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Experience Gained in Bench Scale and Pilot Scale Fluidised Bed Processing

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ABSTRACT

The CSIR has had a strong drive in fluidised bed technology for over two decades. During this period collaboration has been fostered with numerous partners and clients. By being able to draw on their resources, skills, test and other facilities, considerable progress has been made. The earliest developments were in coal combustion. This was formalised in the formation of the NFBC boiler in the early 1980's for the investigation of utilising discard and duff coals.

Work has been done on in-bed sulphur capture to reduce the sulphur dioxide off-gas content. Additionally, work has been conducted on a number of sulphur bearing ores. More recently attention has been placed on the oxidative and reductive roasting of ilmenite to affect the downstream recovery of titanium dioxide.

Expertise in the design and commissioning of industrial-scale plants has led to the supply (through licensees) of a biomass sludge incinerator/boiler generating 26t/h steam, a 20 MW high-sulphur pitch incinerator and a 12 MW fluidised bed deodoriser.

In the late 1990's the Fluidised Bed Centre of Excellence was formed to combine the expertise and facilities of the CSIR and Kumba Resources, particularly in the field of combustion and minerals beneficiation.

INTRODUCTION

The activities of the CSIR are extremely diverse, ranging from food technology to aeronautics. Fluidised bed technology has been a major initiative within the CSIR for over two decades, originally concentrating on combustion, but later expanding to include minerals treatment. A great deal of progress has been made in the field, and such progress would certainly not have been achieved were it not for the privilege of collaborating with a wide range of partners and clients during this period.

THE EARLY YEARS

In the mid 1970's, the CSIR commenced investigation into fluidised bed coal combustion and gasification. For this purpose an initial refractory-lined bubbling fluidised bed test rig was constructed consisting of a 0.25 m^2 bed area with a freeboard height of approximately 1.5 m.

The research became more focused in the early 1980's when investigation was initiated into the use of discard and duff coals by means of Fluidised Bed Combustion (FBC) technology. This work was funded by the Department of Minerals and Energy Affairs (DMEA) and resulted in the establishment of The National Fluidised Bed Combustion (NFBC) boiler in 1984.

The NFBC was designed with a bed area of 9.3 m^2 and was rated at 12 t/h of steam. This steam was generated utilising a membrane wall, a bi-drum convection bank and in-bed tubes. The facility was primarily used for thermal and combustion efficiency trials where, ironically, the chief hurdle was not the combustion of the coal, but rather in achieving accurate, repeatable results. The test procedure evolved to a stage where this accuracy could be ensured. The effects of load, grit re-firing and bed temperature on the thermal and combustion efficiencies were investigated during this phase of development.

Sulphur reduction trials were conducted with the aim to determine the ability of South African sorbents to reduce sulphur dioxide emissions. This study was conducted in collaboration with the University of Cape Town and the Energy Research Institute.[1]

Additionally, anthracite discard combustion trials were conducted to determine ignition temperatures and suitability for utilisation in an FBC. Although the anthracite was able to ensure a relatively high bed temperature after an extended slump period, the low reactivity thereof meant that there was a high carbon content in the ash and hence a very low combustion and thermal efficiency.

BIOMASS FLUIDISED BED INCINERATOR

In 1990/1991 theoretical studies commenced on a fluidised bed incinerator for co-firing biomass with coal. This was followed by small scale combustion trials on the initial test rig mentioned previously. This work confirmed the theoretical studies and established the actual thermal balance over the bed. Subsequent large scale fluidised bed combustion trials were undertaken in the NFBC. Although this work is covered in detail in the paper presented by Brian North, it is worthwhile noting here that the trials conducted in the NFBC were instrumental in establishing, amongst others, the balance required between the degree of in-flight flash evaporation and over-bed combustion and for optimisation of the dispersion of the feed sludge.[2]

The successful theoretical studies, test work and favourable economics led to the construction and commissioning of a commercial plant in January 1994. The plant has been running successfully since.

A SLICE OF REAL LIFE

The success of the industrial scale biomass fluidised bed incinerator was achieved due to the concentrated effort placed into the upfront theoretical studies and test work. It was not possible, however, to determine one of the main criterion (determination of in-flight flash evaporation) on the initial test rig due to its lack of adequate freeboard height. This issue was resolved when tests were subsequently conducted on the NFBC.

