Introduction

Oxidation of zinc concentrate in fluidized bed roasting

Zincor, a zinc refinery to the east of Johannesburg in South Africa. The process used is described in Part II of this paper. Historically, since fluidized beds have been used, air has been the oxidizing medium to convert ZnS to ZnO. In an attempt to increase roaster throughput Air Products and Chemicals, Inc. (APCI) were the first to introduce O₂ enrichment into the fluidized air in April 1983. Zincor followed suit in 1996 and use up to 26% O₂ enrichment (21% O₂ in air and 5% O₂ from a 99.5% O₂ source) to increase throughput capabilities. O₂ enrichment increases the rate of the oxidation reaction,

\[
\text{ZnS} + 1.5\text{O}_2 \rightarrow \text{ZnO} + \text{SO}_2
\]  

and therefore feed rates to the roasters can be increased. The enrichment allows the same total flow rate of fluidizing gas, and thus the loss of fines through entrainment is not increased.

O₂ enrichment allows roaster throughput to be increased, while the feed particle-size distribution can be maintained. If, however, a coarser feed particle size could be used, the flow rate of fluidizing air could be increased, also allowing an increased roaster throughput. This is feasible, as the fluidizing air supplies O₂ in excess of the stoichiometric requirements. One way of increasing the feed particle sizes is through full or partial pelletization of the feed. However, as will be shown later, larger particles require more time to convert to ZnO, and thus the maximum size of such pellets would have to be controlled. The resistance to attrition and breakdown of the pellets will also have to be maximized.

Transformation processes during roasting

The phase transformation processes occurring when zinc concentrate is oxidized have been well researched. Graydon and Kirk give a good description of the phase transformation process when zinc concentrate is roasted. FeS₂ phases in solid solution with ZnS oxidize first. The second stage involves a rise in the O₂ partial pressure at the reacting interface and the onset of oxidation of mainly ZnS to ZnO.
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which forms in solid solution with the remaining oxidized iron as Fe2+. Concurrently, some iron will diffuse from the Fe3O4 rim into the newly formed ZnO. Upon completion of the ZnS oxidation, the O2 partial pressure in the particle rises to the roaster atmosphere pressure, and the remaining Fe2+ in solid solution is oxidized to Fe3+. Simultaneously, the Fe2O3 rim with condensed ZnO is transformed into the refractory zinc ferrite, ZnFe2O4.

In the last stage of the transformation process, the Fe3+ that was formed reacts with ZnO to produce separate ZnFe2O4 and pure ZnO phases.

Chen and Dutrizac\(^\text{6}\) studied the mineralogical changes that occur when zinc concentrate is roasted. The calcine consisted of unreacted ZnS and uniform, porous, banded masses of (Zn,Fe)O or ZnO and ZnFe2O4. The surface of the particles contains a fine-grained uniform and porous mass of ZnO + ZnFe2O4. A dense (Zn,Fe)O phase appears between the sphalerite and ZnO + ZnFe2O4 layer. Also present are small pores attributed to the evolution of SO2 gas during roasting or differences in density of the sphalerite and the reaction products that form\(^\text{6}\).

Micro-pellet roasting in this study is expected to follow the same transformation processes. The micro-pellets are larger than the non-pelletized concentrate particles, and incomplete roasting with a remaining ZnS core could result. Should this occur, it would indicate that the particle residence time inside the roaster was insufficient\(^\text{1}\), if all other control variables (i.e. bed temperature, fluidizing air rate) are kept constant.

Variables affecting micro-pellet roasting

**ZnS pelletization**

Zinc concentrates were pelletized for a horizontal-type fluidized bed roaster as early as 1954\(^\text{7}\). Subsequently, numerous methods to pelletize zinc concentrates were successfully implemented\(^\text{1}\). The underlying purpose has always been to reduce fines, or to increase bed stability and to reduce entrainment.

Denoisieux et al.\(^\text{7}\) produced pellets using a granulating drum and controlled the size between 0.5 mm and 4 mm. The sulfur present as either a sulfide (denoted S/S) or a sulfate (denoted S/SO4\(^2-\)) in the roasted material is used to indicate how successfully ZnS was converted to ZnO. In the entrained particles, S/S and S/SO4\(^2-\) were 0.26% and 0.35% higher, respectively, than in particles in the bed overflow. The longer residence time of pellets in the roaster bed was given as respectively, than in particles in the bed overflow. The longer residence time of pellets in the roaster bed was given as respectively, than in particles in the bed overflow. The longer residence time of pellets in the roaster bed was given as respectively, than in particles in the bed overflow.

