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A STUDY INTO THE USE OF A FATTY ACID AND XANTHATE COLLECTOR COMBINATION IN THE RECOVERY OF BASE METAL SULPHIDES: A HOLISTIC APPROACH

By

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Abstract

In this study, a collector combination between xanthate and a fatty acid collector (Betacol 364) is tested on an ore containing chalcopyrite, pentlandite and pyrrhotite. An increase in the rate of pentlandite and pyrrhotite recovery was measured when the fatty acid and xanthate combination was tested against the baseline (xanthate as sole collector), using the standard laboratory scale batch flotation test procedure.

The rate of mineral recovery in a batch flotation test is not only influenced by particle floatability, but also by the amount of air bubble surface area available in the pulp and the recovery of value mineral from the flotation froth. This is given as $k = P.S_b.R_f$ where k is the rate constant (min^{-1}), P is a parameter which represents the floatability of a mineral, R_f is the froth recovery factor and S_b bubble surface area flux.

A test unit developed for this study (based on the Bikerman foam test) indicated that the fatty acid collector used (Betacol 364) has froth stabilising characteristics and thus may have increased the froth recovery (R_f) during the batch flotation test. The increase in the rate of pyrrhotite recovery measured can therefore not only be explained by an increase in pyrrhotite floatability. In a case where the batch flotation tests are followed by plant testing and the rate of flotation increase measured during the batch test was only or partially as a result of an increase in froth recovery (R_f), the batch test would be an unreliable test to predict plant performance.

Plant operators have many variables available to manage froth recovery (R_f), for example pulp level and frother dosing rate. For a laboratory scale batch test to be a reliable test for predicting a rate increase in plant recovery, it is necessary that an increase in the rate of recovery will be as a result of an increase in particle floatability (P). It is recommended that a collector batch flotation test campaign

must include a test to measure if the collector has frothing characteristics. The Bikerman froth stability column is demonstrated as a possible test to measure if a collector has froth stabilising characteristics.

The Bikerman froth stability column test method confirmed that the fatty acid collector tested (Betacol 364) has froth stabilising properties. Additional tests are therefore recommended to confirm that the rate of recovery increase measured is as a result of an increase in pyrrhotite and pentlandite floatability (P). Micro flotation tests performed on pyrrhotite mineral specimens confirmed that a combination between Xanthate and Betacol 364 increases the particle floatability $(P)$of pyrrhotite. Laboratory scale flotation tests performed on artificial ores, consisting of a mixture of quartz and pyrrhotite mineral specimens, confirmed that the combination of Betacol 364 and xanthate increases the floatability of pyrrhotite (P).

TABLE OF CONTENT

1. Introduction 1

2. Research problem and objectives 3

3. Literature review 4

3.1. Testing collector combinations 4

3.2. Using the kinetic model for froth flotation to predict and anticipate the possible effect a collector change may have on laboratory and plant test results 5

3.3. Bench flotation test 10

3.3.1. Equipment 10

3.3.2. Ore sampling 11

3.3.3. Water quality and temperature 11

3.3.4. Entrainment (mechanical carryover) 12

3.3.5. Milling and conditioning 13

3.3.6. Reagents 15

3.3.7. Interpretation of bench-scale flotation test data 16

3.3.8. Quality control of batch-scale flotation test data 18

3.4. Bikerman froth stability column 19

3.5. Measurement of bubble size in a flotation pulp 20

3.6. Testing of mineral floatability by micro flotation 20

3.7. Flotation of chalcopyrite, pyrrhotite and pentlandite 22

3.7.1. Flotation of oxidised minerals 28

3.7.2. Effect of stiogemetry on the flotation of pyrrhotite 32

3.7.3. Flotation of chalcopyrite, pentlandite and pyrrhotite with a mixture of xanthates or mixture with xanthate and a co-collector 33

3.8. Plant testing of reagents 34

3.8.1. Alternating ON-OFF testing 35

3.8.2. Before, during and after testing of collector combinations on plant scale 36

3.8.3. Analysis of Variance (ANOVA) 37

3.8.4. Analysing plant data by comparing confidence intervals 37

3.8.5. Test size and plant test duration 38

4. Experimental procedure 41
4.1. Introduction 41
4.2. Bench flotation test 41
4.2.1. Description of experimental procedure 41
4.2.2. Verification of method 46
4.2.2.1. Reproducibility of results 46
4.2.2.2. Comparison with plant and published data 48
4.3. Bikerman froth stability column 50
4.3.1. Design of Bikerman froth stability column 50
4.3.2. Description of experimental procedure 57
4.3.3. Verification of method 58
4.3.3.1. Reproducibility of results 58
4.3.3.2. Comparison with published data 61
4.4. Micro flotation cell 64
4.4.1. Design of the Bubble-Flux micro flotation cell 64
4.4.2. Description of experimental procedure 71
4.4.3. Verification of method 73
4.4.3.1. Reproducibility of results 73
4.4.3.2. Comparison with published data 74
4.4.3.3. Effect of entrainment 76
4.5. Pilot plant test of fatty acid based co-collector (Betacol 364). Description of method used. 80
4.6. Short plant scale test (crash test) of fatty acid co-collector (Betacol 364). Description of method used. 81
4.7. Analytical techniques 82
4.8. Description of flotation reagents used 83
4.8.1. Xanthate 83
4.8.2. Copper Sulphate 83
4.8.3. Betacol 364 83
4.8.4. Betacol 316 83
4.8.5. Betacol 44A 83
4.8.6. Betacol 25 83

4.9. Characterisation of ore samples used 84
4.9.1. Pyrrhotite 84
4.9.2. Chalcopyrite 87
4.9.3. Quartz 90
4.9.4. Laboratory prepared mixed ore 92
4.9.5. Nickel ore 92
4.9.6. Description of ore sample from of a South African UG2 concentrator. 98
5. Results and discussion 104
5.1. Laboratory scale batch flotation test using a base metal sulphide ore 104
5.2. Investigation into the effect a collector and co-collector combination has on froth characteristics 127
5.3. Laboratory scale batch flotation test using a synthetic base metal sulphide ore consisting of quartz and chalcopyrite or pyrrhotite 133
5.4. Investigation into the floatability of chalcopyrite and pyrrhotite with xanthate and co-collectors as flotation collectors using a micro flotation technique 140
5.5. Short pilot plant test of a fatty acid co-collector (Betacol 364) against the baseline condition being; no co-collector. 151
5.6. Short term (crash test) plant test of a fatty acid co-collector (Betacol 364) against the baseline condition being; no co-collector. 154
5.6.1. Interpretation of plant results using the t-test. 154
5.6.2. Interpretation of plant results by ANOVA. 159
5.6.3. Interpretation of plant results by confidence intervals. 161
5.6.4. Summary of results and conclusions drawn from this set of tests. 167
6. Conclusions and recommendations 168
7. Appendix A: Analytical techniques i
7.1. Sodium peroxide digestion with AAS finish i
7.1.1. Description of method i
7.1.2. Validation of method iii
7.2. Leco sulphur analyser v

7.2.1. Description of method v
7.2.2. Validation of method v
7.3. Scanning Electron Microscope with EDS v
7.3.1. Preparation of 25 mm polished stub v
7.3.2. Description method to study a polished stub using a SEM/EDS. vii
7.4. XRD identification and quantification ix
7.4.1. Description of method ix
8. Appendix B: Methods used to purify xanthate and measure xanthate activity ix
8.1. Purification of xanthate ix
8.1.1. Description of method ix
8.1.2. Validation of method x
8.2. Measuring activity of xanthate by titration Acetone and Phenolphthalein titration methods. xi
8.3. Method to determine the activity of a xanthate solution xiv
9. Appendix C: Test data xviii
9.1. Bench flotation test with natural ore containing chalcopyrite, pentlandite and pyrrhotite xviii
9.2. Mineral grade and recovery relationships determined by XRD quantification xix
9.3. Mineral grade and recovery calculated from assay results xx
9.4. Investigation into the recovery of chalcopyrite and pyrrhotite from a synthetic ore consisting of value mineral and quartz xxiii
9.5. Short pilot plant test of a fatty acid co-collector (Betacol 364) against the baseline condition being; no co-collector. xxv
10. Additional Notes xxvi
10.1. Ore sampling xxvi

TABLE OF FIGURES

Figure 1: Collectorless flotation selectivity between chalcopyrite, pentlandite and pyrrhotite under various conditions**. (Kelebek, et al., 1996) 23

Figure 2: Flotation recovery of a typical Merensky ore without the addition of a collector. (Newell, et al., 2006) 24

Figure 3: Effect of collector Sodium Isobutyl Xanthate (SIBX) concentration on the flotation of sulphide minerals in unoxidised Merensky ore samples (Newell, et al., 2006) 25

Figure 4: Flotation recovery vs. time after stainless steel (SS) milling and no copper sulphate addition. (30 g/t Potassium Amyl Xanthate (PAX) and 30 g/t Dithiophosphate (DTP) (Bradshaw, et al., 2006) 26

Figure 5: Iron sulphide (pyrrhotite) grade vs. recovery obtained for flotation tests with ore milled with mild steel (MS) and stainless steel (SS) milling media with 50 g/t copper sulphate as specified ($CuSO_4$). (30 g/t potassium amyl xanthate (PAX) and 30 g/t DTP) (Bradshaw, et al., 2006) 27

Figure 6: Ni(pentlandite) grade vs. recovery obtained for flotation tests with ore milled with mild steel (MS) and stainless steel (SS) milling media with 50 g/t copper sulphate as specified ($CuSO_4$). (30 g/t PAX and 30 g/t DTP) (Bradshaw, et al., 2006) 27

Figure 7: Cu (chalcopyrite) grade vs. recovery obtained for flotation tests with ore milled with mild steel (MS) and stainless steel (SS) milling media with 50 g/t copper sulphate as specified ($CuSO_4$). (30 g/t PAX and 30 g/t DTP) (Bradshaw, et al., 2006) 28

Figure 8: Effect of oxidation upon sulphide mineral flotation recoveries-Merensky ore. (Ultrasonic treatment and 1.37×10^{-2} M SIBX) (Newell, et al., 2007). 29

Figure 9: Pentlandite recovery-time for fresh ore and stockpile sample - On a ping area of Falconbridge Ltd (Kelebek & Nanthakumar, 2007) 30

Figure 10: Pyrrhotite recovery-time for fresh ore and stockpile sample - On a ping area of Falconbridge Ltd (Kelebek & Nanthakumar, 2007)............................30

