



**Froth flotation of an Nkomati mineral ore using mixtures of thiol  
collectors**

**By**

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## **LIST OF PRESENTATIONS**

Maree, W., Kloppers, L., Hangone, G. and Oyekola, O. 2015. The effects of mixtures of potassium amyl xanthate and IPETC collectors on grade and recovery in the froth flotation of an Nkomati nickel mineral ore. Presented at *Minerals Engineering Conference Nickel Processing '15*, Falmouth.

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## **DECLARATION**

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## **ABSTRACT**

Nickel, a valuable base metal, is the predominant product from the Nkomati mine in South Africa. High-grade nickel mineral ores (2%) from the Massive Sulphide Body have been depleted leaving those of low grade (0.3%). The beneficiation of this ore presents a challenge to the minerals processing industry. In response, batch froth flotation tests were used to explore the effects of mixtures of potassium amyl xanthate (PAX) and IPETC (Isopropyl ethyl thionocarbamate) on the grades and recoveries of nickel. In the mixtures, the xanthate accounted for 95.5, 90, 85 and 80 mole% respectively. Generally an increase in the nickel grade and recovery was observed with the mixtures relative to PAX. IPETC gave a significant increase in the cumulative water recovery with a significant decrease in cumulative nickel grade relative to PAX.

PAX gave the highest cumulative nickel grade for the singular collector tests (1.9%), while obtaining the lowest cumulative recovery (77%). Out of the collector mixtures, mixtures 85% PAX: 15% IPETC and 90% PAX: 10% IPETC produced the joint highest cumulative grades (1.8%). These mixtures both gave recoveries of 82%. Collector mixture 95.5% PAX: 4.5% IPETC gave the second highest grade (1.7%) and the highest nickel recovery (85%). Although there were differences in the cumulative nickel grades and recoveries there were statistically no significant improvements observed with the use of the mixtures of PAX and IPETC in comparison to the industry mixture (95.5% SIBX: 4.5% IPETC).

Tests were performed using the three best performing collector mixtures at molar dosages of 1.3, 0.65 and 0.325mmol/t. The highest cumulative nickel grades were obtained at the lowest collector dosages (at a molar dosage of 0.325mmol/t of 95.5% SIBX: 4.5% IPETC) with collector mixture 95.5% PAX: 4.5% IPETC being the most selective with a nickel grade of 2%. It was also observed that an increase in collector dosage, generally increased the cumulative nickel recovery with collector mixture 95.5% PAX: 4.5% IPETC at a molar dosage of 1.3mmol/t gave the highest cumulative recovery out of the tested mixtures (85%).

The study also indicated that an increase in selectivity (i.e. cumulative grade) was at the expense of cumulative recovery. With a decrease in dosage, there was no significant improvement in the cumulative nickel grade and recovery for the tested mixtures compared to the industry mixture. There was however a significant decrease in the water recoveries achieved with PAX and collector mixtures 95.5% PAX: 4.5% IPETC at a molar dosage of 1.3mmol/t of as well as mixture 90% PAX: 10% IPETC at a molar dosage of 0.65mmol/t.

This suggests using PAX at a molar dosage of 1.3mmol/t and mixture 90% PAX: 10% IPETC at a molar dosage of 0.65mmol/t would be beneficial in application, because of the lower reagent requirement for PAX (no IPETC required) and a lower dosage requirement for mixture 90% PAX: 10% IPETC (a molar dosage of 0.65mmol/t instead of 1.3mmol/t). These changes would allow savings on reagents as well as decrease the water recovery without compromising the nickel grade and recovery.

When the frother dosage was increased from 12g/t to 50g/t, the cumulative nickel grades and recoveries for the xanthate collectors (PAX and SIBX) was significantly higher ( 2cm higher). This was due to a more stable froth phase. At a frother dosage of 12g/t, the froth phase formed was inadequate to help facilitate efficient flotation. It was also evident that apart from the increase in water recovery, there were no benefits associated with the use of frother at 50g/t as opposed to 12g/t for the mixtures.

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## **DEDICATION**

- To my late grandfather, Mr Eric Bethren Ludick, who did not live long enough to see what I have accomplished and the person I've become.
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## GLOSSARY OF TERMS

- **Grade** is the mass percentage of the desired valuable metal in an ore body.
- **Collectors** are chemical reagents used to selectively alter the hydrophobicity of a particular mineral's surface.
- **Recovery** is the mass percentage of desired metal recovered in the concentrates.
- **Ore** is rock that contains minerals with significant economic value and is normally extracted from the earth through mining.
- **Pulp** is the homogeneous mixture of a crushed ore and water which is also referred to as slurry. The ore has to be in constant suspension in the water for effective froth flotation to occur in a flotation cell.
- **Froth** is a mass of small bubbles above the pulp in a flotation cell. A collective of these small mineral containing bubbles form the froth which is removed for beneficiation.
- **Froth layer/phase** is the accumulation of froth bubbles above the pulp in a flotation cell.
- **Frothers** are chemical reagents used to decrease the surface tension of water and stabilise air bubbles passing through the pulp to the froth layer.
- **Modifiers** are chemical reagents used to alter pulp conditions and to preferentially alter the surface properties of minerals. They are neither collectors nor frothers.
- **Mixing** is the process of increasing the homogeneity of a medium by using physical methods.
- **Agitation** is the physical or mechanical process used to change the flow patterns of a fluid or pulp.
- **Milling** is the processes of size reduction used to reach the target particle size distribution.

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## **LIST OF ABBREVIATIONS**

|                     |  |
|---------------------|--|
| C1                  | First concentrate, collected from 0th to 2nd minute    |
| C2                  | Second concentrate, collected from 2nd to 6th minute   |
| C3                  | Third concentrate, collected from 6th to 12th minute   |
| C4                  | Fourth concentrate, collected from 12th to 20th minute |
| Ca(OH) <sub>2</sub> | Calcium hydroxide                                      |
| k                   | Klimpel rate constant                                  |
| XRF                 | X-ray fluorescent spectrometry                         |
| R                   | Percentage recovery                                    |
| rpm                 | Revolutions per minute                                 |
| PAX                 | Potassium Amyl Xanthate                                |
| SIBX                | Sodium Isobutyl Xanthate                               |
| IPETC               | Isopropyl ethyl thionocarbamate                        |
| CMC                 | Carboxy methyl cellulose                               |
| SCMC                | Sodium carboxy methyl cellulose                        |
| MMZ                 | Main Mineralised Zone                                  |
| PCMZ                | Peridotitic Chromatic Mineralized Zone                 |

# CHAPTER 1

## 1. Introduction

### 1.1 Background

Nickel production in Africa is estimated to be 80 900 metric tons per annum. South Africa produces approximately half of that, while Zimbabwe and Botswana produce the rest. In South Africa, nickel is mainly produced as a by-product from the Bushveld platinum mines (Mbendi Information Services, 2014). However, the Nkomati mine in Mpumalanga is South Africa's only primarily nickel producing mine. The mine's estimated nickel reserves are approximately 408 million tonnes of ore at a grade of 0.33% and currently it processes approximately 650 kt ore/ month (Cockburn, 2013).

Froth flotation is the most important minerals processing technique used for the separation of valuable minerals from gangue material (Wills and Napier-Munn, 2006). To achieve effective separation by froth flotation, there are many influential variables e.g. the dosages of the various reagents, the types of collectors or collector mixtures used, agitation rate, air flow rate, particle size distribution, pulp pH and pulp density.

The separation depends largely on the differences in hydrophobicity of the various minerals. In froth flotation, collectors are used to selectively impart hydrophobicity onto the desired mineral surfaces. Other chemicals reagents used include frothers (used to increase bubble stability) and modifiers (used to create favourable pulp conditions).

Nickel, like copper, is one of the base metals for which there is a high demand. This is due to the fact that nickel is one of the major metal constituents in stainless steel (Bradley, 2011). In addition, nickel sulphide ore reserves are declining and most of the remaining ores are of low nickel grade (Cockburn, 2013). For this reason, new collectors and collector mixtures are being tested to improve the grade and recovery of nickel bearing minerals, without using excessive dosages of collectors. It is thus important that new collectors and collector mixtures not only contribute towards an increase in revenue by simultaneously improving the grade and recovery, but also promote cost cutting by using lower quantities of collectors as well.

The adsorption of collectors on the surfaces of minerals involve complex processes. The smallest change in adsorption caused by a change in pulp pH, under or over dosage of collector mixtures or unfavourable zeta potentials at the minerals surfaces could yield substantial differences in the grades and recoveries. When single collectors are used to float complex ores, the grades and recoveries of the desired minerals may be unacceptably low. This may be due to low collector

selectivity. Potential improvements may then be investigated, by optimising collector dosages and collector compatibility in mixtures to promote increased synergism.

Furthermore, sodium isobutyl xanthate (SIBX) and potassium amyl xanthate (PAX) are traditional thiol collectors used in bulk and selective froth flotation of nickel sulphide mineral ores. However, with the declining grades of complex nickel sulphide ores, these alone are not selective enough for the efficient froth flotation of these ores. Hence, new collectors and collector mixtures that are more selective, but not sensitive to deteriorating feed grades, are desired (Senior et al., 1995).

Other classes of thiol collectors such as the dithiocarbamates (DTC), dithiophosphates (DTP) and thionocarbamates (TC) improve the recovery of coarse particles, flotation rate and require smaller dosages in comparison to the traditional xanthate collectors (McFadzean et al., 2013). Isopropyl ethyl thionocarbamate (IPETC) is one such collector from the thionocarbamate (TC) class that is used due to its increased selectivity, low dosage requirements and added frothing properties. Although, thiol collectors might have improved synergistic effects when used in mixtures, some of them are known to act as frothers when used as a co-collector in collector mixtures (McFadzean et al., 2013).

Although the dithiophosphates, dithiocarbamates and thionocarbamates may be more selective than the xanthates, these are generally more costly. To save on costs and improve the grade and recovery of the desired minerals, dithiophosphates, thionocarbamates and dithiocarbamates are used in mixtures with the traditional xanthate collectors with the xanthates generally being the major constituent.

Mixtures of SIBX and IPETC are used in industry. In this study, the effects of using mixtures of PAX and IPETC at molar equivalents of 1.3mmol/t of 95.5% SIBX: 4.5% IPETC (molar dosages of 1.3mmol/t) on nickel grades and recoveries were explored at different molar ratios of the constituents. Different molar concentrations and dosages of the three best performing mixtures were used to investigate possible improvements. The effects of this mixture have not been reported in open literature. Hence, this study will provide new data that could be used for future optimisation.

Furthermore, SIBX and PAX were mixed in a 50:50 molar ratio and introduced at a molar equivalent of 1.3mmol/t of 95.5% SIBX: 4.5% IPETC (molar dosages of 1.3mmol/t) to investigate effects of this mixture on nickel grade and recovery in comparison to the industrially used collector mixture.

## 1.2 Statement of research problem

Nickel is a valuable base metal. In South Africa, the Nkomati mine is the only primarily nickel producing mine. Currently, nickel mineral ores are of low grade as those of higher grade have been depleted. Consequently, the recovery of this base metal presents a challenge to the minerals processing industry. An increase in grade and recovery of nickel from the complex sulphide ores at the Nkomati mine will have significant positive economic effects. Froth flotation is a process used to beneficiate mineral ores by increasing the grade and recovery of the desired minerals. The effects of using mixtures of collectors in froth flotation are not well understood as that of pure collectors. Therefore, the effects of mixtures of collectors at different dosages and molar ratios will be investigated and compared to a mixture used at the mine.

## 1.3 Key Questions

1. What are the effects of using the industrial mixture, 95.5 SIBX: 4.5 IPETC mol%, at a dosage of 1.3mmol/t (molar dosage of 1.3mmol/t) with frother Senmin XP200 at 50g/t and depressant Senmin Finfix 300 at 240g/t on nickel recoveries and grades; mass and water recoveries; and the rate of flotation?
2. What are the effects of changing the constituents' ratios in the mixture of PAX and IPETC at a molar dosage of 1.3mmol/t with Senmin frother XP200 at 50g/t and depressant Senmin Finfix 300 at 240g/t on nickel recoveries and grades; mass and water recoveries; and the rate of flotation. The ratios investigated for PAX and IPETC are 95.5:4.5, 90:10, 85:15 and 80:20; the xanthate being the more abundant constituent in the mixtures?
3. What are the effects of changing the dosages of the three best performing mixtures of PAX and IPETC with frother Senmin XP200 at 50g/t and depressant Senmin Finfix 300 at 240g/t on nickel recoveries and grades; mass and water recoveries; and the rate of flotation. The dosages investigated for PAX and IPETC were 0.65mmol/t and 0.325mmol/t?
4. What are the effects of using a mixture of PAX (50 mol%) and SIBX (50 mol%) at a molar dosage of 1.3mmol/t with frother Senmin XP200 at 50g/t and depressant Senmin Finfix 300 at 240g/t on nickel recoveries and grades; mass and water recoveries; and the rate of flotation?
5. What are the effects of changing the constituents' ratios in the mixture of PAX and IPETC at a molar dosage of 1.3mmol/t with frother Senmin XP200 at 12g/t and depressant Senmin Finfix 300 at 240g/t on nickel recoveries and grades; mass and water recoveries; and the rate of flotation. The ratios investigated for PAX and IPETC are 95.5:4.5, 90:10, 85:15 and 80:20; the xanthate being the more abundant constituent in the mixtures?

#### 1.4 Aim and Objectives

The aim is to investigate the effects of using different mixtures and dosages of a traditional xanthate collector potassium amyl xanthate (PAX) and a more selective isopropyl ethyl thionocarbamate collector (IPETC) on the grade and recovery of a nickel mineral ore.

The objectives for the current study are as follows:

- 1- Investigate the effects of an industrially used collector mixture on grade and recovery.
- 2- Investigate the effects on grade and recovery of flotation with no collector, potassium amyl xanthate (PAX) and Isopropyl ethyl thionocarbamate (IPETC).
- 3- Investigate the effects on grade and recovery of mixtures of PAX and IPETC at different molar ratios and dosages. Molar ratios of 95.5:4.5, 90:10, 85:15 and 80:20 were investigated. The effect of molar dosages 1.3, 0.65, and 0.325mmol/ton of the three best performing mixtures in terms of grade and recovery were investigated.
- 4- Investigate the effects on grade and recovery of PAX and SIBX in a 50:50 mixture.
- 5- Investigate the effects on grade and recovery of mixtures of PAX and IPETC at different molar ratios at a lower frother dosage (12g/t). Molar ratios of 95.5:4.5, 90:10, 85:15 and 80:20 were investigated.

## CHAPTER 2

### 2. Literature Review

#### 2.1 Nkomati Mine and Mineral Ore

The Nkomati nickel joint venture exploits the main mineralised zone (MMZ) and the peridotitic chromititic mineralised Zone (PCMZ) ores of the Uitkomst Complex near Machadodorp in the Waterval Boven district in South Africa's Mpumalanga Province (Figure 1). The ore is an early age Bushveld layered lenticular mafic-ultramafic intrusion into the basal sediments of the Transvaal Sequence. The deposit is approximately 9 km long and 1.5km wide (Cockburn, 2013).

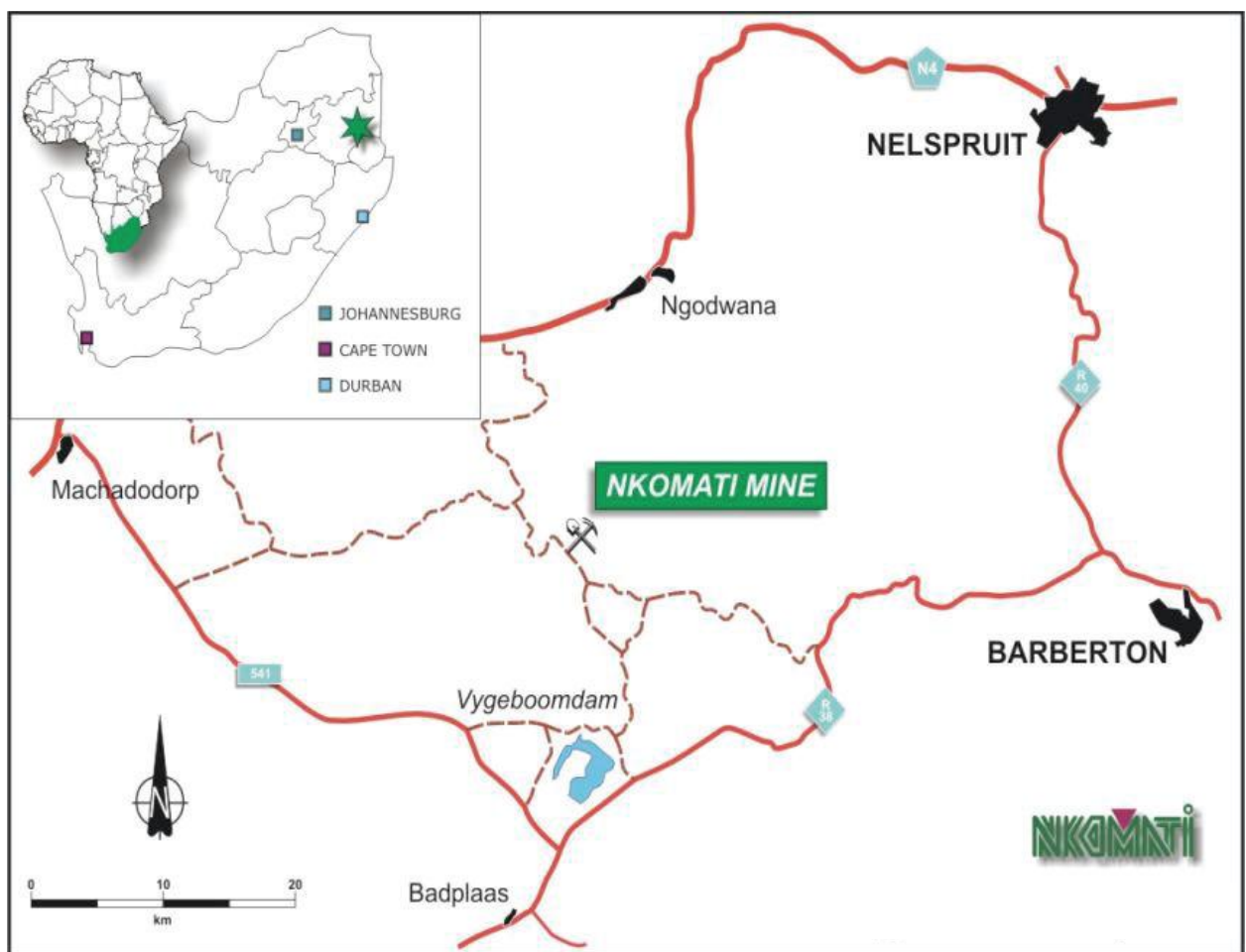


Figure 1: Location of the Nkomati mine (Cockburn, 2013)

The ore found at the Nkomati mine has multiple zones of sulphide mineralisation. The massive sulphide body mined since 1997 had a nickel grade of approximately 2%, but has since been depleted and is no longer mined. Currently the main mineralised zone (MMZ) being explored has a nickel grade of between 0.3-0.7% and an average grade of 0.37% (Cockburn, 2013).

The peridotitic chromititic mineralised zone (PCMZ) deposit currently exploited has an approximate nickel grade of between 0.2-1% and an average grade of 0.23%. It is noteworthy that the PCMZ is a chrome rich ore, nevertheless, contains a significant amount of nickel. There is a third nickel deposit, known as the basal mineralised zone, but is presently unexploited (Cockburn, 2013).

Currently only the MMZ and PCMZ deposits are being mined. There are both open pit and underground operations at the Nkomati mine. The current open pit production is approximately 650kt/month, where 300kt is produced by PCMZ and 350kt is produced from MMZ. The MMZ underground operation produces approximately 50kt/month (Cockburn, 2013).

## **2.2 Mineralogy of the Nkomati Mineral Ore**

Nickel is mainly contained within pentlandite and as much as 15% occurs in a solid solution within pyrrhotite and 1-2% within chlorite. The copper present in the Nkomati mineral ore occurs primarily within chalcopyrite with 1-2% occurring in bornite. The main mineralised zone (MMZ) is very similar to the Merensky Reef minerals. The main difference being the MMZ has lower platinum group metals (PGM's) and higher sulphide base metals (Cockburn, 2013).

The PCMZ minerals are similar to the UG2 (upper ground 2) Reef minerals with chrome grades of between 7-15% in the form of  $\text{Cr}_2\text{O}_3$ . Geologists regard the two ores as being the same, with the only difference being the chrome grades. Process engineers however regard the ores as being significantly different, based on the required liberation grinds. The target liberation grind for a MMZ ores is 67% mass passing 75 $\mu\text{m}$  and for PCMZ ores 80% mass passing 75 $\mu\text{m}$  (Brits, 2008).

It should be noted that MMZ mineral recoveries are relatively insensitive to grinds, whereas PCMZ minerals are very sensitive to grinds. With PCMZ minerals, significant losses in recovery occur with lower grind values (Brits, 2008). Below is a split core image of the Nkomati ore (Figure 2). It should be noted that sulphide mineralisation is normally concentrated in rich silicate areas.

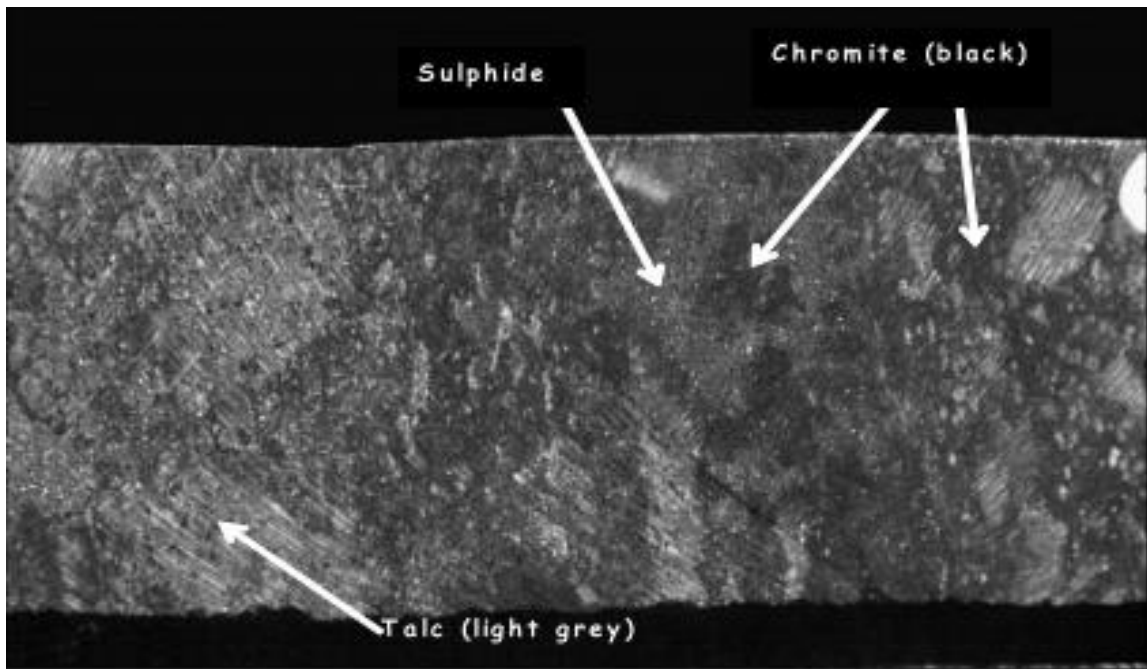


Figure 2: A split core image of the Nkomati PCMZ ore (Bowers & Smit, 2007)

The range of minerals present in the Nkomati ore are presented in Table 2. Pentlandite ores bearing both pyrrhotite and talc are normally floated in two stages as the first stage is talc rejection and the second selective separation of pentlandite from pyrrhotite (Ngobeni, 2013)



Table 1: Order of Mineral Constituency of Nkomati MMZ Feed (Nyambayo, 2014)

| Major Minerals | Composition (%) | Minor Minerals | Composition (%) | Trace Minerals  | Composition (%) |
|----------------|-----------------|----------------|-----------------|-----------------|-----------------|
| Actinolite     | 24.62           | Lizardite      | 8.97            | Pyrrhotite      | 3.42            |
| Diopside       | 20.46           | Chromite       | 8.14            | Quartz          | 3.05            |
| Talc           | 17.71           | Biotite        | 7.82            | Enstatite       | 2.76            |
|                |                 |                |                 | Chalcopyrite    | 1.29            |
|                |                 |                |                 | Forsterite iron | 0.68            |
|                |                 |                |                 | Pyrite          | 0.57            |
|                |                 |                |                 | Pentlandite     | 0.53            |

### 2.3 Froth Flotation

Froth flotation is both a physical and chemical (physico-chemical) process used to separate valuable minerals from the gangue material. Beneficiation of mineral ores using froth flotation is a complex separation process as it involves dynamic interactions among three phases of matter: solid, liquid and gas. Although it has been used for more than 100 years it is still not entirely understood (Wills and Napier-Munn, 2006). There are a number of key aspects to consider during any froth flotation operation. The most important aspects are summarised in figure 3 to enable a comprehensive and inclusive flotation design.

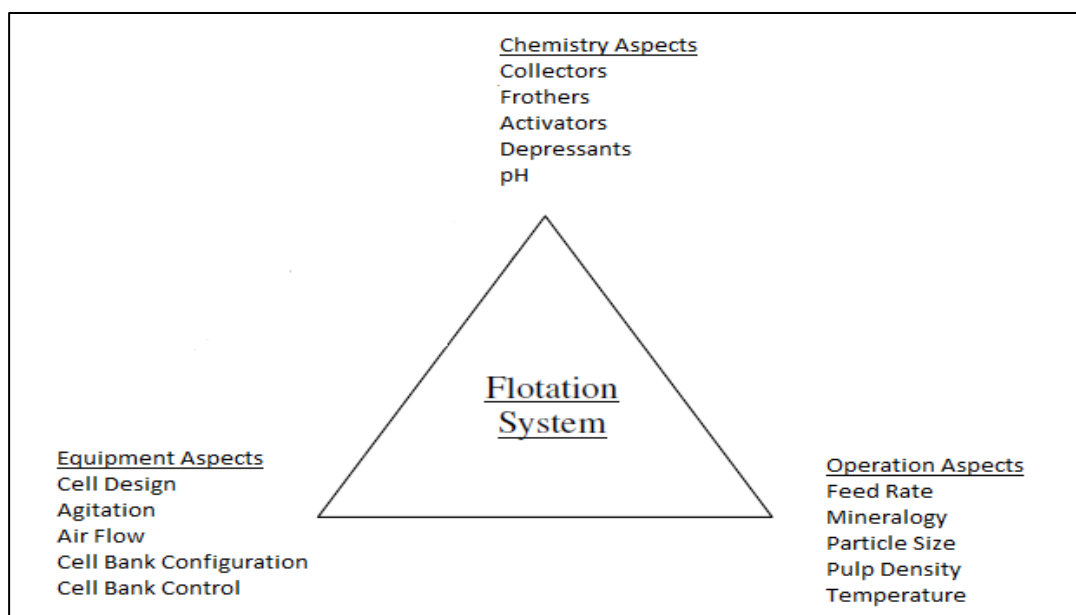


Figure 3: Key operational aspects for consideration in flotation systems (Klimpel, 1984)

After mining, the ore undergoes crushing and milling to liberate the desired minerals present. Wet milling is normally used which results in a slurry being formed and subsequently transferred to the flotation cell. When complex ore bodies are beneficiated a flotation circuit may be applied with more than one flotation stage. This is done to ensure that the separation is as selective as possible (Wills and Napier-Munn, 2006). Collectors, frothers and modifying reagents are then added to the slurry, also referred to as pulp in the flotation cells to condition the ore and impart hydrophobicity to the desired minerals. Modifying reagents used include pH regulators, activators and depressants.

Collectors selectively adsorb onto the surfaces of desired minerals through chemisorption and change the surface properties of those minerals in the slurry. The adsorbed collectors render the selected minerals hydrophobic. Frothers are reagents used to stabilise the froth phase and promote the formation of stable air bubbles in the pulp phase. A froth phase that is stable enough to allow the removal of the mineral-carrying bubbles before they collapse is desired (Bulatovic, 2007).

Modifiers either increase or prevent the adsorption of collectors on the surfaces of minerals. When they increase the rate of adsorption they are referred to as activators. If they prevent adsorption, they are referred to as depressants (Bulatovic, 2007).

Mineralised air bubbles move upwards through the pulp and contribute to the froth phase (Figure 4). The froth stability is an important factor influencing the grade and the recovery of the entire flotation process. This needs to be considered and improved to contribute towards the formation of a stable froth phase. The froth flotation process is effective when applied to relatively fine particles. When the particles are too large, the forces of adhesion between them and the air bubbles may be less than the weight of the particles. This would result in detachment from the mineral-carrying bubbles (Wills and Napier-Munn, 2006).

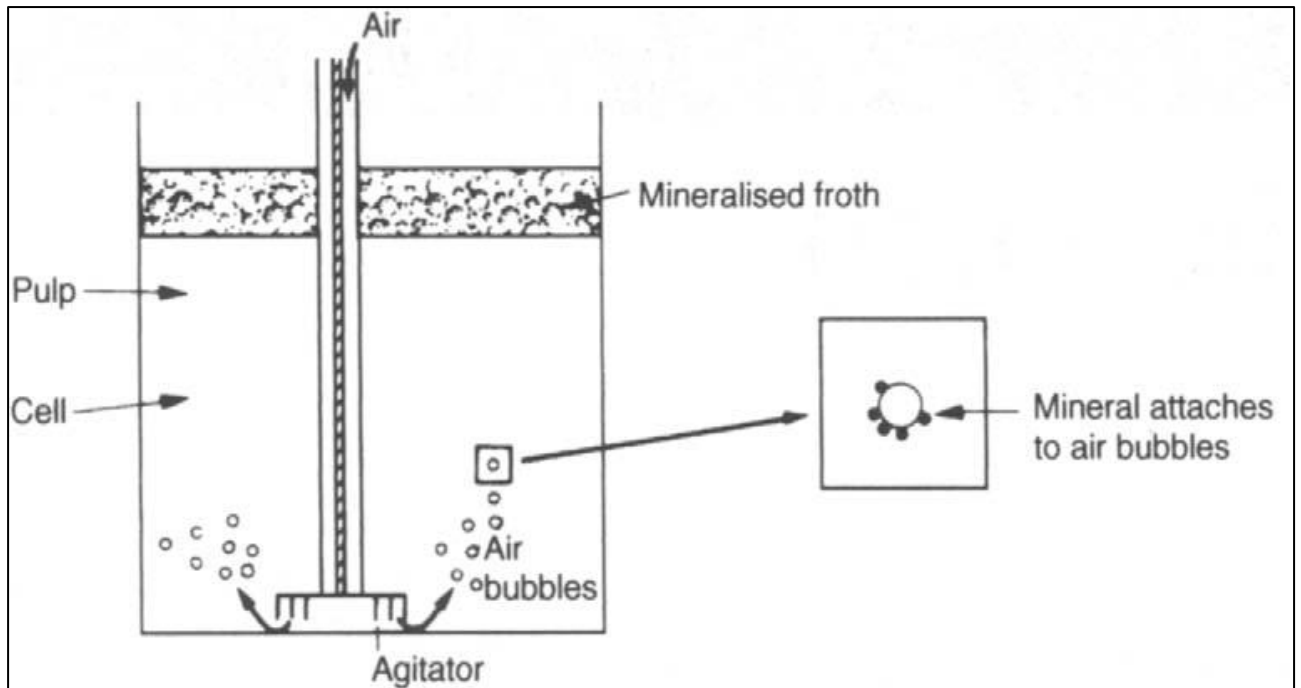


Figure 4: A batch froth flotation operation and cell (Wills and Napier-Munn, 2006)

In *direct* froth flotation, the valuable or desired minerals are concentrated in the froth phase, while the less valuable minerals remain in the pulp phase (Rao, 2004). In *reverse* froth flotation the valuable mineral remains in the pulp phase while the gangue minerals are concentrated in the froth phase.

The recovery of valuable minerals by froth flotation may involve three mechanisms:

- Selective attachment of desired minerals on passing air bubbles (true flotation).
- Entrainment to the froth layer of desired minerals in the water film around the bubble.
- Physical entrapment of the desired minerals in the froth layer.

The separation of desired minerals from gangue material is achieved by exploiting the differences in hydrophobicity of the desired and undesired minerals. In direct froth flotation, the hydrophobic desired minerals selectively attach to the hydrophobic air bubbles and are transported to the froth phase. This transport mechanism is referred to as true flotation. This is easy to control and the selective attachment of minerals on the air bubbles may be manipulated. True flotation is the dominant and most important mechanism in froth flotation (Wills and Napier-Munn, 2006).

Entrainment is the mechanism that transports the minerals to the froth layer via the water film surrounding the air bubbles. This mechanism is not affected by changes in the hydrophobic surface properties of the minerals and is therefore non-selective. This transport mechanism is

affected by the physical properties of the minerals such as the particle size and density. Therefore, increases in water recovery, generally increases the degree of entrainment (Bradshaw et al., 2005).

In physical entrapment, the desired minerals are trapped between gangue or larger hydrophobic minerals in the froth phase and during bubble transportation. Physical entrapment, much like entrainment, is not selective since it depends on the physical properties of the minerals, such as particle size (Wills and Napier-Munn, 2006).

### 2.3.1 Grade vs Recovery

Grade vs recovery plots are the primary means of comparison to evaluate the performance of collector suites with particular ores. Generally in froth flotation as the recoveries increase the grades decline (Figure 5). It is therefore important to find a suitable collector mixture, where high grades are achieved without significantly decreasing the recoveries.

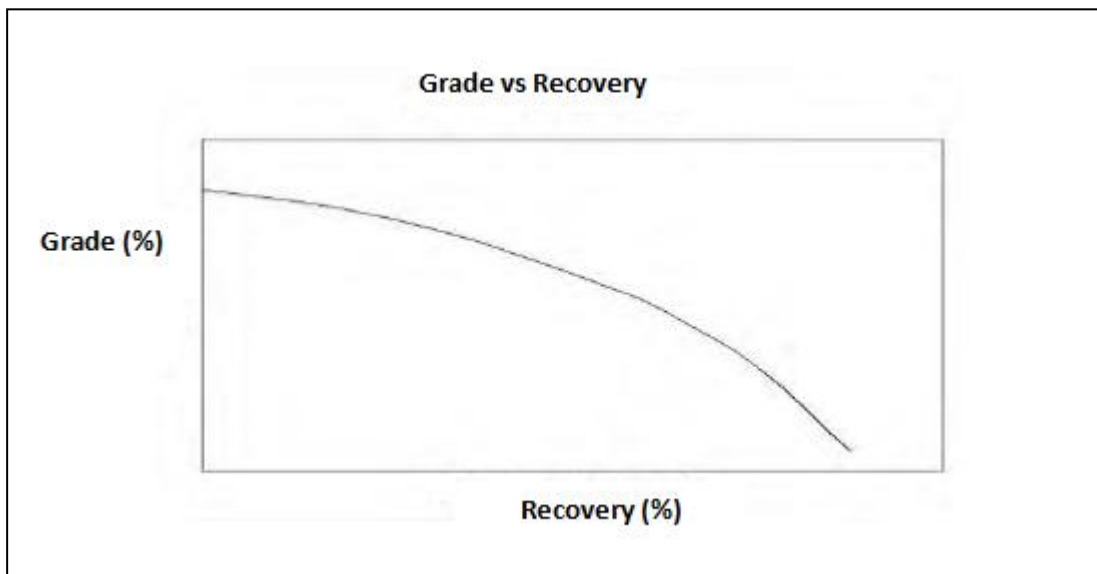


Figure 5: The relationship between grade and recovery in froth flotation.

Other variables affecting the grade and recovery are particle size, pulp density, agitation rate, air flow rate, collector dosage and frother dosage. For this study, the frother dosage, agitation rate and air flow rate were held constant and will not be discussed further.

Higher recoveries are normally achieved with finely ground particles, whereas high concentrate grades are normally achieved with coarser particle sizes (Al-Thyabat, 2009). The rates of

recoveries achieved initially with coarse particles are lower than that of finely ground particles (Bradshaw and O'Conner., 1994). This may decrease the final cumulative grades as more gangue will report to the concentrates towards the latter stages of flotation. With coarse particles, the desired minerals may not be sufficiently liberated and therefore not exposed to the collectors for adsorption. This results in lower grades and recoveries.

The density of the pulp is another important variable to consider in froth flotation. Favourable grades and recoveries may be achieved at a number of pulp densities (Wills & Napier-Munn, 2006). High pulp densities maintain particles in suspension and are generally used when floating coarse particles. An increase in the pulp density normally allows for an increase in recovery, but at the expense of grade. Low pulp densities usually result in low recoveries but high concentrate grades (Kirjavainen, 1996).

