

Chapter 3: Research design & methodology

In chapter 2 I reviewed the current body of knowledge on ilmenite roasting and concluded the chapter with a discussion on how I derived my test program from this scholarship review. In chapter 3 I discuss the design and methodology of this test program in more detail by first identifying the key concepts and variables, then stating how these key variables were measured. Sample preparation and data collection are discussed. The chapter concludes with statements on data capturing methods. Tasks in chapter 3 were as follows:

- 3.1 Formulate hypotheses and overall experimental plan
- 3.2 Source and prepare crude ilmenite and LSR
- 3.3 Characterize crude ilmenite, LSR and chromite in LSR
- 3.4 Determine optimum roasting conditions for LSR and compare with crude ilmenite
- 3.5 Prepare fractionation curves for crude ilmenite and LSR before and after roasting at optimum roasting conditions
- 3.6 Source and prepare chromite
- 3.7 Characterize chromite
- 3.8 Data capturing

3.1 *Formulation of hypotheses and overall experimental plan*

In chapter 2 I decided to test the following hypotheses based on the current body of knowledge on ilmenite roasting for chromite removal:

"It is possible to produce an ilmenite product suitable for ilmenite smelting by subjecting LSR to roasting and subsequent magnetic separation, using the roasting conditions recommended for crude ilmenite by Nell and Den Hoed (1997) or Bergeron and Prest (1974)"

and

"The magnetic susceptibility of chromite remains constant during magnetizing roasting of an ilmenite concentrate under the oxidizing conditions reported by Nell and Den Hoed (1997)."

To test the first hypothesis the key concepts are that once a sample of the LSR⁵ described by Beukes and Van Niekerk (1999) was prepared this sample would have to be characterized and the results compared with that of the material described in literature. I would then determine the optimum roasting conditions for the LSR by conducting small-scale batch tests under conditions similar to those published by Nell and Den Hoed (1997) and Bergeron and Prest (1974). 'Optimum roasting conditions' were defined as the conditions where the maximum increase in magnetic susceptibility occurs in the roasted sample. Once the optimum roasting conditions have been determined a larger sample would be roasted at these conditions and a separability curve constructed to determine to which extent the separation of ilmenite from chromite would be possible. These results would also be compared with those published in literature for Southern African East Coast Deposits.

To test the second hypothesis the key concepts are to determine the chemical composition of the chromite in the LSR in order to be able to source a natural chromite with a similar composition. Then the sourced chromite would have to be prepared, characterized, roasted under the conditions used for the LSR, and fractionated at unroasted and optimum roasting condition.

From the literature survey in chapter 2 the key variables in the study were the oxidizing roasting conditions - temperature and pO_2 of the roasting atmosphere - and the resultant change in the magnetic susceptibility of the ilmenite, due to crystal chemical changes in the ilmenite particles themselves. To characterize the LSR before, and in some cases after, roasting:

- The chemical composition of the LSR was important because the client specified the chemical composition for the final product.
- The mineralogical composition of the LSR was important to determine what mineral components were included in the LSR and how these components would influence the roasting or magnetic separation process.

⁵ Low susceptibility rejects

- The crystal chemical composition of the ilmenite in the LSR was important because it would influence the choice of pO_2 of the roasting atmosphere.

For the chromite only the chemical composition was determined.

To measure and/or control the key variables a test program was developed that consisted of the major steps defined in figure 3.1.

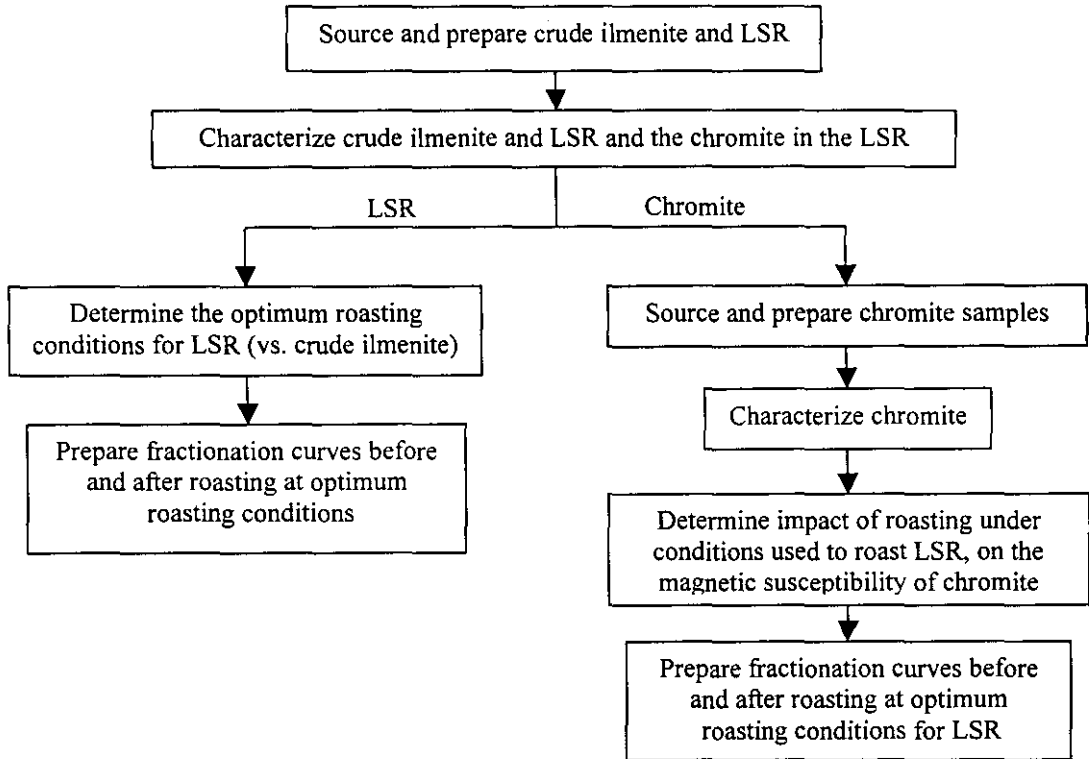


Figure 3.1: Overall experimental plan for this study.