Figure 11: Comparison of the final flotation mass recovery and reactivity number (RN) for the different Pyrrhotite samples at pH 7. (Becker, et al., 2010)..32

Figure 12: Comparison of the final flotation mass recovery and reactivity number (RN) for the different Pyrrhotite samples at pH 7 (Becker, et al., 2010)...33

Figure 13: The number of samples needs to be taken (x-axis) can be read if the approximate standard deviation of the data is known (legend) and required error is known (y-axis) for a 90% confidence interval. 39

Figure 14: Copper recovery results for 4 repeat bench flotation tests using a nickel ore described in section 4.9.5...48

Figure 15: Nickel recovery results for 4 repeat bench flotation tests using a nickel ore described in section 4.9.5...49

Figure 16: Histogram of copper recovery. Plant shift production data and laboratory –scale replica test for a nickel ore described in section 4.9.5 49

Figure 17: Histogram of nickel recovery. Plant shift production data and laboratory –scale replica test for a nickel ore described in section 4.9.5 50

Figure 18: Pump curve for QDN 1.5-4.5-0.08 kw submersible pump 52

Figure 19: Rise velocity for a single air bubble measured in water and in glycerol (Talaia, 2007). 53

Figure 20: Flow diagram for the Bikerman froth stability column 55

Figure 21: GA drawing of the Bikerman froth stability column manufactured by Betachem (Pty) Ltd (left and right view). 56

Figure 22: Photos of the Bikerman froth stability column (manufactured by Betachem (Pty) Ltd) in operation...57

Figure 23: Stable froth height measured with Bikerman froth stability column for 5 repeats**..59

Figure 24: Stable froth height measured with Bikerman froth stability column for 5 repeats**..61

Figure 25: Foamability versus concentration profile of MIBC at a range of gas flow rates (Tan, et al., 2005) 62

Figure 26: The steady state foam height of the PPG frothers (measured with a froth stability column) compared to MIBC as a function of the specific gas flow rate (concentration 20 ppm) at a range of gas flow rates. (Tan, et al., 2005) 63

Figure 27: Stable froth height measured for frothers consisting of Dowfroth 200 (medium strong frother), Dowfroth 250 (strong frother) to Dowfroth 400 (very strong frother) using the Bikerman froth stability column**. 63

Figure 28: Stable froth height measured for three talc depressants** 64

Figure 29: Flow diagram for the bubble flux micro flotation unit 70

Figure 30: Photograph of the micro flotation cell used in this study. 71

Figure 31: micro flotation test with chalcopyrite and pyrrhotite mineral specimens crushed and screened to +75 and -106 micron and floated directly after size reduction**. 75

Figure 32: Flotation recovery of a typical Merensky ore without the addition of collector (Newell, et al., 2006) 75

Figure 33: Micro flotation test with quartz and particle size limits as indicated** 78

Figure 34: Micro flotation test with quartz and particle size limits as indicated** 78

Figure 35: Micro flotation test with Quartz at frother concentration as indicated** 79

Figure 36: Analysis of pyrrhotite and magnetite particle by EDS. Pyrrhotite specimen from Munali Mountains. (The EXD does not detect light elements like Oxygen) 85

Figure 37: SEM images for pyrrhotite specimen from the Munali Mountains in Zambia after dry milling to 212 micron and magnetic fraction separate with rare earth magnet**. 86

Figure 38: Analysis of chalcopyrite and sphalerite particles by EDS. 88

Figure 39: SEM images for chalcopyrite specimen from Ward's natural Science after dry milling to 212 micron **. 89

Figure 40: Analysis of quartz particle by EDS. (The EXD does not detect light elements like Oxygen). 91

Figure 41: SEM images for quartz specimen after dry milling to 212 micron and magnetic fraction separate with rare earth magnet**. 92

Figure 42: Analysis of pyrrhotite, chalcopyrite and pentlandite particle by EDS. Natural ore specimen from South African nickel mine. 94

Figure 43: Scanning electron microscope images of nickel ore ROM sample milled in laboratory to 75% passing 75 micron**. 96

Figure 44: Histogram of the nickel content in pentlandite (atom %) as measured by the EDS attached to the Jeol 6400 SEM used. Results for 30 randomly selected pentlandite particles. 98

Figure 45: Copper recovery and time plot for bench scale flotation test. Natural ore**........ 107

Figure 46: Copper grade and recovery plot for bench scale flotation test. Natural ore**........ 108

Figure 47: Nickel recovery and time plot for bench scale flotation test. Natural ore**........ 109

Figure 48: Nickel grade and recovery plot for bench scale flotation test. Natural ore**........ 110

Figure 49: Sulphide sulphur recovery and time plot for bench scale flotation test** 112

Figure 50: Sulphide grade and recovery plot for bench scale flotation test**113

Figure 51: Mineral concentration measured by quantitative XRD (y axis) compared to calculate mineral concentration from Cu and Ni assay (x axis)........ 116

Figure 52: Chalcopyrite recovery and time plot for bench scale flotation test calculated from Cu, Ni and S assay values**........ 117

Figure 53: Chalcopyrite recovery and time plot for bench scale flotation test** 118

Figure 54: Pentlandite recovery and time plot for bench scale flotation test calculated from Cu, Ni and S assay values**........119

Figure 55: Pentlandite recovery and time plot for bench scale flotation test** 120

Figure 56: Pyrrhotite recovery and time plot for bench scale flotation test calculated from Cu, Ni and S assay values**........121

Figure 57: Pyrrhotite recovery and time plot for bench scale flotation test** 122

Figure 58: Gangue (non sulphides) recovery and time plot for bench scale flotation test calculated from Cu, Ni and S assay values** 123

Figure 59: Gangue (non-sulphide) recovery and time plot for bench scale flotation test**........124

Figure 60: Stable froth height measured using the Bikerman froth stability column** 129

Figure 61: Stable froth height measured using the Bikerman froth stability column** 131

Figure 62: Stable froth height measured using the Bikerman froth stability column** 132

Figure 63: Bench flotation test using D12 equipment. Ore tested is a lab mixture of 90% quartz and 10% chalcopyrite**........136

Figure 64: Bench flotation test using D12 equipment. Ore tested is a lab mixture of 90% quartz and 10% chalcopyrite**........137

Figure 65: Bench flotation test using D12 equipment. Ore tested is a lab mixture of 90% quartz and 10% pyrrhotite**. 138

Figure 66: Bench flotation test using D12 equipment. Ore tested is a lab mixture of 90% quartz and 10% pyrrhotite**. 139

Figure 67: Micro flotation test results using micro-bubble flux micro flotation equipment. Quartz ore specimen dry milled to -106 ,+ 75 micron and left in air for 14 days before flotation (aged surface)**. 142

Figure 68: Micro flotation test results using micro-bubble flux micro flotation equipment. Chalcopyrite ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)** 143

Figure 69: Micro flotation test results using micro-bubble flux micro flotation equipment. Chalcopyrite ore specimen dry milled to -106 ,+ 75 micron and left in air for 14 days before flotation (aged surface)**. 144

Figure 70: Micro flotation test results using micro-bubble flux micro flotation equipment. Pyrrhotite ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**. 145

Figure 71: Micro flotation test results using micro-bubble flux micro flotation equipment. Pyrrhotite ore specimen dry milled to -106 ,+ 75 micron and left in air for 14 days before flotation (aged surface)**. 146

Figure 72: Micro flotation test results using micro-bubble flux micro flotation equipment. Pyrrhotite ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**. 147

Figure 73: Micro flotation test results using micro-bubble flux micro flotation equipment. Quartz ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**. 148

Figure 74: Micro flotation test results using micro-bubble flux micro flotation equipment. Pyrrhotite ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**. 149

Figure 75: Micro flotation test results using micro-bubble flux micro flotation equipment. Quartz ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**. 150

Figure 76: Copper assay data from pilot plant flotation test. Test -1 and test -2 are replica tests of baseline condition plus 50 g/t Betacol 346 **. 152

Figure 77: Nickel assay data from pilot plant flotation test. Test -1 and test -2 are replica tests of baseline condition plus 50 g/t Betacol 346 **. 152

Figure 78: Copper assay data from pilot plant flotation test. Test -1 and test -2 are replica tests of baseline condition plus 50 g/t Betacol 346 **. 153

Figure 79: Nickel assay data from pilot plant flotation test. Test -1 and test -2 are replica tests of baseline condition plus 50 g/t Betacol 346 **. 153

Figure 80: PGM (4E) g/t feed grade measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period. 163

Figure 81: PGM (4E) g/t final concentrate grade measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period. 164

Figure 82: PGM (4E) g/t final flotation tails grade measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period. 165

Figure 83: PGM (4E) g/t plant recovery measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was

added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period. 166

Figure 84: Sulphur (by Leco) final concentrate grade measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for sulphur by Leco. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period. 167

Figure 85: Photo of Struers grinding and polishing equipment used to prepare 25 mm polished stubs. vi

Figure 86: Example of 25 mm polished stubs prepared for SEM analysis. vi

Figure 87: Photo of gold sputter coater used to coat polished stubs with Gold vii

Figure 88: Joel JSM -6300 SEM with EXD used. This instrument is located at IMMRI a division of the University of Pretoria viii

Figure 89: Relative sampling precision calculated using the Gy equation for a chalcopyrite ore example xxix

Figure 90: Relative sampling precision calculated using the Gy equation for a pentlandite ore example. xxix

Figure 91: Relative sampling precision calculated using the Gy equation for a pyrrhotite ore example xxx

Figure 92: Relative sampling precision calculated using the Gy equation for a gold containing ore sample. xxx

Figure 93: Relative sampling precision calculated using the modified Gy equation (n-=1.5) for a gold containing ore sample xxxi

Figure 94: Relative sampling precision calculated for taking a subsample form a 100 kg sample, using the Gy equation for a Chalcopyrite ore example............xxxii

Figure 95 : Relative sampling precision calculated for taking a subsample form a 10 kg sample, using the Gy equation for a Chalcopyrite ore example.............xxxiii

TABLE OF TABLES

Table 1: Example of the flotation sequence where 5 minutes wet milling is required and xanthate is added after milling followed by 1 minute conditioning time. 45

Table 2: Four repeat bench flotation results conducted using a nickel ore containing pentlandite, chalcopyrite and pyrrhotite**. 47

Table 3: Four repeat bench flotation results conducted using a nickel ore containing pentlandite, chalcopyrite and pyrrhotite**. 47

Table 4: Four repeat bench flotation results conducted using a nickel ore containing pentlandite, chalcopyrite and pyrrhotite **. 47

