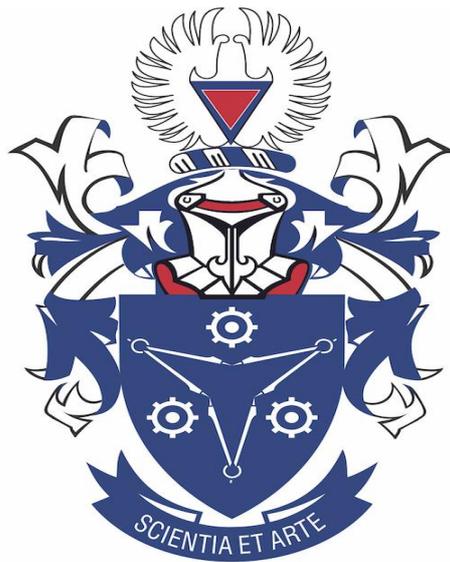


**Development of a beneficiation route to upgrade Sishen iron ore jig slimes for
iron making**

Molebiemang G.M

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VUT

Vaal University of Technology

Department: Metallurgical Engineering

Faculty of Engineering and Technology

Vaal University of Technology

Vanderbijlpark

Supervisor: Prof I.O. Otunniyi

Co-supervisor: K.C. Thubakgale

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PREFACE

I declare that the contents of this thesis, submitted to the Vaal University of Technology for the degree of Magister Technologic (M-tech) in Metallurgical Engineering, are original and have not been submitted prior to this for an academic examination towards any qualification at this university or any other institution.

Signed:

Gracious Mmathuto Molebiemang

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ABSTRACT

Kumba Iron Ore's Sishen mine is located in South Africa's Northern Cape Province and produces about 40 million tons (Mt) of iron ore per annum. The ore body consists mainly of laminated and massive type hematite ore of which 30 Mt is being beneficiated by Dense Medium Separation (DMS), while the remaining 10 Mt is processed by the jig plant that was added as an expansion to the DMS processing route. The jigs at the jig plant are employed to treat coarse, medium and fine Run of Mine Ore (ROM). Due to the presence of finer particles on the screened ROM, hydrocyclones are employed on the secondary screen that feed the finer jig to produce fines on the overflow that is fed to the thickeners for dewatering by sedimentation, and the formation of clear liquid that can be recycled to the Jig plant. However, it was suspected that a substantial amount of the iron gets lost to the hydrocyclone overflow with slimes. These slimes consist of very fine particles (about 90% -45 μ m) which make treatment with gravity concentration methods difficult. Sishen mine attempts to beneficiate the slimes using Pulsating High Gradient Magnetic Separators (PHGMS). However, PHGMS produce a concentrate consisting of 64.0% iron and 5.0% silica which render the slimes not suitable for pelletizing. Normally the slimes are considered as waste and are rejected to the slimes ponds. Rejecting slimes is not only a loss of resources, but it also poses severe long-term environmental problems. Recovery of iron minerals from the tailings produced in a large iron ore operation is of importance as it will result in environmental and economic benefits which indicate the importance of utilising those accumulated fines.

An investigation was carried out to determine a possible beneficiation route for Sishen mine iron ore slimes with the aim of achieving concentrate suitable for pelletizing to be used in iron making. Two samples were used in the investigation namely; Direct Process Slimes (DPS) and Magnetic Separation Processed Slimes (MSPS) from Sishen mine. Both DPS and MSPS were first characterised by Scanning Electron Microscopy (SEM), X-ray Fluorescence (XRF) and particle size and deportment analyses for mineral and elemental composition. Beneficiation routes explored were: direct and reverse flotation on the DPS using different collectors and depressants at varying dosages; reverse flotation on MSPS using conditions derived from reverse flotation of DPS; reverse flotation over narrow size distribution on DPS and MSPS.

The SEM image and particle size deportment analyses on both DPS and MSPS show that the slimes contain more iron in finer to very fine fractions with silica in medium to coarse fraction. This was corroborated by XRF results which showed that DPS contained 44.0% iron, 20.4% silica and 8.7% alumina whereas MSPS contained 63.7% iron, 4.8% silica and 1.9% alumina. Reverse flotation on

DPS using diamine collector (lilaflot D817M) attained very minimal enrichment, giving 51.2% iron and 16.9% silica in the concentrate. In reverse flotation of MSPS iron content was increased from 63.7% to 66.1% with silica and alumina being reduced from 4.8% to 2.8% and 1.9% to 0.5%, respectively. However, reverse flotation over a narrow particle size distribution on MSPS produced a product containing 68.0% iron with silica of 1.6% and alumina of 0.1% (1.7% silica + alumina) which rendered the ore slimes suitable for pelletizing (which requires > 63.0% iron and <5.0% silica + alumina). Therefore, the proposed route to beneficiate the slimes is to pre-concentrate using sLon PHGMS, screening at 25 µm and conducting two-staged reverse flotation on the -25 µm fraction with lilaflot D817M collector at 75 g/t (which in total is 150.0 g/t from two stages) and corn starch depressant at 1000 g/t. For industrial purposes, the two-staged reverse flotation on whole size fraction of MSPS can be used, as it also meets the target specification for pelletizing and constraints might occur to classify and beneficiate at fine-ultrafine fractions.

ABBREVIATIONS

DMS	Dense Medium Separation
DIR	Direct Iron Reduction
EAF	Electric Arc Furnace
PZC	Point of Zero Charge
RH	Ruhrsthal Heraeus
SAIMM	South African Institute of Mining and Metallurgy
STEM	Science, Technology, Engineering and Mathematics
VOD	Vacuum Oxygen Degassing Technology
VUT	Vaal University of Technology

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CHAPTER 1

1. INTRODUCTION

1.1 Background Information

The Northern Cape's Sishen and Kolomela and Limpopo's Thabazimbi mines are operated by Anglo American Kumba Iron Ore. Having the high-quality resource base in both South Africa and Brazil, the Anglo American Kumba Iron Ore is one of the world's top five iron ore producers and, exports 36.1 Mt of high-grade iron ore annually to their customers around the world; including China, South Korea and Japan as map in Figure 1.1 illustrate (Anglo American, 2012:23-35). Sishen mine is the leader in the three mines operated by Anglo American Kumba Iron Ore. The ore body at Sishen mine consists mainly of laminated and massive type hematite and, is mined through an open pit method which involves drilling and blasting. The mined ore is trucked to the beneficiation plants namely; Dense Medium Separation (DMS) and Jig plants, where 40 million tons (Mt) of high grade iron ore is produced per annum; 30 Mt is being beneficiated by DMS, while the remaining 10 Mt is processed by the Jig technology. The jigs were added to increase the processing capacity on that achieved using the DMS plants (Myburg, 2009).

During the beneficiation of the ore by DMS and jigs, ~ 4.9 Mt slimes are generated per annum due to crushing, washing and separation of coarse, medium and fine materials by wet screening. In general slimes are described as the portion of an ore which is too fine to be commercially exploited by the process developed for coarser size fractions. Normally these slimes are considered as waste and are rejected to the slimes ponds. Rejecting slimes is not only a loss of resources, but it poses severe long-term environmental problems. The environmental and economic benefits of recovering iron minerals from the slimes produced, in a large iron ore operation, was addressed and the importance of utilizing accumulated slimes or fines was demonstrated (Santos, 2003). The production of hematite ore by Sishen mine is one of the major sources of revenue in South Africa. In view of the depleting reserves of high-grade ore, recovery of mineral values from the slimes, being lost to the tailing dams, is of importance in an attempt to meet the rapidly growing demand.



Figure 1.1: Anglo American Iron ore Ltd operations in South Africa (right) and customers (left) around the world (Anglo American, 2012)

Iron (Fe) ores occur in the form of magnetite (Fe_3O_4), hematite (Fe_2O_3), goethite ($\text{FeO}(\text{OH})$), limonite ($\text{FeO}(\text{OH}) \cdot (\text{H}_2\text{O})$) or siderite (FeCO_3) and these ores are mainly used as raw material to make pig iron for production of steel (Filippov *et al.*, 2014:62). The steel industry needs increased of iron ore concentrates to be used in blast furnaces, which requires concentrates with iron content greater than 63.0% with minimal impurities such as silica (SiO_2) and alumina (Al_2O_3). This is to minimise the negative effects these components have on the mechanical properties of steel products. In addition, other gangue minerals such as those bearing sulphur (S) and phosphorus (P) must also be minimised because they induce negative mechanical effects such as brittle fracture and hot shortness on the steel products (Filippov *et al.*, 2014:62; Ma, 2012:56-58).

The slimes generation and formation is not favourable but it cannot be avoided, especially for iron ores that are soft laminated or lateritic. As in general, accumulation of slimes in most ores is due to weathering and decomposition of certain rock components. Subsequently, slimes can also be produced from comminution, washing and screening of the ore (Ansari, 1997:93). Historically, these slimes were considered as waste in the mineral processing industry and discarded to tailings dams which resulted in mineral and metal value losses and also caused environmental and social problems (Ansari, 1997:93). Due to the hazards associated with discarding these slimes, considerable interest is growing in developing processes and in improving old processes for recovery of values. The processes used are: froth flotation, flocculation, magnetic separation, centrifugal jiggling, Reichert cones, Spherical agglomeration etc. (Ansari, 1997:93-94).

Of the processes mentioned above, froth flotation and magnetic separation are usually considered to beneficiate iron ore slimes. Froth flotation is a process in which valuable minerals are separated from the gangue minerals by exploiting the differences in their surface properties and consists of two routes namely direct and reverse flotation (Wills, 2006:267-268). In direct flotation, valuable mineral particles are carried to the froth leaving the gangue minerals in the pulp and the opposite is true for reverse flotation. In the froth flotation process, the ore is crushed and ground to a finer size (Wills, 2006:267-269). Finer grinding liberates the valuable mineral particles from the waste rock and other particles (Ives, 1984). Pulp is then processed in the flotation cells, which agitate the mixture and introduce air to form small bubbles. The mineral particles are attached to air bubbles through addition of flotation reagents and rise through the pulp to the surface of the froth that is present in the flotation cell. The mineral is physically separated from the remaining pulp material and is removed for further processing (Wills, 2006:267-270).

Historically, direct flotation of iron ore was the first process used to float iron using anionic collectors such as petroleum sulphonate, fatty acids and hydroxamates. However, the presence of hydrolysable cations (such as calcium, magnesium and iron) in flotation pulp, significantly reduced the selectivity of the direct flotation route, though it still appears desirable for some low grade iron ores that contain a vast amount of quartz (Ma, 2012:57). In addition, other flotation routes used are reverse cationic (where amine collectors are used to float quartz bearing minerals and iron bearing minerals are depressed by starches) and reverse anionic flotation (where quartz bearing minerals are first activated by lime and then floated using fatty acids and iron bearing minerals are depressed by starches) (Ma, 2012:56-61). The flotation routes found their widest application in beneficiating iron ore slimes or fines in the mineral processing industry with reverse cationic being the most frequently used (Thella *et al.*, 2012:418-426; Das *et al.*, 2005:737-742; Rocha *et al.*, 2010:842-845; Kumar *et al.*, 2010:637-648; Kumar and Gopalknshma, 2013:1-6; Oliveira, 2006; Viera and Peres, 2010:124-128; Suresh *et al.*, 2012:5273-5281; Tohry and Dehghani, 2016:28-33).

Magnetic Separation is a physical concentration process that utilises the differences in magnetic properties of various minerals present in the ore body. The magnetic and non-magnetic fractions produced from the process are regarded as either valuable or gangue depending upon its end use in a particular process, e.g., separation of magnetite (magnetic) from quartz (non-magnetic), separation of tin bearing mineral cassiterite (non-magnetic) from magnetite (magnetic) impurity etc. (Das and Roy, 2007). Using both low and high magnetic separation for beneficiation of fine, weakly magnetic, particles can result in low recoveries which can be resolved by increasing magnetic field and field

intensity to more than 20 000 gauss through the use of High Gradient Magnetic Separators (HGMS) (Das and Roy, 2007). However, high gradient magnetic separators sometimes face challenges such as matrix clogging and entraining of non-magnetic particles which can be solved by the use of Pulsating High Gradient Magnetic Separators (PHGMS). The PHGMS are able to beneficiate fine and weakly magnetic particles because of the vertically rotating ring and the mechanism of slurry pulsation. The separator also has a higher beneficiation ratio, the matrix cannot be easily clogged and it is flexible and adaptable to various particle sizes compared to other magnetic separators (Xiong *et al.*, 1989:947-952; Liu *et al.*, 1991:152-159; Yang *et al.*, 1993:211-221; Xiong *et al.*, 1998:111-127; Zeng and Dahe, 2003:219-224).

Table 1.1 shows iron ore slimes beneficiation scenarios by employing the beneficiation techniques discussed above separately and in combination. From the various approaches, it can be projected that flotation and magnetic separation, separately or in combination should be able to produce concentrates of improved iron grade from Sishen mine slimes. At slimes particle sizes below 40 μm , kinetics of particles under stratification forces will vary widely, as mass effect varies in multiple orders from finest particles to the coarsest. For instance, 40 μm particles will be eight times heavier than the 5 μm particles. Classifying the slimes into narrow size distribution before further exploration of the beneficiation technique should give improved response. The goal of this study is to investigate a suitable beneficiation route for processing the Kumba iron ore jig slimes, for the attainment of a concentrate that would satisfy the pelletizing specification, as shown in Table 1.2 (Viera and Peres, 2010:124-125; Mbele, 2012:221-223; Ola *et al.*, 2009:405-416; Ma, 2012:56-58).

Table 1.1: Different approaches for treating iron ore slimes

Scenario	Beneficiation approach	Inferences	Reference
Processing of iron ore slimes by magnetic separation at Kiribaru mine in India	Wet High Intensity Magnetic Separator (WHIMS) after classification by cyclone	The results gave 63 % Fe with a recovery of 56 % and 3.3 % alumina	Prasad <i>et al.</i> , 1988:102-112
Investigation of iron ore slimes generated at Indian mines during beneficiation	Convectonal hydrocyclone and WHIMS	Hydrocyclone and magnetic separation indicated that 61-65 % Fe could be obtained with 59-78 % recovery, 2.5 % silica and 1 % alumina	Das <i>et al.</i> , 2002:285-289
Beneficiation of slimes generated during washing for	Hydrocyclone and high intensity magnetic separator	The results showed Fe could be increased from 55-58 % to 64 %	Gupta <i>et al.</i> , 2006:1916-1921

upgrading hematite ore in India		with recovery of 40 and 50 %	
Beneficiation of two Indian iron ore slimes samples by cell and column flotation	Slimes were de-slimes by a hydrocyclone followed by direct/reverse flotation using oleic acid and dodecyl amine as a collector through both the conventional and column cell	Sample 1 yield 65.5 % Fe with recovery of 50 %, 2.3 % silica and 1.8 % alumina using oleic acid. Sample 2 gave 63.1 % Fe with a recovery of 63.6% using a combination of gum acacin, sodium hexametaphosphate and dodecyl amine	Das <i>et al.</i> , 2005:737-742
Beneficiation of Indian iron ore slimes	The slimes were de-slimes followed by column flotation by varying collector dosages and assessing the froth height and air velocity	Recovery of the concentrate could be increased to 63 % with a grade of 64.3% Fe, 1.9 % silica and 2.2 % alumina	Dash <i>et al.</i> , 2015:58-65
Beneficiation of low grade iron ore from Gua mines by grounding the ore to fine sizes	The ore was ground to -105 micron and de-slimes. De-slimes product was subjected to reverse flotation	The de-slimes product gave 61.7 % Fe from feed of 57.6 %. Reverse flotation increased Fe to 64.5 %	Jyoti <i>et al.</i> , 2010:590-596
Processing of tailings (slimes) from a flotation circuit of large iron ore concentrate in Brazil	WHIMS followed cationic reverse flotation	WHIMS yield 34 % Fe with recovery of 80 %. Reverse flotation cleaning stage produced a product consisting of 1% silica and specular amount of hematite content	Viera and Peres, 2010:124-128
Beneficiation of iron ore slimes from Bellary-Hospet India	The slimes were subjected to direct flotation followed by magnetic separation on flotation tailings	The resulted indicated that an overall product of 62.9 % Fe from 34.6 % was obtained	Suresh <i>et al.</i> , 2012:5273-5281
Investigation of ultrafine tailings (slimes) from iron ore concentrator	De-slimes and reverse cationic flotation tests were conducted using different collector and depressant	A valuable product grade of less than 1 % silica was obtained with 12 % Fe in reject product	Rocha <i>et al.</i> , 2010
Beneficiation of iron ore slimes (less than 37 μ m) using guar gum flocculant	Slimes were subjected to selective flocculation tests using guar gum as a flocculant at different dosages, dispersant dose, pH and settling time	A 66 % Fe product was obtained from 58 % having 3.5 % alumina with a recovery of 68 %.	Tammishetti <i>et al.</i> , 2014

Development of a laboratory scale selective flocculation process on ultrafine (less than 10 µm) iron ore tailings	Selective flocculation tests on slimes using different starches and polyacrylamides.	A feed containing 46.6 % Fe was upgraded to a concentrate with 57-58 % Fe iron at 65-75 % recovery	Weissenborn <i>et al.</i> , 1994:191-213
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Table 1.2: Iron and oxides requirement in the pellets for iron making (Viera and Peres, 2010:124-128)

Elements and oxides	Value (%)	Tolerance
Iron	65	Minimum
Silica+Alumina	5	Maximum
Calcium oxide	0.03	Maximum
Magnesium oxide	0.06	Maximum

1.2 Statement of the Problem

Sishen mine produces about 40 Mt of high grade hematite ore per annum. The 10 Mt of the ore is being processed by Jigs technology to make up for the annual production of 40 Mt. The Jigs at the Jig plant are employed to treat coarse, medium and fine Run of Mine Ore (ROM). Due to the presence of finer particles on the screened ROM, hydrocyclone's are employed on the secondary screen that feeds the finer jig to produce fines on the overflow that are fed to the thickeners for dewatering by sedimentation, and formation of clear liquid that can be recycled back to the Jig plant. However, iron is lost to the hydrocyclone's overflow slimes. These slimes consist of very fine particles (about 90%-45µm) which make treatment using gravity concentration methods difficult. Sishen mine proposed the use of sLon pulsating high gradient magnetic separator to beneficiate the generated slimes as it would increase the production of hematite ore and decrease the environmental hazards associated with discarding the slimes. Two-staged sLon PHGMS was used as concentration method and produced concentrate with a reasonable iron content of 63.0%. However, the silica grade was 5.0% which is high for the pelletizing specification (<5.0% Silica+Alumina) (Viera and Peres, 2010:124-125; Mbele, 2012:221-223; Ola *et al.*, 2009:405-416; Ma, 2012:56-58).

In many plants around the globe, iron ore slimes have been processed successfully via different techniques, including froth flotation. Froth flotation process is not used anywhere in the Sishen mine operation, and amenability of the ore slimes to be beneficiated by the process separately or in

combination with magnetic separation using proposed reagents in this present work has not been investigated. It is therefore considered worthwhile to explore the potential of upgrading these slimes via different flotation schemes. Responses of the slimes to known flotation reagents have to be investigated first and composition determined to establish a basis for recovery analysis. From literature, applicable reagents in iron ore flotation are starches (depressant), amines (collectors), sodium hydroxide (NaOH) and hydrochloric acid (HCl) as pH adjustors (Araujo *et al.*, 2005:219-224). Probable reagents for investigation will be selected from this range of chemicals. Therefore a suitable processing route will be explored to beneficiate the slimes by separating the valuable iron minerals from the associated gangue minerals and thus maximizing the use of slimes of the Kumba iron ore deposit for the steel industries.

1.3 Aim and Objectives

The aim of the project was to develop a mineral processing route to upgrade the iron content of the Kumba Iron Ore's Sishen mine slimes generated prior to jig operations for iron making. The general objective is to explore different froth flotation schemes towards obtaining a concentrate grade suitable for iron making at acceptable recoveries.

The specific objectives are to:

- i. Determine mineralogical and chemical compositions of the Direct Process Slimes (DPS) and Magnetic Separation Processed Slimes (MSPS) as-received and over narrow particle size distributions;
- ii. Conduct direct and reverse flotation tests on the Direct Process Slimes to assess the response of the slimes under the different schemes and reagents (at different amount of dosages and pulp density);
- iii. Repeat reverse flotation at inferred optimum reagent dosages on MSPS, and on fractions screened to narrow particle size intervals from both DPS and MSPS

1.4 Significance of Research

The research is of importance as there is presently no literature report of studies carried out to determine the amenability of Kumba Iron Ore's Shishen mine slimes (generated prior to the jig process) to alternative mineral processing methods such as froth flotation separately or in combination with magnetic separation. The alternative concentration techniques and routes being proposed in this study are considered capable of substantially increasing the iron content of the slimes while reducing the deleterious contents like silica and phosphorus. The successful accomplishment

of this aim will render the slimes suitable for pelletizing that can be fed to the conventional blast furnace and probably the Midrex for direct iron making processes.

CHAPTER 2

2. LITERATURE REVIEW

2.1 Characterization of Iron

Iron is an element found in the earth's crust with a relative abundance of 2.0 to 3.0% in sedimentary rocks and to 8.5% in basalt and gabbro. Deposits of iron must contain a percentage of iron greater than 25.0% to be considered an ore that can be economically exploited. It is however possible for a deposit to be lower than 25.0% and still be exploited if the ore exists in a large deposit and can be concentrated and transported at low cost (Weiss, 1985).

2.1.1 Occurrence of Iron

Iron is one of the most-abundant elements, which make up about 5.0% by mass of the earth's crust. Iron can be found in small amounts in water, plants and even blood. The reason for iron occurring so widely spread is that it can easily combine chemically with other elements in a wide variety of physical and chemical environments. There are seven iron-bearing minerals which can be deemed as sources of iron where iron can be recovered economically. These iron minerals are (Weiss, 1985):

- Hematite (Fe_2O_3) 70.0% Fe
- Magnetite (Fe_3O_4) 72.0% Fe
- Goethite ($\text{FeO}\cdot\text{OH}$) 61.0% Fe
- Siderite ($\text{FeO}\cdot\text{CO}_2$) 48.0% Fe
- Limonite ($\text{Fe}_2\text{O}_3\cdot 3\text{H}_2\text{O}$) 59.0-63.0% Fe
- Lepidocrocite ($\text{FeO}\cdot\text{OH}$) 61.0% Fe
- Chamosite ($3\text{FeO}\cdot\text{Al}_2\text{O}_3$) \pm 35.0% Fe
($2\text{SiO}_2\cdot 6\text{H}_2\text{O}$).

From the above list, the main sources of iron are hematite, magnetite, and goethite. Magnetite (Fe_3O_4) forms a black magnetic iron ore and huge deposits are mostly in Russia and Sweden. Hematite (Fe_2O_3) is a red iron ore that occurs almost in all forms (from solid rock to loose earth) in large quantities throughout the world whilst Goethite ($\text{Fe}_2\text{O}_3\cdot\text{H}_2\text{O}$) is a brown ore containing iron and Limonite ($\text{Fe}_2\text{O}_3\cdot\text{H}_2\text{O}$) and is a yellow-brown iron ore that is a collective term for impure goethite and a mixture of hydrated iron oxides. Quartz, feldspar, calcite, dolomite, clayey substances, and carbonaceous

matter make up the gangue minerals associated with iron ore deposits. The common deleterious constituents of iron ores are phosphorus, potassium, alumina, silica, titanium, sulphur, zinc, manganese, and arsenic (Weiss, 1985).

2.2 Iron ore Mining and Beneficiation

Iron ore is commonly mined in surface operations with methods such as open pit cast. Practically, the ore can be mined underground provided that the technique is independent of the proximity of the ore body to the surface. Surface mining is designed to extract ore from surface deposits and underground mining is used to extract ore that lies deep underground (United States Steel, 1973; U.S. EPA, 1982). Beneficiation of iron ore incorporates concentration, commonly by physical removal of unwanted gangue. Iron or steel processing from concentrated product involves the use of pyro-metallurgical techniques. Historically, iron ore was crushed and shipped directly to a blast furnace. However, most of the ores mined these days must undergo various beneficiation procedures to upgrade the iron content and prepare the concentrate for the blast furnace. The blast furnace operation requires ore feed of a specific size, structure, and chemical make-up for optimum efficiency (Weiss, 1985). A simplified schematic flowsheet showing the mining of iron ore and beneficiation process is shown in Figure 2.1.

Furthermore, there are various methods of beneficiation that can be used to prepare iron ores, depending on the iron content in the ores. Iron ores containing more than 60.0% Fe can be prepared for further beneficiation by crushing and blending. Contrary to this, preparations of some iron ores include screening and concentrating of the raw iron ore. Predominantly, operations needed before iron ore is considered a finished product include (Weiss, 1985):

- **Crushing & Screening-** Following blasting, the ore is loaded on a mining truck and transported. Two or three stages of crushing are normally employed in the mines, for example primary crusher use conveyors to transport the crushed ore to secondary and tertiary crushers or mills. In crushing stages iron ore is reduced from heavy, big stones, to suitable sizes as final product. Furthermore, the size reduction of the ore is done by milling operations which are fed by the crusher product (U.S. EPA, 1982).
- **Grinding-** Rod, ball and autogenous mills are used in grinding circuits to grind the ore down into even smaller uniformed sized particles. Normally the ore can be ground to less than 325

microns (μm), and in some cases less than (45-25 μm) to help facilitate the liberation of the iron minerals from the rock (U.S. EPA, 1982).

- **Concentration** – Involves separation and concentration of valuable minerals from the milling circuit product which becomes the feed to the concentration circuit. Gangue minerals are then separated. Magnetic separation is used to concentrate iron ores with taconite being the most concentrated due to the magnetic properties of magnetite. Conventional flotation can also be a method of concentration for hematite ores; where the concentrate is passed to pelletizing and sintering operations. (Weiss, 1985; U.S. EPA, 1982).
- **Agglomeration**- For iron-making to occur at the blast furnace, iron ores, coke, and limestone that are sent to the furnace need to be permeable to allow for an adequate flow of gas through the system. Furthermore, very fine concentrates in crude ores that are used as feedstock to blast furnaces need to be agglomerated to prevent blockage in the furnace (Weiss, 1985). Agglomeration processes are divided into:
 - **Sintering** – Where the iron-bearing minerals such as ore fines or concentrates are mixed with fuels, spread on surface beds and ignited by gas burners. The fine particles are fused by the high temperature heating process, approximately at 1100 degree Celsius ($^{\circ}\text{C}$). The sinter is then sized and the fines recycled (U.S. EPA, 1982).
 - **Pelletizing**- In which the concentrate is fed into a balling machine and binders (such as soda ash, bentonite and organic compound) are added to strengthen pellets. The concentrate and binder agglomerate into pellets of a consistent size as the drum rotates, after which any remaining fine fraction of the material is recycled back through a process known as indurating. Marbles or balls produced at appropriate sizes are strong enough to retain their shape when shipped to the blast furnaces for iron making (U.S. EPA, 1982).
 - **Briquetting** – Where fine ore is heated and pressed into briquettes, once cooled they are sent directly to the blast furnaces (U.S. EPA, 1982).
 - **Nodulising** - Where fine ore is tumbled in a rotary kiln at a temperature close to melting (U.S. EPA, 1982).

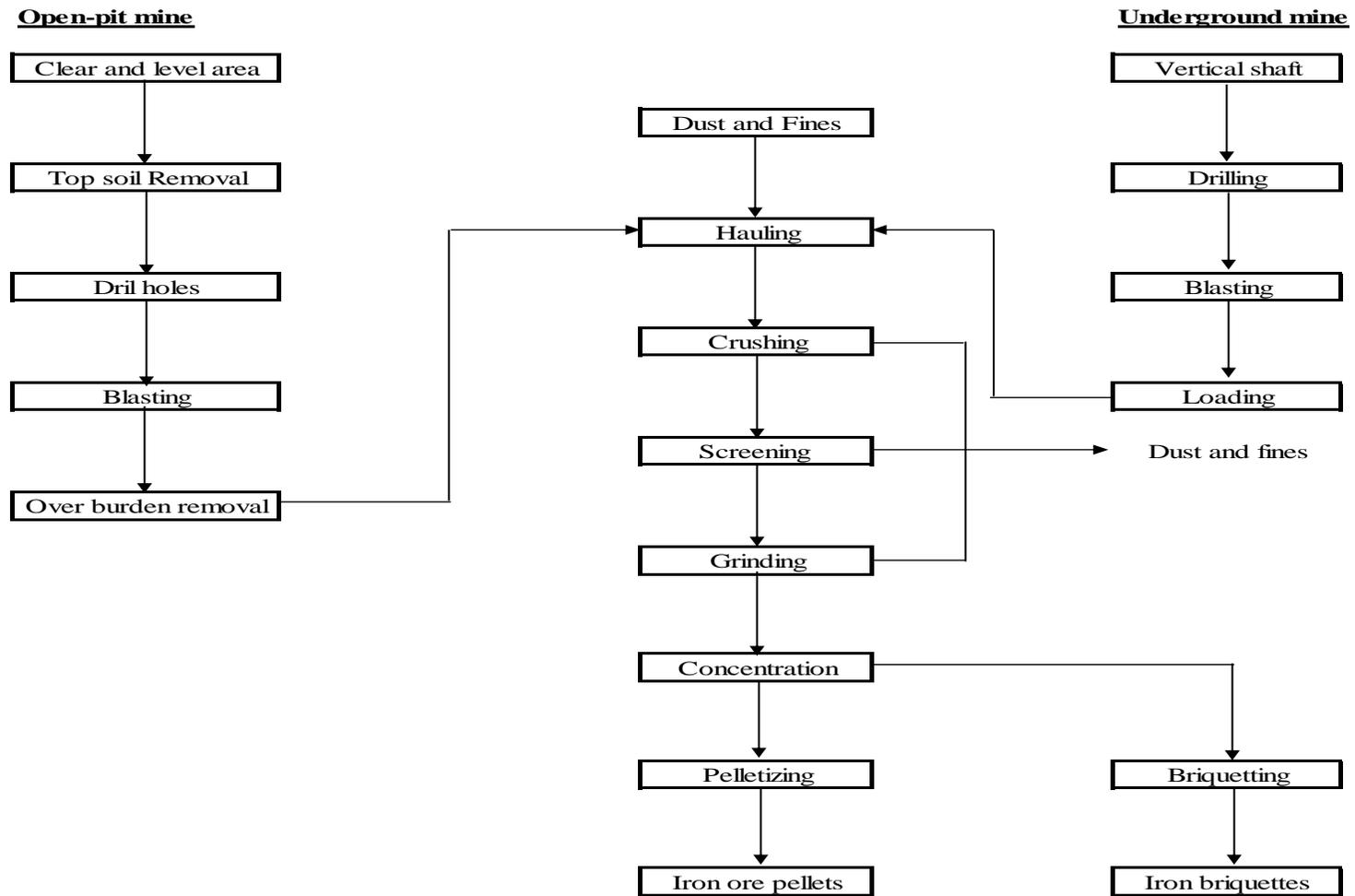


Figure 2.1: A schematic flowsheet simplifying the mining and processing of iron ore (Weiss, 1985)

2.3 Slimes formation and generation during Iron ore Mining and Processing

Iron ore slimes refer to small particles of ore that cannot be beneficiated by techniques that were developed for coarse particles and are usually discarded as waste during mining and processing of iron ores. Slimes are stored in tailings dams/ponds or as stock piles at mining sites. Primarily, the iron ore slimes are formed from soft laminated and laterite iron ores due to weathering and decomposition of the ore and certain rock bodies associated with it. Secondly, formation of slimes is due to mining processing, comminution of ore to its liberated size and handling (Ansari, 1997:93-94).

A large quantity of slimes is generated in washing plants at mines and they contain about 48.0-60.0% Fe and are normally discarded as tailings. In India, for example, 10-15 million tonnes of iron ore is lost as slimes and this makes up about 18.0-25.0% of iron ore processed. These slimes are normally found in finer sizes (<150 microns), assaying 55.0-60.0% iron and 6.0-8.0% alumina, preventing the need to crush to finer sizes. Brazil is also one of the largest producers of iron ore fines or slimes generated during the washing process; a project managed by Sarmarco Mineraco produced 10.4 Mt of pellets with a capacity of 12 Mt/year. The feed to the pelletizing plant was 1.94t of iron ore slimes generated during washing processing of high grade ore (Roy *et al.*, 2007:3271-3287; Moore, 2011:8).

2.3.1 Why slimes are considered as a problem during mining and processing

Slimes are considered a problem, because of the difficulties in handling them due to having small mass, high surface area and high surface energy. Small mass of the slimes causes low particle momentum, hetero-coagulation, particle entrainment in concentrates (e.g., froth), low probability of collision with a bubble and difficulty in overcoming the energy barrier between particle and particle as well as particle and bubble. The high surface area causes a high dissolution rate in water, adsorption of large quantity of chemicals, rigidity of froth, high pulp viscosity and undesirable coating of the valuable particles by ultrafine gangue particles. It is believed that the high surface energy per unit area of the slimes is caused by the imperfect crystallisation, increased cracks, dislocations, edges and other high energy sites which can result in difficulties such as nonspecific adsorption of reagents, increased hydration, rapid surface reaction, and increased solubility (Ansari, 1997:95-97)

2.3.2 The effects of discarding slimes generated during mining and processing

It is estimated that about 70.0-75.0% of the total production of iron ore production is fines which occur at the time of mining or become fines during subsequent handling or conversion of lumps into

calibrated lump ore (CLO) (Roy and Das, 2008:168). These slimes cannot be directly utilised in iron making due to their low iron content, unfavourable granulometry and high content of impurities such as alumina and silica. Therefore, the mining industry is forced to discard the slimes which end up accumulating at mine sites where they are dumped in stockpiles which occupy space that could have been used for other purposes. The neighbouring agriculture and water bodies might also be affected, because during the raining season slimes or fines are washed out by water and spill all over and increase the percentage of suspended particles in water bodies.