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**Figure 1—Design bed particle size distribution\(^\text{13}\)**

![Figure 1—Design bed particle size distribution\(^\text{13}\)](image-url)
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O₂. The pellet sizes varied from 4.1 mm to 16 mm. It took approximately 25 minutes to convert a 4.1 mm pellet. The time required for oxidation increases substantially with increasing pellet size; at 70 minutes the 16 mm pellet had not reached 60% conversion. Natesan and Philbrook16 also studied the roasting kinetics of various particle sizes of non-pelletized concentrate in a 4-inch laboratory scale fluidized roaster. Roasting was performed in a 1-inch bed depth on particles of 0.18 mm, 0.32 mm, and 1 mm in a 21% O₂ atmosphere. Consistent with their earlier study, it was found that the smaller particles are converted at a faster rate.

Micro-pellet production

The first step in the study was to determine the optimum conditions to produce micro-pellets that resist attrition during roasting. Three procedures were tested for the production of suitable micro-pellets:

1. Increasing the final moisture content
2. Use of a binder consisting of different ratios of impure ZnSO₄ solution and water. Impure ZnSO₄ is a product of the neutral leaching step of the roasted calcine. The ZnSO₄ concentration was varied between zero and 220 g/l.
3. Use of different pelletizer speed settings.

Micro-pellets were produced with the Loedige KM 300 ploughshare mixer. This pelletizer was chosen based on previous work performed at Zincor. The unit has a capacity of 8 t/h wet feed. It was installed in parallel to the existing concentrate feed system. The main shaft of the pelletizer, which conveys the concentrate from the inlet to the outlet, has two possible speed settings of 70 or 140 r/min. In addition, two chopper motors with a shaft and blades also have a combined two-speed setting of low and high speeds.

Samples were taken of the non-pelletized concentrate and of the produced micro-pellets. The samples were stored in sealed bags. The samples were used to determine the feed and product moisture content. The samples were weighed before being dried in a muffle furnace at 120°C for 24 hour. The dried sample weight was then determined.

Oxygen concentration

Cannon and Denbigh's17 study using ZnS crystals showed that an increase in the O₂ concentration resulted in an increase in the rate of the oxidation reaction. Sanchuan *et al.*13 also showed that the reaction rate of ZnS oxidation is proportional to the O₂ concentration at 750°C and 950°C. Natesan and Philbrook16,18 studied the effect of different O₂ levels in the fluidizing air on the reaction rates of non-pelletized and pelletized concentrate. In both instances, it was shown that increased levels of O₂ in the fluidizing air increases the rate of the ZnS oxidation reaction.

Current production rates at Zincor can be maintained by enhancing the reaction rates through O₂ enrichment. This is due to the short average residence times in industrial roasters.1 Residency times may be improved by micro-pelletization, thus possibly reducing the need for O₂ enrichment.

Experimental procedure

Micro-pellet strength tests

Using compressive strength tests or fall impact tests as used by previous investigators10,13 to test the strength of the micro-pellets would not be practical due to the micro-pellet's size, which might influence the accuracy of the results. Although the bulk of the unpelletized material was smaller than 75 μm, the only requirement was to know the fraction of fines below 500 μm (this is the largest particle that is entrained) and the fraction of coarse material above 1000 μm (this is the largest micro-pellet to be produced to ensure sufficient residence time for conversion). To determine if the strength of the micro-pellets would be sufficient to prevent excessive break-up and attrition the micro-pellets were roasted for 1 hour in an air atmosphere in a laboratory scale fluidized roaster controlled at 950°C. Size distributions were determined for particles before and after roasting. The difference in pelletized and unpelletized particle size distributions was used to determine the strength of the pelletized concentrate. Particle size analysis was performed with sieve sizes 75, 212, 300, 500, 850, 1000, 1400, 2360, 3150, and 4000 μm. The laboratory scale roaster set-up is shown in Figure 2.

A 20 mm porous-bottom silica glass reactor tube in a thermocouple-controlled vertical-tube furnace was used in all the laboratory tests, as shown in Figure 5.
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The reactor tube was placed inside an external silica holding tube with an entry point for the fluidizing gas. The holding tube and the reactor formed a seal at the necks of the two units. This configuration formed a sleeve between the holding tube and the reactor that allows the fluidizing gas to enter the porous bottom of the reactor. The required flow rate and ratio for the preferred fluidizing gas mixture were controlled with rotameters. Only air, O₂ or Ar were used during the tests.