Table 5: Stable froth height measured with Bikerman froth stability column for 5 repeats**. 59

Table 6: Stable froth height measured with the Bikerman froth stability column for 5 repeats** 60

Table 7: Percentage standard deviation for repeat tests done using the micro flotation cell. Purified sodium isopropyl xanthate (P-SIPX) at 3.3x 10^{-6} mol/l (50 g/t solids). Minerals used and treatment are as indicated. 74

Table 8: Entrainment quantified with the micro flotation cell using quartz with particles size as indicated and Betafroth 190 concentration of 0.03 g/l. 77

Table 9: Entrainment quantified with the micro flotation cell using quartz with particles size as indicated and Betafroth 190 concentration of 0.06 g/l. 77

Table 10: Mineral identification and quantification by XRD for a pyrrhotite specimen from the Munali Mountains in Zambia 84

Table 11: Mineral identification and quantification by XRD for a chalcopyrite sample obtained from Ward's Natural Science 87

Table 12: Mineral identification and quantification by XRD for a quartz sample after iron removal by magnetic separation 90

Table 13: Mineral identification and quantification by XRD for natural ore sample for a South African nickel mine. 93

Table 14: Characteristics of 50 sulphide particles randomly selected from a nickel ore Run of mine (ROM) sample milled in laboratory to 70% passing 75 micron 97

Table 15: Quantitative XRD results for UG2 final concentrate 99

Table 16: Selected SEM images of a South African platinum concentrator final flotation concentrate. 100

Table 17: Selected SEM images of a South African Platinum concentrator final flotation tails 102

Table 18: Summary of bench scale flotation test results**. 106

Table 19: Sulphur assay (by LECO) for concentrate samples from bench flotation test conducted with two co-collectors and the baseline**. 111

Table 20: Mineral identification and quantification by XRD and calculated mineral content from Cu, Ni assay (by AAS) and S (by Leco) for bench flotation test**. 115

Table 21: Xanthate activity measured using the phenolphthalein titration method (section 8) for the four xanthates used for Bikerman froth stability column test work before and after purification by re-precipitation (section 8). 130

Table 22: PGM (4E) g/t and sulphide (by Leco) measured during a two day plant test on a South African opencast Platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay and sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. T-test used to calculate if the data during the test period is significantly (at 80% confidence level) different than the baseline period. For this calculation each of the two test days are analysed separately. Day 1 156

Table 23: PGM (4E) g/t and sulphide (by Leco) measured during a two day plant test on a South African opencast Platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON.

Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay and sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. T-test used to calculate if the data during the test period is significantly (at 80% confidence level) different than the baseline period. For this calculation each of the two test days are analysed separately. Day 2 157

Table 24: PGM (4E) g/t and sulphide (by Leco) measured during a two day plant test on a South African opencast Platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay and sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. T-test used to calculate if the data during the test period is significantly (at 80% confidence level) different than the baseline period. For this calculation data for both test periods are combined. 158

Table 25: PGM (4E) g/t and Sulphide (by Leco) measured during a two day plant test on a South African opencast Platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by Fire assay and Sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. ANOVA used to calculate if the data during the test period is significantly (at 80% confidence level) different than the baseline period. Each test bloc treated as a separate treatment 160

Table 26: PGM (4E) g/t and sulphide (by Leco) measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay

and sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. Confidence intervals calculated based on a 60% confidence interval......162

Table 27: Settings used to Assay for Cu, Ni and Fe the Spectra 600 AA..........ii

Table 28: Repeat Copper reading using Spectra 600 AAS using the same digested ore sample and repeat AAS reading from six ore digestions by sodium peroxide fusion compared to three referee laboratory assay values..........iv

Table 29: Repeat Nickel reading using Spectra 600 AAS using the same digested ore sample and repeat AAS reading from six ore digestions by sodium peroxide fusion compared to three referee laboratory assay values..........iv

Table 30: Activity of purified SIPX measured with the Acetone and phenolphthalein method directly after preparation..........x

Table 31: Activity of commercial SIPX measured with the acetone and phenolphthalein method directly after preparation and 48 hours later...........xi

Table 32: Molecular weight for a number of xanthate types and acid volume needed for acetone titration...........xiii

Table 33: Bench scale flotation test results. Four co-collectors tested against baseline using a D12 bench flotation machine...........xviii

Table 34: Mineral identification and quantification by XRD for concentrate samples from bench flotation test conducted with two co-collectors and the baseline..........xix

Table 35: Calculated mineral content from Cu, Ni assay (by AAS) and S (by Leco) for bench flotation test...........xx

Table 36: Laboratory scale flotation test work done using 1.3 litre Denver cell and 100 gram synthetic ore (90% quartz and 10% chalcopyrite)...........xxiii

Table 37: Laboratory scale flotation test work done using 1.3 litre Denver cell and 100 gram synthetic ore (90% quartz and 10% pyrrhotite)...........xxiv

1. Introduction

The first successful commercial froth flotation process was installed by the Elmor brothers in 1897, at the Glasdir copper mine in North Whales (Maelgwyn Mineral Services Limited, 2009). Since this first froth flotation process was installed, which used oil to separate copper from gangue, it is still being used extensively in the beneficiation of a range of minerals including copper, nickel, lead, zinc, platinum group metals, gold, phosphate, coal, iron and fluorspar.

In essence the modern froth flotation is a process of separating value minerals from gangue by exploiting differences in the hydrophobicity of the minerals and phases. Hydrophobicity differences between valuable minerals and waste gangue are increased through the use of surfactants and wetting agents (Wikipedia, 2011). These reagents are commonly described as collectors (Rao, 2004).

For the separation of sulphide minerals like chalcopyrite, pentlandite and pyrrhotite, xanthate is commonly used as collector. Xanthates were discovered in 1822 and introduced into froth flotation in 1923 (Rao, 2004). Dithiophospates, thionocarbamate, mercaptabenzothiazole (Rao, 2004) and dithiocarbamates (Lotter & Bradshaw, 2009) are often used in combination with xanthate, or (less commonly) on its own. In some instances a combination of at least two xanthates with different chain lengths is used (Bulatovic, 2007).

Each froth flotation plant usually operates with a unique collector combination and specific dosing rate. This collector suite is usually selected to reduce the losses of the value mineral to the plant tailing stream, to reduce the amount of gangue reporting to the flotation concentrate stream and to consider operating cost (Chidley, 2008). A large number of factors influence the collector combination selection, which explains the large number of collector suites in use today. These include (Klimpel, 1995);

- Value mineral: type, particle size distribution, liberation size, degree of surface alteration.

- Gangue mineral: type, particle size distribution, liberation size, degree of surface alteration, particle shape, density and porosity.
- Process water: temperature, total dissolved solids, pH, dissolved ion type and concentration, oxygen concentration and pulp redox potential (Eh).
- Process: flow sheet design, flotation equipment employed, water circuit and process control strategy.
- Flotation reagents: availability, logistics, storage, make-up and dosing equipment.

For an operating froth flotation plant, an alternative collector suite is usually tested on laboratory scale and compared to the baseline collector suite in use. If sufficient potential is shown, a short industrial-scale test (normally one or two shifts of 8 hours each) is conducted and results compared with the baseline. If the test showed promise, a longer industrial-scale test (normally 5 days) is planned. If this is successful, a long term test, lasting 30 days to 3 months, is conducted (Shaw, 1998). A collector suite change will normally be motivated if the long term test was successful. The selection and successful implementation of an alternative collector suite is a lengthy process. It requires a considerable amount of risk, counts on skilled personnel, and involves accurate and robust testing methods. But even after all this rigorous testing, it is often not possible to demonstrate potential on an industrial-scale, owing to normal process variations occurring during the collector test (Lotter & Fragomeni, 2010). For this reason it is likely that some flotation plants are not using the most efficient and effective collector suites.

2. Research problem and objectives

Laboratory scale flotation tests are commonly used to predict if a collector change will result in an increase in flotation kinetics of a value mineral. The problem often encountered is how to conduct this laboratory scale test if the collector has froth stabilising or destabilising characteristics. By changing the froth character a collector change can influence the froth recovery (increasing or decreasing froth stability) and this may be interpreted as an increase or decrease in the floatability of the value mineral. If a plant test is motivated based on this conclusion, it will probably not be a good prediction of plant performance. Operators manage froth structure with frother addition (amongst others operational parameters to their disposal) and would eliminate the change in froth character a collector change may introduce. The same risk in mis-predicting the results of a collector with froth destabilising characteristics is also present.

In this study a set of additional test is described, which should be used in conjunction with the normal laboratory batch flotation test, to test a collector. The test procedure suggested measures the frothing characteristics a collector change introduces into a flotation system. If it is detected that a collector increases or decreases the froth stability, then a set of additional tests is recommended to ensure the collector changes the floatability of the value mineral.

In this study the effect a combination of a fatty acid based co-collector and xanthate has on the flotation of chalcopyrite, pentlandite and pyrrhotite is measured as a case study.

3. Literature review

3.1. Testing collector combinations

Arguably the best way, and perhaps the only way accepted by industry, to test and demonstrate the claimed benefits of a collector combination, is to be able to measure the benefit on plant scale. This poses two important challenges in the development and commercialisation of collector combinations: the first is that the cost and risk to do collector research and development work on plant scale is prohibitive. The second is that the normal variation and data noise on an industrial plant is usually so large, that an abundance of data is needed to statistically verify the effect of a collector change (Xiao, et al., 2009).

Although the aim should always be to demonstrate the collector benefit on commercial scale, a set of tests is needed which can be used by the flotation engineer to develop new collector combinations and to screen existing combinations. In doing so only a very limited number of collector combinations are tested on a commercial scale. It is further important that the test preceding industrial scale testing phase should simulate plant conditions accurately, so that the probability of a successful plant test can be increased. The ideal would be that collector combinations can be so accurately tested in the laboratory that the plant test is merely used as confirmation.

Pradip (1988) recommends a test method where the first step to commercial utilisation of a collector is micro-flotation tests on pure mineral specimens and synthetic mixtures followed by bench-scale batch flotation tests on the ore (Pradip, 1988). Some operators prefer that a collector combination, after successful testing on bench-scale, be tested in a pilot plant before it is tested on industrial scale. There is appreciation for this approach, since plant testing is risky, expensive and often inconclusive. The Amira P9 project has focused on using a pilot plant test rig (FCTR) to simulate a flotation plant test, and, amongst others, uses this method to test reagent combination prior to industrial tests (Coleman, et al., 2007).