Air pollution in mining areas is a well-known problem and occurs due to drilling blasting and plant operation. Additionally, air pollution might also be caused by the generation of slimes during processing and dumping of the slimes as stock piles and transportation of ore. As a result, the environment and ecology can be damaged considerably. For example, people can be affected as fine particles that are inhaled easily sink deep into lungs where they can remain embedded for long periods of time and result in premature death due to lung diseases. People with lung disease such as asthma also suffer from air pollution caused by the generation or discarding of slimes. Furthermore, asthma flare-ups might be triggered which could cause people with sensitive airways to wheeze, cough and have respiratory irritation. Infants and children may also be affected because of greater exposure to air pollution due to their faster breathing rates and increased amounts of time spent outdoors. Pregnant women are also considered as another sensitive group that needs to be protected from air pollution as it results in infants being born with low weight, pre-term and having increased risk of mortality. Therefore, on average life span might be shortened by one to two years (Roy and Das, 2008:119-171; Ansari, 1997:93-94).

2.3.3 The significance of beneficiating the slimes generated during mining and processing

In the past, iron ore slimes generated at mine site were piled due to their lower demand than the fines generated at steel plants where fines are dumped. Recently utilization of slimes has become essential because it results in economic and ecological benefits; surface degradation, ground water pollution and destruction of forests will be minimised as the slimes are reduced. The mineral wealth loss will also be prevented and environmental issues avoided which are caused by long storage of fines/slimes on the soil (Kumar and Mukherjee, 1994:1-6; Ansari, 1997:93-94; Roy and Das, 2008:213-231; Roy and Das, 2008:173).

2.4 Kumba Iron Ore's Sishen mine

Sishen mine is located in the Northern Cape Province of South Africa near the mining town of Kathu (as Figure 2.2 illustrates) and is one of the biggest open-pit mines in the world. The mine has been operating since 1953 and has sufficient reserves to sustain it for a further 19-years of production. The Sishen mine is operated by Anglo American's Kumba Iron ore, and the operation accounts for the majority of iron ore production of Kumba. With the first ore exported from the mine in 1956, more than 900 Mt of iron ore has been produced over close to 60 years of operation with about 3.9 Mt of its production supplied to Arcelor Mittal South Africa in 2013. The mining method used at the Sishen mine is opencast mining (as illustrated in Figure 2.3); where the ore is transported to the beneficiation plant, crushed, screened and beneficiated. The mine is one of the hematite ore producers in the world to fully beneficiate its product through Dense Medium Separation and Jig technology (Sishen mine, 2012).

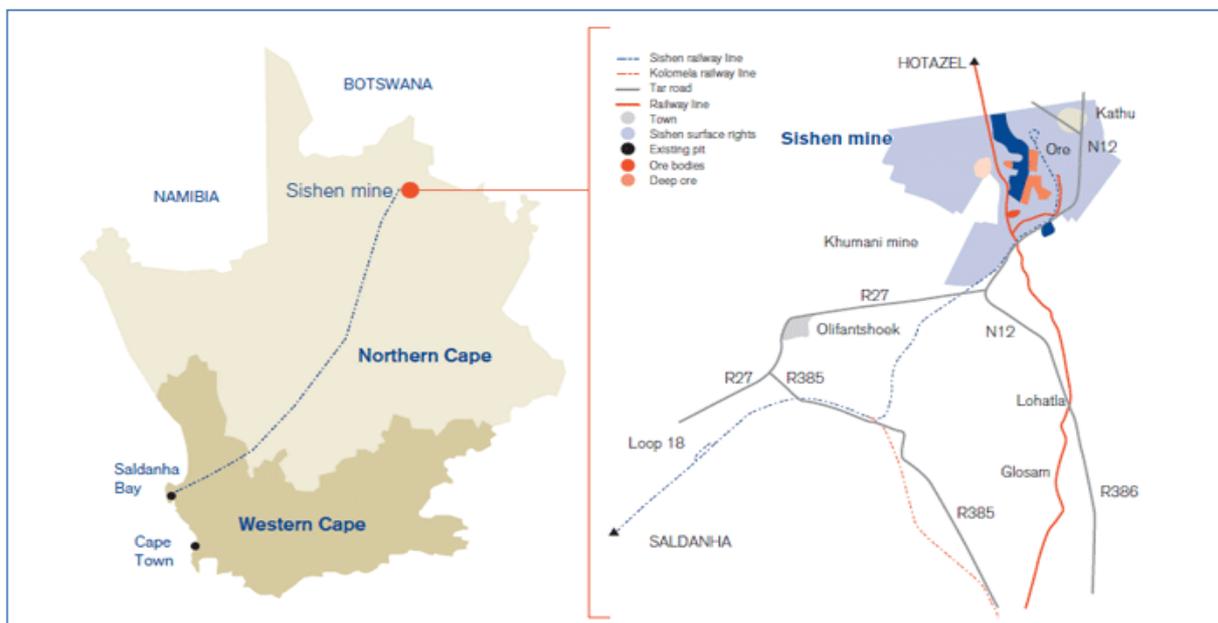


Figure 2.2: Map showing location of Kumba Iron Ore's Sishen mine (Sishen mine, 2012)



Figure 2.3: Sishen mine open pit mining (Sishen mine, 2012).

2.4.1 Geology of the Sishen mine

High-grade hematite iron ore (laminated, massive, conglomeratic and breccia types) is found in the upper parts of a Lake Superior-type, banded iron formation succession of the Griqualand West Supergroup; the Transvaal Supergroup, as it is known, where it occurs in the Northern Cape. Griqualand West Supergroup, the Campbellrand Group dolomites, and the youngest deposits in the area, the Kalahari Group sands, were formed due to a number of uplift and erosion cycles and deformation events which are recorded in the rock record as part of the long geological time interval between the depositions of the oldest rocks. At the mine, the deposits are located on the western flank of the Maremane Anticline, which strikes north-south and dips $\sim 11^\circ$ in the west. In general, the high-grade laminated and massive ore are preserved in synclinal and pseudo-graben structures, which are the result of multiple deformation events. Medium- and lower-grade conglomeratic and brecciated ores are preserved within deep paleo-sinkhole structures in the southern portions of the deposit. The structures are the result of karstification of the underlying dolomites during periods of uplift and erosion (Sishen mine, 2012).

2.4.2 Mining and processing of iron ore in Sishen mine

The mining process of Sishen mine involves conventional topsoil removal and stockpiling, followed by drilling and then blasting of waste lithologies and ore. Overburden is backfilled in the pit or hauled to waste rock dumps on the edge of the pit. Iron ore is then loaded according to blend (grade)

requirements and transported to the beneficiation plants (as shown in Figure 2.4 and 2.5) where it is crushed, washed and screened. Once each size fraction is separated, dense medium separation or jigging processes are used to beneficiate the ore before being stockpiled on the product beds. Plant slimes are pumped into evaporation dams and the plant discard material is stacked on a separate waste dump (Myburg, 2009).

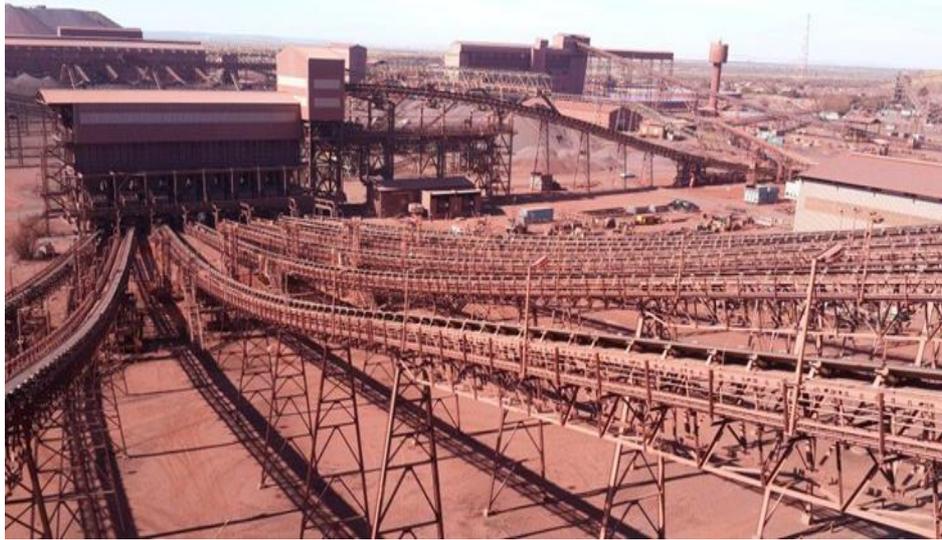


Figure 2.4: Transportation of feed sample to the Jig plant



Figure 2.5: The Jig plant at Kumba Iron ore's Sishen mine

Sishen mine has a concentrate production capacity of 40 million tonnes per annum (Mtpa) (Sishen mine, 2012). The ore body consists mainly of laminated and massive type hematite ore of which 30 Mt is beneficiated by Dense Medium Separation, and the remaining 10 Mt by Jigs technology. The

Jigs at the Jig plant are employed to treat coarser, medium and finer iron ore particles. Iron ore products of different chemical and physical specifications are then produced, reclaimed from the product beds, loaded onto trains and transported to local steel mills and to Saldanha Bay for export to international markets. The products of Sishen include 25mm 65.0% Fe lumpy product from the DMS plant, 25mm 64.0% Fe lumpy product from the jig plant, 27mm and 20mm 66.0% Fe lumpy product, and 8mm 64.5% Fe fines (Myburg, 2009).

2.4.3 The generation of slimes during processing of iron ore in Sishen mine's Jig plant

Sishen mine Jig plant produce 10 Mt of iron ore out of the annual production of 40 Mt. The ROM that is fed to the Jigs includes 15.0% hard ore, 65.0% medium ore and 20.0% fine ore. Due to the presence of the fine material in the ROM, hydrocyclones are employed prior to the finer Jig to produce fines on the overflow that can be fed to the thickeners for water recovery. The water recovered is recycled back to the Jig plant. The slimes generated are about 1.74 Mt per annum (Myburg, 2009).

2.4.4 Attempts at Processing Sishen Iron Ore Slimes – Pelletizing and Magnetic Separation

Kumba Iron Ore's Sishen mine is one of the largest producers of iron ore and major source of revenue in South Africa. Continuous mining of high grade ore will eventually lead to depletion of the ore and the mine would then be faced with challenges to keep producing the product of the required grade. Hence, the need to preserve the raw iron ore by recovery of the iron from slimes. Utilization of slimes will result in increasing the profits of the mine. The environmental and ecological hazards associated with discarding the slimes will be reduced significantly. Due to recognising the importance of utilising slimes, Sishen mine has previously made attempts to process slimes generated prior to the jig operations. The only beneficiation method that has a potential to produce slimes product that meet requirements for pelletizing is the sLon pulsating high gradient magnetic separator though the silica grade was not satisfactory. Mbele (2012:221-223) also conducted pelletizing on the slimes, however, origin of the slimes was not presented clearly and hence assumption cannot be made that it was the slimes generated prior to the jig process. With all the attempts tried on the slimes, to date there is not enough literature in support of studies carried out to determine the amenability of slimes generated prior to the jig process to alternative mineral processing methods such as froth flotation, separately or in combination with magnetic separation.

Froth flotation is considered as a potential beneficiation method for the slimes as it is known to beneficiate minerals with particle sizes below 150 μm by exploiting the differences in their surface

properties. It is, therefore, hypothesised that froth flotation separately or in combination with magnetic separation should give products meeting pelletizing requirements.

Pelletizing processes enables conversion of iron ore slimes (in the size of 80.0% passing 75 microns or lower) into uniform sized iron ore pellets that can be easily charged to a blast furnace or used for DIR production. The key features that promote the process, is that it permits enrichment of iron content by blending blue dust (100.0% Fe_2O_3) with low grades of ore ($\text{Fe}_2\text{O}_3 < 92.0\%$). Pellets are preferred to feed blast furnaces and DIR as they possess metallurgical properties such as high reducibility, better softening behaviour, better bed permeability, and general material handling (Gupta, 2013:235-256). Although the process can face difficulties such as high energy consumption needed for the conversion of green balls in to hard and strong pellets and the need of small amount of binders for strengthening the pellets (Gupta, 2013:235-256). It is still be considered plausible to agglomerate beneficiated Sishen mine slimes as the top size of the slimes is 90.0% passing 45 microns. The produced pellets can be exported to the existing customers or new customers, and can expand the life span of the mine.

The option of froth flotation is, therefore, worthwhile to investigate. However, care should be given to the amount of the reagents used, as it might result in an economical setback.

2.5 Froth Flotation

2.5.1 Basic principles of froth flotation

Froth flotation is a physico-chemical separation process that utilises the differences in surface properties of materials to separate valuable minerals from gangue minerals and depends on the nature and properties of the mineral-water interface. Interaction of water molecules with the mineral surface, both in liquid and gaseous environments and the electrical double layer at solid-water interface, are the two important factors associated with the process (Wills, 2006:267). In flotation processes, valuable mineral particles are transferred to the froth leaving the gangue mineral particles in the pulp or tailing. This is called direct flotation as opposed to reverse flotation, in which the gangue mineral particles are separated into the float fraction. The surfaces of mineral particles can be rendered hydrophobic or hydrophilic through addition of flotation reagents (collectors, frothers, activators and depressants), as a results of ionic interactions and exchanges that occur at the double layer. Hydrophobic particles attach to air bubbles and are carried upwards through the slurry to a froth layer that forms at the top of the flotation cell (as illustrated in Figure 2.6). The froth layer must persist

long enough to either flow over the discharge lip of the cell by gravity or be removed by mechanical froth scrapers (Wills, 2006:267-268).

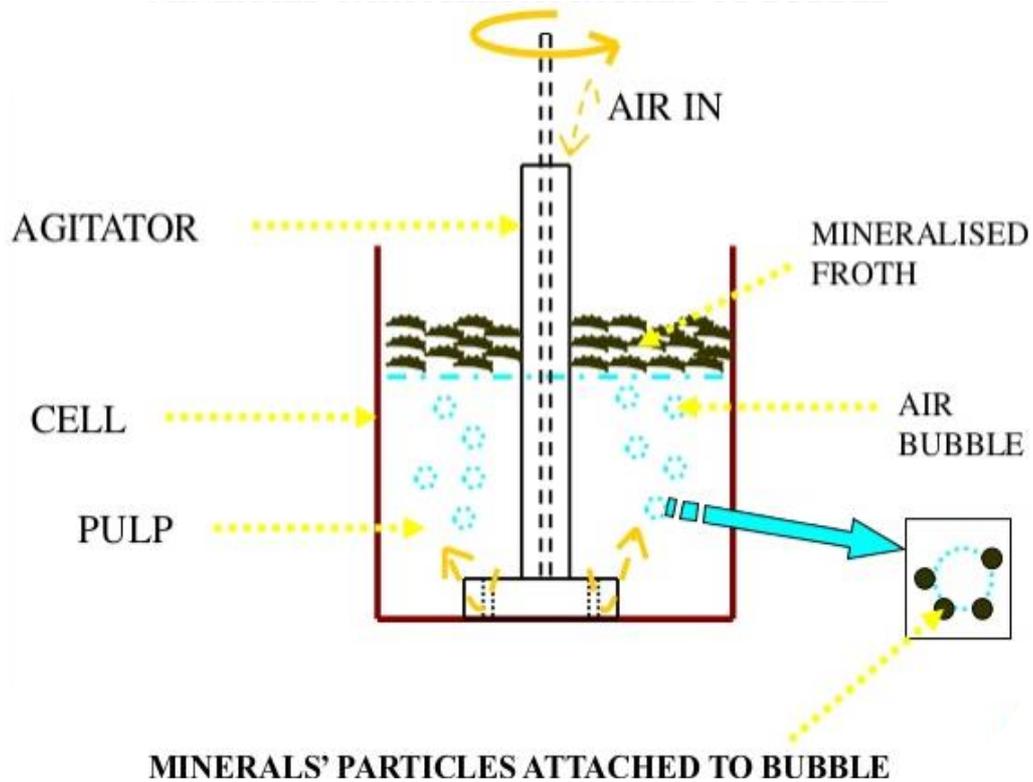


Figure 2.6: Diagram depicting the froth flotation mechanism (Wills, 2006:268)

The activity of a mineral surface in relation to flotation reagents in water, depends on the forces which operate on that surface. Figure 2.7 shows forces tending to separate a particle and a bubble. The forces that lead to the development of a contact angle between the mineral surface and the bubble surface are tensile forces. At equilibrium (Wills, 2006:268),

$$\gamma_{S/A} = \gamma_{S/W} + \gamma_{W/A} \cos \theta \dots\dots\dots (2.1)$$

where $\gamma_{S/A}$, $\gamma_{S/W}$ and $\gamma_{W/A}$ are the surface energies between solid-air, solid-water and water-air, respectively, and θ is the contact angle between the mineral surface and the bubble.

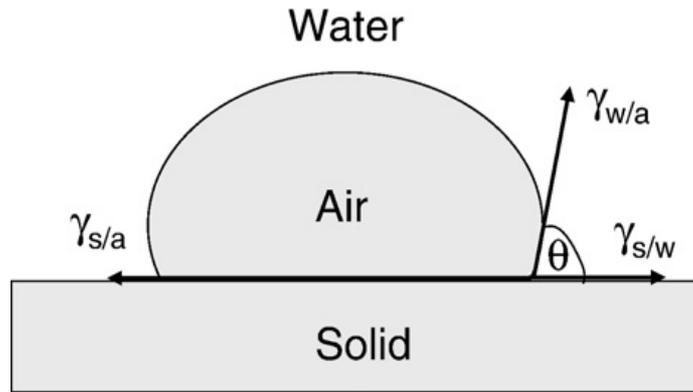


Figure 2.7: Schematic representation of the equilibrium contact between an air bubble and a solid particle immersed in a liquid (Wills, 2006:268)

Work of adhesion ($W_{S/A}$) force is required to break the particle-bubble interface and is equal to the work required to separate the solid-air interface and produce separate air-water and solid-water interfaces (Wills, 2006:268-269), i.e

$$W_{S/A} = \gamma_{W/A} + \gamma_{S/W} - \gamma_{S/A} \dots \dots \dots (2.2)$$

Combining equation (2.2) and (2.3) gives

$$W_{S/A} = \gamma_{W/A} (1 - \cos \theta) \dots \dots \dots (2.3)$$

It can be seen that the greater the contact angle the greater is the work of adhesion between particle and bubble and the more resilient the system is to disruptive forces (Wills, 2006:268-269).

2.5.2 Flotation mechanism

Flotation process mechanism involves attachment of mineral particles to air bubbles and then to be carried to the surface of the ore pulp, where they can be removed. For flotation processes to occur, the ore must be ground to a size sufficiently fine to liberate the valuable minerals from one another and from their associated gangue minerals. Three steps can then follow, namely: adherence of the desired minerals to air bubbles through the addition of flotation reagents, rising of the air bubbles in the ore pulp and formation of mineral-laden froth on the surface of the ore pulp and lastly the removal thereof (Wills, 2006:267-268; Fuerstenau, 1995).

Grinding of the ore might not be a part of flotation, but it does have an important bearing on the process. In grinding, the valuable minerals are nearly separated completely from the gangue minerals

and from one another so that optimum flotation results can be obtained. Usually ball mill or rod mill grinding is used in flotation though considerable gangue slimes may be formed which will complicate subsequent flotation steps (Wills, 2006:268; Fuerstenau, 1995).

Flotation machines produce mechanical agitation of the ore pulp and direct introduction of air under pressure, which are both responsible for a rising current of air bubbles (Wills, 2006:304-315).

2.5.3 Flotation reagents

Generally four types of reagents are used for effective flotation. They are activators, collectors, frothers, and depressant (Wills, 2006:268-269):

- **Activators**- Are specific compounds that promote collector adsorption onto surfaces that they could not attach to (Wills, 2006: 278).
- **Collectors**- Are organic compounds which render surfaces of valuable mineral particles water repellent. The collectors are found in two forms as ionising compounds that dissociate completely in water or non-ionising compounds that are insoluble in water. Ionising collectors are classed with the type of anion or cation that produces the water-repellent effect in water (as classified in Figure 2.8). Time is needed for the interaction of the collector with the particle surface. This is achieved by either chemically or physically bonding to the surface of the mineral. (Crozier, 1992; Jan, 1982; Wills, 2006:270-276).

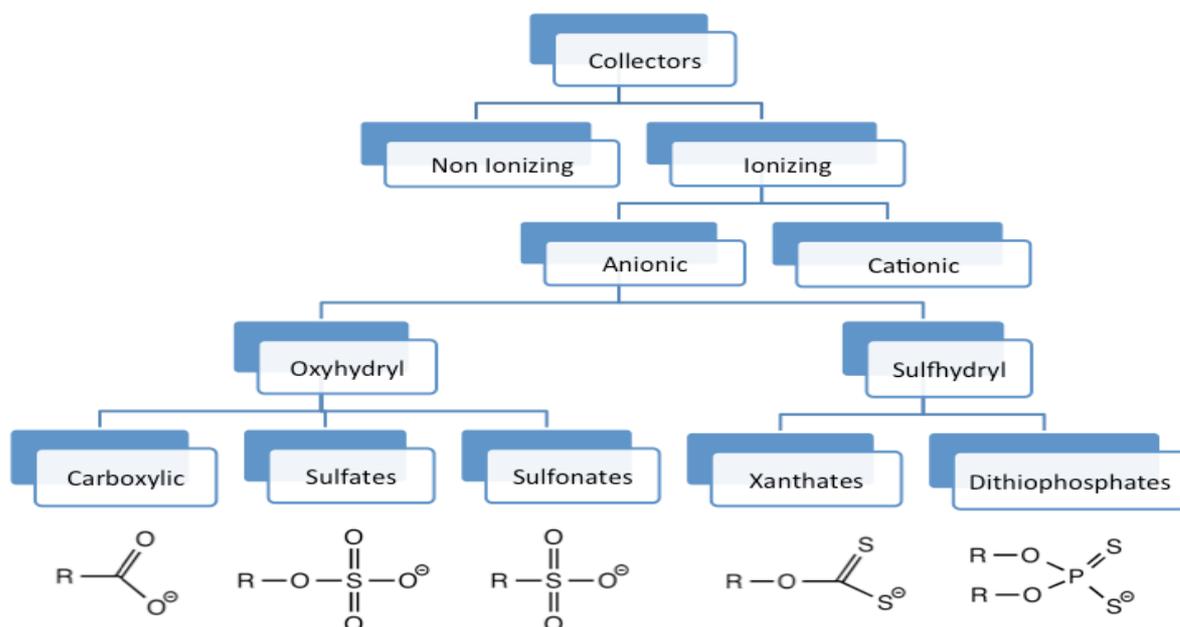


Figure 2.8: Classification of collectors (Wills, 2006:273)

- **Anionic collectors-** Are widely used and subdivided into oxhydryl (when the solidophilic group is based on organic and sulfo-acid ions), and sulfhydryl collectors, (when the solidophilic group contains bivalent sulphur) based on the structure of the solidophilic group. The newest version of anionic collectors is sulphides, R-S-R, containing an active sulphur atom with no other electron donors. It is noted that these collectors are something in between oxhydryl and sulfhydryl groups and unique due to sharing of sulphur (in the R-S-R) lone pair of electrons for bond formation with metals on sulphide mineral surfaces. The effectiveness of these collectors is strictly related to pulp chemistry because sulphur is the only electron-donating atom (Wills, 2006:272; Bulatovic, 2007:8).
- **Cationic collectors-** Are classified as organic compounds that have a positive charge when in aqueous solution. A nitrogen group with unpaired electrons (as structure shown in Figure 2.9) is the common element shared by all cationic collectors and covalently connected to hydrogen atom and hydrocarbon group. Flotation characteristics of amines are determined by change in the number of hydrocarbon radicals connected to nitrogen (Bulatovic, 2007:33).

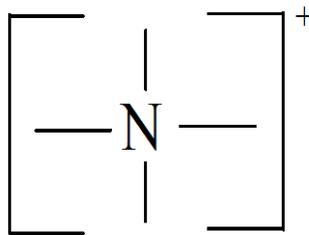


Figure 2.9: Nitrogen structure (Bulatovic, 2007:33)

Amines can be divided into primary, secondary, tertiary and quaternary (as illustrated in Figure 2.10) depending on the number of hydrocarbon radicals attached to the nitrogen bond. The quaternary amine is formed by the fourth hydrogen replaced by hydrocarbon group with A as an anion and usually chloride or sulfate.

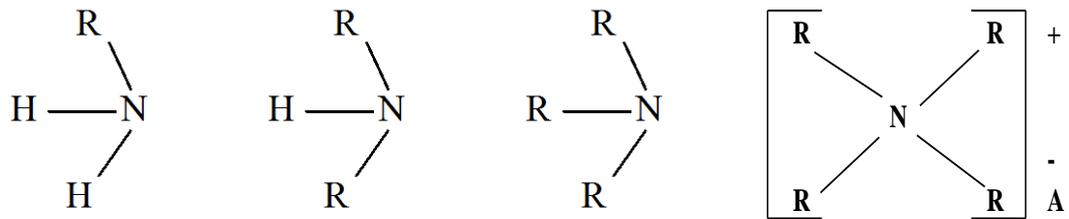


Figure 2.10: Structural formula of primary, secondary, tertiary and quaternary amines (Bulatovic, 2007:33-36)

Another classification of amines includes alkyl amines, aryl amines and alkyl-aryl amines according to whether the nitrogen atom is attached to a carbon atom of a chain or to a carbon atom of a cyclic structure or to both (Wills, 2006:276-278; Bulatovic, 2007:33);

- **Frothers-** Are heteropholic surface active reagents that can stabilise the bubble formation in the pulp to allow for selective drainage of entrained gangue minerals by stabilising the froth phase to a certain extent. Frothers can be divided into two categories that are slightly soluble (like aliphatic alcohols, alkoxy paraffin and natural oils like eucalyptus) or those that are completely soluble in water (like polyglycol and polyglycerol). The frothers play an important role in flotation. They stabilise the bubble formation in the pulp to allow for selective drainage of entrained gangue minerals by stabilising the froth phase to a certain extent (Wills, 2006:276-278).
- **Depressants-** Are used to selectively render certain minerals hydrophilic to avoid recovery in the concentrate (Bradshaw *et al.*, 2005:239-246). This means the undesired particles will not be able to attach to the surface of the air bubble and possibly selectivity of the process will be improved. There are two types of depressants; inorganic and organic (Wills, 2006:279-281). Examples of inorganic depressants include sodium cyanide, zinc sulphate, sulphuric acid etc. These are used when the properties of two or more particles are so similar that a collector can't selectively adsorb onto the surface of the valuable particle (Croezier, 1992:101-103). The natural polysaccharides (i.e. tannic acid, starch and glues etc.) or synthetically produced polyglycols are other types of depressant that can be used. Polymeric organic depressants have found their popularity use in flotation (Wills, 2006:279-281).

2.5.4 Parameters affecting flotation

Croezier (1992:101-103) mentioned that there are many variables which affect the flotation process, both directly and indirectly and listed up to 25 clearly identifiable parameters that can have an effect on performance of the process though it can be more fully described by many variables. Klimpel (1984) divided the major variables into three interactive groups, namely, equipment components, operation components, and chemical components as shown in Figure 2.11.

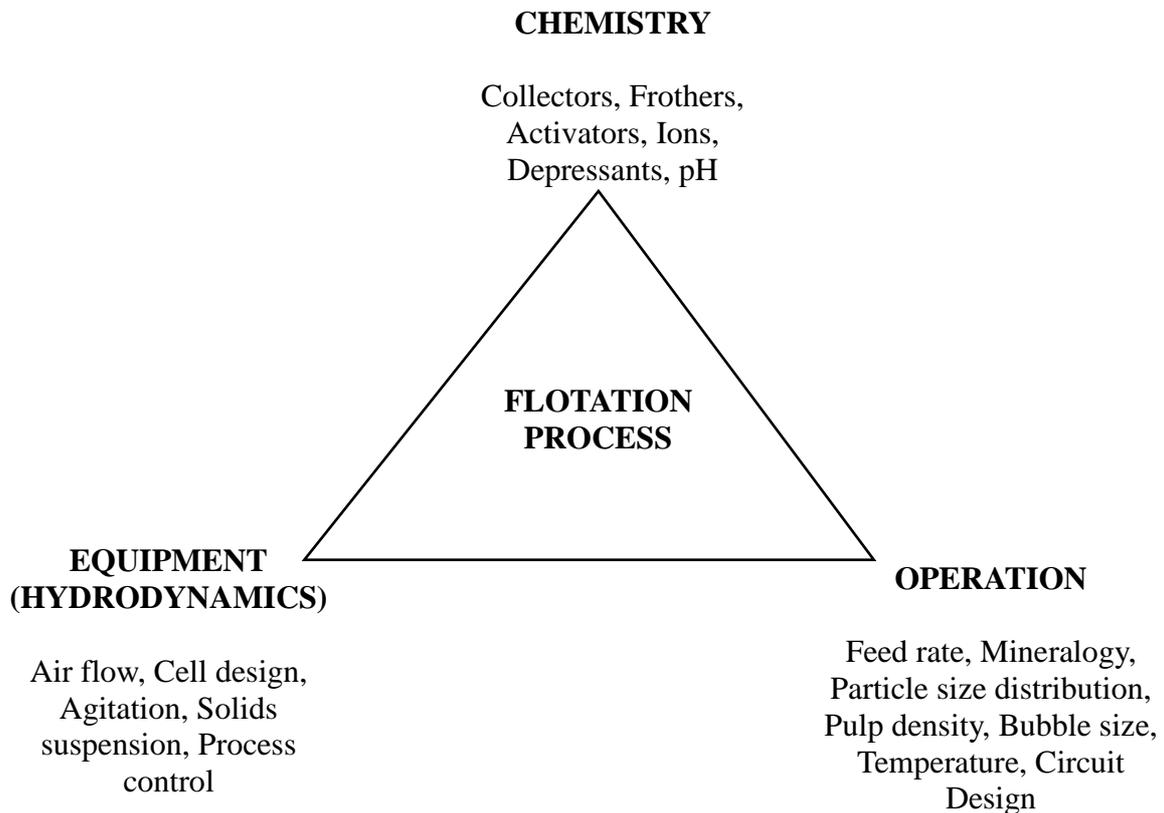


Figure 2.11: Summary of the variables in the flotation system (Klimpel, 1984)

2.5.5 Entrainment

In froth flotation, particles report to the froth phase either by true flotation or entrainment. True flotation is a process where a particle–bubble aggregate forms and subsequently floats to the froth phase (Trahar and Warren, 1976:289-327). Entrainment is a non-selective process by which particles report to the concentrate and occurs as a result of mineral particle concentration in the pulp which enters the froth phase (Savassi *et al.* 1998:243-246). The entrainment process generally occurs in

two-steps. Firstly, the mineral particles ascend upwards into the froth phase from the region below the pulp/froth interface, and secondly, the entrained particles in the froth are transferred to the concentrate in the water (Wang *et al.*, 2015:77-91). These processes can occur simultaneously with true flotation in a flotation cell. As a result, valuable and gangue mineral particles report to the froth. The mass transfer throughout a flotation cell is described in Figure 2.12 which shows (Savassi *et al.* 1998:243-246).

- 1) True flotation due to valuable mineral particles being transferred to the froth from the pulp
- 2) True flotation due to valuable mineral particles being transferred to the concentrate from the froth
- 3) Entrainment due to mineral particles being transferred to the froth from the pulp
- 4) Entrainment due to entrained mineral particles being transferred to the concentrate from the froth by entrainment
- 5) Mineral particles being transferred from the froth to the pulp due to the drainage of detached particles and entrained particles

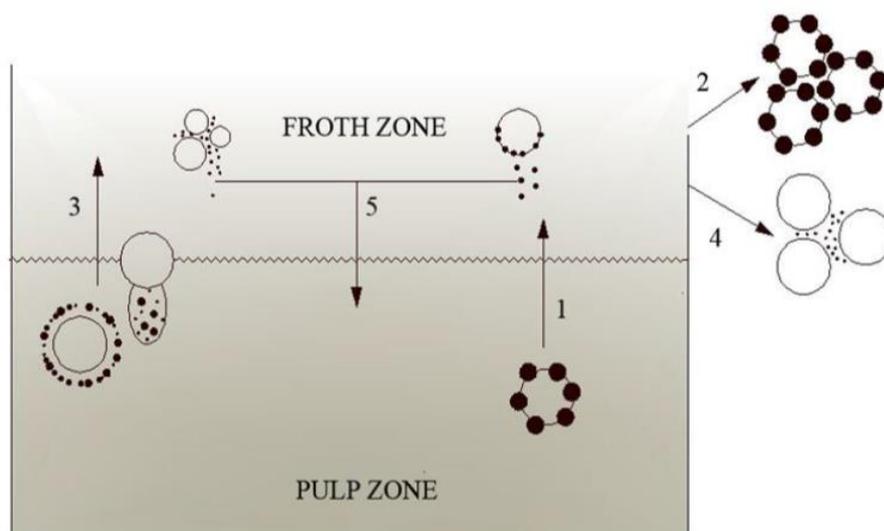


Figure 2.12: Mechanisms of transfer of fully liberated mineral particles in a flotation cell (Savassi *et al.* 1998:243-246).

As outlined in literature, three mechanisms known as: Boundary Layer Theory – transfer of entrained mineral particles to the froth phase in the bubble lamella, i.e. the thin hydrodynamic layer of water surrounding the bubble (Gaudin, 1957; Moys, 1978:21-38; Hemmings, 1981; Bascur and Herbst, 1982:17-23; Wang *et al.*, 2015:77-91); Bubble Wake Theory- transfer of fine mineral particles

suspended in the water to the froth phase in the wake of an ascending bubble (Smith, 1984; Yianatos *et al.*, 1988:279-292); and Bubble Swarm Theory – where water and suspended mineral particles move from the region below the pulp/froth interface to the froth, have been proposed to explain how mineral particles in the flotation cell travel across the pulp/froth interface from the pulp to the froth by entrainment.