Firstly gross samples of crude ilmenite and LSR were prepared. Then these gross samples were characterized. The optimum oxidizing roasting conditions for LSR were determined and lastly larger samples of LSR and crude ilmenite were roasted at the optimum roasting conditions to compare their results with each other. The chromite in the LSR was characterized and then the natural chromite sample sourced, prepared and characterized. Then the impact of roasting on the magnetic susceptibility of the chromite, when roasted under the conditions used for LSR, was determined. Lastly fractionation curves were prepared for both unroasted and roasted chromite primarily to determine whether or not the bimodality in the chromite observed in crude ilmenite was also observed in this chromite sample.

Details of these major steps follow in the rest of this chapter.

All test work were conducted at Kumba R&D in Pretoria West, South Africa except for magnetic susceptibility measurements, QEMSEM and QEMscan, some of the WHIMS test work and preparation of the natural chromite sample. Magnetic susceptibility measurements were conducted at Geotron in Potchefstroom, QEMSEM and QEMscan at Mintek in Randburg, WHIMS tests at Readings, Australia and chromite sample preparation at the University of Pretoria.

3.2 Sourcing and preparation of crude ilmenite and LSR

Heavy Minerals Concentrate (HMC) for the determination of optimum roasting conditions for LSR was produced during a pilot plant trial in South Africa on a typical Southern African East Coast Heavy Minerals Deposit. Crude ilmenite was produced in Australia from the HMC with WHIMS machines⁶.

⁶ Details of settings not available

Back in South Africa a 380-mm diameter commercially available full-scale DMDS was used to remove HSR at 2600 Gauss. The feed rate was 4.5 tons per hour per meter (tph/m) and the rotation speed 65 rpm for the first pass and 55 rpm for the second and third. A 610-mm diameter commercially available full-scale DMDS was used to remove LSR at 6500 Gauss. The feed rate was 8.85 tph/m and the rotation speed 70 rpm for the first pass and 85 rpm for the second. All three fractions were riffled to produce two representative samples each. All of this work was conducted by others for detail flow sheet design (not part of the scope of this thesis). After the abovementioned test work the crude ilmenite was reconstituted for *this* study by blending the one product and one LSR fraction in the ratios that they were produced. Both the crude ilmenite and the LSR samples were then split with a rotary splitter into representative 40g samples. Preparation of these samples is illustrated in figure 3.2.