Collector concentrations and dosages play an important role in influencing the grades and recoveries achieved. It is widely accepted that with an increase in collector dosage and subsequent concentration in the pulp, an increase in recovery is observed. However collectors used at low dosages may cause a decrease in recovery due to insufficient amounts of collector available for adsorption, but will generally yield a higher grade. This is because the dominant mechanism is assumed to be selective bubble-mineral attachment (Rao and Forsberg, 1997).

An increase in frother dosage normally results in an increase in froth stability. This generally results in an increase in recovery, but at the expense of grade. Excessive frother dosages (surplus of frother) results in an over-stable froth layer. This will result in low concentrate grades due to poor detachment of gangue from the froth bubbles (Goodall & O'Connor, 1991).

### **2.3.2 Classification of Minerals**

Based on the surface properties and characteristics of the minerals, they may be classified as either polar or non-polar. (Wills and Napier-Munn, 2006). Normally the surfaces of the minerals classified as non-polar have relatively weak molecular bonds, composed of covalent molecules bonded together by van der Waals forces. Due to this characteristic, non-polar minerals do not readily attach to the water dipoles and are therefore considered hydrophobic.

Minerals considered polar, have strong covalent or ionic surface bonding. They have a higher free energy for bonding and react strongly with water molecules in solution. These characteristics render minerals hydrophilic (Wills and Napier-Munn, 2006). Some of the common polar minerals are classified below:

| <i>Group 1</i>        | <i>Group 2</i> | <i>Group 3(a)</i> | <i>Group 4</i> | <i>Group 5</i> |
|-----------------------|----------------|-------------------|----------------|----------------|
| Galena                | Barite         | Cerrusite         | Hematite       | Zircon         |
| Covellite             | Anhydrite      | Malachite         | Magnetite      | Willemite      |
| Bornite               | Gypsum         | Azurite           | Gothite        | Hemimorphite   |
| Chalcocite            | Anglesite      | Wulfenite         | Chromite       | Beryl          |
| Chalcopyrite          |                |                   | Ilmenite       | Feldspar       |
| Stibnite              |                | <i>Group 3(b)</i> | Corundum       | Sillimanite    |
| Argentite             |                | Fluorite          | Pyrolusite     | Garnet         |
| Bismuthinite          |                | Calcite           | Limonite       | Quartz         |
| Millerite             |                | Witherite         | Borax          |                |
| Cobaltite             |                | Magnesite         | Wolframite     |                |
| Arsenopyrite          |                | Dolomite          | Columbite      |                |
| Pyrite                |                | Apatite           | Tantalite      |                |
| Sphalerite            |                | Scheelite         | Rutile         |                |
| Orpiment              |                | Smithsonite       | Cassiterite    |                |
| Pentlandite           |                | Rhodochrosite     |                |                |
| Realgar               |                | Siderite          |                |                |
| Native Au, Pt, Ag, Cu |                | Monazite          |                |                |

Table 2: Classification of common polar minerals (Wills and Napier-Munn, 2006)

Minerals may be arranged in five groups which are ordered according to the degree of polarity (Table 2). Group 1, predominantly sulphide minerals, contains the minerals that have the lowest polarity and therefore a relatively high natural hydrophobicity. Group 5 contains the minerals with the highest polarity and therefore low natural hydrophobicity. The polarity increases from group 1 to group 5. (Wills and Napier-Munn, 2006).

Pentlandite is a group 1 mineral and is therefore considered to have a relatively high natural hydrophobicity (Table 2). The Nkomati ore is a pentlandite rich ore and also contains pyrite and chalcopyrite; also group 1 minerals. These are contributing factors in the classification of the Nkomati ore as a complex mineral ore.

### 2.3.3 Bubble-mineral attachment

Bubble-mineral interactions and subsequent attachments are very important in any flotation system. In order for favourable bubble-mineral attachment to occur, the desired mineral surfaces should be hydrophobic. Although froth flotation is a surface chemistry based process, there are many simultaneously occurring phenomena at the solid-liquid and solid-bubble interfaces (Othmer, 2007).

The contact angle,  $\theta$  between an air bubble and mineral surface is one of the measurements used to define the strength of bubble-mineral attachments. The higher the contact angle ( $\theta$ ), the stronger the bubble-mineral attachment (Figure 6). If the air bubble displaces all of the water on the solid surface, then the contact angle is  $180^\circ$ . If the air bubble fails to displace any of the water on the solid surface of the mineral, then the contact angle will be  $0^\circ$ . A contact angle of  $90^\circ$  is generally considered to be sufficient for froth flotation. However,  $70^\circ$  has been proven to be the optimum contact angle (Rao, 2004).

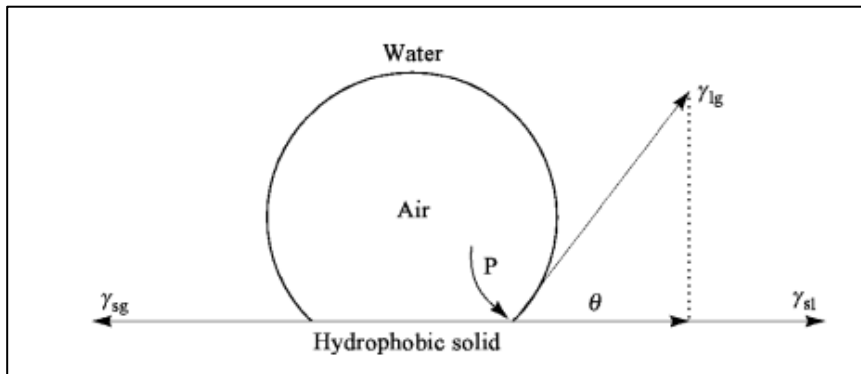


Figure 6: Solid – liquid – air interface, with contact angle (Othmer, 2007)

The contact angle depends largely on the interfacial force between solid, liquid and gas (air bubbles). Young’s equation (Equation 1) may be used to relate the thermodynamic relationship between these forces and the contact angle (Othmer, 2007):

$$\gamma_{s/a} = \gamma_{w/a} \cos \theta + \gamma_{s/w} \dots\dots\dots \text{Equation 1}$$

Where,

$\gamma_{s/a}$  = Interfacial force between solid and air

$\gamma_{w/a}$  = Interfacial force between water and air

$\gamma_{s/w}$  = Interfacial force between solid and water

$\theta$  = Contact angle between the solid and the air bubble

When the contact angle for a specific mineral is between 60° and 90°, then there is a strong chance that the mineral will report to the froth phase. However, generally it is agreed that the contact angle does not provide comprehensive information on the floatability of a specific mineral due to the effect of other factors such as particle size and hydrodynamics (Fuerstenau et al., 2007).

### 2.3.4 Kinetics of froth flotation

The kinetics involved in froth flotation is important; since it serves as an indication of how “fast” specific minerals float. By determining the flotation kinetics of a particular mineral, one could obtain a rate constant. This information could be used, to improve the rate of flotation, in the case of slow

floating minerals (small rate constant). It is widely accepted that the flotation of minerals follow first order kinetics (Sutherland, 1955).

The rate of flotation is a measure of the efficiency of the froth flotation. It is expressed as the amount of floated particles per unit time. It is considered to be the most reliable source of describing the flotation kinetics (Bulatovic, 2007). The Klimpel rate equation (Equation 2) may be used to determine the rate constant:

$$r = R_{\infty} \left[ 1 - \left( \frac{1}{kt} \right) (1 - \exp^{-kt}) \right] \dots\dots\dots \text{Equation 2}$$

Where,

$R_{\infty}$  = Maximum recovery at an infinite amount time (%)

$r$  = Recovery at a specific time  $t$  (%)

$t$  = Specific time (minutes)

$k$  = Klimpel rate constant ( $\text{min}^{-1}$ )

To confirm the order of a flotation reaction, a plot of the experimental data recorded (cumulative mineral recovery % vs flotation time) may also be used. The shape of the graph should illustrate a linear relationship for first order reactions (Nyambayo, 2014).

## 2.4 Chemical Reagents

As previously mentioned, the chemical reagents involved in froth flotation are collectors, frothers and modifiers. Each chemical reagent performs a different function in improving the selectivity of the flotation. The chemical reagents used in this study will now be discussed.

### 2.4.1 Collectors

Various collectors are used depending on the surface properties of the minerals present in the ore. The main objective of a collector is primarily to induce hydrophobicity of the desired minerals. Hydrophobicity and floatability assist in forcing a selected mineral to float to the top of the flotation cell with the aid of air bubbles. Hydrophobicity is the thermodynamic characteristic while floatability is the kinetic characteristic influencing the particles motion (Wills and Napier-Munn, 2006).



Collectors may be ionic or non-ionic compounds. Ionic collectors are more widely used in froth flotation and are generally heteropolar molecules. Due to the presence of chemical, electrical and physical attractions between the surfaces of minerals and polar groups of the collectors, they adsorb onto the desired minerals' surfaces. The non-polar (hydrophobic) groups of the collectors are exposed to the pulp, thus rendering the minerals hydrophobic (Bulatovic, 2007). The addition and subsequent adsorption of collectors onto the surfaces of minerals is called the conditioning period (Laskowski, 2005; Woods, 1997) (Figure 7).

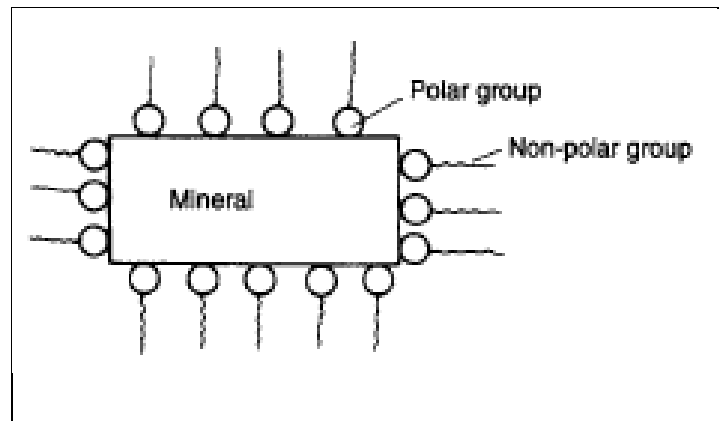


Figure 7: The hydrophobic collector coating mechanism (Wills and Napier-Munn, 2006)

The use of collectors with longer hydrocarbon chains increases the froth flotation time span, without decreasing the selectivity in comparison to collectors with shorter hydrocarbon chains (Wills and Napier-Munn, 2006). Due to the large variety of minerals in complex ores, collectors are subdivided based on their individual and collective properties. For this study only ionic collectors will be discussed.

#### 2.4.2 Ionic collector group

Ionising or ionic collectors used in froth flotation are complex molecules and are heteropolar (i.e. the molecules contain a non-polar hydrocarbon group and a polar group). The non-polar group is usually a hydrocarbon chain with water-repellent properties which induces the hydrophobicity on the minerals' surfaces. The polar group is usually charged and is able to react and bond with minerals' surfaces. The normal selection of collectors used in froth flotation could be determined as shown in Figure 8.

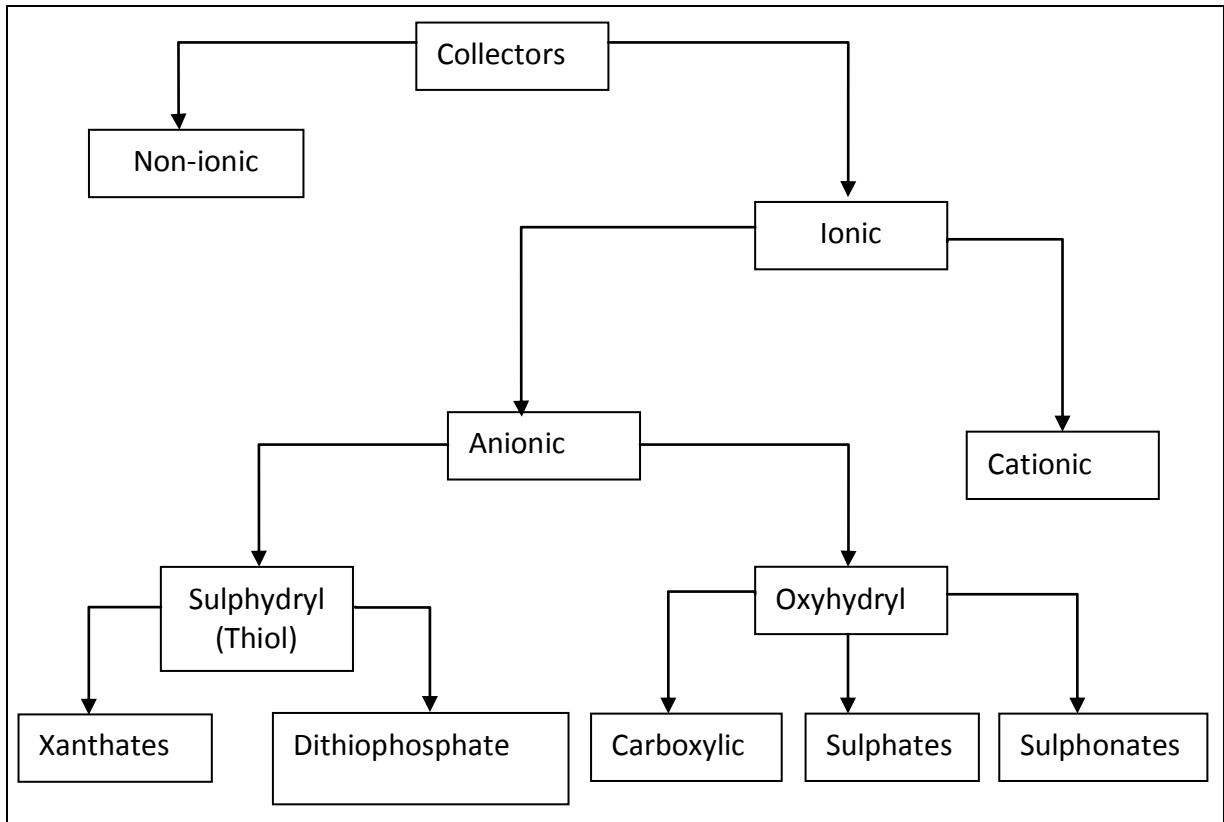


Figure 8: Path of collector selection (Bulatovic, 2007)

It is generally more difficult to remove a collector once adsorbed on a mineral surface than to prevent its initial adsorption. The excessive use of collectors at high dosages and concentrations may adversely affect the grade and recovery of desired minerals. This is due to a multi-layered accumulation of collector on the minerals' surfaces leading to their reduced hydrophobicity. This leads to less non-polar hydrocarbon chains exposed to the slurry, thus decreasing the floatability of desired minerals. To prevent these phenomena, low dosages of collectors are used to promote the formation of monomolecular layers on minerals' surfaces (Wills and Napier-Munn, 2006).

The recovery limit (time limit) of the froth flotation may be extended by manipulation without compromising the selectivity. This may be done by using collectors with long hydrocarbon chains. Although longer hydrocarbon chained collectors are used, the normal length of the hydrocarbon chain in a collector is accepted to be two to five carbon atoms long. The length of the hydrocarbon chain and its structure affects its solubility. Branched chains have higher solubility than straight chains (Smith, 1989). Since sulphydryl collectors are most relevant to sulphide ore beneficiation, only these will be discussed herein.

### 2.4.3 Anionic collectors

Anionic collectors are the most widely used in froth flotation. The sulphhydryl or thiol sub-division of anionic collectors are used in the froth flotation of sulphide minerals. The functional groups of the sulphhydryl collectors contain a bivalent sulphur atom. Sulphhydryl collectors are strong and have a high selectivity to specific minerals involved in the froth flotation of sulphide ores (Bulatovic, 2007). The most commonly used sulphhydryl collectors are the xanthates, dithiophosphates, dithiocarbamates and thionocarbamates. This study will however only focus on the xanthate and thionocarbamate class of collectors.

### 2.4.4 Xanthate collectors

The xanthates are the most important collectors used in the froth flotation of sulphide ores. The xanthate collectors, normally have sodium or potassium as a cation as part of their heterpolar structure (Figure 9). These cations do not take part in the surface reaction (Wills and Napier-Munn, 2006). The reactivity and selectivity of the sodium-containing alkyl xanthates decreases with time due to the absorption of water from the atmosphere. In contrast, the performance of the potassium-containing alkyl xanthates do not suffer as a result of the absorption of water.

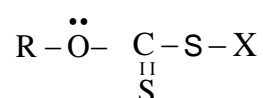


Figure 9: The general structure of a xanthate molecule, where R represents a hydrocarbon bond and X represents a cation bond with either sodium or potassium

### 2.4.5 Mechanism of adsorption of xanthates

The type of adsorption mechanisms proposed for xanthate collectors depends on the mineral involved. Some minerals float due to the formation of dixanthogens (Figure 10), while others float due to the formation of metal-thiolates (Figure 11) on their surfaces.

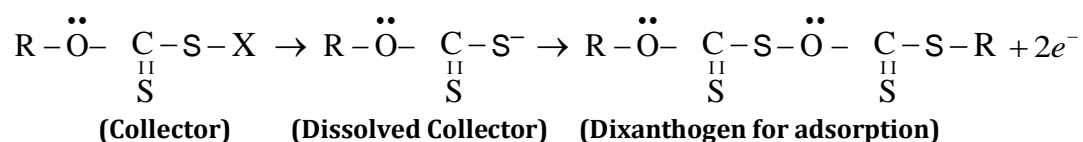


Figure 10: Reaction 1- A reaction illustrating dixanthogen formation, where R represents a hydrocarbon bond and X represents a non-active cation

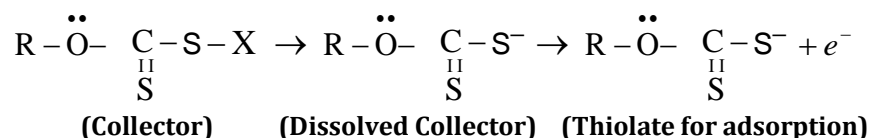


Figure 11: Reaction 2- A reaction illustrating the thiolate formation, where R represents a hydrocarbon bond and X represents a non-active cation

Generally, in acidic pulps xanthates tend to form dixanthogens, whereas in alkaline pulps xanthates tend to form metal-thiolates (Crozier, 1991). Chalcopyrite, pyrite and pyrrhotite are floated due the formation of dixanthogens on their surfaces, while pentlandite floats due to the formation of a metal-xanthate complex on its surface (Lotter and Bradshaw, 2010).

The adsorption of xanthate on mineral surfaces takes place via two steps. The first step is catalytic oxidation, where the polar group of the xanthate collectors donate their electrons to the minerals and adsorbs onto their surfaces. When the xanthate collector donates its electrons to the minerals surfaces, the transfer is referred to as electron deposition. During electron deposition, the electrons move to the oxygen-rich sites on the mineral surfaces and subsequently react with the oxygen (Crozier, 1991).

The second step in the adsorption of xanthate is the chemical reaction of the collector on the mineral surfaces (chemisorption). With a sulphide ore, the chemical bonds between the collector and minerals take place via the sharing of electrons between the sulphur atoms of the collector and mineral surface (Crozier, 1991).

#### 2.4.6 Thionocarbamate collectors

The thionocarbamates are relatively new sulphide collectors and are often used in the froth flotation of copper and zinc sulphide minerals that are activated by copper (Huang, 2010). More recently they have been used in the froth flotation of other sulphide minerals where an improved selectivity was observed particularly between copper sulphide minerals and galena as well as sulphide gangue minerals such as pyrite (Sheridan et al., 2002).

Previous work done, indicated that thionocarbamates have frothing properties, caused by the amino group (NH) (Figure 12). The extent of frothing properties also depends on the alkyl chain lengths (Sheridan et al., 2002) . Thionocarbamates are usually more expensive than xanthates, dithiocarbamates and dithiophosphates. As a result, they are not as popular or as widely used as the other thiol collectors

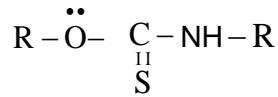


Figure 12: The general structure of a thionocarbamate molecule, where R represents hydrocarbon alkyl groups

#### 2.4.7 Mechanism of adsorption of thionocarbamate

Xanthate collectors are known to be more powerful (non-selective) collectors than thionocarbamates. However, thionocarbamates have distinct advantages over xanthates as they are relatively stable compounds in solution and require a lower pH for adequate flotation (Fairthorne et al, 1996). Due to the modest amount of research done on the thionocarbamates, it was difficult to identify a mechanism on pentlandite.

The collecting power of thionocarbamates may be proportional to the electron density at the reactive centre of the molecule (Glembotskii, 1977). It was also noted, by further investigation, that electron donating groups improved the performance of the collector by increasing the electron density, whereas an electron accepting substituent had the opposite effect.

Bogdanov et al. (1980) explained how thionocarbamates formed complexes with metal cations using the molecular orbital theory. The theory suggests that the d-orbitals of the metal cations overlap with the empty d-orbitals of the sulphur atoms present in the thionocarbamate collectors. These overlapping d-orbitals form a dative  $\pi$ -bond. The theory also predicts that electron donating substituents will decrease the mineral-collector bond strength due to reduced d-orbital overlap between the metal cations and the sulphur atoms of the thionocarbamates (Fairthorne et al., 1996).

Ackerman et al (1984) investigated the effects of different alkyl substituents on the performance of thionocarbamates. It was concluded that the collecting power increased with the number of carbon atoms in the alkyl group attached to the thionocarbamate. It was also suggested that the nature of the O-alkyl and N-alkyl substituents present in dialkyl thionocarbarnates, affected the hydrophobicity of the surface.

Work by Liu et al. (2008) showed that interactions between the copper cations on sulphide mineral surfaces and thionocarbamates formed six membered chelate rings. It was concluded that this interaction and formation of the chelate ring was responsible for selectivity against iron sulphides. While work by Sheridan et al. (2002) indicated that an increase in collector chain length increased the recovery at the expense of the grade, thionocarbamates were found to perform differently with increases in both grade and recovery. Despite the research done on thionocarbamates and on O-

isopropyl-N-ethyl thionocarbamate specifically, the absorption mechanism remains unresolved (Buckley et al., 2014).

## 2.5 Collectors

### 2.5.1 Potassium Amyl Xanthates (PAX)

Potassium amyl xanthate is a yellow powder mostly found in the form of a pellet with a distinctive smell. It has a molecular mass of 199 g/mol, purity of >90% and has a density of 0.50 kg/l. Potassium amyl xanthate is soluble in water and easily decomposes in solution. It is one of the most powerful collectors used. PAX is the collector of choice when floating tarnished or oxidized sulphide minerals (Senmin handbook, 2006). It has the following molecular structure (figure 13):

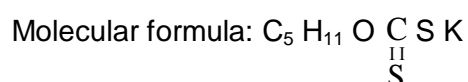
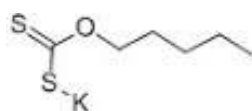


Figure 13: The structure and molecular formula of PAX

### 2.5.2 Sodium Isobutyl Xanthate (SIBX)

Sodium isobutyl xanthate like PAX is a yellow powder mostly found in the form of a pellet with a distinctive smell. It is also readily soluble in water and has a molecular mass of 172 g/mol, with a purity of >90% and a density of 0.65 kg/l. SIBX is used in the flotation of minerals where collecting efficiency and selectivity are important. SIBX is also the preferred collector when floating platinum group metals or slow floating copper and nickel sulphides (Senmin handbook, 2006). SIBX has the following molecular structure (Figure 14):

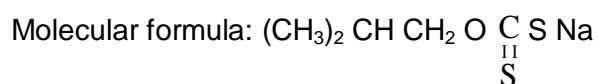
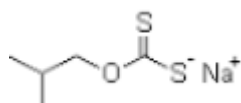


Figure 14: The structure and molecular formula of SIBX

### 2.5.3 IPETC (O-Isopropyl-N-Ethyl Thionocarbamate)

O-isopropyl-N-ethyl thionocarbamate is a reagent that forms part of the thionocarbamate collector series and is sold as IPETC by Senmin. This collector is a yellow oily liquid at room temperature. It

has a density of 0.95 Kg/l at 25°C, a purity of >90% and a molecular mass of 147 g/mol. This collector is normally used to selectively separate copper sulphides from sphalerite, pyrite and galena (Senmin handbook, 2006). IPETC has the following molecular structure (Figure 15):

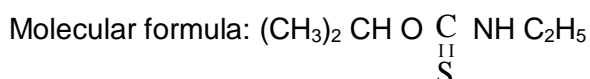
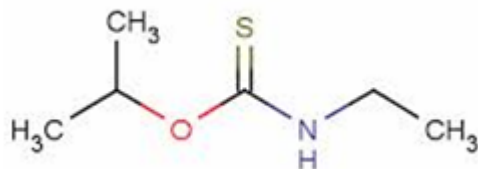


Figure 15: The structure and molecular formula of IPETC

Below is a summary of the main uses of different thiol collectors at specific pH ranges:

Table 3: A summary of the uses of different thiol collectors (Wills and Napier-Munn, 2006)

| Reagent  | pH range | Main uses  | References                     |
|--|----------|--|--------------------------------|
| O-alkyl dithiocarbonates<br>(Xanthates)            | 8-13     | Flotation of sulphides, oxidised minerals such as malachite, cerussite, and elemental metals | Leja (1982); Rao (1971)        |
| Dialkyl dithiophosphates<br>(Aerofloats)           | 4-12     | Selective flotation of copper and zinc sulphides from galena                                 | Mingione (1984)                |
| Dialkyl dithiocarbamate                            | 5-12     | Similar uses to xanthates, but more expensive  | Jiwu et al. (1984)             |
| Isopropyl thionocarbamate<br>(Minerec 166 I/Z-200) | 4-9      | Similar uses to xanthates, but more expensive  | Ackerman et al. (1984)         |
| Mercaptobenzothiazole<br>(R404/425)                | 4-9      | Flotation of tarnished or oxidised lead and copper minerals. Floats pyrite at pH 4-5         | Fuerstenau and Raghavan (1986) |

## 2.6 Mixtures of collectors

Collectors are mixed in order to improve the grades and recoveries of desired minerals (Ngobeni, 2013; Nyambayo, 2014; Ramonotsi, 2011; Bradshaw et al., 1998). The improved grades and recoveries are achieved by higher selective adsorption and reaction rates on the desired minerals' surfaces. Mixtures of collectors could also reduce the total collector dosage requirements (Bradshaw et al., 1998).

A mixture of sodium di-methyl-dithiocarbamate (di-C1-DTC) with sodium ethyl xanthate (SEX) at a 30:70 mole ratio gave an improvement over pure collector sodium ethyl xanthate in nickel recoveries (22% improvement) with a pentlandite ore. Also nickel grades and recoveries increased (5% nickel grade and 3% nickel recovery) relative to pure collector potassium amyl xanthate when a mixture of sodium di-methyl-dithiocarbamate (di-C1-DTC) with potassium amyl xanthate (PAX) at a mole ratio of 10:90 for the same ore (Ngobeni and Hangone, 2013).

Similarly Nyambayo (2014) showed that mixtures of collectors have some advantages over single collectors in the froth flotation of an Nkomati sulphide ore. However, the components of the mixtures should be well-matched. When the components are well matched at the optimum mole ratios, higher mineral recoveries are observed in contrast to that of individual constituents. This occurrence is referred to as synergism, where the combined effects of the collector mixtures exceed the sum of the individual collector effects (Bradshaw et al., 1998).

Nyambayo (2014) also found that a mixture consisting of 30 mol % dithiophosphate (DTP) and 70 mol % of a mixture of sodium isobutyl xanthate (SIBX) and sodium ethyl xanthate (SEX) improved the nickel grades and recoveries by up to 1% and 20% respectively compared to DTP. It was also shown that a mixture of dithiocarbamate (DTC) at 10% and a mixture of sodium isobutyl xanthate (SIBX) and sodium ethyl xanthate (SEX) at 90% improved the nickel grades and recoveries by up to approximately 4% and 7.5% respectively compared to DTC.

Ramonotsi (2011) found that when using a mixture of 60 mol % thionocarbamate and 30 mol % xanthogen formate with a makeup of 10 mol % isobutanol in the froth flotation of a platinum ore, an increase in selectivity of copper sulphide minerals over pyrite was observed. Also a marginal increase in recovery relative to pure thionocarbamate containing 90 mol % thionocarbamate with a 10 mol % isobutanol makeup was observed. When using a thionocarbamate mixture with a xanthate, no improvement in the recovery of copper sulphide minerals nor selectivity of the copper sulphide minerals over pyrite was observed.



Collector mixtures and more specifically thiol collector mixtures are used in the froth flotation of base metals and platinum bearing ores, instead of single collectors due to their synergistic effects (McFadzean et al., 2013). In industry, froth flotation operations using various collector mixtures are common practice and often result in greater effectiveness than singular collectors (Bradshaw et al., 1998).

The synergism of collector mixtures not only influences the recovery, but may also influence the selectivity of specific minerals in differential froth flotation (Bradshaw et al., 1998). Some of the claimed synergistic effects for collector mixtures include lower collector dosage requirements, improved coarse particle recovery, increased flotation rate and in some cases co-collectors act as frothers, thus decreasing the required frother dosages (McFadzean et al., 2013).

Mixtures of collectors have been widely used, since it has been shown to enhance flotation performance. Some of these enhancements include lower collector dosage requirements, improved mineral selectivity and improved recoveries. When mixtures of collectors (collector suites) are used, a greater extent of adsorption has been observed on the mineral surfaces. The increase in adsorption could either enhance the overall hydrophobicity of the desired minerals or it could cause an adsorbed collector layer on the surfaces of minerals which will be more suitable for frother-collector interactions (Bradshaw et al., 1998).

Table 4 illustrates some froth flotation experiments conducted where mixtures of collector were used:

Table 4: A summary of the experimental observations made when using mixtures of collectors (Castelyn, 2011)

| <b>Reagents<br/>(mole ratio tested)</b>            | <b>Mineral(s)</b>    | <b>Observation</b>   | <b>Reference</b>            |
|--|----------------------|--|-----------------------------|
| Butyl X: Butyl DTP<br>(50:50)                      | Galena               | Preferential DTP adsorption from mixture with no increased mass picked up by bubble. | Wakamatsu and Numata (1979) |
| Isopropyl DTC:<br>Isopropyl xanthate (1:2<br>mass) | Chalcopyrite ore     | Better results with DTC:X mixture than with pure DTC                                 | Falvey (1990)               |
| Di-isobutyl DTP: iso                               | Platinum group metal | Recovery improved by   | Mingione (1984)             |

|  |                             |  |  |
|--|-----------------------------|--|--|
| butyl<br>(30:70, 50:50, 70:30<br>mass)                                       | (PGM) ore                   | 11% when pure X was<br>replaced with a 70:30<br>mixture  |  |
| n-butyl X: cyclohexal<br>DTC (90:10)   | Pyrite                      | Increased bubble<br>loading  | Bradshaw (1997)                            |
| Isopropyl X: dicresyl<br>DTP (95:5)<br>SEX:SEDTP                             | Mixed copper sulfide<br>ore | Enhanced copper<br>recovery with mixture,<br>from 80% to 83% and<br>improved rate of<br>recovery   | Adkins and Pearse<br>(1992)                |
| SEX:SEDTP<br>(10:90; 50:50; 90:10; )<br>SEX:SEDTC<br>(10:90; 50:50; 90:10; ) | Galena and pyrite           | Recovery improved for<br>the following mixtures;<br>10:90 SEX:SEDTP<br>(17% increase in<br>recovery)<br>90:10 SEX:SEDTC<br>(29.5% increase in<br>recovery) | McFadzean, Castelyn<br>and O'Connor (2012) |

### 2.6.1 Adsorption mechanism of collector mixtures

The proposed mechanism for systems where collectors are mixed by introducing them into the pulp sequentially have stated that the weaker collector will tend to adsorb onto the strong sites on a mineral surface and the stronger collector will adsorb onto the weaker sites on a mineral surface. The strong sites on mineral surfaces with low oxidation spots, while the weak sites are those mineral surfaces with higher oxidation spots (Bradshaw and O'Connor, 1994). The increase in the hydrophobicity of the mineral is as a result of more available adsorption sites on the mineral surfaces. If the collector suite was pre-mixed and introduced into the pulp, an increase in hydrophobicity might not be observed in comparison to the sequential introduction. It is thus important to note that the synergism of a particular collector mixture depends on the sequence of addition as well as the mixture itself (Bradshaw et al., 1998).

The proposed mechanisms for systems where collectors are pre-mixed and introduced into the pulp assume that when a thiol collector mixture is used on a sulphide mineral ore, the chemical adsorption is the dominant mechanism (Buckely et al., 2003). It is assumed that the xanthate collector adsorbs onto the desired mineral surfaces through charge transfers between the xanthate

and the mineral surface. The dithiophosphate, dithiocarbamate or thionocarbamate will adsorb by forming metal-thiolates on the mineral surfaces (Bradshaw, 1997; Makanza et al., 2008).

In the co-adsorption mechanism, one collector in the collector mixture adsorbs onto the desired minerals surfaces and in turn provides easy adsorption sites for the second collector (Bradshaw and O'Connor, 1994). Much like the sequential mixture mechanism, the stronger collector in the collector mixture will adsorb onto the weaker sites on the minerals' surfaces and the weaker collector in the collector mixture will adsorb onto the stronger sites (Bradshaw & O'Connor, 1994). According to the co-adsorption mechanism high collector dosages would result in unfavourable co-adsorption, due to the formation of multi-layers of collectors on the surfaces of minerals.

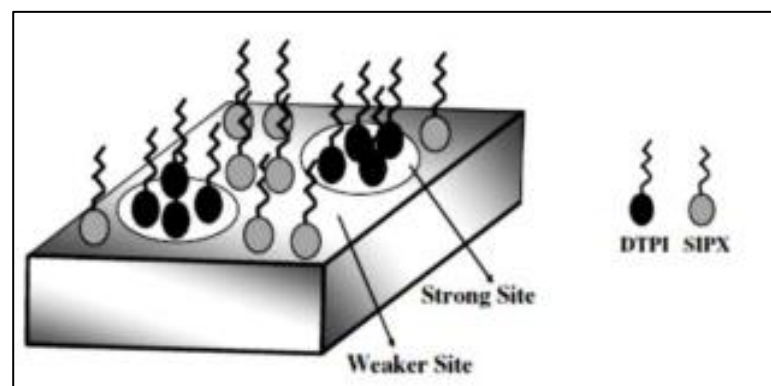


Figure 16: The collector and mineral site interaction (Bacgi et al., 2007)

In figure 16, the DTPI (Di-isobutyl dithiophosphinate) represents the weak collector (more selective collector) and SIPX (Isopropyl xanthate) represents the strong collector (less selective) collector (Bacgi et al., 2007). It is furthermore illustrated that the “strong” sites attract the “weak” collectors and the “weak” sites attract the “strong” collectors.

Ngobeni (2013) also summarised four proposed mechanisms by which collector adsorption takes place when collector mixtures are used. It was indicated that collector adsorption depends largely on the type of collectors used; the minerals present; the pre-treatment of the ore or the subsequent combination of these variables. The summarised mechanisms are as follows:

**Mechanism 1:** The formation of a metal-thiol agglomerate on the desired mineral surface (e.g. a xanthate on a pentlandite surface).

**Mechanism 2:** A metathetical substitution on the mineral surface. This is the displacement of the oxidation product (Usually a metal oxide) on the mineral surface by the collector (e.g. a xanthate on an oxidized pyrrhotite surface).

**Mechanism 3:** The occurrence of catalytic oxidation. This is the physical adsorption of a neutral dithiolate compound on the mineral surface (e.g. a DTP on chalcopyrite surface.).

## 2.7 Frothers

Frothers are chemical reagents added to the slurry in order to stabilise the froth layer and transport air bubbles in the pulp. A stable froth layer will ensure sufficient water drainage and inhibit entrainment. It also prevents mineral-carrying bubbles from collapsing, thus increasing the recovery of desired minerals (Bulatovic, 2007). Frothers are very similar to ionic collectors in structure. They are also heteropolar surface active compounds, containing a polar group and a hydrocarbon radical group, capable of adsorbing in the water-air interface. Therefore, many well-known collectors are also powerful frothers (e.g. oleates).

Some collectors are however too powerful to be used as frothers, as they produce froths that are too stable and do not promote efficient transportation, nor bubble breakage which is critical for further processing. Good frothers should have low collecting ability and should produce froths that are not too stable, but that are stable enough to transfer the desired minerals from the froth layer. Once the froth is removed from the froth layer, the bubbles should be weak enough to break and therefore release the desired minerals (Bulatovic, 2007).

Froth forms as a result of air bubbles introduced into the pulp while being agitated. The frother adsorbs onto the bubble-water interfaces and reduces the surface tension of the water, thus stabilising the air bubbles formed. A good frother should be soluble in water and distribute evenly in aqueous solutions. Most frothers have a heteropolar molecular structure which promotes adsorption as the non-polar group positions towards the air and the polar group towards the water (Figure 17).