Particle size and shape, pulp density, particle density, water recovery, froth structure and froth residence time are factors mostly affecting entrainment (Savassi *et al.*, 1998: 243-256; Smith and Warren, 1989:123-145). Suggestion was made that the ratio of the recovery of non-floating gangue to water can be used as a measure of entrainability or the degree of entrainment (Savassi *et al.*, 1998:243-246). The degree of entrainment has been shown to be a strong determining factor of the flotation separation efficiency between valuable minerals and gangue minerals, and is a function of time (Zhang *et al.*, 2006; Smith and Warren 1989:123-145). As particle size decreases, recovery by entrainment increases whereas for true flotation it decreases. Mineral particles smaller than 50 µm in size (which are referred to as slimes) are known to be easily recovered by entrainment (Savassi *et al.*, 1998). As in the pulp phase, fine particles have more chances to travel up through the froth to the concentrate as they are easily suspended in the water or the water film surrounding the bubbles in the region below the pulp/froth interface (Savassi *et al.*, 1998:243-246). Therefore, recovery of fine mineral particles to the concentrates with valuable minerals particles is detrimental to flotation as it leads to significant deterioration in the concentrate grade. This often happens in flotation of slimes, as the majority of the mineral particles have small particle sizes.

Wang *et al.*, (2015:77-91) described three methods that can be used to determine if recovery of minerals was by true flotation or entrainment. The methods are:

- **Trahar**- In which two batch flotation tests are used to measure the recovery of solid particles and water. In one test collector and frother dosages are varied and on the other frother only is the varied. Other parameters are kept constant on both tests (Trahar, 1981:289–327).
- **Warren**- Which involves two batch flotation tests conducted at identical time but with froth height and froth removal rate varied to change water recovery. Other chemical conditions kept the same (Warren, 1985:33-44).
- **Ross**- Where only one batch flotation test is used to quantify mineral particles recovery and occurs as function of time (Ross, 1989).

Models such as those of Maachar and Dobby (1992:167-172), Moys (1978) and Neethling and Cilliers (2002:123-134) have also been proposed to characterise and predict entrainment with the objective of better controlling or predicting flotation performance (Bisshop & White, 1976; Moys, 1978:21-38; Ross and Van Deventer, 1998:39-129; Maachar and Dobby, 1992:167-172; Savassi *et al.*, 1998; Neethling and Cilliers, 2002:123-134). The models are categorised as those that can predict entrainment flow directly or those that use degree of entrainment and water recovery to predict entrainment flow (Wang *et al.*, 2015:77-91).

2.5.6 The importance of pH

Generally, pH is a measure of the activity of H^+ and OH^- . In flotation pulps, adsorption of collector on specific mineral or activity of the modifying reagents is determined by the pH of the liquid phase. Interaction and behaviour of majority of species in the liquid phase including reagents is also determined by pH of liquid phase (Wills, 2006:282; Bulatovic, 2007:1-5). For slurried iron ore, pH is the most important factor as it defines the surface acidity of the mineral constituents in the slurry (Fuerstenau and Fuerstenau, 1982; Fuerstenau *et al.*, 1970). The surface site distributions of oxide minerals show that the surfaces can carry either a positive, negative or neutral charge depending on the pH of the aqueous medium. When the number of positively charged surface sites and the number of negatively charged surface sites are equal, the corresponding pH value is known as the point of zero charge (PZC). When the pH is above the PZC, the surface has a net negative surface charge. When the pH is below the PZC, the surface has a net positive surface charge. The PZC of each iron oxide and silicate minerals present individually has an effect on flotation behaviour when using a specific collector due to different PZC values of the same mineral from different ore deposits which can differ from synthesised minerals (Fuerstenau *et al.*, 1970). Table 2.1 shows the difference in PZC values of synthesised hematite and other various hematites determined experimentally. Table 2.2 shows different PZC values of certain silicate minerals determined experimentally (Fuerstenau and Fuerstenau, 1982).

Table 2.1: PZC values of hematite (Fuerstenau *et al.*, 1970)

Material	PZC	Method
Synthesised Hematite	pH 9.00	Titration
Synthesised Hematite	pH 8.40	Titration
Synthesised Hematite	pH 8.30	Electrophoresis
Hematite (Quabec)	pH 5.40	Titration
Hematite (Quabec)	pH 8.70	Titration
Hematite Brazil	pH 5.40	Stream potential
Hematite Brazil	pH 5.70	Titration
Hematite (minn)	pH 6.7	Electrophoresis
Hematite	pH 6.70	Electrophoresis

Table 2.2: PZC values of silicate minerals (Fuerstenau and Fuerstenau, 1982)

Silicate class	Minerals	Chemical formula	PZC
Sheet silicate	Kaolinite	$Al_4(Si_4O_{10})(OH)_8$	pH 3.40
Sheet silicate	Muscovite	$K_2Al_4(Al_2Si_6O_{20})(OH,F)$	pH 1.00
Frame work silicate	Quartz	SiO_2	pH 1.80-3.70

As outlined in literature, each different mineral surface has a different distribution of positively, negatively and neutrally charged surface sites at different pH values. Inherently, this causes each surface to carry a different surface potential, ψ , at each pH value. Figure 2.13 shows the surface potential of each mineral as a function of the pH value. The surface potential of the slurry particles attracts or repels ions in the process water and is responsible for effective reagent adsorption that drives separation processes such as flotation. The information presented in Figure 2.13 helps explain why certain collectors and surfactants have a narrow effective pH range and why cations and anions are attracted to and bond to surfaces in specific pH ranges (Cornell *et al.*, 1996).

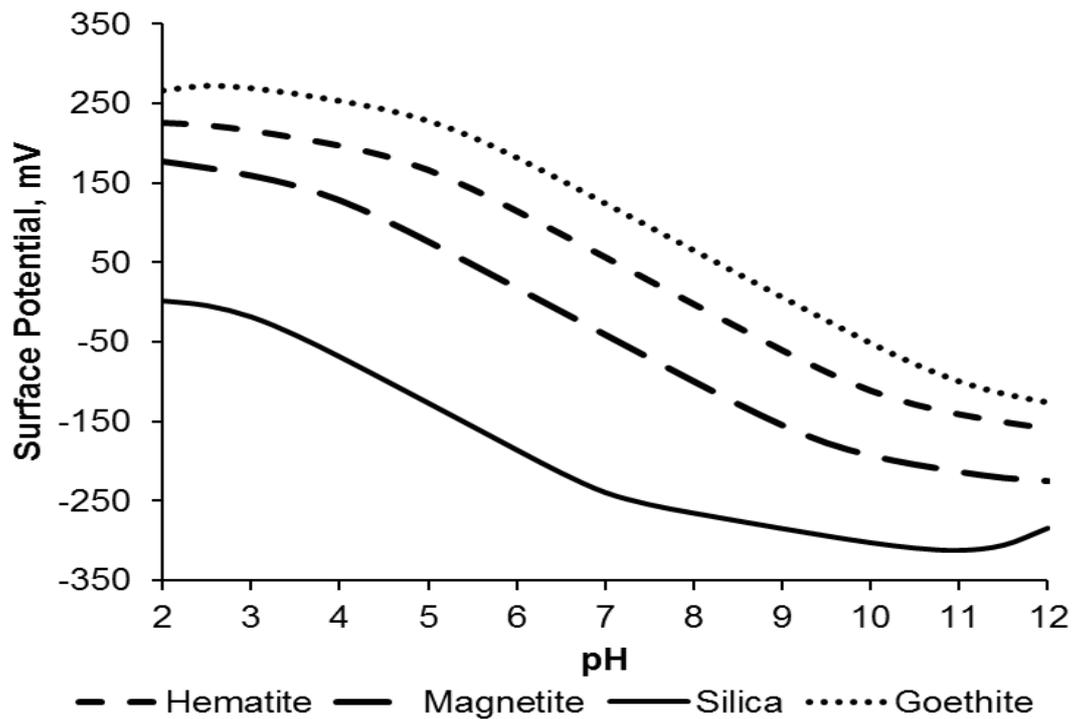


Figure 2.13: Surface potentials as a function of pH for Silica, Magnetite, Hematite and Goethite. Slurry particle characteristics measured from filter feed at hematite ore concentration plant. Site density for mineral surface sites found in literature. (Cornell *et al.*, 1996)

2.5.7 The effect of mineral liberation and particle size distribution

The flotation process can be limited by two main factors: mineral liberation (the process where ore is crushed to produce separate particles of either value or gangue mineral that can be removed from the ore) and particle size (which is used to compare dimensions of particles) as they are both interlinked due to the fact that the liberation of a specific mineral depends on the degree of intergrowth between the different solid mineral phases, which in turn depends on the grain size at which the individual particles are constituted of single minerals (Rao, 2004; Runge *et al.*, 2007:6-12).

The grind size of particles in the flotation feed can have a huge influence on the flotation process, which can be contributed to the fact that coarse particles have slow flotation kinetics because of their poor liberation and actual size, whilst for fine particles, their flotation kinetics is impeded by the poor flotation collision of the particles (Runge *et al.*, 2007:6-12).

The limit of a maximum particle size for flotation is usually dictated by the adhesion forces between the particle and the bubble/oil, which is dependent on the density of particles due to gravitational forces acting on the particles. Some flotation systems depict a reduced efficiency in flotation of particles smaller than 10 micron, but other systems rely on the readily floatable character of colloidal sized particles (Jameson *et al.*, 1977:722-736).

2.5.8 *The effect of impeller speed*

The change in impeller speed during flotation plays an important role as it determines the efficiency of the process. As in general for any given particle size, a high decrease in impeller speed results in particles not maintained in suspension and thus, settle significantly at the base of the cell. In a case of a high increase in impeller speed, the turbulence in the cell is sufficient to rupture the bond between particles and bubbles, and so recovery decreases (Fan *et al.*, 2010:317– 338). It is well accepted that slimes or fine particles have a low momentum due to having small mass; hence, they have a tendency of floating slowly (Ansari, 1997:95-97). Therefore, it is recommended that their low flotation rate can be increased by enhancing agitation (Sivamohan, 1990:247-288). The flotation rate is mainly governed by particle-bubble collision, particle-bubble attachment and particle-bubble detachment, as shown in Figure 2.14 (Fan *et al.*, 2010:317– 338):

- **Particle-bubble collision** – In which a particle has to collide with a bubble, reach a separation distance at which forces start to operate. The mechanism of particle-bubble attachment involves the evaluation of forces that cause a particle to deviate in its trajectory from liquid stream lines near the bubble surface and collide with a bubble;
- **Particle-bubble attachment** – Before a particle can attach to a bubble, a thin film of liquid between the particle and bubble needs to drain. These conditions are required, because once the intervening film becomes sufficiently thin it can rupture and allow the particle to penetrate the skin of a bubble. Therefore, successful attachment can occur if a stable three-phase contact has been established before the fluid stream line starts to diverge from the bubble.
- **Particle-bubble detachment** – A particle can only detach from a bubble due to the effects of stresses that are induced by the turbulence in the flotation cell. The process is dominated by the weight of the particle and the inertia of the particle during acceleration of the bubble that is induced by turbulent eddies in the fluid.

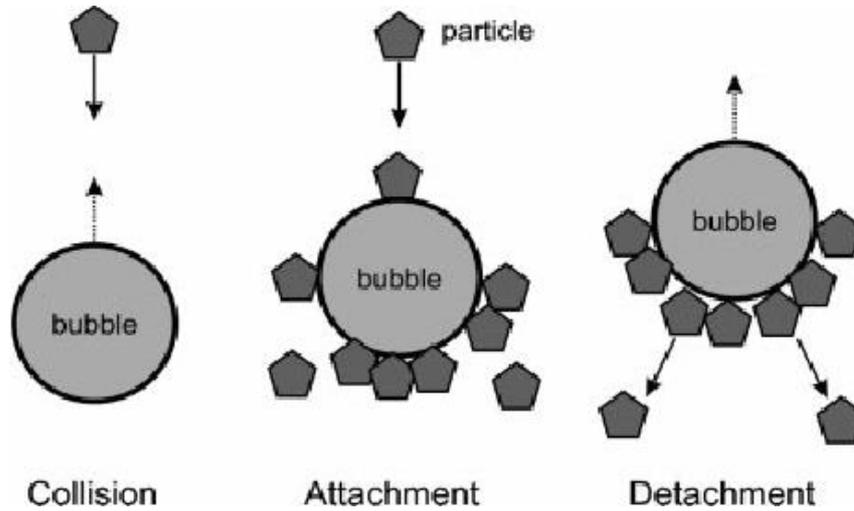
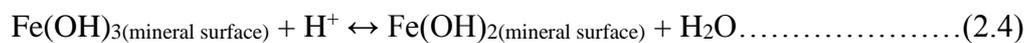


Figure 2.14: Schematic of bubble- particle collision, attachment and detachment (Fan *et al.*, 2010:317– 338)

2.6 The Physical and Chemical characteristics of Hematite and Quartz

The hematite (Fe_2O_3) structure consists of six oxygen atoms forming an octahedral group around a Fe atom, whilst each oxygen atom is in co-ordination with 4 Fe atoms (Partridge and Smith, 1971:229-234). Hematite is weakly magnetic and has a specific gravity of 5.3 to 5.6 and a hardness of 5-6 (on the Mohs scale). When hematite particles are immersed in aqueous solution, the OH^- ions from the solution interact with the Fe cations on the mineral surface forming the surface hydroxide $\text{Fe}(\text{OH})_3$. Hydration is then followed by the dissociation of the surface hydroxide, inducing an electrical charge on the mineral surface. The induced electrical charge is dependent on the pH of the solution (Fuerstenau *et al.*, 2007). Equation 2.4 and 2.5 represent the surface charge on hematite and show that the binding of a hydrogen ion on a hydroxide surface results in FeOH^{+2} groups dominating in acidic conditions, causing the mineral surface to be positively charged. For the mineral surface to be negatively charged, when pH is increased to alkaline, the hydroxide surface takes up hydroxyl ions forming FeO_2^- groups which actually dominate the mineral surface (Rao, 2004).



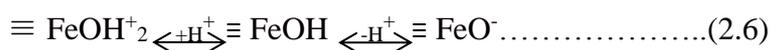
Quartz (SiO_2), possessing a specific gravity of 2.65 and a hardness of 7 (on the Mohs scale) consists of a crystal structure that has a silicon atom surrounded by four oxygen atoms forming a tetrahedral structure. The physical structure of silica is formed by a tetrahedron and bonds between the oxygen

and silicon atoms and is 40.0% ionic in character and about 60.0% covalent (King, 1982). The silicate framework is represented by quartz mineral's physical structure and consists of a three dimensional network of tetrahedrals formed from the polymerisation of the tetrahedral. The surface charge on the silica mineral is generated by chemical interactions, preferential dissolution of surface ions and lattice substitution. Like hematite, when quartz particles are submerged in aqueous solutions, the H⁺ and OH⁻ ions from the aqueous solution are adsorped onto the mineral surface's anionic and cationic sites. At equilibrium, the SiOH dissociates, behaving as Bronsted acid, releasing H⁺ ions to the solution and subsequently leaving the mineral surface negatively charged (Fuerstenau *et al.*, 2007).

2.7 Processing of Iron Oxides by Flotation

Intensive research on iron oxides flotation started in America during the 1930s (Ma, 2012:57). Beneficiation of iron oxides using flotation process depends on the nature of the gangue associated with the iron-containing mineral, which can be siliceous or acidic (rich in silica) and calcareous or basic (rich in calcium oxide). For flotation of iron oxides to occur, the iron particles are reduced to flotation size which results in particles having a high surface-to-volume ratio and a proportion of the atoms are located at the particle surface on breakage. The surface of iron atoms in the presence of water coordinates with hydroxyl ions, or water molecules, and result in particles acquiring an electric charge through ionisation of surface hydroxyl groups. The charge is dependent on protonation/deprotonation of the hydroxyl groups upon changes in pH of the solution, as shown in Equation 2.6. This charge on the iron particles influences the distribution of ions in the aqueous phase- where counter-ions are attracted or repelled towards the surface and co-ions to ensure that the interfacial region is electrically neutral. The electrical double layer is, therefore, formed from the charged surface and a layer of counter-ions in the solution phase. For oxide and silicate, potential determining ions (OH⁻ and H⁺) present on the electric double layer influence the charge created on the mineral surface. Hence, the layer's potential becomes zero when the surface charge is zero. The condition is known to be a point of zero charge (Fuerstenau, 1982:17-30; Fuerstenau, 1995; Shaw, 1992; Potapova, 2009; Fuerstenau and Raghavan, 1978).

Equation 2.7 shows the surface potential of oxides.



$$\varphi_o = \frac{RT}{Z_+F} \cdot \ln \cdot \frac{a^+}{a_o^+} \quad \text{or} \quad \varphi_o = \frac{RT}{Z_-F} \cdot \ln \cdot \frac{a^-}{a_o^-} \dots \dots \dots (2.7)$$

Where T is Temperature ($^{\circ}\text{K}$), R is gas constant ($\text{cal}/^{\circ}\text{K}\text{-mole}$), and F is Faraday constant ($96,500\text{C}/\text{mole}$). Z_+ and Z_- are vacancies of potential determining ions in solution inclusive of sign: a^+ and a^- as positive and negative activity of potential determining ions, and a_0^+ and a_0^- as positive and negative activity of potential determining ions. For oxides, the activities of H^+ ions in solution are considered, and at point of zero charge will be a^+ and a_0^+ , and under corresponding conditions a^- and a_0^- will be considered as the activities of OH^- ions. Thus, oxides in solution that are more acidic will carry a positive charge and a negative charge in solutions that are more alkaline (Quast, 2012: 62-63).

For iron oxides, surface charge plays an important role in the adsorption of ionic species at mineral-water interfaces due to electrostatic interaction. However, apart from electrostatic interaction, specific adsorption can occur due to the affinity of certain species to the surface. Specific adsorption can be either physical or chemical depending on the forces contributing to adsorption. Figure 2.15 shows the Stern model of the electric double layer with counter-ions in the liquid phase. In Figure 2.15, the Stern layer is represented by δ and it is where the counter-ions have the highest concentration and are bound to the surface. The concentration of counter-ions decreases beyond the Stern layer, until it reaches the bulk concentration. In the inner Stern layer, chemisorption of collector on mineral surfaces prevails, whereas, physisorption occurs on the outer layer. (Fuerstenau, 1982:17-30; Fuerstenau, 1995; Shaw, 1992; Potapova, 2009).

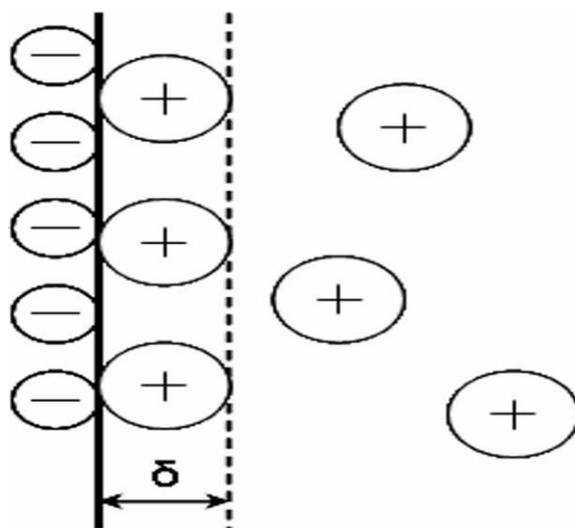


Figure 2.15: Electric double layer schematic figure at a liquid/solid interface (Potapova, 2009)

In physical-adsorption, molecules or ions from solution become reversibly associated with the surface and attachment is due to electrostatic attraction or Van der Waals bonding. The substances that are physically adsorbed can be desorbed from the surface if conditions such as pH or composition of the solution changes. The physical-adsorption process is less selective because collectors have a tendency of adsorbing on any surface that has the correct electrical charge or degree of natural hydrophobicity. For oxides and silicate minerals, collector adsorb on the surface physically due to electrostatic interaction with the knowledge of PZC minerals in question. In chemisorption, molecules or ions from the solution undergo a chemical reaction with the surface and become irreversibly bonded. As a result, the nature of the surface becomes permanently changed. The chemical-adsorption process is highly selective, because chemical bonds are specific to particular atoms. It is commonly noted, that during chemical-adsorption, formation of a monolayer and covalent bonding occur. The collectors can also adsorb on to oxide and silicate mineral surface by surface precipitation/reaction, which involves collector interaction with the mineral surface and with metal atoms that have moved from their lattice sites. Depressants are usually used to reduce the hydrophobicity of desired minerals (in reverse flotation) or undesired minerals (in direct flotation). For example, in reverse flotation adsorption of starch on to hematite minerals by chemisorption is desired to render hematite minerals hydrophilic (Fuerstenau, 1982:17-30; Fuerstenau and Fuerstenau, 1982; Rao, 2004).

The flotation of iron oxides is divided into three routes namely; direct anionic, reverse anionic and reverse cationic (Ma, 2012:57). In direct anionic flotation, iron oxides are floated from siliceous gangue minerals using anionic collectors such as fatty acids (mainly oleic acid) at pH values where the surface of iron oxide is positively charged or neutral. In reverse cationic flotation, silicate minerals are floated from iron oxides with cationic collectors such as mono amines or diamines, when their surfaces are negatively charged and iron oxides are depressed by starches. In reverse anionic flotation, quartz is first activated with lime and then floated with anionic collectors such as myristic, palmitric, caprylic and valeric and iron oxides are depressed by starches (Ma, 2012:57; Uwadiae, 1992:11-129; Yang, 1988:579).

Some iron oxides contain vast amounts of gangue minerals bearing phosphate and alumina, and flotation can be used as the beneficiation route. For example, in iron oxide containing calcareous phosphate bearing minerals as the main gangue, reverse anionic flotation occurs due to those gangue

minerals being floated from iron oxides with the use of modified fatty acids as collectors and sodium silicates or starch as the depressant (Ma, 2012:59; Smith, 1976).

2.8 Reagents used in Flotation of Iron Oxides

2.8.1 Fatty acids collector's mechanism and adsorption on hematite

The anionic fatty acids (mainly oleic acids) such as alkali sulphates, sulfonates and hydroxamate have been primarily used during flotation of iron oxides to float iron bearing minerals from siliceous gangue minerals. All these collectors are saturated aliphatic monocarboxylic acids and used at pH values less than 7 though sometimes the highest recovery of hematite is obtained at pH 7 and 8. The condensed molecular formula of oleic and linoleic acid is: $\text{CH}_3-(\text{CH}_2)_7-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{COOH}$ and $\text{CH}_3-(\text{CH}_2)_4-\text{CH}=\text{CH}-\text{CH}_2-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{COOH}$ respectively. Figure 2.15 shows the structural formula of oleic acid and Figure 2.16 shows the linoleic acid structural formula. These collectors belong to the eighteen aliphatic monocarboxylic acids homologous series which contain steric acids that are completely saturated; oleic acid with one unsaturated double bond and linoleic acid with two and linoleic acids with three non-conjugated double bonds. The dissolution of these fatty acids in water is accompanied by dissociation of the molecules into ions in a manner similar to that of inorganic acids (Buckenham & Mckenzie, 1961:450-454; Gaudin, 1957).

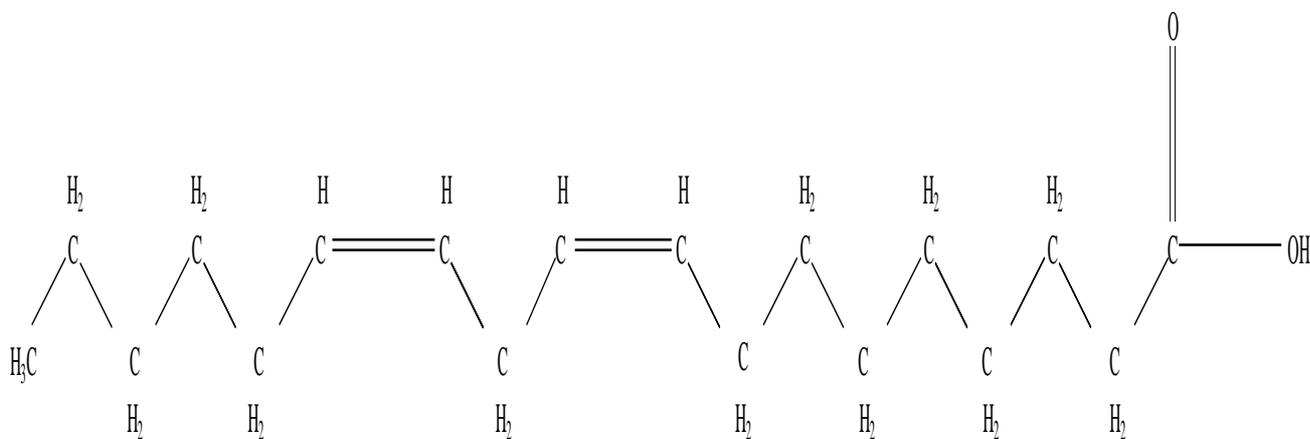


Figure 2.16: Structural formula of oleic acid (Gaudin, 1957)

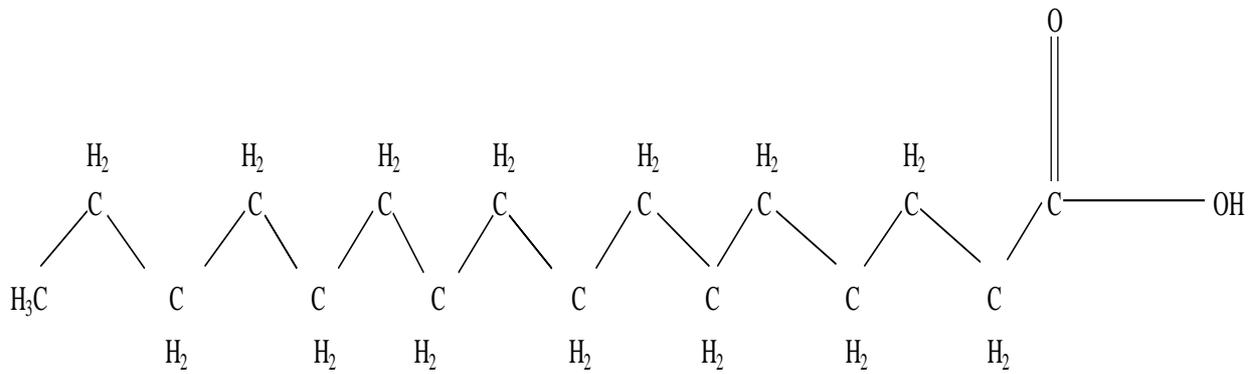


Figure 2.17: Structural formula of linoleic acid (Gaudin, 1957)

It is well accepted that the dissociated anionic portion of these fatty acids or its soap adsorb in the electrical double layer with the polar part of the chain closest to the mineral and the nonpolar part oriented towards the water (Gaudin, 1957). At low adsorption concentrations in the double layer, when the adsorbed species are not compressed by close neighbouring species into a fixed position, the hydrocarbon chain will most likely choose a horizontal orientation with respect to the surface (Jaycock and Ottewill, 1962:497). This orientation will be imposed on it by the surrounding hydrogen bonded water molecules into the structure of which the hydrocarbon chain does not easily fit. Flexible models of 18 carbon aliphatic monocarboxylic acids show that the carbon chain of stearic acid is straight, of the oleic acid is bent once and that of the linoleic acid is bent twice as shown in Figure 2.17. Thus, when adsorbed on a mineral, the area occupied by each acid increases with increasing unsaturation (Buckenham and Mckenzie, 1961:450-454). When the adsorption concentration of the fatty acid ions is high, the hydrocarbon chains have to assume a more vertical orientation with respect to the surface of the mineral so that more anions may be accommodated on it (Gaudin, 1957; Fuerstenau, 1982).

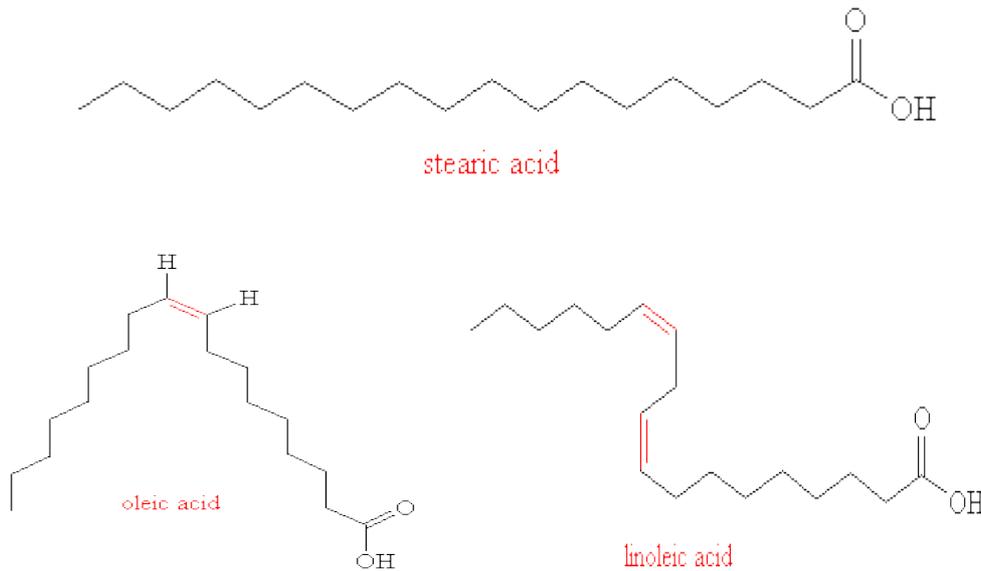
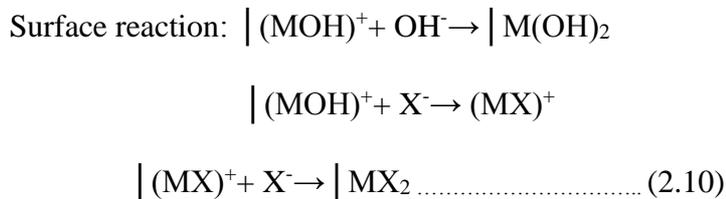
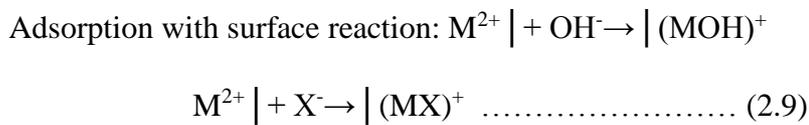
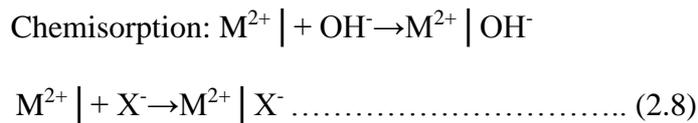


Figure 2.18: Structural representation of carbon chain length of stearic, oleic and linoleic acid (with the red highlighted section showing the bend of the chain) (Gaudin, 1957)

Fatty acids adsorb on hematite by chemisorption which is driven by covalent bond linkages at pH less than the isoelectric point. When a reagent chemisorbs, it can only reach monolayer status because the chemisorbing species dislodges the ion from the lattice. This process formed is, therefore, called adsorption with surface reaction. The surface reaction can take place by reagents reacting with hydrolysed species as shown in the reactions below (Gaudin, 1957):



Where M^+ presents cations, X^- represents anions and OH^- represents hydroxides.

2.8.2 Alkyl ether amine collector's mechanism and adsorption on silicate minerals

Amines are termed cationic organic derivatives of ammonium hydroxide and exist in cationic form below certain pH levels, and this is also consequently the basis of their collecting action (Lovell, 1982). The general structure of amines is $(R-(OCH_2)_3-NH_2)$, where R is the alkyl chain and can vary depending on the compound chemistry. The amines adsorb onto mineral surfaces through hydrogen bonding due to the presence of the $-NH_2$ group, in addition to electrostatic bonding via NH^{3+} cations that are present when ether amines dissociate in aqueous solutions. The covalent C-O bond within the amine molecular structure characterises amine reagents. Alkyl ether amines are formed through modifying aliphatic amines by insertion of ether $(O-CH_2)_3$ group. When alcohol reacts with acrylonitrile, alkyl ether monoamines are formed with the general formula of $R-O-(CH_2)_3-NH_2$. Alkyl ether diamines are formed by second reaction of alcohol and acrylonitrile acid form alkyl and characterised by the formula $R-O-(CH_2)_3-NH-(CH_2)_3-NH_2$. The distinct difference between the monoamines and diamines is the presence of the extra $NH-$ group which possesses an extra lone pair of electrons. The extra pair of electrons enhances the probability of hydrogen bond formations as the electrons are readily available for hydrogen bonding. The ionisation of amines in water occurs according to the following equation (Filippov *et al.*, 2014:62-69):

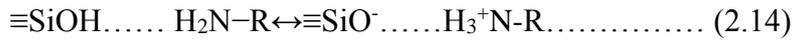
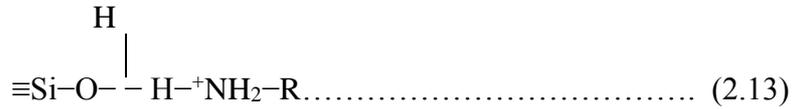


Equation 2.12 represents a saturated system where precipitation of the amine molecule occurs:



Alkyl amines perform best as collectors at slightly alkaline pH values in the range of pH 8 to pH 11 with quaternary ammonium salts at a wide pH range i.e. pH 6 to pH 12. In this pH range, there is a co-existence of the neutral molecular amine and its cationic species. The presence of the neutral molecules reduces electrostatic repulsion between cationic amine groups on the mineral surface, hence, more collector cations can adsorb onto the mineral surface. Such conditions ensure maximum collector coverage resulting in improved quartz collection and subsequent flotation (Viera & Peres, 2007:1008-1013). The co-adsorption phenomenon is best attained when there is 50.0% dissociation of the amine molecule, hence, at pH values equivalent to the collectors' pKa value, maximum flotation is possible (Gaudin, 1957; Yuhua and Jianwei 2005:116-122).