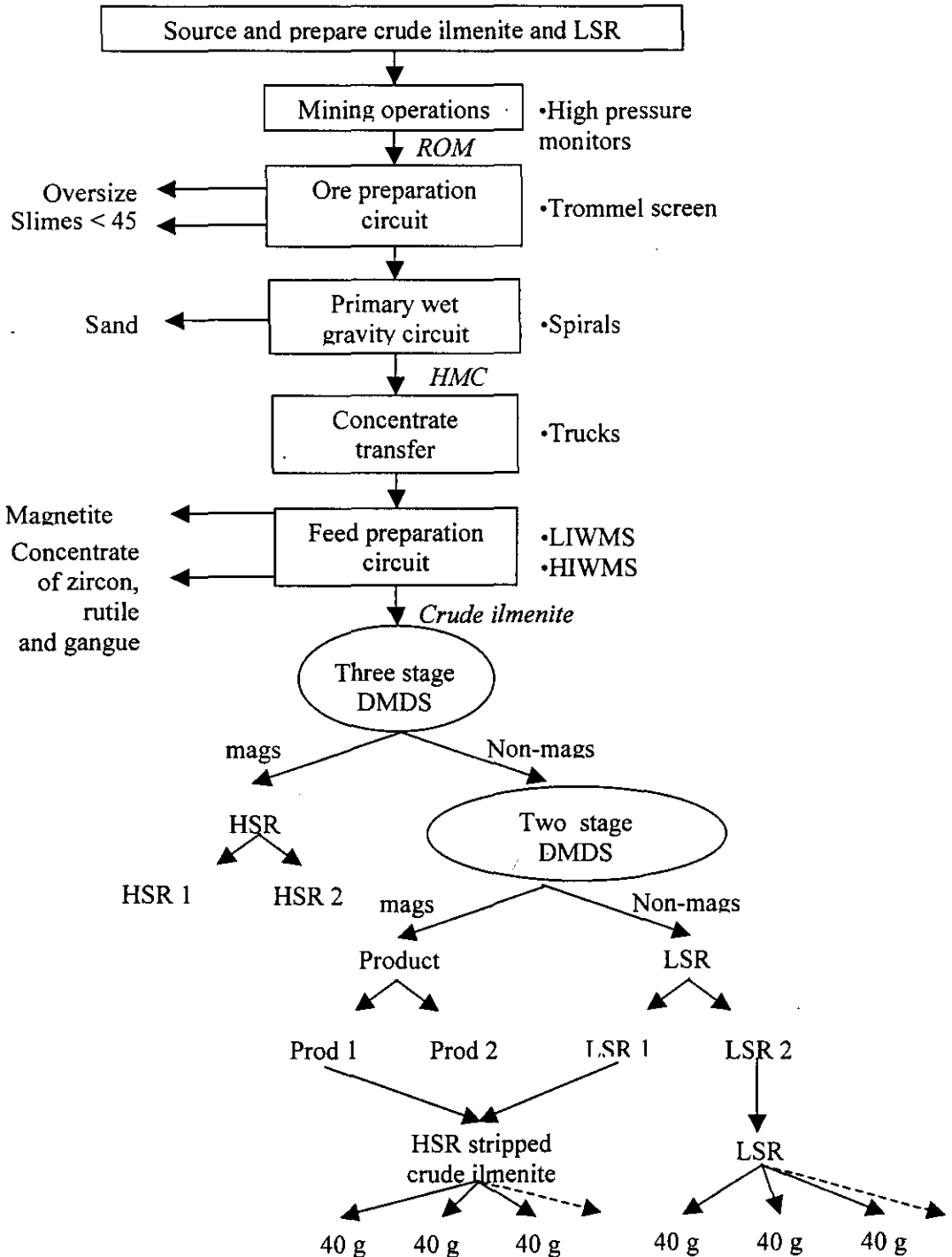


Figure 3.2: Flow sheet for the sourcing and preparation of crude ilmenite and LSR for this study.

One of the difficulties experienced at the stage was to obtain suitable samples to conduct the roasting tests on. The size of the 40g samples was therefore determined by the amount of material available on the one hand, and the minimum size sample that can be roasted in the batch fluidized bed reactor (in which these studies were to be conducted) on the other.

HMC for fractionation after optimum roasting was produced during another pilot plant trial on a typical Southern African East Coast Heavy Minerals Deposit. The crude ilmenite was prepared on commercially available full-scale WHIMS⁷ equipment. The LSR was prepared in a similar fashion as described above, with 250-g samples instead of 40-g samples as final result. Although the samples used in the fractionation at optimum roasting conditions were prepared according to the same flow sheet as the material used in the tests to determine the optimum roasting conditions, the results of the two types of experiments were not directly comparable. The reasons are that the locations within the heavy minerals deposit, from which these samples were prepared, differed, as did the WHIMS equipment used.

Personnel from a heavy minerals producer on the Southern African East Coast prepared the two HMC concentrates, one in 1995 and the other in 1999. All the sample preparation work to determine the optimum roasting conditions, from receiving the HMC onwards, was conducted by research personnel at the pilot plant of ISCOR R&D⁸ in Pretoria West in 1999. Sample masses were determined with laboratory type scales and recorded manually onto a log sheet.

Crude ilmenite, to fractionate after roasting at optimum roasting conditions, was produced by personnel from a heavy minerals producer on the Southern African East Coast at Readings in Australia in 2000. The rest of the sample preparation work, from receiving the crude ilmenite onwards, was conducted by research personnel at the pilot plant of ISCOR R&D⁹ in Pretoria West in 2001.

3.3 Characterization of crude ilmenite, LSR and chromite in LSR

The following chemical analyses were conducted on crude ilmenite and LSR:

- XRF-analysis with an ARL9400 sequential XRF analyzer to determine the chemical composition *quantitatively* - refer to figure 3.3;
- Titration to determine the Fe²⁺ and Fe³⁺ content *quantitatively*.

The following chemical analyses were conducted on the chromite in the LSR:

- Energy dispersive X-ray analyses (EDX) with a JEOL Low Vacuum 58100 Scanning Electron Microscope.
- Wavelength dispersive spectrometry (WDS) with a JEOL super probe 733.

The following mineralogical analyses were conducted on crude ilmenite and LSR:

- XRD to determine the mineralogical composition *qualitatively*;
- Reflected light microscopy to describe mineralogical properties of the samples with special reference to textural and weathering properties of the ilmenite *qualitatively*;
- Micro-analyses to investigate the chemical composition of unaltered mineral grains and alteration products *qualitatively*;
- Particle counting (using optical microscope images) to determine the textural properties of the minerals *semi-quantitatively*.
- QEMSEM, using a Leica Q 600 image analyser (IA) integrated with a LEO S440 scanning-electron microscope (SEM) and an ISIS energy-dispersive X-ray analysis system (EDX), to determine the modal¹⁰ composition of each sample *qualitatively*.
- QEMscan, using a Leica Q 600 image analyser (IA) integrated with a LEO S440 scanning-electron microscope (SEM) and an ISIS energy-dispersive X-ray analysis system (EDX), to determine the degree of liberation in which iron oxides occur in the samples *qualitatively*.

