup Rake Sand Magnetics Fraction XRF Al₂O₃ Results of High Temperature Digest

The Fe₂O₃ content of the residues from the high temperature digest ranges from 73% in the 150 to 250 µm fraction to 86% in the + 850 µm fraction.

Figure 4.23 shows that the largest increase in the Fe₂O₃ level is in the coarsest fractions, this is most likely due the coarser fractions having a higher aluminous goethite content than the finer fractions. The finer fractions have higher aluminous haematite content than the coarser fractions.

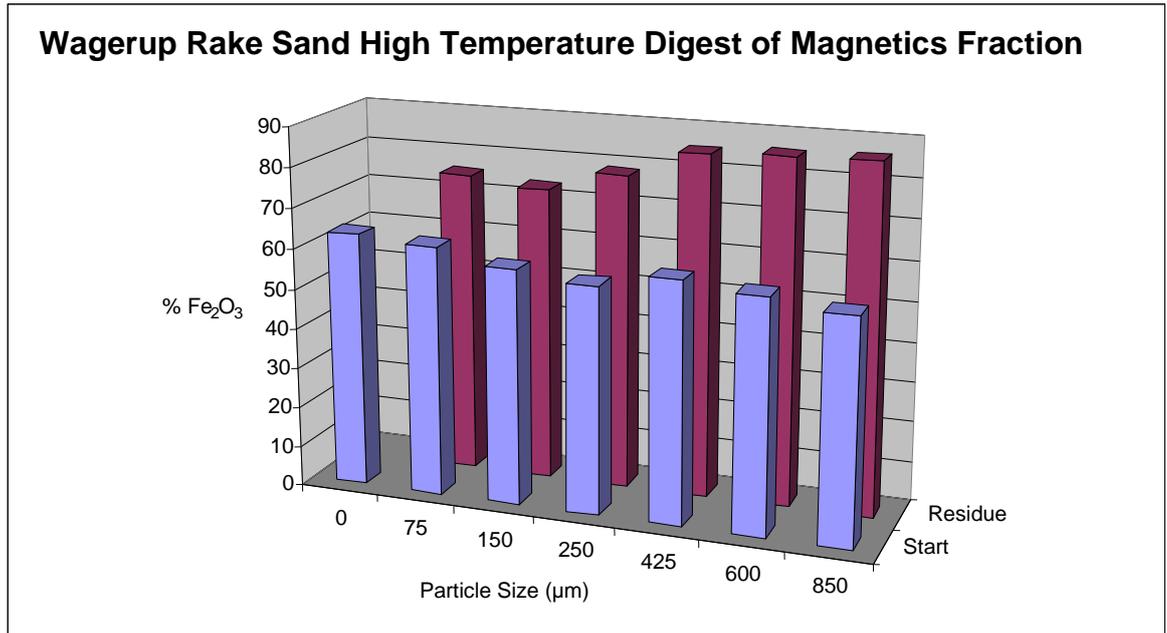


Figure 4.23 Wagerup Rake Sand Magnetics Fraction XRF Fe₂O₃ Results of High Temperature Digest

4.4.6 Conclusions

Magnetic separation can be used to produce a fraction high in silica (non-magnetic fraction) and low in iron oxides that may be able to be used as a building material, and a fraction high in iron oxides which may be able to be used as an iron ore substitute if the aluminium oxide level can be reduced.

The alumina content of the non-magnetic fraction and some of the middlings fraction can be recovered through an atmospheric digest. The remaining alumina in the middlings fraction and most of the alumina in the magnetic can be recovered through a high temperature digest. Recovery of the alumina may be able to offset some of the cost of the magnetic separation.

Atmospheric digest of residue sand prior to magnetic separation would allow the digesters to be run at higher blow off ratios which would result in a production increase for the refinery. This would result in the non-magnetic fraction having a lower Al₂O₃ content. This may make it more suitable for use as a building material.

Atmospheric digest results are in agreement with previous work by Dye and Collett (2002 & 2005), with coarser fractions having higher available alumina than the finer fractions.

Separation can be successfully performed on either rake sand or sand from the sand separation plant at the residue storage area. Other factors such as the cost of replacement due to abrasion of the sand line to the residue area and available space within the refinery will influence the optimum location of any magnetic separation plant.

4.5 High Temperature Alumina Extraction from Magnetically Separated Sand

The production of iron ore suitable for use in steel manufacture has been investigated using Alcoa Red Sand™ (Jamieson, E. (2006). Production of an Iron Ore. Alcoa Report AWA 7.05 CAB004). Tests to date have demonstrated that a high iron fraction which may be suitable as a low-grade iron ore can be separated from the red sand. However the aluminium content of the high iron sand makes it unsuitable for use in direct induction furnaces. The following is a summary of Jones, A. (2008). Alumina Recovery from High Iron Red Sand. Alcoa Report AWA 7.05 CAB010.

Most of the alumina present in the high iron samples is present as aluminous goethite. A high temperature digestion (235°C) should allow the goethite to dehydroxylated and recrystallise as haematite, expelling the alumina into solution. In a high temperature digest the SiO₂ (present as quartz) in the samples will dissolve and result in DeSilication Product (DSP, an alumino-silicate) precipitation, which not only consumes caustic but will also remove much of the alumina recovered from the aluminous goethite.

To determine if it would be feasible to reduce the aluminium content of the high iron fraction of Red Sand™ via high temperature digestion, available alumina and reactive silica analyses were carried out at 235°C. Available alumina and reactive

silica are two parameters which are often used to assess the viability of a bauxite deposit.

High temperature available alumina and reactive silica digests using 8% NaOH were carried out on the Magnetics fraction of Wagerup residue sand separated using a Wet High Intensity Magnetic Separator (WHIMS), and the upgraded Magnetics fraction. The Magnetics sample was reprocessed by passing over an induced roll dry magnetic separator to upgrade the sample by removal of silica.

RESULTS

Table 4.11 XRD Results for Digested and Undigested High Iron Red Sand™ samples

| Sample | Major | Minor | Trace |
|-----------------------------------|-----------|-----------------------------|-------------------------------------|
| Magnetics | Quartz | Haematite, goethite | Gibbsite |
| Magnetics digest residue | Haematite | Quartz, goethite, magnetite | DSP, sodium aluminosilicate hydrate |
| Upgraded Magnetics | Haematite | | Magnetite, quartz, goethite |
| Upgraded Magnetics digest residue | Haematite | | Magnetite, quartz, goethite, DSP |

XRD results for the digest residues show that some DSP was formed during the digests.

Table 4.12 High Temperature Available Alumina Results for High Iron Red Sand™

| Sample | % Available Alumina |
|--------------------|---------------------|
| Magnetics | 1.00 |
| Upgraded Magnetics | 19.40 |

The available alumina results for the high temperature digest of the high iron Red Sand are very low (1%) for the Magnetics fraction. The upgraded Magnetics fraction has a higher available alumina (19.40%); the difference in the available alumina results is due to the difference in the silica content of the samples (table 4.14 XRF results for high temperature digests of high iron Red Sand).

Table 4.13 High Temperature Reactive Silica Results for High Iron Red Sand™ samples

| Sample | % Reactive Silica |
|--------------------|-------------------|
| Magnetics | 16.81 |
| Upgraded Magnetics | 1.82 |

The results for the high temperature reactive silica analysis of the high iron Red Sand show that at the digest temperature the silica in the sample is soluble. The reactive silica in the Magnetics sample is significantly higher than that of the upgraded Magnetics.

Table 4.14 XRF Results for High Temperature Digests of High Iron Red Sand™

| SAMPLE NAME | Al ₂ O ₃ | Fe ₂ O ₃ | SiO ₂ | TiO ₂ | Na ₂ O | CaO | K ₂ O | P ₂ O ₅ | Ga ₂ O ₃ | Total |
|-----------------------------------|--------------------------------|--------------------------------|------------------|------------------|-------------------|--------|------------------|-------------------------------|--------------------------------|-------|
| | % | % | % | % | % | % | % | % | % | % |
| Magnetics | 15.79 | 45.39 | 31.90 | 2.846 | 0.12 | 0.1390 | 0.0326 | 0.038 | 0.0117 | 96.27 |
| Magnetics digest residue | 14.39 | 48.57 | 20.92 | 3.491 | 7.92 | 0.1464 | 0.0111 | 0.0371 | 0.0050 | 95.49 |
| Upgraded Magnetics | 23.74 | 67.41 | 2.82 | 3.780 | 0.17 | 0.1746 | 0.0395 | 0.0482 | 0.0187 | 98.20 |
| Upgraded Magnetics digest residue | 6.05 | 82.69 | 2.74 | 4.321 | 1.77 | 0.2136 | 0.0102 | 0.0413 | 0.0063 | 97.84 |

CONCLUSIONS

The iron oxide content of the Magnetics sample increases only slightly (45% to 48%) after digestion at 235°C. Combined with only a slight decrease in aluminium oxide level the digest residue would not be any more suitable as an iron ore substitute than the original Magnetics sample.

The reactive silica level of the Magnetics sample was approximately 17% with an available alumina content of 1%.

The reactive silica is an important measure in the Bayer process as the silica that is taken into solution is subsequently precipitated as sodium aluminium silicates which is a significant cost in the process due to the consumption of both caustic soda and alumina.

The results from the available alumina and reactive silica analyses clearly indicate that this sample is not suitable as a feed for a high temperature digestion process, due to the extremely high reactive silica level.

The XRD results show quartz present as the major mineral phase in the Magnetics fraction. The major mineral phase in the Upgraded Magnetics is haematite with quartz present as a minor mineral phase. The XRD results are consistent with the XRF silica results of 31.9% for the Magnetics sample and 2.8% for the upgraded Magnetics sample. The XRD and XRF results show that the Magnetics sample can be reprocessed through a magnetic separator to produce a sample that has a very low silica content.