The maximum amine collector action on quartz can be found within a narrow pH range and is explained by three stages of adsorption: the initial transfer of collectors to the solid surface, which starts the contact angle development, region of rapid increases in the adsorption of amine species in association with pre-adsorbed species and finally the region of less active adsorption (Fuerstenau, 1957:365-367). Vidyadhar *et al.* (2002:59-72) proposed that the adsorption of molecular amines on silanol groups is achieved by charge transfer in the strong hydrogen bond between nitrogen atoms and the silanol group. The authors also described adsorption as taking place in three stage; hydrogen bond of ammonium cations with the silanol group according to Equation 2.13 (where the dashed line represents a hydrogen bond), hydrogen bonded to the silanol group together with the ammonium cation hydrogen bonded to the deprotonated silanol group produce monolayer patches of closely packed amine molecules (process occurs when the CHC is reached and is two dimensional, the equilibrium established between the amine molecules and cations is shown in Equation 2.14) and by attenuating the electrostatic repulsion forces of the cationic heads in the hemi-micelles, the neutral molecules alter the adsorption layer, increase the density of adsorped material in the monolayer and result in a bilayer.



Fuerstenau and Jia (2004:223-231) and Kou *et al.*, (2010:75-83) suggested that at low concentrations of amines; amines adsorb onto silicate minerals by physisorption through electrostatic interactions, at medium concentrations; hemi-micelles form and there is co-adsorption of cationic species with their molecular species at high enough concentrations, surface precipitation of the neutral molecule occurs on the mineral surface.

Since amines usually attach to minerals' surfaces with opposite charges (i.e. negatively oxidized surface), the condition in the flotation cell needs to be altered in such a manner that the pH, type of ions and concentration, allow only the surface of the mineral to be collected, to be negatively charged. Due to constraints in achieving such conditions in the cells, modifiers or depressants are used in conjunction with amines (Fuerstenau and Fuerstenau, 1982; Vidyadhar *et al.*, 2002:59-72).

Chain length and neutralisation degree are known as having effects on the performance of amine collectors (Scott and Smith 1991). Scott and Smith (1991) investigated the effect of chain length on amine collectors. For a specific set of amine concentrations, diamine collectors with longer chain lengths yielded better quartz flotation. This phenomenon has been attributed to the influence of the chain length on surfactant adsorption at the solid-liquid interface. At the critical hemi-micelle concentration, where there is enough alkyl amine collector to form micelles and collector patches on a mineral surface through van der Waals interactions, there are increases in contact angles and flotation recovery. Longer chain alkyl amines attaching through stronger van der Waals forces, easily form long hydrophobic aggregates of collector on the mineral surface in comparison to short chain alkyl amines. The result is the lowering of the critical hemi-micelle concentration, thereby, improving the flotation recovery and recovery rate (King, 1982; Rao, 2004; Rao *et al.*, 2001:26-29). Cassola *et al.*, (2011) in their investigation of amine collectors, the neutralisation degree effect was investigated. As high degrees of neutralisation deteriorate the flotation performance by limiting the formation of the collector/mineral complex even though solubility is much enhanced. The results obtained in the investigation demonstrated that maximum collector efficiency can result from achieving the optimum balance between: chain length, hydrophobicity and the degree of neutralisation.

2.8.3 Starch and dextrin depressants mechanism and adsorption on iron minerals

The starches and dextrin (with molecular formula of $C_{18}H_{32}O_{16}$) are defined as polymers of dextrose. These polymers of dextrose are structurally linked, for linear chains (amylose) through 1-4 glycosidic linkages and for branched points (amylopectin) 1-6 linkages. Dextrin is formed by fragmentation of chains and recombining as lower molecular weighted, but highly branched structures (Caesar and Radley, 1968). It adsorbs on minerals through chemisorption, physisorption or hydrophobic-hydrophobic interactions. Starch is used in its natural state, typically as maize, potato or pearl barley. It can also be used as partially hydrolysed which yields more soluble dextrans (Araujo *et al.*, 2005:219-224). It is well accepted that starch bond with hematite through chemisorption (Fuerstenau and Fuerstenau, 1982). Filippov *et al.*, (2014:66) also made a suggestion that hydrogen bonding is the underlying adsorption mechanism for starches with the oxide minerals, due to the presence of the hydroxyl group both in starch and mineral oxides. According to Peres and Correa (1996), high protein starches (such as zein), are as effective in depressing hematite, as conventional corn starch and amylopectin (Araujo *et al.*, 2005:219-224). In a study conducted by Pinto *et al.*, (1992:469-478) they compared amylose and amylopectin (the principal constituents of corn starch) with conventional starches. The study revealed that amylopectin is the constituent responsible for the central depressing action of hematite. Oil is also a crucial element of starches as high oil content is detrimental to the froth, according to theories of the elasticity of films which surround gas bubble (Araujo *et al.*, 2005:219-224).

2.9 Iron Oxides flotation routes

2.9.1 Direct anionic flotation

Iron bearing minerals (mostly hematite) are floated using anionic collectors such as petroleum sulphonate, fatty acids and hydroxamates and siliceous gangue such as quartz left in the tailings. For some low grade iron ores that contain a vast amount of quartz, this flotation route still appears desirable (Fuerstenau and Pradip, 1984; Ma, 2012:57; Araujo *et al.*, 2005:221; Filippov *et al.*, 2004:63-64). For instance, at Republic Mine, Michigan, U.S.A., a hematite ore is upgraded from 36.5% Fe to 65.4% Fe with a Fe recovery of 82.5% using the direct flotation route (Ma, 2012:57). In some cases this flotation route is facing difficulties in successfully reducing gangue minerals with the use of sodium silicate as a depressant and anionic fatty acids or amphoteric as collectors (Filippov *et al.*, 2004:63-64). Reagents that are mostly used are:

- **Collectors** - Fatty acids such as lauric acid, linoleic acid, oleic acid and tall (a distilled tall oil containing approximately 91.0% oleic and linoleic acids, 6.0% rosin acid and 3.0% unsaponifiables) are used as the collector of iron oxides, with dosages ranging between 0.5 and 0.7 kg/t (Yang, 1998; Ma, 2012:57-58). The adsorption on the surfaces of hematite is normally through chemical bonding and surface precipitation (Ma, 2012:58). Hydroxamates are also used and behave similarly to fatty acids in solution and their adsorption mechanism on hematite is classified as classical chemisorption (Ma 2012:58; Fuerstenau and Pradip, 1984). Fuerstenau *et al.* (1970) showed that it is possible to upgrade pure hematite from 42.0% to 67.0% Fe at a recovery of 19.0% using hydroxamates or fatty acids as collectors in direct flotation of iron ore.
- **Depressant** - Sodium silicate is usually used as depressant to selectively reduce silica and silica bearing gangue minerals and its depressant effect is highly sensitive to pH and it can sometimes exhibit a depressant effect in a narrow pH range (Fuerstenau *et al.*, 1967; Fuerstenau, 1995). Sodium silicate hydrolysis products are monomeric, polymeric, and colloidal species, depending on the pH value, concentration, and ratio of SiO₂ to Na₂O (sodium silicate modulus) (Lagerstrom, 1959:722-736; Ingri, 1959:758-775). Bench flotation tests were conducted on iron ore samples and it was observed that the use of sodium silicate as depressant could increase Fe content in the concentrate to more than 55.0% with recovery of more than 90.0% (Arantes and Lima, 2013:157-160).
- **pH regulator** – Hydrochloric acids is commonly used as pH regulator to render the pulp acidic because flotation is noted at acid (< pH 7) to neutral pH (pH 7-8.5) of the solution. The recovery of hematite by direct flotation reaches its maximum at neutral pH (Pope and Sutton, 1973).

2.9.2 *Reverse cationic flotation*

Reverse cationic flotation removes quartz using ether amine as a collector that has been partially neutralised by acetic acid conditioning or a combination of ether diamines and ether monoamine, while depressing iron oxide with the use of corn or dextrin starch in alkaline pulps adjusted by NaOH. In most cases, the pH ranges from 10 to 10.5 (Viera and Peres, 2010:124-128; Sastry *et al.*, 1976). Papini *et al.* (2001:1-5) performed numerous rougher bench scale flotation experiments on an iron ore from the Iron Quadrangle, Brazil using different cationic collectors: fatty mono-amine, fatty di-amine, ether mono-amine, ether di-amine, condensate, and kerosene combined with amine. Fatty

amines and condensates yielded concentrates with very high silica contents. Ether mono-amines proved to be more efficient collectors than ether di-amines for the ore investigated, in disagreement with the expectation that the presence of a second polar group would reinforce the collecting power. Concurrently for the same ore type, di-amines were more effective than mono-amines when utilised in conjunction with kerosene. Reagents that are commonly used are:

- **Collectors** – The amine group: ether amine, ether diamine and ether monoamine are collectors widely used to selectively float silica bearing gangue minerals and leaving the iron oxides in the pulp. The adsorption mechanism of amines on quartz is electrostatic in nature, stabilised by hydrophobic association of the hydrocarbon chains (Ma, 2012:58).
- **Depressant** - Starch is employed to improve the flotation selectivity of other silicate minerals by rendering the surfaces of iron bearing minerals hydrophilic. The starch depression action occurs due to its strong adsorption characteristics with mineral surface (Araujo *et al.*, 2005). There is a variety of starches derived from corn, tapioca, rice, potato, maize and other plants such as guar gum, and gum acacia. Soluble starch is widely used in depressing minerals like hematite and diaspore (Turrer and Peres, 2010:1066-1069). For example, corn starch has its huge application in many industries as the depressant for iron bearing minerals (Turrer and Peres, 2010:1066-1069). In Brazil, this starch is commonly used in flotation of iron ore, silvinitite, copper sulphide etc. (Peres and Correa, 1996). Similarly, the corn starch has been used in selective flocculation of mineral fines like bauxite, coal, phosphate, chromite, hematite and magnetite using starch as the flocculating agent (Wang and Ren, 2005:116-122; Pradip, 1994:179-194; Pradip *et al.*, 1993:1289-1294).
- **Frothers** – Are rarely used during the reverse cationic flotation of iron oxides, because the partially neutralised ether mono- and diamines used as collectors also act as frothers (Araujo *et al.*, 2005:223). However, several alcohol-based frothers were used during the reverse cationic flotation of iron ores in the USA and in Canada (Houot, 1983). According to Araujo *et al.* (2005:223-224) in Brazilian iron ore flotation, replacing approximately 10.0% of the total amine dose with polyglycol-type frothers, such as Flotanol D14 and Flotanol C7 increases the quartz recovery for the laboratory scale single mineral flotation. However, the results needed confirmation using industrial scale tests.
- **pH regulators** – Sodium hydroxide is used as pH regulator to render the pulp alkaline and enable attachment of silica bearing minerals to cationic collectors during flotation. The same

activator can also be used in the reverse anionic flotation of iron oxides (Araujo *et al.*, 2005: 219-224).

2.9.3 *Reverse anionic flotation*

In reverse anionic flotation, quartz is rejected by first activating it by using lime, and then floating it using fatty acids as collectors (Fuerstenau and Elgillani, 1966; Fuerstenau and Palmer, 1976:148-196). Historically, the anionic reverse flotation of activated quartz was a route employed for flotation of quartz when amines were not available to mineral processors (Ma, 2012:58; Filippov *et al.*, 2014:63-64). China's iron ore industry vouches for this process as it has achieved excellent results. Three reverse anionic flotation circuits were built in China's major iron ore area, Anshan in 1998. In 2003 the circuits processed 6.77 million tons of iron ore, with a feed grade of 29.9% Fe, a concentrate grade of 67.5% Fe, a tailings grade of 8.31% Fe, and a Fe recovery of 82.1% (Zhang *et al.*, 2006). Reagents commonly used are:

- **Collectors** – fatty acids such as myristic, palmitric, caprylic and valeric are normally used as collectors to float activated quartz (Araujo *et al.*, 2005:221; Filippov *et al.*, 2014:65-66).
- **Depressant** – the use of starches as depressants to selectively depress iron minerals and allowing silica gangue minerals to be floated are also used in the reverse anionic flotation (Araujo *et al.*, 2005:221-222).
- **Activator** – lime is normally used to activate quartz prior to flotation (Araujo *et al.*, 2005:221-222).

2.9.4 *The application of iron oxides flotation routes to process iron ore slimes*

The flotation of very fine particles is of interest nowadays regardless of the held view that they do not float (Pease *et al.*, 2005:831; Miettinen, 2007:5). According to Pease *et al.*, (2005:831-832), some ores must be ground to sizes below 10 μm to reach the required liberation degree for achieving concentrate grades within the specifications. For example "at Xstrata's McArthur River Mine – MRM, grinding to P₈₀ 7 μm is crucial to produce a saleable concentrate", and "at Mt Isa, grinding streams to P₈₀ of 12 μm and 7 μm is crucial to get adequate recovery". In these plants, creating slimes is definitely important for good flotation recovery. The overall flotation concentrate production of these concentrators reached 1Mt per year, with predominance of particles smaller than 10 μm , at

recovery levels higher than 80.0% (as shown by Figure 2.19 (the recovery–size curve for sphalerite from rougher concentrate at Mt Isa)) (Pease *et al.*, 2005:832). Therefore, it can be concluded that fine particles do float (Pease *et al.* 2005:832; Miettinen, 2007:5-6).

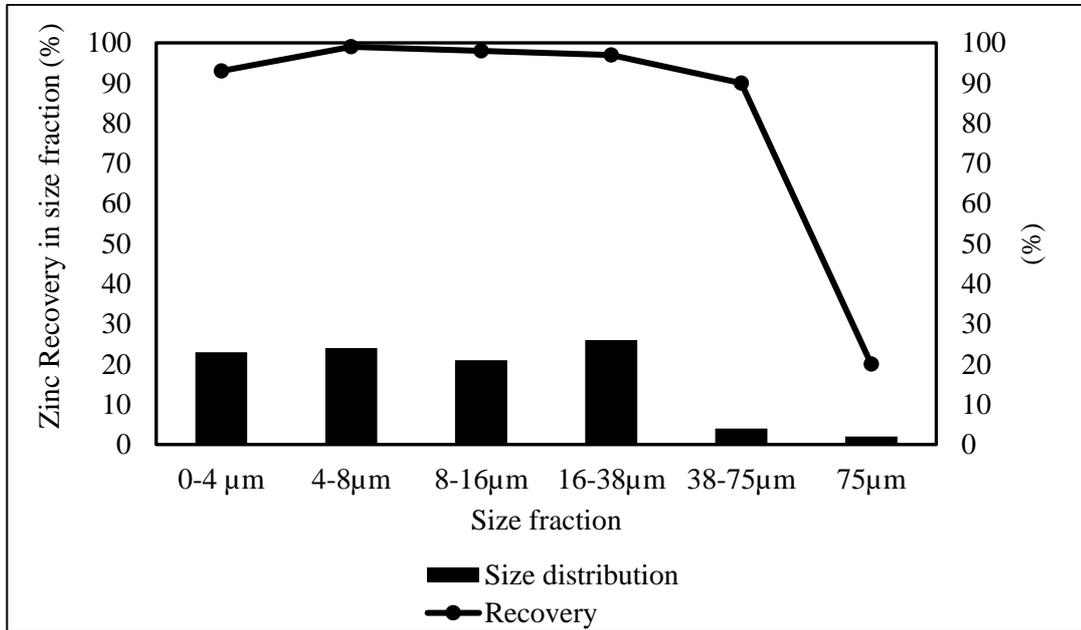


Figure 2.19: Mt Isa recovery from rougher concentrate by size (Pease *et al.*, 2005:831-832)

Literature shows that it is possible to beneficiate iron ore slimes by adopting flotation routes discussed in section 2.7.4 (Thella *et al.*, 2012:418-426; Das *et al.* 2005:737-742; Rocha *et al.*, 2010:842-845; Kumar and Gopalknshma 2013; Oliveira, 2006; Viera and Peres, 2010:124-128; Viera and Peres, 2007:1008-1013; Suresh *et al.*, 2012:5273-5281; Turrer and Peres, 2010:1066-1069; Tohry and Dehghani, 2016:28-33; Rocha, 2008). Of these routes, the most widely used is reverse cationic flotation. For example Thella *et al.* (2012:418-426) conducted two-stage reverse cationic flotation of iron ore slimes consisting of 80.0% of the particle passing 25 μm using a Denver flotation cell. The reagents used were amine collector at a dosage of 1000 g/t on the first stage and 500 g/t on the second stage to float silica bearing gangue minerals and corn starch at a dosage of 200 g/t to depress iron minerals. pH 9.5 was maintained throughout the experiments with pulp made of 20.0% solids. About 64.5 % Fe with 2.2% Al₂O₃ and 1.7% SiO₂ was obtained in the concentrates with Fe recovery of 28.7%. Similarly, Kumar and Gopalkrishma (2013) investigated the beneficiation of iron ore slimes made of 80% -45 μm particles by reverse cationic flotation using cationic collectors and maize starch.

Their results indicated that the use of cationic collectors increased iron content from 60.3% to 64.4% with silica of 3.5% and alumina of 2.1%. The recovery of Fe was 53.8%. Kumar *et al.* (2010:643-646) also performed reverse cationic flotation on Indian iron ore low grade slimes (80% -45.6 μ m) using a Denver flotation cell. The tests were conducted using different cationic collectors at various dosages in combination with starch as depressant. Among the collectors tested Sokem 524C (chemical composition not revealed) managed to increase iron content in the concentrate from 63.2% to 63.4% with silica of 2.5% from 5.3%. The tests were conducted on pulp made of 40.0% solids, pH 10, starch at a dosage of 1000 g/t, collector at a dosage of 300 g/t and impeller speed of 1250 rpm.

Reverse anionic flotation was commonly employed for flotation of iron oxides/slimes when mineral processors did not have access to amines (Filippov *et al.*, 2014:63-64). Even though, recently the processors have access to amines, the route is still found to be favourable. For example, Tohry and Dehghani (2016:28-33) investigated the use of sodium silicate by reverse anionic flotation of iron ore slimes (80%-39 μ m). The tests were conducted in a Denver flotation cell using Alky (fatty acids based) and Dinol as collectors, a mixture of NaOH;NaCO₃ as pH modifiers and sodium silicate as a depressant. Their results indicated that an iron ore concentrate made of 67.0% iron, 1.5% silica and 0.1% phosphorus can be obtained from a feed consisting 57.2% iron, 6.4% silica and 0.1% phosphorus.

According to Araujo *et al.* (2005:220), the direct anionic flotation route of oxides seemed attractive for processing of low grade iron ore although it faced difficulties in reducing gangue minerals such as quartz. Investigation was carried out on Indian iron ore slimes of D₅₀ 25 microns. The slimes were subjected to direct anionic flotation tests using fatty acids as collector at a dosage of 2000 g/t and pine oil as a frother. The tests were conducted with a pulp made of 20.0% solids at pH 9.5 and impeller speed of 1000 rpm. The results showed that Fe was upgraded from 58.28% to 62.9% with recovery of 57.9%. Silica and alumina were reduced to 2.9% and 2.7 % from 4.8% and 3.4% respectively (Thella *et al.*, 2012:418-426).

Direct anionic flotation of slimes might seem unfavourable due to its difficulty in reducing silica bearing minerals. However, the route is still employed by combining it with the famous reverse cationic flotation. For example, Thella *et al.* (2012:418-426) performed two staged flotation tests of

iron ore slimes (80% -45 μ m) with high alumina content. Reverse flotation was conducted in stage one by using amine collector, hexametaphosphate as a dispersant and starch as a depressant. Concentrates from stage one was further subjected to direct anionic flotation using fatty acids (stage 2) as a collector at a dosage of 4000 g/t and pH 9.5. A concentrate consisting of 63.5% iron, 2.7% alumina and 2.5% silica was obtained from 54.9% iron, 6.6% alumina and 7.5% silica.

2.9.5 Behavior of slimes during flotation

Discussion were presented (section 2.5.5, 2.5.7, 2.5.8 and 2.9.4) on the different behaviours of slimes in flotation with special attention to high energy flotation machine, special impellers, small bubble size, attritioning, different reagents and entrainment (Pease *et al.*, 2005:832; Ansari, 1997:95-96 Miettinen, 2007:1-2; Thella *et al.*, 2012:418-426; Das *et al.*, 2005:737-742; Rocha *et al.*, 2010:842-845; Kumar and Gopalknshma 2013; Oliveira, 2006; Viera and Peres, 2010:124-128; Viera and Peres, 2007:1008-1013; Suresh *et al.*, 2012:5273-5281; Turrer and Peres, 2010:1066-1069; Tohry and Dehghani, 2016:28-33; Rocha, 2008). The conclusion drawn from the discussion indicated that slimes can float. However, they respond differently because they have small mass which can result in fine particles being carried to the froth after being either entrained in liquid or mechanically trapped with particles being floated. The slimes have a tendency of consuming a lot of reagents during flotation which is due to their high surface area to volume ratio. The large surface area of the slimes or fines is normally accompanied by the high specific surface energy which causes collectors to adsorb non-specifically resulting in flotation of undesirable minerals. The slimes can be subjected to slime coating during flotation where fine particles of valuable mineral coat gangue mineral, and the grade of the concentrate becomes poorer. Inversely, when gangue mineral consists of fine particles and coat the coarse particles of valuable mineral, the attachment of the bubble to coarse particles are prevented which results in low recoveries. Slimes also tend to be more affected by water chemistry and ions in solution and have less momentum (and as results flotation rates will be lower) (Pease *et al.*, 2005:832; Ansari, 1997:95-96 Miettinen, 2007:1-2).

2.9.6 Approach of slimes flotation

Pease *et al.* (2005:834-840) designed good flotation principles for fines or slimes because they tend to not float well in many circuits due to being mixed with coarse particles and composites, needing more collector and flotation time. The principles designed involve:

- I. Achieving correct mineral liberation by projecting target grade and recovery

- II. Achieving target liberation by applying the most efficient grinding method, in the place that needs least grinding power.
- III. Making clean surfaces in grinding, and float as soon as possible before surfaces are oxidised again.
- IV. Minimising circulating loads and grinding cleaner feed rather than cleaner tail
- V. Designing launder and pumping systems for more tenacious froths made by fine particles.
- VI. Designing thickening and filtering for finer particles.

Sivamohan (1990:247-288) also revealed that the low flotation rate of slimes may be increased by enhanced agitation, keeping smaller bubbles, increasing the residence time in machine and providing counter current flows of particles. Figure 2.20 shows a simple conceptual solution—tailored to flotation conditions of different size distributions to achieve high recovery across many size ranges Pease *et al.*, 2005:834-840).

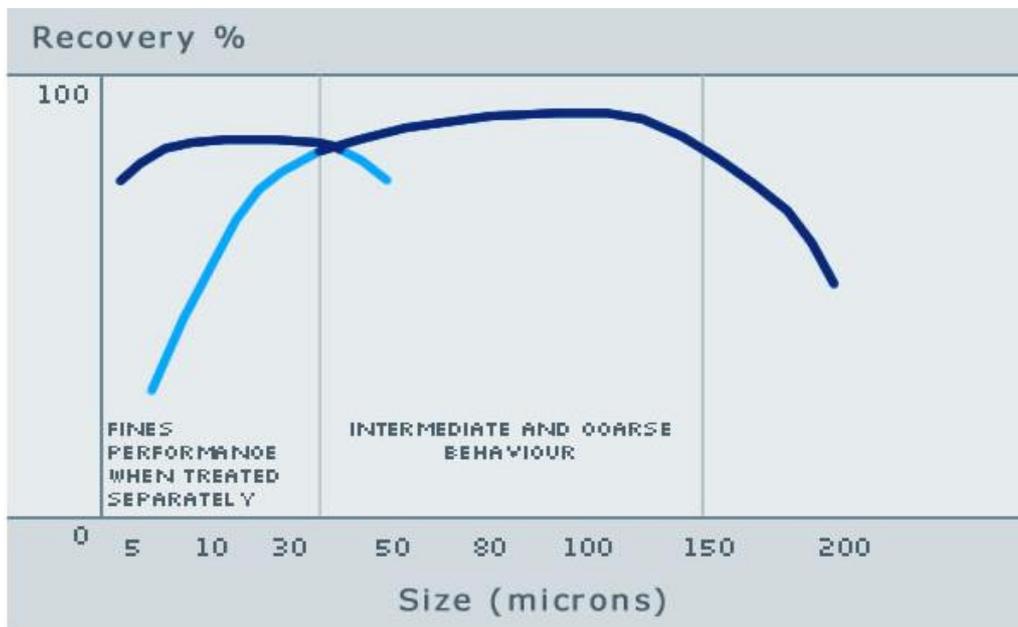


Figure 2.20: Conceptual staged grind-float circuit performance (Pease *et al.*, 2005:834-840)

2.9.7 Laboratory flotation tests

Batch flotation tests can be cheap and simple to perform in a laboratory that is equipped with a suitable laboratory flotation cell and equipment associated with the processing of flotation product. Runge (2010) conducted a research on flotation testing and recommended the following for bench scale flotation:

- I. Various bench scale cells may be used, whether top-driven (Denver D12) or bottom driven (Runge engineering)
- II. Samples should be floated as soon as possible after being collected at the froth
- III. The weight of the sample is independent on the required pulp density (which should be low)
- IV. The aeration rate is independent on the bubble-loading characteristics
- V. Water added in the test should simulate the actual plant water and
- VI. Aeration rate, impeller speed, froth depth and froth scraping intervals should be kept constant

Types of batch flotation tests that can be carried out are (Wills, 2006: 292):

- I. Rougher flotation to determine the best feed particle size distribution and to optimise rougher flotation chemicals and conditions
- II. Different stages of cleaning with or without re-grinding of the rougher concentrate to determine the number of cleaning stages and conditions required to achieve the required concentrate grades.
- III. Rougher and cleaning stages rate flotation to determine the sample's flotation kinetics and the best flotation times for rougher and cleaner flotation.

2.9.8 Laboratory flotation tests results interpretation

Runge (2010) announced that the ideal graphs to use for interpretation of batch-scale flotation tests are timed mineral recovery, grade recovery. Mineral curves indicate the difference in flotation time rate between fast-floating minerals. The mineral release curves can also be used for reagent evaluation, where increase in the flotation rate or the overall attainable amount of floatable mineral can be detected. For iron oxides flotation, the grade and recovery curve for iron values is commonly accompanied by silica values. Typical example is shown in Figures 2.21 and 2.22.

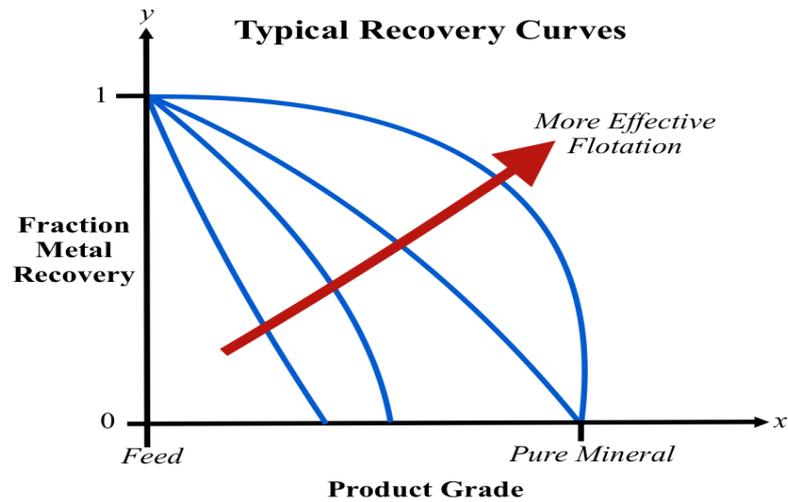


Figure 2.21: Grade vs recovery curve (Runge, 2010; Wills, 2006:296)

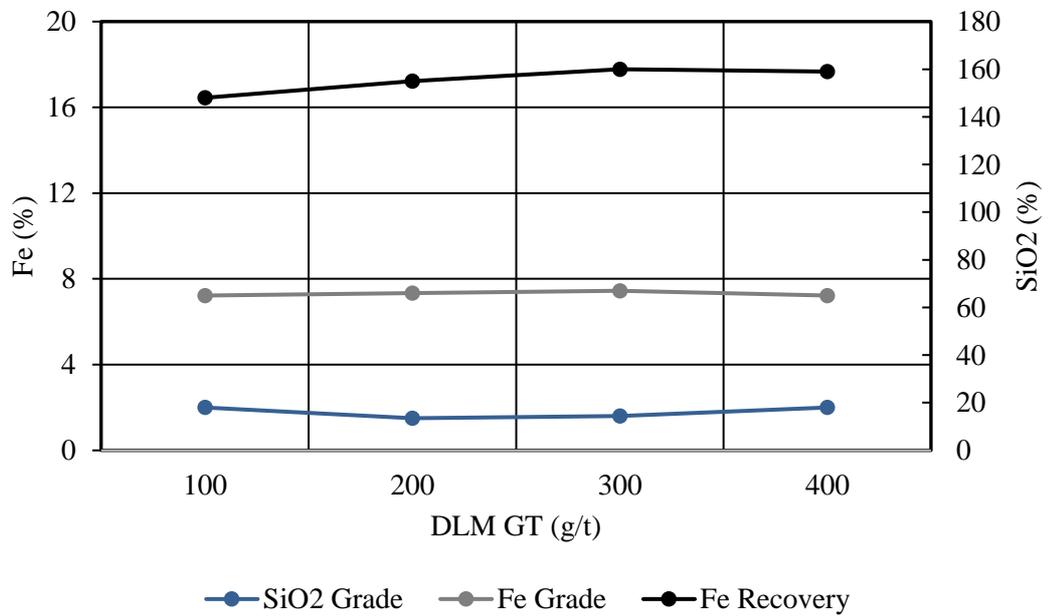


Figure 2.22: Effect of depressant in iron flotation (Praes *et al.*, 2013:3-5)

2.10 Magnetic Separation

2.10.1 Basic principle of magnetic separation

Magnetic separation is a physical separation of particles according to their different magnetic properties by combining tractive magnetic forces; gravitational, frictional or inertial, and attractive or repulsive inter-particle forces to act on those particles. Figure 2.23 represents the principal elements of the operation of a magnetic separator (Das and Roy, 2007:89).

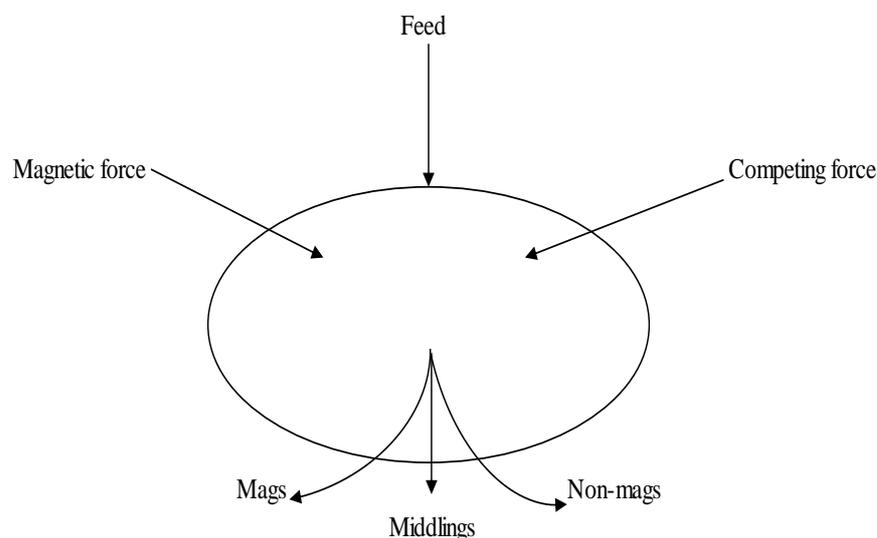


Figure 2.23: The principle elements of the magnetic separator operation (Das and Roy, 2007:89)

When the feed is fed into a magnetic separator the separation is done into two or more components; the concentrate produced is called the magnetic components and the tailings non-magnetic components. The other less magnetic components are called middling. Each of the second component streams must be transported into, through, or out of the device. The force tending to reduce degree of separation is magnetic and competing gravitational, friction, hydrodynamic, or inertial forces. Attractive inter-particle forces tend to reduce the degree of separation. Magnetic separation can be considered complete in limited cases, because chances are that during the separation magnetic and non-magnetic particles may be found in the tails, or middling (Oberteuffer, 1974:223-237; Das and Roy, 2007:92-93).

2.10.2 Mechanism of magnetic separation

Magnetic separation is a process used to separate magnetic materials from those that are less magnetic or non-magnetic. All materials have a response when placed into a magnetic field and those that are strongly affected (magnetised) are known as ferromagnetic those that are lesser affected are known as paramagnetic. Ferromagnetic materials attach to weak magnetic fields and devices used to separate these materials usually have magnets that are permanently magnetised and no electricity is required to maintain their magnetic fields. Paramagnetic materials require stronger magnetic fields that can only be achieved and maintained by electromagnet coils (Oberteuffer, 1974:223-237; Das and Roy, 2007:89-102).

2.10.3 Parameters affecting magnetic separation

Particle size of feed materials is one of the most important parameters affecting the separating effect of magnetic separators. The small particle size of feed-ore results in a high degree of mineral separation, which can obtain satisfactory separation indexes and the opposite is true for coarse particle size feed-ore. Slurry concentration (which mainly refers to the size of overflow concentration) is also one of the parameters affecting the magnetic separation process. High slurry concentration results in a high concentration that will have an effect on the quality of the concentrate. Low slurry concentration produces separation concentration that is too low and cause an increase in flow velocity separation. Therefore, the maximum slurry concentration used is normally 35.0% solids by weight, which is generally controlled at about 30%. Some other parameters include the specific assemblage of minerals and grades as well as their corresponding magnetic susceptibility, magnetic field intensity, and drum rotating speed (Dobbins *et al.*, 2007; Das and Roy, 2007:89-94).