⁷ Details of settings not available

⁸ Also referred to as ITEC or Kumba Technology

⁹ Also referred to as ITEC or Kumba Technology

¹⁰ Composition of a rock or mineral stream in terms of the relative amounts of minerals present

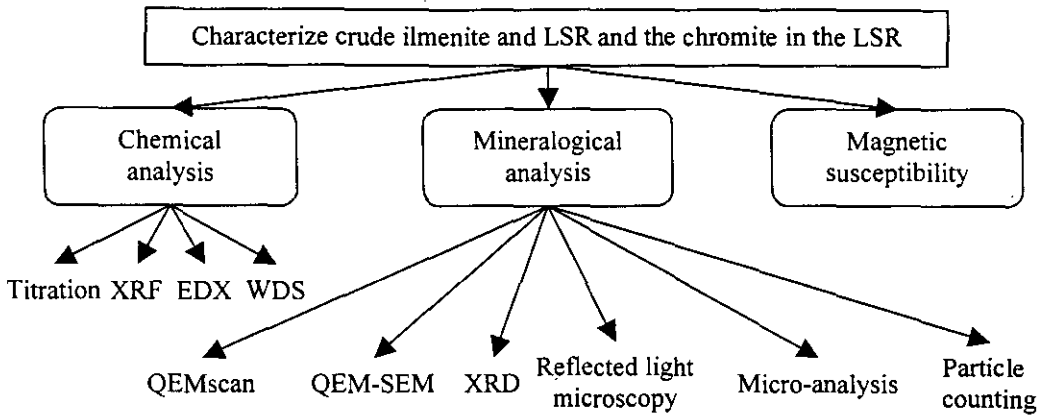


Figure 3.3: Flow sheet for the characterization of crude ilmenite and LSR for this study.

When conducting QEMSEM analysis on mineral samples these samples were divided into size fractions to minimize segregation during preparation and to improve mixing with the graphite matrix. Size fractions for screening were selected on the basis of the particle size distribution of the sample. 106 microns would be typical for beach sands, which have a very narrow particle size distribution between about 75 microns and 300 microns (Nell 2002). The as received samples of crude ilmenite and LSR were screened into two size fractions: +106 μm and -106 μm . Polished sections of each sample were prepared. Over four thousand grains in each sample - 2000 per size fraction - were classified according to their mineral composition. Their mineral composition was based on their chemical composition determined during QEMSEM analysis by raw EDX counts. The definitions used for each phase were indicated in table 3.1. Research personnel at Mintek in Randburg conducted the QEMSEM analysis in 1999.

Table 3.1: Definitions used to classify different grains as different minerals based on the occurrence of elements in raw EDX counts, or QEMscan* results, on a polished sample of crude ilmenite or LSR.

Phase	Definition
Ilmenite*	EDX spectrum indicates Fe and Ti
Fe-oxide*	EDX spectrum indicates Fe only
Chromite	Classified on major elements present i.e. Cr and Fe (Coetzee and Coetzee 1992)
Zircon	Classified on major elements present i.e. Zr and Si
Rutile*	EDX spectrum indicates Ti only
Siliceous Leucoxene*	EDX spectrum indicates Fe, Ti and Si
Quartz*	Classified on major elements present i.e. Si only (Coetzee and De Villiers 1992)
Other silicates*	Classified on major elements present i.e. Si and other elements (Coetzee and De Villiers 1992)
Monazite	Classified on major elements present i.e. Ce and Th (Von Backström 1992)
Sulphide	Classified on major elements present i.e. S (Hammerbeck 1992a)

To obtain more information on the degree of liberation within grains each sample was subjected to QEMscan analysis where EDX analysis was conducted along a grid pattern with a spacing of 4 μm . A very short counting time, in the order of 13 milliseconds (Nell, 2002), was used and therefore only the major elements present were detected. Light elements such as O and C were not considered. Definitions for minerals were as in table 3.1. Over one thousand grains in each sample - 500 per size fraction - were analysed. Research personnel at Mintek in Randburg conducted the QEMSEM analysis in 1999.

The magnetic susceptibility of all samples was determined with a KLY-2 Kappabridge (Agico, Brno in the Czech Republic). Results were reported as specific magnetic susceptibility (cm^3/g) – refer to

chapter 2 – utilizing a bulk density of 2.4 g/cm³. Nell and Den Hoed (1997) reported their results in a similar fashion. Research personnel from the pilot plant of Kumba R&D conducted the analysis at Geotron in Potchefstroom in 2000.

3.4 *Determination of the optimum roasting conditions for LSR and comparison with crude ilmenite*

To determine the optimum roasting conditions for LSR tests were conducted on 40g samples as per figure 3.4.

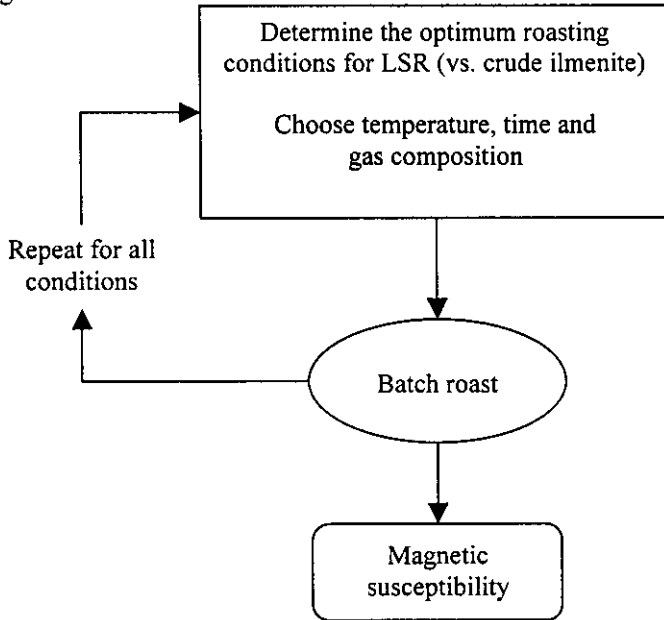


Figure 3.4: Flow sheet for the determination of optimum roasting conditions for LSR.