The need quickly became apparent for a replacement facility for the initial test rig – a test facility that would incorporate the functionality and freeboard height of the NFBC with the flexibility of the smaller initial test rig. Hence, in 1992, the Multi Purpose Fluidised Bed Combustor (MPFBC) was commissioned in Pretoria West with a bed area of 0.375 m^2. The unit was designed and built with a relatively tall freeboard (4.5 m) in order to draw comparisons with an earlier industrial plant, the Slagment Fluidised Bed Hot Gas Generator commissioned in 1989. It was felt that the MPFBC was a good representation of a vertical slice through the Slagment plant, which led to the coining of the phrase: "A *Slice of Real Life*".

Gamsberg Ore Concentrate

One of the initial projects conducted on the MPFBC was for Gold Fields who required a relatively small amount of Gamsberg ore concentrate (essentially zinc sulphide) processed at a higher than normal excess air level. This made it impossible to process in existing industrial scale roasters, and the flexibility of the MPFBC was consequently chosen. This production was part of a much larger project the purpose of which was to ascertain whether Gamsberg ore could be utilised successfully, with the main concern being the high levels of manganese in the ore. It was hypothesised that a fluidised bed roaster operated at higher excess air levels would be able to overcome the problem producing a roasted material allowing easier removal of the manganese via subsequent leaching.

Ten tons of the ore concentrate was produced by Mintek and then roasted in the MPFBC to produce the calcine for further treatment by Gold Fields. The bed was operated at 910°C with a superficial gas velocity of 0.5 m/s and a feed rate of 40 kg/h. The off-gases were cooled in a heat exchanger after which the bulk of the product was removed with a bag filter. Thereafter, the gas (containing 8% sulphur oxides) passed through a wet scrubber before being vented though a stack.

The fluidised bed roasting proved highly successful with the concentrate being combusted autothermally even at a fairly high excess air level of approximately 75%. Despite the success achieved, a much more attractive method was identified after completion of the roasting work. This involved direct smelting and subsequent fuming of the zinc from the calcine. Consequently, further work on the roasting option was terminated.

Fragmented Marine Shell

An interesting application was explored in 1992 when the ability of a gas-fired fluidised bed was investigated for the calcination of an on-shore marine shell deposit for final use in the production of pool chemicals. 50 tons of lime (calcium oxide) was to be produced from the marine shell deposit (calcium carbonate) for subsequent hydration by Bredasdorp Lime and final chlorination by Aquachlor. Fluidised bed technology was chosen for producing a high quality lime due to the ability to control the reactor temperature accurately, which enabled a close control of slaking characteristics.

The MPFBC's bed was maintained between 900 and 950°C with a calcium oxide residence time of 3.9 hours and a superficial fluidising velocity of 1 m/s. Many operational problems were encountered during the investigation including the generation of a hard, glass-like 'clinker' at the base of the bed after two to three days' operation. Additionally, there was a build-up of lime on the walls of the reactor, in the cyclone and in the heat exchanger. The build-up in the heat exchanger was the most serious, and could necessitate a shut-down every two to three days. Finally, a hard deposit was also found in the duct to the cyclone. This was assessed to be calcium carbonate formed due to recarbonation of fine CaO in the off-gas on cooling of the gas in the ducts. These problems were eventually overcome and the required 50 tons of lime was produced.

Unfortunately, despite the technical success of the project, an unforeseen economic shift meant that the project did not go ahead in South Africa. Lime is currently imported from abroad.

LPG injection nozzles

A large degree of uncertainty surrounded the fluidised bed calcination of the fragmented marine shell, which necessitated up-front development work. Chief amongst these were the development of a new LPG nozzle for the direct and continuous firing of LPG in the bed as a heat source. LPG firing had been used successfully to start up FBC's. However, with continuous firing of gas in the bed itself, problems were encountered. Melting of the bed material occurred due to the high flame temperature at the nozzles. Additionally, the flame would occasionally burn back into the riser of the nozzles and melt the distributor cap. To overcome this, a new nozzle was developed that split the gas into two streams – an air rich and a gas rich stream. These streams combined and burnt within the bed, successfully avoiding the high flame temperatures.

Calcination Test Rig

The experience gained during the marine shell calcination work highlighted the need for a smaller and more flexible fluidised bed test facility with a great degree of heat recovery. This led to the development of the Fluidised Bed Calcination Rig (FBCR). This was designed with a bed area of 0.04 m^2 and pendulum heat exchanger in the expanded freeboard (0.16 m^2) for preheating of the fluidising air.