2.11 Processing of Iron Oxides by Magnetic Separation

In magnetic separation processes, iron ore is separated from the non-magnetic associated gangue materials by taking advantage of the difference in the magnetic properties of the different materials. The magnetic separation operations can be conducted in either dry or wet environments which can either be low or high intensity. Low intensity magnetic separators use magnetic fields of 1000 to 3000 gauss and are mostly used to beneficiate magnetite ore as it is inexpensive. High intensity separators use magnetic fields as strong as 20,000 gauss and are used to separate weakly magnetic iron ores such as hematite, from nonmagnetic or less magnetic gangue materials (Das and Roy, 2007:89-95). Types of magnetic separation technologies commonly used for iron oxides are:

- **Wet and dry, low intensity magnetic separation (LIMS):** This process is used to concentrate ferromagnetic particles such as iron of abrasion, magnetite and some pyrrhotites. The limited feed size is up to 3.175 mm or even finer with field strength of 1000 to 3000 gauss. Two well-known application are the concentration of magnetic taconite ores and the recovery of magnetite media in heavy media separation plants. For example, LIMS separators are installed at the Kiruna plant KA1 in Sweden to process a low phosphorus magnetite ore containing over 60.0% Fe (Das and Roy, 2007:95-96).
- **Wet high intensity magnetic separation (WHIMS):** this process is used for the separation of weakly magnetic materials from non-magnetic materials such as beneficiation of hematite

and goethite from their associated gangue minerals, separation of ilmenite, wolframite and chromite from gangue minerals, and ferro-oxides and ferrotitanium oxides from cassiterite, zircon and rutile. The slurry is fed into the magnetic matrix, which is contained in a stainless steel ring moving at controlled speed between the poles of the powerful stationary electromagnets. The key features of WHIMS are exceptional performance on even weakly magnetic materials, high magnetic field intensity, middling splitter setting with field of 15 000 to 20 000 gauss (Das and Roy, 2007:96-97; Oberteuffer, 1974:223-225). Prasad *et al.* (1988:102-112) have studied iron ore slimes processed by magnetic separation at the Kiriburumines in India. A concentrate containing 63.0% Fe and 3.3% Al₂O₃ with an iron recovery of 56.0% was obtained when using WHIMS with slimes composed of hematite, goethite, and kaolinite after the classification via cyclone.

- **High gradient magnetic separation (HGMS):** here use is made of a matrix of shaped iron pieces which produce high field gradients to act as collection sites for paramagnetic particles in the field strength of 20 000 gauss or more. The frequently used matrixes to form the high gradient sites are balls, rods, grooved plates, expanded metal and fibres. The high gradient magnetic separators also use the uniform field of a solenoid. The core is filled with a matrix of secondary poles such as ball bearings or wire wool to obtain the high gradient (Xiong *et al.*, 1998:111-127; Oberteuffer, 1973; Zeng and Dahe, 2003:219-224). Xiong *et al.* (1998:111-127) explained that conventional wet high gradient magnetic separator/wet high intensity magnetic separator (WHGMS/WHIMS) can face several problems. The most common problems are matrix clogging and mechanical entrainment of non-magnetic particles which weaken the magnetic forces and reduce the quality of the magnetic product. These problems can be solved by employing pulsating high gradient magnetic separation (PHGMS) such as sLon pulsating high gradient magnetic separator (PHGMS) which has been in development since 1981 (Xiong *et al.*, 1989:947-952; Liu *et al.*, 1991:152–159; Yang *et al.*, 1993:211-221; Xiong *et al.*, 1998:111-127; Zeng and Dahe, 2003:219-224).

2.11.1 The principles and mechanism of sLon pulsating high gradient magnetic separator

sLon PHGMS beneficiate fine weakly magnetic particles by utilising the combined force fields of magnetic force, pulsating fluid and gravity. Slurry is fed into the feeding box and enters the ring through slots in the upper yoke. Magnetic particles are flushed out into the concentrate box by being attracted from slurry onto the surface of the matrix and brought to the top of the ring. Due to passing

through the matrix, the non-magnetic particles enter the tailing box through slots in the lower yoke as results of action contributed by combination of slurry pulsation, gravity and hydrodynamic force (Xiong, 1997:89-100). The ability of sLon to beneficiate fine, weakly magnetic particles is due to its feature of the vertically rotating ring and the slurry pulsation mechanism. The separator also has a higher beneficiation ratio, matrix cannot be easily clogged and it is flexible and adaptable to various particle sizes compared to other magnetic separators (Xiong, 1997:89-100; Xiong, 2002:1-8; Xiong *et al.*, 1998:111-127; Zeng & Dahe, 2003:219-224; Xiong *et al.*, 1989:947-952; Hearn and Dobbins, 2007).

2.11.2 Operating parameters affecting sLon pulsating high gradient magnetic separator

The sLon pulsating high gradient magnetic separator is well known to beneficiate fine, weakly magnetic particles. However, the working efficiency of the magnetic separator can mainly be challenged by operating parameters such as: Feed velocity- due to its influence on the hydrodynamic competing force on particles in the matrix; as it increases, recovery and separation efficiency decreases, slurry pulsations- as it increases the rate of collisions between the magnetic particles and the matrix, favouring the recovery of magnetic particles. Competitive forces acting on the non-magnetic particles are also increased by pulsation. Therefore, increasing pulsation velocity increases the grade of magnetic particles (Xiong, 1997:89-100; Xiong *et al.*, 1998:111-127; Xiong *et al.*, 1989:947-952; Hearn and Dobbins, 2007).

2.11.3 The application of magnetic separation techniques to process iron ore slimes

Historically, magnetic separation techniques were used for removal of tramped iron from a variety of feed material and concentration of iron ores. The techniques have also found its wide application in processing of iron ore slimes (Oberteuffer, 1974:223). For instance, an investigation was carried out on three iron ore slimes generated at Barsua, Bolani and Meghahatuburu iron ore mines of India. The slimes were treated by hydrocyclones and wet high intensity magnetic separator. The results indicated that iron concentrate of 61.0% - 65.0% Fe could be obtained by recovering 58.0% - 78.0% of the iron value present in the slimes. The alumina and silica levels were brought down to 2.5% and 1.0% respectively (Das *et al.*, 2002:285-289). Prasad *et al.*, (1988:102-112) also beneficiated iron ore slimes produced from washing plants and tailings ponds of Kiriburu mines in India using WHIMS followed by classification in a hydrocyclone. The results showed that a concentrate assaying 63.0% iron and 3.3% alumina could be produced with an overall iron recovery of 56.0%. Similarly, Das *et*

al., (1994) used hydrocyclones followed by wet high intensity magnetic separation to beneficiate iron ore slimes from Barusa, Bolani and Kiriburu in India. The results obtained indicated that a concentrate assaying 60% to 65% Fe with 60% to 80% recovery can be obtained. Rao (2004) studied recovery of iron values from iron slimes of Donimala Tailing Dam. The study was carried out by subjecting the ore slimes to hydrocyclones followed by magnetic separation. A concentrates assaying 65.9% Fe, 1.5% SiO₂, 1.9% Al₂O₃ and recovery of 60.0% from feed of 49.4% Fe, 13.5% SiO₂ and 8.4% Al₂O₃.

High gradient magnetic separator devices have been developed to extend the useful application of magnetic separation to very weakly magnetic materials of small particle size (Dobbins *et al.*, 2007; Zheng and Dahe, 2003:219-224). Several industries and researchers are using the high gradient magnetic separators. For example, Gushan Iron Mine use sLon PHGMS to process hematite ore. The ore is first ground to 48.0% -0.074 mm using a ball mill. Four wet high-intensity magnetic separators (SQC WHIMS) are used as rougher to remove the non-magnetic particles, magnetic particles are reduced to about 87% -0.074 mm, then subjected to two WHIMS (used as cleaner) and two SLon-1500 (as scavengers). The total results showed that grade and recovery of iron concentrate was 2.9% and 13.8% higher than the ones obtained from gravity flowsheets (Xiong *et al.*, 1998:111-127).

2.11.4 Laboratory magnetic separation tests

There is a variety of laboratory magnetic separation devices used, the most commonly used is a high intensity dry device which uses a rotating iron disc to concentrate the magnetic field and remove the magnetic particles from the nonmagnetic. The device consists of an electromagnet which produces a magnetic field across the air gap and a belt which passes through the air gap. Mineral particles are fed from a vibrating feed on to the belt and through the magnetic field. The magnetic particles are attached to the disc above the belt and from there removed by the disc's rotation. The non-magnetic particles are collected at the end of the belt (Das and Roy, 2007:89-102; Oberteuffer, 1974:234-235).

2.11.5 Laboratory magnetic separation tests results interpretation

Oberteuffer, (1974:224) explained that efficiency of magnetic separation can be expressed by both recovery – ratio of magnetic material in the mags relative to that in the feed, and grade – fraction of magnetic material in the mags. These independent measures are mostly employed for the separators effectiveness. A typical example of the graph is shown in Figure 2.24.

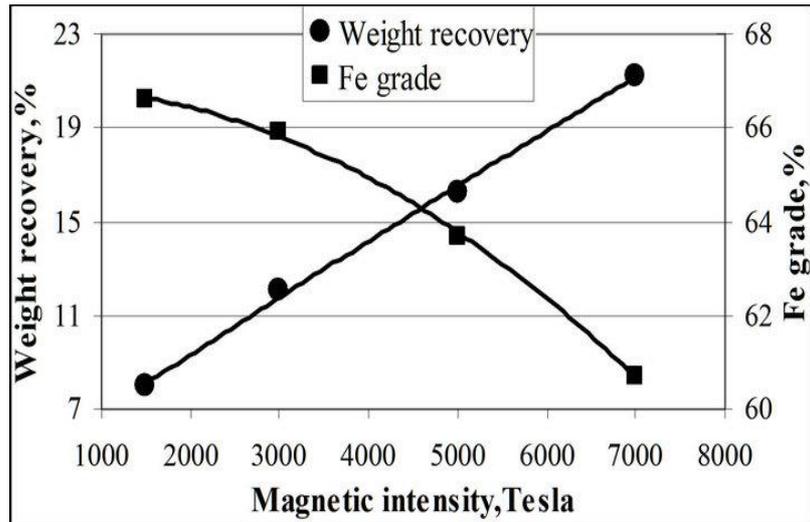


Figure 2.24: Influence of magnetic field intensity on product weight recovery and Fe grade (Umadevi *et al.*, 2012:55-60)

2.12 Iron making

In the iron making process, iron needs to be reduced from the oxidation state by carbon (which is referred to as a reducing agent) as it has greater affinity for oxygen (Sundholm *et al.*, 1999:381–546; Turkdogan and Fruehan, 1999:37–160). Carbon (C) reacts with the oxygen associated with the iron as solid carbon, dissolved in a metal phase, or present in a gaseous phase as carbon monoxide gas (Burgo, 1999:699-740;). The source of C is coal and anthracite, or products derived from them. There are different technologies available to exploit these reactions, with the blast furnace being the main technology (Burgo, 1999:669-740; Feinman, 1999:741-780). The furnace consists of a refractory-lined steel shaft in which a charge (iron ore, sinter, or pellets; coke; and limestone or dolomite) is continuously added to the top through a gas seal. Pre-heated air is blown into the shaft near the bottom of the furnace and coke is combusted to produce carbon monoxide which produces iron from iron ore. Limestone is used to flux silica and alumina in the ore and coke ash to form a slag that absorbs much of the sulphur from the charge. Molten iron and slag are then tapped from the hearth at the bottom, drawn off and processed. Pig iron is also removed and transported to steel mill operation for further processing in either an electric arc furnace or basic oxygen furnace (Burgo, 1999:669-740).

Iron ores contain not only Fe but also other elements such as silicon (Si), manganese (Mn), phosphorus (P), aluminium (Al), magnesium (Mg), calcium (Ca), titanium (Ti), potassium (K),

Sodium (Na) and Sulphur (S), which are present at higher oxidation states in the form of minerals (Burgo, 1999:669-740; Poveromo, 1999:547-642). During the iron making process, the oxidation states of some of these elements – especially Si, Mn, and P – are reduced to zero. Apart from Fe and C, the liquid pig iron tapped from a blast furnace also contains Si (1.5%), Mn (1.0–2.0%) and P (< 0.4 %) as major components. The coal and anthracite used, as source of carbon, also contain other elements in the form of gangue minerals – most importantly S. This S, together with the S present in the iron ore, will remain in the pig iron, resulting in S contents of 0.1% or less, typical for liquid pig iron tapped from a blast furnace (Burgo, 1999:669-740).

2.13 Steel making

The production of steel, suitable for various applications, requires refining of the liquid pig iron tapped from a blast furnace for reduction of C, Si, Mn, S, and P present in the pig iron. In the first refining step, O₂ gas is blown under controlled conditions into the liquid pig iron, where it reacts with C, Si, and Mn (Fruehan, 1998), increasing their oxidation states from zero to C²⁺ or C⁴⁺ as carbon monoxide (CO) or carbon dioxide (CO₂) gas, and to Si⁴⁺ as SiO₂, and Mn²⁺ as MnO, both of the latter compounds presenting themselves to a liquid slag phase. Phosphorus is also oxidised from zero to a higher oxidation state, and sulphur to S²⁺ by forming CaS through a process controlled by temperature and slag chemistry. Both P and S occur in the slag phase. Similarly as for iron making, different technologies are available for steelmaking to exploit these reactions, with the basic oxygen furnace (BOF) being widely applied in the treatment of liquid pig iron (Fruehan and Nassaralla, 1998:743-759; Jones *et al.*, 1998:525-660; Miller *et al.*, 1998:475-524). During tapping from BOF, the refined steel is separated from the slag. Since after oxygen blowing, the dissolved O content of steel is too high for casting purposes a reduction process is needed. This reduction is achieved by adding Al or Si, which react with the dissolved O to form Al₂O₃ or SiO₂. The reaction products report to the slag phase under ideal conditions and remain in the steel as non-metallic inclusions under non-ideal conditions.

Other steel producers actively manage the inclusion content and morphology of their products through the introduction of Ca additions and soft purging with argon (Ar) gas. The treatment occurs in the Ladle Furnace (LF) which includes further reduction of the S content of the steel through synthetic slag additions as well as final adjustments to the chemical composition of the steel. During tapping of the steel from the primary vessel (typically a BOF) or arcing at the ladle furnace, N in the air dissolves into the steel. Many of the alloys and synthetic slag components added to the steel at the

ladle furnace contain water, which has the potential to increase the dissolved hydrogen content of the steel. In many instances the levels of these gases have to be reduced with VOD technology – one of the technologies available. Vacuum Oxygen Degassing Technology (VOD) is also used to reduce the carbon content of ultralow carbon steel grades (Kor and Glaws, 1998:661-713).

2.14 The effects of Silica and other impurities in Iron and Steel making

The silica used in steelmaking depends on the intended purpose of the addition, the practice involved, the condition of the steel, the aim residual silicon level, and the experience and preference of the individual operator. The presence of high silica and other impurities on the ore to be charged to the furnace results in high addition of limestone and the furnace is blocked (Burgo, 1999:669-740). The other effects of silica in the form of silicon and other elements on the steel properties are discussed in Table 2.3.

Table 2.3: The effects of elements on steel properties (Kor and Glaws, 1998:661-713; Burgo, 1999:669-740)

Element	Positive effect	Negative effect
Silicon	It is a principal deoxidizer; helps remove bubbles of oxygen from the molten steel and used to produce steel in the range of 0.4-1.0% Fe.	High amounts of silicon can decrease ductility which will result in cracking problems. For galvanising, more than 0.04% of silicon in steel can affect the thickness and appearance of galvanised coating
Aluminum	It is also use as a deoxidizer and help form a more fine-grained crystalline microstructure	High aluminum content in steel will results in a decrease of steel grade's toughness

Sulphur	It improves machinability It has an adverse effect of impact properties on steel	It has an adverse effect of impact properties on steel
Phosphorus	Increase the tensile strength of steel and improves machinability.	It has embrittling effects. Its content in steel is limited in the range of 0.03-0.1% P.

2.15 The technologies of Steel making applied in South Africa

Arcelor Mittal South Africa is one of the biggest steel making companies that source their high grade iron ore lumps (64% Fe) and sintered ore fines (63.5% Fe) from Kumba Iron Ore's Sishen mine which supplied almost 6.25 Mt per annum from an agreement that was made in 2011 (Sishen mine, 2012). The company has four operating steelworks named after the towns they are based in. Vanderbijlpark Works is the largest integrated steel plants and utilises blast furnaces for the production of pig iron and rotary kilns for DRI (ArcelorMittal, n.d.-a; ArcelorMittal, n.d.-b). Lumpy iron ore and sintered ore fines are used in pig iron production and in DRI. BOF is used for the production of primary steel (Fruehan and Nassaralla, 1998:743-759; Jones *et al.*, 1998:525-660; Miller *et al.*, 1998:475-524). Secondary steelmaking is conducted in ladle furnaces, followed by degassing in either a RH or VAD. Other three plants are Saldanha Works, Newcastle Works, and Vereeniging Works.

2.16 Conclusion

The production of high grade iron ore is being driven by the ever increasing use of steel for which iron ore is needed. In view of the fact that high grade ore is being depleted, processing of slimes to recover iron values is of importance to meet the rapidly growing demand of steelmaking. Processing of slimes will not only serve to provide economic benefit but environmental and ecological hazards will also be prevented.

Froth flotation is one of the mineral processing methods known for beneficiation of mineral particles below 150 microns by exploiting their difference in physical properties. However, parameters such as particle size and size distribution, pulp density, reagents and agitation are of importance as they

have a huge effect on flotation to attain target concentrate grade. Test investigations are also necessary as flotation responses vary among deposits from different mining areas for the same mineral.

CHAPTER 3

3. METHODOLOGY

3.1 Materials

3.1.1 *Sampling and collection*

Two samples were used for investigation, namely direct process slimes (DPS) and magnetic separation processed slimes (MSPS) from Kumba Iron Ore's Sishen mine located in the Northern Cape Province of South Africa. The direct process slimes were generated by secondary screening in which the underflow product was fed to the hydrocyclones. The overflow product of the hydrocyclones was then fed to the thickeners for water recovery. Thereafter, thickener's underflow products were sampled in the form of slurry into 25 litre buckets for test work purposes.

The direct process slimes was used to produce magnetic separation processed slimes using two-staged sLon Pulsating High Gradient Magnetic Separation (PHGMS). The operation was conducted in two stages by adopting the sLon operation (as discussed in section 2.10.1) where about 100 kg/h of slurry (at 30.0 wt.% solid) was introduced to a 2 mm rod matrix which agitated the slurry at a pulsation rate of 250 rpm. The rougher concentrate (from first stage namely rougher sLon) was subjected to the second stage (cleaner sLon) where the rod matrix and pulsation rate was increased to 3 mm and 350 rpm respectively, to produce the cleaner products. The cleaner fraction was then sampled into 25 litre buckets for flotation test work purposes.

3.1.2 *Pulp and solid density determination*

Hematite is a dense ore with a specific gravity of 4.9-5.1 hence it can be concentrated by methods such as DMS. Therefore, it is important to determine the density of the hematite slimes sample before tests are conducted on it. To determine the density of the pulp, a 500 ml beaker was weighed and the scale tarred off to remove the weight of the beaker. Thereafter, the slurry was added to the beaker and weighed to give a mass of 738.65 g. The mass of the slurry was noted carefully and the density of the pulp was calculated by:

$$D = \frac{m}{v} \dots \dots \dots (3.1)$$

where D-density, m-mass and v-volume (Wills, 2006). Therefore, density of the pulp was:

$$D = \frac{738.65 \text{ g}}{500 \text{ ml}}$$

$$= 1.477 \text{ g/ml}$$

$$1 \text{ g/ml} = 1000 \text{ kg/m}^3$$

Therefore 1.477 g/ml = 1477 kg/m³

The density of the pulp was verified by using Mercy's pulp density scale (as shown in Figure 3.1) and gave density of ~ 1483 kg/m³. The density of the pulp was continuously checked daily using the pulp scale to check if it has changed due to the possibility of solid settling in the pulp that might affect the percentage solid of the slurry.



Figure 3.1: Mercy scale

To determine the density of the solid, a 50 ml density bottle was first washed with acetone to remove traces of grease and dried at 40 °C. After cooling, the bottle and stopper were weighed in a balance and the weight was recorded as M₁. A dried sample (~ 10g) was added to the bottle and the weight was recorded as M₂. Distilled water was then added to the bottle till half full and placed in desiccator to remove air entrained within the sample. The vessel was evacuated

for ~ 2 minutes. Thereafter, the bottle was removed from the desiccators and topped up with distilled water. The stopper was then inserted into the bottle (as shown in Figure 3.2 (a)) and put on the balance and the mass was recorded as M_3 . Afterwards, the sample was washed out and the bottle refilled with distilled water (as shown in Figure 3.2 (b)), put on the scale and the mass was recorded as M_4 (Wills, 2006).



Figure 3.2: (a) Density bottles with iron ore slimes (b) Density bottles with distilled water

The density of the solid was calculated by (Wills, 2006:41-43):

$$S = \frac{M_2 - M_1}{(M_4 - M_1) - (M_3 - M_2)} \times D_f \text{ kg/m}^3 \dots\dots\dots (3.2)$$

$M_1 = 29.22$, $M_2 = 41.22$, $M_3 = 90.80$, $M_4 = 81.86$. Therefore, the density of the solid was:

$$\begin{aligned} S &= \frac{41.22 - 29.22}{(81.86 - 29.22) - (90.8 - 41.22)} \times 1000 \\ &= 3.92 \times 1000 \\ &= 3920 \text{ kg/m}^3 \end{aligned}$$

The test was conducted in duplicate to check reproducibility and eliminate any errors that might have occurred. The results obtained were:

$M_1 = 34.66$, $M_2 = 46.60$, $M_3 = 92.20$, $M_4 = 83.39$.

$$\begin{aligned} S &= \frac{46.60 - 34.66}{(83.39 - 34.66) - (92.20 - 46.60)} \times 1000 \\ &= 3.82 \times 1000 \\ &= 3820 \text{ kg/m}^3 \end{aligned}$$

Therefore, an average of the two densities was calculated by:

$$\begin{aligned} \text{Average} &= \frac{3920 + 3820}{2} \\ &= 3870 \text{ kg/m}^3 \end{aligned}$$

Thereafter, the percentage solid of the slurry was calculated by (Wills, 2006:41-42):

$$X = \frac{(100s(D-1000))}{D(s-1000)} \dots\dots\dots (3.3)$$

Where, X= percentage solids by weight D= pulp density (kg/m³) and s= density of solid (kg/m³). Therefore the percentage solid of the slurry was:

$$\begin{aligned} \%S &= \frac{100(3870) X (1477 - 1000)}{1477 X (3870 - 1000)} \\ &= 43.55\% \\ &= 44.0\% \end{aligned}$$

To calculate percentage moisture of the filter cake, the following equation was used (Wills, 2006:41-42):

$$\% \text{ moisture} = \frac{\text{wet weight} - \text{dry weight}}{\text{wet weight}} \times 100 \dots\dots\dots (3.4)$$

$$\begin{aligned} \% \text{ moisture} &= \frac{406.004 - 325.006}{406.004} \\ &= 19.950 \\ &= 20.0\% \end{aligned}$$

3.1.3 Sample preparation

Upon receipt, the DPS and MSPS samples were weighed and stored in suitably closed containers to avoid contamination. The stored DPS (in the form of slurry) was well stirred daily to prevent solids from settling which might agglomerate and have an effect on the percentage solids in the slurry. The MSPS was air dried whenever test work needed to be conducted to prevent moisture absorption from the environment.

Sub-samples of both slimes were then prepared for:

- **Mineralogy** – For direct process slimes, a sub-sample of 500 ml was filtered and the filter cake was air dried. The sub-sample was well blended and three 50 g representative sub-samples prepared by cone and quartering and labelled to be analysed by SEM and XRF. The same procedure was followed for magnetic separation processed slimes.
- **Particle size and mineral department analysis**- About two, four litre samples of DPS and MSPS were poured into separate containers and closed to avoid contamination. This was used for particle size distribution and mineral department analysis.
- **Froth flotation** – For direct flotation on DPS, about 2 litres of the slurry was sub-sampled for test work. For reverse flotation on DPS, the sub-sample of the slurry was first filtered to produce a filter cake consisting of 20.0% moisture (as shown in section 3.1.2). To obtain a slurry of 60.0% solids, the filter cake was weighed (taking into account the moisture content) to give 1385.85 g of wet mass which was thereafter mixed with 419.09 g of water. The pulp was agitated in Denver D12 machine (as shown in Appendix B Figure 7.2) to allow it to mix well. A similar procedure was followed when diluting pulp to 40.0%, 35.0% and 25.0% solids by weight as mass of the filter cakes and water shown in Table 3.1 (with consideration of the volume of the cell). For MSPS, lumps were broken off the air dried samples and flotation feed made of 25.0% solids by weight.

Table 3.1: Pulp preparation for flotation tests

Wt. % solids	Mass of filter cake (g)	Mass of water (g)
60.00	1385.85	419.09
40.00	1385.85	1371.56
35.00	1200.00	1650.00
25.00	864.00	2036.00

3.1.4 Reagents

For direct flotation, two fatty acid anionic collectors were used to float iron bearing minerals namely tall oil (containing approximately 91.0% oleic and linoleic acids, 6.0% rosin acid and 3.0% unsaponifiables) and linoleic acid. The structural formula of oleic and linoleic acid is

represented as $\text{CH}_3-(\text{CH}_2)_7-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{COOH}$ and $\text{CH}_3-(\text{CH}_2)_4-\text{CH}=\text{CH}-\text{CH}_2-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{COOH}$ respectively. The collectors were used undissolved in water. For reverse flotation, two diamine cationic collectors were used to float silica namely Lilafлот D817M with active constituent- 1,3-Propanediamine, N-[3-((C₁₁-C₁₄,C₁₃-rich)oxy)propyl]) and Flotigam 3135 with active constituent- Dipropylene diamine, R-O-(CH₂)₃-NH-(CH₂)₃-NH₂). The choice of using cationic collectors was based on quartz flotation which is supported by the statistical analysis of the data pertaining to flotation tests conducted under standard and identical conditions using these reagents and they are well known for floating quartz at medium to coarse sizes (Kumar and Raju, 2015). Detailed MSDS of the reverse flotation reagents is shown in Appendix A. In preparation for use in flotation tests, the collectors were made of 1.0% solution (meaning 1 g per 100 ml of distilled water) (Akzonoble; Clariant; Protea Chemicals).

Dextrin and corn starch were used as depressants for hematite. The starches were prepared by weighing approximately 2.5 g of sodium hydroxide (NaOH) in a previously weighed 500 ml beaker containing a magnetic agitation bar, adding approximately 300 ml of distilled water, and putting the set on the magnetic stirrer. The solution was allowed to mix well. About 10g of the starch was also weighed in a previously weighed 1000 ml beaker containing a magnetic agitation bar to which about 600 ml of distilled water was added and placed on the magnetic stirrer. The solution was stirred until well dissolved. Both the starch and NaOH solutions were poured into a 1000 ml beaker with magnetic stirrer, and the heat turned on until the solution started boiling. Thereafter, the solution was allowed to cool, and approximately 100 ml of distilled water was added to make up a 1000 ml solution. The ratio of starch: NaOH was four parts of starch to one part of sodium hydroxide. A separate 5.0% sodium hydroxide solution was prepared (5 g NaOH in 100 ml of distilled water) to be used as a pH regulator (Araujo *et al.*, 2005:219–224).

3.2 Tests products handling

All products produced from flotation tests were washed into a filtrate chamber and filtered in a laboratory pressure filter (with 4-3 micron filter paper), before being dried overnight in a laboratory oven at 90° C. Once dry, each product was weighed and recorded before a

representative sample was taken and further sub-sampled using cone and quartering before subsequently packaged for XRF analysis.

The pressure drum filter used is shown in Appendix B Figure 7.4, operated at 5 bar (abs), to filter wet ore samples. Compressed air was delivered via a tube. A bucket was placed underneath to collect the filtrate or as a sample safety mechanism in the event of filter paper failure.

3.3 Experimental Work

Figure 3.3 shows a summary layout of the experimental plan.

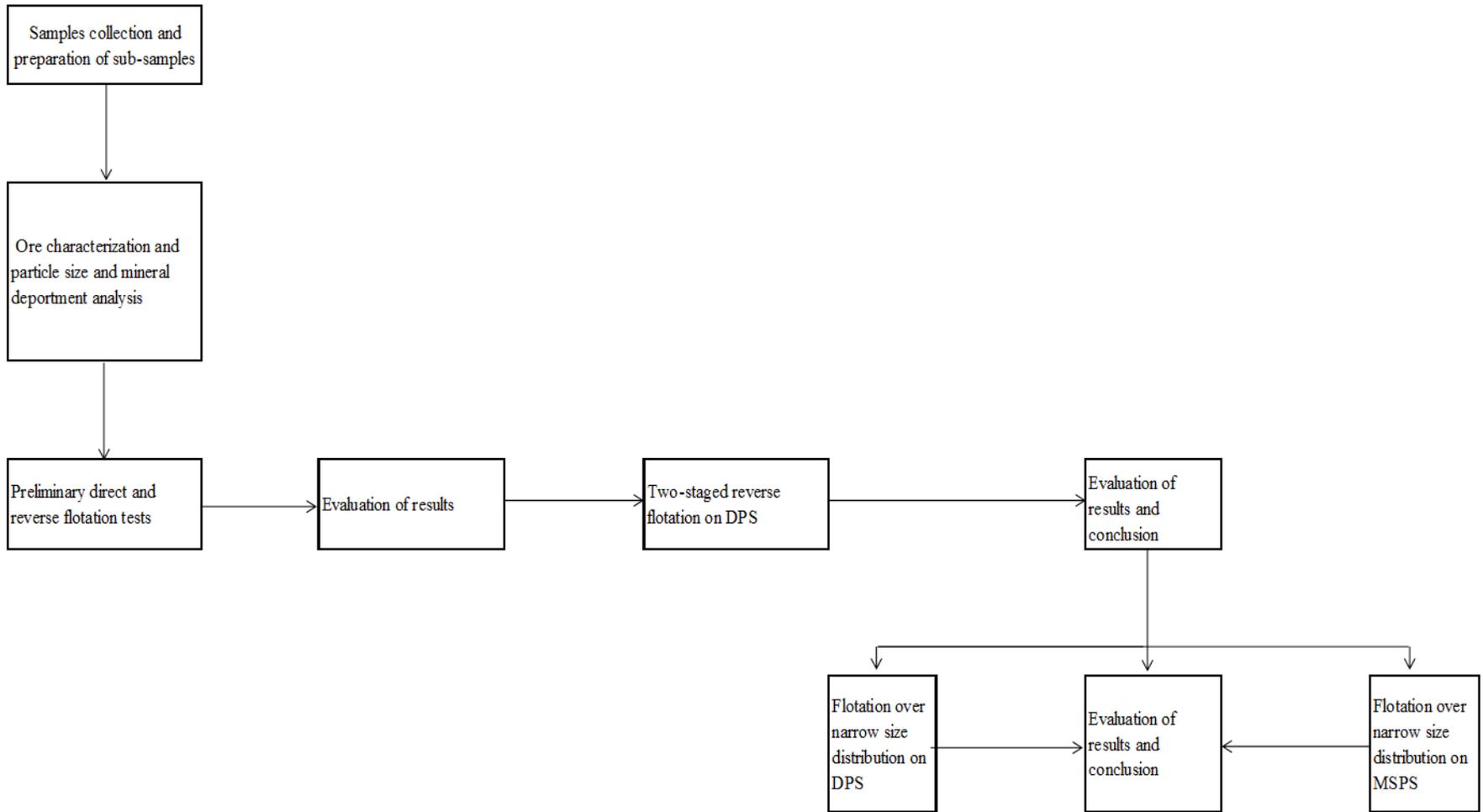


Figure 3.3: Experimental plan for beneficiation of iron ore slimes

From Figure 3.3, the DPS and MSPS were first characterised by SEM and XRF for mineral and elemental composition. Thereafter, the samples were screened at 75 to 5 micron (using square of 2 to determine the sizes in between) to determine mineral composition and deportment across the size range. The DPS was then subjected to preliminary direct and reverse flotation tests to assess the response of the ore slimes to flotation. Though reverse flotation was the preferred route, due to the high proportion of iron in the slimes, direct flotation tests were also conducted to give more information of the slimes' response to fatty acids collectors.

Two-staged reverse flotation was then conducted on the DPS. The two-stage dosage and conditioning was chosen based on the preliminary test results and that it is commonly noted that the collector is added at more than one point in a full-scale-flotation circuit and because it can reduce the number of flotation tests required, as it allows two different collector dosages and results in the same flotation test. The best conditions derived from flotation on the direct process slimes were adopted in flotation over narrow size DPS and MSPS at 25.0% solids by weight.

3.3.1 Mineralogical and chemical composition analysis

Mineralogical and chemical analyses were conducted on the ore DPS and MSPS. The purpose of the tests was to characterise the slimes with the aim of determining the mineral and elemental compositions present using SEM and XRF

3.3.1.1 Particle size and deportment analyses

Particle and deportment analyses were conducted on both the DPS and MSPS to determine the mineral deportment across size range using screen sizes 75, 53, 38 and 25 μm . The sieves were arranged in a stack in descending order of the sieve sizes selected according to $\sqrt{2}$ rule, the sieve with the largest aperture of 75 μm being at the top most position and that with the smallest aperture of 25 μm being at the bottom next to the collecting pan. The slurry was first screened using 25 μm sieve to remove the very fine particles (by pouring the slurry on the sieve placed on a shaker, switch on the shaker and allow the slimes to be screened for about 10 minutes continuously adding wash water to avoid agglomeration). The +25 μm fraction was collected dried to be screened using the sieves selected above and the -25 μm fraction was carefully stored in a closed container to be combined with the -25 μm fraction from the +25 μm screened

fraction. The nest of sieves was then placed on a Pascal sieve shaker; shaker switched on and allowed to run for about 10 minutes. The combined -25 μ m product was further screened using a 10 and 5 μ m cloth in an MXB Ultrasonic bath. Thereafter, all the products were filtered, dried in an oven at 90⁰C overnight. The weight of the dried products from each size fraction was measured and noted. For deportment analysis, the dried product of each size fraction was prepared to be analysed by XRF to determine the mineral deportment across size range (Wills, 2006:90-108).

In order to differentiate in the size fractions of the Sishen mine slimes, five size fractions were designated namely medium-coarse (mainly in the size fraction of +75 μ m), medium (mainly in the size fraction of -75+53 μ m), fine (mainly in the size fraction of -53+25 μ m), very fine (mainly in the size fraction of -25+5 μ m) and ultra-fine (mainly in the size fraction of -5 μ m) (Ansari, 1997:93-102; Viera and Peres, 2007:1008-1013).