The available alumina content of the Upgraded Magnetics was 19.40% with a reactive silica content of 1.8%. The difference in silica (quartz) content between the Magnetics and the Upgraded Magnetics is the reason for the difference in the reactive silica and available alumina results for the 2 samples.

The iron oxide content of the upgraded Magnetics sample increased from 67.4% to 82.7%, and the aluminium oxide content decreased from 23.7% to 6.0% in the digest residue. The increase in the iron oxide content and reduction in aluminium oxide content of the upgraded Magnetics sample after digestion at 235°C may make it more attractive for use as a low grade iron ore feed for pig iron production.

The available alumina content however is likely to be too low to make high temperature digestion of the high iron fraction of red sand economic for alumina recovery alone, and would need revenue raised from the sale of the digest residue as

an iron ore substitute to help offset the cost of doing the separation of the sand (and possibly reprocessing if the silica content is too high) as well as the cost of the high temperature digest.

4.6 Wet Magnetic Separation of Sand

A large scale sample (approximately 16 tons) of Wagerup residue sand was sent to CSIRO Minerals at Waterford for wet magnetic separation trials.

Operational conditions of the WHIMS (Wet High Intensity Magnetic Separator) unit were modified to provide the best separation of materials. The CSIRO / Alcoa report DMR 2717 (2005) provides operational details. Results were also published in Jamieson, E. et al (2006).

All of the residue sand was wet screened using a Kason vibrating screen to remove +1 mm fraction which would otherwise cause a problem with the narrow plate setting of the WHIMS. Approximately 15% of the sand was removed as + 1 mm, screening of the sand produced a large amount of fines.



Figure 4.24 Kason Vibrating Screen

The sand after screening was to be passed through a LIMS (model Eriez L8) to remove the highly magnetic fraction, prior to the sample being fed into the WHIMS.



Figure 4.25 Eriez Low Intensity Magnetic Separator (LIMS)

The WHIMS separator was fed from a single 200 litre drum with a speed-controlled mixer and impeller to suspend the solids. The suspended feed was pumped by a diaphragm pump ~5 metres up to a small header tank that then drained into the WHIMS inlet.

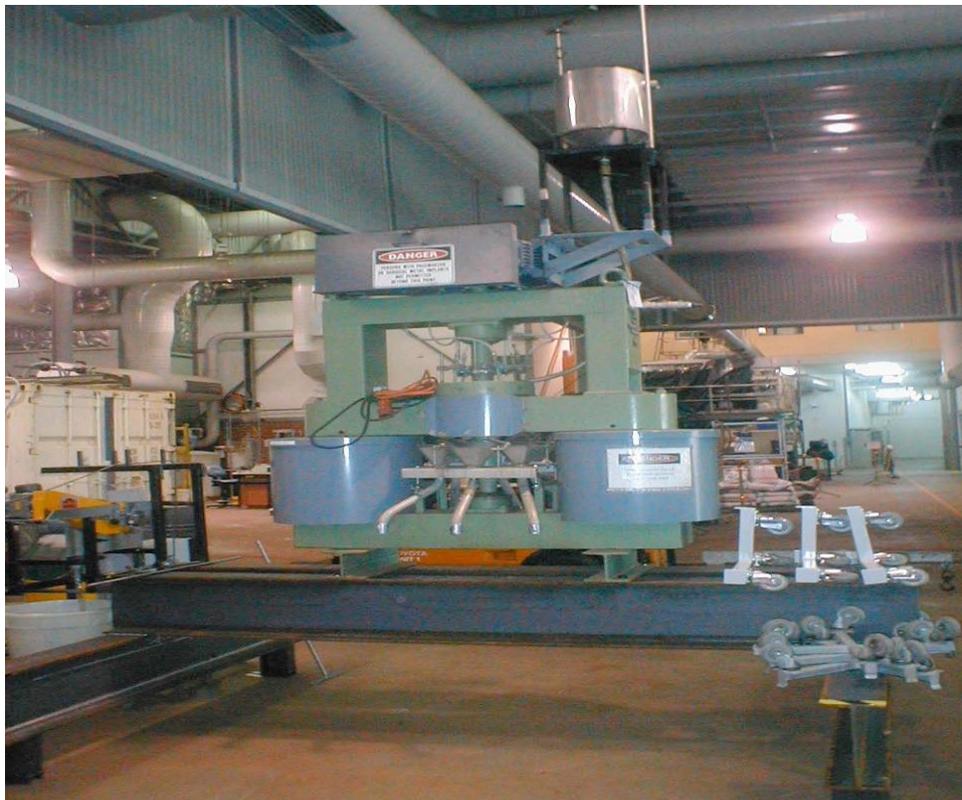


Figure 4.26 Humboldt Jones P40 Wet High Intensity Magnetic Separator

4.6.1 LIMS (Low Intensity Magnetic Separator)

Field strength of 1000 Gauss was applied to a 30% (v/v) sand: water slurry, fed at a rate of 2 L/min. This feed rate proved insufficient to be able to treat the sample size supplied and was hence the LIMS was removed from the process.



Figure 4.27 Drum of Eriez Low Intensity Magnetic Separator (LIMS)

XRF Analysis of the LIMS product shows a high iron content (see Table 4.15).

Table 4.15 XRF Analysis of LIMS Concentrate

| Sample Name | Fe ₂ O ₃ | Al ₂ O ₃ | TiO ₂ | SiO ₂ | CaO | Ga ₂ O ₃ | K ₂ O | Na ₂ O | P ₂ O ₅ | Total |
|-------------|--------------------------------|--------------------------------|------------------|------------------|--------|--------------------------------|------------------|-------------------|-------------------------------|-------|
| | % | % | % | % | % | % | % | % | % | % |
| LIMS | 68.95 | 26.87 | 1.914 | 1.81 | 0.1140 | 0.0212 | 0.0390 | 0.190 | 0.0265 | 99.93 |

4.6.2 WHIMS (Wet High Intensity Magnetic Separator)

With the removal of the LIMS pre-screening step, a certain level of blocking of the WHIMS was expected. Hence the process screens were cleaned regularly with high pressure water and occasionally had to be removed. This was time-consuming but was required to prevent blocking of the plates and to allow clean separation. The plates can be seen below (figure 4.28), as are the separated products of Non-Magnetics, Middlings and Magnetics.



Figure 4.28 Humboldt Jones P40 Wet High Intensity Magnetic Separator



Figure 4.29 Magnetic, Middlings, and Non-Magnetic Fractions from WHIMS

4.6.3 Microscopy of the WHIMS Middlings Fraction

Previous work (Roach 1992) suggests that approximately 50% of the iron oxide in residue sand is present as discrete particles, with the remaining 50% being conglomerate particles containing other mineral phases. Magnetic separation requires the presence of discrete mineral grains for good separation. The following chapter is

a summary of Jones, A. (2006). Microscopy Studies of WHIMS Separated Red Sand. Alcoa Report AWA 7.05 CAB012.

Magnetic separation of Red Sand TM produces 3 fractions, one high in iron oxides (magnetic fraction), and typically about 40% as Fe. The second fraction has high silica (non-magnetic fraction) content (ca 93% as SiO₂). The third fraction comprised of a mixture of iron and silica (middlings fraction). It was thought that this middlings fraction may possibly be comprised of conglomerate particles. The other possibility is that that this fraction is comprised of discrete particles resulting from inefficient separation.

Optical microscopy and backscattered FEGSEM analysis was conducted in order to determine if the middlings fraction is composed of discrete particles of iron oxide and silica (quartz) as a result of an inefficient separation process or is comprised of conglomerate particles which would require further leaching to liberate this fraction into discrete particles.

A sample of each of the 3 fractions (magnetic, middlings, and non-magnetic) resulting from the magnetic separation of Wagerup Red Sand using the WHIMS unit was examined via optical microscopy and also submitted for backscattered FEGSEM analysis.

Optical microscopy images (Figures 4.30 to 4.32) show mainly iron oxide particles with some entrained silica in the magnetic fraction, the middlings fraction shows discrete silica (quartz) particles and iron oxide particles as either possibly 2 different forms - possibly haematite and goethite (XRD analysis shows both haematite and goethite to be present) or as conglomerate particles along with discrete iron oxide particles. The non-magnetic fraction shows mainly silica (quartz) particles with some iron oxide particles.

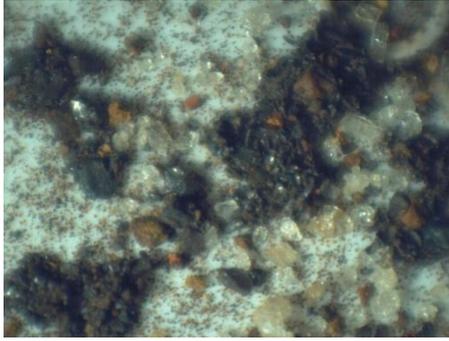


Fig 4.30 Magnetic fraction

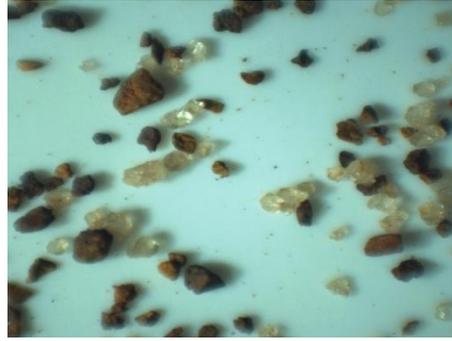


Fig 4.31 Middlings fraction

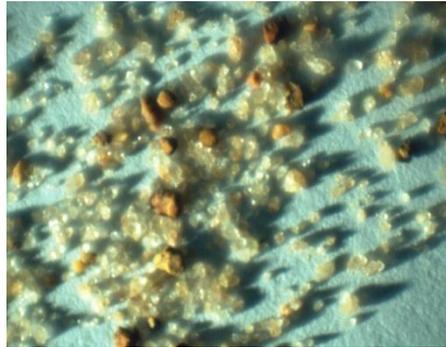


Fig 4.32 Non-Magnetic fraction

In backscattered SEM images higher (Figures 4.33 to 4.35 below) atomic weight components such as iron oxide show up lighter than lower atomic components such as silica (quartz) and gibbsite, thus making this technique ideally suited for the purpose of determining if the middlings fraction of Red Sand™ is composed of discrete particles or conglomerate particles.