The final stage in testing a collector combination is to demonstrate the benefit on industrial-scale. Xiao et al. (2009) describe the planning necessary and the statistical t-test recommended to interpret the plant data in order to verify the hypothesis that the collector combination has advantages over the baseline (Xiao, et al., 2009). They conclude "that with the application of these statistical tools in planning a plant trial and analyzing the results, a more convincing and accurate conclusion about the real benefit of selecting a collector can be drawn".

3.2. Using the kinetic model for froth flotation to predict and anticipate the possible effect a collector change may have on laboratory and plant test results

If an analogy with classical chemical reactor design is evoked to model froth flotation, flotation is described to be the reaction of bubbles with particles and is expressed as:

$$\frac{dC_p(t)}{dt} = -k(t).C_p^m(t).C_b^n(t) \qquad (1)$$

where $C_p(t)$ and $C_b(t)$ are the concentrations of the particles and bubbles at time t, respectively. The exponents, m and n, are the respective order for particles and bubbles and k(t) is a pseudo rate constant that depends on various parameters governing the flotation process, and may vary with time. (Polat & Chander, 2000).

Polat and Chandler (2000) describe how this equation can be simplified when the rate of bubble particle coalition is first order, the concentration of bubbles remains constant and the material to be floated consists of particles which rate of flotation (k) could be expressed by a continuous distribution function, f(k). The recovery at time t, R (t) will be equal to:

$$R(t) = R_\infty[1 - \int_0^\infty fk(t)e^{(-kt)}\,dt] \qquad (2)$$

where R_∞ is the ultimate recovery at long flotation residence time (Polat & Chander, 2000).

If one floatable component is present (with a single rate constant), the equation can be simplified where the recovery of the floatable component at time t, R(t) will be equal to:

$$R(t) = R_{\infty}[1 - e^{-kt}] \tag{3}$$

R_{∞} is the maximum recovery at infinite time or maximum recovery possible (%), k is the flotation rate constant of the floatable component (min $^{-1}$), t is the flotation time or residence time (min). It should be noted that this is an oversimplification and even if only one mineral is to be floated, factors like liberation, particle size and surface oxidation will affect the rate constant and the use of a single rate constant is not correct. One solution to this representation is to group the minerals as fast floating and slow floating, and assuming the fast floating fraction can be modeled with a single rate constant and the slow floaters can be modeled with a single rate constant (Hay & Rule, 2003). This equation is given as:

$$R(t) = (100 - \emptyset).\left[1 - e^{-k_f.t}\right] + (\emptyset).[1 - e^{-k_s.t}] \tag{4}$$

where Ǿ is the fraction slow floating mineral, k_f is the rate constant for the fast floating fraction (min^{-1}) and k_s is the rate constant for the slow floating fraction (min^{-1}).

Hay and Rule (2003) has indicated how the Kelsall equation can be effectively used to model and then predict plant performance (Hay & Rule, 2003).

Gorain et al. (1999) have shown that the rate constant (k) can be described as:

$$k = P.S_b.R_f \tag{5}$$

where k is the rate constant (min^{-1}), P is a parameter which represents the floatability of an ore, R_f is the froth recovery factor and S_b the bubble surface area flux.

Gorain et al. (1999) have also shown that there exists a relationship between the bubble surface flux (S_b), bubble size (D_b) and superficial gas velocity (J_g), and that this can be expressed as (Gorain, et al., 1999):

$$S_b = \frac{6.J_g}{D_b} \tag{6}$$

where S_b is the bubble surface area flux (m^2/m^2 sec), J_g is the superficial gas velocity (m/sec) and D_b is the Sauter mean bubble size (m).

If it is assumed that equations 3 to 6 can be used to describe a flotation process accurately, this model is helpful to predict what may be measured when a collector change is made on plant- and/or laboratory-scale. When a collector test program is designed and results evaluated, this has important implications. Conclusions which can be made are:

- If a collector has frothing characteristics (most have) and also reduces the bubble size in a laboratory-scale flotation cell, the value D_b will decrease, S_b will increase (Equation 6), the rate constant will increase (Equation 5) and the valuable mineral will be recovered faster (Equation 3). An increase in recovery at a set time in this instance is therefore not related to an increase in the floatability of the mineral (P). The same results would be measured by an increase in frother dosage. A conclusion that this collector combination will result in higher recovery during the industrial test is likely to be incorrect. It is thus necessary to test and compare the bubble size in a test flotation cell to avoid this possible error.
- If a collector has frothing characteristics, a more stable froth might be produced which might lead to an increase in froth recovery – R_f (Equation5). In this event, a batch flotation test might record faster value mineral recovery and the conclusion might be drawn that the collector increases the floatability –P (Equation 5). This will not be reproducible in the industrial test, since the plant operators are likely to compensate by reducing the frother dosage. The plant test is likely to be unsuccessful. The laboratory scale test program should therefore include a measurement that can be used to compare the frothing characteristics of a collector combination.
- A collector combination may increase the floatability of a value mineral -P (Equation 5). This is a desirable outcome and can be measured as more of the value mineral being recovered at the same flotation time. The test collector combination and the baseline collector suite are likely to have the same infinite

time recovery -R_∞ (Equation 3). Both batch flotation tests (baseline and test collector) will thus approximate the same recovery value at extended flotation time since only P is increased and not R_∞. Laboratory scale flotation tests are often conducted over a time which is similar to the flotation time in the industrial plant. If only one concentrate and discard sample is taken at the end of the batch test, it is likely that this test measures the infinite time grade and recovery -R_∞ (Equation 3) and that the desirable effect of a collector combination, which increases value mineral floatability (P -in Equation 5) is not recognised. To measure an increase in the value mineral floatability P (Equation 5), grade and recovery data measured at flotation time lower than time when infinite time recovery is reached needs to be compared. Opportunities cannot be recognised if this is not incorporated into the test program.

- If it is assumed that the J_g and R_f values in the batch flotation test is higher than industrial plant and the D_b is smaller. The rate of flotation in the batch test is then likely to be faster than the industrial plant and then the infinite time recovery will be reached faster. It is therefore likely that the batch flotation residence time needs to be shorter than the plant residence time to achieve the same flotation results.
- If a collector combination increases the value mineral floatability - P (Equation 5) and not the infinite time recovery R_∞ - (Equation 3) ,as is likely to happen, the benefit of the increase in rate of flotation will only be measured during a plant test if the residence time in the plant is not sufficient to reach infinite time recovery R_∞- (Equation 3) with the baseline collector suite. A collector combination successfully tested under laboratory conditions, which increases the rate of flotation, might not result in an increase in recovery in the plant.
- The air flow rate in the batch flotation test needs to be measured and controlled to be equal for all collector combinations tested. If the air is self-induced by the flotation machine, the air flow rate may be different for a different collector combination. An increase in the Jg value (Equation 6), will result in faster

flotation kinetics and higher recovery measured at the same residence time. If a plant test is motivated based on these results, it is likely to be unsuccessful.

- The froth is usually artificially removed in a laboratory batch cell by scraping the froth with a manual or automatic scraper. If this froth scraping is not consistent for each test, the froth recovery –R_f (Equation 5) is likely to be different for different tests and any recovery difference measured might not be as a result of a change in the value mineral floatability. A plant test motivated using this data set is likely to be unsuccessful.
- A micro flotation test conducted on pure mineral specimens and batch flotation tests done on synthetic ore made from pure mineral specimens can be used in conjunction with batch tests done on ore samples to confirm that an increase in the rate of flotation k- (Equation 3) or increase in infinite time recovery - R_∞ - (Equation 3) is a result of an increase in value mineral floatability -P (Equation 5). This should increase the probability of a successful plant test.
- A collector combination can increase the rate of flotation of the slow floating fraction k_s- (Equation 4) or the fraction slow floaters Ø (Equation 4). This is arguably the most desirable effect of a change in collector combination and has a high probability of being successfully tested on an industrial-scale. The slow floating fraction usually consists of a slow floating value mineral; some reasons may include particle size and liberation. A sound understanding of mineral recoveries and not only value element recoveries is needed to confirm this.

3.3. Bench flotation test

3.3.1. Equipment

The Denver laboratory flotation machine (D-12) or equivalent is perhaps the most widely used laboratory device used to test collector and collector combinations on a laboratory scale. This is essentially a scaled down, batch version of the commercial Denver flotation machine.

It should be noted that no ASTM procedure has been documented to conduct a laboratory scale flotation test and a considerable amount of variation in the flotation procedures and methods can be found. Normally each flotation plant has a standard test procedure which is developed to simulate the process as accurately as possible.

Reproducibility of the Denver laboratory flotation test is a challenge and many researches have suggested variations to improve this. Luttrell and Yoon (1983) have shown that by controlling the level in the batch flotation cell and by measuring and controlling the air flow rate, the ease of operation and reproducibility can be improved (Luttrell & Yoon, 1983). Linddell and Dunne (1984) have shown that by controlling the air flow rate, by employing specially designed scrapers to limit the depth of froth scraping and by removing the froth according to a set routine, the reproducibility could be improved (Lindell & Dunne, 1984). The Leeds flotation cell developed by the University of Leeds incorporates these modifications in the design and it is accepted that this machine can produce improved reproducibility.

Irrespective of the equipment used to conduct the laboratory scale tests, if reproducibility is acceptable and the test can be shown to simulate the plant practice sufficiently, it can be used to test and compare collector combinations.

3.3.2. Ore sampling

Lotter and Fragomeni (2009) comments that high-confidence flotation testing "methodology is based on two principles: one, to ensure that the ore sample is representative and has been well blended and subsampled; two, to perform the flotation test in sufficient numbers of replicas with appropriate quality controls, so as to improve reproducibility of test data" (Lotter & Fragomeni, 2010).

Additional notes on taking samples for a laboratory scale flotation test is discussed in the appendix under Section 10.1

3.3.3. Water quality and temperature

The effect of water quality and water temperature on performance of flotation plants is well known by flotation operators (du Plessis, 2009). Corin et al. (2011) studied the effect of ion concentration on batch flotation tests on a typical platinum group minerals (PGM) - Merensky ore with chalcopyrite, pentlandite and pyrrhotite as the main value minerals. It was concluded that a change in ion concentration has a measurable effect on value mineral and gangue mineral flotation and speculated that this effect is probably related to a difference in froth stability (Corin, et al., 2011).