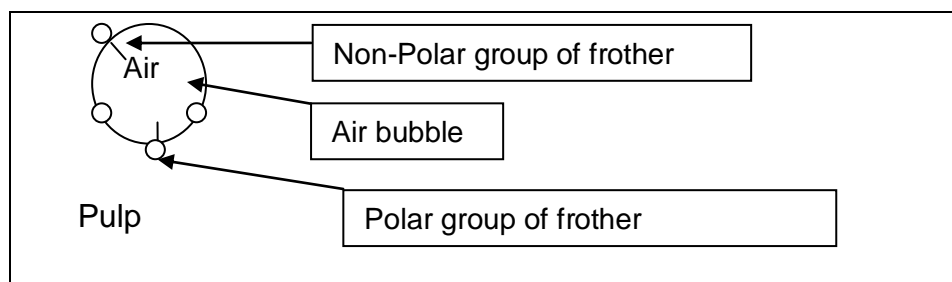


Figure 17: The adsorption of frothers.

The frother to be used for this study is a polyether also referred to as a polyester alcohol. Its commercial name is Senfroth XP 200 and is supplied by Senmin. It is a frother derived from the polyether polyol chemical family and is a widely used frother in the froth flotation of sulphide minerals.

Alcohols are ideal for frothing as they are highly soluble in water and they have limited collecting ability. Various alcohols of high molecular mass are used and have some advantages over earlier frothers such as pine oil and cresol; the greatest being that their composition is more stable (Bulatovic, 2007).

When a very high dosage of frother is used, frother molecules may accumulate on the surface of the desired minerals and thus decrease their hydrophobicity as the alkyl chains of the frother and collector molecules may attach to one another by van der Waals forces. Furthermore, frothers are therefore able to form a hydrogen bond with the oxygen atom in the collector molecule. These interactions however only take place when a mineral is present (Bradshaw et al., 1998).

It has been shown that the presence of collectors may affect the frothing properties of a particular frother, resulting in that frother negatively affecting the hydrophobicity of the desired minerals. The surface activity of a frother and the resulting frothing is sensitive to the presence of small amounts of other substances such as collector molecules or impurities. Certain frother-collector combinations may result in a decrease of conditioning time and an increase in both attachment efficiency and tenacity of attachment, resulting in better recoveries and flotation kinetics (Bradshaw et al., 1998)

## **2.8 Regulators**

Regulators, or modifiers, are used in froth flotation to manipulate the activity of the collectors. Regulators either increase or decrease the hydrophobicity or other surface properties of selected minerals. Regulators render collector activity more selective towards desired minerals. They may be classified as activators, depressants and pH modifiers. In this study, only depressants and pH modifiers are discussed as no activators were used.

### **2.8.1 Depressants**

Depression regulators or depressants are used to increase the selectivity of the froth flotation, by rendering certain minerals hydrophilic, thus restricting their flotation. They are used in the froth flotation of complex ores such as platinum and nickel sulphide ores. Since the activity of

depressants are not fully understood, it is difficult to control their effects in comparison to other types of reagents (Bradshaw et al., 2005).

There are many mechanisms proposed for depressant activity. These include the following (Bulatovic, 2007):

1. The desorption of collectors adsorbed on minerals' surfaces and the formation of an insoluble collector compound with the desorbed collectors that is not capable of re-adsorbing on the surfaces of minerals.
2. The interaction of depressant and activator on the surfaces of minerals, where the activator is desorbed from the minerals surfaces.
3. The interaction of depressants with cations in the slurry, resulting in the prevention of cation adsorption on the surfaces of minerals thus causing activation.
4. The adsorption of the depressant on the surfaces of minerals as a monolayer, resulting in the prevention of collector adsorption on selected minerals.

For the purpose of this study, the depressant used is sodium carboxy methyl cellulose (SCMC). This depressant is sold as Sendep 300 and is produced by Senmin. The use of this particular depressant was suggested, since the Nkomati ore is a complex nickel ore and is regarded as a problematic ore due to the inclusion of many naturally hydrophobic minerals such as chalcopyrite, pyrite, pyrrhotite and talc. It is desired that the hydrophobic gangue minerals such as pyrite, pyrrhotite and talc are depressed.

### **2.8.2 pH modifiers**

The pH of the pulp plays an important role in froth flotation, as it regulates its ionic composition. In froth flotation circuits, the selectivity and separation of minerals in complex ores are dependent on the collector concentration as well as the pH of the pulp. Froth flotation is normally carried out under alkaline conditions, since most collectors including xanthates are fairly stable here and the corrosion of the flotation cell and agitator are minimised relative to acidic conditions (Bulatovic, 2007).

When alkaline conditions are desired, the pH of the pulp is normally controlled by the addition of calcium hydroxide (lime). By adding calcium hydroxide to the pulp, it dissociates and the calcium and hydroxide ions are released. The increased addition of calcium hydroxide will result in an increase of hydroxide ions in the pulp. This decreases the concentration of hydrogen ions and subsequently alters the ionic composition of the pulp. Alternatively, sodium hydroxide or ammonia

are used to increase the pH. The use of calcium hydroxide is however more cost effective than sodium hydroxide and ammonia (Bulatovic, 2007).

For acidic conditions, sulphuric acid is normally added to decrease the pH of the pulp. Sulphuric acid is relatively cost effective and is therefore widely used. Due to the nature of this study, the froth flotation experiments were conducted in alkaline conditions and therefore only alkaline pulp conditions will be discussed further.

Calcium hydroxide is added to regulate the pH of the pulp in the froth flotation of base metal sulphide ores. The costs of pH modifiers specifically calcium hydroxide are lower than either collectors and frothers. However, it should be noted that the combined cost of pH regulators per tonne of treated ore are generally greater than the cost of collectors and frothers (Wills and Napier-Munn, 2006).

pH regulators may be used as depressants (Bulatovic, 2007) as hydroxyl and associated hydrogen ions, alter the electrical double layer surrounding the selected mineral's surface, thus influencing its floatability. Critical pH values for the froth flotation of certain minerals exist and depend largely on the type of minerals present, the type of collectors used and the operating temperature (Sutherland and Wark, 1955). Figure 18 illustrates the critical pH values for two of the major gangue minerals (pyrite and chalcopyrite) present in the Nkomati ore with sodium diethyl dithiophosphate as collector.

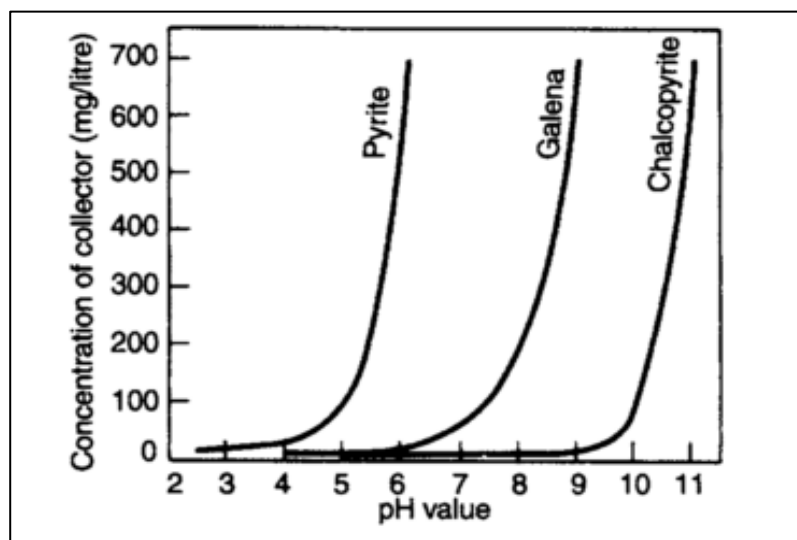


Figure 18: The relationship between sodium diethyl dithiophosphate collector and pH (Sutherland and Wark, 1955)

Conventional lime or soda ash is added to the pulp during its conditioning in order to achieve the desired pulp alkalinity and precipitate heavy metal salts (Wills and Napier-Munn (2006). With

xanthate collectors, alkali pulps can depress a broad range of sulphide minerals. For a particular concentration of xanthate collector there is a specific pH value below which a desired mineral will float and above it will not.

From the curves (Figure 18) it is clear that using sodium diethyl dithiophosphate collector at a dosage of approximately 50mg/litre and at a pulp pH of 8 a separation of chalcopyrite from both galena and pyrite would occur. At the same pH (pH 8), with an increase in collector dosage to 100mg/litre, galena would separate from both chalcopyrite and pyrite. Similarity at the same initial collector dosage (50mg/litre), but at a pulp pH of 6 a separation of galena and chalcopyrite from pyrite would result. It is therefore possible to identify pH ranges for specific minerals at defined collector concentrations (e.g. pH range for pyrite flotation is 2.5-6, relative to the concentration of collector used) (figure 18).

## **2.9 Particle size**

Particle size plays a significant role in determining the time required for flotation and the achievement of acceptable grades and recoveries. The particle size influences the rate of collector adsorption (Bulatovic, 2007). It is widely accepted that fine particles lead to higher recoveries, whereas coarse particles generally lead to higher concentrate grades.

There are two critical size limits of particles that influence their floatability. The first is a lower limit below which they will not float as particles that are too fine generally experience a lower number of bubble-particle interactions. This is due to having small inertial forces as a result of their small masses. The second limit is an upper limit above which they will also not float as coarse particles may be heavy enough to detach from air bubbles during transportation to the froth (Schubert, 2008).

These critical particle size limits differ from one mineral to another. The lower and upper particle size limits for most minerals are 10 to 100 $\mu$ m (Schubert, 2008). Since finer particles have lower numbers of bubble-particle interactions in comparison to coarser particles, turbulent conditions are recommended to increase the rate of interaction (Schulze, 1984). A typical particle size and recovery trend further emphasises the critical particle size limits for froth flotation (figure 19):



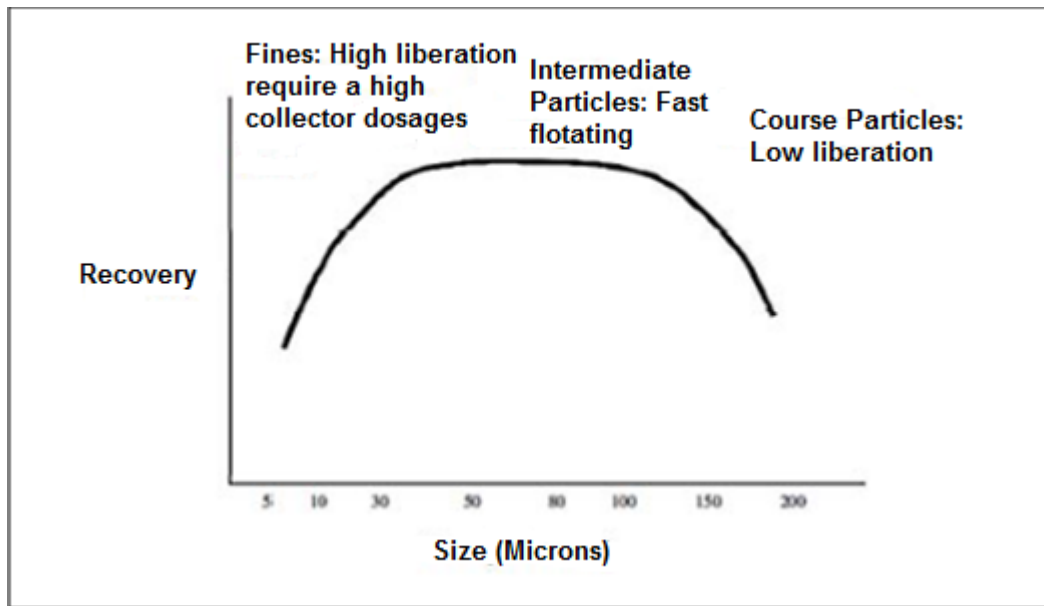


Figure 19: The relationship between particle size and mass recovery (Pearse et al., 2006)

In small commercial plants, jaw and cone crushers are used to crush the ore to sizes of between 0.5cm and 1mm. Since wet milling is normally used to decrease the particles size and liberate the desired minerals, care should be taken to restrict the oxidation of their surfaces. If the desired minerals float readily, a coarser grind may sometimes be used (Wills and Napier-Munn, 2006).

Coarse particles initially have lower flotation rates and recoveries compared to finer particles. They also require higher pulp densities, higher collector dosages and generally require longer flotation times than that of finer particles. Advantages associated with floating coarser particles include a decrease in entrainment and slime coatings, due to the lower number of fine particles present in the pulp. It should be noted that although coarse particles may float more readily in certain cases, the incomplete liberation of minerals may occur (Wills, 2006; Schubert, 2008).

There are many advantages and disadvantages associated with using fine particle sizes. Some of the advantages are that:

- Maximum liberation of the desired minerals is allowed.
- A lower collector dosage for surface coverage in comparison to coarser particles.
- Bubble-particle transportation and attachment is more effective in contrast to coarser particles (Klimpel, 1984).

The disadvantages of using fine particle sizes were summarised by Ngobeni (2013) and include the following:

- They collectively have a large surface area and are therefore more easily oxidised.
- They are susceptible to entrainment and as a result decrease the concentrate grades.
- They have low collision rates and therefore require more pulp turbulence in order to promote bubble-particle interactions and collisions. This leads to an increase in the agitation rate and subsequent increase in power consumption.
- The dominant transport mechanism is entrainment, due to turbulence caused by an increase in the agitation rate. This further decreases the concentrate grades.

The disadvantages however outweigh the advantages and care should be taken not to exceed the lower limits of desired minerals. Generally long chained collectors are used in the flotation of fine particles, while short chained collectors as well as collector mixtures are used to float course particles (Szymula et al., 1996). The desired mineral is a pentlandite in the Nkomati ore used in this study. Figure 20 illustrates its recovery as a function of particle size.

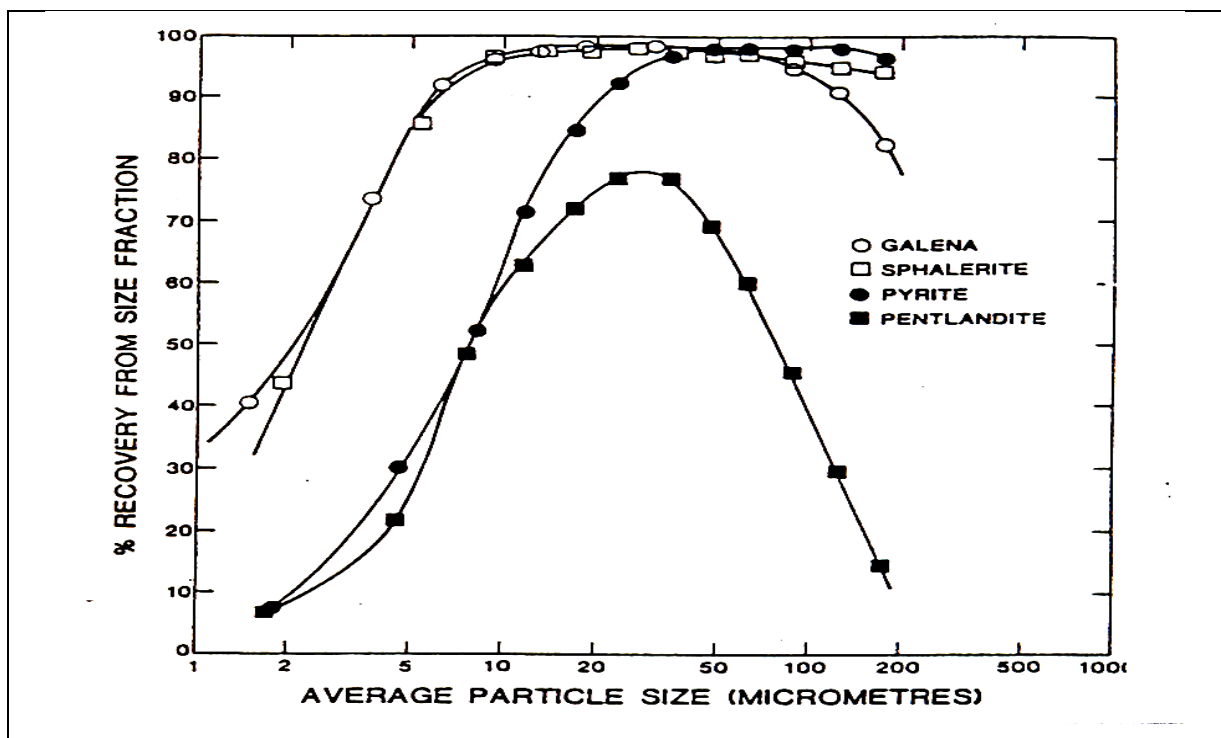


Figure 20: Galena, sphalerite, pyrite and pentlandite mineral sizes; typically used for froth flotation (Senior et al., 2005: 211)

There are many different particle size distribution suggested for the froth flotation of pentlandite ores, using a laboratory batch froth flotation cell. It is widely accepted that the upper particle size limit should not be finer than 80% passing 75µm.

Bafubiandi and Medupe (2007) performed tests with various particle size distributions and found that a grind finer than 80% passing 75  $\mu\text{m}$  yielded high concentrate grades initially and that they dramatically decreased during the rest of the flotation. It was further found that at particle size distribution 80% passing 75  $\mu\text{m}$ , the highest mass and nickel recoveries were recorded accompanied by the lowest grades. The decrease in grades was suggested to be due to an increase in entrainment due to the finer particles.

When floating fine particles, a higher mass recovery and a decrease in grade is usually expected (Deglon, 2005). It was therefore decided that a particle size distribution of 80% passing 75  $\mu\text{m}$  will be used in this study based on previous work done (Ngobeni, 2013; Szymula et al., 1996; Bafubiandi and Medupe, 2007) as well as the peak recovery area (figure 20).

### **2.10 Air flow rate**

The air flow rate into the froth flotation cell and pulp affects the degree of entrainment, which consequently affects the grades and recoveries (Tuteja et al., 1995). It is expected that high air flow rates would result in high mass recoveries, with lower grades.

As the air introduced into the flotation pulp is increased, the bubble generation increases and provides more bubbles for mineral-bubble interaction and mineral transportation. Due to the increase in the number of bubbles, a thicker froth phase is expected. Since more froth bubbles are available for mineral-bubble attachment, the resulting effects are an increase in mineral-bubble interaction, an increase in recoveries and a possible decrease in concentrate grades (Tuteja et al., 1995).

The formation of large bubbles with high velocities plays a significant role in entrainment of gangue minerals. This results in an increase in recoveries and decrease in grades (Goodall & O'Connor, 1991). When the bubbles have a high velocity, an increase in entrainment and a decrease in selectivity will result. This will furthermore cause a decrease in grade, but an increase in recovery (Tuteja et al., 1995). Longer drainage times are recommended when floating at high air flow rates.

When low air flow rates are used, they result in a decrease in bubble generation, which provides fewer bubbles for mineral-bubble interaction. These effects will cause a decrease in mineral-bubble attachment, in comparison to that of increased air flow rates, thus resulting in a decrease in recovery. The lower air flow rates also allow for a longer bubble residence time in the pulp, which could result in more selective flotation and a possible increase in concentrate grades (Moses and Petersen, 2000).

In this study an air flow rate of 7ℓ/min was used as it was found to be standard commonly used air flow rate for batch 3ℓ laboratory benchtop froth flotation machine (Ngobeni, 2013; Nyambayo, 2014).

### **2.11 Agitation speed**

Agitation is important in any froth flotation process, since it promotes the dispersion of air bubbles and mineral particles in the pulp. A higher agitation rate generally produces an increase in the gas and particle dispersion. This promotes mineral-bubble interaction and consequent attachment.

The agitation rate required for the froth flotation of a particular ore depends largely on the air flow rate and particle size distribution. Low agitation rates are preferred for coarse particles and high agitation rates are preferred for finely ground particles (Deglon, 2005; Grano, 2006; Wills, 2006; Schubert, 2008; Al-Thyabat, 2009). The bubble sizes of the froth are also affected by the agitation rate. Smaller bubbles are usually formed with higher agitation speeds while larger bubbles format lower agitation speeds.

There are negative effects of a high agitation speed if the particle size distribution is not chosen correctly. High agitation speeds create turbulence within the pulp. This causes an increase in the existing shear forces, resulting in higher shear forces greater than the adhesive forces between the desired minerals and froth bubbles. This results in the detachment of desired minerals and a decrease in recoveries and grades (Deglon, 2005; Grano, 2006; Schubert, 2008).

High agitation rates also increase the collision rates of minerals and bubbles which result in an increase in entrainment (Deglon, 2005; Grano, 2006). With low agitation rates, large desirable mineral particles may tend to settle from the pulp, which prohibits attachment to froth bubbles. Nyambayo (2014), Wiese (2007) and McFadzean (2013) used 1200rpm with a particle size distribution 60% passing 75µm. The agitation rate chosen for this study was also 1200rpm to enable standard comparison and to create turbulence which is needed when floating finer particles.

### **2.12 Pulp density**

Pulp density refers to the solid to liquid mass ratio and is typically expressed as a mass percentage of solids. The widely accepted upper limit of solids in a pulp is normally 55% by mass and the lower limit is accepted to be 8% by mass. Industrial froth flotation circuits use pulp densities of between 25% to 40% solids (Kirjavainen, 1996).

Generally, higher pulp densities aid in maintaining an effective suspension. However, high pulp densities may compromise, the selective drainage of entrained gangue minerals and result in increased entrainment, higher recoveries and lower grades (Kirjavainen, 1996).

High pulp densities also require flotation cells of smaller volume and subsequently require smaller dosages of flotation reagents. Consequently, high pulp densities are preferred in the froth flotation of coarse and dense minerals. By increasing the pulp density, the potential recovery of desired minerals increases, and the grades decrease. In contrast, lower pulp densities require higher reagent dosages in order to ensure that the reagent concentration levels are maintained for adequate froth flotation (Kirjavainen, 1996). Low pulp densities are preferred during the cleaning stages of industrial flotation circuits, since they reduce the recovery of fine hydrophilic minerals (Kirjavainen, 1996). It is thus widely accepted that a decrease in pulp density will result in a decrease in recovery, but an increase in grade. A pulp density of 30% was chosen for this study, based on previous work done by Ngobeni (2013), Bacus (2000) and Nyambayo (2014).

### **2.13 Collector dosage**

With an increase in collector dosage to a particular flotation pulp, a higher recovery could be expected. At low collector dosages, the low collector concentration in the pulp could cause a low rate of adsorption of the collector onto the desired minerals' surfaces. This would result in a decrease in recovery since selective attachment of minerals on bubbles would decrease. The grade on the other hand may be higher since the dominant mechanism of recovery may be selective froth flotation (Rao and Forssberg, 1997).

The concentration where maximum recovery takes place is referred to as the critical micelle concentration (CMC). Here, collectors are very selective and do not form multiple collector layers on the minerals' surfaces. The collectors' selectivity depends on its ability to form a single layer on the minerals' surfaces. The single layer will render the minerals hydrophobic, whereas a double layer will result in the mineral reverting to a hydrophilic state (Sheridan et al., 2002; Senior et al., 2005).

## 2.14 Industrial froth flotation circuits

In the processing of low grade ores, one froth flotation stage is usually not adequate to achieve sufficient concentration for further processing. Froth flotation is therefore carried out in more than one stage. When two or more stages are used the process is referred to as a froth flotation circuit (Wills and Napier-Munn, 2006).

In a froth flotation circuit there are usually three main stages which include the rougher stage, scavenger stage and the cleaning stage. There is no general standard of arrangement for the order of the flotation stages, since they differ from ore to ore (Heikki et al., 2002). In industrial froth flotation circuits, true flotation is usually the desired mechanism and hydraulic entrainment and physical entrapment cannot be completely controlled. As a result, multiple froth flotation stages are used rather than a single stage (figure 21):

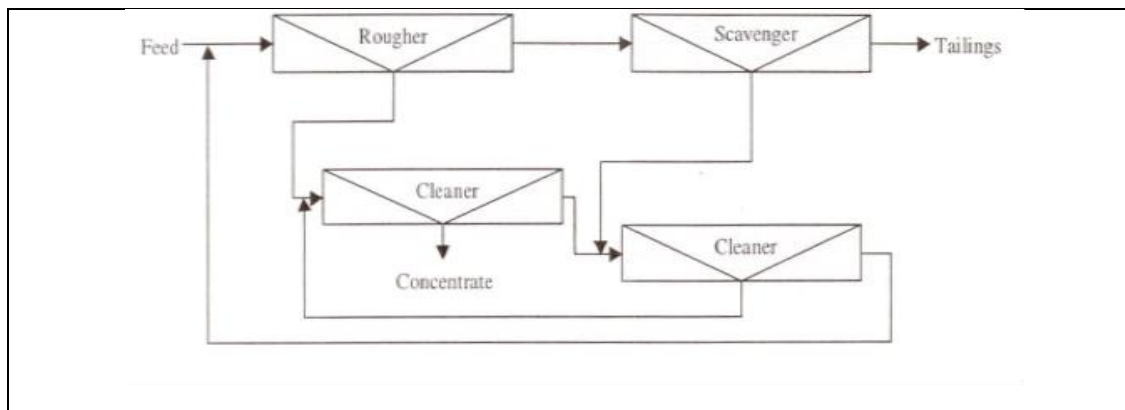


Figure 21: A typical flotation circuit (Wills and Napier-Munn, 2006)

The rougher stage is used to process fresh feeds and the desired minerals are roughly separated from the gangue. This initially results in high recoveries and high grades (Heikki et al., 2002). The primary function of the rougher stage is to maximise the recovery of the desired minerals while ensuring the maximum rejection of gangue. The gangue from the rougher stage is sent to the scavenger stage, where further processing and subsequent recovery of desired minerals are done. Since the feed sent to the scavenger stage is of a poor grade, high reagent dosages and air flow rates are usually used (Heikki et al., 2002). In the cleaner stage in froth flotation circuits the concentrates of the rougher stages are further processed and floated to reject entrained and entrapped gangue. The concentrates of the cleaner stages are then either leached or smelted.

## **2.15 Laboratory batch froth flotation**

The objectives of laboratory froth flotation tests are to validate the results obtained from plant analyses or to explore different flotation options to improve grades and recoveries. Laboratory scale batch froth flotation is an easy and inexpensive method used for the replication of plant process conditions. It is therefore possible to investigate the effects of changing certain variables in a particular stage, which may assist in diagnosing problems for industrial flotation circuits.

Standard operating procedures and conditions as well as techniques based on previous work done by Ngobeni and Hangone (2013) are used to assess the floatability of an ore. Laboratory flotation experiments are used to determine the optimum feed grind size and predict the change in performance of different ore types. It also allows the determination of important floatability parameters, such as flotation rate constants and the proportion of minerals in different floatable classes.

# CHAPTER 3

## 3 Methodology

### 3.1 Experimental Design

The first experimental design (Table 5) was done to evaluate the performance of the industrial mixture, single collectors and proposed mixtures at an industrially recommended frother dosage (12g/t). These tests were also all performed at constant collector and depressant dosages.

Table 5: The experimental design with frother at 12g/t

| Test Run | Collector    | Constituents ratio  | Collector Dosage<br>( depressant at 240g/t)   |
|----------|--------------|---------------------|---|
| 1.       | No Collector | -                   | 0   |
| 2.       | SIBX:IPETC   | 95.5%SIBX:4.5%IPETC | Molar equivalent of 95.5% SIBX:<br>4.5% IPETC at 220 g/t<br>Or<br>(molar dosage of 1.3mmol/t) |
| 3.       | PAX          | 100%                |   |
| 4.       | SIBX         | 100%                |   |
| 5.       | IPETC        | 100%                |   |
| 6.       | SIBX:IPETC   | 95.5%SIBX:4.5%IPETC |   |
| 7.       | PAX:IPETC    | 90%PAX:10%IPETC     |   |
| 8.       | PAX:IPETC    | 85%PAX:15%IPETC     |   |
| 9.       | PAX:IPETC    | 80%PAX:20%IPETC     |   |



The second experimental design (Table 6) was done to evaluate the change in performance (nickel grade and recovery) with a decrease in the collector dosage. The frother dosage was increased to 50g/t and kept constant, to ensure a stable frother phase. Depressant was also once again added at a constant dosage of 240g/t.

Table 6: The experimental design with frother at 50g/t

| Test Run   | Collector    | Constituents ratio  | Collector Dosage<br>(depressant at 240g/t)   |
|------------|--------------|---------------------|--|
| 10.        | No Collector | -                   | 0  |
| 11.        | PAX          | 100%                | Molar equivalent of 95.5%<br>SIBX:4.5% IPETC at 220 g/t<br>Or<br>(molar dosage of 1.3mmol/t) |
| 11. Repeat | PAX          | 100%                |  |
| 12.        | SIBX         | 100%                |  |
| 12. Repeat | SIBX         | 100%                |  |
| 13.        | IPETC        | 100%                |  |
| 13. Repeat | IPETC        | 100%                |  |
| 14.        | SIBX:IPETC   | 95.5%SIBX:4.5%IPETC |  |
| 14. Repeat | SIBX:IPETC   | 95.5%SIBX:4.5%IPETC |  |
| 15.        | PAX:IPETC    | 95.5%PAX:4.5%IPETC* |  |
| 15. Repeat | PAX:IPETC    | 95.5%PAX:4.5%IPETC  |  |
| 16.        | PAX:IPETC    | 90%PAX:10%IPETC*    |  |
| 16. Repeat | PAX:IPETC    | 90%PAX:10%IPETC     |  |
| 17.        | PAX:IPETC    | 85%PAX:15%IPETC*    |  |
| 17. Repeat | PAX:IPETC    | 85%PAX:15%IPETC     |  |
| 18.        | PAX:IPETC    | 80%PAX:20%IPETC     |  |
| 18. Repeat | PAX:IPETC    | 80%PAX:20%IPETC     |  |
| 19.        | PAX:SIBX     | 50%PAX:50%SIBX      |  |
| 19. Repeat | PAX:SIBX     | 50%PAX:50%SIBX      |  |

Mixtures with a \* were further tested to explore the effects of collector dosages on nickel grade and recovery.

### 3.2 List of Equipment Required

In order to perform a standard laboratory froth flotation test, a specified list of equipment is scoped (Table 7). The tabled list of equipment is based on previous work done by Ngobeni and Hangone (2013) which was in accordance to the standard froth flotation test procedure.

Table 7: The equipment list and number of equipment units required

| Equipment                     | No of units needed |
|-------------------------------|--------------------|
| Stainless steel rod mill      | 1                  |
| 3l Batch froth flotation cell | 1                  |
| XRF analyser                  | 1                  |
| pH meter                      | 1                  |
| Water bottles                 | 7                  |
| Concentrate containers        | 4                  |
| Laboratory balance scale      | 1                  |
| Buchner Funnel                | 1                  |
| Oven                          | 1                  |
| 1ml Pipette                   | 1                  |
| 25ml Volumetric flask         | 3                  |
| 200ml Volumetric flask        | 1                  |
| 5ml Volumetric flask          | 1                  |
| 110mm Filter paper            | 50                 |
| 320mm Filter paper            | 50                 |
| 50ml syringe                  | 2                  |
| 25ml syringe                  | 2                  |
| Pipette bulb                  | 1                  |
| Fluid dropper                 | 2                  |
| Laboratory Spatula            | 1                  |
| Froth scrappers               | 2                  |
| Stop watch                    | 1                  |

### 3.3 Health and Safety Considerations

The health and safety considerations of any experiment are of the utmost importance and should be considered before attempting any experiment. Froth flotation is a process which is relatively safe in comparison to many other industrial processes. The main health and safety concerns are those regarding the chemical reagents used. Most of the reagents used in the froth flotation of mineral ores are relatively safe and are not directly harmful, unless consumed or exposed to sensitive areas on the body such as the eyes.

However, when handled in a safe and responsible manner, there is no major harm upon exposure. It is however, recommended that safety gloves and spectacles be worn when working with froth flotation reagents. The extra make up solutions may be discarded down a drain, except for cyanide based flotation reagents and other harmful specified reagents.

### 3.4 Experimental Procedure

1. Record the mass of the ore sample and add it and Cape Town municipal water to the rod mill to achieve 40% water and 60% ore by mass.
2. Mill for 28 minutes (clean the mill shell and rods before use).
3. Calibrate the pH meter and prepare the chemical reagents required into solutions (frother, depressant and collectors).
4. Record the masses of the filled water bottles, the empty concentrate containers and the dry filter papers to be used in the Buchner funnel and pressurised filtration vessel.
5. Add water to the flotation cell to cover the agitator.
6. Switch only the agitator on and set the agitation speed to 1200rpm.
7. Pour the milled sample into the flotation cell and add water to raise the pulp to the appropriate level (to achieve a froth height of 2cm upon the introduction of air) and allow agitation for 1 minute.
8. Insert the pH meter into the pulp and raise its pH to 10 with the addition of lime ( $\text{Ca(OH)}_2$ ). Once the pH is constant at approximately 10 for 1 minute, remove the pH meter from the pulp and rinse the probe of the pH meter over the flotation cell using water bottle 6, to ensure minimal sample loss.

9. Take a 20ml feed sample.
10. Add the collector or collector mixture to the pulp and condition for 2 minutes.
11. Add the frother and allow it to condition for 1 minute.
12. Switch on the air feeder and set to 7ℓ/min.
13. Use a scrape interval of 10 seconds.
14. Scrape the froth into (And the rest) container 1 for 2 minutes.
15. Scrape the froth in container 2 for 4 minutes.
16. Scrape the froth in container 3 for 6 minutes.
17. Scrape the froth in container 4 for 8 minutes.
18. Shut off the air feed supply and wait for one minute before taking a 20ml tailings sample.
19. Remove the tailings from the flotation cell by draining it into a bucket, then switch off the power.
20. Transfer the tailings from the bucket into the pressurised filtration vessel and allow filtration until a filter cake is achieved on the 320mm filter paper.
21. After pressure filtration, place the filter cake, feed and tailing samples in an oven to dry.
22. Record the masses of the concentrates as well as the used water bottles to establish the amount of water added to the cell and the water entrained.
23. Filter each the concentrates in a Buchner funnel and place the filter paper cakes in an oven to dry.
24. Measure the dry masses of the tailings, concentrates and calculate the mass and water recoveries.
25. Store all dried samples in zip locked bags for XRF analysis.
26. Repeat steps 1 to 26 for the experiments specified within the experimental design.

### 3.5 Laboratory Equipment

#### 3.5.1 Sample splitter

The bulk sample was received as 20 × 2kg sub-samples. Three sample bags were thus randomly added together to obtain the required 6kg sample for splitting. A vibratory rotary splitter was used to separate a 6kg sample into 6, 1kg samples (Figure 22).



Figure 22: A photograph of the 6 split splitter

### 3.5.2 The rod mill

The purpose of the rod mill is to decrease the particle sizes and liberate the desired minerals in order to perform adequate froth flotation experiments (Figure 24). Wet milling was used as it is the industry standard for size reduction with the aim of performing laboratory batch froth flotation experiments.

The rod mill uses a drive rotation motion at a set rotation speed which allows the rods contained in the shell to grind the material. The rod mill had a total of 20 rods, where 12 rods were 30cm x 25mm in diameter and 8 were 30cm x 15mm in diameter. A rotation speed of 59 rpm was used.



Figure 23: A photograph of the rod mill

### 3.5.3. The flotation cell

The flotation cell to be used to conduct the experiment is a bench top batch froth flotation cell (Figure 23). The batch flotation cell contains an agitator, air feeder and a water feeder. The flotation cell has a maximum capacity of 3ℓ, the agitation rate was maintained at 1200rpm and the air flow rate maintained at 7ℓ/min throughout the froth flotation experiments. Four sample dishes were used to collect the concentrates.



Figure 24: A photograph of the batch flotation cell

### 3.5.4 X-ray fluorescence spectrometer

A portable X-ray fluorescence (XRF) analyser was used to determine concentration of the metal elements in the mineral concentrates (Figure 25). The process of XRF analysis operates on the principle of exciting a specimen with high energy X-radiation and the detection of lower energy x-ray fluorescence radiation. In the process, electrons from the inner electron shells of the specimen are knocked out of their shells and outer electrons fill these voids, while emitting a fluorescence radiation. The fluorescence radiations emitted by these outer electrons are unique for particular metal element. The fluorescence radiation is evaluated using a detector.



Figure 24: A photograph of the laboratory XRF used for analysis (United Spectrometer Technologies cc, n.d)



### 3.5.5 Malvern Particle Size Analysis

A Malvern Mastersizer hydro 2000G was used to perform the particle size analysis of the concentrates (Figure 26). Due to this instruments small measuring range capability, it is the preferred particle size analyser in most flotation laboratories. The instrument is able to measure particle sizes from 0.05 $\mu\text{m}$  to 3480 $\mu\text{m}$  using laser diffraction.



Figure 25: The laboratory Malvern Mastersizer 2000 used for analysis

### 3.5.6 Sulphur Analysis

To ensure that sulphide minerals was indeed concentrated and recovered preferentially, elemental sulphur analysis was done. The measuring instrument used for the sulphur analysis of the feed, tailings and concentrate samples was the Leco SC-432 sulphur analyser (Figure 27). This Leco sulphur analyser loads and weighs samples with no preparation required and is supplied with pure oxygen to enable a combustion process. From the combustion process, the sulphur present in the sample oxidises to form  $\text{SO}_2$ . The resulting gases are then detected using an infrared sulphur detecting measuring cell.