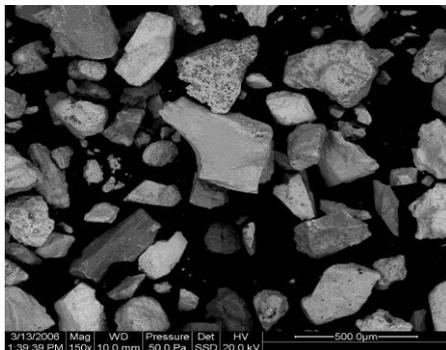


Fig 4.33 Magnetic fraction

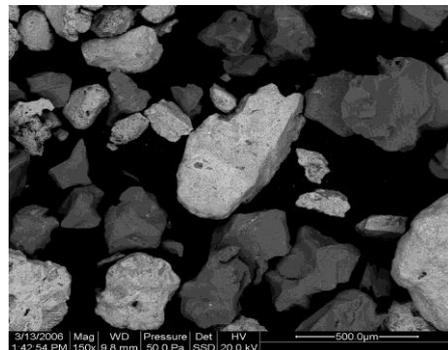


Fig 4.34 Middlings fraction

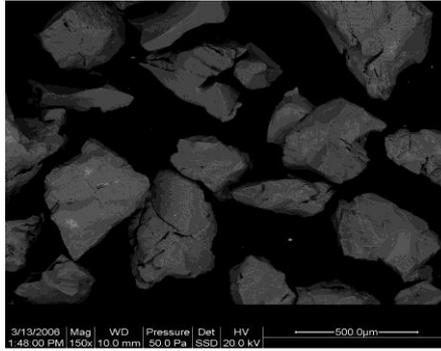


Fig 4.35 Non-Magnetic fraction

The brighter particles in the magnetic and middlings fractions are higher in Fe (iron oxides), while the darker particles are higher in Al and Si (i.e. gibbsite and quartz). The backscattered image for the non-magnetic fraction shows darker particles only indicating that it is comprised mainly of Al and Si (i.e. gibbsite and quartz).

The middlings sample did not show portioning of the particles between high and low atomic number components but it did show discrete particles of high and low atomic number components indicating that it is not made up of conglomerate particles.

The optical microscopy images and the backscattered FEGSEM images indicate that Red Sand™ is comprised mainly of discrete particles of iron oxide and silica (quartz) that should be able to be separated magnetically into two (magnetic and non-magnetic) fractions.

It is recommended that further trials be conducted upon the middlings fraction to confirm that it can be separated cleanly into two fractions of magnetic and non magnetic particles as indicated above.

4.5.4 Dry Magnetic Separation of the WHIMS Middlings Fraction

This section is a summary of Jones, A. (2008). Magnetic Reprocessing of WHIMS Middle Sand. Alcoa Report AWA 7.05 CAB003. Magnetic Separation of Red Sand™ produces three fractions - one high in iron oxides (magnetic fraction, typically about 40% as Fe); one fraction high in silica (non-magnetic fraction containing 93% as SiO₂); and the third fraction, comprised of a mixture of iron

oxides and silica (middlings fraction). Microscopy analysis suggests that the middle fraction should be able to be separated into high iron and high silica fractions.

The middlings fraction was reprocessed using dry drum and rare earth roll magnetic separators to determine if further separation of this fraction is possible. If further separation is possible it would demonstrate that the original separation process was inefficient and that a multi-stage separation process is required to successfully separate Red Sand™ by magnetic separation.

To test this process, the middle fraction was reprocessed using both a dry drum and a rare earth roll magnetic separator, in an attempt to further separate these discrete fractions.

Approximately 30 kg of the middlings fraction from the CSIRO LIMS/WHIMS trials was reprocessed using a dry drum magnetic separator and a rare earth roll magnetic separator at Outokumpu's (now Outotec) Physical Separation Technology Laboratory in Kewdale, Western Australia.

Dry Drum Separator

Table 4.16 Middlings Fraction Reprocessed using a Dry Drum Magnetic Separator

| Sample | Mass (kg) | Mass % of Middlings Fraction |
|---------------|-----------|------------------------------|
| “Magnetite” | 0.14 | 0.47 |
| Mags 3 | 0.14 | 0.47 |
| Mags 4 | 0.46 | 1.53 |
| Mags 5 | 11.53 | 38.42 |
| Non-Magnetics | 17.74 | 59.11 |
| Total | 30.01 | 100 |

Upon further reprocessing of the middlings fraction nearly 60% reported to the non-magnetics fraction, approximately 38½% to the Mags 5 fraction (this fraction had the lowest magnetic susceptibility after the non-magnetics fraction), with the remaining

2.5% being split between the other three fractions.

The sample labelled as “Magnetite” was the portion of the sample that was sticking to the drum of the magnetic separator (see figure 1) and was thought possibly to be magnetite. The samples labelled as Mags 3 to Mags 5 refers to the segment in which the fraction was collected and how close the segment is to the magnetic drum, with Mags 1 being the closest segment to the drum and then Mags 2 etc. No sample was collected in either the Mags 1 or Mags 2 segments.



Figure 4.36 Reprocessing of Middlings sand using a Dry Drum Magnetic Separator

Table 4.17 XRD Analysis Results for Middlings fraction reprocessed using a Dry Drum Magnetic Separator

| Sample | Significant | Trace |
|---------------|---------------------------------------|----------------------------------|
| “Magnetite” | magnetite, haematite | quartz, anatase, goethite |
| Mags 3 | goethite, ilmenite, haematite | gibbsite, anatase, quartz |
| Mags 4 | goethite, ilmenite, haematite, quartz | gibbsite, anatase |
| Mags 5 | goethite, haematite, quartz | gibbsite, anatase |
| Non-Magnetics | Quartz | gibbsite, haematite, goethite |

The separation of the middlings fraction using the dry drum magnetic separator shows that the middlings fraction was composed mainly of quartz, with goethite and haematite being the next largest mineral phases present. Reprocessing of the middlings fraction shows that it can separate into high silica and high iron fractions.

Table 4.18 Total Composition Analysis Middlings Fraction Reprocessed using a Dry Drum Magnetic Separator

| | Si | Fe | Al | Ca | Ti | Na | As | Cr | Zr |
|-------------|------|------|------|------|------|------|-----|-----|-----|
| Sample Id | % | % | % | % | % | % | ppm | ppm | ppm |
| “Magnetite” | 0.86 | 47.2 | 13.3 | 0.11 | 1.37 | 0.08 | 13 | 550 | 928 |
| Mags 3 | 2.37 | 42.3 | 11.7 | 0.18 | 3.71 | 0.13 | 8 | 500 | 530 |
| Mags 4 | 4.58 | 44.3 | 7.35 | 0.22 | 3.51 | 0.16 | 8 | 500 | 612 |
| Mags 5 | 4.61 | 43.8 | 6.74 | 0.26 | 1.40 | 0.23 | 21 | 500 | 737 |
| Non-Mags | 35.3 | 10.9 | 3.65 | 0.07 | 0.41 | 0.07 | 6 | 250 | 289 |

The “Magnetite” sample has the highest iron content (47%), the lowest Si (0.86%), and also the highest aluminium content (13%). XRD analysis shows that the mineral phase of the iron in the “Magnetite” sample is indeed magnetite.

The Al present is likely to be as aluminous goethite and aluminous haematite.

The “Magnetite” fraction comprises less than half a percent of the middlings sand.

The Mags 3 and Mags 4 fractions comprise two percent of the middlings sand.

The non-magnetic fraction has the highest silica content (35% Si), the lowest iron content (~11% Fe), and the lowest Al (3.65%) content. The non-magnetics fraction makes up 60% of the middlings sand.

The Si in the non-magnetic fraction is present as quartz. XRD analysis showed trace amounts of gibbsite in the non-magnetic fraction, the Mags 3 to Mags 5 also had gibbsite present but some of the Al may also be present as aluminous goethite and aluminous haematite.

Rare Earth Roll Separator



Figure 4.37 Reprocessing of Middlings sand using a Rare Earth Roll Magnetic Separator

Table 4.19 Middlings Fraction Reprocessed using a Rare Earth Roll Magnetic Separator

| Sample | Mass (g) | Mass % of Middlings Fraction |
|----------|----------|------------------------------|
| Mags 1 | 220.00 | 13.29 |
| Mags 2 | 420.00 | 25.38 |
| Mags 3 | 207.67 | 12.55 |
| Mags 4 | 63.34 | 3.83 |
| Mags 5 | 17.81 | 1.08 |
| Mags 6 | 20.71 | 1.25 |
| Mags 7 | 45.28 | 2.74 |
| Non-Mags | 660.00 | 39.88 |
| Total | 1654.81 | 100 |

Further reprocessing of the middlings fraction using a rare earth roll separator (the magnetic field intensity for the reprocessing unfortunately was not measured) resulted in about 40% of the sample reporting as non-magnetic, 25% reporting to the Mags 2 fraction with the Mags 1 and Mags 3 combined making up a further 25%.

The Mags 1 to Mags 7 represents how close to the rare earth roll the collecting segment is with the Mags 1 being the closest to and the non Mags furthest from the rare earth roll. The Mags 1 fraction contains the mineral phases with the highest magnetic susceptibility whilst the non-magnetics fraction contains the mineral phases with the lowest magnetic susceptibility.