Batch-roasting tests were conducted in a custom made experimental set-up (refer to figure 3.5 for details). The set-up consisted of a quartz glass tube with a diameter of 30 mm and a length of 400 mm inside a tube furnace. An inner tube with a diameter of 25 mm was placed inside the outer tube. The bottom of the inner tube consisted of porous sintered glass disc. The sample was placed in the inner tube on the sintered disc.

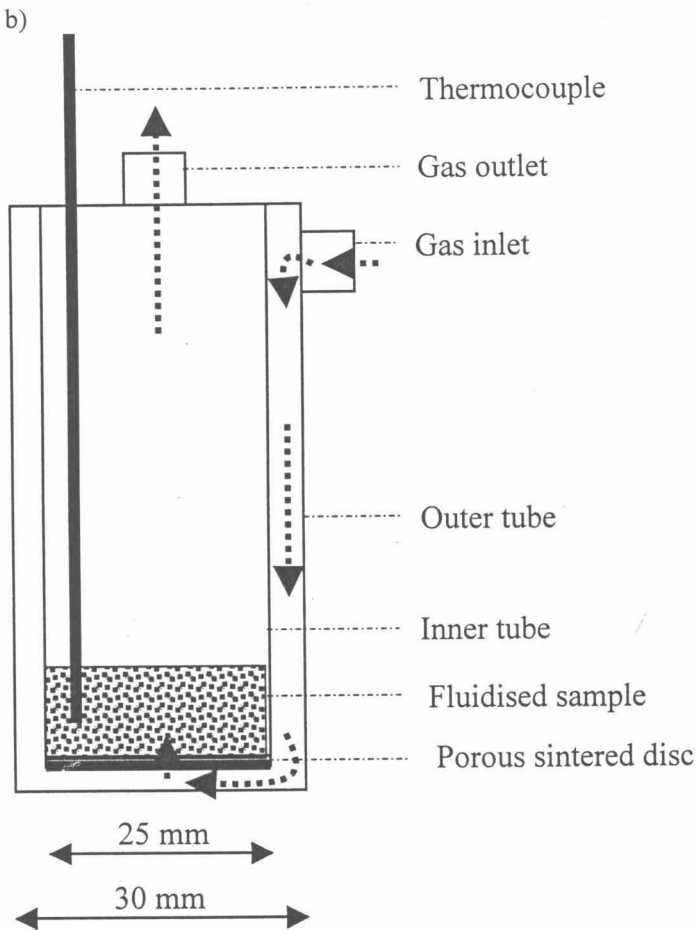
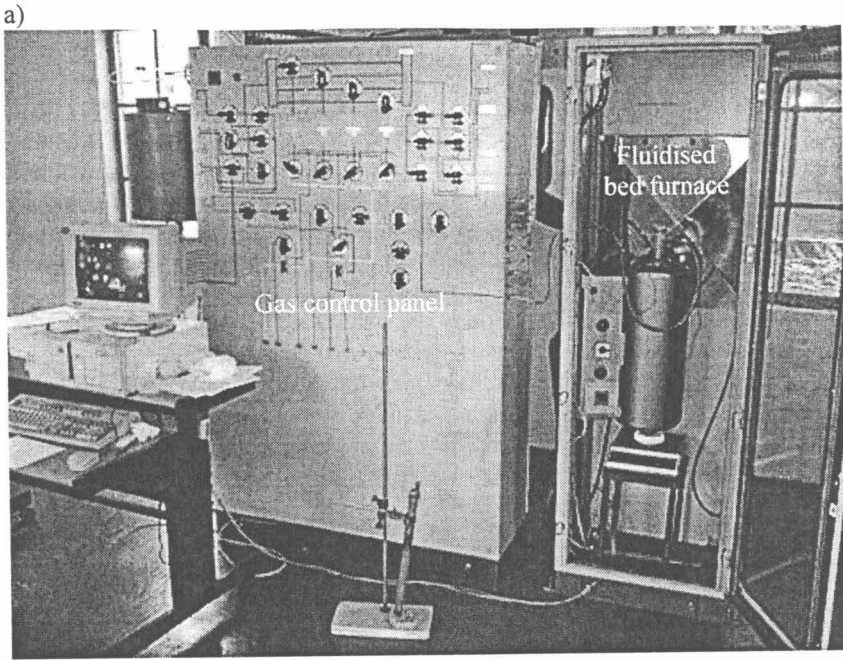


Figure 3.5: a) Experimental set-up for the fluidised bed roaster b) schematic layout of the quartz glass inner and outer tube arrangement for the fluidised bed roaster.

The 40g sample was placed in the reaction tube and the reaction tube was then placed in a pre-heated furnace. The required atmosphere was then introduced from gas cylinders to the outer tube from where it passed through the sintered disc into the inner tube. This allowed the sample to be fluidised. A thermocouple in a silica sheath was also placed in the inner tube to measure the sample temperature

during the experiment. The furnace temperature was initially set to $\sim 100^{\circ}\text{C}$ below the required temperature.