The gas exiting the reactor was cooled by passing it through two glass cooling towers on the tube side. Cool pressurized air was passed through the cooling tower on the shell side. The cooled gas then passed through a resin water trap to ensure that the gas was dehydrated and so prevent sulphuric acid formation before entering the SO₂ gas analyser. Sulphuric acid will form if trace levels of SO₃ and moisture are present in the gas. The off-gas was then released to the atmosphere.

The Ultramat 6F infrared gas analyser, produced by Siemens®, was used to measure the percentage SO₂ (%/v/v) concentration. The analyser was calibrated by first performing a zero calibration with the oxidizing atmosphere for the specific test, and then with a 5% chemical grade SO₂ calibration gas. The roaster was set to the required temperature for the specific test and left to stabilize. Prior to each roasting test, the reactor was flushed with Ar for 1 minute to ensure an inert atmosphere. Data logging was started before Ar flushing. The micro-pellets were then placed inside the reactor and the required fluidizing atmosphere was set for the specific test. All tests were performed at a total gas flow rate of 7.6 L/min. This flow rate represents flow conditions present inside a Zincor industrial sized roaster and is equivalent to 500 Nm³/h/m². Each test was run until no more SO₂ was measured. The sample was then removed from the reactor and placed in a sealed polytop container for analysis.

The effects of micro-pellet size and O₂ enrichment

Tests were performed to determine the time required to roast different sizes of micro-pellets and to gain a better understanding of how micro-pellets would react inside a roaster. The size of the micro-pellets (1000 μm to 4750 μm) and O₂ enrichment (air and 23%, 24.5%, and 26% O₂) were varied. The micro-pellets used in the tests were produced with the pelletizer at the settings for optimal strength micro-pellets. All the tests were conducted at 950°C using 0.33 g of sample, and each test was done in triplicate. The extent of conversion of the roasted particles was determined by calculation from the original known sulfur content, the measured %SO₂ (%/v/v) and the gas flow rate.

The roasted particles were studied using scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDX) analysis to acquire semi-quantitative information about the samples. A JOEL JSM 6300 instrument was used. The samples were set in an epoxy resin and were polished through to the sample for analysis.

Results and discussion

Micro-pellet strength measurement

The results for (1) water only as a binder, (2) pelletizer settings, and (3) ZnSO₄ as a binder are discussed in the following section.

Extra test work to confirm repeatability was not possible due to the pelletizer being available to Zincor for only a limited time. However, comparing the trends for the different tests does provide sufficient confidence in the results. The samples used for the test work were taken from a continuous process, which should mitigate some of the variability inherent in batch processes.

Water as a binder

The first phase of the tests was to determine the effect of increasing the moisture content of the micro-pellets. The results are shown in Figure 4. The data points represent single experimental runs.

The content of ~500 μm particles was reduced from 87% in the unpelletized feed to 51% through micro-pelletization with 9.5% moisture. The results for unroasted particles show that increasing amount of binder decrease the percentage of fines (~500 μm), with concomitant increases in the coarser fractions. This confirms that moisture plays an important role in forming bigger particles, consistent with findings by previous investigators, as discussed above. Most of the agglomerated particles fall in the size fraction +1000 μm. The trends seen in particle size as a function of binder content for unroasted particles also largely unchanged by the roasting procedure. There is evidence of attrition, but there are still net gains obtained by increasing the amount of binder. Beyond about 7% moisture, an increasing amount of binder does not seem to result in particles that are more resistant to attrition, as the trends for unroasted and roasted particles are more or less parallel.

Ideally, with an optimized micro-pelletization process, the particles would all be in the +500 –1000 μm size range when leaving the roaster. Roasting of the +500 –1000 μm particles resulted in attrition, decreasing the content of this size fraction from 28% to 14% at all the moisture levels tested, except at 6.1% and 6.6% moisture.

Producing roasted micro-pellets in this desired size range is as important as not producing excessive amounts of +1000 μm roasted pellets. Attritioning of the +1000 μm micro-pellets during roasting is desirable therefore and beneficial to the overall efficiency of the process by lowering the risk of producing low quality calcine.

![Figure 4—Results using water as a binder](image-url)
At 6.1% moisture, agglomeration of the particles occurred during roasting, which reduced the –500 μm particle content from 78% to 69%.

The particles were roasted for only 1 hour and it is reasonable to assume that further particle attrition will occur during longer periods of roasting. Particles in the bed have a calculated average residence time of 4 hours, and longer roasting time might lower the fraction of +1000 μm particles even further. This will be important, recalling that Zincor controls the +1000 μm particles to around 20% to prevent defluidization of the bed.