Table 4.20 XRD results for Middlings Fraction Reprocessed using a Rare Earth Roll Magnetic Separator

| Sample | Significant | Trace |
|---------------|--|--|
| Mags 1 | goethite, haematite | quartz, anatase, ilmenite, gibbsite, magnetite |
| Mags 2 | goethite, haematite | muscovite, anatase, gibbsite, quartz |
| Mags 3 | goethite, haematite | muscovite, anatase, gibbsite, quartz, boehmite, kaolin |
| Mags 4 | goethite, haematite, quartz, gibbsite | boehmite, muscovite, anatase |
| Mags 5 | goethite, haematite, quartz, gibbsite | boehmite, rutile, muscovite, anatase |
| Mags 6 | goethite, haematite, quartz, gibbsite, muscovite | boehmite, rutile, anatase |
| Mags 7 | quartz, gibbsite | haematite, boehmite, muscovite, goethite, rutile |
| Non-Magnetics | quartz | gibbsite |

The non-magnetics fraction had the highest Si content (~45%), the lowest Fe content (0.8%) and also the lowest Al content. The main mineral phase of the non-magnetics fraction was quartz with gibbsite present in trace amounts. The Mags 1 had the highest iron content (49% Fe), which was present mainly as goethite and haematite

with a trace amount of magnetite. The Al in the Mags 1 was likely to be present as aluminous goethite and or aluminous haematite.

Further reprocessing of the middlings fraction showed that it could be separated into a high iron fraction (Mags 1 to 3) and a high silica (non-magnetics) fraction which made up 91% of the sample. The remaining fractions (Mags 4 to 7) had lower levels of iron and Si, further reprocessing of these samples may possibly be able to be separate them into non-magnetic and magnetic fractions.

Table 4.21 Total Composition Analysis Middlings fraction Reprocessed using a Rare Earth Roll Magnetic Separator

| Sample Id | Fe | Si | Al | Ca | Ti | Na | As | Cr | Zr |
|---------------|-------|-------|-------|------|------|------|-----|-----|------|
| | % | % | % | % | % | % | ppm | ppm | ppm |
| Mags 1 | 49.00 | 0.96 | 6.85 | 0.27 | 1.65 | 0.23 | 22 | 550 | 877 |
| Mags 2 | 45.00 | 1.02 | 7.94 | 0.28 | 1.48 | 0.25 | 22 | 500 | 859 |
| Mags 3 | 42.20 | 1.80 | 9.47 | 0.27 | 1.49 | 0.27 | 19 | 500 | 797 |
| Mags 4 | 31.90 | 3.58 | 13.90 | 0.24 | 1.63 | 0.25 | 16 | 500 | 805 |
| Mags 5 | 18.50 | 11.70 | 16.10 | 0.25 | 4.06 | 0.20 | 11 | 350 | 1730 |
| Mags 6 | 17.30 | 7.94 | 19.60 | 0.21 | 1.88 | 0.20 | 14 | 300 | 922 |
| Mags 7 | 6.95 | 26.80 | 12.20 | 0.10 | 0.95 | 0.10 | 6 | 300 | 765 |
| Non-Magnetics | 0.81 | 44.90 | 1.02 | 0.01 | 0.06 | 0.02 | -1 | 250 | 217 |

The Mags 5 sample has the highest Titanium and Zircon concentration of the reprocessed middlings fraction. The Mags 5 sample however only comprises 1% of the middlings fraction.

Conclusions

Further reprocessing of the middlings fraction from the WHIMS by both rare earth roll and dry drum magnetic separator indicated that almost all of it could be further separated into high iron and high silica fractions. It may be possible that the remaining fractions of the middlings sand can also be separated into high iron and high silica fractions by further reprocessing. The rare earth roll separator produced

fractions with a higher iron and silica content than the dry drum separator.

These results are consistent with the microscopy analysis which showed that the middlings fraction is comprised mainly of discrete particles of iron oxide and silica (quartz) that should be able to be separated magnetically into two (magnetic and non-magnetic) fractions.

The results show that the original separation was inefficient and that a multi-stage magnetic separation of Red Sand™ would likely to be able to completely separate it into high iron and high silica fractions.

4.6.5 Implications of the WHIMS Middle Fraction for Upstream Processing

Results from the previous chapters clearly show that the coarse residue sand could be separated into different magnetic fractions. There are two reasons why it is believed that the Wagerup sample did not suitably separate using the WHIMS technique.

Firstly the samples were not pre-treated with LIMS to remove the highly magnetic fraction. It is believed that this fraction stuck to the WHIMS plates and induced carry-over for all samples, effectively creating the middle fraction.

Secondly the Wagerup sample was shown to have a high level of “fines” associated with the sand. This was partly removed by the screening process, but agitation and washing was unable to remove it all. This resulted in at least some mud coating to the sand particles and possibly making the quartz particles behave as slightly magnetic particles.

The implications to the treatment of sand are clear.

- Sand should have any fine fraction, removed by either cyclone or filtration process or both.

- Sand should not be dried in the presence of mud as it subsequently difficult to separate. Thus it is preferable if the sand to be separated can be taken straight from the refinery process.
- Wet magnetic separation of sand can only be done on clean sand and as part of a two stage process. First, strongly magnetic material must be removed by LIMS prior to treatment by WHIMS.
- Magnetic separation is simplest on dry sand using rollers. Hence a process that produces clean sand and stockpiles it to dry would benefit subsequent beneficiation processes.

4.7.1 Production of Clean Red Sand™ - Laboratory Assessment

A variety of uses for bauxite residues has previously been outlined by Cooling and Jamieson (2004) and work has now indicated that a suitable product can be made to meet the desired criteria. An attempt was made to use the findings from magnetic separation trials to form a process that would produce sand suitable for several uses, including subsequent magnetic separation.

To meet the Red Sand™ criteria, it is necessary to remove any residual fine mud and soluble salts from the coarse residue and then neutralise any remaining alkalinity through a process of carbonation (neutralisation by CO₂) and washing. This process was first assessed using bench scale trials.

Bench scale samples were obtained by manual sampling of the sand classifier at Alcoa's Pinjarra Refinery. (This was a particularly arduous task as it involved sampling 500 ml a time from a cramped site high in the refinery structure.) The sand was then batch washed in a 200 litre drum (see Figure 4.38) into which CO₂ was sparged.



Figure 4.38: Washing & carbonation of small scale sample

The criterion that the washed sand was required to meet was that the pH of the slurry must be below 10.5 (as below pH 10.5 the sand is not considered a “dangerous good”).

The washed and carbonated sand was then provided to Main Roads Western Australia (MRWA) for physical testing (compaction and load bearing) to determine its suitability as a road pavement material.

This test is known as the California Bearing Ratio (CBR). The California Bearing Ratio Test (CBR Test) is a penetration test developed by California State Highway Department (U.S.A.) for evaluating the bearing capacity of sub-grade soil for design of flexible pavement.

The California Bearing Ratio test is one of the most commonly used in Civil Engineering to evaluate the strength of a sub base, sub grade soil, or base course material for design thickness of roads

Although the CBR rating was developed for measuring the load-bearing capacity of soils used for building roads, the CBR can also be used for measuring the load-bearing capacity of unimproved airstrips or for soils under paved airstrips. The harder the surface, the higher the CBR rating.

MRWA (Bond 2006) reported.....

“From this testing the following conclusions can be made:

- The high CBR results previously obtained by Alcoa are confirmed.

- The soaked CBR results are similar to the unsoaked CBR results, therefore the material is not moisture sensitive, which is a similar attribute to limestone.
- The CBR strength of this material is density dependent.
- The CBR strength of this material is similar to the limestone used as a pavement material on numerous State and Local roads.
- The CBR strength of this material is similar to other naturally occurring sands used as a sub-base material on several State Roads in the South West that now have a service life near 30 years.
- The CBR strength at all the densities and moisture contents tested satisfy the criteria for sub-base material as defined in Engineering Road Note 9 (ERN9) and only requires a minimum basecourse thickness.”

Following this success, a larger scale was attempted to produce one tonne of sand. This would allow assessment of technology to be used in any later pilot plant. Specifically the trial was to use a flat bottom classifier. Flat bottom classifiers (FBC, sometimes also known as Teeter bed classifiers or Hydrosizers) are used in the mineral sands industry for the removal of fine particles and the neutralisation of sand from the synthetic rutile process. However they have not been applied to angular manufactured sand such as coarse bauxite residue.

Screening of the sand (see Figure 4.39) was also required to remove particles greater than two millimetres to prevent blockage of the valves on the flat bottom classifier.



Figure 4.39 Trommel screen for removal of oversize material

After removal of oversize particles the sand was fed into the flat bottom classifier (FBC). Clean water permeated upward through the settling chamber, discharging through the overflow weir. Carbon dioxide (CO_2) was injected into the wash water. The interaction of the upward flow and the settling solids created a fluidised bed which inhibited the settling of fine sized material, whilst allowing the heavier coarser particles to pass to the bottom of the chamber (see Figure 4.40).



Figure 4.40 Flat Bottom Classifier Washing & Carbonating Residue Sand

Process modelling was used to determine the optimal conditions required to minimise CO₂ and water use while maintaining the required level of washing and neutralisation of the finished product.

Table 4.22 Results for sand washed & carbonated using a Flat Bottom Classifier

| Wash rate 1 kL water per tonne of sand | | | | |
|--|------|-------|-------|--------------------------------|
| | pH | TC | TA | Al ₂ O ₃ |
| Feed | 13.1 | 6.11 | 11.60 | 1.56 |
| OF | 11.5 | 0.29 | 2.56 | 0.23 |
| UF | 7.8 | -1.23 | 1.47 | <0.04 |
| Under-Flow solids 65.4% w/w | | | | |

The pH of the sand was reduced from 13.1 in the feed to 7.8 in the under-flow, whilst the pH of the overflow water was 11.5.