Figure 26: Leco sulphur analyser

# CHAPTER 4

## 4 Results and Discussion

This chapter outlines the results obtained from the batch froth flotation tests with the use of:

- Single collectors and no collector
  - The experimental results obtained will serve as a basis for comparison. It will also indicate which effects each collector has on the froth flotation grades and recoveries.
- A collector mixture used in industry namely 95.5%SIBX: 4.5%IPETC at 1.3mmol/t (To determine the industrial grades and recoveries achieved).
  - The results obtained will thus also be used as a basis for comparison for the proposed collector suites.
- Collector mixtures of PAX and IPETC at a molar dosage of 1.3mmol/t used for the industry comparison.
- Collector mixtures of PAX and IPETC at decreased dosages.
- Collector mixtures of PAX and IPETC as well as the industry collector mixture at decreased frother dosages (12g/ton).
- Collector mixture using PAX and SIBX at a 50:50 mole ratio.

Although batch froth flotation is an effective method used to analyse the performance of certain collectors on various ores, they do not provide a total explanation as to what the ultimate cause of enhanced flotation performances are. To elucidate these occurrences, the following test were performed:

- Batch froth flotation kinetics modelling using the Klimpel rate equation.
- Leco sulphur analysis. To confirm that sulphide minerals are indeed the predominant minerals being recovered.
- Malvern size analysis. To investigate the particle size distribution of minerals reporting to each of the concentrates for selected batch froth flotation tests.

## 4.1 Reproducibility

### 4.1.1 Feed particle size distribution

The feed sample has to be consistent in terms of particle size distribution to ensure reproducibility and to usefully compare the different flotation tests, especially the effects of single variables such as collector or frother dosages. To evaluate the uniformity of the feed samples, three randomly chosen samples were analysed using a Malvern particle size analyser. The analysis of each feed sample was repeated 5 times and the average particle size distribution was calculated. The particle size distributions were analysed between 1-1000µm. However only the particle size range between 1-110µm (the upper and lower target liberation ranges) was used to generate Figure 28.

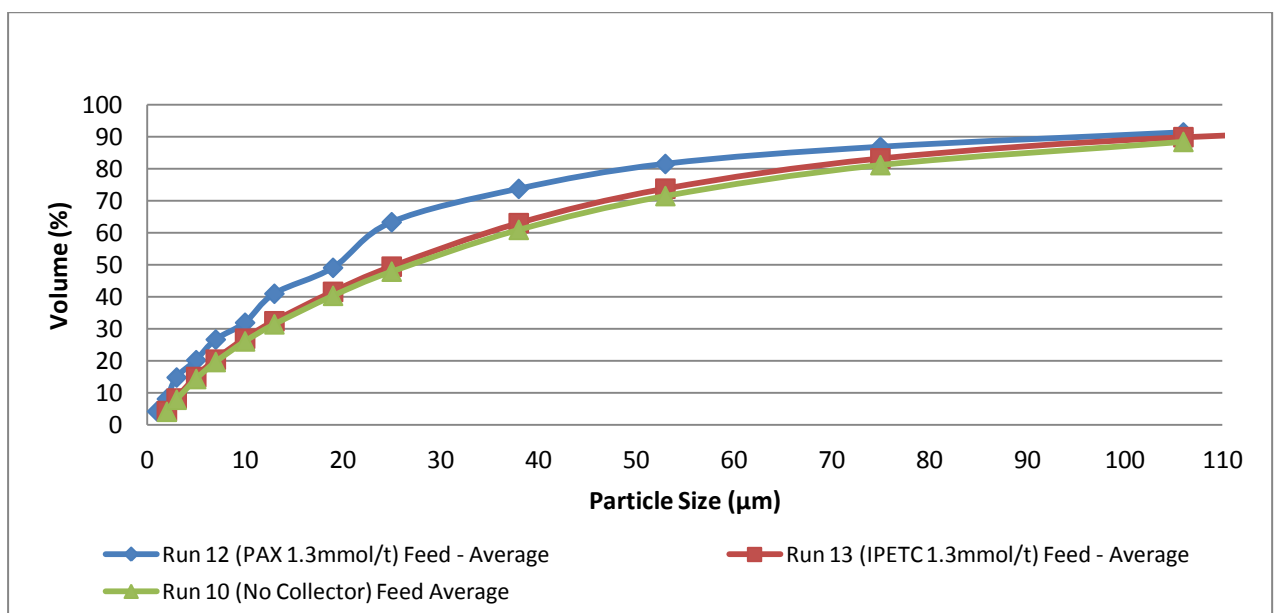


Figure 27: Particle size distribution of three randomly chosen samples

The three curves follow the same trend and are shown to have reproducible particle size distributions when milled for a target of  $P_{80\%} - 75\mu\text{m} \pm 5\mu\text{m}$ . It may be concluded that the milling of the ore samples and the subsequent particle size distribution of the feed is reproducible. A milling curve was plotted to find the required milling time to achieve a target grind size of 80% mass passing 75µm. By interpolation this was found to be 28 minutes (Figure 29).

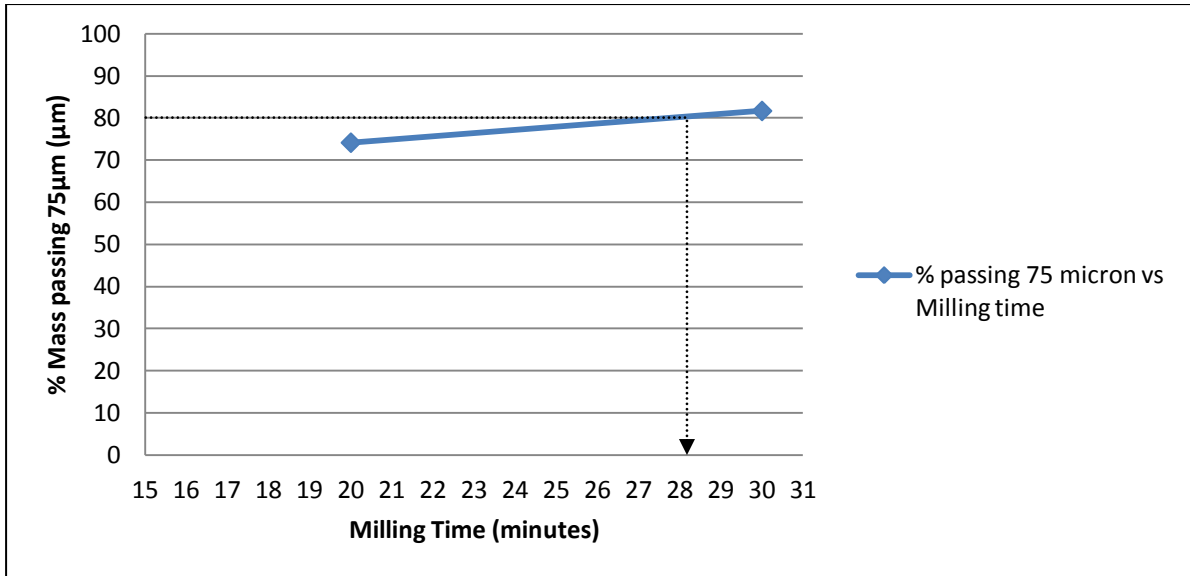


Figure 28: % Mass passing 75µm vs milling time

#### 4.1.2 Batch Froth Flotation Test

The batch froth flotation tests were all repeated, with the exception of the test performed at 12g/ton of frother and the collectorless test. The standard deviations for each test was calculated and shown as error bars in all subsequent graphs. To establish if there was a significant difference in the results obtained with the industrial mixture, a test of hypothesis for a mean procedure was used (Perry, et al., 1997).

A two-tailed t-test with 5% significance or 95% confidence was the preferred test for hypothesis testing, as the mean and standard deviations of the data was calculated (A z-test could have also been used, since repeats on the industry mixture allowed the calculation of both the mean and the associate standard deviations). The Null hypothesis ( $H_0$ ) stated that there is an insignificant change in the 95% confidence level and was accepted if the calculated t sample was lower than the t-critical values and rejected if it was found to be higher (Perry, et al., 1997).

## 4.2 Single Collectors and Mixtures at Frother Dosage 12g/ton

### 4.2.1 Summary of Results

Table 8: Results summary for nickel using collector suites

| Nickel                                   |            |  |        |                               |        |                                |        |                             |        |  |                                       |
|--|------------|--|--------|-------------------------------|--------|--------------------------------|--------|-----------------------------|--------|--|---------------------------------------|
| Collector<br>(molar dosage of 1.3mmol/t) | Mole Ratio | Cumulative Concentrate Mass Recovery (%) |        | Cumulative Water Recovery (%) |        | Cumulative Nickel Recovery (%) |        | Cumulative Nickel Grade (%) |        | Klimpel Rate Constant K (min <sup>-1</sup> ) | Maximum Recovery R <sub>max</sub> (%) |
|  |            | 2 min                                    | 20 min | 2 min                         | 20 min | 2 min                          | 20 min | 2 min                       | 20 min |  |                                       |
| IPETC                                    | 100        | 11.4                                     | 29.4   | 15.9                          | 83.2   | 51.4                           | 74.8   | 2.4                         | 1.3    | 1.4  | 76.1                                  |
| SIBX: IPETC                              | 95.5: 4.5  | 13.2                                     | 26.0   | 16.5                          | 56.0   | 42.8                           | 79.4   | 1.6                         | 1.5    | 0.8  | 82.4                                  |
| PAX: IPETC                               | 95.5: 4.5  | 8.9                                      | 15.2   | 8.6                           | 14.1   | 41.2                           | 68.6   | 2.4                         | 2.3    | 1.0  | 70.3                                  |
| PAX: IPETC                               | 90 : 10    | 9.5                                      | 14.0   | 9.2                           | 11.9   | 42.2                           | 66.0   | 2.1                         | 2.3    | 1.2  | 66.0                                  |
| PAX: IPETC                               | 85 : 15    | 9.4                                      | 16.9   | 9.3                           | 18.4   | 44.6                           | 73.3   | 2.3                         | 2.1    | 1.0  | 75.1                                  |
| PAX: IPETC                               | 80 : 20    | 9.3                                      | 18.9   | 9.6                           | 23.6   | 40.9                           | 74.3   | 2.1                         | 1.9    | 0.8  | 78.2                                  |

### 4.2.2 Nickel Grade versus Recovery

The cumulative nickel grades displayed the following trend: 95.5%PAX:4.5%IPETC = 90%PAX:10%IPETC > 85%PAX:15%IPETC > 80%PAX:20%IPETC > 95.5%SIBX:4.5%IPETC > IPETC, while their corresponding numerical values were 2.3%, 2.3%, 2.1%, 1.9%, 1.5% and 1.3% respectively (Figure 30 and Table 8). The cumulative nickel grades for the xanthate collectors (SIBX and PAX) as well as no collector were insignificant due to the inadequate formation of a froth phase.

Mixtures of PAX and IPETC gave cumulative nickel grades in the range of 1.9 to 2.3%, while IPETC and its mixture with SIBX gave a cumulative nickel grade of 1.5%. This suggests that the collector mixtures of PAX and IPETC may be more selective than the industrial mixture (95.5%SIBX:4.5%IPETC) and IPETC on its own. This could be as a result of compatible mixtures of collectors at an optimum ratio that improves both the concentrate grade and recovery (Buckely *et al.*, 2003).

Cumulative nickel recoveries decreased in the following order (Figure 30): 95.5%SIBX:4.5%IPETC > IPETC > 80%PAX:20%IPETC > 85%PAX:15%IPETC > 95.5%PAX:4.5%IPETC > 90%PAX:10%IPETC. The cumulative nickel recoveries obtained were 79.4%, 74.8%, 74.3%, 73.3%, 68.6% and 66.0% respectively (Table 8). IPETC as well as its mixtures with PAX resulted in a decrease in cumulative nickel recovery as opposed to the industrial mixture (95.5%SIBX:4.5%IPETC).

As expected the generally higher cumulative nickel grades came at the expense of cumulative nickel recoveries. With an increase in the concentration of IPETC in the collector mixtures, no significant increases in grades were observed. However, there were significant increases in the cumulative nickel recoveries. This may be due to increased frothing properties with the higher concentration of IPETC, resulting in higher froth stability, entrainment, lack of adequate mineral “drop-back”, resulting in increase nickel recoveries and poorer grades.

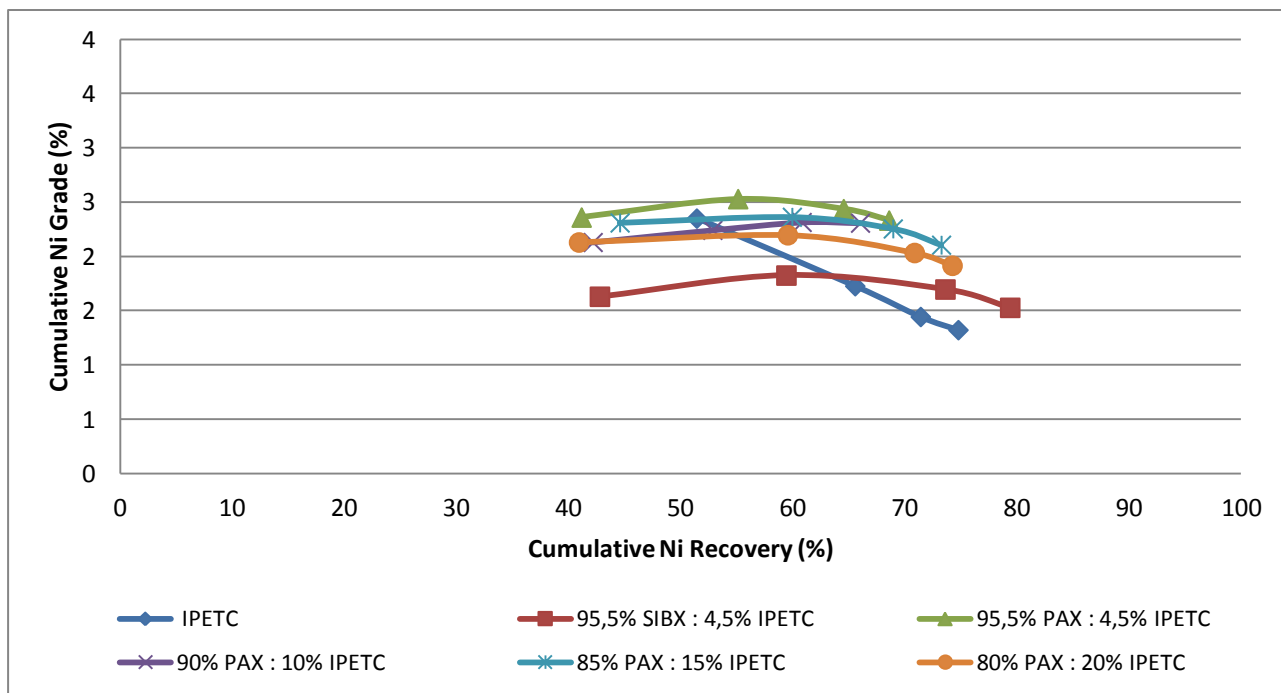


Figure 29: Percentage cumulative nickel recovery vs grade obtained with IPETC and its mixtures with 95.5, 90, 85 or 80 mol% PAX at a molar dosage of 1.3mmol/t and frother dosage of 12g/ton.

#### 4.2.3 Water versus Mass Recovery

The cumulative water recoveries of the collector mixtures at a frother dosage of 12g/t, decreased in the following order: IPETC > 95.5%SIBX:4.5%IPETC > 80%PAX:20%IPETC > 85%PAX:15%IPETC > 95.5%PAX:4.5%IPETC > 90%PAX:10%IPETC (Figure 31). The cumulative water recoveries were 83.2, 56.0, 23.6, 18.4, 14.1 and 11.9% respectively (Table 8). There seems to be a trend with IPETC and PAX, with a higher concentration of IPETC resulting in higher water and mass recoveries. It was however observed that the cumulative water recovery for the industrial collector mixture (95.5%SIBX:4.5%IPETC) was higher than all of the other mixtures and the highest cumulative water recovery was obtained upon the use of IPETC.

The cumulative mass recovery, similarly to the water recovery decreased in the following order: IPETC > 95.5%SIBX:4.5%IPETC > 80%PAX:20%IPETC > 85%PAX:15%IPETC > 95.5%PAX:4.5%IPETC > 90%PAX:10%IPETC (Figure 31). Their corresponding numerical values were 29.4, 26.0, 18.9, 16.9, 15.2 and 14.0% respectively (Table 8). It was expected that an increase in water recovery would result in a significant increase in the cumulative mass recovery due to entrainment.

This was observed for collector mixtures of PAX and IPETC, although the increase in mass recovery was not significant. The high water recoveries achieved with the industrial mixture and IPETC may have increased the cumulative mass recovery, but the effects were not clearly discernible. The grade and recovery data however suggest that the predominant mechanism for mass recovery was indeed entrainment and not collector-mineral interaction. There was a decrease in the cumulative mass and water recoveries with the mixtures compared to IPETC, which could be due to the frothing properties of IPETC. A stable froth phase with large bubbles was observed with an increase in IPETC concentration. The bubbles however became smaller in size towards the later stages of flotation (this may be due to the decrease in IPETC concentration in the pulp).

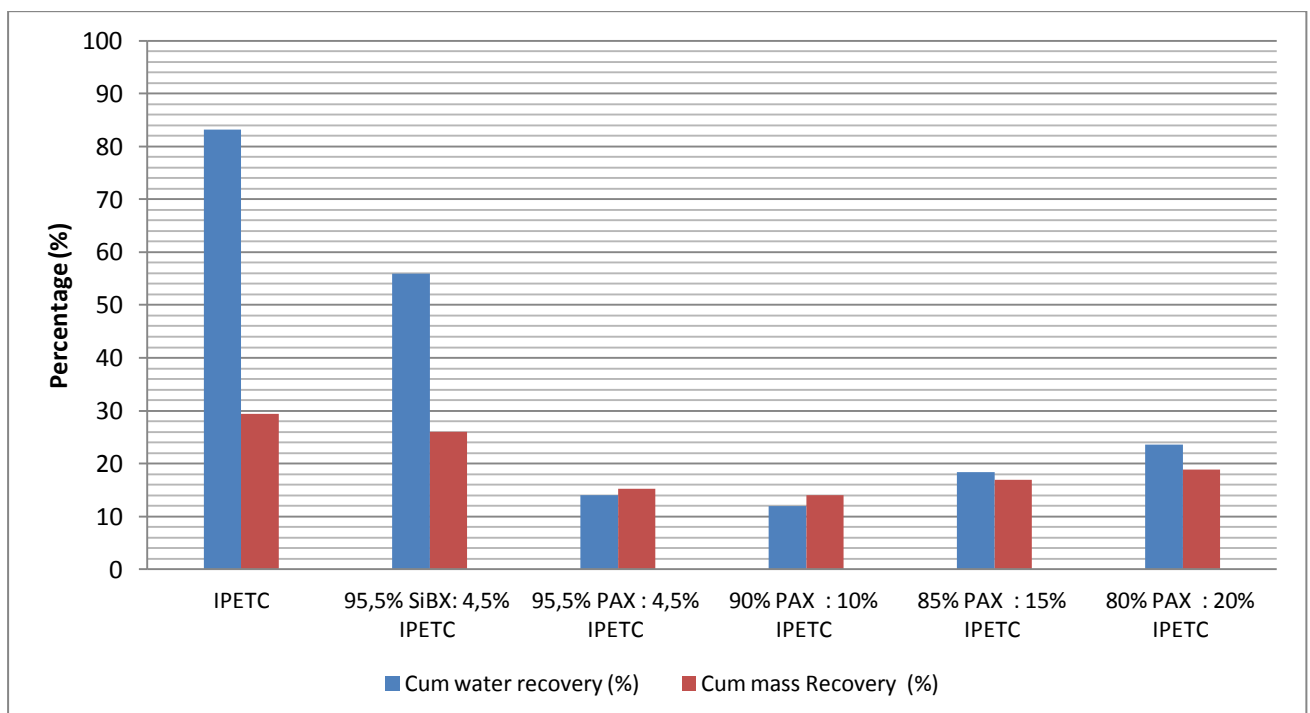


Figure 30: Cumulative mass versus cumulative water recoveries with an industrially used mixture and pure IPETC and its mixture with 95.5, 90, 85 or 80% PAX at a molar dosage of 1.3mmol/t



#### 4.2.4 Flotation Rates

The Klimpel rate constants were calculated (Table 8) to distinguish between the different rates for nickel flotation. The Klimpel rate constants displayed the following trend for the tested collectors and mixtures: IPETC > 90%PAX:10%IPETC = 95.5%PAX:4.5%IPETC = 85%PAX:15%IPETC > 95.5%SIBX:4.5%IPETC = 80%PAX:20%IPETC (Figure 32). The corresponding numerical values were 1.4, 1.2, 1.0, 1.0, 0.8 and 0.8min<sup>-1</sup> respectively (Table 8).

The highest Klimpel rate constant was achieved with the use of the IPETC (1.4min<sup>-1</sup>). The high water recovery and resulting entrainment may have contributed to the slightly higher Klimpel rate constant. The mixtures of PAX with IPETC together with the industrial mixture (95.5%SIBX:4.5%IPETC) gave similar results (approximately 1min<sup>-1</sup>). This suggested that the proposed collector mixtures of PAX and IPETC do not significantly improve the rate of nickel recovery compared to the industrial mixture at a frother dosage of 12g/t.

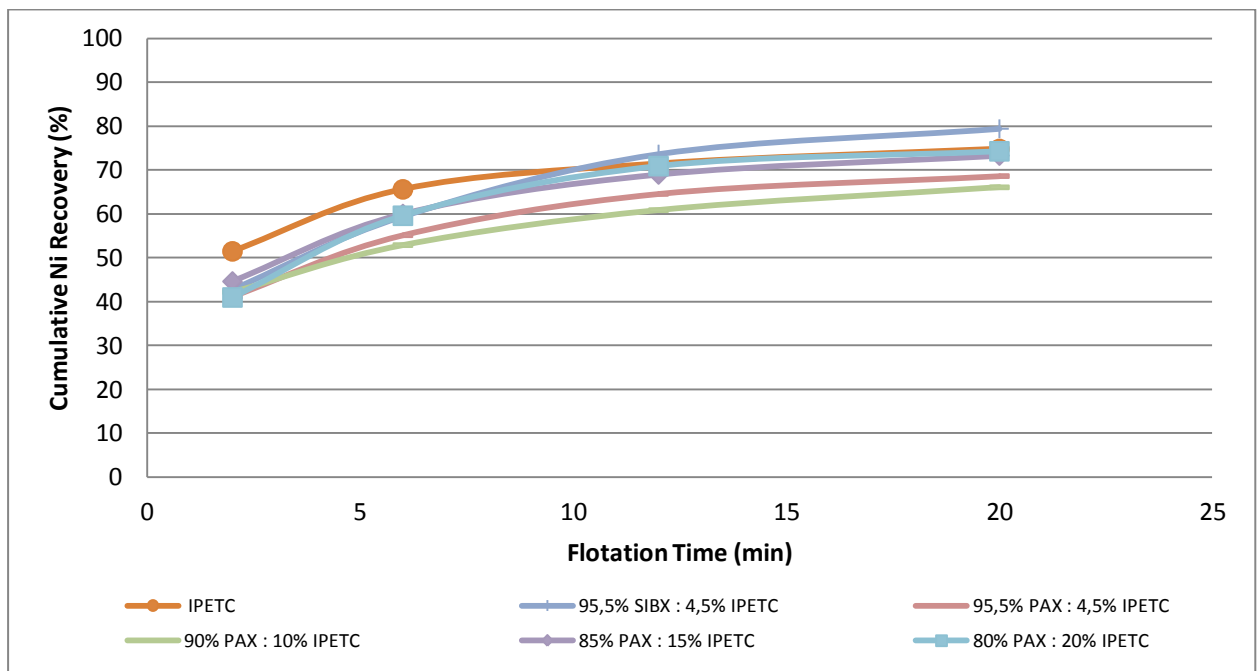


Figure 31: Percentage cumulative nickel recovery obtained with the use of IPETC and its mixtures with 95.5, 90, 85 or 80 mol% at a molar dosage of 1.3mmol/t

### 4.3 Single Collectors at Frother Dosage 50g/t

#### 4.3.1 Summary of Results

Table 9: Results summary for nickel using single collectors

| Nickel                                |            |   |        |                                    |        |                                     |        |                                  |        |  |                                       |
|---------------------------------------|------------|---|--------|------------------------------------|--------|-------------------------------------|--------|----------------------------------|--------|--|---------------------------------------|
| Collector (molar dosage of 1.3mmol/t) | Mole Ratio | Ave. Cumulative Concentrate Mass Recovery (%) |        | Ave. Cumulative Water Recovery (%) |        | Ave. Cumulative Nickel Recovery (%) |        | Ave. Cumulative Nickel Grade (%) |        | Klimpel Rate Constant K (min <sup>-1</sup> ) | Maximum Recovery R <sub>max</sub> (%) |
|                                       |            | 2 min   | 20 min | 2 min                              | 20 min | 2 min                               | 20 min | 2 min                            | 20 min |  |                                       |
| No Collector                          | 0          | 4.5   | 9.8    | 6.7                                | 17.4   | 11.3                                | 17.8   | 1.1                              | 0.8    | 1.1  | 18.1                                  |
| PAX                                   | 100        | 12.0  | 19.8   | 13.7                               | 25.9   | 48.7                                | 77.0   | 1.9                              | 1.9    | 1.1  | 80.1                                  |
| SIBX                                  | 100        | 11.5  | 20.8   | 12.7                               | 28.8   | 48.9                                | 79.1   | 2.1                              | 1.9    | 1.1  | 92.0                                  |
| IPETC                                 | 100        | 15.0  | 33.8   | 20.0                               | 96.4   | 61.8                                | 82.2   | 2.1                              | 1.2    | 1.9  | 83.2                                  |

#### 4.3.2 Cumulative Ni Grade versus Recovery

The cumulative nickel grades observed displayed the following trend: SIBX = PAX > IPETC > No collector (Figure 33). Their numerical values were 1.9%, 1.2% and 0.80% respectively (Table 9). Due to the frothing properties of IPETC, an increase in froth stability resulted, which furthermore caused inadequate water drainage. A lack of adequate water drainage may have caused a slow “drop-back” of entrained gangue minerals, which furthermore contributed to the low cumulative nickel grades (Figure 33).

A high water recovery was also observed, resulting in high entrainment of gangue minerals. This may have also contributed to the low grade achieved with IPETC. Smaller air bubbles were observed during the flotation of concentrates 3 and 4. It is known, that smaller bubbles do not readily rupture and cause poor drainage and a decrease in selectivity and grade (Wills and Napier-Munn, 2006). These concentrates also displayed a darker colour when compared to the first two concentrates. This could be due to a possible decrease in froth stability and collector selectivity during the later periods of flotation (conc.3-4). Much of the desired mineral may have also been collected at the earlier stages of flotation (conc.1-2).

There are differences in the chemistry between the collectors and the minerals on the surfaces of ore particles. It has been proposed that a metal-thiolate complex forms with the use of the xanthate collectors while chelating formations form with the use of the thionocarbamate. The sulphur atoms bonded to the cations in the xanthate collectors are assumed to bond and react with the sulphur atoms of pentlandite. This chemisorption reaction forms the metal-thiolate complex. With IPETC, the most important atoms for chelating agents are nitrogen, oxygen and sulphur (Fairthorne et al., 1997).

The sulphur atom in the functional group of IPETC is assumed to bond and react with the copper atoms in chalcopyrite (Fairthorne et al., 1997). For pentlandite however, the same mechanism is proposed to take place with the only difference being preferential selectivity to first chalcopyrite and then pentlandite. This mechanism for IPETC was however hard to distinguish, because the high water entrainment nullified any effects thereof.

Collectors with branched or short-chain hydrocarbon alkyl groups are generally more selective than their more powerful longer chained counterparts (Wills and Napier-Munn, 2006). This was not observed as the SIBX and PAX gave similar final cumulative nickel grades. There was however a significant difference in grade between PAX and IPETC.

The cumulative nickel recoveries decreased in the following order; IPETC > SIBX > PAX > no collector (Figure 33) and the numerical values were 82.2%, 79.1%, 77.0% and 17.8% respectively (Table 9). Although IPETC is accepted to be a selective collector (Sheridan et al., 2002), its selectivity may have been masked by its frothing properties. The consequent entrainment, thus increased the cumulative nickel and gangue recovery. There was however no significant difference in the recoveries obtained with SIBX, PAX or IPETC. Collectorless flotation resulted in the lowest cumulative nickel recovery as it relied on the natural floatability of the nickel minerals. As expected, a significant increase in nickel recovery was observed with the addition of collectors.

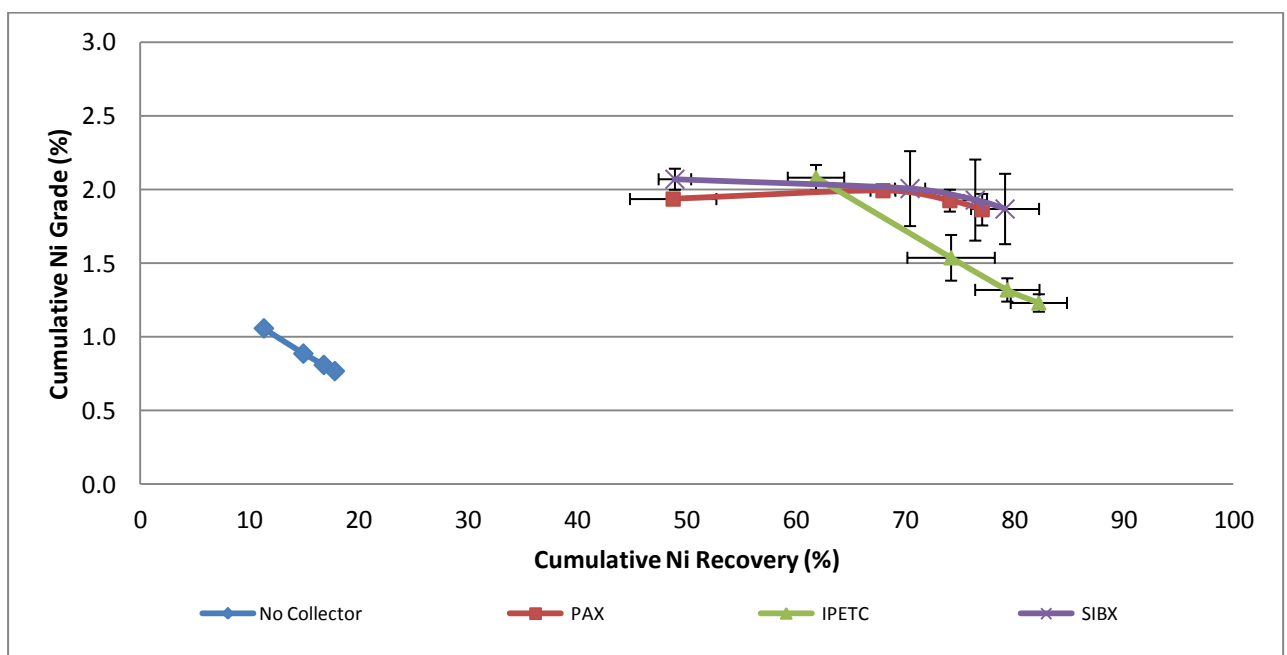


Figure 32: Cumulative nickel grade versus cumulative nickel recovery with PAX, SIBX and IPETC at a molar dosage of 1.3mmol/t

### 4.3.3 Cumulative Water and Mass Recoveries

The cumulative water recoveries decreased in the following order: IPETC > SIBX > PAX > no collector (Figure 34) and their numerical values were 96.4%, 28.8%, 25.9% and 17.4% respectively. The significantly higher cumulative water recovery obtained with IPETC may be attributed to the frothing properties of the thionocarbamates (Sheridan et al., 2002).

An increase in the froth stability, the formation of thick bubble lamellae and a consequent decrease in the rate of water drainage contributed to the high cumulative water and mass recovery. Also, fine particles in the lamellae tend to increase the froth height and stability, resulting in slow water drainage and increased entrainment (Aktas et al. 2008). This will further result in a decrease in nickel grade and an increase in water recovery.

The cumulative mass recoveries decreased in the following order: IPETC > SIBX > PAX > no collector and their corresponding numerical values were 33.8%, 20.8%, 19.8%, and 9.8% respectively (Figure 34). An increase in mass recovery may be attributed to an increase in the rate of adsorption of collectors on the surfaces of minerals and the entrainment of fine particles due to the high water recoveries (Aktas, Cilliers, & Banford, 2008). In the test done using the IPETC, it is unlikely that the first mechanism (increase in rate of adsorption) was the determining factor, since a high water recovery was achieved. The differences in the cumulative mass and water recoveries obtained with the xanthate collectors were not significant.

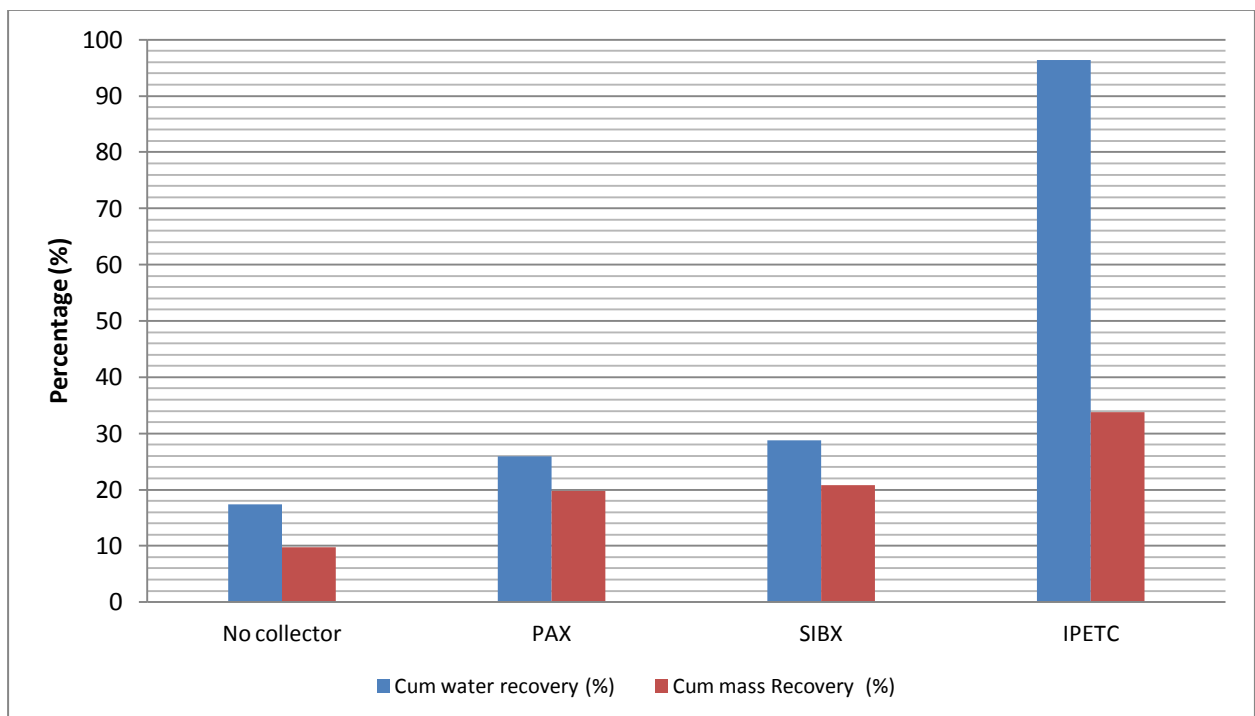


Figure 33: Cumulative mass and cumulative water recovery obtained with the use of PAX, SIBX and IPETC at a molar dosage of 1.3mmol/t

#### 4.3.4 Flotation Rates

The Klimpel rate constant model was used to distinguish between the different flotation rates. The Klimpel rate constants ( $k$  value) for the xanthate collectors and collectorless tests were all found to be  $1.1\text{min}^{-1}$ , while the Klimpel rate constant for IPETC was found to be  $1.9\text{min}^{-1}$  (Table 9). Collectors with longer hydrocarbon chains generally have lower adsorption rates on mineral surfaces and solubility (Wills and Napier-Munn, 2006).

The effects of the different hydrocarbon chains on collectors as well as the entrainment may account for the different rates of flotation observed. The rate of adsorption of IPETC on the mineral surfaces may have been greater than that of PAX and SIBX (Table 9). However, the high water recovery and resulting entrainment may have been the mechanism responsible for a significantly higher Klimpel rate constant. The Klimpel rate constants were similar for the xanthate collectors and the collectorless test (Table 9) i.e. Although the addition of xanthate collectors increased the grade and recovery of nickel they did not change the rate of flotation (Figure 35).