Argon at a flow rate of 1 litre per minute was passed through the reactor once it was placed in the furnace. The sample was then heated to the reaction temperature over a period of 30 minutes. Once the required temperature was reached, the experiment was started through the introduction of the required atmosphere. The sample temperature was continuously monitored for the duration of the experiment. After the required reaction time argon was once again introduced to the reactor and the sample was then removed from the furnace and allowed to cool down. The roasted samples were subjected to various analytical procedures. The magnetic susceptibility, the Fe(II) and Fe(III) content of the samples and the complete chemical analyses of the samples were determined by XRF analysis.

Roasting tests were conducted at temperatures ranging from $700\text{--}850^{\circ}\text{C}$ for residence times of 5-40 minutes primarily in air but also in a mixture of 50 per cent air and 50 per cent CO_2 gas. For comparison purposes tests were also conducted on crude ilmenite at 750°C and 800°C for periods ranging from 5 minutes to 40 minutes in air. Details of the roasting experimental plan are shown in table 3.2.

Table 3.2: Roasting conditions utilized for the determination of optimum roasting conditions for LSR.

Time intervals [min]	0; 5; 10; 20; 30; 40
Temperatures [$^{\circ}\text{C}$]	700; 750; 800; 850
Atmosphere	Air; 50:50 Air: CO_2

At least two experiments were conducted for each condition. The difference between similar experiments was within 5 per cent, based on the observed increase in magnetic susceptibility. Research personnel from ISCOR R&D conducted the tests at the pilot plant in Pretoria West in 2000.

3.5 Preparation of fractionation curves for crude ilmenite and LSR before and after roasting at optimum roasting conditions

To prepare separability curves for roasted LSR and roasted crude ilmenite tests were conducted on 250g samples as per figure 3.6. For comparison purposes separability diagrams for unroasted LSR and crude ilmenite were prepared in a similar fashion, excluding the roasting step.

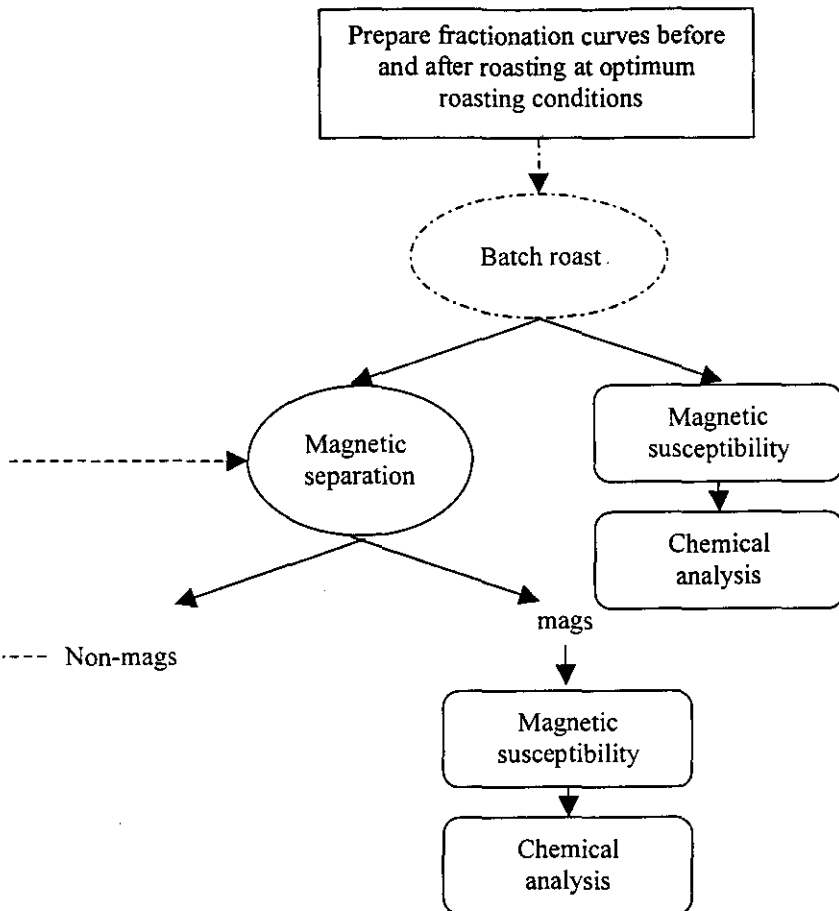


Figure 3.6: Flow sheet for the preparation of fractionation curves for crude ilmenite and LSR before and after roasting

After the optimum roasting conditions for LSR were determined, bulk samples of LSR and crude ilmenite was roasted in a custom made, Linn-type rotary furnace (internal diameter 150 mm and length 540 mm) at 800°C for 10 minutes; the furnace was rotated at 3 rpm. The externally heated furnace reactor was preheated to an operating temperature of 800°C whilst being rotated. On reaching the temperature, an oxidising gas was introduced at 6 liters per minute. The sample was loaded into the furnace reactor and roasted for 10 minutes. After 10 min the sample was cooled under rapidly flowing air and then discharged from the reactor. The roasted 250g sample was then split into two samples of 200g and 50g. The 50g sample was used for analysis of the roasted material (the magnetic susceptibility and the complete chemical analyses by XRF). The 200g sample was used for magnetic separation at various magnetic field strengths, and subsequent characterization of each fraction.