Conclusions

Initial test work has indicated that a process including using a flat bottom classifier washing and carbonation of the coarse residue fraction can produce clean sand that meets Red Sand™ standards for use as general fill.

The operation of screening and dewatering are essential for the successful operation of the flat bottom classifier:

- Trash screen to remove large particles that could block the underflow valve.
- Dewatering screen for removal of process liquor which contains soluble salts.

4.7.2 Production of Clean Red Sand™ - Pilot Plant

Following success of laboratory and bench scale trials, production of clean residue sand was to be up-scaled to a pilot plant. This chapter is a summary of Jones, A. (2009). Wagerup Sand Wash and Carbonation Pilot Plant. Alcoa Report AWA 7.05 CAB020. It has also been reported in CSR report for projects 3B4 (pilot plant trials), 3B5 (road construction trials) and Jones (2007).

A pilot plant was set up at Wagerup to produce clean sand (with a pH of less than 10.5 and a salt content of less than 5 g/l) for demonstration projects. This section outlines the basic process of the pilot plant.



Figure 4.41 Sand Wash & Carbonation Pilot Plant (photo courtesy of Garside Images)

The sand pilot plant takes a feed (10 tph, 22 kL/h) from a sand relay line (35% w/w solids). There are two sand relay lines which are fed from the sand buffer tank which is fed sand from the refinery with lake water make-up.

The two sand relay lines are used to pump sand to the various residue drying areas (RDAs) at Wagerup. One of the sand relay lines is a four pump system, the other a two pump system. The sand slurry feed density to the pilot plant varies depending on which of the sand relay lines is running at the time and also to which of the RDAs the sand is being pumped. The sand feed density is higher on the two pump system than on the four pump system. If the RDA to which the sand is being pumped is one of the ones further away from the pilot plant the sand feed density is lower due to more lake water being added to aid the pumping.

A higher sand feed density results in a thicker cyclone underflow and a thicker sand bed on the dewatering screen and thus more sand entering the Flat Bottom Classifier (FBC). It also results in more liquor per hour being carried over into the FBC. This extra sand results in a shorter retention time in the FBC (i.e. it takes less time to achieve the set bed height).

Table 4.23 Design Process Flow Data

| Process Stream in P&ID | | 1 | 4 | 5 | 11 | 13 | 14 | 15 | 16 | 17 | 18 | 19 |
|--------------------------------|-----------|---------------------------------------|---------------------------|---------------------------|-----------------------------------|-----------------------------------|------------------|-----------------|--------------------------|-----------------------------|-----------------|---------|
| | Units | SRT feed sand slurry line | Trash screen underflow | Trash screen discharge | Dewatering screen discharge | Dewatering screen underflow | FBC discharge | CO ₂ | Fresh water to FBC | Product pumping water | FBC overflow | Product |
| Slurry | kLph | 58 | 58 | 0.1 | 5 | 53 | 10 | NA | 8 | 12 | 4 | 21 |
| Sand | tph | 10.0 | 9.9 | 0.1 | 9.9 | 0 | 9.5 | NA | 0 | 0.0 | 0.4 | 9.5 |
| Liquor | tph | 57 | 57 | 0.1 | 2 | 55 | 6 | NA | 8 | 12 | 3 | 18 |
| Liquor | kL/hr | 55 | 55 | 0.1 | 2 | 53 | 6 | NA | 8 | 12 | 3 | 18 |
| Slurry | tph | 67 | 66 | 0.2 | 12 | 55 | 16 | NA | 8 | 12 | 4 | 28 |
| Solids | (gpl) | 464 | 464 | 759.7 | 1964 | 0 | 997 | NA | 0 | 0 | 118 | 450 |
| TC/TA | | 1 | 1 | 0.6 | 1 | 1 | 1 | NA | 0 | 0 | 1 | 1 |
| A/TC | | 0 | 0 | 0.5 | 0 | 0 | 0 | NA | 0 | 0 | 0 | 0 |
| TC | (g/L) | 17 | 17 | 17.0 | 17 | 17 | -3 | NA | 0 | 0 | 3 | -1 |
| TA | (g/L) | 28 | 28 | 28.3 | 28 | 28 | 5 | NA | 0 | 0 | 5 | 2 |
| Al ₂ O ₃ | (g/L) | 8 | 8 | 7.8 | 8 | 8 | 0 | NA | 0 | 0 | 1 | 0 |
| Density Liquor | (g/mL) | 1 | 1 | 1.0 | 1 | 1 | 1 | NA | 1 | 1 | 1 | 1 |
| Density Solids | (g/mL) | 3 | 3 | 3.0 | 3 | 3 | 3 | NA | 3 | 3 | 3 | 3 |
| Slurry Solids | (% wt/wt) | 15 | 15 | 50.0 | 85 | 0 | 60 | NA | 0 | 0 | 11 | 34 |
| CO ₂ | (kg/hr) | NA | NA | NA | NA | NA | NA | 40 | NA | NA | NA | NA |

The trash screen has a spray bar with fresh water to help the sand pass through the screen. The water from the spray bar also helps to reduce the TA of the liquor in the slurry.

The sand slurry that passes through the trash screen falls into a sump with fresh water make-up, and is then pumped to a second Linatex 250 Genesis hydrocyclone.

The second cyclone separates off more of the liquor and mud from the sand slurry. The underflow discharges onto a second vibrating (dewatering) screen, with 300 μm aperture screen panels. The dewatering screen removes the process water from the sand slurry and discharges a sand bed with a moisture content of approximately 20%.

Table 4.24 Normal operating conditions for the pilot plant process streams

| Parameter | Normal operating conditions |
|---|-----------------------------|
| Trash screen cyclone inlet pressure | 130 to 150 KPa |
| Dewatering screen sand bed height | 70 to 80 mm |
| Sand bed level in FBC | 70% |
| CO ₂ flow rate (as measured using the mass flow meter) | 25% to 35% |
| Water pressure | 400 to KPa |
| Trash screen sump makeup water flow rate | 15 to 20 KL/hr |
| FBC wash water flow rate | 10 to 20 KL/hr |
| FBC sump make up water flow rate | 5 to 10 KL/hr |
| Dewatering screen sump make up water flow rate | 30 to 40 KL/hr |

Note that the fresh water make-up into the dewatering screen sump is only required to enable the undersize sand from the dewatering screen to be pumped away and that lake water could be substituted for fresh water in a full scale plant. The fresh water make-up into the sump plays no role in washing and neutralisation of the final product.



Figure 4.43 Sand Bed coming off the Dewatering Screen

The sand bed coming off the end of the dewatering screen (figure 4.43) is transferred by conveyor belt to a 1.5 m (figure 4.44) diameter flat bottom classifier.



Figure 4.44 Sand Travelling up the Conveyor into the FBC

In Figure 4.45 the sand enters the FBC through a central feed well (1) where an impact plate prevents the sand feed from plunging into the suspension, and allows the sand to then be uniformly distributed into the settling chamber (2).

Clean water is injected to a plenum chamber (3) located under the settling chamber. The clean water permeates a membrane (4) and flows upward through the settling chamber, discharging over the overflow weir (5). CO₂ is injected into the wash water through a Lightnin Series 45 Inline mixer. The mixer has a helical element design that creates a distinct pattern of flow splitting and rotating vortices which disperse the gas bubbles in the water. Dispersion of the gas bubbles is essential for efficient neutralisation of any residual caustic.

low a flow will fail to sufficiently fluidise the sand and will result in a poorly washed, high pH sand product.

The set point for the bed height in the FBC is critical in that too low a set point results in a smaller fluidised bed, thus decreasing retention time in the tank and resulting in high pH sand product. If the set point is too high; the finer fractions of the sand can be lost to the overflow resulting in the final product being too coarse. Also running with the set point of the sand bed too high can result in operational issues with the tank reading the bed level as greater than 100% and starts to continuously discharge sand into the sump (thus blocking the sump), this can occur if there is a sudden increase in the water pressure which usually occurs when refinery (residue operations) personnel turn on the residue area sprinkler system (used for dust suppression in the residue drying areas).

The underflow of the tank discharges into a sump (the sump has fresh water make up) and the sand slurry is pumped from the sump to the stockpile.



Figure 4.46 Sand Slurry Discharging to build the Stockpile

The sump pump discharge is sampled and monitored for pH. The pH reading of the sample is affected by the timing of the sampling – i.e. the elapsed time between when the underflow from the FBC was discharged into the sump was the sample taken.

The sand that is discharged from the end of the pipe into the stockpile had a lower pH than that of the FBC sump discharge due to the mixing that occurs in the pipe whilst it is being pumped to the stockpile.