The vibrating screen (shown in Appendix B Figure 7.3) was used for wet screening of the sample at 25 microns, prior to screening of the oversize from 75 microns to 5 microns. These tests were required to determine mineral deportment across size range.

3.3.2 Preliminary direct and reverse flotation tests on the direct process slimes

The flotation procedure applied was similar to that described by Ma (2012:56-61) and all tests were conducted at natural pH of the ore slimes as maximum flotation of hematite using fatty acids is highest in the neutral pH regions (Pope and Sutton, 1973; Parks, 1965). Two collectors were used separately named fatty acids; tall oil (containing approximately 91.0% oleic and linoleic acids, 6.0% rosin acid) and linoleic acid at dosages of 5, 7.5 and 10 g/t (using reagent dosage calculation shown in Appendix A) (Ma, 2012:56-61). Both collectors were obtained from Protea Chemicals South Africa. No frother or depressant was added because the collectors acted as frothers and depressants such as sodium silicate can sometimes face difficulties in depressing the undesirable minerals like silica (Araujo *et al.*, 2005:219-224). Three batch flotation tests were then conducted by:

- I. Pouring about 2 litres of sub-sampled slurry (at 40.0% solids) into a 2.5 litre cell on a Denver D12 machine and agitated at an impeller speed of 1200 rpm.

- II. Adding collector undissolved in water using a syringe and allowing to mix with the slurry for 3 minutes
- III. Introducing air and resuming the flotation process to produce a concentrate that is collected by scraping off the froth at 15 seconds intervals for 9 minutes and leaving the sinks which are known as tailings in the pulp.

Three rougher rate flotation tests were also conducted using tall oil and linoleic acid at a dosages of 5, 7.5 and 10 g/t (as shown in Appendix A) to determine the samples' best flotation times. The choice of collector dosages was based on visual observation of froth, as high dosages caused the froth to overflow. The sub-samples were also made up of about 2 litres of slurry (at 44.0% solid by weight), and tests were conducted using the same procedure described above with air introduced and floats collected by scraping off the froth at 15 seconds intervals for 1, 3, and 5 minutes (Ma, 2012:56-61).

Reverse flotation tests were conducted using lilafлот D817M and flotigam 3135 separately to float the silica and corn starch was added to depress the iron. The collectors were made up of 1.0% solution and starch was causticized as described in section in 3.1.4. For the flotation test, the slurry of 40.0 wt. % (as indicated in Table 3.1) was used, and 500 g/t starch added. Pulp pH was measured and adjusted to 10.5 using 5.0% solution of NaOH. The collector was then added at 50 g/t and the pulp conditioned for 1 minute. Air was introduced and float (silica concentrate) was collected by scraping off the froth at 15 seconds interval for 3 minutes (Thella *et al.*, 2012:418-426; Rocha *et al.*, 2010; Das *et al.*, 2005:737-742).

All flotation tests were carefully conducted in duplicate to obtain data that reproduce closely and products were collected, dried and prepared for analysis by XRF. The same procedure was followed for all flotation tests conducted in the investigation.

The machine used for all flotation tests was a Denver D 12 flotation cell as is shown in Appendix B (Figure 7.2). The cell is mechanically agitated, the speed of rotation of the impeller being variable and simulate the large-scale models commercially available. Introduction of air to the cell is normally via a hollow standpipe, the volume being controlled by a valve and the speed of the impeller. The air stream is shared into fine bubbles by the impeller which then

rises through the pulp to the surface, where any particles picked up are removed as a mineralised froth (Wills, 2006:267-280).

3.3.3 Two-staged reverse flotation tests on the direct process slimes

Two-staged reverse flotation tests were conducted using lilafлот D817M and flotigam 3135 collectors in conjunction with corn starch. All tests were conducted using the procedure described in section 3.3.2 on the first stage. For the second stage, about 400 ml of water was added and pH of the pulp adjusted to 10.5 using 5.0% NaOH solution. Another 50 g/t of collector was then added and allowed to mix with the slurry for 1 minute. Air was introduced and the float was collected by scraping off the froth at 15 seconds interval for 5 minutes. Two more flotation tests were conducted using the same test conditions but with collector addition increased to 75 and 100 g/t which equated to 50.0% and 100.0% increases in the dosages (Thella *et al.*, 2012:418-426; Rocha *et al.*, 2010:842-845; Das *et al.*, 2005:737-742).

Another set of two-staged reverse flotation tests was conducted on each collector by increasing the depressant dosage by 50.0% of the initial dosage of 500 g/t and later on by another 50.0% to make double dosage of 1000 g/t. The purposes of the tests were to determine the effect of the depressant dosages in determining the suitable reagent suite for flotation of iron ore slimes.

To further investigate the effect of collectors and depressants dosages in determining the suitable reagents suite for flotation of slimes, dextrin starch was employed as a second depressant. More two-staged flotation tests were conducted using the same procedures as described above.

Due to the poor results obtained from the above flotation tests (presented and discussed in Section 4.4), further investigations were carried out to determine the effects of:

- pH,
- impeller speed,
- Pulp density, and
- Particle size distribution (which in the investigation is referred to as flotation over narrow particle size distribution).

Investigation of the parameters was considered necessary because:

- Iron oxides minerals are subjected to varying surface charges which influence the pH at which flotation is performed for a chosen collector (Thella *et al.*, 2012:418-426).
- The low flotation rate of fine-ultrafine particles may be improved by increasing the collision rate and the inertia moment (Sivamoha, 1990:247-288).
- Wills (2006:287-294) stated that “The optimum pulp density is of great importance, as in general the more dilute the pulp, the cleaner the separation”.
- The wide particle-size distribution is approached by splitting into narrow size ranges and treating them differently (Pease *et al.*, 2005:247-288).

To assess these parameters, two-staged reverse flotation tests were conducted using lilafлот D817M at dosage of 75 g/t and corn starch at 1000 g/t as follows, judging from results obtained, (discussed in Section 4.4):

- **pH** - tests were conducted by increasing the pH values from 10.5 to 11 and 11.5 because cationic collectors can only react with a mineral surface if the pH used is above its PZC (PZC of quartz is between 1.8-3.7 and that of amines is between 5 to 11-11.5) (Smith and Scott, 1990). Hence, it was thought that increasing the pH from optimum pH of 10.5 to 11 and 11.5 might result in floating more quartz to promote separation from iron.
- **Impeller speed** - tests were conducted by increasing impeller speed from 1200 rpm to 1300 rpm and 1400 rpm.
- **Pulp density** - tests were conducted by diluting the pulp density from 40.0% to 35.0% and 25.0% solids by weight.

3.3.4 Two-staged reverse flotation tests on the magnetic separation processed slimes

As in the investigation plan, two-staged flotation tests were also conducted on the magnetic separator processed slimes using lilafлот D817M at 75 g/t with corn starch at 1000 g/t. The flotation tests procedures used were the same as described in section 3.3.3, with 25.0 wt.% solid slurry, judging from results of investigation on effect of feed percentage solid, as discussed in section 4.4.3.

3.3.5 Flotation over narrow particle size distributions

Particle kinetics over the whole slimes size range certainly will differ. Hence, flotation over a narrow particle size range is expected to enhance kinetics and separation efficiency (Pease *et al.*, 2005:831-840; Thella *et al.*, 2012:418-426; Trahar, 1981:289-327). Reverse flotation tests were conducted on -53, -53+38, -38+25 and -25 μm fractions. The fractions were obtained by screening the slimes using 53, 38 and 25 micron sieves (as discussed in section 3.3.1.1) thereafter, collecting the +53, +38, +25 and -25 fractions separately. The process was repeated until enough mass was obtained for flotation tests. Flotation tests were conducted adopting the same procedure described in section 3.3.3 at pulp percentage solids of 25.0% (judging from results of previous tests discussed in section 4.4.3). Tests were done on both the DPS and MSPS.

3.4 Analytical Techniques used in the investigation

This section gives brief information on the equipment used when conducting the research. Scanning Electron microscope and X-ray fluorescence (as shown in Appendix B Figure 7.1) were used for mineralogical study to determine the minerals and elements present in the ore slimes.

The following methods were used:

- **Scanning Electron Microscope (SEM)** - About 8 g sample of the direct slime powder was spread on double sided adhesive carbon discs, and the powder surface coated with graphite. SEM study was done in energy dispersive X-ray spectroscopy mode, to excite and identify elemental constituents of the minerals present. A surface elemental map was then produced to give a visual indication of the proportion of different elements which is an indication for the different minerals (McMullan, 2006).
- **X-ray Fluorescence (XRF)** - an analytical technique which uses the interaction of x-rays with a target material to determine its elemental composition. Fused beads were made by mixing 0.6 g slimes powdered with a flux in a flux/sample ratio of 5:1 and then heated to 1000⁰C in a platinum crucible. The sample was dissolved in the flux and cast into a mould with a flat bottom which resulted in a glass disc or fused bead that is a homogenous representation of the sample and free of mineral structures. The fused

bead was then analysed by (XRF) to determine the range of elements present and their proportions in the ore slimes (Beckhoff *et al.*, 2006).

CHAPTER 4

4. RESULTS AND DISCUSSION

4.1 Mineralogical and Chemical Analysis of the Direct Process Slimes and Magnetic Separation Processed Slimes

Mineralogical and chemical analyses were conducted on the DPS and MSPS using SEM and XRF to characterise the slimes by determining the mineral and elemental composition present. The obtained summarised chemical analyses results are shown in Tables 4.1 and 4.2 and SEM images in Figures 4.1 and 4.2.

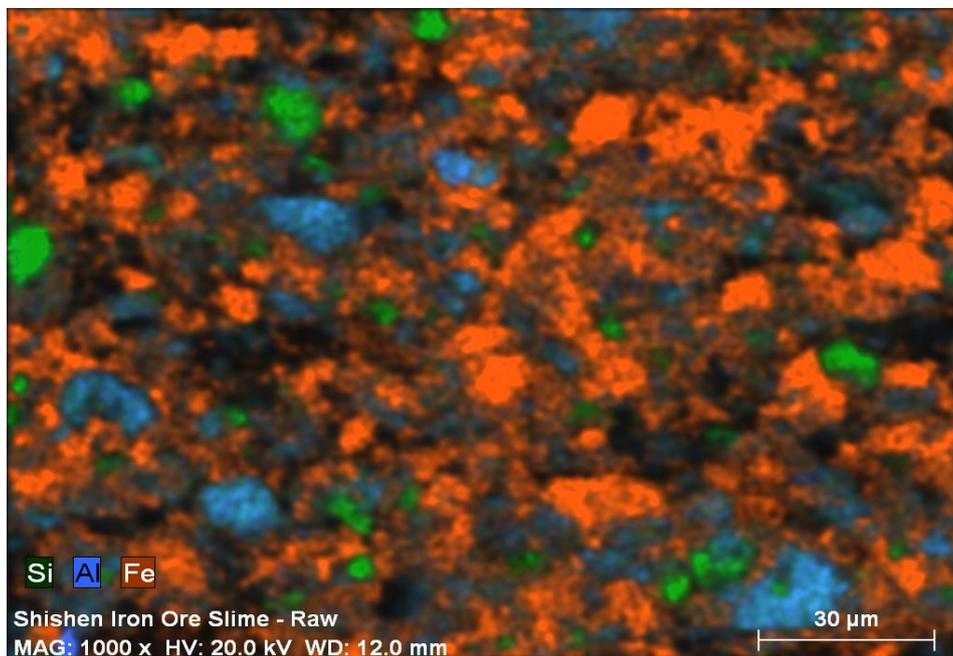


Figure 4.1: SEM-EDS elemental map of the direct process slimes powder indicating proportion of silicon, iron and aluminium in the ore slimes

Table 4.1: Chemical composition of the direct process slimes obtained using XRF

Product	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	P ₂ O ₅ (%)	K ₂ O (%)	CaO (%)
Direct process slimes	44.00	20.40	8.70	0.26	1.70	1.20

The SEM-EDS images show that iron is possibly liberated from silica and occurs in higher proportion than the silica. This is corroborated by the summarised composition analyses results (as shown in Table 4.1) too, which shows that the slimes consist mainly of 44.0% iron with

silica and alumina (Al_2O_3) contents of 20.4% and 8.7% respectively. This justifies the need to beneficiate the ore slimes, before it can be used to produce pellets for iron making.

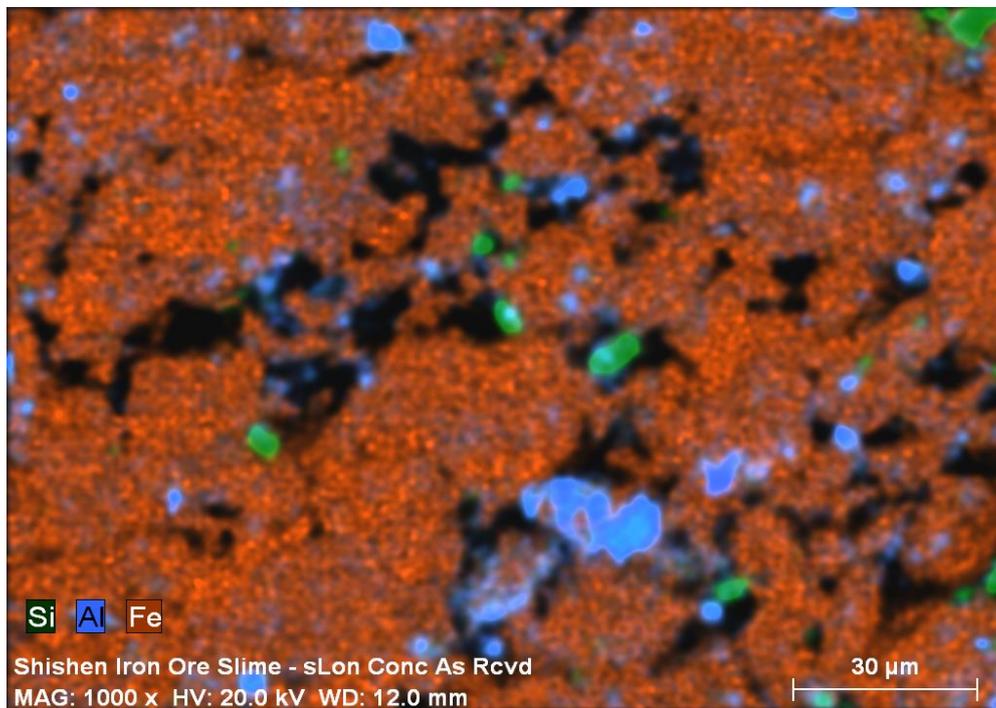


Figure 4.2: SEM-EDS elemental map of the magnetic separation processed slimes powder indicating proportion of silicon, iron and aluminium in the ore slimes

Table 4.2: Chemical composition of the magnetic separation processed iron slimes obtained using XRF

Product	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	P ₂ O ₅ (%)	K ₂ O (%)	CaO (%)
Magnetic separation processed slimes	63.70	4.80	1.90	0.11	0.13	0.10

From the analysis of composition shown in Table 4.2, the use of pulsating high gradient magnetic separator resulted in iron content of the slimes being upgraded to 63.7% with 4.8% silica and 1.9% alumina (Al_2O_3) from feed of 44.0% iron, 20.4% silica and 8.7% alumina. The SEM image presented in Figure 4.2 also shows that the slimes consist of much more iron. The results obtained from PHGMS nearly meet the requirement for pelletizing (Viera and Peres, 2010:124-125; Mbele, 2012:221-223; Ola *et al.*, 2009:405-416; Ma, 2012:56-58).

4.2 Particle size and Department Analyses

4.2.1 Particle size and department analyses of the direct process slimes

Particle size and department analyses (screening and assaying) were conducted on the direct process slimes to determine particle size distribution and the mineral department across size range. The results are shown in Figures 4.3-4.6. Raw data for this analysis is shown in Appendix C Tables 8.1 and 8.2.

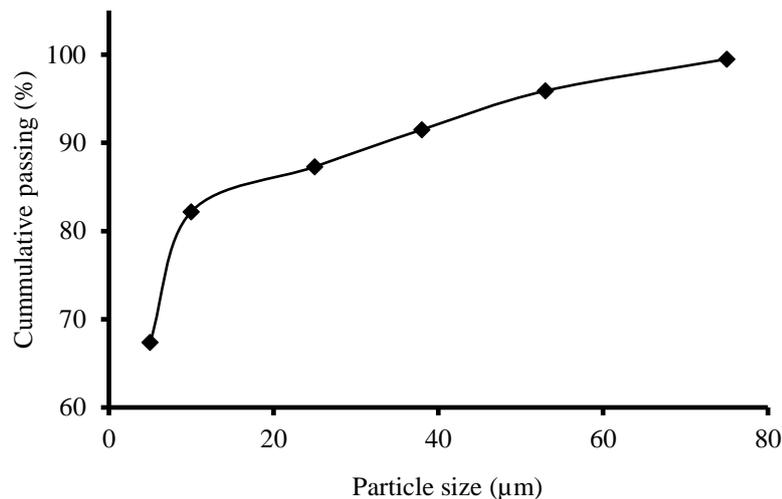


Figure 4.3: Particle distribution curve of the direct process slimes

The particle size distribution (Figure 4.3) shows that the slimes consist of about 90.0% particles below 40 µm, indicating that they are made-up of very fine particles thus precluding beneficiation methods such as gravity concentration. However, methods such as flotation, selective flocculation and magnetic separation are often considered worthy of consideration when beneficiating such finer particles because of commonly known to treat mineral particles below 150 microns.

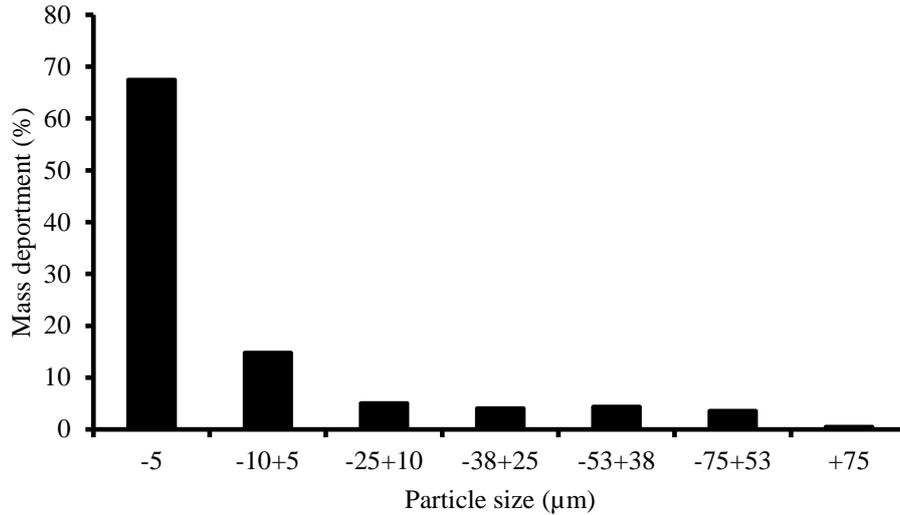


Figure 4.4: Mass department across size range of the direct process slimes

Figure 4.4 shows that most of the mass reported to the finer fraction with 82.3% reporting to the -10 µm, while most of the iron is also in the finest fractions. Hence, it could be suggested that the ore slimes are beneficiated by separating at 10 µm, but it might not be suitable for industry purposes, because of possible constraints on the availability of industrial scale equipment that will efficiently screen the mineral in the slimes at 10 µm.

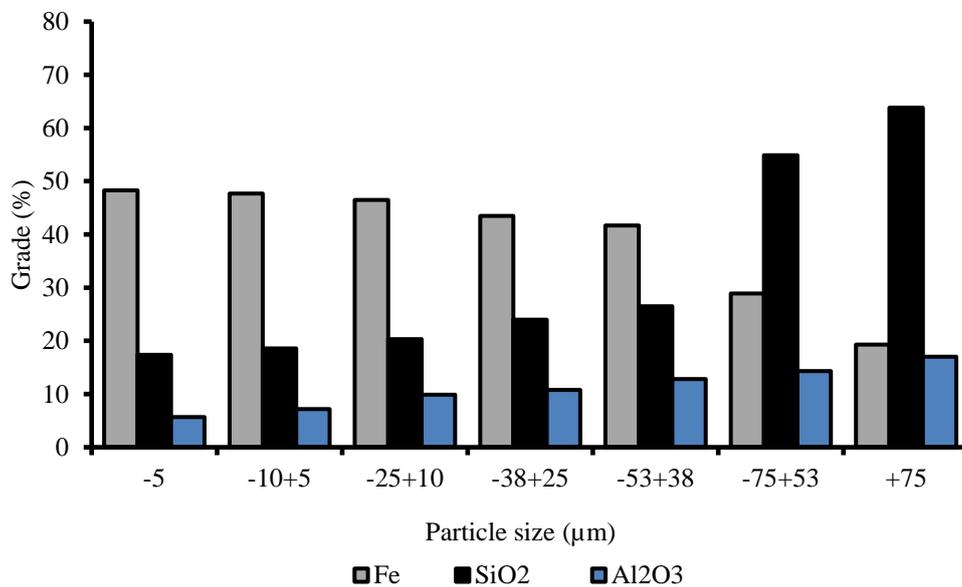


Figure 4.5: Mineral department across size range of the direct process slimes

The results (Figure 4.5) show that there is more iron present in the fine and ultrafine fractions. The silica content is higher in the medium-coarse and medium fractions with some quantity in

the fine-ultrafine fractions. The results obtained corroborate with the SEM image. The selective deportment of the target iron values and the gangue to different size fractions indicates that separation of the slimes at some sub-sieve size will amount to a pre-concentration, and processing over the narrower particle size range can have positive effects in beneficiation.

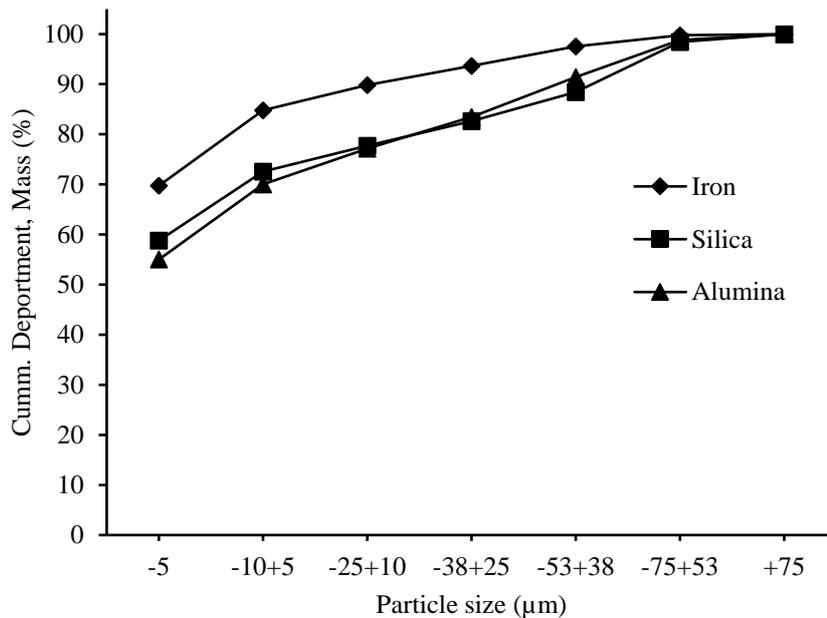


Figure 4.6: Cumulative mass and assay over size in the minerals of direct process slimes

From results depicted in Figure 4.6, Fe is higher in the finer fractions; the -5 μm contains about 70.0% of the Fe values which left most of the silica and aluminium appearing in the coarser fraction.

4.2.2 Particle size and deportment analyses of the magnetic separation processed slimes

The results of particle size and composition analysis conducted on magnetic separation process slimes obtained are shown in Figures 4.7- 4.10.

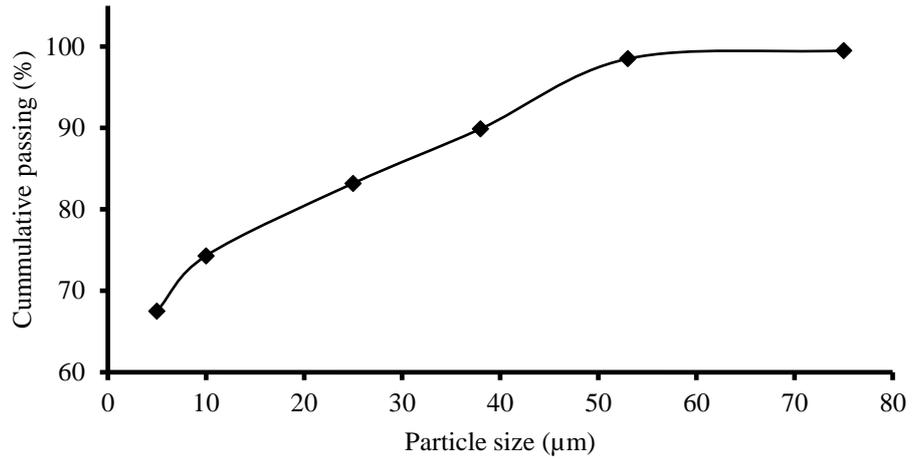


Figure 4.7: Particle size distribution curve of the magnetic separation processed slimes

The particle size distribution of magnetic separation slimes was similar to that of the direct processed slimes (Figure 4.3) in that most of the mass and Fe reported to the finest fraction.

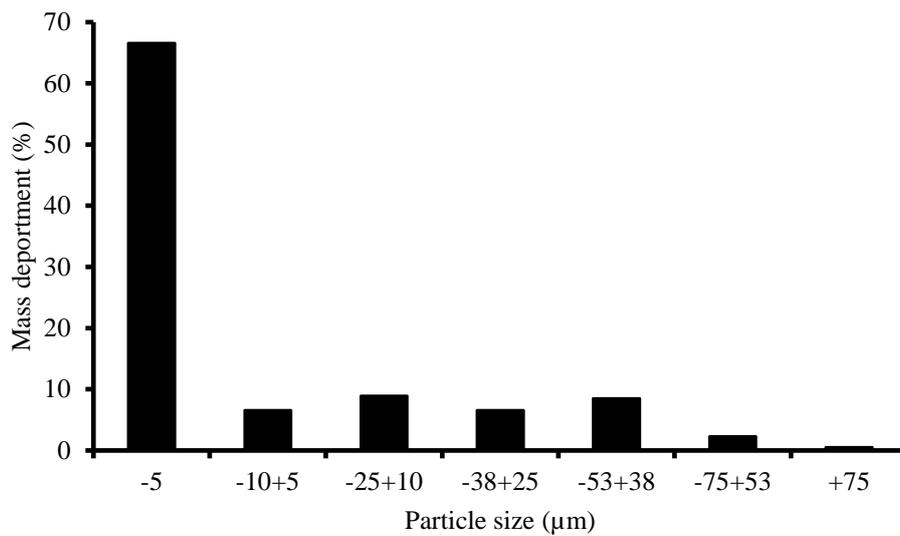


Figure 4.8: Mass department across size range of the magnetic separation processed slimes

Like the direct process slimes, Figure 4.8 shows that more Fe mass reported to the ultrafine fraction size in the magnetic separation processed slimes.

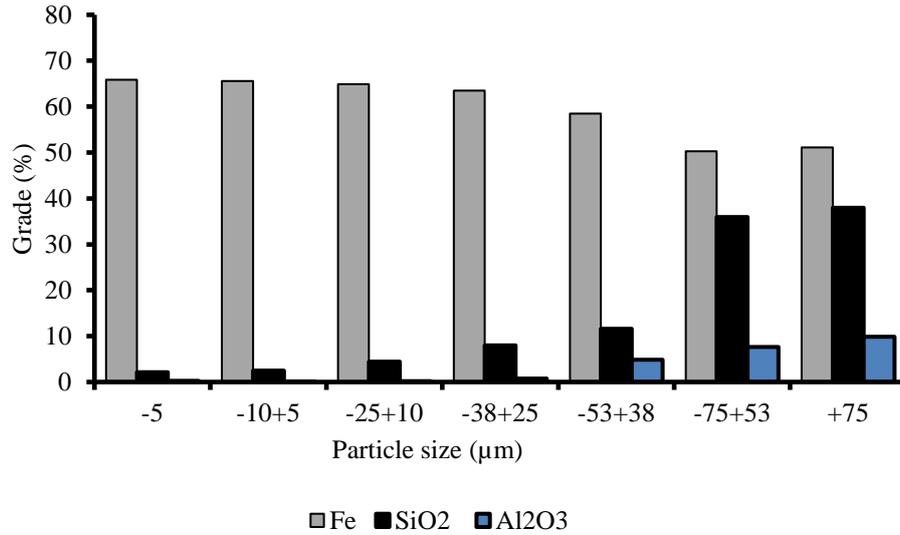


Figure 4.9: Mineral department across size range of magnetic separation processed slimes

The results (Figure 4.9) indicated that more Fe reported to the -38 μm fraction with silica mostly at the +38 μm.

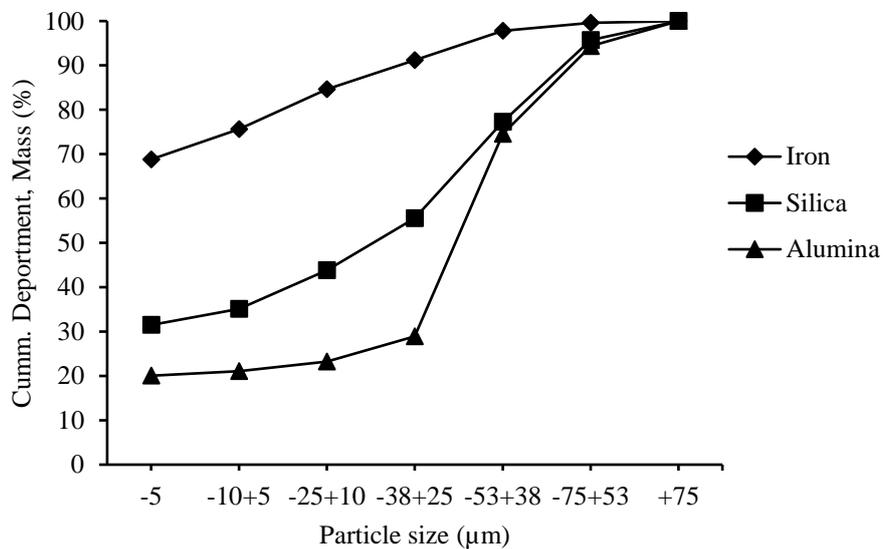


Figure 4.10: Cum. mass and assay over size in the minerals of magnetic separation processed slimes

The low concentration of silica in the finest fraction suggests that PHGMS is effective in rejecting silica from the ultrafine feed particles. However, the option of flotation is still being explored further as coarse silica can possibly be dealt with in the flotation stage.

4.3 Preliminary Direct and Reverse Flotation tests of the Direct Process Slimes

4.3.1 Response of the direct process slimes to direct flotation

Three batch flotation tests were conducted on the direct process slimes using tall oil and linoleic acid (as described in section 3.3.2). The results obtained are shown in Tables 4.3 and 4.4.

Table 4.3: Response of DPS under direct flotation test using tall oil as a collector

				Grade			Recovery		
Products	Time (min)	Mass (g)	Mass (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)
Float	9	190.06	15.97	46.00	19.99	9.23	16.50	15.30	15.08
Sink		999.99	84.03	44.24	21.04	9.88	83.50	84.70	84.92
Head (calc)		1190.05	100.00	44.52	20.87	9.78	100.00	100.00	100.00

Table 4.4: Response of DPS under direct flotation test using linoleic acid as a collector

				Grade			Recovery		
Products	Time (min)	Mass (g)	Mass (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)
Float	9	170.05	14.53	45.51	20.09	9.11	14.96	14.03	11.61
Sink		1000.01	85.47	44.00	21.12	9.49	85.04	85.96	88.39
Head (calc)		1170.06	100.00	44.22	20.97	9.43	100.00	100.00	100.00

From Tables 4.3 and 4.4, tall oil yield better results compared to linoleic acid with 46.0% Fe reporting to the float at recovery of 83.5% and SiO₂ content of 19.9%. However, the sinks still contains very high amounts of Fe values which suggest that both collectors hasn't been effective in floating iron.

The results obtained when conducting rougher rate flotation tests with various collectors (tall oil and linoleic acid) dosages are shown in Figures 4.12-4.15.

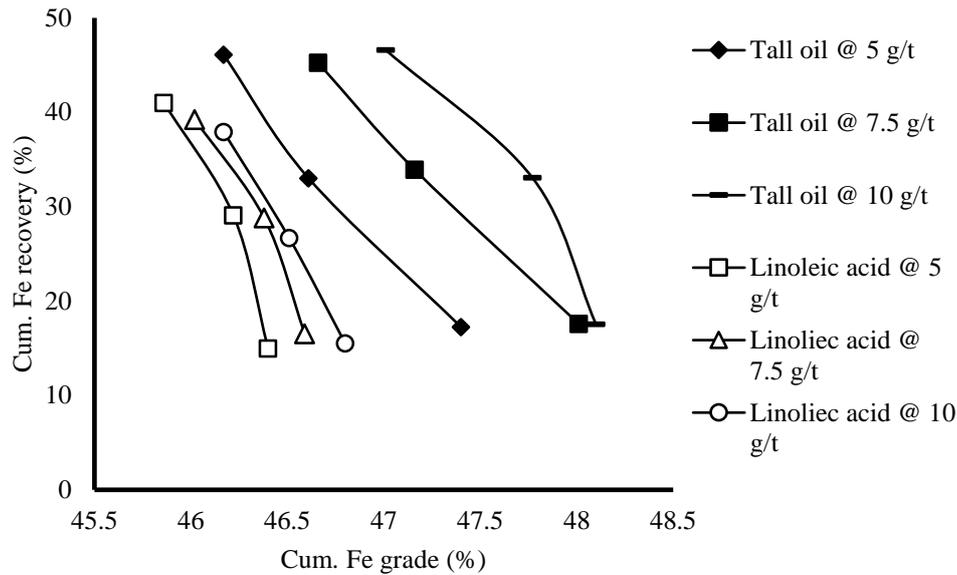


Figure 4.11: Cumulative grade-recovery curves of Fe reporting to the float at various collectors (tall oil and linoleic acid) dosages in direct flotation of DPS

The highest overall Fe recovery for the combined float was 46.6% along with a grade of 47.0% obtained at 10 g/t of tall oil. Increasing linoleic acid from 5 to 10 g/t resulted in a slight Fe increase and SiO₂ decrease. Overall, tall oil performed better than linoleic acid as the final Fe grades for the combined float ranged between 46.0% and 47.0% for all the collector dosages compared to linoleic acid with Fe ranging between 45.0% and 46.0%. The better performance of tall oil might be due to its content of oleic acid whose distinct iron flotation characteristic is recognised and that hematite reaches its maximum flotation at neutral pH (Fuerstenau and Cummins, 1967; Fuerstenau *et al.*, 1967; Fuerstenau *et al.*, 1970; Fuerstenau and Pradip, 1984). The results obtained were as expected, due to the high proportion of iron in the ore slimes, and the resulting heavy mass pull.