**Pelletizer settings**

Subsequently, mechanical settings on the pelletizer were varied for micro-pellets containing levels of moisture above 9%. The results in Table 1 compare unroasted with roasted micro-pellets.

It was found that an appreciable decrease in the amount of fines (–500 μm) resulted from the combination of a high transfer shaft speed and a low chopper speed. The product micro-pellets were mainly in the +1000 μm size fraction. These trends hold for the unroasted pellets and the pellets after the attrition caused by roasting. Most of the loss due to attrition was from the +500–1000 μm size fraction. The high shaft speed and a low chopper speed resulted in the least degradation by attritioning. Based on these results, a high shaft speed and low chopper speed setting were used to produce the micro-pellets required for the test work.

**Impure ZnSO₄ electrolyte as a binder**

Clear trends in the extent of agglomeration, or the extent of attrition through roasting, were not found. Therefore ZnSO₄ solution was not used as a binder to produce micro-pellets.

The particle size distribution for micro-pellets produced at optimal conditions compared with non-pelletized feed is shown in Figure 5.

The optimal conditions for micro-pellet production with the pelletizer are moisture levels of 9.5% to 10.5%, a high shaft speed and a low chopper speed. These trends hold for the unroasted pellets and the pellets after the attrition caused by roasting. Most of the loss due to attrition was from the +500–1000 μm size fraction. The high shaft speed and a low chopper speed resulted in the least degradation by attritioning. Based on these results, a high shaft speed and low chopper speed setting were used to produce the micro-pellets required for the test work.

**Laboratory-scale micro-pellet roasting**

**Micro-pellet size**

The results for roasting variously sized micro-pellets in air are shown in Figure 6.

Unexpectedly, the +1400–2360 μm micro-pellets reached full conversion before the +1000–1400 μm micro-pellets. This can be explained by particle attrition and degradation of the micro-pellets. These +1400–2360 μm pellets, for an inexplicable reason, were found to be more prone to degradation and attrition.

The +3150–4750 μm sized micro-pellets required approximately 17 minutes for full conversion. The result compares reasonably with the Natesan and Philbrook study; a single 4.1 mm particle required approximately 25 minute to be converted. Table II shows the measured conversion times and calculated residence times for various particle sizes. The particle sizes used for the residence time calculations differ slightly from the sizes used in the test work, as shown.

The reaction times shown in these tests are far less than the calculated residence times inside a Zincor roaster, or the measured retention times as found by Spira. Therefore,

![Comparison of the particle-size distributions for blended unpelletized feed concentrate and the best result achieved for roasted vs. unroasted micro-pellets](image)

**Figure 5**

**Conversion for various sized micro-pellets (+1000–1400 μm, +1400–2360 μm, +2360–3150 μm and +3150–4750 μm)**

![Conversion for various sized micro-pellets](image)

**Figure 6**

---

**Table 1**

<table>
<thead>
<tr>
<th>Mass fraction of particles retained at different pelletizer settings to optimize the pelletization process</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chopper speed</td>
</tr>
<tr>
<td>----------------</td>
</tr>
<tr>
<td>Transfer shaft speed (r/min)</td>
</tr>
<tr>
<td>+500 μm</td>
</tr>
<tr>
<td>+1000 μm</td>
</tr>
<tr>
<td>+1000 μm</td>
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</tbody>
</table>

![Table 1](image)

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Oxygen enrichment

The results for the roasting of +1000–1400 μm pellets in O₂-enriched fluidizing air are shown in Figure 9.

The results show the influence that O₂ enrichment has on the reaction rate. It took approximately 8 minutes to roast the micro-pellet to completion in air, compared with approximately 3.5 minutes in 26% O₂. The influence that O₂ enrichment has on fluidized bed roasting is well known and is used at Zincor.

SEM images of the +1000–1400 μm micro-pellets roasted in O₂-enriched air are shown in Figure 10.

Both particles are from the same test. The particle in Figure 10A once again exhibits a dense ZnO core surrounded by a ZnO + ZnFe₂O₄ outer rim. This is a typical roasted particle and consistent with the depiction of Chen and Dutrizac [305] of their partially roasted zinc concentrate – phase 2 (ZnO) and phase 3 ZnO + ZnFe₂O₄. In general, the morphologies of the roasted particles are similar to those found by Graydon and Kirk [203] and by Chen and Dutrizac [305].