A Frantz barrier magnetic separator was used for fractionation. A vibrator feeder was used to feed a single layer of particles past the magnet. The angle of feed lane was kept at 25°. Nell & den Hoed (1997) used the same instrument settings during their study. For both material types splitting was conducted from a lower setting up to a higher setting. During fractionation a multimeter was connected in series to correlate the magnetic field strength – measured with a Gauss Meter - with the current (Ampere) settings. The following current settings were used:- 10mA, 20mA, 30mA, 40mA, 50mA, 100mA, 150mA, 200mA, 250mA, 300mA, 350mA, 400mA and 450mA. The correlation table between the field strength and current can be seen in table 3.3.

Table 3.3: Correlation table for current (mA) and magnetic field strength (Gauss) readings.

CURRENT (MA)	10	20	30	40	50	100	150	200	250	300	350	400	450
Magnetic field strength (Gauss)	250	384	481	584	751	1375	2150	2735	3450	4050	4650	5550	6210

Each fraction was analysed to determine the distribution of chemical components and the magnetic susceptibility. The magnetic susceptibility was determined as described elsewhere and the chemical analyses of the samples were determined by XRF.

To confirm the results of roasting at 800°C for 10 minutes samples of crude ilmenite and LSR were roasted in a similar fashion at another optimum roasting condition: 750°C for 20 minutes. Once again both samples were fractionated in a similar fashion at various magnetic field strengths and each fraction was chemically analysed in a similar fashion to determine the distribution of the components in each fraction. Research personnel from ISCOR R&D conducted the tests at the pilot plant in Pretoria West in 2000.

3.6 Sourcing and preparation of chromite

A natural chromite sample was sourced from rock in the UG1 layer in the western limb of the Bushveld Igneous Complex, South Africa. The gross sample was prepared from this rock by crushing it (by, in sequence a jaw crusher, a gyratory crusher, and a laboratory rod mill) to a top size of 850 µm, screening with a laboratory sieve at 106 µm, and finally by performing magnetic separation with a Readings magnetic separator (set to reject the non-chromite gangue). The magnetic fraction was used as the gross sample for this study. In figure 3.7 the flow sheet for this part of the study was indicated. The sample was sourced by academic staff and preparation work conducted by a post graduate student at the University of Pretoria in 2001.

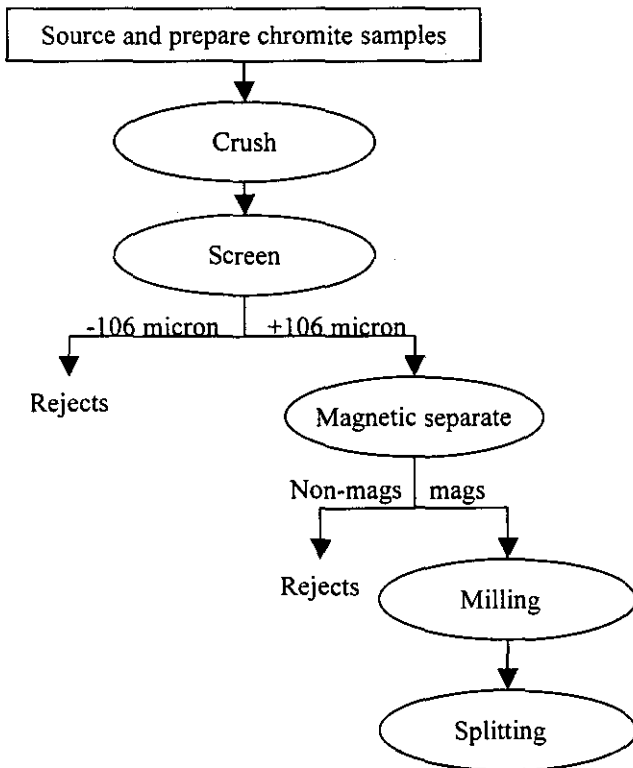


Figure 3.7: Flow sheet for the sourcing and preparation of chromite for this study.

3.7 Characterization of chromite

As indicated in figure 3.8 the gross sample was characterized by determining its chemical composition, magnetic susceptibility and size distribution. The chemical composition was determined with WDS analysis utilizing an ARL SEMQ microprobe. The magnetic susceptibility was determined with a Barrington MS2 magnetic susceptibility meter. The size distribution was determined with a Malvern size analyzer. Secondary samples of the chromite were prepared by milling with a Bond ball mill, determining the size distribution with a Malvern size analyzer and splitting with a rotary splitter.

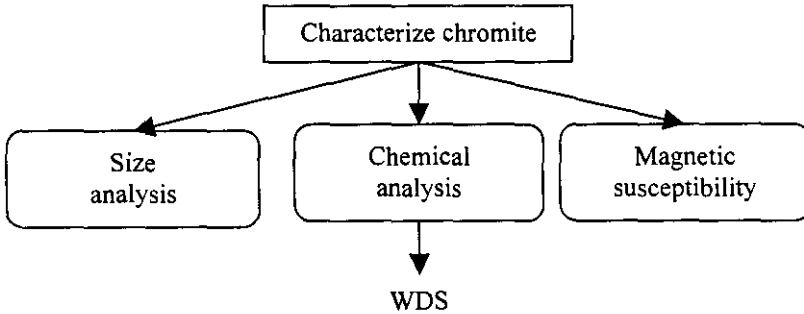


Figure 3.8: Flow sheet for the characterisation of chromite for this study.