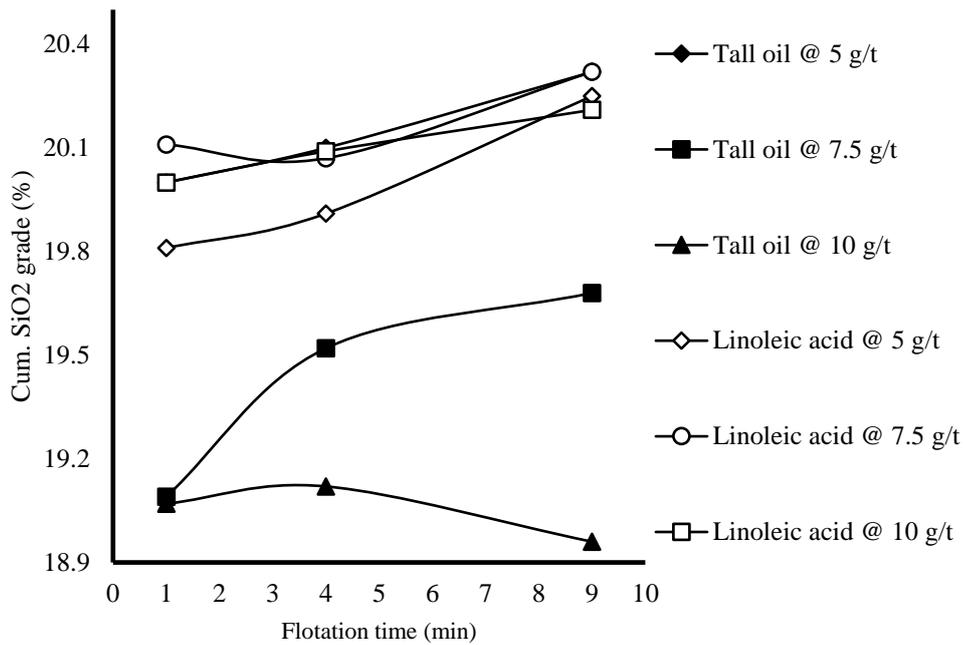


Figure 4.12: Cumulative SiO₂ grade reporting to the float at collectors (tall oil and linoleic acid) dosages in direct flotation of DPS

From results shown in Figure 4.12, it was observed that higher dosage of tall oil (at 10 g/t) was slightly favourable for direct flotation of DPS as silica reporting to the float was lower with an overall of ~18.9%. Linoleic acid, on the other hand, was worse for silica reduction in the concentrates as an overall grade of ~ 20.2% was obtained at the high dosage of 10 g/t.

From the results presented in Figure 4.13 and Figure 4.14, the iron recoveries and mass reporting to the floats were close for all the dosages of both collectors used. Hence, the conclusion that the collectors used in this study did not effectively reduce silica and increase iron content.

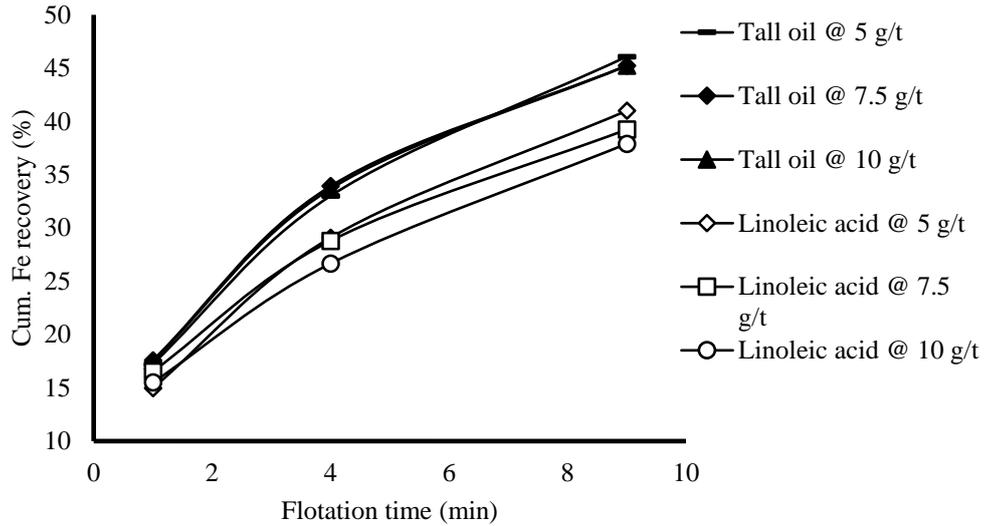


Figure 4.13: Cumulative Fe recovery vs. flotation times at various collectors (tall oil and linoleic acid) dosages in direct flotation of DPS

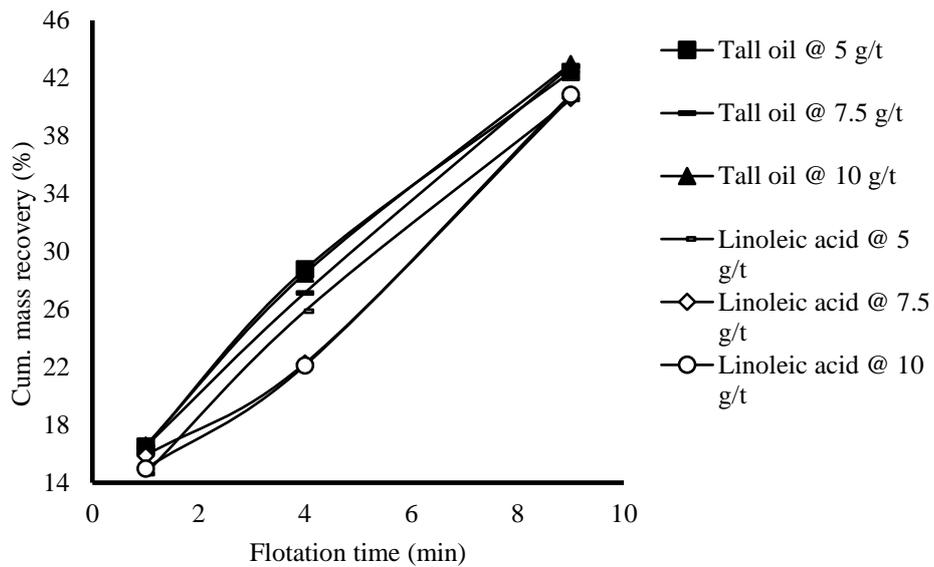


Figure 4.14: Cumulative mass recovery vs. flotation times at various collectors (tall oil and linoleic acid) dosages in direct flotation of DPS

4.3.2 Conclusion on results of preliminary direct flotation tests of direct process slimes

Although the history of iron ore flotation started with direct flotation of iron oxides using anionic collectors such as petroleum sulphonate, fatty acids and hydroxamates (Ma, 2012:56-58), the fatty acids employed in this study were not effective on the ore slimes since the highest

grade that could be obtained was 47.0% iron and 18.9% silica when using tall oil at a dosage of 10g/t. It was suggested that, the poor performance of direct flotation might have been due to very fine iron mineral particles being carried to the froth via entrainment (Savassi *et al.*, 1998:243-256). The entrainment might have been supported by iron being in fine size in the slimes and in larger proportion than quartz as shown in results obtained in Figures 4.1 and 4.3-4.6. Therefore, direct flotation may not serve as a recommended route to beneficiate Sishen mine iron ore slimes.

4.3.3 Reverse flotation tests of the direct process slimes

The results obtained on preliminary reverse flotation tests conducted on the direct process slimes using lilafлот D817M and flotigam 3135 as collectors in conjunction with corn starch are shown in Tables 4.5 and 4.6.

Table 4.5: Response of DPS under preliminary reverse flotation test using lilafлот D817M collector

				Grade			Recovery		
Products	Time (min)	Mass (g)	Mass (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)
Float	3	239.21	20.80	40.33	21.28	10.29	18.23	21.81	26.34
Sink		910.70	79.20	47.51	20.04	7.56	81.77	78.19	73.66
Head (calc.)		1149.91	100.00	46.01	20.30	8.13	100.00	100.00	100.00

Table 4.6: Response of DPS under preliminary reverse flotation test using flotigam 3135 collector

				Grade			Recovery		
Products	Time (min)	Mass (g)	Mass (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)
Float	3	224.56	21.91	43.52	20.60	9.41	20.94	21.67	24.38
Sink		800.30	78.09	46.09	20.89	8.19	79.06	78.33	75.62
Head (calc)		1024.86	100.00	45.53	20.83	8.46	100.00	100.00	100.00

From depicted results in Tables 4.5 and 4.6, lilafлот D817M performed better than flotigam 3135 as Fe content in the sink was 48.0% with recovery of 81.8 % and silica of 20.0%. The better performance of lilafлот D817M might be due to the longer hydrocarbon chain length and different neutralisation degree compared to flotigam 3135 (as shown in Appendix A) (Fuerstenau *et al.*, 1964; Rao *et al.*, 2001:26-29). Therefore, the lilafлот D817M collector might be a favourable collector for flotation of slimes and this is investigated in more flotation tests.

4.4 Two-staged Reverse Flotation of DPS using Cationic Collectors (diamines) with Starch depressant

The response of the ore slimes to flotation tests using lilafлот D817M and flotigam 3135 collectors in conjunction with corn starch as a depressant at various dosages are shown in Figures 4.15 to 4.20 (the raw data is given in Appendix D Tables 9.19 and 9.2).

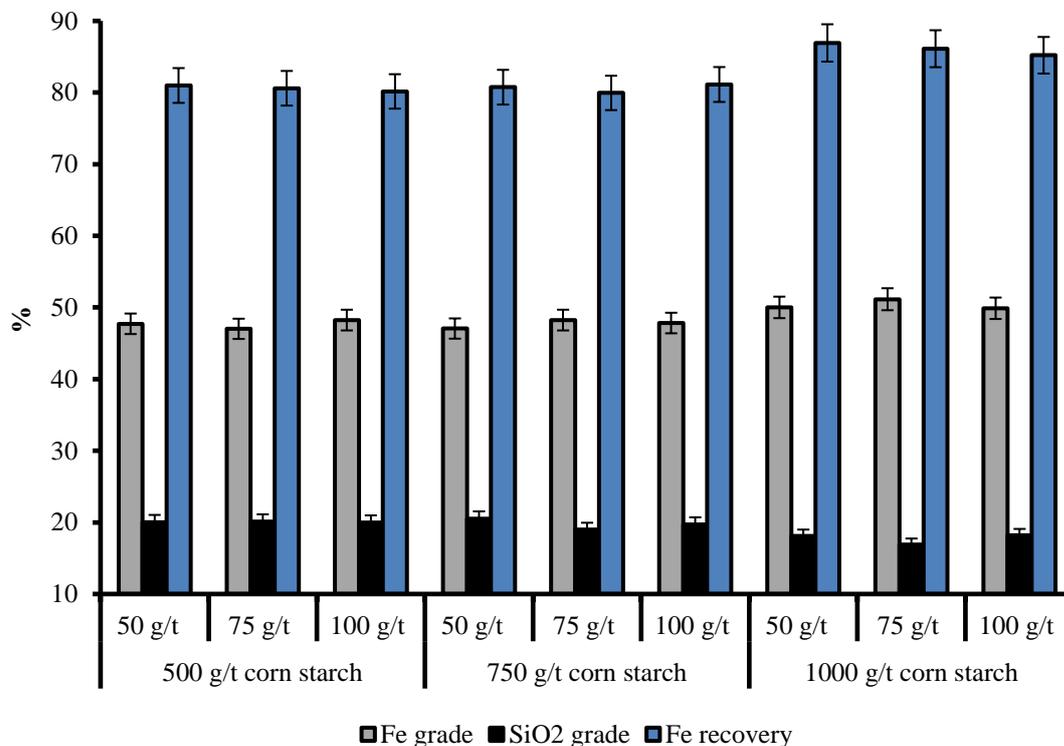


Figure 4.15: Iron grade, silica grade and iron recovery in the sink fraction from two-stage reverse flotation of DPS using lilafлот D817M collector and corn starch depressant at different dosages.

Lilafлот D817M at dosage of 75 g/t showed a progression on reducing silica in the sink fraction as it was decreased to 16.9% with Fe content of 51.2% and recovery of 86.1%. The collector

thus functions as projected in being able to selectively adsorb onto and float silica particles, leaving a Fe-enriched sink. Similar behaviour of cationic collector dosages in terms of silica being floated was obtained in flotation of iron ore slimes by Thella *et al.*, (2012:418-426). The high Fe content in the sink attainable at 1000 g/t of corn starch is also recognised in flotation of iron ore slimes (Rocha *et al.*, 2010:842-845; Lima *et al.*, 2013:152-159). These results show that the slimes might respond well to the reverse flotation. However, the iron grade remains far too low and the silica grade is still higher than required.

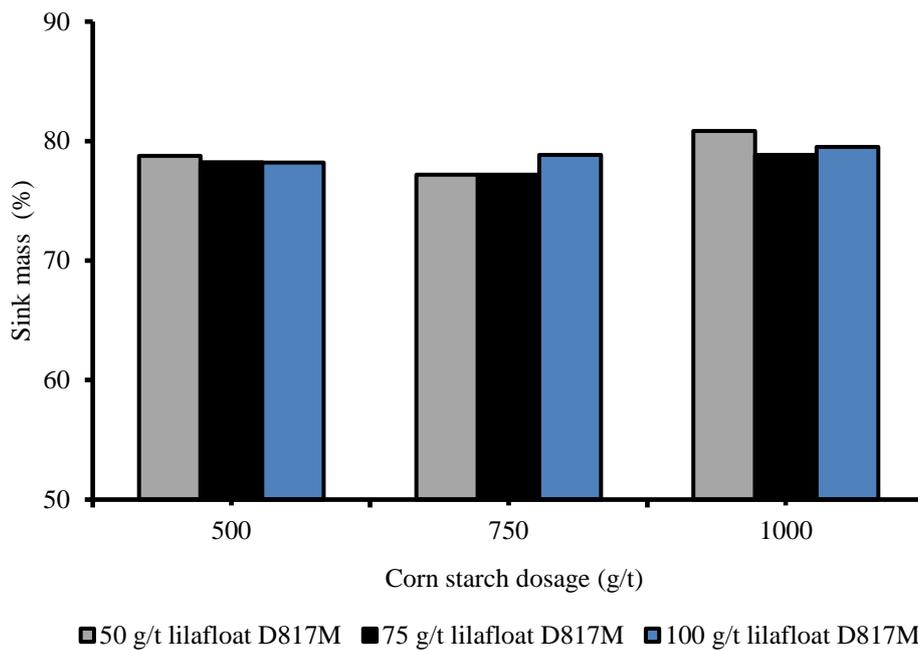


Figure 4.16: Sink mass in the two-stage reverse flotation of DPS at various lilafloat D817M and corn starch dosages

Increasing the starch dosage from 500 g/t to 750 g/t did not clearly show the depressant effect, but at 1000 g/t, some clear increase can be seen in the final sink at all collector dosages. More iron was recovered to the sink as indicated in Figure 4.15.

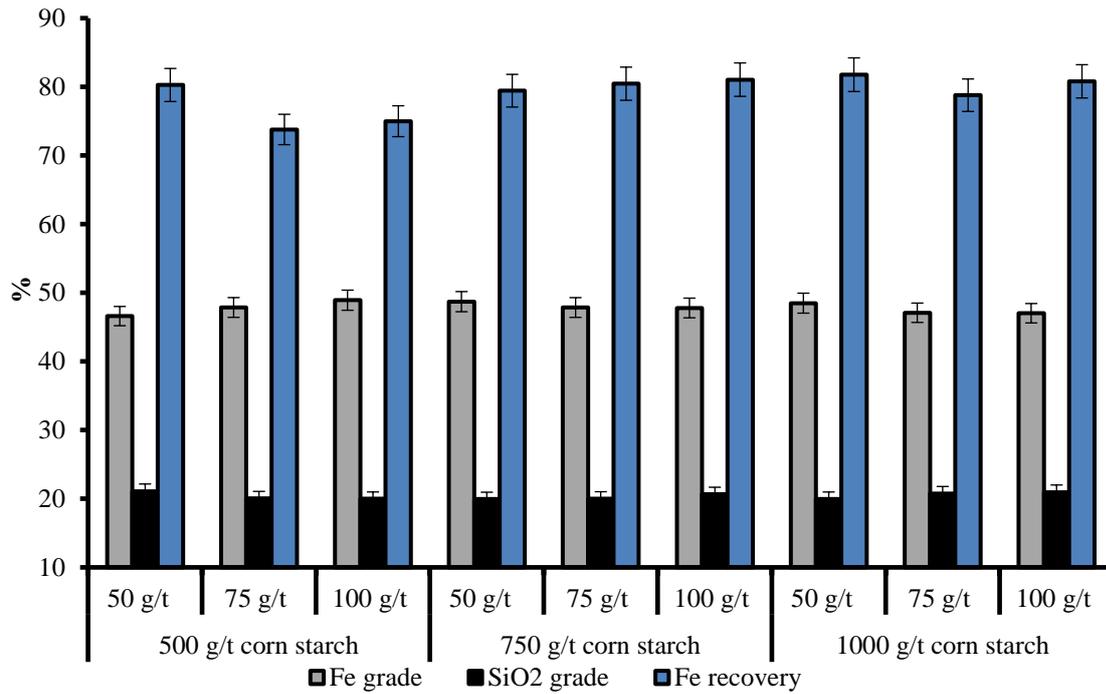


Figure 4.17: Iron grade, silica grade and iron recovery in the sink fraction from two-stage reverse of DPS flotation using flotigam 3135 collector and corn starch depressant at different dosages.

The use of the flotigam 3135 collector for the flotation of ore slimes had a slight effect on increasing the Fe content in the sink fraction which ranged from 46.0% to 48.0% in all dosages of both the collector and depressant. The highest depressant dosage gave the highest recovery of Fe in the sink fraction at 50 g/t collector, but these changed slightly at higher collector dosage. SiO₂ grades were discouraging at ~21.0% on average in all collector and depressant dosages. Lilafлот D817M collector still performed better than flotigam 3135, but neither met the target.

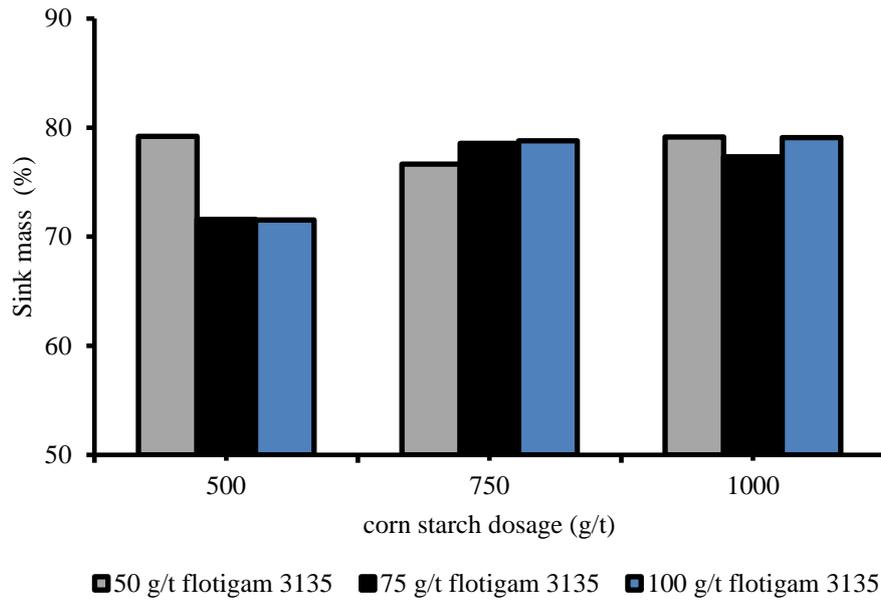


Figure 4.18: Sink mass in the two-stage reverse flotation of DPS at various flotigam 3135 and corn starch dosages

From results shown in Figure 4.18, the use of corn starch at 500 g/t with 50 g/t flotigam 3135 resulted in more mass reporting to the float. Hence, the results obtained were considered unreliable. Increasing the collector dosage from 50 g/t to 100 g/t on starch at dosage of 750 g/t resulted in more mass reporting to the sink. However, the same trend was not followed when starch was further increased to 1000 g/t: the mass reporting to the sink was 79.1% at 50 g/t of flotigam 3135 but decreased to 77.3 % at 75 g/t and to 79.1% at 100 g/t. Low dosages of starch left less mass in the sink concentrates.

Similarly, the response of the DPS to flotation tests using lilafлот D817M and flotigam 3135 collectors in conjunction with dextrin starch at various dosages is shown in Figures 4.19-4.24 (the raw data is given in Appendix E Tables 10.1 and 10.2).

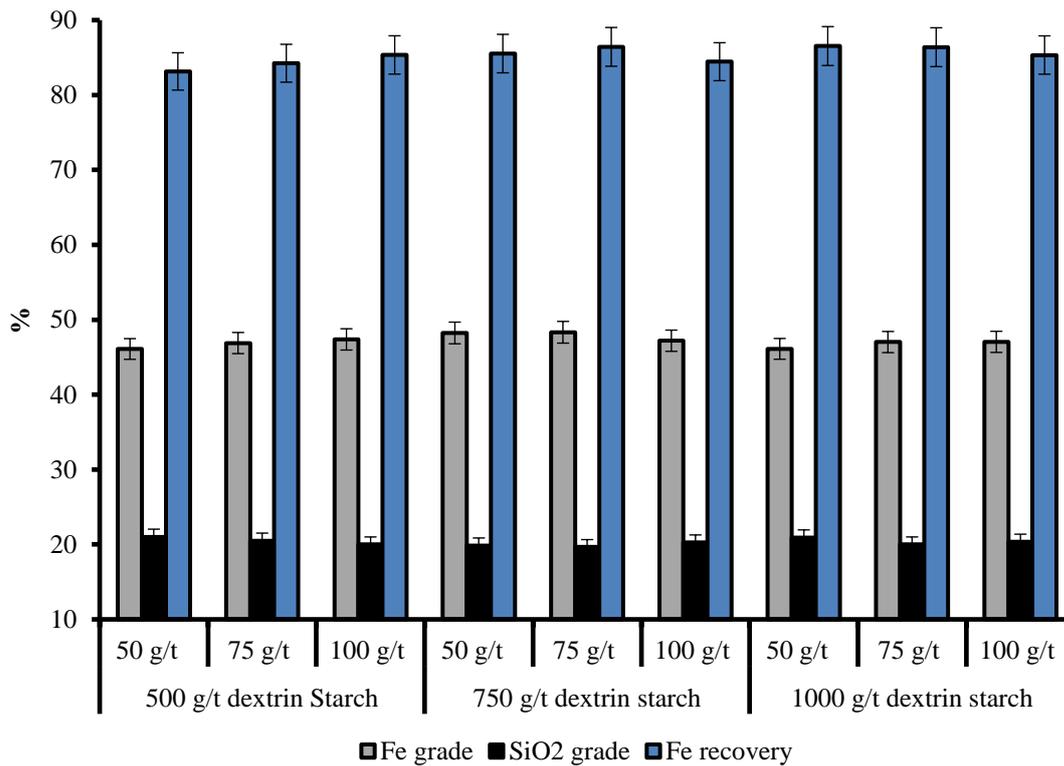


Figure 4.19: Iron grade, silica grade and iron recovery in the sink fraction from two-stage reverse flotation of DPS using lilafлот D817M collector and dextrin starch depressant at different dosages.

Results depicted in Figure 4.19 shows that the use of dextrin starch in conjunction with lilafлот D817M did not have any significant impact on the flotation performance of the ore slimes. However, increasing collector dosage from 50 g/t to 100 g/t in two depressant dosages of 500 and 1000 g/t resulted in silica being decreased slightly in the sink with the lowest being 20.0% at 100 g/t of the collector for both starch dosages.

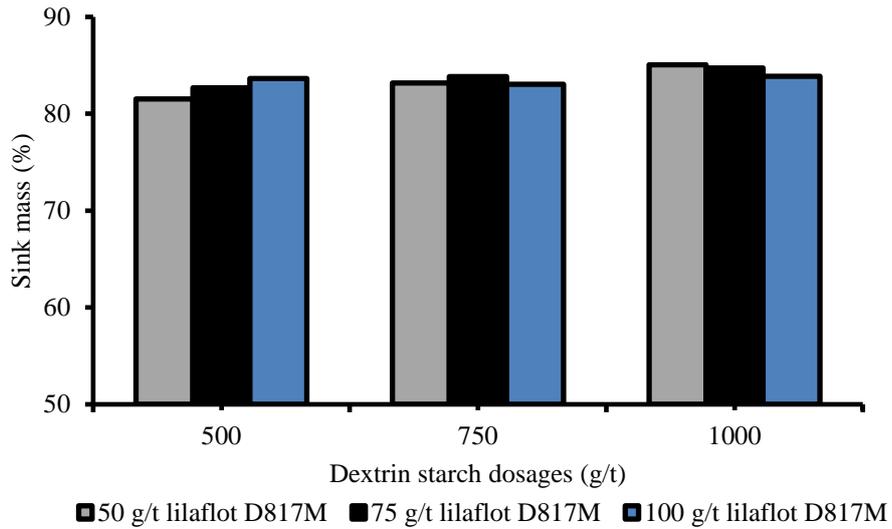


Figure 4.20: Sink mass in the two-stage reverse flotation of DPS at various lilafлот D817M and dextrin starch dosages

The use of lilafлот D817M collector at high dosage of dextrin starch resulted in more mass reporting to the sink compared to the dosage of 500 and 750 g/t.

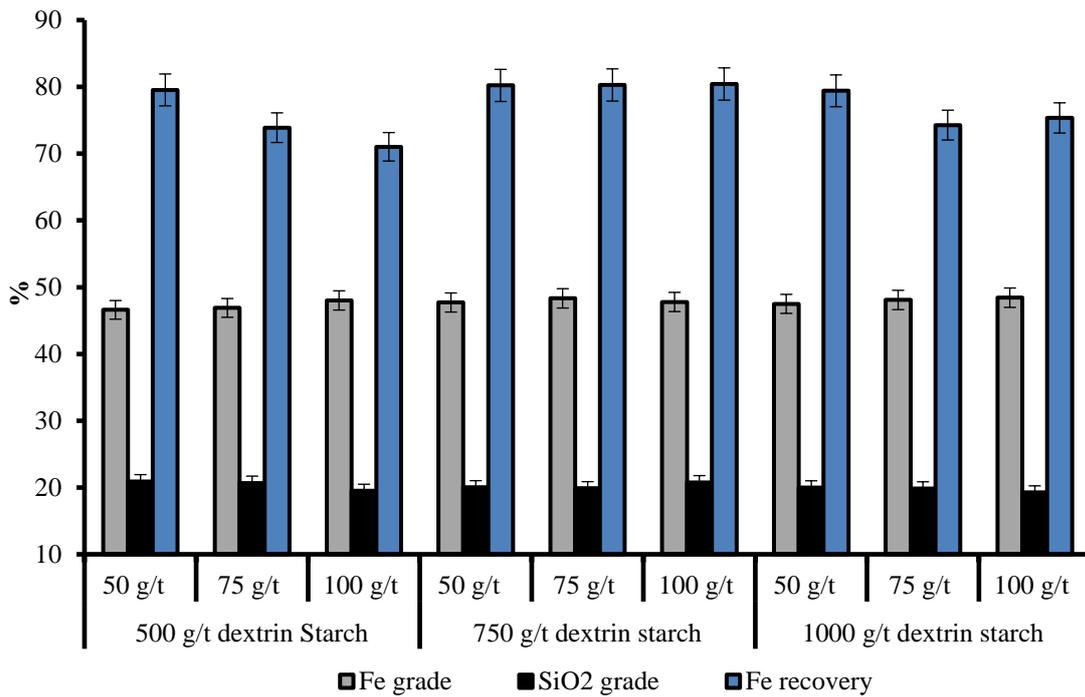


Figure 4.21: Iron grade, silica grade and iron recovery in the sink fraction from two-stage reverse flotation of DPS using flotigam 3135 collector and dextrin starch depressant at different dosages.

The results shown in Figure 4.21 are discouraging as silica was not reduced. The Fe content ranged between 46.0% and 48.0% in all collector dosages with the highest recoveries obtained at 750 g/t of dextrin. The results suggest that dextrin starch might not be an effective depressant in the reverse flotation of iron ore.

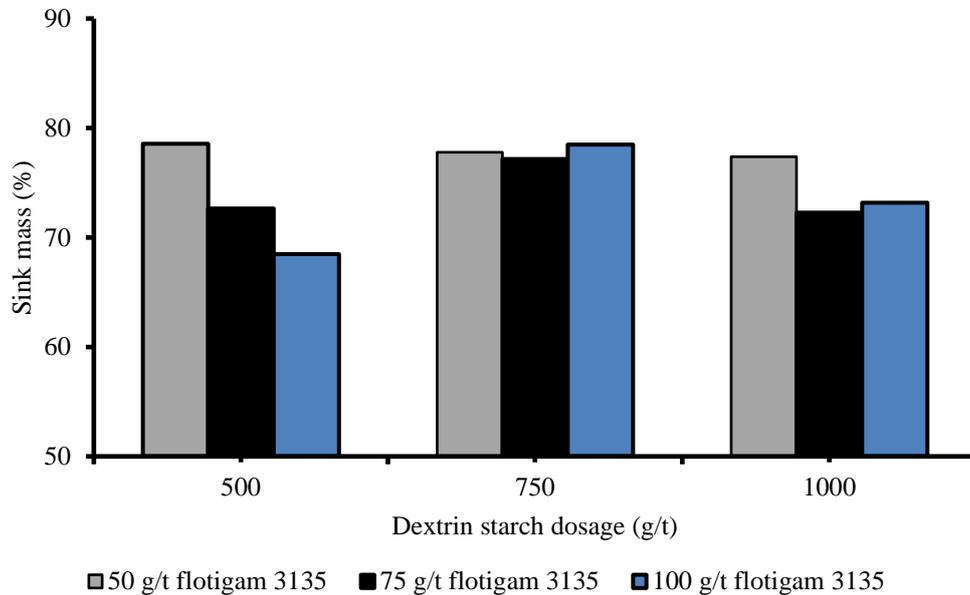


Figure 4.22: Sink mass in the two-stage reverse flotation of DPS at various flotigam 3135 and dextrin starch dosages

The data depicted in Figure 4.22, indicate that more mass reported to the sink at 750 g/t of dextrin starch, which correlates with the high recoveries shown in Figure 44.16 at the same dosage of starch. Furthermore, the use of dextrin at a dosage of 1000 g/t yielded a lower mass reporting to the sink although there was a significant increase at 50 g/t of flotigam 3135.

The ineffectiveness of dextrin to depress iron minerals might be attributed to the fact that it is derived from corn starch that is modified by the action of temperature, acidic hydrolysis or certain enzymes. Although dextrin is hydrophilic, it suffers a drastic reduction in the number of glucose units and has a too short chain length to establish "bridges" between the particles; it is still recommended to depress hematite because it is also a non-ionic surfactant (Peres and Correa, 1996; Rao, 2004; Fuerstenau and Fuerstenau, 1982).

The overall iron grade and recovery thus being far from satisfactory, necessitated that variations in other parameters such as pH, pulp density, and agitation were explored. The

further investigations were conducted using lilaflot D817M @ 75 g/t and corn starch @ 1000 g/t, based on the relatively better results obtained under these conditions, as discussed.

4.4.1 Conclusion on results of two-staged reverse flotation of direct process slimes

Amines are well known as the only cationic collectors used by industries in reverse flotation of iron ores because they ionise in water solution by protonation (Brandão, 2005). Moreover, Scott and Smith (1991) mentioned that the use of 1,3 diaminopropanes of 8C, 12C and 16C alkyl chain lengths during flotation of quartz gave better results than those observed by monoamines and that their effect is due to the doubly charged amine. However, the diamines used in this study showed only a slight progression on floating silica though it was expected that selective flotation should occur due to the ability of diamines to float quartz at coarse to medium size fractions (Kumar and Raju, 2015:13; Vieira and Peres, 2010:124-128; Rao *et al.*, 2001:27-29). It was speculated that the inability of diamines to fully float quartz might be attributed to collector being adsorbed on both silica and iron minerals as a result of the narrow pH values that occur when quartz and hematite are oppositely charged during amine flotation of silicates (Scott and Smith, 1991).

For the work of Ma *et al.* (2012:56-61) non-selectivity during flotation of quartz from hematite can also be attributed to amines adsorbing on both quartz and hematite minerals. The SEM image and particle size and deportment analyses results showed that the slimes consist of iron being more in the fine-ultrafine fractions. Therefore, it is also suspected that the non-selective flotation of the slimes might be due to fine iron particles being carried to the froth due to entrainment although it was believed they would have been depressed by starch. Hetero-coagulation of ultrafine quartz particles with coarser hematite particles and hetero-coagulation of ultrafine hematite particles with coarser quartz particles might also have contributed to the non-selective flotation of slimes, because they mask the surface properties of coarser particles and significantly reduce the selective adsorption of starch on hematite particles (Fuerstenau *et al.*, 1985); Usui, 1972:223-226). Slime coating was also suspected to be one of the factors that contributed to the non-selective flotation of slimes, because the fine iron particles might have coated the medium and medium-coarse silica particles which resulted in silica grade still being present in the sink fraction.