Palm et al. (2010) used synthetic plant water for wet milling and flotation which was prepared using reverse osmosis water containing the following ions: Mg^{2+} 80 ppm, Ca^{2+} 80 ppm, Na^{+} 135 ppm, Cl^{-} 270 ppm, SO_4^{2-} 250 ppm NO_3^{-} 135 ppm, NO^{2-} 40 ppm CO_3^{2-} 40 ppm. The TDS value was 1030 and the ionic strength was 3.5E-02 (Palm, et al., 2010). It can be concluded that, as a minimum requirement, it is necessary to conduct a set of comparative tests using the same water source.

The standard when conducting micro flotation tests is to prepare synthetic plant water. When Vermaak et al. (2005) studied the flotation of PGM in a micro flotation cell they prepared synthetic plant water with the following ion concentrations by adding soluble salts to distilled water: Mg^{2+} 80 ppm, Ca^{2+} 80 ppm, Na^{+} 135 ppm , Cl^{-} 270 ppm, SO_4^{2-} 250 ppm NO_3^{-} 135 ppm, NO^{2-} 40 ppm CO_3^{2-} 40 ppm (Vermaak, 2005).

O’Conner and Mills (1990) studied the effect of pulp temperature on the flotation of pyrite (from a South African gold mine) using SMBT (sodium mercaptobenzothiol) and TEB (triethoxybutane) as flotation reagents. It was found that conducting the test at below ambient temperature, thus simulating winter conditions, the rate of flotation for fast floating particles decreased, with little observed effect on the rate of slow floating particles. As the temperature increased above the ambient temperature, the main effect was a reduction in water recovery and an increase in concentrate grade (O'Conner & Mills, 1990). This has important implications for batch flotation test work, especially done on the South African Highveld where day temperatures can vary from 5 to 28 degrees.

It is therefore important that not only the water quality (ion concentration, pH and TDS) needs to be kept constant for a set of comparative tests, but also, and possibly more importantly, the temperature of the water used to conduct the flotation test.

3.3.4. Entrainment (mechanical carryover)

Gangue and value minerals are recovered to the flotation product as a result of true flotation and entrainment (also called mechanical carryover or mechanical flotation) (Konopacka & Drzymala, 2010). When testing a collector combination, only the effect of true flotation is relevant (based on the argument in Section 2.2) and an attempt must be made to either keep entrainment constant for the test program or account for it.

Gangue and other particles are mechanically carried to the froth launder with the water in the froth. Konopacka et al. (2010) describe and model five types of possible relationships between the entrainment of gangue and value minerals and the recovery of water to the flotation concentrate (Konopacka & Drzymala, 2010).

Wiese et al. (2005) plots mass units of value mineral recovered against mass units of water recovered for different reagent combinations. By comparing mass units of value mineral recovered with the same mass units of water recovered, the effect of collector on true recovery can be interpreted (Wiese, et al., 2005).

Zheng et al. (2006) has shown that a number of models can successfully model water recovery (Zheng, et al., 2006). From these models it is concluded that if froth depth, air flow rate, bubble size, bubble stability and froth retention time in a batch test program can be kept constant, there is a good chance that entrainment will be similar. Froth depth, air flow rate and retention time are normally kept constant in a batch test. Bubble size and froth stability are normally froth characteristics attributed to the frother used and the frother characteristics of the collector combination tested. It can then be proposed that if two collector combinations tested have similar frother characteristics (bubble size and froth stability), entrainment would be more or less similar and any recovery differences measured will be as a result of a change in true recovery. If one of the collectors tested has significant different frother characteristics, (bubble size and froth stability) any change in recovery cannot be attributed to true flotation only and it might be possible that this recovery difference could not be reproduced on a plant scale.

Measuring the frothing characteristics of a collector combination (bubble size and froth stability) seems therefore advisable. If large differences in collector combinations are measured, care should be taken when the data is analysed. Plotting mass units of value mineral recovered against mass units of water recovered for the different reagent combinations is then necessary to assist in interpreting the data (Wiese, et al., 2005).

3.3.5. Milling and conditioning

Buswell et al. (2002) has shown that the type of grinding media used (mild steel or stainless steel) influences chalcopyrite, pentlandite and pyrrhotite recovery, when floated in a batch test cell. Differences were also measured when the ore was milled in the laboratory or when extracted from the float feed stream in the plant (Buswell, et al., 2002).

Palm et al. (2010) studied the flotation of sphalerite prepared for flotation using a dry milling or wet milling method. It was found that, irrespective of the crushing procedure (HPGR or conventional), dry milling resulted in the highest grades and recoveries of zinc (Palm, et al., 2010). Seke (2005) reported similar results (Seke, 2005).

These differences are likely to be related to the oxygen concentration in the mill and possible reactions between oxygen, dissolved iron species and the value mineral. (Martins, et al., 1991). The adsorption of xanthate collectors onto sulphide minerals chalcopyrite, pyrrhotite and pentlandite is recognised to occur via an electrochemical mechanism, which is in turn influenced by the oxygen concentration and mineral reversible potential in the pulp during and after milling (Buswell, et al., 2002).

Leppinen et al. (2000) studied the effect of grinding media on sulphide flotation and concluded that the iron of the mild steel in the grinding media used, created a reducing environment in the slurry, with low concentrations of dissolved oxygen. The formation of iron hydroxides can suppress flotation of sulphides when the electrochemical conditions are altered by aeration (Leppinen, et al., 2000).

Milling of the ore sample before a batch flotation test is therefore not only about particle size reduction and liberation, but also influences the floatability of the value mineral (Martins, et al., 1991) and the interaction between the value mineral and xanthate (Buswell, et al., 2002). It is thus not only necessary that the milling stage should aim to simulate the particle size distribution found in the plant, but also the pulp chemistry of the plant.

Greet et al. (2004) describes a test method and equipment where the pulp chemistry in the grinding mill is measured and controlled by injecting gasses and reagents into the mill. This work highlighted the importance of the milling stage on flotation pulp chemistry and indicated that plant conditions can be simulated on laboratory scale (Greet, et al., 2004).

In the absence of this equipment, it is important that the flotation metallurgist recognises the value of pulp chemistry and the grinding step on flotation test results and reproducibility and should, as an oversimplification, aim to keep the laboratory milling time as close as possible to the residence time in the industrial mill.

For a 100 t/h plant with one 4 meter diameter mill with a 10 meter length, 40% ball charge and 60% void between the grinding balls, the theoretical open circuit milling residence time is calculated to be approximately 7 minutes if a mill feed density of 30%

solids by mass is assumed. Using a safety factor of 2, the residence time of a particle in a mill would be less than 15 minutes. A batch flotation test procedure should thus aim to achieve the specified particle size distribution without having to mill for more than 15 minutes. It is equally important to include a wet milling stage before the flotation batch test of no shorter than 5 minutes to simulate the effect wet milling has on flotation. The guideline used in the Betachem mineral processing laboratory is to wet mill for at least 4 minutes but not longer than 10 minutes (Grobler, et al., 2008). This is achieved with a dry milling step before wet milling.

For the same reasons as listed above, it is also important to simulate the conditioning of the pulp after milling and before flotation as practiced in the industrial plant when conducting a batch flotation test. During the conditioning stage surface reactions between the collector and value mineral can take place, but equally important is the reaction of oxygen and ions in solution with the value mineral and the collector combination. At the Betachem mineral processing laboratory, milling and flotation is seen as a single continuous test; the timed test sequence is started when the water is placed in the wet mill and finishes when flotation is complete (Grobler, et al., 2008).

3.3.6. Reagents

To simulate the reagent dosing rate and dosing sequence in the laboratory is normally not difficult. The following should however be considered:

The purity of xanthate often varies considerably, especially if there is an extended period between manufacture and usage. For a test program where different xanthate types are tested and compared, the xanthate purity should be taken into account. Venter (2009) used the method suggested by Rao to purify commercial grade xanthate in the laboratory (Venter, 2009).

Thiol collectors like xanthates degenerate when mixed with water. Zhonghi et al. (1997) studied the degeneration of ethyl xanthate at various temperatures and different pH mediums and found the half-life of potassium ethyl xanthate (purified with Rao method) to be approximately 7 days, at ambient temperature of 25 degrees dissolved in water with pH 6.6. This half- life decreased significantly at higher temperatures and when dissolved with plant water at pH of 8 (Zhongxi-Sun & Forsling, 1997). The best practice is to prepare a fresh xanthate solution each day. The same method is recommended for all collectors being tested (Grobler, et al., 2008).

3.3.7. Interpretation of bench-scale flotation test data

Laboratory batch flotation data is commonly communicated through a plot of flotation recovery of the value element vs. the concentration or grade of the value element in the flotation concentrate. The effect of collector combinations can be established by comparing the cumulative grade - recovery plot for the two conditions. The disadvantage of this method is that it is difficult to differentiate between an increase in flotation kinetics of the value mineral and a change in infinite time recovery (Section 2.2).

An alternative method is to plot the recovery of the value element or value mineral against time. It is often difficult to comment on selectivity and gangue flotation with this method alone. This can be overcome by also plotting the recovery of gangue as a function of time.

Hay and Rule (2003) have demonstrated how the Kelsall's unmodified equation (Equation 4 Section 2.2) can be used to fit batch flotation data and how it can then be used to predict plant performance (Hay & Rule, 2003). Hay and Rule (2003) use model constants (like fast floating mineral fraction, fast floating mineral rate, slow floating mineral rate, fast floating gangue fraction, fast floating gangue rate and slow floating gangue rate) to characterise batch flotation data. The fitted constants can then be used to compare batch flotation data for an alternative collector combination.

In ores (for example South African Merensky reef ores), consisting of a mixture of value minerals all containing a value element, such as chalcopyrite, pentlandite and pyrrhotite containing platinum group elements, it is often misleading to analyse the platinum group elements data on its own. For these ores, measuring and comparing the flotation data for each value mineral is useful. Newell et al. (2006) studied different methods to improve the recovery of chalcopyrite, pentlandite and pyrrhotite using an ore from Nkomati Nickel mine in South Africa (Newell, et al., 2006). Newell et al. (2006) found it useful to plot mineral recoveries as a function of time. The authors used Cu, Ni and Fe assay values obtained by Atomic Absorption Spectroscopy (AAS) and S assay (measured by Leco sulphur analyser) to calculate mineral recoveries. Cu and Ni assay values were used to calculate chalcopyrite and pentlandite recoveries and the unaccounted S used to calculate pyrrhotite recovery. This approach was also followed by Bradshaw et al. (2006) studying chalcopyrite, pentlandite and pyrrhotite recovery in Merensky ore. The head value obtained using this chemical method compared well with QEM*SEM analysis of the feed ore which validated this method as a low cost alternative to mineralogical methods (Ekmekci, et al., 2006).