Amongst other projects, this rig was successfully utilised in a production campaign for the calcination of 200 kg of calcium carbonate in July 1999, and the production of magnesium oxide from a magnesium chloride solution.

HIGH SULPHUR PITCH INCINERATION

As mentioned earlier, considerable experience was achieved in the fluidised bed capture of sulphur in fluidised beds utilising limestone addition in the NFBC. This formed the basis for the commencement of test work in the early 1990's for a fluidised bed incinerator for a high sulphur pitch and a waste water stream contaminated with organic compounds.

Sasol produces the high sulphur pitch (HSP) waste stream from sulphuric acid stripping of Low Neutral Oil Depitched Tar Acids (LNO-DTA), being a by-product of the coal gasification process. Normal incineration of the HSP without gas cleaning would lead to high levels of SO_2 in the flue gas due to the high sulphur content (7-10%) in the pitch.

Test work was initiated on the MPFBC in order to determine the level of sulphur capture achievable with different types of limestone, as well as an operable pitch injection mechanism due to the high viscosity of the HSP at room temperature. Excessively high freeboard temperatures were experienced causing molten limestone deposits in the exit duct. This was due to over-bed combustion of the HSP. The freeboard temperature was optimised in the MPFBC by a combination of injecting the HSP directly into the bed, utilising an increased bed height and introducing secondary air into the freeboard. This test work showed that most sulphur capture was achieved in the bed (~85%) and provided the design parameters for a 2 500 kg/h HSP fluidised bed incinerator with the capability of processing an additional 2 000 kg/h of an organic effluent stream.[3]

The installed plant consisted of a 21 m^2 bed with 80% of the air being directed to the bed for fluidisation and the remainder utilised as secondary air in the freeboard. The flue gas leaving the incinerator was passed through a waste heat boiler where 20 tonnes/h (at 20 bar) of steam was generated for use elsewhere in the plant. A baghouse was used to de-dust the flue gas before venting to atmosphere. The plant was commissioned in 1996 and has since been running successfully with SO_2 emissions to atmosphere of less than 400 ppm.

SULPHIDES REVISITED

Nickel Sulphide Roasting

Falconbridge, a major nickel producing company based in Canada, was interested in investigating a novel process for the production of nickel from nickel sulphide ore. The key steps in the process were the dead roasting of the ore in a fluidised bed followed by smelting of the calcine. The existence of a good synergy between Mintek and the CSIR, coupled with their proximity of each other meant that approximately fifty five tons of wet (20% moisture) nickel sulphide concentrate could be dead roasted in the MPFBC before carrying out smelting tests at Mintek.

A production campaign was undertaken in December 1997 in the MPFBC with the aim to produce a blended calcine product having a maximum sulphur content of 0.75%. Additional heating was required to achieve a bed temperature of 950°C. This was accomplished by direct firing of LPG through the bubble caps.

The elutriated fines were initially recovered from the cyclone and baghouse. However, the baghouse quickly proved to be unsuccessful due to the considerable acid attack of the filter bags. Since over 90% of the elutriated fines were reporting to the cyclone, the baghouse was bypassed for the remainder for the production run.

By far the biggest technical challenge was in the management of the off-gas cleanup. The off-gas had a high acid dew point resulting in severe corrosion of the Induced Draft (ID) fan impeller. This, coupled with the erosion of the impeller caused by dust carry over, resulted in the fan going out of balance frequently. The main reason was the low efficiency of the wet scrubber where little or no removal of SO_3 occurred. Various modifications and improvements were made in the scrubber design and scrubbing medium. Although this did help in reducing downstream corrosion of the ID impeller, additional measures still needed to be employed to protect the impeller directly. Hence various attempts were made to coat the impeller with different acid resistant coatings, but with no lasting degree of success.

Despite these problems the project was very successful in terms of the quality of the product. Approximately forty four tons of dead roasted calcine, having the required sulphur content, was produced on time for smelting at Mintek.

In spite of the technical success of this technology, it has subsequently been banked in favour of utilising nickel laterites for the associated environmental benefits.

PGM Sulphide Roasting

Building on the synergy between the CSIR and Mintek, a similar roast and smelt evaluative campaign was undertaken a year later. This work was conducted for Western Platinum. Approximately thirty tonnes of PGM sulphide concentrate were required to be dead-roasted in a fluidised bed before utilising the calcine to carry out tests on smelting and leaching treatment processes.