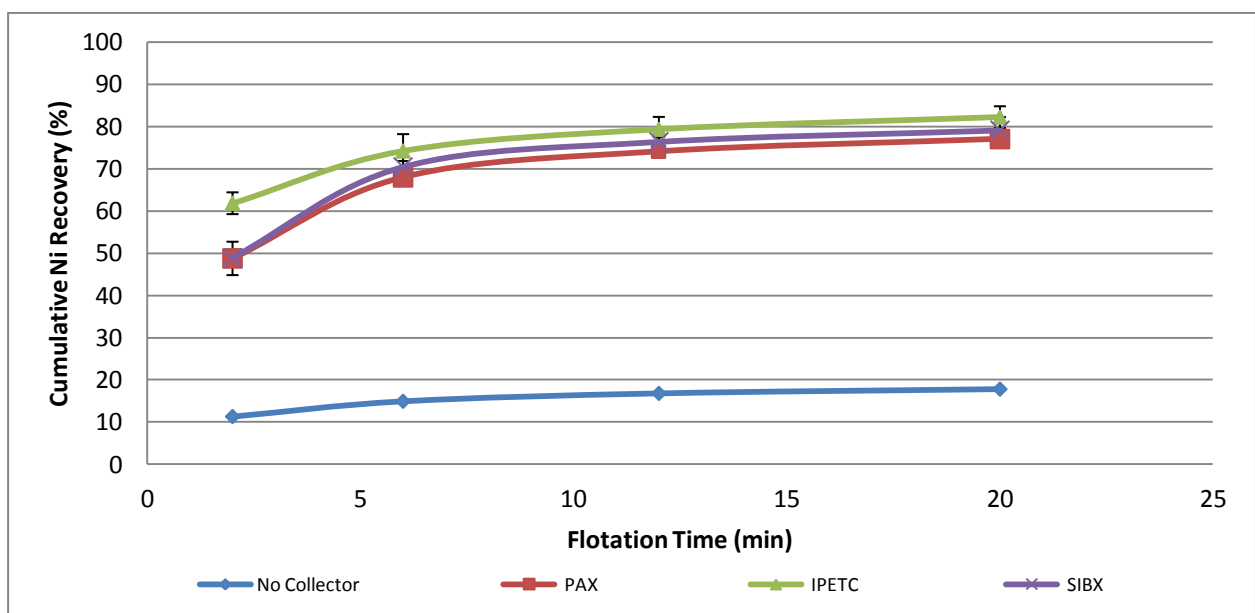


Figure 34: Cumulative nickel recovery versus time with the use of PAX, SIBX and IPETC at a molar dosage of  $1.3\text{mmol/t}$

## 4.4 Collector Mixtures at Frother Dosage 50g/t

### 4.4.1 Summary of Results

Table 10: Results summary for nickel using collector mixtures

| Nickel                                |            |   |        |                                    |        |                                     |        |                                  |        |  |                                       |
|---------------------------------------|------------|---|--------|------------------------------------|--------|-------------------------------------|--------|----------------------------------|--------|--|---------------------------------------|
| Collector (molar dosage of 1.3mmol/t) | Mole Ratio | Ave. Cumulative Concentrate Mass Recovery (%) |        | Ave. Cumulative Water Recovery (%) |        | Ave. Cumulative Nickel Recovery (%) |        | Ave. Cumulative Nickel Grade (%) |        | Klimpel Rate Constant K (min <sup>-1</sup> ) | Maximum Recovery R <sub>max</sub> (%) |
|                                       |            | 2 min   | 20 min | 2 min                              | 20 min | 2 min                               | 20 min | 2 min                            | 20 min |  |                                       |
| SIBX: IPETC                           | 95.5: 4.5  | 12.7  | 23.5   | 15.8                               | 39.9   | 50.8                                | 82.0   | 1.8                              | 1.6    | 1.1  | 85.7                                  |
| PAX: IPETC                            | 95.5: 4.5  | 12.3  | 23.1   | 14.9                               | 35.9   | 50.3                                | 84.5   | 1.9                              | 1.7    | 1.0  | 87.9                                  |
| PAX: IPETC                            | 90 : 10    | 11.3  | 22.1   | 13.0                               | 33.2   | 47.3                                | 80.2   | 2.0                              | 1.7    | 1.0  | 84.5                                  |
| PAX: IPETC                            | 85 : 15    | 8.7   | 22.3   | 9.3                                | 41.1   | 40.0                                | 80.3   | 2.2                              | 1.7    | 0.7  | 87.3                                  |
| PAX: IPETC                            | 80 : 20    | 11.7  | 25.0   | 15.5                               | 49.0   | 46.4                                | 82.6   | 2.0                              | 1.6    | 0.9  | 87.8                                  |
| PAX: SIBX                             | 50 : 50    | 11.0  | 20.2   | 12.3                               | 27.6   | 45.4                                | 78.7   | 2.0                              | 1.9    | 0.9  | 96.2                                  |

### 4.4.2 Cumulative Ni Grade versus Recovery

The cumulative nickel grades observed displayed the following trend: 50%PAX:50%SIBX > 95.5%PAX:4.5%IPETC = 90%PAX:10%IPETC = 85%PAX:15%IPETC > 80%PAX:20%IPETC = 95.5%SIBX:4.5%IPETC (Figure 36). Their numerical values were 1.9%, 1.7%, 1.7%, 1.7%, 1.6% and 1.6% respectively (Table 10).

Although the PAX:SIBX mixture gave a higher cumulative nickel grade compared to the other collector mixtures (Figure 36 and Table 10) the differences were not significant. The constituents of a collector mixture affect the recovery and it is accepted that the ratio of compatible collectors in a mixture may affect the grade. Consequently, a compatible mixture of collectors at an optimum ratio may improve both the concentrate grade and recovery (Buckely *et al.*, 2003).

The cumulative nickel recoveries decreased in the following order; 95.5%PAX:4.5%IPETC > 80%PAX:20%IPETC > 95.5%SIBX:4.5%IPETC > 85%PAX:15%IPETC > 90%PAX:10%IPETC > 50%PAX:50%SIBX (Figure 36) and the respective numerical values were 84.5%, 82.6%, 82.0%, 80.3%, 80.2% and 78.7% respectively (Table 10). Although there were differences in the cumulative recoveries for the tested mixtures, the differences proved to be insignificant compared to the industrial mixture (95.5%SIBX:4.5%IPETC). This was evident as the cumulative nickel recoveries for the tested mixtures of PAX:IPETC where within a 2% increase or decrease of the industrial mixture (Table 10)

The small increases in cumulative grades and recoveries may have been due to different rates of adsorption of the collectors in the mixtures onto the stronger and weaker sites of the mineral surfaces; thus resulting in more hydrophobic mineral surfaces (Bradshaw & O'Connor, 1994). These effects may have been at work as the industry mixture (95.5%SIBX:4.5%IPETC) and 80%PAX:20%IPETC gave the lowest cumulative nickel grade (1.6%) and was therefore the least selective mixture. It should however be noted that due to the strong frothing properties of IPETC, it was the more powerful phenomenon at work, resulting in the increases or decreases in the cumulative nickel grade and recovery.

Furthermore, the mixture 50%PAX:50%IPETC gave the lowest cumulative nickel recovery (78.7%), but gave the highest cumulative grade (1.9%). However, the industry collector mixture (95.5%SIBX:4.5%IPETC) gave encouraging results as it obtained the joint second highest cumulative grade (1.7%) and the highest cumulative nickel recovery (84.5%). However the phenomena on the surfaces of the minerals were obscured by the added water recovery due to the frothing properties of IPETC. Collector mixtures 95.5%PAX:4.5%IPETC, 90%PAX:10%IPETC, 85%PAX:15%IPETC were thus the three collector mixtures identified for dosage testing and evaluation based on their relatively constant performance with regards to nickel grade and recovery.

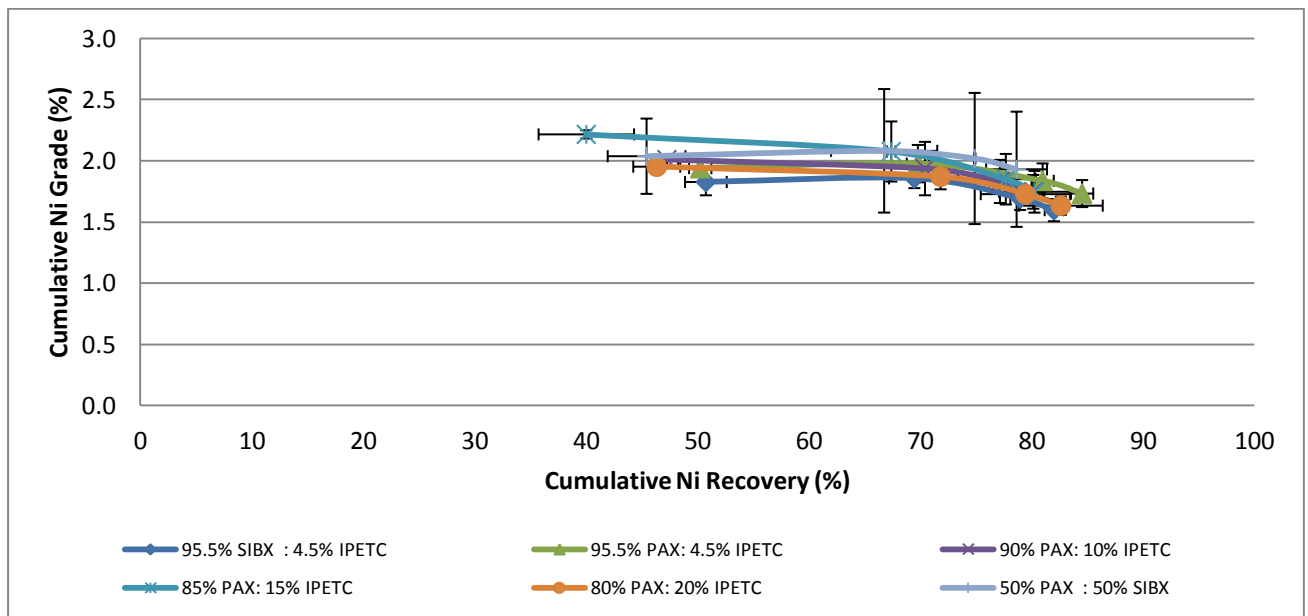


Figure 35: Cumulative nickel grade versus nickel recoveries of the industrial mixture, PAX with its mixtures with SIBX and 4.5, 10, 15 or 20% IPETC at a molar dosage of 1.3mmol/t

#### 4.4.3 Cumulative Water versus Mass Recovery

The cumulative water recoveries decreased in the following order: 80%PAX:20%IPETC > 85%PAX:15%IPETC > 95.5%SIBX:4.5%IPETC > 95.5%PAX:4.5%IPETC > 90%PAX:10%IPETC > 50%PAX:50%SIBX (Figure 37) and their cumulative water recoveries were 49%, 41.1%, 39.9%, 35.9%, 33.2% and 27.6% respectively (Table 10). It was observed that the mixture with the highest concentration of IPETC (80%PAX: 20%IPETC) gave the highest cumulative water recovery.

Due to IPETC having short-branched alkyl groups, it dissociated and adsorbed at a high rate, resulting in a relatively fast reaction on the mineral's surfaces. The induced frothing properties of the collector plus the addition of a frother, may have led to collector-frother interaction (Bradshaw et al., 1998). This could also lead to high froth stability, which could further increase the cumulative water recovery since inadequate draining would occur.

The cumulative water recoveries for the industry mixture (95.5%SIBX: 4.5%IPETC) and mixture 85%PAX: 15%IPETC gave cumulative water recoveries that were statistically significantly different. This suggested that the addition of PAX decreased the water recovery significantly. Furthermore, possible synergistic interactions both between the two collectors in the mixture as well as the collector mixture and frother could have occurred and due to the high dosages of collector, it may have caused unfavourable effects such as high water recoveries, with decreasing grades and recoveries (Bradshaw et al., 1998). Besides the fact that the two collector mixtures with the highest concentration of IPETC (85%PAX: 15%IPETC and 80%PAX:20%IPETC) gave the highest cumulative water recoveries, there were no discernible trends and no significant differences in water recovery for the other collector mixtures (except 85%PAX: 15%IPETC) compared to the industry mixture.

The cumulative mass recovery decreased in the following order; 80%PAX:20%IPETC > 95.5%SIBX:4.5%IPETC > 95.5%PAX:4.5%IPETC > 85%PAX:15%IPETC > 90%PAX:10%IPETC > 50%PAX:50%SIBX and their corresponding numerical values were 25.0%, 23.5%, 23.1%, 22.3%, 22.1% and 20.2% respectively (Table 10 and Figure 37). It was expected that with an increase in cumulative water recovery, an increase in cumulative mass recovery would follow. This would be due to the expected entrainment, which could increase the concentration of minerals in the froth layer.



The expected increases in mass recoveries as a result of the increases in water recoveries were not clearly discernible. No significant differences in the mass recoveries compared to the industry mixture were achieved. This suggests that the effects of entrainment were largely insignificant.

Generally it was observed that with an increase in the cumulative water recoveries for collector mixtures of IPETC and PAX, there were no differences in the cumulative mass recoveries greater than 5%. This suggested that the concentration of IPETC only has a significant influence on the water recovery and any additional minerals that may be recovered could be due to entrainment caused by a high water recovery.

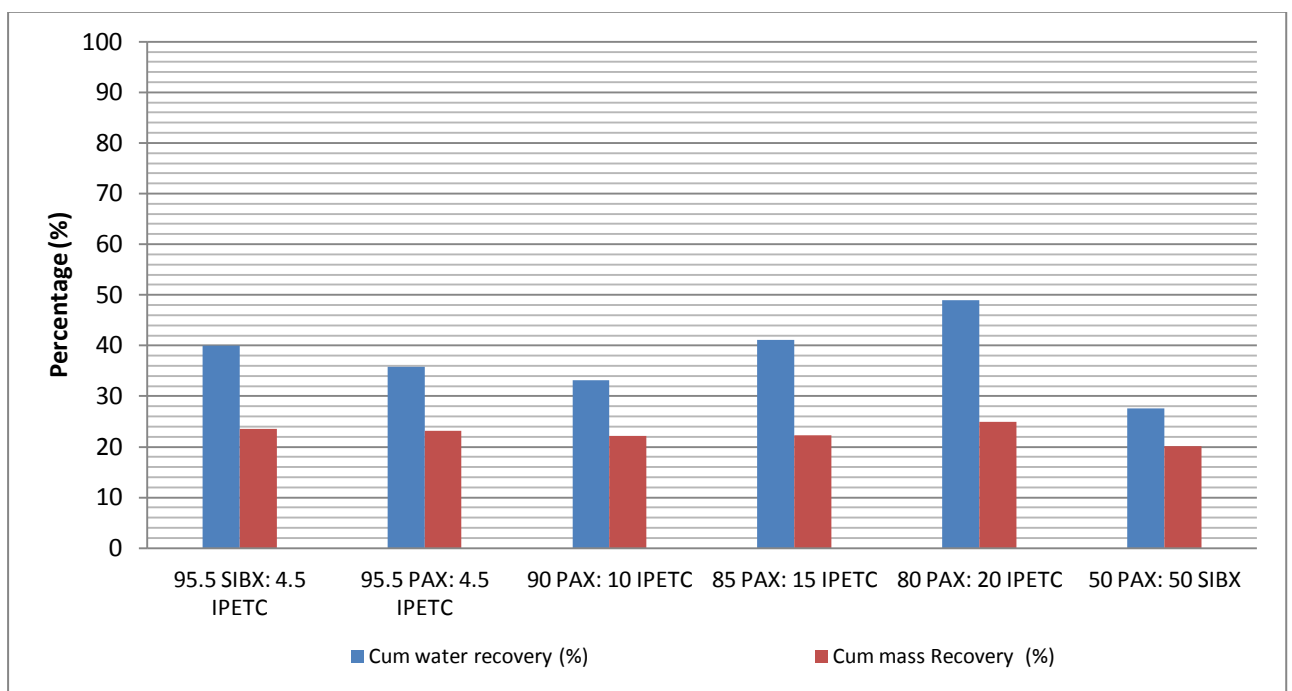


Figure 36: Cumulative mass versus cumulative water recoveries of the industrial mixture, PAX with its mixtures with SIBX and 4.5, 10, 15 as well as 20% IPETC at a molar dosage of 1.3mmol/t

#### 4.4.4 Flotation rates

The Klimpel rate constants were calculated (Table 10) to establish the rate of flotation for nickel for every mixture. The highest Klimpel rate constant was achieved with the use of the industrial mixture (95.5%SIBX: 4.5%IPETC). The Klimpel rate constants calculated displayed the following trend (Figure 38): 95.5%SIBX:4.5%IPETC > 95.5%PAX:4.5%IPETC = 90%PAX:10%IPETC > 80%PAX:20%IPETC = 50%PAX:50%SIBX > 85%PAX:15%IPETC. The

numerical values obtained for the Klimpel rate constants were 1.1, 1.0, 1.0, 0.9, 0.9 and 0.7min<sup>-1</sup> respectively (Table 10).

The trend in the flotation rate constant (Figure 38) suggest that there were no significant differences between the Klimpel rate constants achieved with the industry mixture, compared to the other mixtures. These findings further demonstrate that the use of the mixtures of collectors may increase the flotation rate (Bradshaw and O'Connor, 1994), but no significant improvements were observed. Pentlandite, although not as fast floating as chalcopyrite floats readily, however it has been reported that due to the presence of chalcopyrite and pyrrhotite, a delay in the froth flotation of pentlandite may occur. This is due to the depressive nature of chalcopyrite (Mishra et al., 2013).

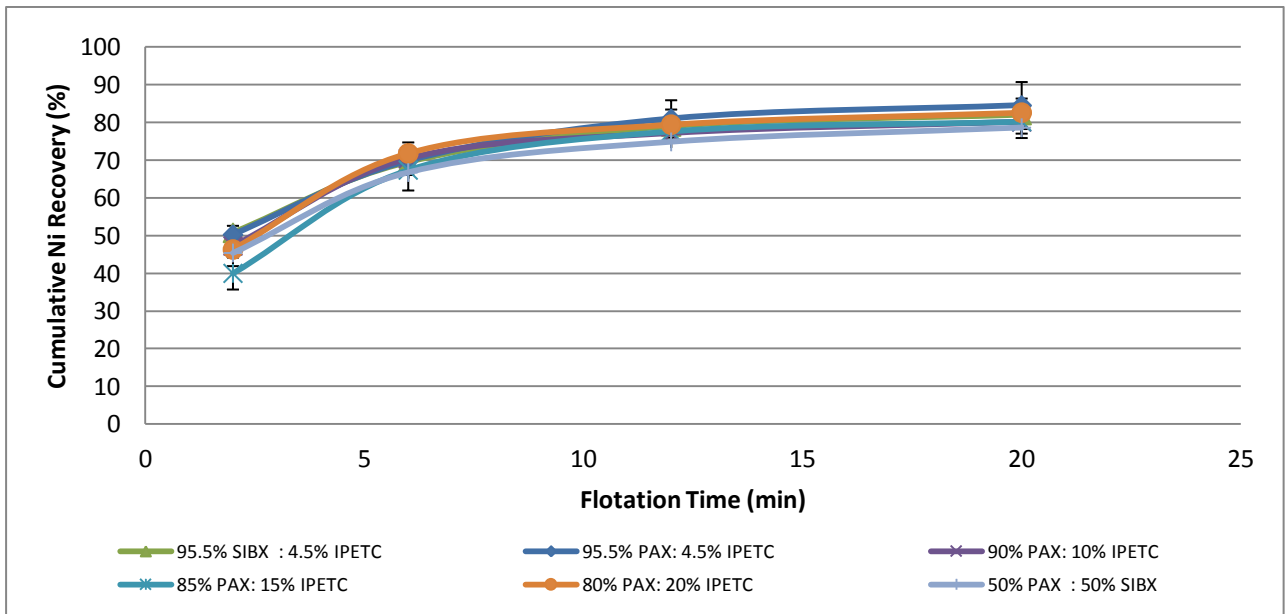


Figure 37: Cumulative nickel recoveries (%) of the industrial mixture, PAX with its mixtures with 4.5, 10, 15 as well as 20% IPETC at a molar dosage of 1.3mmol/t

## 4.5 Collector Mixtures at Decreased Dosages

### 4.5.1 Summary of Results

Table 11: Results summary for nickel using collector suites at decreased dosages

| Nickel                    |            |                                      |        |                           |        |                                     |        |                                  |        |                        |          |
|---------------------------|------------|--------------------------------------|--------|---------------------------|--------|-------------------------------------|--------|----------------------------------|--------|------------------------|----------|
| Collector                 | Mole Ratio | Ave. Cumulative Concentrate Mass (%) |        | Ave. Cumulative Water (%) |        | Ave. Cumulative Nickel Recovery (%) |        | Ave. Cumulative Nickel Grade (%) |        | K (min <sup>-1</sup> ) | Rmax (%) |
|                           |            | 2 min                                | 20 min | 2 min                     | 20 min | 2 min                               | 20 min | 2 min                            | 20 min |                        |          |
| PAX: IPETC (1.3mmol/t.)   | 95.5: 4.5  | 12.3                                 | 23.1   | 14.9                      | 35.9   | 50.3                                | 84.5   | 1.9                              | 1.7    | 1.0                    | 87.9     |
| PAX: IPETC (0.65mmol/t.)  | 95.5: 4.5  | 8.0                                  | 17.7   | 12.3                      | 32.8   | 46.1                                | 77.5   | 2.6                              | 2.0    | 1.0                    | 81.5     |
| PAX: IPETC (0.325mmol/t.) | 95.5: 4.5  | 7.5                                  | 18.1   | 7.0                       | 27.9   | 48.0                                | 76.2   | 3.3                              | 2.1    | 1.1                    | 80.0     |
| PAX: IPETC (1.3mmol/t.)   | 90 : 10    | 11.3                                 | 22.1   | 13.0                      | 33.2   | 47.3                                | 80.2   | 2.0                              | 1.7    | 1.0                    | 84.5     |
| PAX: IPETC (0.65mmol/t.)  | 90 : 10    | 10.6                                 | 20.8   | 9.8                       | 33.7   | 42.6                                | 69.6   | 2.2                              | 1.6    | 1.1                    | 72.8     |
| PAX: IPETC (0.325mmol/t.) | 90 : 10    | 9.9                                  | 20.2   | 9.9                       | 30.7   | 55.4                                | 77.8   | 2.9                              | 1.9    | 1.5                    | 80.2     |
| PAX: IPETC (1.3mmol/t.)   | 85 : 15    | 8.7                                  | 22.3   | 9.3                       | 41.1   | 40.0                                | 80.3   | 2.2                              | 1.7    | 0.7                    | 87.3     |
| PAX: IPETC (0.65mmol/t.)  | 85 : 15    | 9.7                                  | 22.3   | 10.2                      | 39.5   | 45.5                                | 80.2   | 2.2                              | 1.7    | 0.9                    | 85.4     |
| PAX: IPETC (0.325mmol/t.) | 85 : 15    | 9.0                                  | 21.3   | 8.7                       | 35.1   | 56.2                                | 79.6   | 3.1                              | 1.8    | 1.5                    | 81.5     |

### 4.5.2 Comparison of Nickel Grade versus Recovery

#### 4.5.2.1.1 Nickel grades and recoveries obtained with mixture 95.5%PAX:4.5%IPETC

The cumulative grades for collector mixture 95.5%PAX:4.5%IPETC at decreased molar dosages of 1.3, 0.65 and 0.325mmol/ton, gave cumulative nickel grades of 1.7%, 2.0% and 2.1% respectively (Table 11). It was observed that there was a general increase in cumulative nickel grade with a decrease in collector dosage (Figure 39). These increases in cumulative nickel grade however proved to be statistically insignificant (Appendix G).

The cumulative nickel recovery for collector mixture 95.5%PAX:4.5%IPETC at the same molar dosages showed that the rate and cumulative nickel recovery decreased in the following dosage order (Figure 41): 1.3, 0.65 and 0.325mmol/ton. The cumulative recoveries obtained were 84.5, 77.5 and 76.2% respectively (Table 11). These decreases in nickel recovery were not statistically significant (Appendix H).

The increase in grade generally came at the expense of nickel recovery. This suggested that with a decrease in dosage there may have been an improvement in selectivity (Bradshaw et al., 1998). Since the major collector in the mixture is PAX, the proposed mechanism suggests that PAX being the “stronger” collector adsorbs onto the “weaker sites” of the minerals surfaces and IPETC adsorbs on the “stronger sites”. However, both PAX and IPETC attaches to the sulphur atom in pentlandite through the sulphur anion.

The increase in cumulative nickel grade and the accompanying decrease in cumulative nickel recovery, may be attributed to possible synergism of the collector mixture or an excess of collector in the pulp. The surplus of collector could possibly decrease the selectivity as a multi-layer of collector could have formed on the minerals’ surfaces instead of a mono-layer (Bradshaw et al., 1998). With the larger dosages, possible interactions between the collector and frother could have occurred resulting in a possible decrease in entrainment. This may have decreased the amount of gangue recovered and increased the grade.

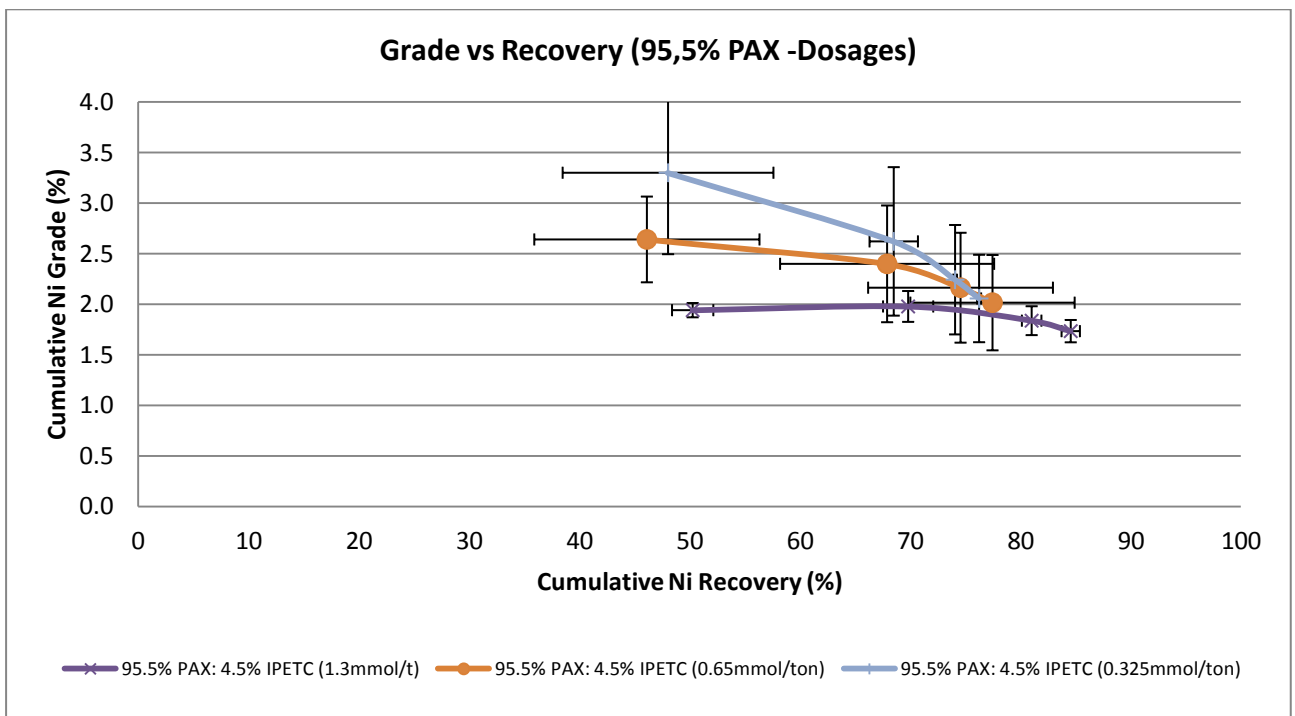


Figure 38: Cumulative nickel grade versus nickel recovery with PAX and its mixtures with 4.5 % IPETC at molar dosages of 1.3, 0.65 and 0.325mmol/t

#### 4.5.2.2 Nickel grades and recoveries obtained with mixture 90%PAX:10%IPETC

The cumulative grades obtained with the collector mixture 90%PAX:10%IPETC at molar dosages of 0.65, 1.3 and 0.325mmol/ton (Figure 40) were 1.7%, 1.6% and 1.9% respectively (Table 11). Unlike the collector mixture 95.5%PAX:4.5%IPETC, there was no discernible trend.

The increase in the concentration of IPETC appears to have caused a general decrease in cumulative nickel grade in comparison to the cumulative grades obtained with collector mixture 95.5%PAX:4.5%IPETC. These differences in grade were also found not to be statistically significant (Appendix G).

The cumulative nickel recovery obtained with the collector mixture 90%PAX:10%IPETC at molar dosages of 1.3, 0.325 and 0.65mmol/ton (Figure 41) were 80.2%, 77.8% and 69.6% respectively (Table 11). It was expected that with an increase in the concentration of IPETC, a higher mass recovery would result due to entrainment, contributing to a higher cumulative nickel recovery at the expense of grade. However there was no statistically significant change in the grade.

It was also expected that an increase in selectivity would result with the use of a lower dosage of the collector mixture, since the probability of multilayer collector formation would decrease, due to limited amount of collector available for adsorption. It is known, that the benefits of using collector mixtures include increases in selectivity at reduced collector dosages due to possible synergism (Mcfadzean et al., 2012).

With the increase in the concentration of IPETC in the collector mixtures, it was expected that a higher cumulative nickel recovery would result due to it being more selective than PAX. Also expected was that the lower dosages of the collector mixture would affect the surface chemistry on pentlandite; hence affecting both the grade and recovery.

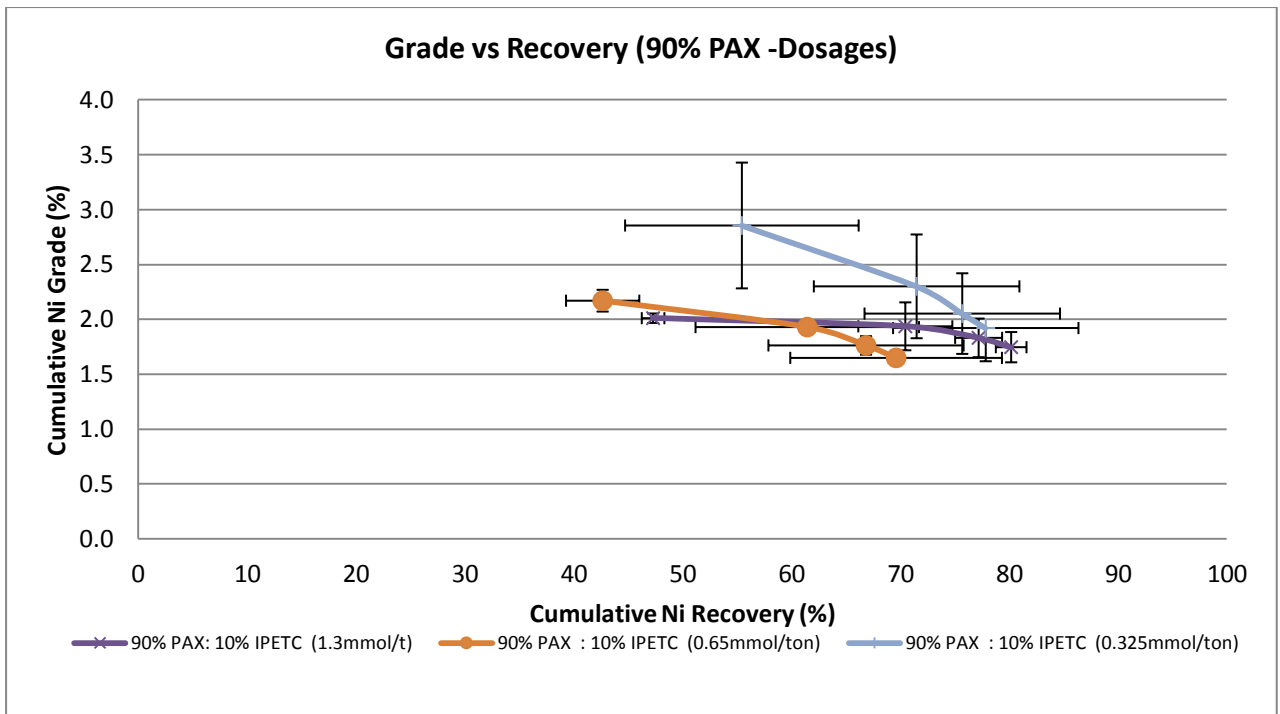


Figure 39: Cumulative nickel grade versus nickel recovery with PAX and its mixtures with 10% IPETC at molar dosages of 1.3, 0.65 and 0.325mmol/t

#### 4.5.2.3 Nickel grades and recoveries obtained with mixture 85%PAX:15%IPETC

The final cumulative grades obtained with the collector mixture 85%PAX:15%IPETC were not significantly different for dosages the 0.65 and 1.30 mmol/ton. The only increase in cumulative grade occurred at 0.325mmol/ton (Figure 41). The corresponding numerical values were 1.7%, 1.7% and 1.8% respectively (Table 11). Unlike the collector mixture 95.5%PAX:4.5%IPETC, there were no discernible trends that could be followed. It appears less evident that the increase in the concentration of IPETC caused a general decrease in cumulative nickel grade when compared to the cumulative grades obtained with collector mixture 90%PAX:10%IPETC at different dosages.

The final cumulative nickel recoveries for collector mixture 85%PAX:15%IPETC at molar dosages of 1.3, 0.65 and 0.325mmol/ton (Figure 41) were 80.3%, 80.2% and 79.6% respectively (Table 11). The differences among the final cumulative nickel recoveries and grades were not significant. No significant benefits resulted from the increased collector dosage of 1.3mmol/ton.

An increase in grade from 1.7% to 1.8% with the use of dosage 0.325mmol/ton came at a relatively small expense of recovery. The cumulative grades and recoveries of this collector mixture at dosages 1.3 and 0.65mmol/ton indicated that there were no significant differences in the performance with a decrease in the collector dosage. As with the other mixtures the lowest dosage of this collector mixture gave the highest cumulative nickel grade with a relatively small decrease in cumulative nickel recovery. Although the differences in final cumulative nickel grades and recoveries were insignificant for the tested mixtures at different dosages, it was however clear that a decrease in collector dosage to a molar dosage of 0.325mmol/ton of the industry mixture could be used to save on collector without compromising the flotation grade or recovery.

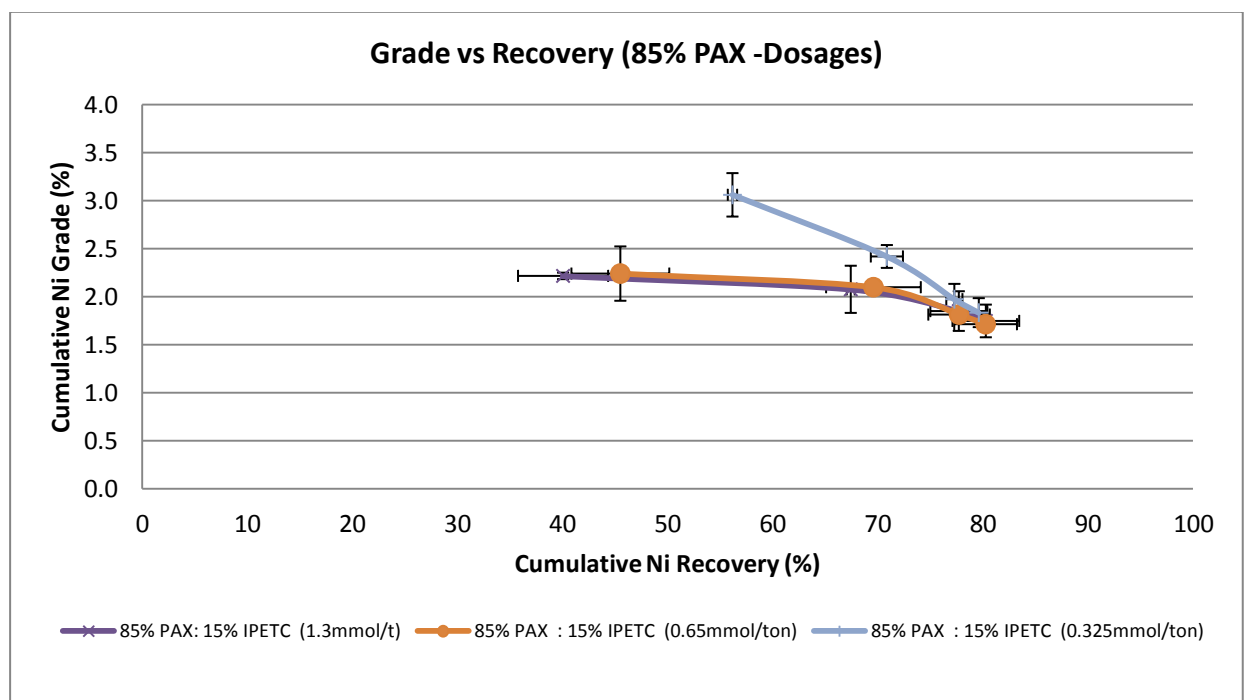


Figure 40: Cumulative nickel grade versus cumulative nickel recovery with PAX and its mixtures with 15% IPETC at molar dosages of 1.3, 0.65 and 0.325mmol/t

#### 4.6 Comparison of Water versus Mass Recovery

The cumulative water recoveries for collector mixture 95.5%PAX:4.5%IPETC for 1.3, 0.65 and 0.325mmol/ton were 35.9, 32.8 and 27.9% respectively. The cumulative water recoveries for collector mixture 90%PAX:10%IPETC for 1.3, 0.65 and 0.325mmol/ton were 33.2, 33.7 and 30.7% respectively. The cumulative water recoveries for collector mixture 85%PAX:15%IPETC for 1.3, 0.65 and 0.325mmol/ton were 41.1, 39.5 and 35.1% respectively (Figure 42 and Table 11). It was generally expected that with an increase in the IPETC concentration in the flotation cell, a higher cumulative water recovery would result. The expected increase in water recovery would be primarily due to the frothing properties of IPETC at a high collector dosage.