When this method is used to calculate mineral recoveries, the following should be noted:

- This method can only be used if a value element is present in only one value mineral. It is known that in the Nkomati ore, nickel occurs as pentlandite, but also in pyrrhotite (de Villiers, 2010). Newell et al. (2006) assumed Ni occurring in pentlandite only and calculated results are therefore only an approximation of mineral recoveries.
- Care should be taken with non-stoichiometric minerals like pentlandite which can have a variable Ni content and pyrrhotite which can have variable Fe content. It is likely that the Merensky ore studied by Bradshaw et al. (2006) contained more than one pyrrhotite crystal structure (Mphela, 2010) and it should thus be concluded that the mineral recoveries calculated is only an approximation.

This method is however a low cost and accessible method available to the flotation metallurgist and when the assumptions are understood and it is accepted that the results are only indicative of mineral recoveries, it is useful.

3.3.8. Quality control of batch-scale flotation test data

Lotter et al. (2009) describe a quality control procedure for high confidence batch flotation used by Xstrata process support as follows (Lotter & Fragomeni, 2010):

- Twelve feed samples are assayed and the average and standard deviation calculated. It is a requirement that the sample standard deviation does not exceed 5%. The average estimates the sample mean grade and is the external reference mean (or assay head grade).
- A minimum of three acceptable flotation replica tests is required. The average results of the three tests are used.
- The first rougher concentrate mass of each replica test must agree within a relative standard deviation of less than 5%.
- For each flotation test set (replica of three), the concentrate (mass and grade) and tails (mass and grade) are used to calculate an internal reference mean (or build up head grade). This internal mean and external mean are compared as a percentage (call factor) and must be between 96.7% and 103.3% (this is for a 95% conference limit). If the call factor is outside these limits the test is discarded and repeated.

A variation of this method, used by the Betachem mineral processing laboratory is the following (Grobler, et al., 2008):

- Use the average of the build up head grade for the complete test program as the external reference mean if the external mean is not measured.
- Use the individual test data to indicate error bars (standard error) on visual and tabulated presentations.
- Calculate the call factor of each individual test and request assay repeat if outside 93% to 107%. If the call factor is still outside 93% to 107% after assay repeat, reject the test.

3.4. Bikerman froth stability column

When collector combinations are tested and compared, under laboratory conditions, it has been shown in Section 2.2 and Section 2.3.4 that the ideal is that the froth characteristics of comparative tests should be similar. Collectors often have inherent frothing characteristics and it is often not possible to test collector combinations without also affecting the froth structure. When testing a new collector combination, it is at least necessary to know if the test collector has different frothing characteristics to the baseline collector. This data should then be used to judge if any difference measured is related to a change of particle floatability (P) or related to a change of only frothing characteristics (like froth recovery and bubble size). (It has been argued in Section 2.2 that if a collector combination changed the particle floatability (P) a subsequent plant test has a high probability of being successful. If the collector combination only changed the froth structure, plant operators are likely to compensate for this during the plant test phase by changing frother addition, pulp level and air flow rate, and a plant test has a low probability of being successful).

Engelbrecht et al. (1998) employed the Bikerman foam test method (ASTM D1173) to test flotation froth characteristics (Engelbrecht, 1998). Barbian et al. (2005) used the Bikerman concept to develop a froth stability column and when used, on a typical South African platinum ore, the experiments indicated that the maximum stable froth height measured is a function of the air flow rate to the froth stability column and the frother concentration (Barbian, et al., 2005). Grobler (2005) showed that the Bikerman froth stability column could be used to test the frothing characteristics of a frother by plotting the stable froth height as a function of frother concentration at the same air flow rate (Grobler, 2005).

The Bikerman froth stability column can therefore be used to test the frothing characteristics of a collector or collector combination. This simple and inexpensive procedure should therefore form part of a collector combination test program.

3.5. Measurement of bubble size in a flotation pulp

The flotation rate constant is influenced by bubble size (Section 2.2). A collector combination with frothing characteristics is likely to produce bubbles with smaller mean diameter in the collection zone of a flotation cell which will result in faster flotation kinetics. Measuring the effect of collector combination on bubble size is useful. This will indicate if a change in flotation response is as a result of a change in the mineral floatability, (which may be tested on a plant), or bubble size, (which would probably not show potential on plant scale).

Chen et al. (2008) used image analysis methods to measure the bubble size distribution in a flotation cell (Chen, et al., 2001). Digital imagery and open source image analysis software for example Image J (http://rsbweb.nih.gov/ij/) is ideally suited to measure bubble size and size distributions.

The UCT bubble analyser (Tucker, et al., 1994) and McGill bubble analyser (Bailey, et al., 2005) can be used to compare the bubble size for comparative tests between collector combinations.

3.6. Testing of mineral floatability by micro flotation

Testing the recovery of pure mineral specimens by micro flotation techniques is widely used. Bradshaw et al. (2006) developed a micro flotation cell which could be used to measure the bubble loading (Bradshaw & O'Conner, 2006). Vermaak used this micro flotation cell to test the floatability of platinum-bismuth telluride (Vermaak, 2005). The method used is described as follows:

- Mineral specimens were freshly ground in porcelain crucible and sieved to obtain 2 gram of +38 – 106 micron material.
- Tests were conducted with 2 grams of mineral and 250 ml water.
- Synthetic water plant was used as described in Section 2.3.3.

- Mineral samples were conditioned in the synthetic water for 1 minute followed by the addition of collector ($5x10^{-5}$ M) followed by 3 minutes of conditioning.
- Flotation concentrates were collected at total times of 3, 6, 10 and 20 minutes, respectively.
- Concentrates were dried, weighted and the data plotted as cumulative recovery as a function of time.
- Air flow rate was set at 3.5 cm^3/min.

The value of this technique is based on the availability of mineral specimens which represents the ore and value mineral under review. It should be noted that large compositional variations for the gangue mineral and value mineral are possible. Pure mineral specimens are not always a good representation of minerals in the ore, for example sphalerite, pentlandite and pyrrhotite. Ideally natural specimens from the plant under investigation should be employed to mitigate this risk.

It should be accepted that the micro flotation technique can only help the flotation metallurgist to decide if the changed laboratory-scale results are related to a change in particle floatability and thus have value as part of a holistic test program.

3.7. Flotation of chalcopyrite, pyrrhotite and pentlandite

Following is summary of published information on the flotation behaviour of chalcopyrite, pyrrhotite and pentlandite relevant to this study. This extract in not intended to be a comprehensive summary of literature on the flotation mechanism of these base metal sulphides.

Kelebek et al. (1996) studied flotation of chalcopyrite, pentlandite and pyrrhotite in the absence of collector (Kelebek, et al., 1996). Results from this work are shown in **Figure 1**.

The authors found that chalcopyrite can be recovered with flotation without the addition of the collector. The addition of $CuSO_4$ did not appreciably affect chalcopyrite flotation. Neither did the addition of sulphur dioxide or DETA (Diethylenetriamine).

Pentlandite floated well without the addition of the collector (**Figure 1**). The hyperbolic shape of the chalcopyrite, pentlandite recovery curve, suggests that chalcopyrite floats faster than pentlandite, but given sufficient flotation time, pentlandite floats well without collector. The effect of surface oxidation of pentlandite and the subsequent formation of a hydrophobic sulphur rich surface in the flotation cell, (with or without the activation by naturally occurring Cu ions), is a possible mechanism. In this study the addition of $CuSO_4$ did not affect pentlandite recovery, (this was explained by the pre activation of pentlandite by Cu ions in the ore), but the addition of sulphur dioxide reduced pentlandite recovery appreciably. The removal of possible sulphur-rich surface layer by the sulphur dioxide was the possible mechanism. The addition of DETA and sulphur dioxide reduced pentlandite recovery further. The removal of Cu ions and other heavy metals on the surface of the pentlandite was the possible mechanism.

Pyrrhotite does not float nearly as well as chalcopyrite without collector addition. The recovery is not only appreciably lower (45%) compared to that of chalcopyrite recovery (95%), but the shape of the curve in **Figure 1** suggests that the pyrrhotite floats slower than chalcopyrite. The addition of 120 g/t copper sulphate increased the pyrrhotite recovery from 45 to 80%. At this dosage, it seems that chalcopyrite floats the fastest, followed by pentlandite and the pyrrhotite floating the slowest. Again the formation of a sulphur-rich oxidation layer was the possible mechanism for the formation of a hydrophobic pyrrhotite surface. Adding sulphur dioxide removes this hydrophobic layer (or prevents it from forming,) and reduces the rate and recovery of pyrrhotite. Like with pentlandite collectorless flotation, the addition of DETA and sulphur dioxide effectively prevents pyrrhotite from floating (Kelebek, et al., 1996).

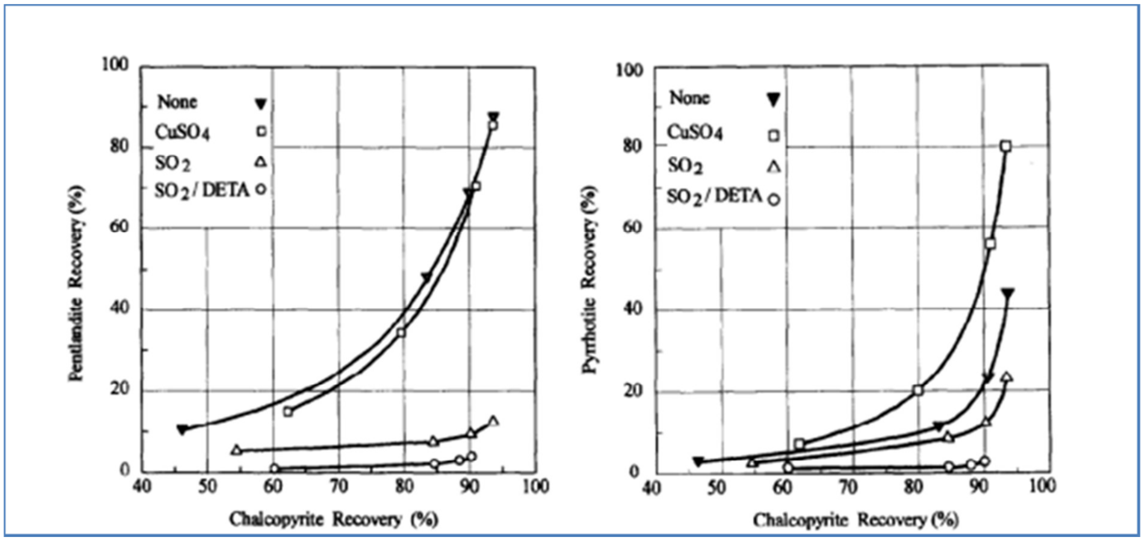


Figure 1: Collectorless flotation selectivity between chalcopyrite, pentlandite and pyrrhotite under various conditions**. (Kelebek, et al., 1996)

*** Reagent dosages: 35 g/t Dowfroth 250 (common to all), 120 g/t $CuSO_4$ (As indicated), 1.1kg/t SO_2 (indicated) and 200 g/t DETA (As indicated). pH approximately 9.5.*

For a typical Merensky ore, chalcopyrite, pentlandite and pyrrhotite can be floated without the addition of collector (**Figure 2**). Chalcopyrite floats reasonably well, but the recovery and rate of flotation of pentlandite and pyrrhotite is low (Newell, et al., 2006).