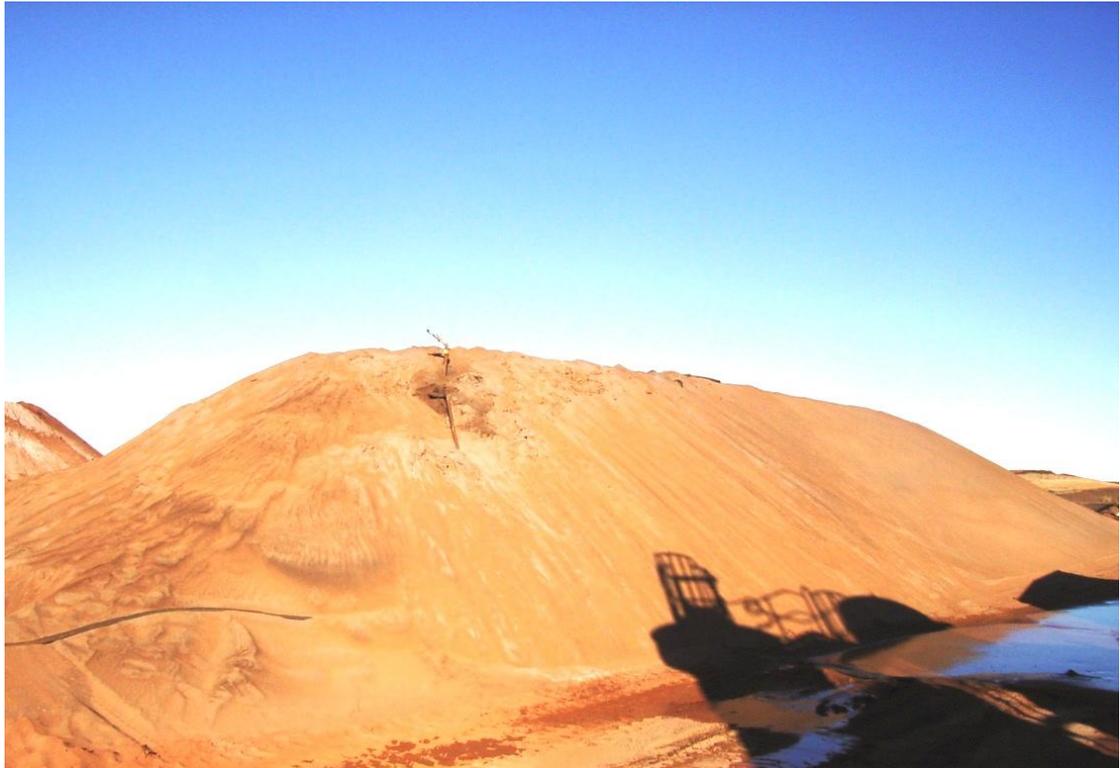


Figure 4.47 Clean Sand Stockpile

Summary:

There are three factors that have the biggest influence on the operating efficiency of the pilot plant. These are:

- Sand slurry feed density. This will influence the retention time of the sand in the FBC (higher feed density means that there is more sand entering the FBC and forming the bed at a faster rate and thus reducing the retention time in the FBC).

- Water pressure – water is used to control the level control of the trash screen sump and the slurry pumped to the dewatering cyclone. If the water pressure is too low there is insufficient water to enable the slurry to be pumped into the dewatering cyclone which can result in the cyclone either blocking or operating inefficiently which in turn, leads to the cyclone underflow having a lower density and thus more liquor being entrained in the sand coming off the dewatering screen. Water is also used for counter-current washing in the FBC and as a make-up in the FBC sump to enable the sand slurry to be pumped to the stockpile. If the water pressure is too low the product pump can become bogged. If the water pressure is too high the water surges into the bottom of the FBC and can send a slug of sand up towards the level probe which can result in either the probe registering the sand bed as being higher and therefore a lower retention time of the sand in the FBC (results in a higher pH of the product), or the level probe reading that the sand bed in the FBC is greater than 100% and thus above its set point and starts to continuously discharge sand slurry from the underflow valve, which is the most common consequence of the water pressure being high
- The height of the fluidised sand bed in the FBC - again this influences the retention time of the sand in the FBC, with a higher bed height increasing retention time of the sand in the FBC and thus more contact time with the water and CO₂. This gives a sand product with a lower pH.

In the case of the pilot plant constructed and operated at Wagerup, only one (the height of the sand bed in the FBC) of the three factors was within control of the pilot plant operators. The other two are determined by the day to day running of Wagerup Residue Operations.

4.7.3 Clean Red Sand™ Assessment

Assessment of Final Red Sand™ Product from Wagerup Pilot Plant

The operation of the pilot plant initially focused upon operational ability and then focused upon product performance. Assessment of pH and conductivity of the Wagerup pilot plant sand was conducted to assist with process operational performance and control measures. It was determined that the process could be controlled by regular conductivity assessment. It was shown that the conductivity of the sand transport-water was strongly correlated with pH (Figure 4.48). It was determined that if conductivity for the sand product was held below 2000 $\mu\text{S}/\text{cm}$, then a maximum pH of 10 or less was maintained. This is acceptable as further processing resulted in the transport-water being drained away as the sand dried. Rain or other wetting of the sand resulted in significant dilution and much lower resultant conductivity. This demonstrates that the sand salts are from the transport water, and are not dissolved from the sand itself.

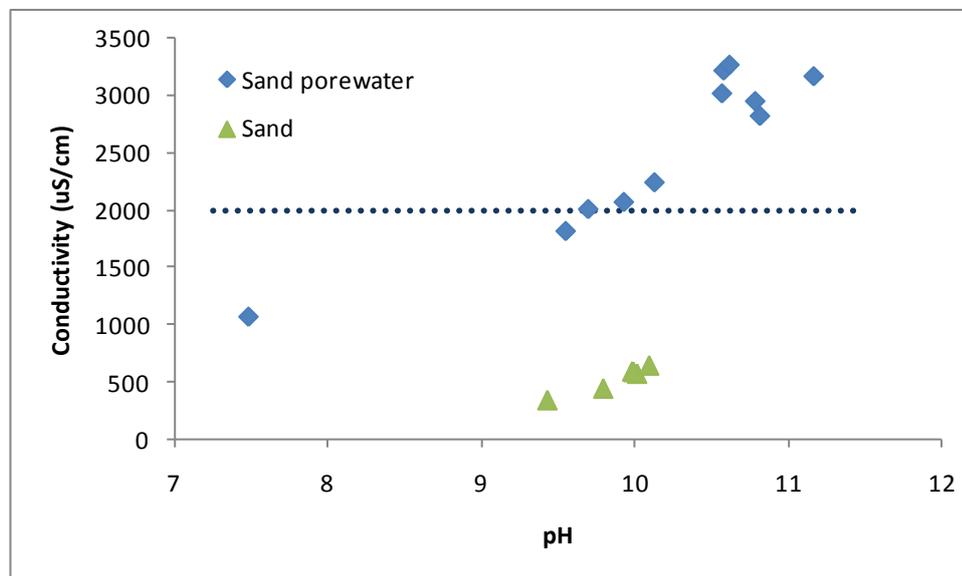


Figure 4.48 pH vs. Conductivity of sand samples' pore-water, and sand after pore-water was decanted

During this process of operational development, product samples were screened for analysis and tested by Main Roads Western Australia (MRWA) for compaction. The MRWA reported that the sand performance had significantly declined from the

original trial samples and this was likely associated with the absence of fines. Process conditions were then changed (cyclone pressures, cyclone drop tails and washing practices) to improve the associated levels of fines in the product. The resulting conditions were then maintained for the production process. The assessment of this material has been reported in Carter, C., Attiwell, S., and Jones, A. (2009. Evaluation of Red Sand Product from the pilot plant. Alcoa Report AWA 7.05 CAB019), but is summarised below.

Results

The particle size distribution (PSD) of the pilot plant product sand was adjusted to increase the level of fines from 3% < 150µm (PSD determined by MRWA) in the original sand product to around 8.3% (Table 4.25). The amount of material > 850 µm also increased; so that the material contained a broader spread of particle sizes (Figure 4.49).

Table 4.25: Particle size distribution (PSD) of pilot plant sand (% by mass)

| Screen Size (µm) | Average | MRWA Range | PSD of Original Sand Product as Determined by MRWA |
|------------------|---------|-------------|--|
| +850 | 5.48 | 2.5 – 8.3 | 1 |
| 600 – 850 | 12.84 | 7.4 – 15.6 | 4 |
| 425 – 600 | 16.12 | 12.2 – 19.0 | 16 |
| 250 – 425 | 33.58 | 30.0 – 36.8 | 56 |
| 150 - 250 | 23.68 | 18.1 – 30.4 | 20 |
| -150 | | | 3 |
| 75 – 150 | 7.71 | 4.0 – 14.4 | |
| 45 – 75 | 0.50 | 0.2 – 1.0 | |
| -45 | 0.10 | 0.05 – 0.15 | |

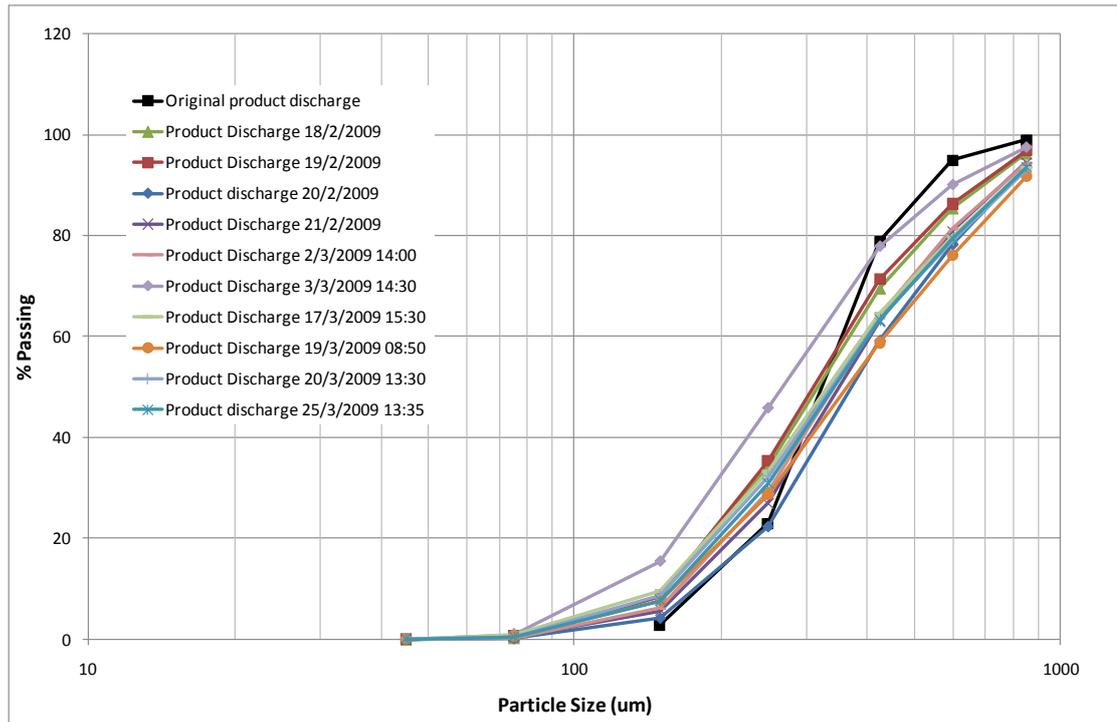


Figure 4.49: PSD (% by mass) of sand pilot plant compared to original sand

The conductivity of the sand transport water varied between 1000 and 3500 μ S/cm, dependent on pH. The pH of the transport water rose with time over the first 5 hours after sampling and then stabilised. Once the transport water was drained and the sand dried, there was no change to the pH.