Generally it was observed that the higher the concentration of IPETC in the collector mixtures, the higher the cumulative water recovery. This was evident with the use of different dosages of collector mixtures 85%PAX:15%IPETC and 95.5%PAX:4.5%IPETC. However, with the collector mixture 90%PAX:10%IPETC at different dosages, it was observed that there was an insignificant difference in water recovery between dosages 1.3 and 0.65mmol/ton. At 0.325mmol/ton the mixture gave a decrease in water recovery, but still statistically insignificant. There was however a significant decrease in the cumulative water recovery with mixtures 85%PAX:15%IPETC at molar dosage of 0.325mmol/t and 95.5%PAX:4.5%IPETC at molar dosage of 1.3mmol/t of the industry mixture.

The cumulative mass recoveries for collector mixture 95.5%PAX:4.5%IPETC for 1.3, 0.65 and 0.325mmol/ton were 23.1%, 17.7% and 18.1% respectively. The cumulative mass recoveries for collector mixture 90%PAX:10%IPETC for 1.3, 0.65 and 0.325mmol/ton were 22.1%, 20.8% and 20.2% respectively. The cumulative mass recoveries for collector mixture 85%PAX:15%IPETC for 1.3, 0.65 and 0.325mmol/ton were 22.3%, 22.3% and 21.3% respectively (Figure 42 and Table 11). There were no significant differences compared to the industry mixture.



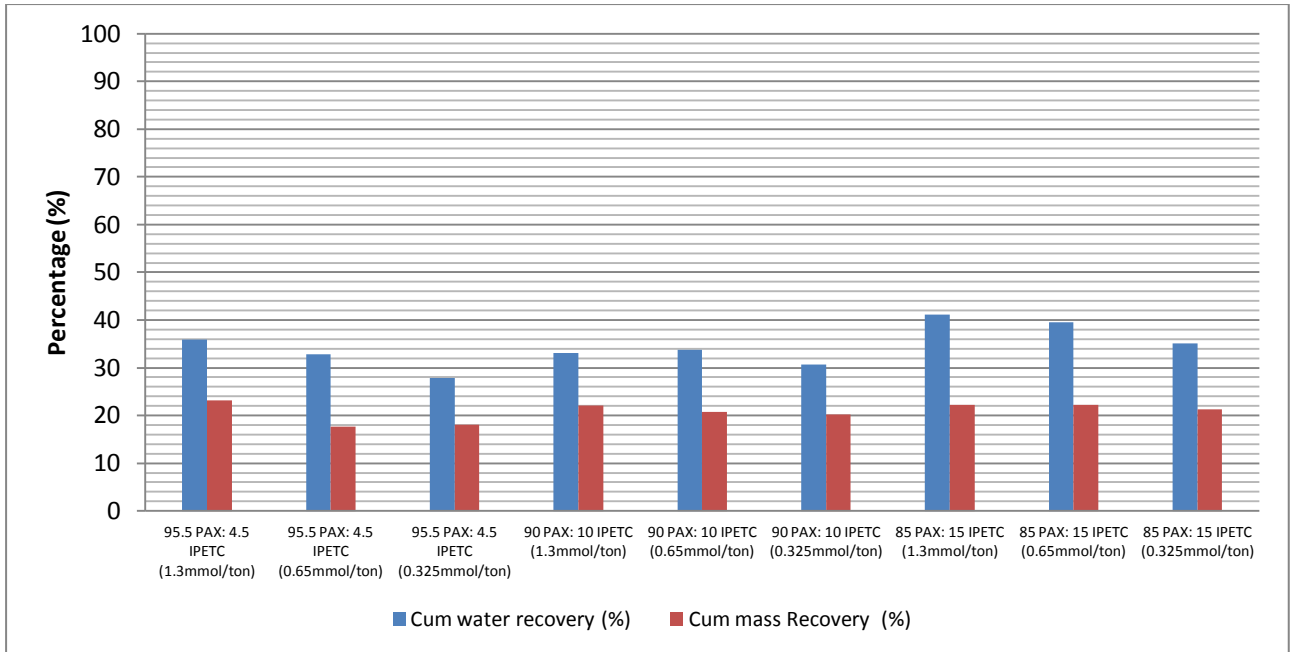


Figure 41: Final Cumulative mass versus cumulative water recoveries with IPETC and its mixtures with 95.5, 90, 85 and 80 mol% PAX at molar dosages of 1.3, 0.65 and 0.325mmol/t

## 4.7 Comparison of Flotation rates

### 4.7.1 Flotation rates obtain with mixture 95.5%PAX:4.5%IPETC

The Klimpel rate constants calculated for this collector mixture at different dosages indicated that the collector dosage at 0.325mmol/ton gave the highest Klimpel rate constant. This suggests that at the lowest collector dosage (0.325mmol/ton), the rate of nickel flotation was higher ( $1.1 \text{ min}^{-1}$ ) with the use of collector mixture 95.5%PAX:4.5%IPETC, in comparison to 1.3mmol/ton ( $1.0 \text{ min}^{-1}$ ) and 0.65mmol/ton ( $1.0 \text{ min}^{-1}$ ). This may have been due to improved adsorption and possible synergistic effects, resulting from a lower dosage as only a monolayer of collector may have formed on the minerals' surfaces. The differences were not statistically significant and no clear conclusion could be drawn (Figure 43).

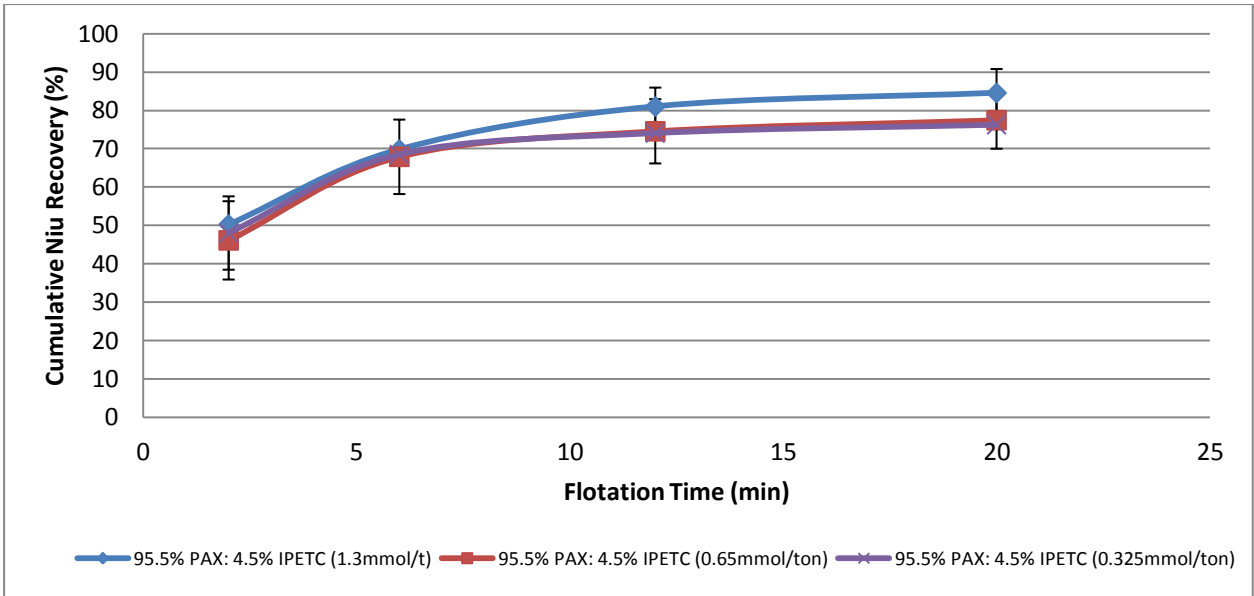


Figure 42: Percentage cumulative nickel recovery obtained with the use of PAX and its mixtures with 4.5 mol% at molar dosages of 1.3, 0.65 and 0.325mmol/t

#### 4.7.2 Flotation rate obtained with mixture 90%PAX:10%IPETC

The calculated Klimpel rate constants for this collector mixture at different dosages increased with a decrease in collector dosage as follows: 1.0, 1.1 and 1.5  $\text{min}^{-1}$  and the dosages were 1.3, 0.65, and 0.325mmol/ton respectively. As with the mixture 95.5%PAX:4.5%IPETC the lowest dosage gave the highest Klimpel rate constant. This further demonstrated the benefit of using collector mixtures at lower dosages (Figure 44).

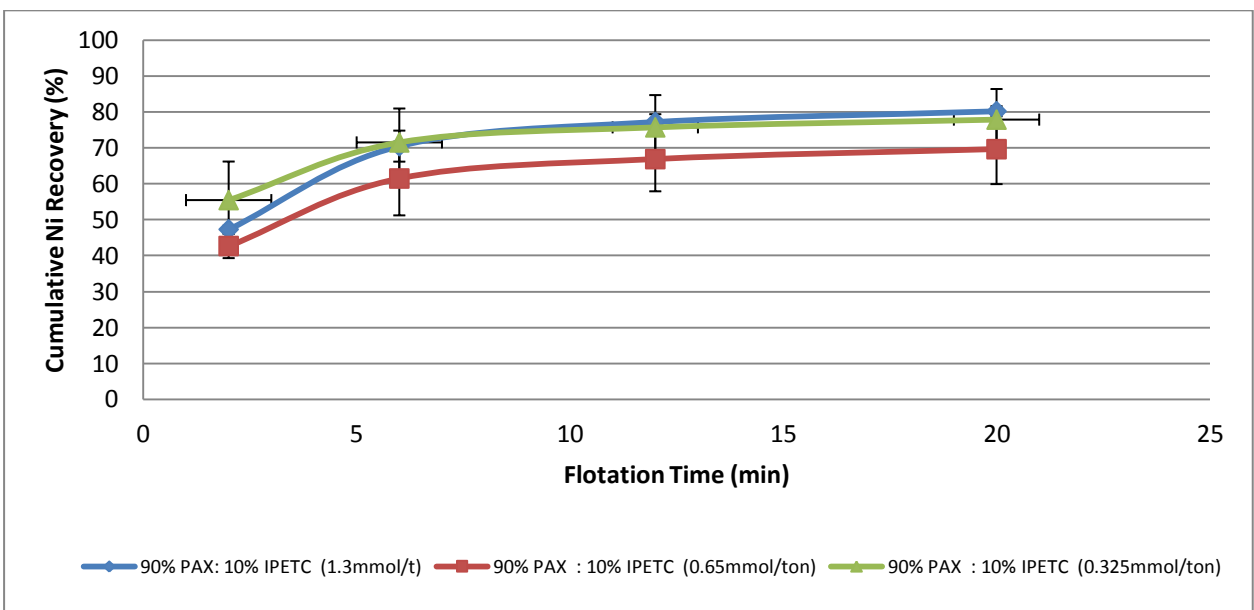


Figure 43: Percentage cumulative nickel recovery obtained with the use of PAX and its mixture with 15 mol% at molar dosages of 1.3, 0.65 and 0.325mmol/t

#### 4.7.3 Flotation rate obtained with mixture mixture 85%PAX:15%IPETC

The calculated Klimpel rate constants for this collector mixture at different dosages increased with a decrease in collector dosage as follows: 0.7, 0.9 and 1.5  $\text{min}^{-1}$  and the dosages were 1.3, 0.65, and 0.325mmol/ton respectively. Collector mixture 85%PAX:15%IPETC performs similar to mixture 90%PAX:10%IPETC with regards to klimpel rate constants in response to the different dosages. Due to the relatively small differences in Klimpel rate constants, this suggested that the rate of surface adsorption of the collectors on the minerals surfaces are all happening at the same rate or the effects of entrainment are similar (Figure 45).

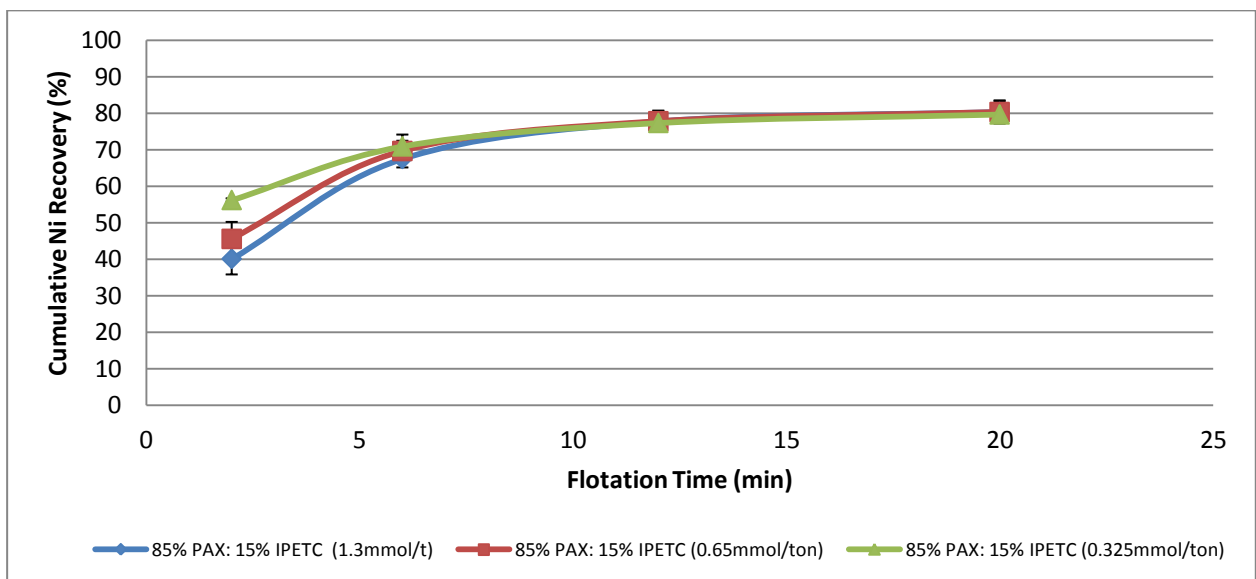


Figure 44: Percentage cumulative nickel recovery obtained with the use of PAX and its mixture with 15 mol% IPETC at molar dosages of 1.3, 0.65 and 0.325mmol/t

## 4.8 Sulphur Grade versus Recovery

As the Nkomati deposit is a predominantly sulphide deposit, with the major base metal sulphides being chalcopyrite and pentlandite, it is imperative to evaluate and confirm the recovery of base metal sulphides as well as gangue sulphides (pyrite and pyrrhotite) which may report to the concentrates. To this end, a cumulative sulphur grade versus cumulative recovery curve was prepared for the concentrates of the various collector mixtures.

As expected the use of no collector yielded the lowest final cumulative sulphur grade and recovery, since natural hydrophobicity and entrainment were the assumed major mechanisms for mineral recovery (Figure 46). However, when compared to the grade and recovery plots of nickel, the grades were significantly higher. This is due to the fact that the sulphur analysed is all sulphur present in each of the concentrates. This included chalcopyrite, pentlandite, pyrite and pyrrhotite that were not depressed or reported to the concentrates via entrainment as these were the only sulphur-containing minerals according to the mineralogy.

It is also evident that the PAX yielded the lowest cumulative sulphur grade and recovery, whereas collector mixture 95.5%PAX:4.5%IPETC at 0.325mmol/t yielded the highest cumulative sulphur grade (Figure 46). The industrial mixture (95.5%PAX:4.5%IPETC) yielded the highest cumulative sulphur recovery. This could have been mainly attributed to the entrainment of sulphide gangue minerals as well as the base metal sulphides since the addition of IPETC increases the water recovery and froth stability.

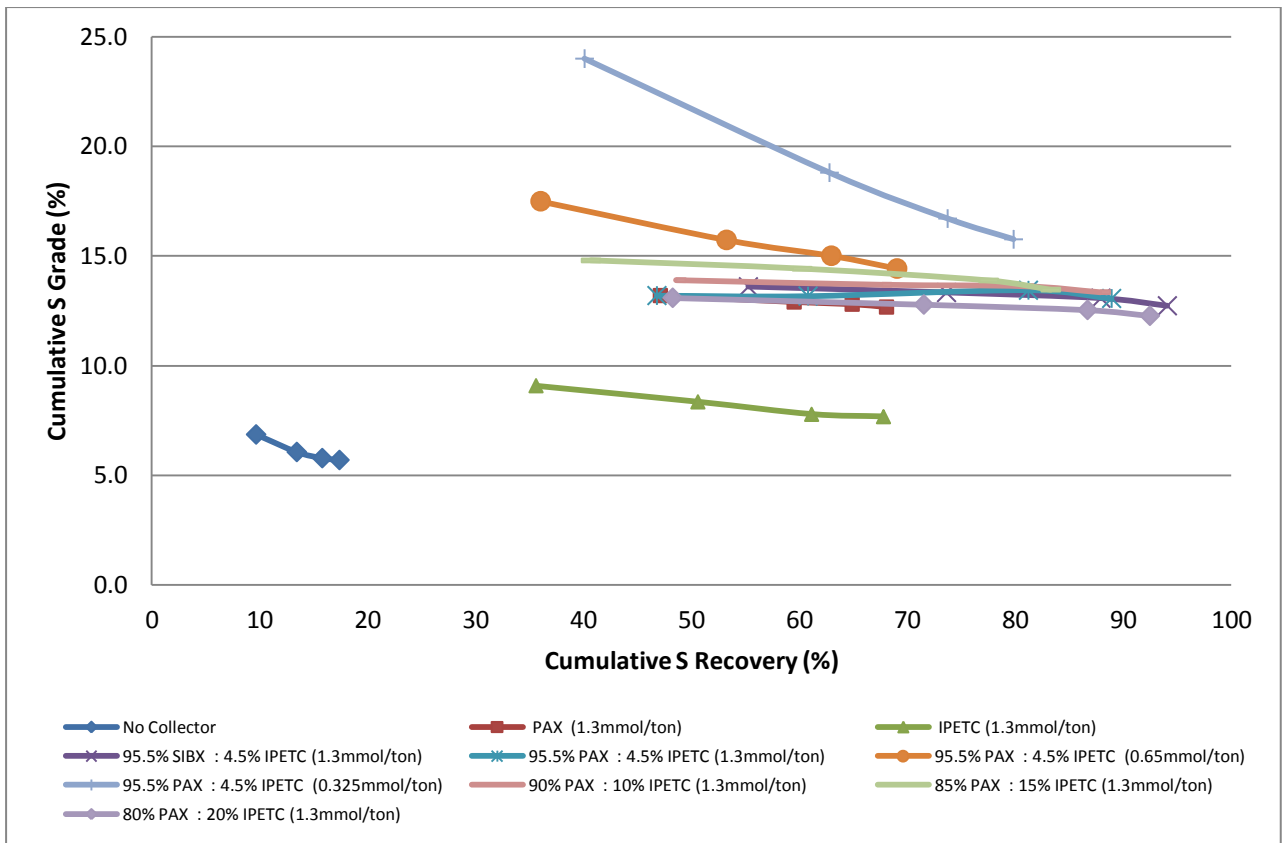


Figure 45: Cumulative Sulphur grade vs cumulative recovery curves for the industrial mixture (95.5%SIBX: 4.5%IPETC), pure IPETC, PAX and its mixtures with 4.5, 10, 15 or 20 mol% IPETC at molar dosage 1.3mmol/t and decreased dosages for mixture 95.5%PAX: 4.5%IPETC.

#### 4.8 Concentrate Particle Size Distribution

Since particle size is important in froth flotation as it is an indication of the interaction between the chemical reagents (collectors, frothers, etc) and minerals as well as the froth stability (Aktas et al., 2008). Particle size ranges may also be used to classify a particle size distribution based on the amount of material reporting to a specified size fraction. Nyambayo (2014) summarised the classification of particle sizes in the following table:

Table 12: Particle size classification (Nyambayo, 2014)

| Particle size range ( $\mu\text{m}$ ) | Description |
|---------------------------------------|-------------|
| -10                                   | ultrafine   |
| -25 +10                               | fine        |
| +25, -75                              | medium      |
| +75                                   | coarse      |

Much like the analysis and validation of the particle size distribution for the randomly chosen feed samples, the particle size distributions of the concentrates were analysed for various collector mixtures. The particle size ranges were plotted for particles between sizes 1-100 $\mu\text{m}$ . The particle size distributions for concentrate 1 and 2 (Figure 47 and 48), indicated that the highest volume % of particles are in the medium to fine regions (Table 12). This was expected and is consistent with the finding of Nyambayo (2014).

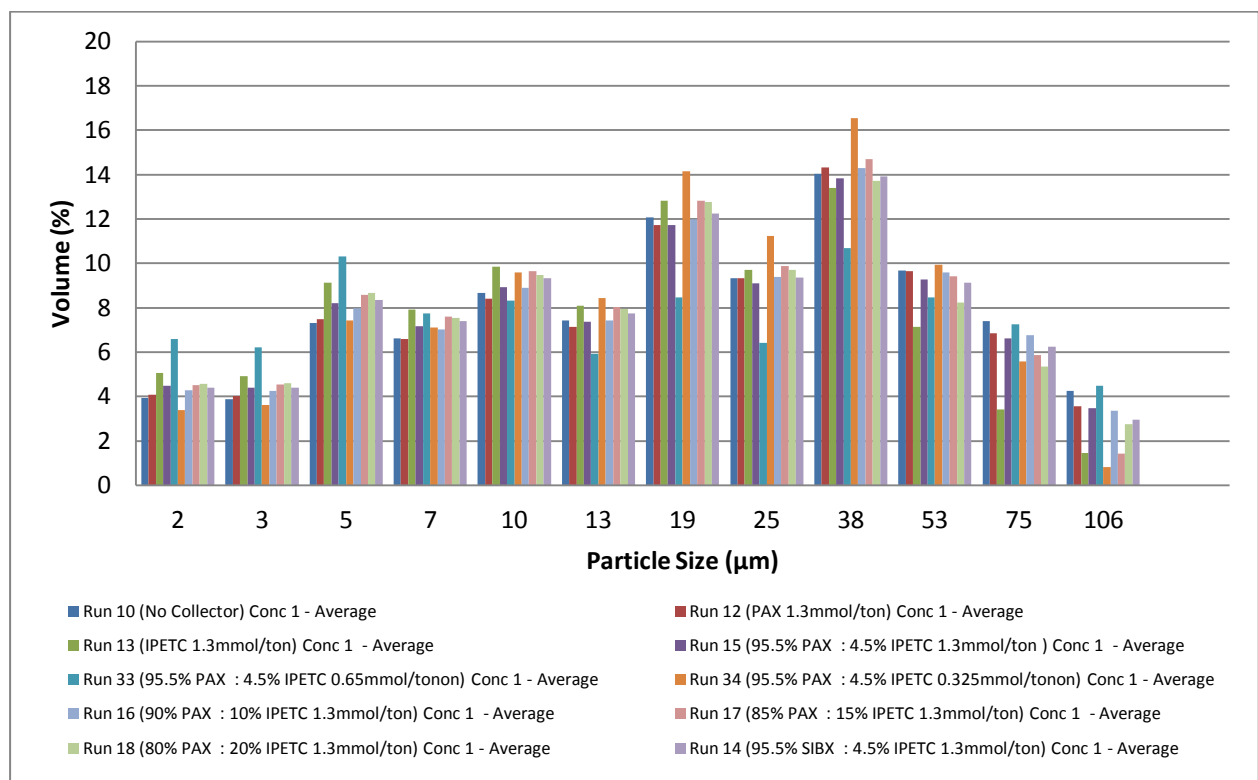


Figure 46: Particle size particle distributions from 1-100 $\mu\text{m}$  for the first concentrate (after 2 minutes)

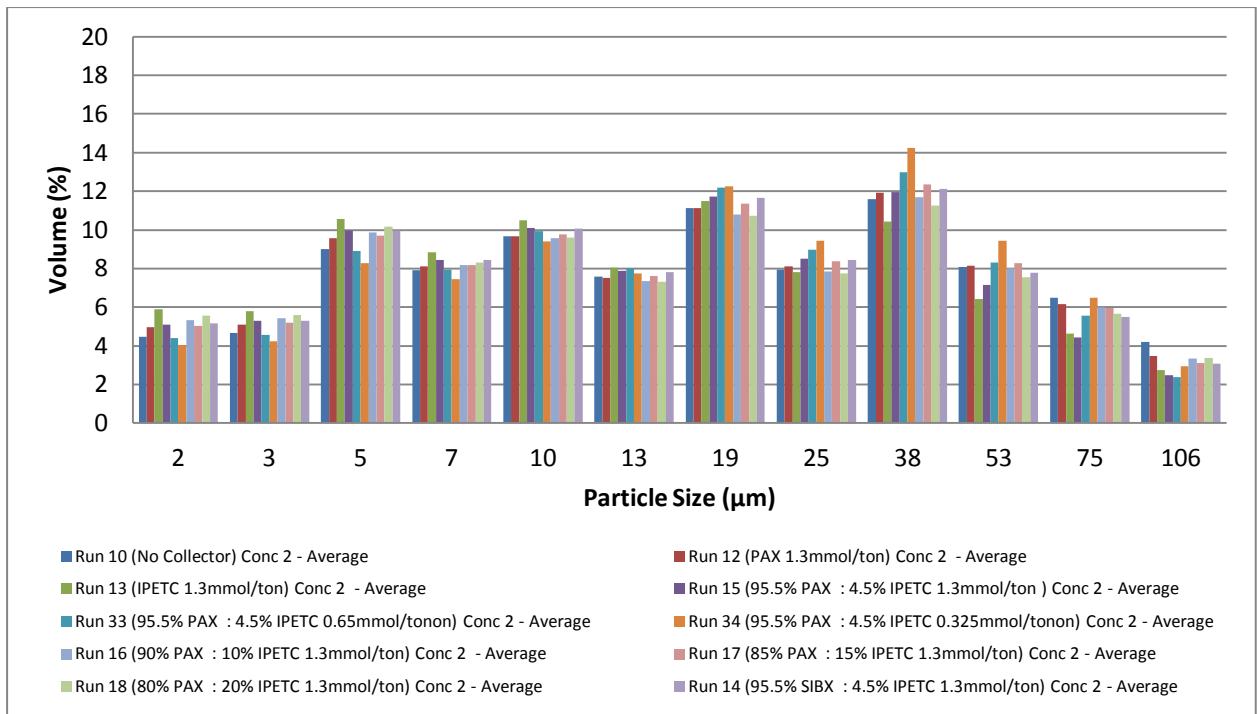


Figure 47: Particle size particle distribution from 1-100 $\mu$ m for the second concentrate (after 6 minutes)

As a base of comparison, the feed particle size distribution for a  $P_{80} - 75\mu$ m grind was a medium to fine ground ore and therefore should reflect this in the particle size distribution of the concentrate. When evaluating the trends for concentrates 2 (Figure 48), it is evident that similarly to all of the other four graphs, all the collector mixtures followed the same trend. However, with concentrate 2 there appears to be a decrease in the peaks within the medium to fine range and no change in peak within the ultrafine region.

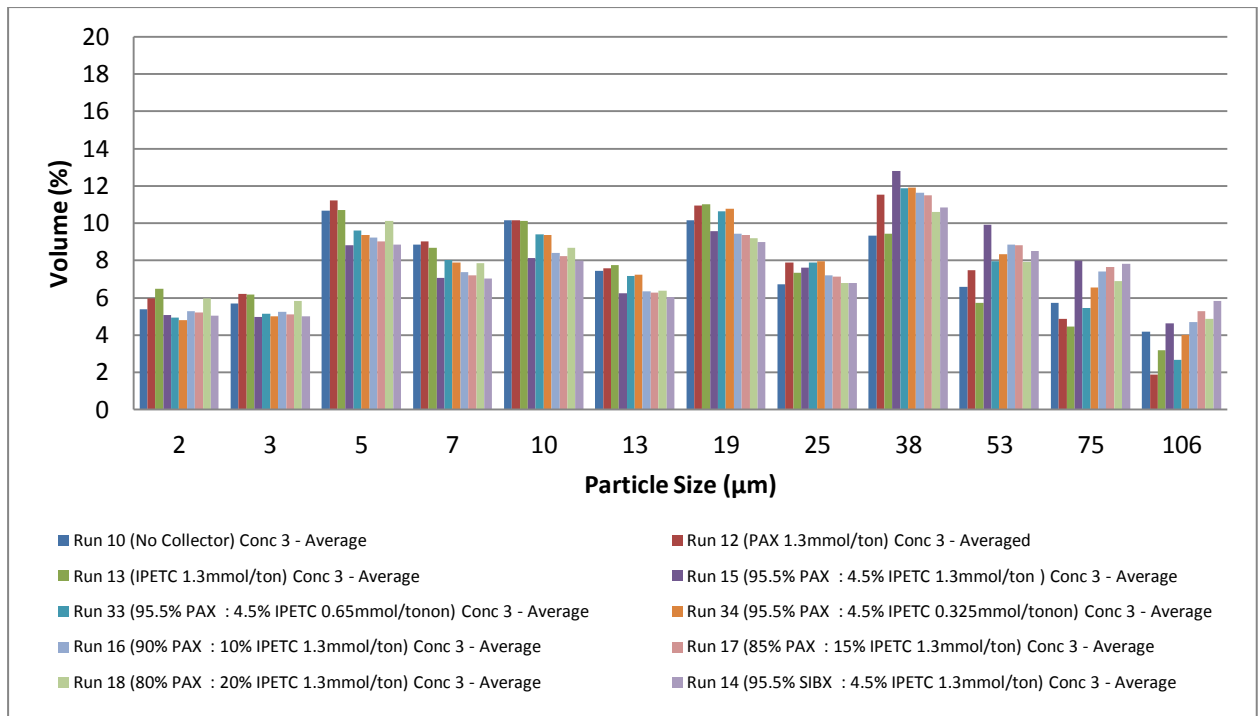


Figure 48: Particle size particle distribution from 1-100µm for third concentrate (after 12 minutes)

The particle size distribution for concentrate 3 follows the same trends for the medium to fine region as that of concentrate 2. There is however a slight decrease in the trend highs when compared to Concentrate 1 and 2 (Figure 47 and 48). This possibly indicates that initially most of the coarse material had been recovered and through the duration of the flotation, the coarse desired particles were recovered to the extent of depletion with the medium, fine and ultra fine sized minerals becoming the predominant particle size class being recovered.



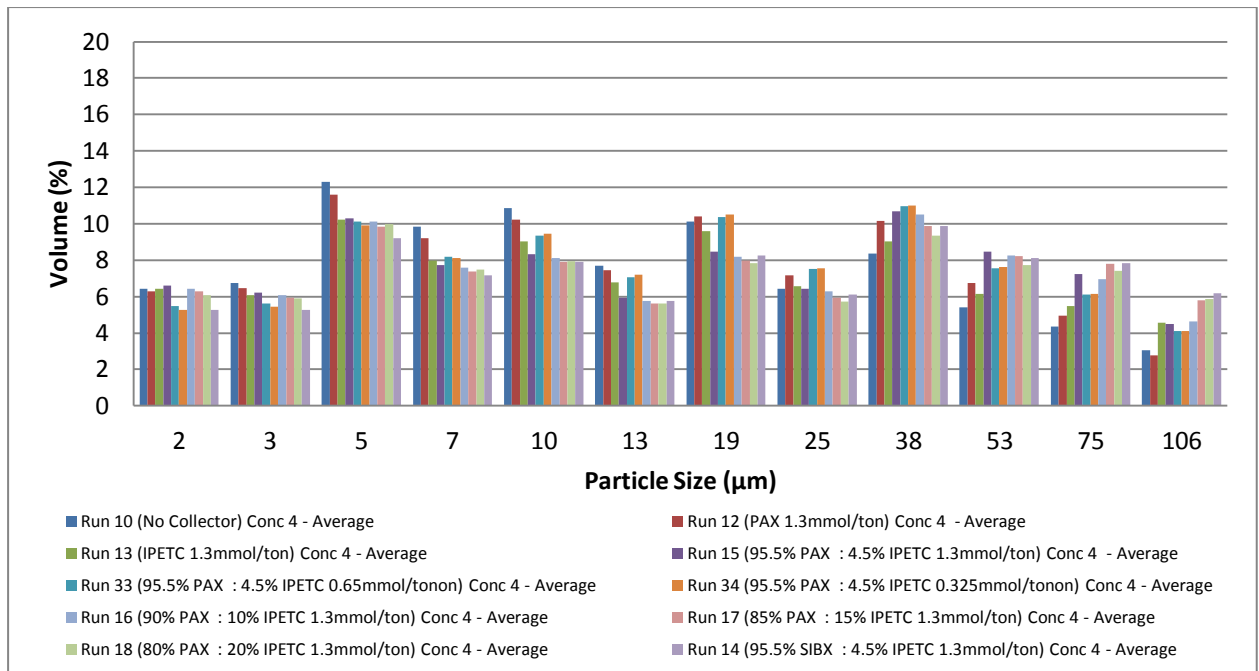


Figure 49: Particle size particle distribution from 1-100μm for the fourth concentrate (after 20 minutes)

The final concentrate indicates a peak high in the ultra fine particle size region, which is totally different when compared to the previous concentrate graphs. This clearly indicates that the predominant particles being recovered are the ultra fine to fine. It is also evident that initially the fine particles had a high recovery. This was then followed by a decrease in fine particle recovery. This could be due to the fact that most of the coarse, medium and fine minerals have been recovered by the third concentrate.

Although there are no clear discernible trends, it is however clear that initially (conc 1-3) the peaks are skewed to the right between 19-38μm indicating the high recovery of coarser to medium minerals. The last concentrate gave a curve that is skewed to the left between 2-10μm, which indicates a high recovery of ultrafine to fine minerals. As expected the collector mixtures generally resulted in a higher recovery over the various particle sizes compared to the pure collectors.

## CHAPTER 5

### 5.1 Conclusions

The aim of this study was to investigate the effects of mixed thiol collectors, namely PAX and IPETC on the froth flotation of an Nkomati sulphide ore. Tests using pure collectors as well as an industrially used collector mixture were done in order to establish a baseline for performance evaluation after assessment. Tests were also conducted using decreased frother and collector dosages (1.3, 0.65 and 0.325mm/ton) of the collector mixtures to assess their metallurgical performances.

At a frother dosage of 12g/t, the cumulative nickel grades for mixtures of PAX and IPETC were generally lower than those achieved at 50g/t of frother. Although there may have been differences in the cumulative nickel grades there was no significant improvement. This suggests that at 12g/t a more selective flotation could be expected due to lower entrainment. With the use of PAX and SIBX, the froth phase generated was significantly lower, which prevented the effective beneficiation (couldn't scrap anything). As expected generally higher cumulative nickel grades come at the expense of cumulative nickel recovery. Following the trend, the cumulative nickel recoveries achieved at 12g/t of frother was generally lower than the cumulative nickel recoveries obtained with 50g/t of frother.

With the use of pure collectors it was found that the water recovery increased as the concentration of IPETC increased. This may be attributed to its frothing properties that could have increased the stability of the froth, decreased the water drainage and mineral drop-back and subsequently increased the non-selective mass recovery due to entrainment. Using IPETC, an increase in the cumulative water recovery and decrease in cumulative nickel grade relative to PAX was observed.

Generally, an increase in recovery was observed with the collector mixtures at the molar dosage of 1.3mmol/t of 95.5%SIBX:4.5%IPETC relative to the pure collectors at the same dosage. Amongst the collector mixtures there were no significant improvements with regards to the nickel grade and recovery when compared to the industry mixture. Of the collector mixtures, 85%PAX:15%IPETC and 90%PAX:10%IPETC produced the joint highest cumulative grades out of the tested mixtures (1.75%). These mixtures gave recoveries of 82.3% and 82.2% respectively. Collector mixture 95.5% PAX: 4.5% IPETC gave the second highest grade (1.73%) and the highest nickel recovery (84.6%).