The sulphide roast was once again conducted in the MPFBC, however, this time due to the fine nature of the concentrate and the dry state in which it was fed, more than 90% of the product was collected in the baghouse. Additional heat was required to keep the bed at operating temperature. This was achieved by co-feeding coal.

The SO_2/SO_3 levels were also lower than those previously experienced during the nickel sulphide work and therefore a decision was made to keep the off-gas hot and dry by not utilising a scrubber. Hence the risk of acid attack of the ID fan was removed. The higher operating temperatures in the baghouse did however necessitate the use of Gore-Tex Teflon bags.

TITANIFEROUS MATERIALS

ISCOR Titania Slag

The first reductive roast utilising the MPFBC was on a titania slag. ISCOR identified a two phased roasting approach for investigation. This involved oxidative roasting of a titania slag, followed by reductive roasting. The aim was to affect the oxidation state of the iron selectively and so aid in downstream separation. Additionally, the aim was to optimise the roasting conditions for industrial scale processing.

The effect of various operating conditions and coal types was evaluated during the oxidative stage. Excess coal was used in the subsequent reducing roast. Supplementary nitrogen was also added to the fluidising air in order to maintain reducing conditions. The purpose was to reduce the Fe_2O_3 to FeO, while controlling the conditions so that titanium remained as TiO_2.

Titanium

Between 1998 and 2001 a considerable focus was placed on the fluidised bed processing of ilmenite. The need for a bench scale fluidised bed rig was identified in order to test small quantities of material over a range of operating parameters with relatively quick turnaround times. The rig consisted of an externally heated reaction tube (one inch ID) fitted with a gas distribution plate. A manifold was included enabling the mixing of various gases, for instance in the simulation of reducing atmospheres. Additionally, a feeder and extractor were added to the rig to enable continuous processing.

This rig was soon put through its paces in the reductive roasting of an ilmenite concentrate for the Process Research Division of Billiton SA. The objective was to determine the effect on the magnetic properties of the product calcine, with the aim in removal of chrome and traces of radioactive impurities.

Following this project, a host of similar ilmenite exploratory work was conducted on the bench scale rig over a two year period. These tests were performed under oxidative conditions with occasional dilution of the fluidising air with nitrogen in order to reduce the oxygen concentration. The aim was to affect the magnetic susceptibility of the ilmenite in order to facilitate the removal of high chromite particles to provide a suitable smelter feed.

Once optimal operating conditions were determined on the bench scale rig, a larger run was conducted on the MPFBC in 2000. After oxidative roasting the material was sent for magnetic separation and smelting. A due diligence study is currently being conducted on the project and all indications are that the project will most likely progress to industrial scale.

FORMATION OF THE FBCoE

In the late 1990's the CSIR and Kumba Resources identified and capitalised on a synergy in the fluidised bed treatment of minerals. In 2000 the two organisations joined forces in the establishment of the Fluidised Bed Centre of Excellence (FBCoE).

The existing MPFBC was moved to Kumba Resources' premises and underwent significant upgrading and automation. This rig now forms part of a range of facilities hosted by the FBCoE.

One particularly interesting project conducted by the FBCoE was for a consortium of Dutch paper mills. The aim was to process paper residue from the de-inking stage in order to produce a mineral product with significant application, amongst others, in the building industry. Considerable alterations were made to the new MPFBC in order to simulate operating conditions in an envisaged industrial scale facility. This included, amongst others, the installation of an electrical preheater in-line before the wind box and atomised liquid fuel injection nozzles (for support heat).

The test work was performed to confirm previous small scale test work results, optimise operating conditions, and provide the consortium with sufficient processed material for testing and marketing purposes. An industrial scale plant has since been constructed in The Netherlands and has successfully commenced operation.

OTHER APPLICATIONS

Apart from the applications mentioned in this paper, several other applications were investigated. These include:

- Reduction of MnO_2
- Pre-reduction of chromite
- Nitriding of titanium dioxide
- Production of char from anthracite and bituminous coal
- Barium sulphate reduction
- Manganese nitriding
- Chlorination of titanium nitride
- Coating of diamonds

CONCLUDING REMARKS

- Fluidised bed technology is robust with unique characteristics rendering it suitable to a host of different applications.
- Despite the advances made in fluidised bed modelling and simulation, in this kind of investigative work, there are always surprises necessitating the need for test work.
- There are great advantages to having access to a range of versatile test facilities, skills and expertise.
- Partnership with industry in solving real problems and developing processes greatly assists in building greater knowledge and expertise in the field.

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