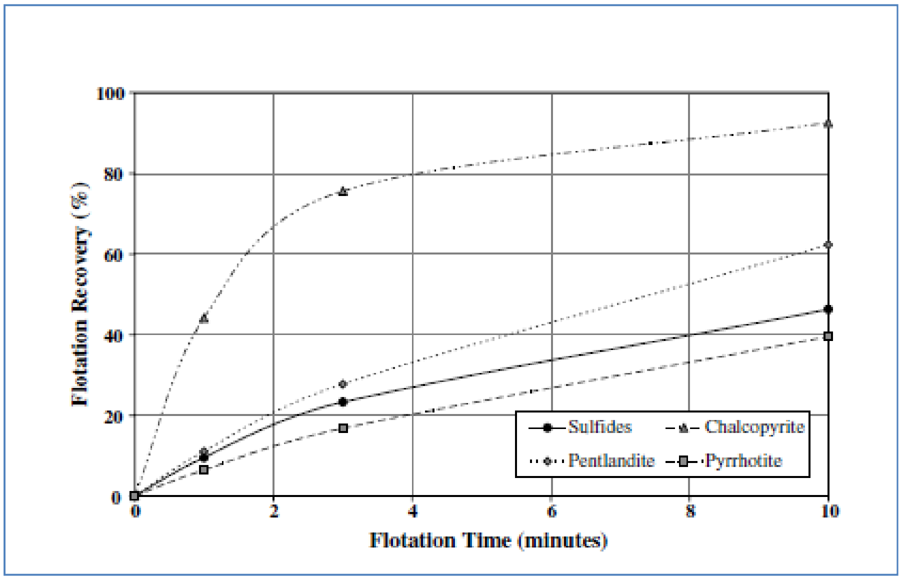


Figure 2: Flotation recovery of a typical Merensky ore without the addition of a collector. (Newell, et al., 2006)

Xanthate is by far the most commonly used collector to float chalcopyrite, pentlandite and pyrrhotite. It is postulated that the mechanism is either the formation of a hydrophobic metal-xanthate salt on the surface of the sulphide mineral (chemisorbed species), or dixantogen can physically adsorb on the surface rendering the mineral hydrophobic (Rao, 2004). A significant amount of work is being done to suggest which of the two accepted mechanisms is responsible for the flotation of different minerals in different environments. Although this is important and certainly interesting, it is outside the scope of this study. It is, however, important to note that the formation of dixantogen is an electrochemical reaction and both pH and electro potential on the mineral surface affects this reaction (Rao, 2004). Woods (2003) describes the adsorption of the collector, under certain conditions, as “a surface species to be formed by an anodic oxidation reaction involving the collector occurring simultaneously with a cathodic process which is usually the reduction of oxygen.” (Woods, 2003).

A typical Merensky ore, where the sulphide mineral is chalcopyrite, pentlandite and pyrrhotite responds well with an increase in xanthate addition as indicated in **Figure 3** (Newell, et al., 2006).

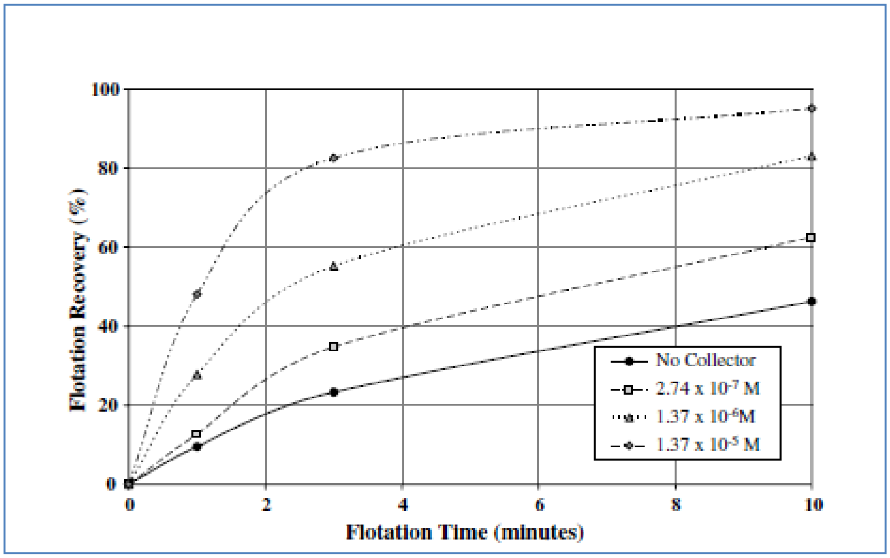


Figure 3: Effect of collector Sodium Isobutyl Xanthate (SIBX) concentration on the flotation of sulphide minerals in unoxidised Merensky ore samples (Newell, et al., 2006).

When **Figure 2** and **Figure 3** (Newell, et al., 2006) are compared, it can be concluded that chalcopyrite floats well without collector (**Figure 2**) and that the increase in sulphide recovery and kinetics (**Figure 3**) is as a result of an increase in pentlandite and pyrrhotite recovery when xanthate is added.

Bradshaw et al. (2006) measured the mineral recovery of a typical Merensky ore in a laboratory flotation test using xanthate and DTP as collector (Bradshaw, et al., 2006). This work indicates that chalcopyrite floats well and fast (**Figure 4**), followed by pentlandite, with pyrrhotite not effectively recovered with xanthate and DTP only.

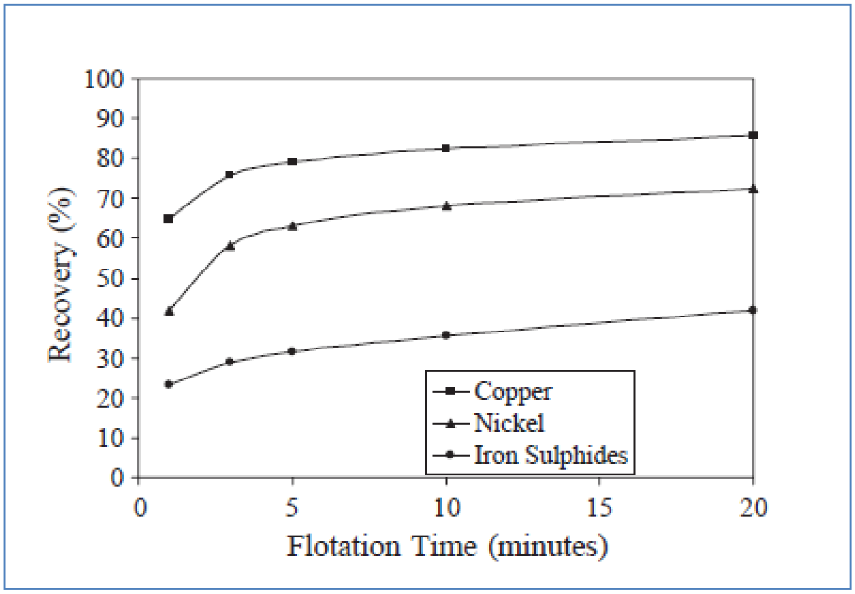


Figure 4: Flotation recovery vs. time after stainless steel (SS) milling and no copper sulphate addition. (30 g/t Potassium Amyl Xanthate (PAX) and 30 g/t Dithiophosphate (DTP) (Bradshaw, et al., 2006)

Adding 50 g/t copper sulphate to the same ore and then adding xanthate and DTP have a marked effect on pyrrhotite recovery as shown in **Figure 5** .

Without the addition of copper sulphate, the recovery of pyrrhotite increases when milled in a stainless steel mill compared to a mild steel mill. Bradshaw et al. (2006) indicated that milling with mild steel, the electro potential on a pyrrhotite electrode remained below the reversible xanthate/dixantogen potential, and when milling with stainless steel media, the potential was higher than the reversible xanthate/dixantogen potential (Bradshaw, et al., 2006). The authors concluded that in the mild steel mill, oxygen was consumed by the grinding media, thus preventing the formation of dixantogen which in turn resulted in low pyrrhotite recovery. The addition of copper sulphate increased the electro potential on a pyrrhotite electrode to above the reversible xanthate/dixantogen potential, therefore allowing dixantogen to form and hence the increase in recovery of pyrrhotite.

The effect of copper sulphate on the recovery of pentlandite and chalcopyrite is less pronounced than its effect on pyrrhotite recovery as is shown in **Figure 6** and **Figure 7**.