The acid neutralisation capacity of Wagerup pilot plant sand was low, meaning that a pH change can be easily achieved through the addition of a small amount of acid or base. If lower pHs are required, then small amounts of gypsum can also be added.

The phosphorus retention index (PRI) was relatively high at 12mL/g; hence the sand can be suitable for phosphorus retention. That is, Wagerup pilot plant sand could be used in applications that require phosphorus pollution control such as on golf courses, effluent treatment etc.

Composition and leach testing was assessed on the initial Wagerup pilot plant samples. There is no suitable legislation for by-products, so an assessment was made

against the Western Australian landfill regulations. Based on ASLP leach data, the sand was classified as inert. Carter, C. and Attiwell, S. (2008). Environmental Review of Processed Residue Sand. Alcoa Report AWA 7.05 CAB008: Attiwell, S., & Carter, C., (2009). Use of Alcoa Residue Sand for Top Dressing Fairbridge oval: A Demonstration Project. Alcoa Report AWA 7.05 CAB018. : Carter, C., Attiwell, S., and Jones, A. (2009). Evaluation of Red Sand Product from the pilot plant; Alcoa Report AWA 7.05 CAB019.

Physical testing of the sand for compaction and load bearing were conducted by MRWA and reported by WML (contractors) to CSR as part of the P129 final report.

“The laboratory Californian Bearing Ratio (CBR) values for the 2009 ReSand® are similar to the laboratory CBR values obtained in 2006 on similar ReSand® material.

The laboratory CBR testing conducted on the 2009 ReSand® compacted at or above 94% of Modified Maximum Dry Density (MMDD) gave values of 60% or greater. These results show that the 2009 ReSand® is potentially strong and has the strength to replace limestone as a sub-base material in the pavement layer where 100mm of gravel basecourse is applied. This promising laboratory result requires confirmation by the construction of field trials.”

Recommendations

- No further characterisation work is necessary; ongoing monitoring of any sand product should occur to ensure that the product remains within specifications.
- Sand is suitable for trials as fill, soil ameliorant and for road construction.

4.8 Use of Clean Red Sand™ in Road Construction

Sand from the pilot plant was used in demonstration trials as part of the Centre for Sustainable Resource Processing (CSRP) project P129. It has been reported in Jamieson, E. (2009). Use of Alcoa Residue Sand in Road construction – a Demonstration project, Alcoa Report AWA 7.05 CAB022: A brief summary is made below.

Some 2000 m³ of Red Sand™ was used in road construction in by Main Roads Western Australia (MRWA) and Southern Road Services. This material has been used as both the sub-grade as well as the sub-base materials. The physical road performance will be monitored by Main Roads. The Department of Agriculture and Food, Western Australia (DAFWA) has been contracted to monitor the environmental performance for a period of 2 years.

Feedback from the Southern Road Services to date has been positive and includes:

- Drainage of Red Sand™ is very good.
- Wetting is even and fast.
- Red Sand™ allows heavy construction vehicle access with minimal preparation.
- Rolling produces a high degree of “polish”.
- Compaction of red sand appears superior to traditional materials allowing a wide range of uses.



Figure 4.50 TDG Pilot Plant producing Red Sand™ for trials



Figure 4.51 Delivery of Red Sand™ to Greenlands Road



Figure 4.52 Grading of Red Sand™



Figure 4.53 Soaking of Red Sand™



Figure 4.54 Red Sand™ collection from Pilot Plant



Figure 4.55 Rolling of Red Sand™

Results of trials are still being collected and will be part of the CSR project P128 (DAFWA leach monitoring) & P129 (MRD/WML physical assessment).

4.9 Suitability of Clean Red Sand™ for Iron Beneficiation

Sand from the pilot plant was separated using an induced roll magnetic separator. Clear separations were possible. As can be seen from the microscopy images below, the separations were clean. The dark coloured particles in the non-magnetics fractions are predominately quartz with a fine coating of mud. The microscopy images indicate that the design and operational control of the pilot plant specification are suitable to produce a product that can be beneficiated using magnetic separation techniques.

Further investigation is recommended on the use of magnetic separation of clean sand as a low grade iron ore substitute.

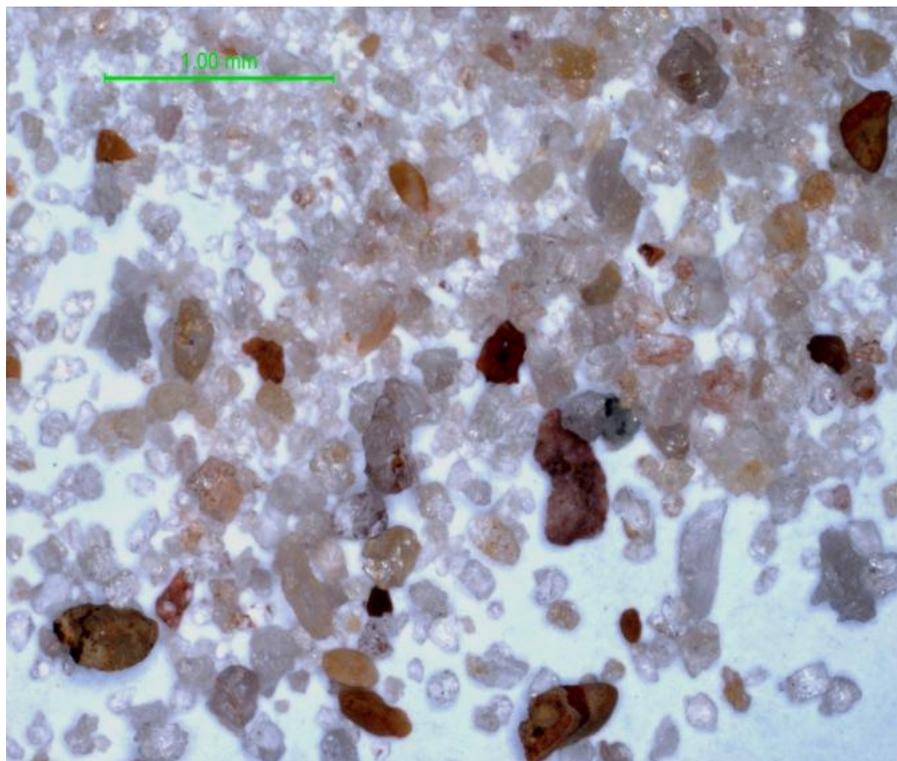


Figure 4.56 Non Magnetics fraction

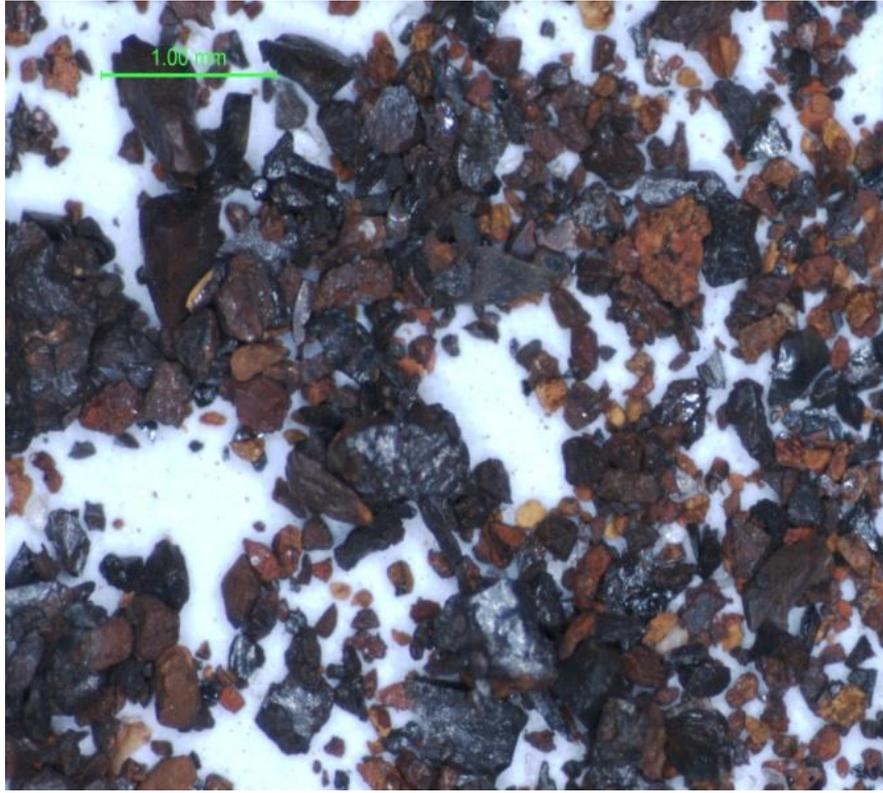


Figure 4.57 Magnetics fraction

5.0 Conclusions

A series of assessments have been conducted to determine if coarse bauxite residue can be used as construction sand. As part of this process various separation techniques were assessed to determine the product criteria that would produce a sand product of widest applicability, both to construction as well as to further beneficiation. Process requirements settled upon were:

- Fresh material not being dried with slimes.
- Separation of slimes is essential.
- Separation of fines must be limited to allow compaction strength for construction.
- Neutralisation of remaining caustic by carbonation.
- Removal of excess salt by washing.