3.8 Determination of the impact of roasting, at conditions used to determine optimum roasting conditions for LSR, on the magnetic susceptibility of chromite

As summarized in figure 3.9 the individual secondary samples were roasted in batches under the different roasting conditions, stated in table 3.4, utilizing a custom-made fluidized bed roaster as described in paragraph 3.2.3. The roasted samples were characterized by determining the magnetic susceptibility thereof with a Barrington MS2 magnetic susceptibility meter, by SEM optical investigation and WDS and EDX chemical analyses.

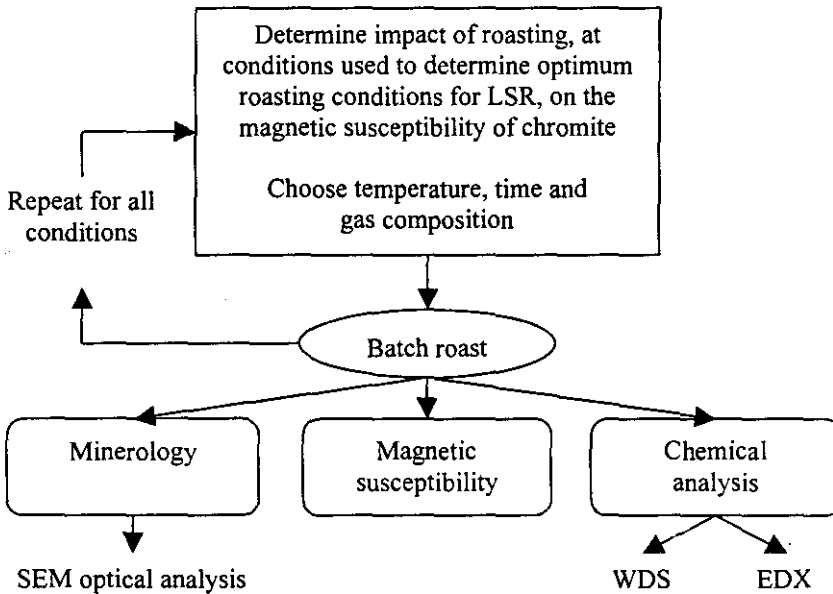


Figure 3.9: Flow sheet to determine the impact of roasting of chromite at the roasting conditions utilized to determine the optimum roasting conditions for LSR.

Table 3.4: Roasting conditions to determine the impact of roasting of chromite.

Time intervals [min]	0; 5; 10; 20; 30; 40
Temperatures [°C]	700; 750; 800; 850
Atmosphere	Air

3.9 Preparation of fractionation curves for chromite before and after roasting at optimum roasting conditions for LSR

As shown in figure 3.10 fractionation curves for the UG1 chromite were constructed for unroasted chromite and for chromite roasted at 750°C for 20 minutes. The method used to construct these is described in paragraph 3.2.4.

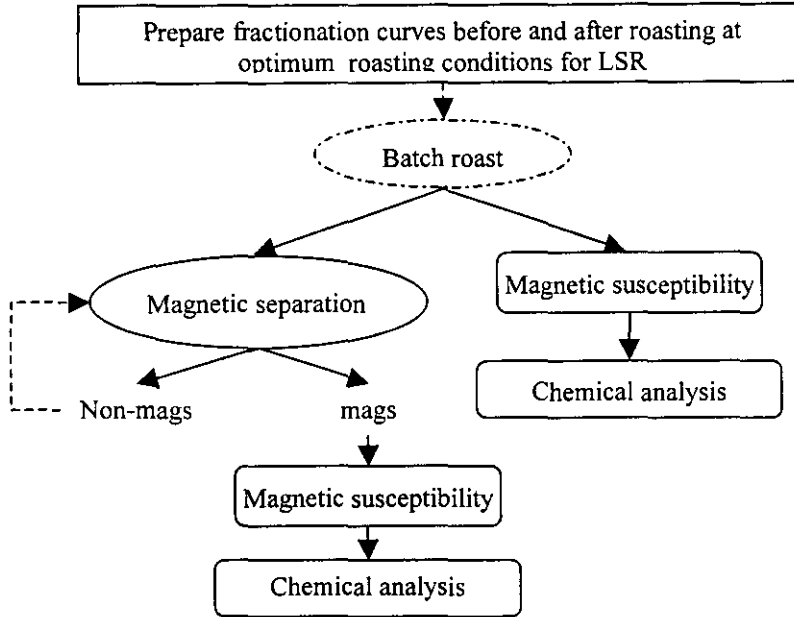


Figure 3.10: Flow sheet for the preparation of fractionation curves for chromite before and after roasting at optimum conditions for LSR.

3.10 Data capturing

Data was captured either electronically or manually as indicated in table 3.5. Table 3.5 also indicates where the data was interpreted by others and submitted as a written report.

Table 3.5: Details on how data was captured

Analysis	Electronic (e) or manual (m)	Interpreted by others
XRF	e	No
Titration	m	No
XRD	e	Yes
Reflected light microscopy	m	Yes
Micro-analysis	m	Yes
Particle counting	m	Yes
QEMSEM	e	Yes
QEMscan	e	Yes
Magnetic susceptibility	m	No

In short: the aim of chapter 3 was to describe the design as well as the execution of the research program to test the hypotheses defined in Chapter 2. Firstly the key concepts and variables in this study were highlighted. The various test procedures used in this study were discussed in detail. A short comment was made on data capturing methods. In chapter 4 the results are presented and discussed.