<table>
<thead>
<tr>
<th>Particle size (μm)</th>
<th>Time for complete conversion in air (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1400 (1000)</td>
<td>8 (209)</td>
</tr>
<tr>
<td>2360 (2000)</td>
<td>7 (234)</td>
</tr>
<tr>
<td>3150 (3350)</td>
<td>13 (238)</td>
</tr>
<tr>
<td>4750 (4750)</td>
<td>17 (239)</td>
</tr>
</tbody>
</table>

Table II

Reaction time and (residence time) in a Zincor roaster

Figure 7—SEM image of a concentrate micro-pellet containing a range of sub-particles bonded together. 1 – ZnS, 2 – FeS₂, 3 – zincian and plumbian dolomite (0.5% Zn; 0.4-0.8% Pb), and 4 – PbS

Figure 8—SEM images of +1400–2360 μm and +2360–3150 μm roasted micro-pellets. 1 – ZnO, 2 – ZnO, and ZnFe₂O₄

Figure 9—Fractional conversion for +1000–1400 μm sized micro-pellets in O₂-enriched fluidizing air

Figure 10—SEM images of +1000–1400 μm micro-pellets roasted in 23% O₂ enriched fluidizing air. 1 – ZnO and 2 – ZnO and ZnFe₂O₄

using micro-pellets should not reduce the specific calcine production rates achieved with O₂ enrichment.

SEM images of an unroasted micro-pellet and roasted micro-pellets are shown in Figures 7 and 8.

The SEM image in Figure 7 shows a typical concentrate micro-pellet that consists of various small mineral particles randomly bonded together. Zn(Fe)S particles are shown as the light to darker grey particles that constitute the bulk of the particles.

Figure 8 shows that the micro-pellets were completely oxidized. The +1400–2360 μm micro-pellet shows a typical image of a roasted particle consisting of the more porous ZnO + ZnFe₂O₄, which passed through the full transformation process. Pores that may be associated with the evolution of SO₂ during roasting are visible. The +2360–3150 μm micro-pellet is distinctly different, with dense ZnO in the middle surrounded by a ZnO + ZnFe₂O₄ outer rim. This is a typical roasted particle and consistent with the depiction of Chen and Dutrizac of their partially roasted zinc concentrate – phase 2 (ZnFe₂O₄) and phase 3 ZnO + ZnFe₂O₄. In general, the morphologies of the roasted particles are similar to those found by Graydon and Kirk and by Chen and Dutrizac.
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by a porous ZnO + ZnFe₂O₄ mass. The particle in figure 10B consists of only the porous ZnO + ZnFe₂O₄ mass. The general morphologies of the particles roasted in O₂-enriched air are similar to those of particles roasted in air only, as discussed in the previous section.

Discussion and conclusions

Production of micro-pellets and strength measurement

The use of micro-pellets produced with the pelletizer was effective in reducing entrainable particles from 87% to 31%. Water was an effective binder at moisture levels of 9.5%. The study was inconclusive regarding ZnSO₄ solution as a binder to increase micro-pellet strength. The strength of +1000 – 4750 μm micro-pellets occurs during roasting, and production of the micro-pellets in this production conditions for this technique.

The speed and low chopper speed setting gave the optimized effective in reducing entrainable particles from 87% to 31%. The general mass. The particle in figure 10B consists of only the porous ZnO + ZnFe₂O₄ mass. The general morphologies of the particles roasted in O₂-enriched air are similar to those of particles roasted in air only, as discussed in the previous section.

Using micro-pellets between +500 – 1000 μm is favoured for the Zinorco roasting process. Attritioning and particle degradation of the +1000 – 4750 μm micro-pellets occurs during roasting, and production of the micro-pellets in this size range can thus be accommodated to a limited extent².

Laboratory-scale micro-pellet roasting

Laboratory-scale roasting in this study showed that the +3150 – 4750 μm sized micro-pellets required approximately 17 minutes to convert fully. The calculated average residence time for particles of this size in the Zinorco roasters is 4 hours³. Likewise, 500 μm (2.5 hours) and 1000 μm (2.5 hours) particle residence times far exceed the required reaction times. This indicated that the introduction of micro-pelletized feed would be feasible.

Roasting micro-pellets in O₂-enriched fluidizing air increased the reaction rate. In air the +1000 – 1400 μm pellets required 6 minutes to convert, while in 26% O₂-enriched air they required only 3.5 minutes.

The general morphologies of the roasted particles are similar to those found by Graydon and Kirk⁵ and Chen and Dutrizac⁶. Some roasted micro-pelletized samples contained a dense ZnO kernel and a porous ZnO + ZnFe₂O₄ outer rim mass.

Acknowledgements

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References