Further analysis has shown that magnetic separation of an iron rich material is possible; however the “iron ore” has a high alumina content. Atmospheric and mid-temperature digestion failed to significantly remove the alumina, however high temperature (>235C) was successful.

The processing of Red Sand through the production of clean sand, and the different and non-magnetic fractions is seen as a route towards the greater utilisation of this material in Bayer Process Residue. The potential of producing an iron rich by product, a silica rich product and the recovery of more aluminium from the bauxite ore are all desirable in the goal of enhancing the sustainability of the aluminium industry in Australia.

6.0 Recommendations for Further Research

A variety of suggestions for further work have been made throughout the text however key recommendations are listed below.

- Continue demonstration trials for multiple applications of clean Red Sand™.
- Conduct analytical assessment of magnetic fraction from the pilot plant clean sand.
- Conduct trials on drum magnetic separation of pilot plant sand.
- Conduct trials on high iron clean sand for its suitability in iron production.
- Conduct further studies on the recovery of aluminium from red sand.

It would appear that the production of sand from bauxite residue is both possible and produces a product superior to virgin natural sand. The angular nature of the sand improves its compaction strength making it better suited to load-bearing. The high iron content can also be beneficiated with potential use as iron ore, agriculture, soil amendment and environmental remediation.

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Appendix A

Glossary

Alian. Alcoa Liquor Analyser is a titration based method based on the Watts Utley (1956) method. The Alian reports values for TC, TA, Al_2O_3 , and ratios of A/TC and TC/TA and are defined as:

The concentration of the aluminate ion ($\text{Al}(\text{OH})_4^-$), that forms during the dissolution stage of gibbsite ($\text{Al}(\text{OH})_3$) from bauxite by the addition of hydroxyl ions (OH^-) from the liquor (sodium hydroxide), is termed alumina (A) and reported as grams per litre Al_2O_3 .

Remaining or excess hydroxyl ions are termed free caustic since they are not bound with an aluminate ion. The total sodium hydroxide concentration containing free and bound hydroxide is termed the Total Caustic (TC), is reported on a grams per litre sodium carbonate basis.

The total alkalinity (TA) is the sum of the total caustic and the sodium carbonate concentration present in the liquor.

TC/TA is the ratio of total caustic to total alkalinity in the liquor and is important to alumina refining as is it the total caustic and not the total alkalinity which determines the amount of alumina that can be dissolved from the bauxite.

A/TC is the ratio of alumina to total caustic in the liquor and is used for process monitoring and control in alumina refineries.

Available Alumina. The amount of Al_2O_3 that can be extracted from bauxite under conditions simulating alumina extraction in the Bayer process is termed available alumina.

Boehmite [γAlOOH]. An aluminium oxide hydroxide mineral component of bauxite ores. The mineral occurs together with gibbsite in deposits of tropical laterites. Boehmite is the second most valuable mineral after gibbsite, found in bauxites. Boehmite requires higher digestion temperatures in order to achieve the alumina to total caustic ratio that is desired for alumina refineries.

California Bearing Ratio. The California Bearing Ratio Test (CBR Test) is a penetration test developed by the California State Highway Department (U.S.A.) for evaluating the bearing capacity of sub-grade soil for design of flexible pavement. The test is performed by measuring the pressure required to penetrate a soil sample with a plunger of standard area. The measured pressure is then divided by the pressure required to achieve an equal penetration on a standard crushed rock material.

Cyclone. A cyclone (sometimes called a hydrocyclone) is piece of equipment used in the Mineral Processing Industry for classification. The principle of how the cyclone works is that a slurry is fed in tangentially which causes it to spiral, the particles are then acted upon by two opposing forces. These forces cause the larger (and heavier) particles which are faster settling to exist through a spigot at the bottom of the cyclone, whilst the slower settling particles are forced upwards and exit through the top of the cyclone.

Desilication Product (DSP). An insoluble sodium aluminium silicate formed by the dissolution of silicate minerals in Bayer liquor. DSP has the formula $3\text{Na}_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot \text{Na}_2\text{X}$, where $\text{X} = \text{SO}_4, \text{CO}_3, \text{OH}, \text{AlO}_2$.

Gibbsite [$\text{Al}(\text{OH})_3$]. An aluminium hydroxide mineral, along with boehmite and diasporite is one of the three main aluminium phases in bauxite. Gibbsite is the principal constituent of bauxites from the tropic region. Gibbsite is the most valuable and desirable of the minerals found in bauxite.

Goethite [FeOOH]. An iron bearing oxide (iron oxyhydroxide) mineral found in soil and other low-temperature environments.

Green Liquor. Process liquor (sometimes known as pregnant liquor) with a high alumina to total caustic ratio.

Haematite [Fe_2O_3]. Ferric oxide mineral and derives its name from the Greek word for blood due to its typical red colour.

Kaolinite [$\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$]. A common clay mineral with a relatively high alumina content (up to a maximum of 39.5% Al_2O_3) and has been used in alternative alumina processes.

Liquor. Solution of sodium aluminate, sodium hydroxide (caustic soda), sodium carbonate as well as numerous organic and inorganic impurities.

Maghemite [$\gamma\text{Fe}_2\text{O}_3$]. Iron mineral formed by weathering or low-temperature oxidation of iron(II) containing minerals such as magnetite.

Magnetite [Fe_3O_4]. An iron mineral which has magnetic properties (which give it its name). Magnetite has a lower iron content than haematite.

Reactive Silica. The amount of siliceous minerals that will react with caustic under Bayer process conditions is termed reactive silica.

Spent Liquor. Process liquor with a low ratio of alumina (Al_2O_3) to total caustic.

Appendix B

The strength of the magnetic field intensity of the Reading Induced Roll Magnetic Separator at Western Australian School of Mines (WASM) is varied by adjusting the dial setting. However there is no indicator of the actual field strength on the magnetic separator. To determine the field strength at a particular dial setting, the maximum magnetic field intensity was measured (using a Hirst Fluxmaster FM70 instrument) at various dial settings.

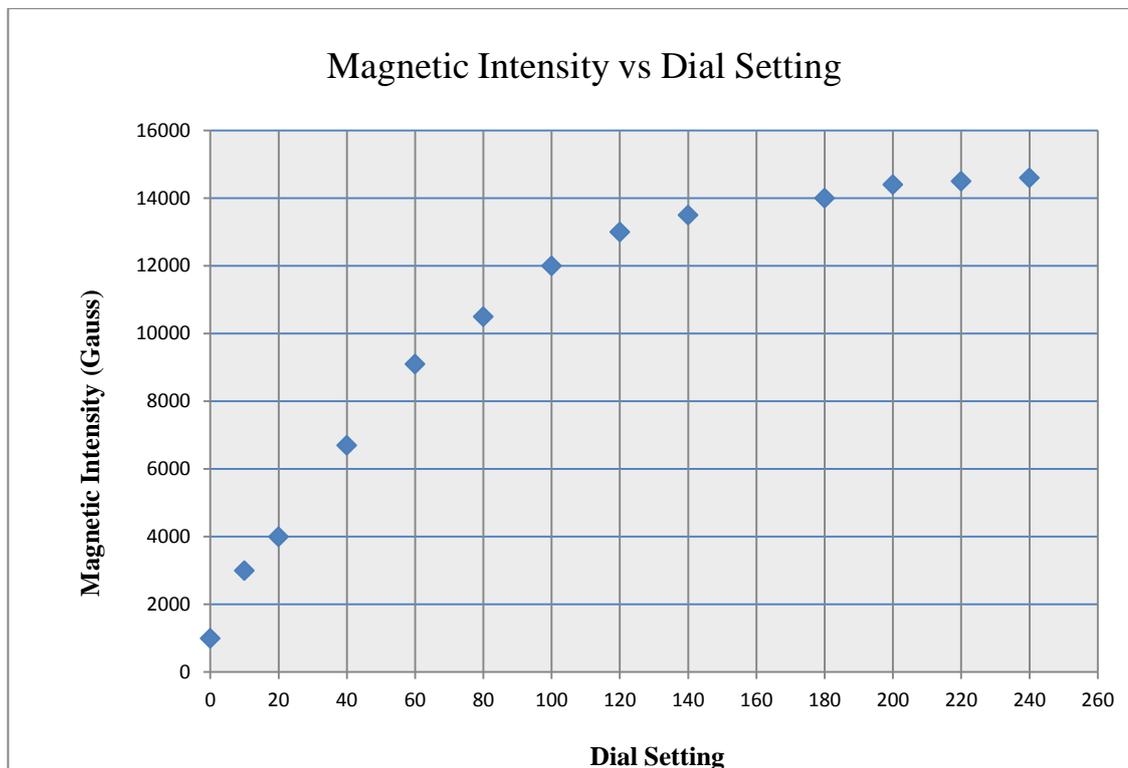


Figure B1 Measured magnetic field intensity at various dial settings on the WASM Reading Induced Roll Magnetic Separator

Appendix C

“Publications” from this project are made up of internal (to Alcoa) reports and external papers.

Internal Reports

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External Publications

Jamieson, E., Jones, A.; Cooling, D. and Stockton, N. (2006). Magnetic Separation of Red Sand to Produce Value. *Minerals Engineering* 19 (2006) 1603–1605.

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