The better performance of corn starch compared to dextrin might be due to zein (most abundant corn protein) content, because it is as efficient as amylopectin and conventional corn starch (Fuerstenau *et al.*, 1967). A study conducted by Pinto *et al.* (1992:469-478) also confirms that corn starch is more effective to depress hematite due to its zein content. The mass percent of the sink fraction obtained from variation on dosages of both collector and depressant studied in this investigation was compared to that of Turrer and Pres (2010:1066-1069) in which it was shown that it is possible to attain a decrease in the percentage iron recovery in the floats when the depressant dosage increases. The same trend was almost observed for the ore slimes under investigation as an increase in depressant dosage resulted in more mass remaining in the sink fraction which would result in less iron recovery in the floats. However, the trend was not consistent for some dosage of depressants used, which might be due to the response of the ore slimes to the collectors dosages used. Also, the requirement for high dosages of both the depressant and collector may be due to the fact that fine particles have higher surface area per mass, and need relatively more reagents (Pease *et al.*, 2005:831-840; Ansari, 1997:93-102). Overall, the iron and quartz grade in the sink fail to meet target concentrate specifications, and other variables had to be explored.

4.5 Effect of pH on the two-staged flotation of direct process slimes

The results of the tests conducted on DPS at different pH values are shown in Table 4.7.

Table 4.7: The response of the DPS on two-staged reverse flotation at different pH values

		Grade			Recovery		
Products	Mass (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)
Float at pH 11	32.02	39.77	23.11	8.13	28.03	35.65	38.26
Sink at pH 11	67.98	48.10	19.65	6.18	71.97	64.35	61.74
Float at pH 11.5	34.39	41.66	22.18	8.38	31.02	36.73	39.10
Sink at pH 11.5	65.61	46.68	20.02	6.84	68.98	63.27	60.90

The results in Table 4.7 show that at pH 11 and 11.5, the iron grade and recovery in the sink fraction decreased, compared to pH of 10.5 which gave Fe content of 51.2% at recovery of 86.1% and silica of 16.9 %. The results are contrary to the expectation because at these pH values investigated amines are expected to be more dissociated and to enhance selective flotation (Ralston *et al.*, 1994:102-112; Rao, 2004). Therefore, this justifies the need not to float at pH values above 10.5.

4.5.1 Conclusion on results of two-staged reverse flotation of direct process slimes at various pH values

It was suggested that increasing pH values from 10.5 to 11.5 (where it is believed that amines are more effective) would result in quartz being more charged and enable electric interaction between amine cations to promote adsorption. However, the results obtained in the investigation showed that negatively charged iron mineral surfaces also responded to the increased ions in solution, hence the grade was detected in the float fraction although it was believed that they would have been depressed by starch (Smith and Scott, 1990).

4.6 Effect of Impeller Speed on the Two-staged Reverse Flotation of the Direct Process Slimes

The results of the tests conducted on DPS at different impeller speeds are shown in Table 4.8

Table 4.8: The response of DPS on two-staged reverse flotation at different impeller speeds

		Grade			Recovery		
Products	Mass (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)
Float at 1300 rpm	33.70	41.91	20.76	8.76	31.49	34.53	39.37
Sink at 1300 rpm	66.30	46.34	20.00	6.99	68.51	65.67	60.63
Float 1400 rpm	33.62	42.67	21.81	8.38	31.96	35.44	38.83
Sink at 1400 rpm	66.38	46.01	20.12	6.89	68.04	64.56	61.17

The SEM image (Figure 4.1) along with the particle and mineral department analyses results showed that most of the silica reported to the medium-coarse and medium fractions with a small quantity to the fine-ultrafine fractions. As the flotation of coarse silica particles would mostly be promoted by the diamine used, it was hoped that increasing the impeller speed would result in increasing the low momentum of fine silica particles and enhance the bubble-particle collision to increase the overall silica flotation from the slime (Pease *et al.*, 2005:831-840; Ansari, 1997:93-102; Kumar and Raju 2015; Sivamohan, 1990:247-288). However, the results obtained in Table 4.8 clearly showed that fine iron particles also responded to the increased momentum as increasing the impeller speed resulted in more iron reporting to the float as well. Although it was believed that iron would be depressed by starch, the higher kinetic energy promoted entrainment and frustrated selectivity.

4.6.1 Conclusion on results of two-staged reverse flotation of direct process slimes at various impeller speed

It was expected that increasing rotor speed from 1200 to 1400 rpm would result in more collisions and increase the interaction of fine particles with reagents for selective flotation to occur (Lima *et al.*, 2013:182). However, this was not the case for the ore slimes investigated, as increasing the rotor speed resulted in more quartz being detected in the sink fraction which might be due to the quartz particles being detached from the air bubbles.

4.7 Effect of Pulp Density on the Two-staged Reverse Flotation of Direct Process Slimes

The results of the tests conducted on DPS at different feed percentage solid are shown in Table 4.9.

Table 4.9: The response of the DPS on the two-staged reverse flotation results at different feed percentage solids

		Grade			Recovery		
Products	Mass (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)
Float at 35.0% solids	21.39	26.94	34.93	9.24	12.29	38.64	23.83
Sink at 35.0% solids	78.61	52.30	15.09	8.03	87.71	61.36	76.17
Float at 25.0% solids	27.63	26.69	34.39	8.71	15.82	49.33	32.23
Sink at 25.0% solids	72.37	54.22	13.49	6.99	84.18	50.67	67.77

From results presented in Table 4.9, decreasing feed percentage solid to 35.0% and 25.0% resulted in an increase of Fe content in the sink. The results were encouraging as silica content was decreased to 13.5% with Fe of 54.2% and recovery of 84.1% at feed percentage solid of 25.0%. Low percentage solids create a great distance between particles hence they can be selectively attached to the air bubbles and this results in better recovery (Wills, 2006:267-274; Thella *et al.*, 2012:418-426). Therefore, further tests were conducted at 25.0% feed percentage solids, with the same conditions used in section 3.3.3.

4.7.1 Conclusion on results of the two-staged reverse flotation on direct process slimes at various pulp density

Diluting the flotation pulp from 35.0% to 25.0% resulted in better selectivity as the iron grade was increased in the sink fraction. Therefore, this indicated that the ore slimes respond better to flotation at pH 10.5, rotor speed of 1200 rpm and pulp density of 25.0%

4.8 Flotation of Magnetic Separation Processed Slimes

The results of flotation tests conducted on the full size distribution of the pre-concentrated slimes are shown in Table 4.10.

Table 4.10: The response of MSPS to two-staged reverse flotation

				Grade			Recovery		
Products	Time (min)	Mass (g)	Mass (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe (%)	SiO ₂ (%)	Al ₂ O ₃ (%)
Float 1	3	90.80	9.66	57.90	9.60	0.99	8.88	19.25	12.86
Float 2	5	130.00	13.83	49.60	13.19	1.92	10.89	37.87	35.71
Final float (calc.)	8	220.80	23.49	53.01	11.71	1.54	19.76	57.12	48.57
Sink		719.00	76.51	66.09	2.70	0.50	80.24	42.88	51.43

Results obtained in Table 4.10, show that introducing pre-concentration by high gradient magnetic separator on the ore slimes followed by reverse cationic flotation can be considered as a favourable route for beneficiating the slimes, as silica content in the flotation concentrate was decreased from 5.0% to 2.7%, and with a minor increase of iron content from 64.0% to 66.0% at a recovery of 80.2%.

4.9 Two-staged Reverse Flotation of DPS and MSPS at Narrow Particle Size Distribution

The results of the tests conducted on DPS at narrow size distribution are shown in Figure 4.23.

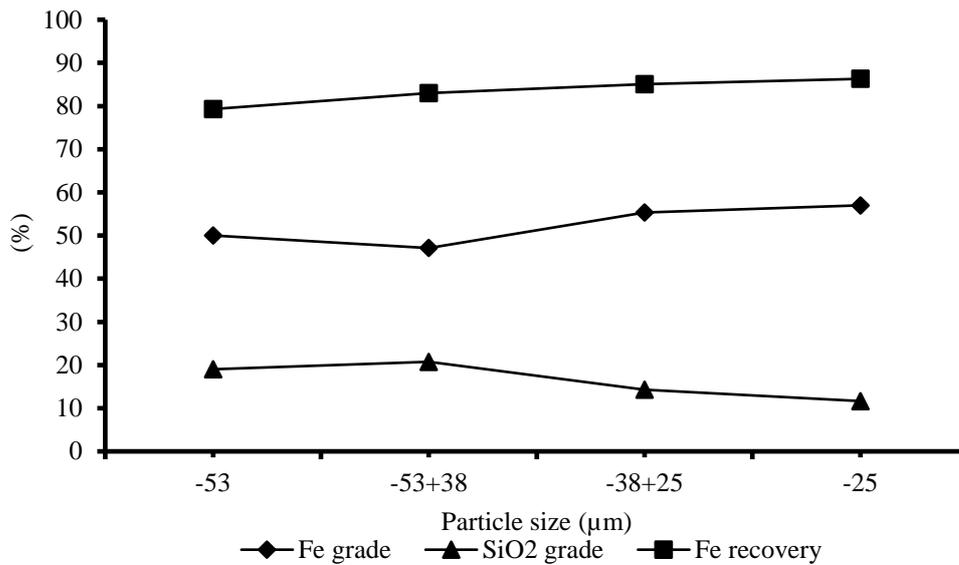


Figure 4.23: Iron grade, silica grade and iron recovery in the sink fraction obtained from two-stage reverse flotation at narrow particle size distribution on the direct process slimes

It can be seen from Figure 4.23 that floating at narrow particle size distribution might be a favourable route to beneficiate the ore slimes. The Fe content was significantly increased to 57.0% with recovery of 86.3% at -25 µm fraction which correlated with the mineral department analyses results and SEM images that revealed most of the iron reported to the very fine fraction. Furthermore, silica content was also significantly decreased from 13.5% to 11.7%. This shows that the best way to beneficiate the ore slimes by flotation is to separately float the -25 µm fraction from the + 25 µm fraction.

Figure 4.24 shows how the froth structure of flotation on the – 25 µm DPS looked like.



Figure 4.24: Flotation at -25 micron fraction on the direct process slimes

Similarly, results obtained from reverse cationic flotation of the MSPS slime at narrow particle size distribution are shown in Figure 4.25.

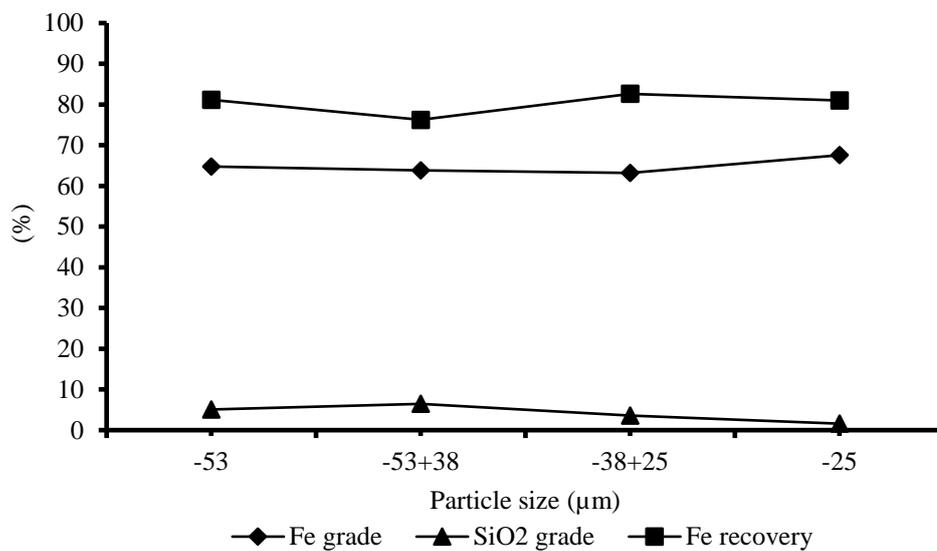


Figure 4.25: Iron grade, silica grade and iron recovery in the sink fraction obtained from two-stage reverse flotation at narrow particle size distribution of MSPS

The results obtained in Figure 4.25 show that – 25 μm fraction gave 1.6% silica and 68.0% iron with recovery of 81.0%. This clearly improved separation might have been attributed to particles likely to be evenly distributed and promoting the ease with which particles can attach

to bubbles. The grades from the +25 μm fraction are poor compared to the -25 μm , implying that a separate flotation regime needs to be investigated for the coarser fraction. In this investigation, the best condition has been sought for the -25 μm fraction which is the larger proportion in the slimes.

4.9.1 Conclusion on results of two-staged reverse flotation on DPS and MSPS at narrow particle size distribution

Slimes in this investigation would certainly have different flotation kinetics responses due to different distribution of mineral particles (Pease *et al.*, 2005:831-840; Thella *et al.*, 2012:418-426. Trahar, 1981:289-327). Hence, it was hypothesised that floating at narrow particle size distribution would result in selective flotation of quartz from hematite. The results obtained on two-staged flotation of DPS at narrow particle size distribution were nearly as expected, as the iron grade in the sink fraction of – 25 microns was higher compared to the flotation of the whole size fraction of the slimes. Furthermore, flotation over a narrow particle size distribution of MSPS gave exceptional results, as iron and silica grades were increased from 63.7% to 68.0% and 4.8% to 1.6%, respectively.

Therefore, pre-concentration via pulsating high gradient magnetic separation before the narrow particle size flotation, will ensure that the target concentrate specification is achieved in the concentrate. The recommended approach for Sishen mine slimes is to pre-concentrate it with sLon pulsating high gradient magnetic separator, screen the slimes at 25 μm and follow a two-stage reverse flotation process as illustrated in Figure 4.26. Reverse flotation of the whole size fraction also meet the specific target and can be considered for industrial purposes as constraints might occur to classify and beneficiate at fine-ultrafine fractions at industrial scale.

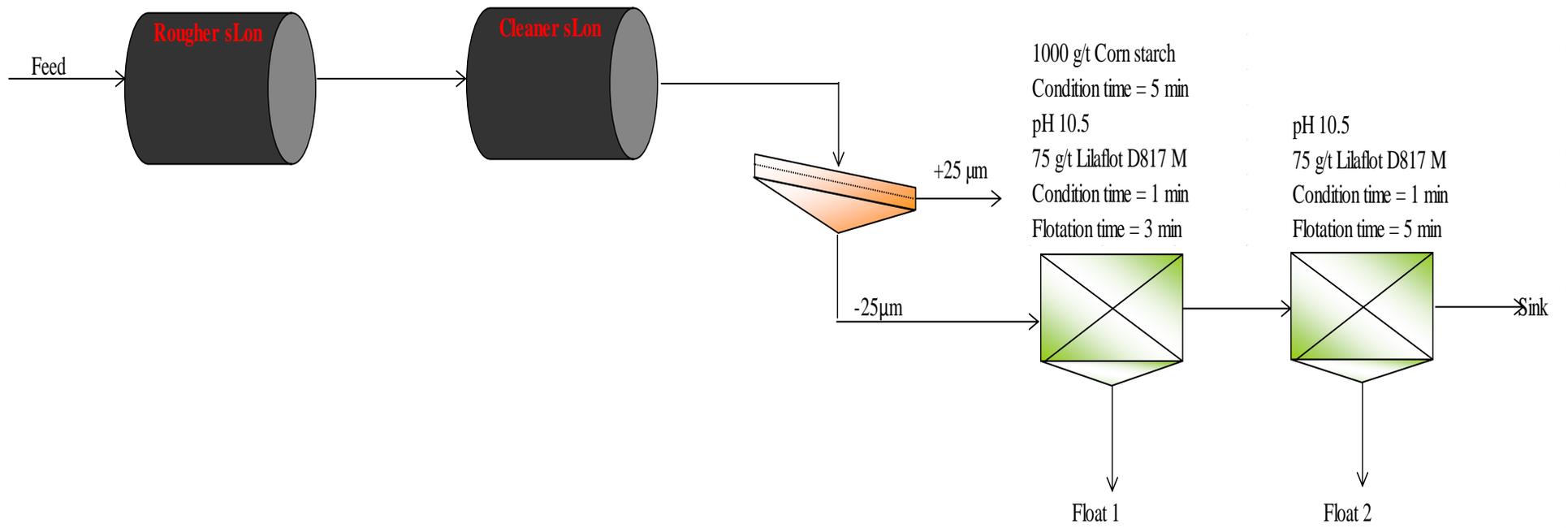


Figure 4.26: Proposed Sishen mine iron ore slimes beneficiation flowsheet

CHAPTER 5

5. CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Suitable beneficiation route for iron ore slimes from Sishen mine were investigated with the aim of achieving a concentrate with less than 5.0% silica + alumina and more than 63.0% iron which will be suitable for pellets that can be fed to a blast furnace for the iron making process. The characterisation study done on the ore slimes as-received showed that the sample contains 44.0% iron with major impurities as silica and aluminium consisting of 20.4% and 8.7% respectively which makes the ore slimes worthwhile to beneficiate.

Preliminary direct flotation tests conducted on the direct process slimes, using tall oil and linoleic acids as collectors were not an effective route to beneficiate the ore slimes as the results obtained were far from meeting the desired concentrate target for pelletizing. This was to be expected because the iron content is much higher in the slimes, and reverse flotation is more appropriate. The test conducted on the -25 micron direct process slimes fraction, using the flotation test conditions derived from the previous two-staged reverse flotation tests resulted in an Fe content of 57.0% with SiO₂ of 11.7%.

Flotation of direct process slimes could not increase the Fe and reduce the silica + alumina content to satisfy pelletizing requirements. However, magnetic separation processed slimes flotation, resulted in a satisfactory product which was obtained by screening at 25 µm before reverse flotation on the -25 µm fraction with lilafлот D817M collector (for silica) and corn starch (to depress iron oxides). At 75 g/t (which in total is 150.0 g/t from two stages) lilafлот D817M collector and 1000 g/ton corn starch depressant, gave the best concentrate of 0.1% Al₂O₃ and 1.6% SiO₂ (which is 1.7% silica + alumina) and 68.0% Fe which will render the product concentrate suitable for pelletizing. Although reverse flotation on the -25 micron magnetic separation processed slimes fraction gave the best concentrate, the 0.5% alumina, 2.7% silica and 66.0% iron obtained from reverse flotation of the whole size fraction also met the specific target and could thus be a recommended route for industrial purposes since it does not require classification at fine-ultrafine fractions.

Therefore, the proposed route to beneficiate the Sishen mine slimes, is to first use sLon pulsating high gradient magnetic separator as pre-concentration technique and subject the

slimes to two-staged reverse flotation using lilafloat D817M collector at 75 g/t and corn starch depressant at 1000 g/t.

5.2 Recommendation

The development of a beneficiation route for Sishen mine slimes (generated prior to jig operations) is still in its initial development stages. The following aspects may also be considered for future work:

- The particle size department analysis showed that more mass and Fe reported to the fine-ultrafine fractions. It is, therefore, recommended that the slimes be treated by beneficiating the fine-ultrafine fractions only.
- More flotation tests should be conducted using ether monoamine collectors in conjunction with corn starch.
- A feasibility study should be conducted into the economic viability of froth flotation as a beneficiation means of Sishen mine slimes.
- Further investigation of favourable kinetics for the coarse size fraction should also be considered.

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6. APPENDIX A

Appendix A show the formulas used to calculate recovery and grade of the minerals. The physical and chemical properties of reagents and dosage calculations is also shown.

Calculation of recovery:

$$\text{Recovery} = \frac{\text{mass (\%)} \times \text{Grade (\%)}}{\text{Head mass (\%)}} \times 100$$

Cumulative recovery = Recovery 1 + Recovery 2 + Recovery 3

Cumulative grade = Grade 1 + Grade 2 + Grade 3

Reagent dosage calculations:

$$X \text{ ml} = \frac{A \text{ (kg)} \times 100 \times B \text{ (g/t)}}{1000} \times C \text{ (\%)}$$

Where X represents required dosage for flotation test in ml, A represents mass of the sample in kilograms, B represents dosage required for flotation test in g/t and C represents strength of the reagent in percentage

Chemical and physical properties of Flotigam 3135

Form:	Liquid
Colour:	Yellow to brown
Odour:	amine-like
pH value:	approx. 9.2 (10 g/t)
Freezing point:	<-5 ⁰ C
Boiling point:	approx. 83 ⁰ C
Flash point:	91 ⁰ C
Solubility in water:	20g/l
Ignition temperature:	>200 ⁰ C
Thermal decomposition:	>21 ⁰ C
Viscosity:	approx. 475mPas (20 ⁰ C)
Carbon Chain Length	C8 - C10

Degree of Neutralization 33 - 37 %

Chemical and physical properties of Lilafлот D817M

Form: Liquid
Density: 918 kg/m³ at 20⁰C
Flash point: >100 ⁰C
Thermal decomposition: >21⁰C
Viscosity: 420mPas (20⁰C)
Viscosity: 100mPas (10⁰C)

Carbon Chain Length C8 - C16

Degree of Neutralization 34 - 40 %

7. APPENDIX B

Appendix B shows the image of XRF machine (Figure 7.1) that was used to analyse the samples. The images of Denver float machine, wet screening and pressure filtering apparatus are also shown in Figures 7.2-7.4 as they were used during tests conducted in the investigation.



Figure 7.1: XRF machine



Figure 7.2: Denver float machine



Figure 7.3: Wet screening apparatus



Figure 7.4: Pressure filtering apparatus

8. APPENDIX C

Appendix C (Tables 8.1 and 8.2) show a summary of raw data for particle size assay conducted on the direct process slimes and magnetic separation processed slimes.

Table 8.1: Particle size assay of direct process slimes

Particle size	Mass	Mass	Cum passing	Grade						Recovery			P ₂ O ₅	K ₂ O	CaO
				Fe	SiO ₂	Al ₂ O ₃	P ₂ O ₅	K ₂ O	CaO	Fe	SiO ₂	Al ₂ O ₃			
(µm)	(g)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
+75	9,2	0,5	99,5	19,3	63,8	17,0	0,3	1,9	2,1	0,2	1,5	1,2	0,5	0,8	0,6
-75+53	69,7	3,6	95,9	28,9	54,9	14,3	0,3	2,2	1,9	2,2	10,0	7,4	3,6	7,2	3,9
-53+38	83,8	4,4	91,5	41,7	26,5	12,8	0,3	2,0	1,8	3,9	5,8	7,9	4,4	7,7	4,5
-38+25	79,2	4,1	87,4	43,5	24,0	10,8	0,3	0,7	1,5	3,8	5,0	6,3	3,9	2,7	3,3
-25+10	97,1	5,1	82,3	46,5	20,3	9,9	0,3	1,4	1,3	5,0	5,1	7,1	5,3	6,2	3,8
-10+5	283,3	14,8	67,5	47,7	18,6	7,2	0,3	1,0	1,3	15,1	13,8	15,0	13,5	13,3	10,4
-5	1295,3	67,5	0,0	48,3	17,4	5,7	0,3	1,0	2,0	69,7	58,8	55,0	68,7	62,1	73,6
Head (calc.)	1917,6	100,0		46,8	20,0	7,0	0,3	1,1	1,8	100,0	100,0	100,0	100,0	100,0	100,0
Head (meas.)	1700			44,0	20,4	8,6	0,3	1,7	1,2						

Table 8.2: Particle size assay of magnetic separation processed slimes

Particle size	Mass	Mass	Cum passing	Grade						Recovery			P ₂ O ₅	K ₂ O	CaO
				Fe	SiO ₂	Al ₂ O ₃	P ₂ O ₅	K ₂ O	CaO	Fe	SiO ₂	Al ₂ O ₃			
(µm)	(g)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
+75	7,7	0,5	99,5	51,1	38,0	9,9	0,2	0,9	0,7	0,4	4,3	5,6	1,3	1,5	2,7
-75+53	34,8	2,3	97,2	50,3	35,9	7,7	0,2	1,2	0,5	1,8	18,4	19,8	7,1	8,8	9,1
-53+38	127,4	8,5	88,7	58,5	11,6	4,8	0,2	0,8	0,2	7,7	21,7	45,7	20,9	20,4	14,8
-38+25	99,2	6,6	82,1	63,5	8,0	0,8	0,1	0,5	0,1	6,5	11,7	5,7	12,2	9,8	6,7
-25+10	133,3	8,9	73,3	64,9	4,4	0,2	0,1	0,3	0,1	8,9	8,7	2,2	11,0	7,3	4,5
-10+5	100,1	6,6	66,6	65,6	2,5	0,1	0,1	0,2	0,2	6,8	3,7	1,0	6,2	3,8	8,7
-5	1002,9	66,6	0,0	65,9	2,1	0,3	0,0	0,2	0,1	68,0	31,5	20,1	41,2	48,5	53,4
Head (calc.)	1505,4	100,0		64,6	4,5	0,9	0,1	0,3	0,1	100,0	100,0	100,0	100,0	100,0	100,0
Head (meas.)	1500			63,7	4,8	1,9	0,1	0,1	0,1						

9. APPENDIX D

Appendix D Tables 9.1 and 9.2 show a summary of raw data for all flotation testwork conducted on the direct process slimes using lilafлот D817M and flotigam 3135 collector (at different dosages) and corn starch (at different dosages).

Table 9.1: Flotation testwork conducted on the direct process slimes using Lilafлот D817M collector (at different dosages) and corn starch (at different dosages)

Corn starch @ 500 g/t

Products	Grade									Recovery								
	Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t			Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Float 1 + Float 2	41,51	21,97	10,96	40,65	23,24	10,84	42,80	23,43	0,26	19,01	22,83	27,06	19,40	24,33	27,02	19,84	24,65	25,48
Sink	47,72	20,04	7,30	47,02	20,12	7,72	48,24	19,98	0,21	80,99	77,17	72,94	80,60	75,67	72,98	80,16	75,35	74,52
Feed (calc.)	46,40	20,45	8,08	45,63	20,80	8,40	47,05	20,73	0,22	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

Corn starch @ 750 g/t

Products	Grade									Recovery								
	Lilafлот @ 50 g/t			Lilafлот @ 75 g/t			Lilafлот @ 100 g/t			Lilafлот @ 50 g/t			Lilafлот @ 75 g/t			Lilafлот @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Float 1 + Float 2	40,22	21,91	10,09	41,46	26,10	0,27	41,45	22,94	0,26	19,25	22,96	23,43	20,25	28,87	28,22	18,87	23,80	24,68
Sink	47,06	20,51	7,58	48,24	19,00	0,20	47,83	19,72	0,21	80,75	77,04	76,57	79,75	71,13	71,78	81,13	76,20	75,32
Feed (calc.)	45,57	20,82	8,13	46,69	20,62	0,22	46,48	20,40	0,22	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

Corn starch @ 1000 g/t

Products	Grade									Recovery								
	Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t			Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Float 1 + Float 2	31,69	28,20	0,25	30,73	31,32	0,21	33,60	27,85	0,23	13,07	27,00	21,67	13,88	33,08	23,21	14,79	28,30	23,22
Sink	50,01	18,09	0,21	51,15	17,00	0,19	49,89	18,18	0,20	86,93	73,00	78,33	86,12	66,92	76,79	85,21	71,70	76,78
Feed (calc.)	46,50	20,03	0,22	46,83	20,03	0,20	46,55	20,16	0,21	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

A summary of raw data is shown in Table 9.2 for all flotation testwork conducted on direct process slimes using Flotigam 3135 collector (at different dosages) and corn starch (at different dosages).

Table 9.2: Flotation testwork conducted on direct process slimes using Flotigam 3135 collector (at different dosages) and corn starch (at different dosages)

Corn starch @ 500 g/t

Products	Grade									Recovery								
	Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t			Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	
Float 1 + Float 2	43,65	20,66	9,23	42,83	23,18	9,72	42,14	22,35	8,58	19,73	20,45	25,28	26,21	31,43	34,42	25,51	30,77	34,54
Sink	46,61	21,09	9,12	47,86	20,07	7,35	48,92	19,99	6,46	80,27	79,55	74,72	73,79	68,57	65,58	74,49	69,23	65,46
Feed (calc.)	46,00	21,00	9,14	46,43	20,95	8,02	46,99	20,66	7,06	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

Corn starch @ 750 g/t

Products	Grade									Recovery								
	Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t			Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	
Float 1 + Float 2	41,44	22,99	9,01	42,55	22,48	9,95	41,51	21,85	8,39	20,56	25,95	29,79	19,54	23,47	27,78	18,95	22,17	26,61
Sink	48,71	19,96	6,46	47,85	20,02	7,06	47,79	20,65	6,23	79,44	74,05	70,21	80,46	76,53	72,22	81,05	77,83	73,39
Feed (calc.)	47,01	20,67	7,05	46,71	20,55	7,68	46,46	20,90	6,69	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

Corn starch @ 1000 g/t

Products	Grade									Recovery								
	Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t			Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	
Float 1 + Float 2	41,02	21,83	9,12	43,30	21,63	9,83	42,23	21,36	9,18	18,23	22,36	27,85	21,22	23,40	28,48	19,19	21,23	23,01
Sink	48,49	19,98	6,23	47,09	20,75	7,23	47,02	20,95	8,12	81,77	77,64	72,15	78,78	76,60	71,52	80,81	78,77	76,99
Feed (calc.)	46,93	20,37	6,83	46,23	20,95	7,82	46,02	21,04	8,34	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

10. APPENDIX E

Appendix E show a summary of raw data (Tables 10.1 and 10.2) for all flotation testwork conducted on direct process slimes using lilafлот D817M and flotigam 3135 collectors (at different dosages) and dextrin starch (at different dosages).

Table 10.1: Flotation testwork conducted on direct process slimes using Lilafлот D817M collector (at different dosages) and dextrin starch (at different dosages)

Dextrin starch @ 500 g/t

Products	Grade									Recovery								
	Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t			Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Float 1+ Float 2	41,73	20,65	9,76	41,94	21,83	10,59	41,53	23,09	10,23	16,85	18,21	21,48	15,76	18,22	21,72	14,65	18,43	19,27
Sink	41,73	20,65	9,76	46,89	20,50	7,99	47,38	20,00	8,39	16,85	18,21	21,48	84,24	81,78	78,28	85,35	81,57	80,73
Feed (calc.)	46,61	21,00	8,08	46,03	20,73	8,44	46,42	20,51	8,69	83,15	81,79	78,52	100,00	100,00	100,00	100,00	100,00	100,00

Dextrin starch @ 750 g/t

Products	Grade									Recovery								
	Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t			Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Float 1 + Float 2	40,31	23,89	10,03	39,40	23,00	10,48	42,56	21,16	10,00	14,47	19,57	20,08	13,57	18,38	19,85	15,55	17,58	20,04
Sink	48,24	19,87	8,08	48,33	19,67	8,15	47,20	20,26	8,15	85,53	80,43	79,92	86,43	81,62	80,15	84,45	82,42	79,96
Feed (calc.)	46,90	20,55	8,41	46,89	20,21	8,53	46,41	20,41	8,46	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

Dextrin starch @ 1000 g/t

Products	Grade									Recovery								
	Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t			Lilafлот D817M @ 50 g/t			Lilafлот D817M @ 75 g/t			Lilafлот D817M @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Float 1 + Float 2	40,78	21,13	10,31	41,26	21,04	10,20	41,99	22,54	10,26	13,46	15,09	18,89	13,61	15,94	18,45	14,66	17,57	19,34
Sink	46,11	20,90	7,78	47,20	20,00	8,12	47,05	20,36	8,24	86,54	84,91	81,11	86,39	84,06	81,55	85,34	82,43	80,66
Feed (calc.)	45,31	20,93	8,16	46,29	20,16	8,44	46,23	20,71	8,57	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

A summary of raw data is shown in Table 10.2 for all flotation testwork conducted on direct process slimes using flotigam 3135 collector (at different dosages) and dextrin starch (at different dosages).

Table 10.2: Flotation testwork conducted on direct process slimes using Flotigam 3135 collector (at different dosages) and dextrin starch (at different dosages)

Dextrin starch @ 500 g/t

Products	Grade									Recovery								
	Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t			Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	
Float 1 + Float 2	43,94	21,87	9,78	44,02	21,69	9,87	42,57	21,84	9,51	20,46	22,22	24,00	26,11	28,31	33,07	25,51	30,77	34,54
Sink	46,61	20,89	8,45	46,90	20,68	7,52	48,02	19,52	8,05	79,54	77,78	76,00	73,89	71,69	66,93	74,49	69,23	65,46
Feed (calc.)	46,04	21,10	8,73	46,11	20,96	8,16	46,30	20,25	8,51	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

Dextrin starch @ 750 g/t

Products	Grade									Recovery								
	Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t			Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	
Float 1 + Float 2	41,19	22,38	9,67	40,07	21,41	10,01	42,43	22,03	9,91	19,78	24,20	27,52	19,71	24,17	27,03	19,57	22,54	25,82
Sink	47,71	20,03	8,09	48,33	19,89	8,00	47,79	20,75	7,80	80,22	75,80	72,48	80,29	75,83	72,97	80,43	77,46	74,18
Feed (calc.)	46,26	20,55	8,44	46,44	20,24	8,46	46,64	21,03	8,25	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

Dextrin starch @ 1000 g/t

Products	Grade									Recovery								
	Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t			Flotigam 3135 @ 50 g/t			Flotigam 3135 @ 75 g/t			Flotigam 3135 @ 100 g/t		
	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃	Fe	SiO ₂	Al ₂ O ₃
(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	
Float 1 + Float 2	42,11	22,20	9,76	43,37	22,95	9,85	43,19	23,82	9,39	20,59	24,50	28,17	25,72	30,72	32,02	24,65	31,18	30,03
Sink	47,50	20,01	8,01	48,09	19,87	8,03	48,43	19,29	8,03	79,41	75,50	71,83	74,28	69,28	67,98	75,35	68,82	69,97
Feed (calc.)	46,28	20,51	8,41	46,78	20,72	8,54	47,02	20,51	8,40	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00	100,00