The collector mixtures at different dosages indicated that at lower dosages apart from the changes

in water and mass recovery, no significant improvement was observed. The increase in grade generally comes at the expense of cumulative nickel recovery. The lowest dosage (at the molar dosage of 0.325mmol/t) for the various collector mixtures obtained the highest calculated Klimpel rate constants, which furthermore indicates that a faster rate of flotation is achieved at lower dosages. Collector mixture 95.5%PAX:4.5%IPETC at the molar dosage of 0.325mmol/t was the most selective mixture out of all the tests, giving a cumulative grade of 2.1% (however it was insignificant compared to the industry mixture). It was also observed that an increase in collector dosage generally increased the cumulative nickel recovery, with collector mixture 95.5%PAX:4.5%IPETC at the molar dosage of 1.3mmol/t giving the highest cumulative recovery (84.5%). The study also indicated that an increase in selectivity (cumulative grade) normally comes at the expense of cumulative recovery.

## 5.2 Recommendations

Based on the findings and conclusions of this thesis, the recommendations for future work are:

- Further Investigation into the frothing properties and effects of isopropyl ethyl thionocarbamate. This should provide more information as to what the dominant mechanism is causing an increase in mineral recovery (entrainment or selective bubble attachment).
- Microflotation test work must be carried out with the pure collectors and collector mixtures at different dosages and frother dosages in order to better understand collector-mineral-frother interactions with thionocarbamates as well as to decouple froth phase effects and pulp phase effects.
- Zeta and pulp potential measurement test work should be carried out in order to investigate the minerals' surface charges as well as charge transfer in the pulp which will contribute to better understanding of the pulp and surface chemistry as well as reactions on the minerals' surfaces.
- An activator should also be used in order to investigate the effects of activation before beneficiation.
- The use of decreasing dosages of mixture 50%PAX: 50%SIBX to investigate possible improvements in the grades and recoveries with more cost effective traditional xanthates.

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## APPENDIX

### Appendix A: The chemical formulae of minerals in the Nkomati ore

Table 13: Mineral composition of the ore

| Mineral         | Chemical Formula  |
|-----------------|---|
| Actinolite      | $\text{Ca}_2(\text{Mg}_{4.5-2.5}\text{Fe}^{2+}_{0.5-2.5})\text{Si}_8\text{O}_{22}(\text{OH})_2$ |
| Biotite         | $\text{K}(\text{Mg, Fe})_3\text{AlSi}_3\text{O}_{10}(\text{OH, F})_2$                           |
| Chalcopyrite    | $\text{CuFeS}_2$  |
| Chromite        | $(\text{Fe, Mg})\text{Cr}_2\text{O}_4$  |
| Diopside        | $\text{MgCaSi}_2\text{O}_6$   |
| Enstatite       | $(\text{Mg, Fe})\text{SiO}_3$   |
| Forsterite iron | $(\text{Mg}_2\text{SiO}_4)$   |
| Lizardite       | $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$   |
| Pentlandite     | $(\text{Fe, Ni})_9\text{S}_8$   |
| Pyrite          | $\text{FeS}_2$  |
| Pyrrhotite      | $\text{Fe}_{1-x}\text{S}$ ( $x = 0$ to $0.2$ )  |
| Quartz          | $\text{SiO}_2$  |
| Talc            | $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$  |

## Appendix B: Calculation

### 1.1.1 Material balances

Equation 3 represents the general form of a material balance.

$$\text{Accumulation} = \text{In} + \text{Generation} - \text{Out} - \text{Consumption} \quad \dots\dots\dots \text{Equation 3}$$

### 1.1.2 Concentration

$$C = \frac{n}{v} \quad \dots\dots\dots \text{Equation 4}$$

### 1.1.3 Moles of Collectors

$$\text{Moles of Collector}_{20g/t}(n) \approx \frac{\text{Mass}}{\text{Molar Mass}} = \frac{0.02g}{\text{molar mass of collector}} \quad \dots\dots\dots \text{Equation 5}$$

### 1.1.4 Required Mass of Collector for Preparation of Solution

$$\text{Collector}_{\text{dosage}} = \left( \frac{\text{volume}_{\text{flask}}}{\text{volume}_{\text{syringe}}} \right) (\text{Collector dosage}) \quad \dots\dots\dots \text{Equation 6}$$

### 1.1.5 Collector Dosage

$$\text{Collector}_{20g/t} \approx \frac{20g}{1000kg\text{Ore}} = \approx 0.02g \text{ per } 1 \text{ Kg of ore} \quad \dots\dots\dots \text{Equation 7}$$

### 1.1.6 Mass Equivalents Needed for New Collector

$$\text{Collector B}_{\text{massequivalent}} \approx (\text{Collector A}_{\text{Molarequivalent}})(\text{Collector B}_{\text{moleculamass}}) \quad \dots\dots\dots \text{Equation 8}$$

### 1.1.7 Mass Equivalents of the collector mixtures

$$\begin{aligned} &60\% \text{ Xanthate collector}/40\% \text{ Thiol collector}_{\text{massequivalent}} = \\ &(\text{Xanthate collector moles}_{20/t})(\text{Xanthate collector ratio}_{60\%})(\text{Xanthate collector}_{\text{MM}}) \\ &+ \\ &(\text{Xanthate collector moles}_{20/t})(\text{Thiol collector ratio}_{40\%})(\text{Thiol collector}_{\text{MM}}) \end{aligned} \quad \dots\dots \text{Equation 9}$$

## Appendix C: Experimental Data

Table 14: Summary of results with 12g/t of frother and 240g/t of depressant

| Nickel                         |                  |                                 |             |                      |             |                                |             |                             |            |            |             |
|--------------------------------|------------------|---------------------------------|-------------|----------------------|-------------|--------------------------------|-------------|-----------------------------|------------|------------|-------------|
| Collector                      | Mole Ratio       | Cumulative Concentrate Mass (%) |             | Cumulative Water (%) |             | Cumulative Nickel Recovery (%) |             | Cumulative Nickel Grade (%) |            | K (/min)   | Rmax (%)    |
|                                |                  | 2 min                           | 20 min      | 2 min                | 20 min      | 2 min                          | 20 min      | 2 min                       | 20 min     |            |             |
| IPETC (1.3mmol/t)              | 100              | 11.4                            | 29.4        | 15.9                 | 83.2        | 51.4                           | 74.8        | 2.4                         | 1.3        | 1.4        | 76.1        |
| <b>SIBX: IPETC (1.3mmol/t)</b> | <b>95,5: 4,5</b> | <b>13.2</b>                     | <b>26.0</b> | <b>16.5</b>          | <b>56.0</b> | <b>42.8</b>                    | <b>79.4</b> | <b>1.6</b>                  | <b>1.5</b> | <b>0.8</b> | <b>82.4</b> |
| PAX: IPETC (1.3mmol/t)         | 95,5: 4,5        | 8.9                             | 15.2        | 8.6                  | 14.1        | 41.2                           | 68.6        | 2.4                         | 2.3        | 1.0        | 70.3        |
| PAX: IPETC (1.3mmol/t)         | 90 : 10          | 9.5                             | 14.0        | 9.2                  | 11.9        | 42.2                           | 66.0        | 2.1                         | 2.3        | 1.2        | 66.0        |
| PAX: IPETC (1.3mmol/t)         | 85 : 15          | 9.4                             | 16.9        | 9.3                  | 18.4        | 44.6                           | 73.3        | 2.3                         | 2.1        | 1.0        | 75.1        |
| PAX: IPETC (1.3mmol/t)         | 80 : 20          | 9.3                             | 18.9        | 9.6                  | 23.6        | 40.9                           | 74.3        | 2.1                         | 1.9        | 0.8        | 78.2        |

Table 15: Summary of results with 50g/t of frother and 240g/t of depressant

| <b>Nickel</b>                     |                  |                          |             |              |                           |             |              |                      |             |              |                   |            |              |            |             |
|-----------------------------------|------------------|--------------------------|-------------|--------------|---------------------------|-------------|--------------|----------------------|-------------|--------------|-------------------|------------|--------------|------------|-------------|
| Collector                         | Mole Ratio       | Ave. Cumulative Mass (%) |             | Ave Std Dev. | Ave. Cumulative Water (%) |             | Ave Std Dev. | Ave. Ni Recovery (%) |             | Ave Std Dev. | Ave. Ni Grade (%) |            | Ave Std Dev. | K (/min)   | Rmax (%)    |
|                                   |                  | 2 min                    | 20 min      |              | 2 min                     | 20 min      |              | 2 min                | 20 min      |              | 2 min             | 20 min     |              |            |             |
| No Collector                      | 0                | 4.5                      | 9.8         | 0.0          | 6.7                       | 17.4        | 0.0          | 11.3                 | 17.8        | 0.0          | 1.1               | 0.8        | 0.0          | 1.1        | 18.1        |
| PAX (1.3mmol/t)                   | 100              | 12.0                     | 19.8        | 2.2          | 13.7                      | 25.9        | 4.4          | 48.7                 | 77.0        | 1.3          | 1.9               | 1.9        | 0.1          | 1.1        | 80.1        |
| SIBX (1.3mmol/t)                  | 100              | 11.5                     | 20.8        | 4.0          | 12.7                      | 28.8        | 10.4         | 48.9                 | 79.1        | 1.8          | 2.1               | 1.9        | 0.2          | 1.1        | 92.0        |
| IPETC (1.3mmol/t)                 | 100              | 15.0                     | 33.8        | 6.2          | 20.0                      | 96.4        | 14.8         | 61.8                 | 82.2        | 3.0          | 2.1               | 1.2        | 0.1          | 1.9        | 83.2        |
| <b>SIBX: IPETC</b><br>(1.3mmol/t) | <b>95,5: 4,5</b> | <b>12.7</b>              | <b>23.5</b> | <b>0.7</b>   | <b>15.8</b>               | <b>39.9</b> | <b>4.0</b>   | <b>50.8</b>          | <b>82.0</b> | <b>1.5</b>   | <b>1.8</b>        | <b>1.6</b> | <b>0.1</b>   | <b>1.1</b> | <b>85.7</b> |
| PAX: IPETC<br>(1.3mmol/t)         | 95,5: 4,5        | 12.3                     | 23.1        | 2.0          | 14.9                      | 35.9        | 5.6          | 50.3                 | 84.5        | 3.2          | 1.9               | 1.7        | 0.1          | 1.0        | 87.9        |
| PAX: IPETC<br>(0.65mmol/t)        | 95,5: 4,5        | 8.0                      | 17.7        | 4.7          | 12.3                      | 32.8        | 18.4         | 46.1                 | 77.5        | 8.9          | 2.6               | 2.0        | 0.5          | 1.0        | 81.5        |
| PAX: IPETC<br>(0.325mmol/t)       | 95,5: 4,5        | 7.5                      | 18.1        | 4.8          | 7.0                       | 27.9        | 13.6         | 48.0                 | 76.2        | 3.0          | 3.3               | 2.1        | 0.6          | 1.1        | 80.0        |
| PAX: IPETC<br>(1.3mmol/t)         | 90 : 10          | 11.3                     | 22.1        | 1.7          | 13.0                      | 33.2        | 4.9          | 47.3                 | 80.2        | 2.2          | 2.0               | 1.7        | 0.1          | 1.0        | 84.5        |
| PAX: IPETC<br>(0.65mmol/t)        | 90 : 10          | 10.6                     | 33.7        | 1.1          | 9.8                       | 28.8        | 4.2          | 42.6                 | 69.6        | 8.1          | 2.2               | 1.6        | 0.1          | 1.1        | 72.8        |
| PAX: IPETC<br>(0.325mmol/t)       | 90 : 10          | 9.9                      | 20.2        | 4.0          | 9.9                       | 30.7        | 12.0         | 55.4                 | 77.8        | 9.4          | 2.9               | 1.9        | 0.4          | 1.5        | 80.2        |
| PAX: IPETC<br>(1.3mmol/t)         | 85 : 15          | 8.7                      | 22.3        | 1.5          | 9.3                       | 41.1        | 6.2          | 40.0                 | 80.3        | 2.6          | 2.2               | 1.7        | 0.2          | 0.7        | 87.3        |
| PAX: IPETC<br>(0.65mmol/t)        | 85 : 15          | 9.7                      | 22.3        | 1.1          | 10.2                      | 39.5        | 1.8          | 45.5                 | 80.2        | 3.8          | 2.2               | 1.7        | 0.1          | 0.9        | 85.4        |
| PAX: IPETC<br>(0.325mmol/t)       | 85 : 15          | 9.0                      | 21.3        | 1.1          | 8.7                       | 35.1        | 4.7          | 56.2                 | 79.6        | 0.9          | 3.1               | 1.8        | 0.2          | 1.5        | 81.5        |
| PAX: IPETC<br>(1.3mmol/t)         | 80 : 20          | 11.7                     | 25.0        | 0.5          | 15.5                      | 49.0        | 7.7          | 46.4                 | 82.6        | 2.9          | 2.0               | 1.6        | 0.1          | 87.8       | 0.0         |
| PAX: SIBX<br>(1.3mmol/t)          | 50 : 50          | 11.0                     | 20.2        | 4.9          | 12.3                      | 27.6        | 13.8         | 45.4                 | 78.7        | 2.8          | 2.0               | 1.9        | 0.5          | 0.9        | 96.2        |

## Appendix D: Sulphur Grade vs Recovery Analysis

Table 16: Leco Sulphur analysis

| <b>Sample Name</b> | <b>Sulphur %</b> | <b>Sample Name</b> | <b>Sulphur %</b> |
|--------------------|------------------|--------------------|------------------|
| Run 10 Feed        | 3.22             | Run 16 Feed        | 3.17             |
| Run 10 C1          | 6.87             | Run 16 C1          | 13.9             |
| Run 10 C2          | 4.68             | Run 16 C2          | 13.2             |
| Run 10 C3          | 4.59             | Run 16 C3          | 13.3             |
| Run 10 C4          | 4.97             | Run 16 C4          | 10.9             |
| Run 10 Tails       | 3.01             | Run 16 Tails       | 0.726            |
| Run 12 Feed        | 3.24             | Run 17 Feed        | 3.38             |
| Run 12 C1          | 13.2             | Run 17 C1          | 14.8             |
| Run 12 C2          | 11.9             | Run 17 C2          | 13.7             |
| Run 12 C3          | 11.8             | Run 17 C3          | 12.3             |
| Run 12 C4          | 10.5             | Run 17 C4          | 9.57             |
| Run 12 Tails       | 1.22             | Run 17 Tails       | 0.663            |
| Run 13 Feed        | 3.24             | Run 18 Feed        | 3.13             |
| Run 13 C1          | 9.09             | Run 18 C1          | 13.1             |
| Run 13 C2          | 7.01             | Run 18 C2          | 12.2             |
| Run 13 C3          | 5.87             | Run 18 C3          | 11.5             |
| Run 13 C4          | 6.82             | Run 18 C4          | 9.31             |
| Run 13 Tails       | 1.43             | Run 18 Tails       | 0.66             |
| Run 14 Feed        | 3.07             | Run 33 Feed        | 3.31             |
| Run 14 C1          | 13.6             | Run 33 C1          | 17.5             |
| Run 14 C2          | 12.6             | Run 33 C2          | 13.0             |
| Run 14 C3          | 11.9             | Run 33 C3          | 12.0             |
| Run 14 C4          | 9.26             | Run 33 C4          | 10.3             |
| Run 14 Tails       | 0.673            | Run 33 Tails       | 1.06             |
| Run 15 Feed        | 3.22             | Run 34 Feed        | 3.09             |
| Run 15 C1          | 13.2             | Run 34 C1          | 24               |
| Run 15 C2          | 13.1             | Run 34 C2          | 13.6             |
| Run 15 C3          | 14.3             | Run 34 C3          | 10.2             |
| Run 15 C4          | 10.1             | Run 34 C4          | 9.37             |
| Run 15 Tails       | 0.711            | Run 34 Tails       | 1.27             |

## Appendix E: Concentrate Particle Size Analysis

Table 17: Particle size range for concentrates

| Sample  | % Particles at Different Size Fractions |       |        |       |        |       |        |       |        |        |       |        |        |        |        |
|---|---|-------|--------|-------|--------|-------|--------|-------|--------|--------|-------|--------|--------|--------|--------|
|   | 2 µm                                    | 3 µm  | 5 µm   | 7 µm  | 10 µm  | 13 µm | 19 µm  | 25 µm | 38 µm  | 53 µm  | 75 µm | 106 µm | 150 µm | 212 µm | 300 µm |
| Run 10 (No Collector) Feed - Average                        | 4.153                                   | 3.766 | 6.477  | 5.308 | 6.443  | 5.329 | 8.910  | 7.547 | 13.046 | 10.561 | 9.674 | 7.244  | 4.537  | 2.308  | 1.079  |
| Run 10 (No Collector) Conc 1 - Average                      | 3.925                                   | 3.874 | 7.312  | 6.613 | 8.677  | 7.438 | 12.063 | 9.319 | 14.044 | 9.671  | 7.404 | 4.264  | 1.748  | 0.383  | 0.085  |
| Run 10 (No Collector) Conc 2 - Average                      | 4.479                                   | 4.672 | 9.017  | 7.925 | 9.676  | 7.582 | 11.134 | 7.944 | 11.579 | 8.085  | 6.495 | 4.202  | 2.326  | 1.107  | 0.465  |
| Run 10 (No Collector) Conc 3 - Average                      | 5.398                                   | 5.687 | 10.667 | 8.866 | 10.152 | 7.449 | 10.168 | 6.741 | 9.350  | 6.591  | 5.722 | 4.178  | 2.725  | 1.786  | 1.069  |
| Run 10 (No Collector) Conc 4 - Average                      | 6.419                                   | 6.751 | 12.293 | 9.826 | 10.846 | 7.692 | 10.120 | 6.420 | 8.373  | 5.418  | 4.364 | 3.053  | 2.027  | 1.444  | 0.936  |
| Run 12 (PAX 1.3mmol/t) Feed - Average                       | 4.259                                   | 3.901 | 6.703  | 5.416 | 6.428  | 5.261 | 9.053  | 8.103 | 14.238 | 10.420 | 7.781 | 5.317  | 4.526  | 3.504  | 1.768  |
| Run 12 (PAX 1.3mmol/t) Conc 1 - Average                     | 4.091                                   | 4.019 | 7.489  | 6.589 | 8.418  | 7.137 | 11.724 | 9.327 | 14.337 | 9.641  | 6.854 | 3.553  | 1.386  | 0.427  | 0.169  |
| Run 12 (PAX 1.3mmol/t) Conc 2 - Average                     | 4.948                                   | 5.108 | 9.559  | 8.101 | 9.657  | 7.505 | 11.123 | 8.097 | 11.928 | 8.141  | 6.140 | 3.482  | 1.516  | 0.546  | 0.325  |
| Run 12 (PAX 1.3mmol/t) Conc 3 - Averaged                    | 5.972                                   | 6.196 | 11.235 | 9.011 | 10.174 | 7.581 | 10.948 | 7.897 | 11.524 | 7.468  | 4.874 | 1.878  | 0.217  | 0.104  | 0.294  |
| Run 12 (PAX 1.3mmol/t) Conc 4 - Average                     | 6.272                                   | 6.452 | 11.580 | 9.193 | 10.231 | 7.456 | 10.392 | 7.181 | 10.171 | 6.732  | 4.966 | 2.762  | 1.270  | 0.704  | 0.571  |
| Run 13 (IPETC 1.3mmol/t) Feed - Average                     | 4.371                                   | 3.929 | 6.703  | 5.434 | 6.551  | 5.429 | 9.185  | 7.874 | 13.598 | 10.770 | 9.454 | 6.632  | 3.782  | 1.602  | 0.547  |
| Run 13 (IPETC 1.3mmol/t) Conc 1 - Average                   | 5.054                                   | 4.923 | 9.127  | 7.915 | 9.851  | 8.098 | 12.824 | 9.696 | 13.410 | 7.131  | 3.407 | 1.444  | 1.623  | 1.464  | 0.565  |
| Run 13 (IPETC 1.3mmol/t) Conc 2 - Average                   | 5.902                                   | 5.798 | 10.547 | 8.855 | 10.499 | 8.043 | 11.477 | 7.805 | 10.429 | 6.436  | 4.640 | 2.740  | 1.465  | 0.824  | 0.397  |
| Run 13 (IPETC 1.3mmol/t) Conc 3 - Average                   | 6.494                                   | 6.186 | 10.723 | 8.672 | 10.132 | 7.748 | 11.019 | 7.340 | 9.431  | 5.737  | 4.467 | 3.186  | 2.199  | 1.446  | 0.738  |
| Run 13 (IPETC 1.3mmol/t) Conc 4 - Average                   | 6.432                                   | 6.060 | 10.209 | 7.968 | 9.028  | 6.762 | 9.589  | 6.554 | 9.023  | 6.161  | 5.485 | 4.549  | 3.597  | 2.655  | 1.525  |
| Run 14 (95.5% SIBX : 4.5% IPETC 1.3mmol/t) Conc 1 - Average | 4.395                                   | 4.397 | 8.348  | 7.390 | 9.342  | 7.738 | 12.251 | 9.363 | 13.928 | 9.127  | 6.256 | 2.949  | 1.012  | 0.443  | 0.176  |
| Run 14 (95.5% SIBX : 4.5% IPETC 1.3mmol/t) Conc 2 - Average | 5.147                                   | 5.302 | 9.967  | 8.454 | 10.059 | 7.830 | 11.652 | 8.454 | 12.109 | 7.769  | 5.497 | 3.077  | 1.228  | 0.261  | 0.000  |
| Run 14 (95.5% SIBX : 4.5% IPETC 1.3mmol/t) Conc 3 - Average | 5.043                                   | 5.006 | 8.847  | 7.047 | 8.004  | 6.048 | 8.978  | 6.781 | 10.851 | 8.527  | 7.818 | 5.833  | 3.608  | 1.949  | 1.241  |



|  |       |       |        |       |        |       |        |        |        |       |       |       |       |       |       |
|--|-------|-------|--------|-------|--------|-------|--------|--------|--------|-------|-------|-------|-------|-------|-------|
| Run 14 (95.5% SIBX : 4.5% IPETC 1.3mmol/t) Conc 4 - Average  | 5.277 | 5.279 | 9.203  | 7.151 | 7.887  | 5.775 | 8.270  | 6.096  | 9.877  | 8.125 | 7.834 | 6.179 | 4.162 | 2.545 | 1.638 |
| Run 15 (95.5% PAX : 4.5% IPETC 1.3mmol/t) Conc 1 - Average   | 4.482 | 4.405 | 8.195  | 7.154 | 8.940  | 7.359 | 11.713 | 9.097  | 13.823 | 9.284 | 6.627 | 3.460 | 1.345 | 0.371 | 0.177 |
| Run 15 (95.5% PAX : 4.5% IPETC 1.3mmol/t) Conc 2 - Average   | 5.091 | 5.304 | 9.953  | 8.451 | 10.096 | 7.873 | 11.738 | 8.518  | 11.972 | 7.139 | 4.449 | 2.483 | 1.895 | 1.349 | 0.527 |
| Run 15 (95.5% PAX : 4.5% IPETC 1.3mmol/t) Conc 3 - Average   | 5.076 | 4.989 | 8.802  | 7.081 | 8.142  | 6.244 | 9.573  | 7.621  | 12.802 | 9.916 | 7.994 | 4.616 | 2.132 | 1.088 | 0.635 |
| Run 15 (95.5% PAX : 4.5% IPETC 1.3mmol/t) Conc 4 - Average   | 6.587 | 6.209 | 10.306 | 7.746 | 8.313  | 5.936 | 8.473  | 6.429  | 10.676 | 8.455 | 7.240 | 4.480 | 1.982 | 1.117 | 1.285 |
| Run 16 (90% PAX : 10% IPETC 1.3mmol/t) Conc 1 - Average      | 4.279 | 4.256 | 7.976  | 7.013 | 8.889  | 7.432 | 11.985 | 9.378  | 14.286 | 9.583 | 6.767 | 3.360 | 1.102 | 0.188 | 0.106 |
| Run 16 (90% PAX : 10% IPETC 1.3mmol/t) Conc 2 - Average      | 5.340 | 5.413 | 9.860  | 8.170 | 9.575  | 7.339 | 10.785 | 7.857  | 11.679 | 8.020 | 6.001 | 3.338 | 1.595 | 1.063 | 0.640 |
| Run 16 (90% PAX : 10% IPETC 1.3mmol/t) Conc 3 - Average      | 5.298 | 5.248 | 9.233  | 7.372 | 8.399  | 6.345 | 9.441  | 7.213  | 11.637 | 8.869 | 7.399 | 4.689 | 2.450 | 1.360 | 1.023 |
| Run 16 (90% PAX : 10% IPETC 1.3mmol/t) Conc 4 - Average      | 6.412 | 6.093 | 10.124 | 7.587 | 8.100  | 5.752 | 8.202  | 6.274  | 10.508 | 8.245 | 6.954 | 4.648 | 2.983 | 2.111 | 1.353 |
| Run 17 (85% PAX : 15% IPETC 1.3mmol/t) Conc 1 - Average      | 4.524 | 4.535 | 8.575  | 7.597 | 9.644  | 8.032 | 12.825 | 9.884  | 14.711 | 9.421 | 5.856 | 1.429 | 0.000 | 0.000 | 0.000 |
| Run 17 (85% PAX : 15% IPETC 1.3mmol/t) Conc 2 - Average      | 5.019 | 5.200 | 9.711  | 8.194 | 9.762  | 7.609 | 11.369 | 8.367  | 12.360 | 8.277 | 5.967 | 3.105 | 1.175 | 0.439 | 0.295 |
| Run 17 (85% PAX : 15% IPETC 1.3mmol/t) Conc 3 - Average      | 5.201 | 5.123 | 9.021  | 7.208 | 8.238  | 6.263 | 9.370  | 7.150  | 11.480 | 8.811 | 7.637 | 5.283 | 3.077 | 1.647 | 0.891 |
| Run 17 (85% PAX : 15% IPETC 1.3mmol/t) Conc 4 - Average      | 6.303 | 5.950 | 9.850  | 7.361 | 7.888  | 5.630 | 7.959  | 5.934  | 9.882  | 8.237 | 7.789 | 5.793 | 3.584 | 2.084 | 1.196 |
| Run 18 (80% PAX : 20% IPETC 1.3mmol/t) Conc 1 - Average      | 4.559 | 4.591 | 8.656  | 7.542 | 9.486  | 7.937 | 12.756 | 9.705  | 13.724 | 8.226 | 5.363 | 2.747 | 1.193 | 0.440 | 0.113 |
| Run 18 (80% PAX : 20% IPETC 1.3mmol/t) Conc 2 - Average      | 5.555 | 5.602 | 10.162 | 8.296 | 9.592  | 7.313 | 10.726 | 7.751  | 11.273 | 7.542 | 5.644 | 3.365 | 1.866 | 1.185 | 0.644 |
| Run 18 (80% PAX : 20% IPETC 1.3mmol/t) Conc 3 - Average      | 5.977 | 5.845 | 10.135 | 7.865 | 8.675  | 6.375 | 9.211  | 6.800  | 10.591 | 7.943 | 6.887 | 4.869 | 2.845 | 1.411 | 0.699 |
| Run 18 (80% PAX : 20% IPETC 1.3mmol/t) Conc 4 - Average      | 6.079 | 5.918 | 9.971  | 7.479 | 7.954  | 5.624 | 7.846  | 5.736  | 9.356  | 7.723 | 7.406 | 5.864 | 4.116 | 2.718 | 1.697 |
| Run 33 (95.5% PAX : 4.5% IPETC 0.65mmol/t) Conc 1 - Average  | 4.101 | 4.156 | 8.031  | 7.300 | 9.509  | 8.146 | 13.413 | 10.521 | 15.440 | 9.471 | 5.755 | 1.500 | 0.000 | 0.000 | 0.000 |
| Run 33 (95.5% PAX : 4.5% IPETC 0.65mmol/t) Conc 2 - Average  | 4.386 | 4.571 | 8.913  | 7.960 | 9.931  | 7.998 | 12.192 | 8.983  | 12.967 | 8.297 | 5.558 | 2.372 | 0.452 | 0.095 | 0.057 |
| Run 33 (95.5% PAX : 4.5% IPETC 0.65mmol/t) Conc 3 - Average  | 4.954 | 5.145 | 9.592  | 8.021 | 9.388  | 7.188 | 10.653 | 7.900  | 11.883 | 7.968 | 5.473 | 2.689 | 1.604 | 1.905 | 1.892 |
| Run 33 (95.5% PAX : 4.5% IPETC 0.65mmol/t) Conc 4 - Average  | 5.489 | 5.624 | 10.135 | 8.177 | 9.352  | 7.070 | 10.349 | 7.501  | 10.976 | 7.540 | 6.096 | 4.090 | 2.293 | 1.117 | 0.561 |
| Run 34 (95.5% PAX : 4.5% IPETC 0.325mmol/t) Conc 1 - Average | 3.393 | 3.611 | 7.416  | 7.101 | 9.597  | 8.432 | 14.144 | 11.236 | 16.553 | 9.939 | 5.589 | 0.832 | 0.000 | 0.000 | 0.000 |
| Run 34 (95.5% PAX : 4.5% IPETC 0.325mmol/t) Conc 2 - Average | 4.026 | 4.229 | 8.292  | 7.447 | 9.404  | 7.745 | 12.261 | 9.452  | 14.243 | 9.435 | 6.499 | 2.951 | 0.818 | 0.425 | 0.231 |
| Run 34 (95.5% PAX : 4.5% IPETC 0.325mmol/t) Conc 3 - Average | 4.800 | 5.003 | 9.353  | 7.887 | 9.354  | 7.250 | 10.790 | 7.949  | 11.918 | 8.339 | 6.556 | 4.010 | 1.962 | 0.855 | 0.510 |
| Run 34 (95.5% PAX : 4.5% IPETC 0.325mmol/t) Conc 4 - Average | 5.269 | 5.426 | 9.899  | 8.128 | 9.438  | 7.194 | 10.508 | 7.551  | 11.015 | 7.612 | 6.152 | 4.100 | 2.383 | 1.307 | 0.652 |

## Appendix F: Statistical Analysis of Cumulative Water and Recovery%

|   |  |                                       |
|---|--|---------------------------------------|
| Cumulative Water Recovery %: Industry Vs SIBX                 |  |                                       |
| t-Test: Paired Two Sample for Means                           |  |                                       |
|   | Industry Mixture (1.3mmol/ton) at 50g/t of frother | SIBX (1.3mmol/ton)                    |
| Mean  | 39.92  | 28.82                                 |
| t Stat  | 2.56   |                                       |
| P(T<=t) two-tail  | 0.24   |                                       |
| t Critical two-tail   | 12.71  |                                       |
| Null Hypothesis:  | Accept H0  | No Significant Difference             |
| Cumulative Water Recovery %: Industry Vs PAX                  |  |                                       |
| t-Test: Paired Two Sample for Means                           |  |                                       |
|   | Industry Mixture (1.3mmol/ton) at 50g/t of frother | PAX (1.3mmol/ton)                     |
| Mean  | 39.92  | 25.89                                 |
| t Stat  | 36.09  |                                       |
| P(T<=t) two-tail  | 0.02   |                                       |
| t Critical two-tail   | 12.71  |                                       |
| Null Hypothesis:  | Reject H0  | Significantly Different               |
| Cumulative Water Recovery %: Industry Vs IPETC                |  |                                       |
| t-Test: Paired Two Sample for Means                           |  |                                       |
|   | Industry Mixture (1.3mmol/ton) at 50g/t of frother | IPETC (1.3mmol/ton)                   |
| Mean  | 39.92  | 96.37                                 |
| t Stat  | -4.94  |                                       |
| P(T<=t) two-tail  | 0.13   |                                       |
| t Critical two-tail   | 12.71  |                                       |
| Null Hypothesis:  | Accept H0  | No Significant Difference             |
| Cumulative Water Recovery %: Industry Vs 95.5%PAX: 4.5%IPETC  |  |                                       |
| t-Test: Paired Two Sample for Means                           |  |                                       |
|   | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (1.3mmol/ton)  |
| Mean  | 39.92  | 35.87                                 |
| t Stat  | 162.31   |                                       |
| P(T<=t) two-tail  | 0.00   |                                       |
| t Critical two-tail   | 12.71  |                                       |
| Null Hypothesis:  | Reject H0  | Significantly Different               |
| Cumulative Water Recovery %: Industry Vs 95.5%PAX: 4.5% IPETC |  |                                       |
| t-Test: Paired Two Sample for Means                           |  |                                       |
|   | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (0.65mmol/ton) |
| Mean  | 39.92  | 32.77                                 |
| t Stat  | 0.57   |                                       |
| P(T<=t) two-tail  | 0.67   |                                       |
| t Critical two-tail   | 12.71  |                                       |
| Null Hypothesis:  | Accept H0  | No Significant Difference             |

|   |           |                           |
|---|-----------|---------------------------|
| Cumulative Water Recovery %: Industry Vs 95.5%PAX: 4.5% IPETC                             |           |                           |
| t-Test: Paired Two Sample for Means   |           |                           |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother 95.5% PAX : 4.5% IPETC (0.325mmol/ton) |           |                           |
| Mean  | 39.92     | 27.92                     |
| t Stat  | 1.47      |                           |
| P(T<=t) two-tail  | 0.38      |                           |
| t Critical two-tail   | 12.71     |                           |
| Null Hypothesis:  | Accept H0 | No Significant Difference |
| Cumulative Water Recovery %: Industry Vs 90%PAX: 10% IPETC                                |           |                           |
| t-Test: Paired Two Sample for Means   |           |                           |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother 90% PAX : 10% IPETC (1.3mmol/ton)      |           |                           |
| Mean  | 39.92     | 33.15                     |
| t Stat  | 6.80      |                           |
| P(T<=t) two-tail  | 0.09      |                           |
| t Critical two-tail   | 12.71     |                           |
| Null Hypothesis:  | Accept H0 | No Significant Difference |
| Cumulative Water Recovery %: Industry Vs 90%PAX: 10% IPETC                                |           |                           |
| t-Test: Paired Two Sample for Means   |           |                           |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother 85% PAX : 15% IPETC (0.65mmol/ton)     |           |                           |
| Mean  | 39.92     | 33.71                     |
| t Stat  | 68.49     |                           |
| P(T<=t) two-tail  | 0.01      |                           |
| t Critical two-tail   | 12.71     |                           |
| Null Hypothesis:  | Reject H0 | Significantly Different   |
| Cumulative Water Recovery %: Industry Vs 90%PAX: 10% IPETC                                |           |                           |
| t-Test: Paired Two Sample for Means   |           |                           |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother 90% PAX : 10% IPETC (0.325mmol/ton)    |           |                           |
| Mean  | 39.92     | 30.71                     |
| t Stat  | 1.44      |                           |
| P(T<=t) two-tail  | 0.39      |                           |
| t Critical two-tail   | 12.71     |                           |
| Null Hypothesis:  | Accept H0 | No Significant Difference |
| Cumulative Water Recovery %: Industry Vs 85%PAX: 15% IPETC                                |           |                           |
| t-Test: Paired Two Sample for Means   |           |                           |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother 85% PAX : 15% IPETC (1.3mmol/ton)      |           |                           |
| Mean  | 39.92     | 41.09                     |
| t Stat  | -2.91     |                           |
| P(T<=t) two-tail  | 0.21      |                           |
| t Critical two-tail   | 12.71     |                           |
| Null Hypothesis:  | Accept H0 | No Significant Difference |

|   |           |  |                                     |  |  |
|---|-----------|--|-------------------------------------|--|--|
| Cumulative Water Recovery %:<br>t-Test: Paired Two Sample for Means |           |  | Industry Vs 85%PAX: 15% IPETC       |  |  |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother                  |           |  | 85% PAX : 15% IPETC (0.65mmol/ton)  |  |  |
| Mean  | 39.92     |  | 39.51                               |  |  |
| t Stat  | 0.12      |  |                                     |  |  |
| P(T<=t) two-tail  | 0.93      |  |                                     |  |  |
| t Critical two-tail   | 12.71     |  |                                     |  |  |
| Null Hypothesis:  | Accept H0 |  | No Significant Difference           |  |  |
| Cumulative Water Recovery %:<br>t-Test: Paired Two Sample for Means |           |  | Industry Vs 85%PAX: 15% IPETC       |  |  |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother                  |           |  | 85% PAX : 15% IPETC (0.325mmol/ton) |  |  |
| Mean  | 39.92     |  | 35.07                               |  |  |
| t Stat  | 8.67      |  |                                     |  |  |
| P(T<=t) two-tail  | 0.07      |  |                                     |  |  |
| t Critical two-tail   | 12.71     |  |                                     |  |  |
| Null Hypothesis:  | Accept H0 |  | No Significant Difference           |  |  |
| Cumulative Water Recovery %:<br>t-Test: Paired Two Sample for Means |           |  | Industry Vs 80%PAX: 20% IPETC       |  |  |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother                  |           |  | 80% PAX : 20% IPETC (1.3mmol/ton)   |  |  |
| Mean  | 39.92     |  | 49.00                               |  |  |
| t Stat  | -3.20     |  |                                     |  |  |
| P(T<=t) two-tail  | 0.19      |  |                                     |  |  |
| t Critical two-tail   | 12.71     |  |                                     |  |  |
| Null Hypothesis:  | Accept H0 |  | No Significant Difference           |  |  |
| Cumulative Water Recovery %:<br>t-Test: Paired Two Sample for Means |           |  | Industry Vs 50%PAX: 50% SIBX        |  |  |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother                  |           |  | 50% PAX : 50% SIBX (1.3mmol/ton)    |  |  |
| Mean  | 39.92     |  | 27.64                               |  |  |
| t Stat  | 1.63      |  |                                     |  |  |
| P(T<=t) two-tail  | 0.35      |  |                                     |  |  |
| t Critical two-tail   | 12.71     |  |                                     |  |  |
| Null Hypothesis:  | Accept H0 |  | No Significant Difference           |  |  |
| Cumulative Water Recovery %:<br>t-Test: Paired Two Sample for Means |           |  | SIBX Vs PAX                         |  |  |
| SIBX (1.3mmol/ton)  |           |  | PAX (1.3mmol/ton)                   |  |  |
| Mean  | 28.82     |  | 25.89                               |  |  |
| t Stat  | 0.74      |  |                                     |  |  |
| P(T<=t) two-tail  | 0.59      |  |                                     |  |  |
| t Critical two-tail   | 12.71     |  |                                     |  |  |
| Null Hypothesis:  | Accept H0 |  | No Significant Difference           |  |  |
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means  |           |  | Industry Vs SIBX                    |  |  |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother                  |           |  | SIBX (1.3mmol/ton)                  |  |  |
| Mean  | 23.50     |  | 20.77                               |  |  |
| t Stat  | 1.14      |  |                                     |  |  |
| P(T<=t) two-tail  | 0.46      |  |                                     |  |  |
| t Critical two-tail   | 12.71     |  |                                     |  |  |
| Null Hypothesis:  | Accept H0 |  | No Significant Difference           |  |  |

|  |  |       |  |  |  |
|--|--|-------|--|--|--|
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  |       | Industry Vs PAX                                    |  |  |
|  |  |       | Industry Mixture (1.3mmol/ton) at 50g/t of frother | PAX (1.3mmol/ton)                      |  |
| Mean   |  | 23.50 |  | 19.82                                  |  |
| t Stat   |  | 2.29  |  |  |  |
| P(T<=t) two-tail   |  | 0.26  |  |  |  |
| t Critical two-tail  |  | 12.71 |  |  |  |
| Null Hypothesis:   |  |       | Accept H0  | No Significant Difference              |  |
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  |       | Industry Vs IPETC                                  |  |  |
|  |  |       | Industry Mixture (1.3mmol/ton) at 50g/t of frother | IPETC (1.3mmol/ton)                    |  |
| Mean   |  | 23.50 |  | 33.78                                  |  |
| t Stat   |  | -2.36 |  |  |  |
| P(T<=t) two-tail   |  | 0.26  |  |  |  |
| t Critical two-tail  |  | 12.71 |  |  |  |
| Null Hypothesis:   |  |       | Accept H0  | No Significant Difference              |  |
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  |       | Industry Vs 95.5%PAX: 4.5% IPETC                   |  |  |
|  |  |       | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (1.3mmol/ton)   |  |
| Mean   |  | 23.50 |  | 23.15                                  |  |
| t Stat   |  | 0.86  |  |  |  |
| P(T<=t) two-tail   |  | 0.55  |  |  |  |
| t Critical two-tail  |  | 12.71 |  |  |  |
| Null Hypothesis:   |  |       | Accept H0  | No Significant Difference              |  |
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  |       | Industry Vs 95.5%PAX: 4.5% IPETC                   |  |  |
|  |  |       | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (0.65mmol/ton)  |  |
| Mean   |  | 23.50 |  | 17.69                                  |  |
| t Stat   |  | 1.88  |  |  |  |
| P(T<=t) two-tail   |  | 0.31  |  |  |  |
| t Critical two-tail  |  | 12.71 |  |  |  |
| Null Hypothesis:   |  |       | Accept H0  | No Significant Difference              |  |
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  |       | Industry Vs 95.5%PAX: 4.5% IPETC                   |  |  |
|  |  |       | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (0.325mmol/ton) |  |
| Mean   |  | 23.50 |  | 18.13                                  |  |
| t Stat   |  | 1.91  |  |  |  |
| P(T<=t) two-tail   |  | 0.31  |  |  |  |
| t Critical two-tail  |  | 12.71 |  |  |  |
| Null Hypothesis:   |  |       | Accept H0  | No Significant Difference              |  |

|  |  |                                     |  |
|--|--|-------------------------------------|--|
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  | Industry Vs 90%PAX: 10% IPETC       |  |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 90% PAX : 10% IPETC (1.3mmol/ton)   |  |
| Mean   | 23.50  | 22.10                               |  |
| t Stat   | 3.34   |                                     |  |
| P(T<=t) two-tail   | 0.19   |                                     |  |
| t Critical two-tail  | 12.71  |                                     |  |
| Null Hypothesis:   | Accept H0  | No Significant Difference           |  |
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  | Industry Vs 90%PAX: 10% IPETC       |  |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 90% PAX : 10% IPETC (0.65mmol/ton)  |  |
| Mean   | 23.50  | 20.78                               |  |
| t Stat   | 7.49   |                                     |  |
| P(T<=t) two-tail   | 0.08   |                                     |  |
| t Critical two-tail  | 12.71  |                                     |  |
| Null Hypothesis:   | Accept H0  | No Significant Difference           |  |
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  | Industry Vs 90%PAX: 10% IPETC       |  |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 90% PAX : 10% IPETC (0.325mmol/ton) |  |
| Mean   | 23.50  | 20.18                               |  |
| t Stat   | 1.57   |                                     |  |
| P(T<=t) two-tail   | 0.36   |                                     |  |
| t Critical two-tail  | 12.71  |                                     |  |
| Null Hypothesis:   | Accept H0  | No Significant Difference           |  |
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  | Industry Vs 85%PAX: 15% IPETC       |  |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 85% PAX : 15% IPETC (1.3mmol/ton)   |  |
| Mean   | 23.50  | 22.28                               |  |
| t Stat   | 6.74   |                                     |  |
| P(T<=t) two-tail   | 0.09   |                                     |  |
| t Critical two-tail  | 12.71  |                                     |  |
| Null Hypothesis:   | Accept H0  | No Significant Difference           |  |
| Cumulative Mass Recovery %:<br>t-Test: Paired Two Sample for Means |  | Industry Vs 85%PAX: 15% IPETC       |  |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 85% PAX : 15% IPETC (0.65mmol/ton)  |  |
| Mean   | 23.50  | 22.26                               |  |
| t Stat   | 0.68   |                                     |  |
| P(T<=t) two-tail   | 0.62   |                                     |  |
| t Critical two-tail  | 12.71  |                                     |  |
| Null Hypothesis:   | Accept H0  | No Significant Difference           |  |

|  |           |                           |
|--|-----------|---------------------------|
| Cumulative Mass Recovery %: Industry Vs 85%PAX: 15% IPETC<br>t-Test: Paired Two Sample for Means |           |                           |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother      85% PAX : 15% IPETC (0.325mmol/ton)      |           |                           |
| Mean   | 23.50     | 21.28                     |
| t Stat   | 6.13      |                           |
| P(T<=t) two-tail   | 0.10      |                           |
| t Critical two-tail  | 12.71     |                           |
| Null Hypothesis:   | Accept H0 | No Significant Difference |
| Cumulative Mass Recovery %: Industry Vs 80%PAX: 20% IPETC<br>t-Test: Paired Two Sample for Means |           |                           |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother      80% PAX : 20% IPETC (1.3mmol/ton)        |           |                           |
| Mean   | 23.50     | 24.97                     |
| t Stat   | -3.12     |                           |
| P(T<=t) two-tail   | 0.20      |                           |
| t Critical two-tail  | 12.71     |                           |
| Null Hypothesis:   | Accept H0 | No Significant Difference |
| Cumulative Mass Recovery %: Industry Vs 50%PAX: 50% SIBX<br>t-Test: Paired Two Sample for Means  |           |                           |
| Industry Mixture (1.3mmol/ton) at 50g/t of frother      50% PAX : 50% SIBX (1.3mmol/ton)         |           |                           |
| Mean   | 23.50     | 20.18                     |
| t Stat   | 1.21      |                           |
| P(T<=t) two-tail   | 0.44      |                           |
| t Critical two-tail  | 12.71     |                           |
| Null Hypothesis:   | Accept H0 | No Significant Difference |

## Appendix G: Statistical Analysis of Cumulative Nickel Grade %

|  |  |                                       |
|--|--|---------------------------------------|
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means | Industry Vs SIBX                                   |                                       |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | SIBX (1.3mmol/ton)                    |
| Mean   | 1.60   | 1.87                                  |
| t Stat   | -2.60  |                                       |
| P(T<=t) two-tail   | 0.23   |                                       |
| t Critical two-tail  | 12.71  |                                       |
| Null Hypothesis:   | Accept H0  | No Significant Difference             |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means | Industry Vs PAX                                    |                                       |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | PAX (1.3mmol/ton)                     |
| Mean   | 1.60   | 1.86                                  |
| t Stat   | -22.88   |                                       |
| P(T<=t) two-tail   | 0.03   |                                       |
| t Critical two-tail  | 12.71  |                                       |
| Null Hypothesis:   | Accept H0  | No Significant Difference             |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means | Industry Vs IPETC                                  |                                       |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | IPETC (1.3mmol/ton)                   |
| Mean   | 1.60   | 1.23                                  |
| t Stat   | 16.54  |                                       |
| P(T<=t) two-tail   | 0.04   |                                       |
| t Critical two-tail  | 12.71  |                                       |
| Null Hypothesis:   | Reject H0  | Significantly Different               |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means | Industry Vs 95.5% PAX : 4.5% IPETC                 |                                       |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (1.3mmol/ton)  |
| Mean   | 1.60   | 1.73                                  |
| t Stat   | -9.64  |                                       |
| P(T<=t) two-tail   | 0.07   |                                       |
| t Critical two-tail  | 12.71  |                                       |
| Null Hypothesis:   | Accept H0  | No Significant Difference             |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means | Industry Vs 95.5% PAX : 4.5% IPETC                 |                                       |
|  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (0.65mmol/ton) |
| Mean   | 1.60   | 2.01                                  |
| t Stat   | -1.55  |                                       |
| P(T<=t) two-tail   | 0.36   |                                       |
| t Critical two-tail  | 12.71  |                                       |
| Null Hypothesis:   | Accept H0  | No Significant Difference             |



|  |  |  |  |
|--|--|--|--|
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means |  | Industry Vs 95.5% PAX : 4.5% IPETC                 |  |
|  |  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (0.325mmol/ton) |
| Mean   |  | 1.60   | 2.06                                   |
| t Stat   |  | -1.90  |  |
| P(T<=t) two-tail   |  | 0.31   |  |
| t Critical two-tail  |  | 12.71  |  |
| Null Hypothesis:   |  | Accept H0  | No Significant Difference              |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means |  | Industry Vs 90% PAX: 10% IPETC (1.3mmol/ton)       |  |
|  |  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 90% PAX: 10% IPETC (1.3mmol/ton)       |
| Mean   |  | 1.60   | 1.75                                   |
| t Stat   |  | -4.48  |  |
| P(T<=t) two-tail   |  | 0.14   |  |
| t Critical two-tail  |  | 12.71  |  |
| Null Hypothesis:   |  | Accept H0  | No Significant Difference              |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means |  | Industry Vs 90% PAX : 10% IPETC                    |  |
|  |  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 90% PAX : 10% IPETC (0.65mmol/ton)     |
| Mean   |  | 1.60   | 1.65                                   |
| t Stat   |  | -0.54  |  |
| P(T<=t) two-tail   |  | 0.68   |  |
| t Critical two-tail  |  | 12.71  |  |
| Null Hypothesis:   |  | Accept H0  | No Significant Difference              |

|  |  |  |                                     |
|--|--|--|-------------------------------------|
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means |  | Industry Vs 90% PAX : 10% IPETC                    |                                     |
|  |  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 90% PAX : 10% IPETC (0.325mmol/ton) |
| Mean   |  | 1.60   | 1.92                                |
| t Stat   |  | -2.16  |                                     |
| P(T<=t) two-tail   |  | 0.28   |                                     |
| t Critical two-tail  |  | 12.71  |                                     |
| Null Hypothesis:   |  | Accept H0  | No Significant Difference           |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means |  | Industry Vs 85% PAX:15% IPETC (1.3mmol/ton)        |                                     |
|  |  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 85% PAX:15% IPETC (1.3mmol/ton)     |
| Mean   |  | 1.60   | 1.75                                |
| t Stat   |  | -2.67  |                                     |
| P(T<=t) two-tail   |  | 0.23   |                                     |
| t Critical two-tail  |  | 12.71  |                                     |
| Null Hypothesis:   |  | Accept H0  | No Significant Difference           |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means |  | Industry Vs 85% PAX : 15% IPETC                    |                                     |
|  |  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 85% PAX : 15% IPETC (0.65mmol/ton)  |
| Mean   |  | 1.60   | 1.71                                |
| t Stat   |  | -1.55  |                                     |
| P(T<=t) two-tail   |  | 0.36   |                                     |
| t Critical two-tail  |  | 12.71  |                                     |
| Null Hypothesis:   |  | Accept H0  | No Significant Difference           |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means |  | Industry Vs 85% PAX : 15% IPETC                    |                                     |
|  |  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 85% PAX : 15% IPETC (0.325mmol/ton) |
| Mean   |  | 1.60   | 1.83                                |
| t Stat   |  | -5.56  |                                     |
| P(T<=t) two-tail   |  | 0.11   |                                     |
| t Critical two-tail  |  | 12.71  |                                     |
| Null Hypothesis:   |  | Accept H0  | No Significant Difference           |
| Cumulative Ni Grade %<br>t-Test: Paired Two Sample for Means |  | Industry Vs 80% PAX:20% IPETC                      |                                     |
|  |  | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 80% PAX:20% IPETC (1.3mmol/ton)     |
| Mean   |  | 1.60   | 1.63                                |
| t Stat   |  | -3.24  |                                     |
| P(T<=t) two-tail   |  | 0.19   |                                     |
| t Critical two-tail  |  | 12.71  |                                     |
| Null Hypothesis:   |  | Accept H0  | No Significant Difference           |

| Cumulative Ni Grade %               |  | Industry Vs 50% PAX:50% SIBX   |  |
|-------------------------------------|--|--------------------------------|--|
| t-Test: Paired Two Sample for Means |  |                                |  |
|                                     | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 50% PAX:50% SIBX (1.3mmol/ton) |  |
| Mean                                | 1.60   | 1.93                           |  |
| t Stat                              | -1.24  |                                |  |
| P(T<=t) two-tail                    | 0.43   |                                |  |
| t Critical two-tail                 | 12.71  |                                |  |
| Null Hypothesis:                    | Accept H0  | No Significant Difference      |  |
| Cumulative Ni Grade %               |  | SIBX Vs PAX                    |  |
| t-Test: Paired Two Sample for Means |  |                                |  |
|                                     | SIBX (1.3mmol/ton)                                 | PAX (1.3mmol/ton)              |  |
| Mean                                | 1.87   | 1.86                           |  |
| t Stat                              | 0.05   |                                |  |
| P(T<=t) two-tail                    | 0.97   |                                |  |
| t Critical two-tail                 | 12.71  |                                |  |
| Null Hypothesis:                    | Accept H0  | No Significant Difference      |  |
| Cumulative Ni Grade %               |  | PAX Vs IPETC                   |  |
| t-Test: Paired Two Sample for Means |  |                                |  |
|                                     | PAX (1.3mmol/ton)                                  | IPETC (1.3mmol/ton)            |  |
| Mean                                | 1.86   | 1.23                           |  |
| t Stat                              | 18.73  |                                |  |
| P(T<=t) two-tail                    | 0.03   |                                |  |
| t Critical two-tail                 | 12.71  |                                |  |
| Null Hypothesis:                    | Reject H0  | Significantly Different        |  |

|                                     |           |  |  |
|-------------------------------------|-----------|--|--|
| Cumulative Ni Grade %               |           | 95.5% PAX :4.5% IPETC Vs 95.5% PAX:4.5% IPETC  |  |
| t-Test: Paired Two Sample for Means |           |  |  |
|                                     |           | 95.5% PAX:4.5% IPETC (1.3mmol/ton)             | 95.5% PAX : 4.5% IPETC (0.65mmol/ton)  |
| Mean                                | 1.73      |  | 2.01                                   |
| t Stat                              | -1.10     |  |  |
| P(T<=t) two-tail                    | 0.47      |  |  |
| t Critical two-tail                 | 12.71     |  |  |
| Null Hypothesis:                    | Accept H0 |  | No Significant Difference              |
| Cumulative Ni Grade %               |           | 95.5% PAX:4.5% IPETC Vs 95.5% PAX:4.5% IPETC   |  |
| t-Test: Paired Two Sample for Means |           |  |  |
|                                     |           | 95.5% PAX:4.5% IPETC (1.3mmol/ton)             | 95.5% PAX : 4.5% IPETC (0.325mmol/ton) |
| Mean                                | 1.73      |  | 2.06                                   |
| t Stat                              | -1.42     |  |  |
| P(T<=t) two-tail                    | 0.39      |  |  |
| t Critical two-tail                 | 12.71     |  |  |
| Null Hypothesis:                    | Accept H0 |  | No Significant Difference              |
| Cumulative Ni Grade %               |           | 95.5% PAX:4.5% IPETC Vs 95.5% PAX : 4.5% IPETC |  |
| t-Test: Paired Two Sample for Means |           |  |  |
|                                     |           | 95.5% PAX : 4.5% IPETC (0.65mmol/ton)          | 95.5% PAX : 4.5% IPETC (0.325mmol/ton) |
| Mean                                | 2.01      |  | 2.06                                   |
| t Stat                              | -1.49     |  |  |
| P(T<=t) two-tail                    | 0.38      |  |  |
| t Critical two-tail                 | 12.71     |  |  |
| Null Hypothesis:                    | Accept H0 |  | No Significant Difference              |
| Cumulative Ni Grade %               |           | 90% PAX:10% IPETC Vs 90% PAX:10% IPETC         |  |
| t-Test: Paired Two Sample for Means |           |  |  |
|                                     |           | 90% PAX:10% IPETC (1.3mmol/ton)                | 90% PAX : 10% IPETC (0.65mmol/ton)     |
| Mean                                | 1.75      |  | 1.65                                   |
| t Stat                              | 0.75      |  |  |
| P(T<=t) two-tail                    | 0.59      |  |  |
| t Critical two-tail                 | 12.71     |  |  |
| Null Hypothesis:                    | Accept H0 |  | No Significant Difference              |
| Cumulative Ni Grade %               |           | 90% PAX:10% IPETC Vs 90% PAX : 10% IPETC       |  |
| t-Test: Paired Two Sample for Means |           |  |  |
|                                     |           | 90% PAX : 10% IPETC (1.3mmol/ton)              | 90% PAX : 10% IPETC (0.325mmol/ton)    |
| Mean                                | 1.75      |  | 1.92                                   |
| t Stat                              | -1.50     |  |  |
| P(T<=t) two-tail                    | 0.38      |  |  |
| t Critical two-tail                 | 12.71     |  |  |
| Null Hypothesis:                    | Accept H0 |  | No Significant Difference              |

|                                     |  |  |  |  |                                     |
|-------------------------------------|--|--|--|--|-------------------------------------|
| Cumulative Ni Grade %               |  |  | 90% PAX : 10% IPETC Vs 90% PAX : 10% IPETC |  |                                     |
| t-Test: Paired Two Sample for Means |  |  |  |  |                                     |
|                                     |  |  | 90% PAX : 10% IPETC (0.65mmol/ton)         |  | 90% PAX : 10% IPETC (0.325mmol/ton) |
| Mean                                |  |  | 1.65                                       |  | 1.92                                |
| t Stat                              |  |  | -1.10                                      |  |                                     |
| P(T<=t) two-tail                    |  |  | 0.47                                       |  |                                     |
| t Critical two-tail                 |  |  | 12.71                                      |  |                                     |
| Null Hypothesis:                    |  |  | Accept H0                                  |  | No Significant Difference           |
| Cumulative Ni Grade %               |  |  | 85% PAX : 15% IPETC Vs 90% PAX : 10% IPETC |  |                                     |
| t-Test: Paired Two Sample for Means |  |  |  |  |                                     |
|                                     |  |  | 85% PAX : 15% IPETC (1.3mmol/ton)          |  | 85% PAX : 15% IPETC (0.65mmol/ton)  |
| Mean                                |  |  | 1.75                                       |  | 1.71                                |
| t Stat                              |  |  | 0.26                                       |  |                                     |
| P(T<=t) two-tail                    |  |  | 0.84                                       |  |                                     |
| t Critical two-tail                 |  |  | 12.71                                      |  |                                     |
| Null Hypothesis:                    |  |  | Accept H0                                  |  | No Significant Difference           |
| Cumulative Ni Grade %               |  |  | 85% PAX : 15% IPETC Vs 90% PAX : 10% IPETC |  |                                     |
| t-Test: Paired Two Sample for Means |  |  |  |  |                                     |
|                                     |  |  | 85% PAX : 15% IPETC (1.3mmol/ton)          |  | 85% PAX : 15% IPETC (0.325mmol/ton) |
| Mean                                |  |  | 1.75                                       |  | 1.83                                |
| t Stat                              |  |  | -6.30                                      |  |                                     |
| P(T<=t) two-tail                    |  |  | 0.10                                       |  |                                     |
| t Critical two-tail                 |  |  | 12.71                                      |  |                                     |
| Null Hypothesis:                    |  |  | Accept H0                                  |  | No Significant Difference           |
| Cumulative Ni Grade %               |  |  | 85% PAX : 15% IPETC Vs 90% PAX : 10% IPETC |  |                                     |
| t-Test: Paired Two Sample for Means |  |  |  |  |                                     |
|                                     |  |  | 85% PAX : 15% IPETC (0.65mmol/ton)         |  | 85% PAX : 15% IPETC (0.325mmol/ton) |
| Mean                                |  |  | 1.71                                       |  | 1.83                                |
| t Stat                              |  |  | -1.02                                      |  |                                     |
| P(T<=t) two-tail                    |  |  | 0.49                                       |  |                                     |
| t Critical two-tail                 |  |  | 12.71                                      |  |                                     |
| Null Hypothesis:                    |  |  | Accept H0                                  |  | No Significant Difference           |

## Appendix H: Statistical Analysis of Cumulative Nickel Recovery%

|                                     |  |                                       |  |
|-------------------------------------|--|---------------------------------------|--|
| Cumulative Ni Recovery %            |  | Industry Vs SIBX                      |  |
| t-Test: Paired Two Sample for Means |  |                                       |  |
|                                     | Industry Mixture (1.3mmol/ton) at 50g/t of frother | SIBX (1.3mmol/ton)                    |  |
| Mean                                | 82.02  | 79.10                                 |  |
| t Stat                              | 1.81   |                                       |  |
| P(T<=t) two-tail                    | 0.32   |                                       |  |
| t Critical two-tail                 | 12.71  |                                       |  |
| Null Hypothesis:                    | Accept H0  | No Significant Difference             |  |
| Cumulative Ni Recovery %            |  | Industry Vs PAX                       |  |
| t-Test: Paired Two Sample for Means |  |                                       |  |
|                                     | Industry Mixture (1.3mmol/ton) at 50g/t of frother | PAX (1.3mmol/ton)                     |  |
| Mean                                | 82.02  | 77.01                                 |  |
| t Stat                              | 9.02   |                                       |  |
| P(T<=t) two-tail                    | 0.07   |                                       |  |
| t Critical two-tail                 | 12.71  |                                       |  |
| Null Hypothesis:                    | Accept H0  | No Significant Difference             |  |
| Cumulative Ni Recovery %            |  | Industry Vs IPETC                     |  |
| t-Test: Paired Two Sample for Means |  |                                       |  |
|                                     | Industry Mixture (1.3mmol/ton) at 50g/t of frother | IPETC (1.3mmol/ton)                   |  |
| Mean                                | 82.02  | 82.19                                 |  |
| t Stat                              | -0.07  |                                       |  |
| P(T<=t) two-tail                    | 0.96   |                                       |  |
| t Critical two-tail                 | 12.71  |                                       |  |
| Null Hypothesis:                    | Accept H0  | No Significant Difference             |  |
| Cumulative Ni Recovery %            |  | Industry Vs 95.5% PAX : 4.5% IPETC    |  |
| t-Test: Paired Two Sample for Means |  |                                       |  |
|                                     | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (1.3mmol/ton)  |  |
| Mean                                | 82.02  | 84.55                                 |  |
| t Stat                              | -0.66  |                                       |  |
| P(T<=t) two-tail                    | 0.63   |                                       |  |
| t Critical two-tail                 | 12.71  |                                       |  |
| Null Hypothesis:                    | Accept H0  | No Significant Difference             |  |
| Cumulative Ni Recovery %            |  | Industry Vs 95.5% PAX : 4.5% IPETC    |  |
| t-Test: Paired Two Sample for Means |  |                                       |  |
|                                     | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (0.65mmol/ton) |  |
| Mean                                | 82.02  | 77.45                                 |  |
| t Stat                              | 0.78   |                                       |  |
| P(T<=t) two-tail                    | 0.58   |                                       |  |
| t Critical two-tail                 | 12.71  |                                       |  |
| Null Hypothesis:                    | Accept H0  | No Significant Difference             |  |

|   |       |  |  |
|---|-------|--|--|
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means   |       | Industry Vs 95.5% PAX : 4.5% IPETC                 |  |
|   |       | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 95.5% PAX : 4.5% IPETC (0.325mmol/ton) |
| Mean  | 82.02 | 76.24  |  |
| t Stat  | 12.63 |  |  |
| P(T<=t) two-tail  | 0.05  |  |  |
| t Critical two-tail   | 12.71 |  |  |
| Null Hypothesis:  |       | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery (%)<br>t-Test: Paired Two Sample for Means |       | Industry Vs 90% PAX : 10% IPETC                    |  |
|   |       | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 90% PAX: 10% IPETC (1.3mmol/ton)       |
| Mean  | 82.02 | 80.16  |  |
| t Stat  | 1.17  |  |  |
| P(T<=t) two-tail  | 0.45  |  |  |
| t Critical two-tail   | 12.71 |  |  |
| Null Hypothesis:  |       | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery (%)<br>t-Test: Paired Two Sample for Means |       | Industry Vs 90% PAX : 10% IPETC                    |  |
|   |       | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 90% PAX : 10% IPETC (0.65mmol/ton)     |
| Mean  | 82.02 | 69.59  |  |
| t Stat  | 1.66  |  |  |
| P(T<=t) two-tail  | 0.34  |  |  |
| t Critical two-tail   | 12.71 |  |  |
| Null Hypothesis:  |       | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery (%)<br>t-Test: Paired Two Sample for Means |       | Industry Vs 90% PAX : 10% IPETC                    |  |
|   |       | Industry Mixture (1.3mmol/ton) at 50g/t of frother | 90% PAX : 10% IPETC (0.325mmol/ton)    |
| Mean  | 82.02 | 77.83  |  |
| t Stat  | 0.63  |  |  |
| P(T<=t) two-tail  | 0.64  |  |  |
| t Critical two-tail   | 12.71 |  |  |
| Null Hypothesis:  |       | Accept H0  | No Significant Difference              |

|                                     |   |                                 |  |
|-------------------------------------|---|---------------------------------|--|
| Cumulative Ni Recovery (%)          |   | Industry Vs 85% PAX : 15% IPETC |  |
| t-Test: Paired Two Sample for Means |   |                                 |  |
|                                     | <i>Industry Mixture (1.3mmol/ton) at 50g/t of frother</i> |                                 | <i>85% PAX:15% IPETC (1.3mmol/ton)</i>     |
| Mean                                | 82.02   |                                 | 80.27                                      |
| t Stat                              | 1.06  |                                 |  |
| P(T<=t) two-tail                    | 0.48  |                                 |  |
| t Critical two-tail                 | 12.71   |                                 |  |
| Null Hypothesis:                    | Accept H0   |                                 | No Significant Difference                  |
| Cumulative Ni Recovery (%)          |   | Industry Vs 85% PAX : 15% IPETC |  |
| t-Test: Paired Two Sample for Means |   |                                 |  |
|                                     | <i>Industry Mixture (1.3mmol/ton) at 50g/t of frother</i> |                                 | <i>85% PAX : 15% IPETC (0.65mmol/ton)</i>  |
| Mean                                | 82.02   |                                 | 80.25                                      |
| t Stat                              | 0.66  |                                 |  |
| P(T<=t) two-tail                    | 0.63  |                                 |  |
| t Critical two-tail                 | 12.71   |                                 |  |
| Null Hypothesis:                    | Accept H0   |                                 | No Significant Difference                  |
| Cumulative Ni Recovery (%)          |   | Industry Vs 85% PAX : 15% IPETC |  |
| t-Test: Paired Two Sample for Means |   |                                 |  |
|                                     | <i>Industry Mixture (1.3mmol/ton) at 50g/t of frother</i> |                                 | <i>85% PAX : 15% IPETC (0.325mmol/ton)</i> |
| Mean                                | 82.02   |                                 | 79.60                                      |
| t Stat                              | 110.90  |                                 |  |
| P(T<=t) two-tail                    | 0.01  |                                 |  |
| t Critical two-tail                 | 12.71   |                                 |  |
| Null Hypothesis:                    | Reject H0   |                                 | Significantly Different                    |
| Cumulative Ni Recovery %            |   | Industry Vs 80% PAX : 20% IPETC |  |
| t-Test: Paired Two Sample for Means |   |                                 |  |
|                                     | <i>Industry Mixture (1.3mmol/ton) at 50g/t of frother</i> |                                 | <i>80% PAX:20% IPETC (1.3mmol/ton)</i>     |
| Mean                                | 82.02   |                                 | 82.65                                      |
| t Stat                              | -0.30   |                                 |  |
| P(T<=t) two-tail                    | 0.81  |                                 |  |
| t Critical two-tail                 | 12.71   |                                 |  |
| Null Hypothesis:                    | Accept H0   |                                 | No Significant Difference                  |
| Cumulative Ni Recovery %            |   | Industry Vs 50% PAX : 50% SIBX  |  |
| t-Test: Paired Two Sample for Means |   |                                 |  |
|                                     | <i>Industry Mixture (1.3mmol/ton) at 50g/t of frother</i> |                                 | <i>50% PAX:50% SIBX (1.3mmol/ton)</i>      |
| Mean                                | 82.02   |                                 | 78.66                                      |
| t Stat                              | 2.50  |                                 |  |
| P(T<=t) two-tail                    | 0.24  |                                 |  |
| t Critical two-tail                 | 12.71   |                                 |  |
| Null Hypothesis:                    | Accept H0   |                                 | No Significant Difference                  |



| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means |                                    | SIBX Vs PAX                                      |  |
|---|------------------------------------|--|--|
|   | SIBX (1.3mmol/ton)                 | PAX (1.3mmol/ton)                                |  |
| Mean  | 79.10                              | 77.01  |  |
| t Stat  | 0.97                               |  |  |
| P(T<=t) two-tail  | 0.51                               |  |  |
| t Critical two-tail   | 12.71                              |  |  |
| Null Hypothesis:  | Accept H0                          | No Significant Difference                        |  |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means |                                    | SIBX Vs IPETC                                    |  |
|   | SIBX (1.3mmol/ton)                 | IPETC (1.3mmol/ton)                              |  |
| Mean  | 79.10                              | 82.19  |  |
| t Stat  | -0.77                              |  |  |
| P(T<=t) two-tail  | 0.58                               |  |  |
| t Critical two-tail   | 12.71                              |  |  |
| Null Hypothesis:  | Accept H0                          | No Significant Difference                        |  |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means |                                    | PAX Vs IPETC                                     |  |
|   | PAX (1.3mmol/ton)                  | IPETC (1.3mmol/ton)                              |  |
| Mean  | 77.01                              | 82.19  |  |
| t Stat  | -2.80                              |  |  |
| P(T<=t) two-tail  | 0.22                               |  |  |
| t Critical two-tail   | 12.71                              |  |  |
| Null Hypothesis:  | Accept H0                          | No Significant Difference                        |  |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means |                                    | 95.5% PAX : 4.5% IPETC Vs 95.5% PAX : 4.5% IPETC |  |
|   | 95.5% PAX:4.5% IPETC (1.3mmol/ton) | 95.5% PAX : 4.5% IPETC (0.65mmol/ton)            |  |
| Mean  | 84.55                              | 77.45  |  |
| t Stat  | 0.73                               |  |  |
| P(T<=t) two-tail  | 0.60                               |  |  |
| t Critical two-tail   | 12.71                              |  |  |
| Null Hypothesis:  | Accept H0                          | No Significant Difference                        |  |

|   |  |  |
|---|--|--|
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means | 95.5% PAX : 4.5% IPETC Vs 95.5% PAX : 4.5% IPETC |  |
|   | 95.5% PAX : 4.5% IPETC (1.3mmol/ton)             | 95.5% PAX : 4.5% IPETC (0.325mmol/ton) |
| Mean  | 84.55  | 76.24                                  |
| t Stat  | 1.94   |  |
| P(T<=t) two-tail  | 0.30   |  |
| t Critical two-tail   | 12.71  |  |
| Null Hypothesis:  | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means | 95.5% PAX : 4.5% IPETC Vs 95.5% PAX : 4.5% IPETC |  |
|   | 95.5% PAX : 4.5% IPETC (0.65mmol/ton)            | 95.5% PAX : 4.5% IPETC (0.325mmol/ton) |
| Mean  | 77.45  | 76.24                                  |
| t Stat  | 0.23   |  |
| P(T<=t) two-tail  | 0.86   |  |
| t Critical two-tail   | 12.71  |  |
| Null Hypothesis:  | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means | 90% PAX : 10% IPETC Vs 95.5% PAX : 4.5% IPETC    |  |
|   | 90% PAX : 10% IPETC (1.3mmol/ton)                | 90% PAX : 10% IPETC (0.65mmol/ton)     |
| Mean  | 80.16  | 69.59                                  |
| t Stat  | 1.80   |  |
| P(T<=t) two-tail  | 0.32   |  |
| t Critical two-tail   | 12.71  |  |
| Null Hypothesis:  | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means | 90% PAX : 10% IPETC Vs 95.5% PAX : 4.5% IPETC    |  |
|   | 90% PAX : 10% IPETC (1.3mmol/ton)                | 90% PAX : 10% IPETC (0.325mmol/ton)    |
| Mean  | 80.16  | 77.83                                  |
| t Stat  | 0.46   |  |
| P(T<=t) two-tail  | 0.72   |  |
| t Critical two-tail   | 12.71  |  |
| Null Hypothesis:  | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means | 90% PAX : 10% IPETC Vs 95.5% PAX : 4.5% IPETC    |  |
|   | 90% PAX : 10% IPETC (0.65mmol/ton)               | 90% PAX : 10% IPETC (0.325mmol/ton)    |
| Mean  | 69.59  | 77.83                                  |
| t Stat  | -9.64  |  |
| P(T<=t) two-tail  | 0.07   |  |
| t Critical two-tail   | 12.71  |  |
| Null Hypothesis:  | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means | 85% PAX : 15% IPETC Vs 85% PAX : 15% IPETC       |  |
|   | 85% PAX : 15% IPETC (1.3mmol/ton)                | 85% PAX : 15% IPETC (0.65mmol/ton)     |
| Mean  | 80.27  | 80.25                                  |
| t Stat  | 0.01   |  |
| P(T<=t) two-tail  | 1.00   |  |
| t Critical two-tail   | 12.71  |  |
| Null Hypothesis:  | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means | 85% PAX : 15% IPETC Vs 85% PAX : 15% IPETC       |  |
|   | 85% PAX : 15% IPETC (1.3mmol/ton)                | 85% PAX : 15% IPETC (0.325mmol/ton)    |
| Mean  | 80.27  | 81.26                                  |
| t Stat  | -0.86  |  |
| P(T<=t) two-tail  | 0.55   |  |
| t Critical two-tail   | 12.71  |  |
| Null Hypothesis:  | Accept H0  | No Significant Difference              |
| Cumulative Ni Recovery %<br>t-Test: Paired Two Sample for Means | 85% PAX : 15% IPETC Vs 85% PAX : 15% IPETC       |  |
|   | 85% PAX : 15% IPETC (0.65mmol/ton)               | 85% PAX : 15% IPETC (0.325mmol/ton)    |
| Mean  | 80.25  | 79.60                                  |
| t Stat  | 0.24   |  |
| P(T<=t) two-tail  | 0.85   |  |
| t Critical two-tail   | 12.71  |  |
| Null Hypothesis:  | Accept H0  | No Significant Difference              |