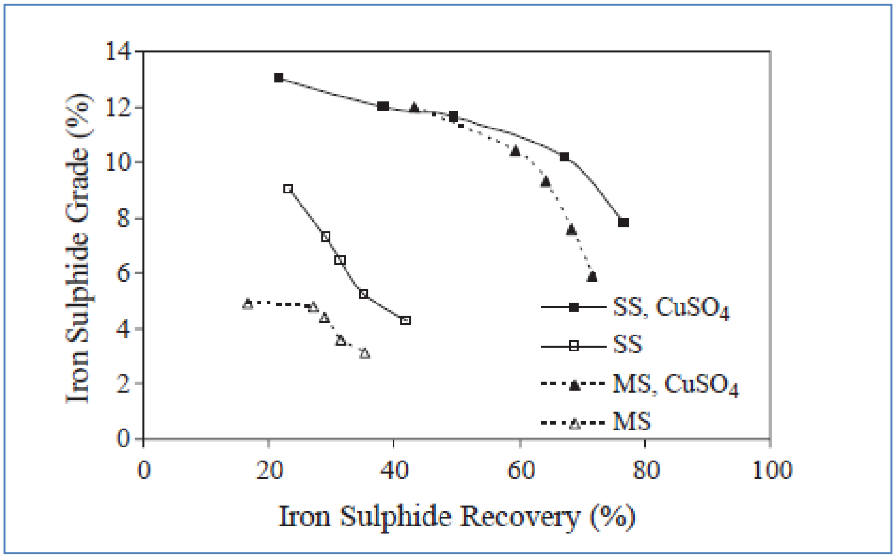


Figure 5: Iron sulphide (pyrrhotite) grade vs. recovery obtained for flotation tests with ore milled with mild steel (MS) and stainless steel (SS) milling media with 50 g/t copper sulphate as specified ($CuSO_4$). (30 g/t potassium amyl xanthate (PAX) and 30 g/t DTP) (Bradshaw, et al., 2006)

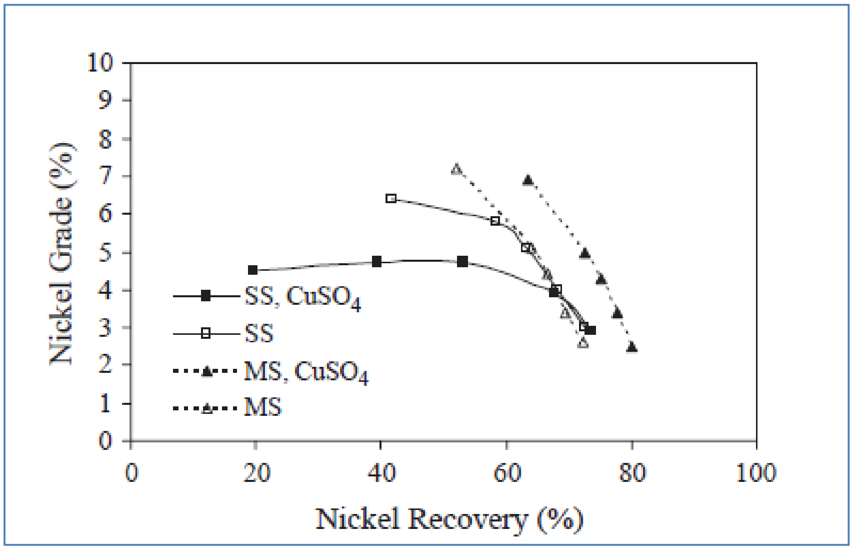


Figure 6: Ni(pentlandite) grade vs. recovery obtained for flotation tests with ore milled with mild steel (MS) and stainless steel (SS) milling media with 50 g/t copper sulphate as specified ($CuSO_4$). (30 g/t PAX and 30 g/t DTP) (Bradshaw, et al., 2006)

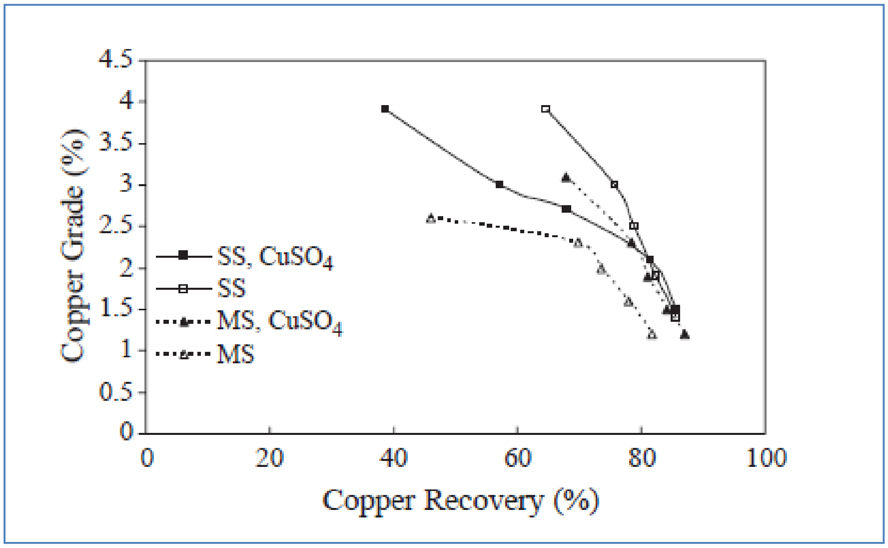


Figure 7: Cu (chalcopyrite) grade vs. recovery obtained for flotation tests with ore milled with mild steel (MS) and stainless steel (SS) milling media with 50 g/t copper sulphate as specified ($CuSO_4$). (30 g/t PAX and 30 g/t DTP) (Bradshaw, et al., 2006)

Brough et al. (2010) studied the effect of copper sulphide on a number of South African Merensky reef samples and reported improvements in flotation grades and recovery for pentlandite and pyrrhotite but not for chalcopyrite. The authors ascribe the improvements of copper sulphate addition to the “suitable preparing the surface of the sulphides (particularly pyrrhotite) for xanthate adsorption and subsequent flotation.” (Brough, et al., 2010)

3.7.1. Flotation of oxidised minerals

Plant operators have known for a long time that the longer sulphides are left on the ore stockpile open to the environment and rain, the more flotation performance is reduced (McLennan, 2004). This was studied by Newell et al. (2007) using an ore sample from Nkomati Nickel (Newell, et al., 2007). The ore was thermally oxidised at 85 °C to establish a thick layer of base-metal oxides and sulphoxides. The results (**Figure 8**) show that flotation recovery decreases with oxidation time. Chalcopyrite is least affected with pentlandite and pyrrhotite showing more or less the same reduction in flotation recovery.

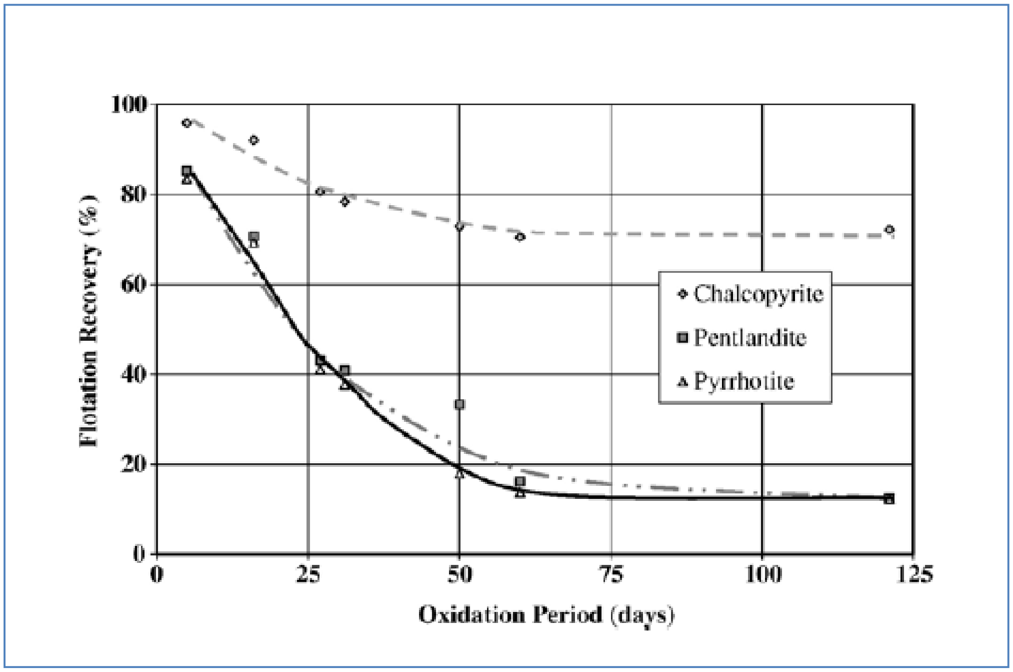


Figure 8: Effect of oxidation upon sulphide mineral flotation recoveries- Merensky ore. (Ultrasonic treatment and 1.37×10^{-2} M SIBX) (Newell, et al., 2007).

Kelebek et al. (2007) studied the flotation difference between fresh and oxidised material from the ore stockpile (Kelebek & Nanthakumar, 2007). The authors found that pentlandite recovery was negatively affected by oxidation on the stockpile as expected (**Figure 9**), but that pyrrhotite recovery improved (**Figure 10**). The result of surface oxidation is not always metal oxide or hydroxide, but can also be a sulphur rich, hydrophobic surface, which is possibly why the pyrrhotite in this ore floats better after mild oxidation. Metal ions can leach out on the stockpile and can precipitate on pyrrhotite surface, thus activating it similarly to copper sulphate activation (Kelebek & Nanthakumar, 2007).

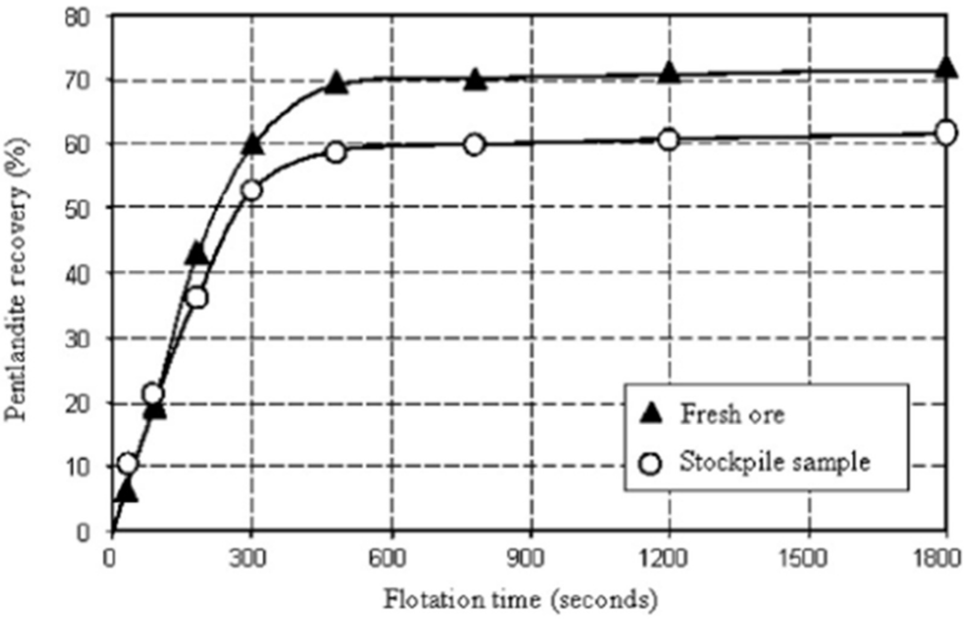


Figure 9: Pentlandite recovery-time for fresh ore and stockpile sample - On a ping area of Falconbridge Ltd (Kelebek & Nanthakumar, 2007).

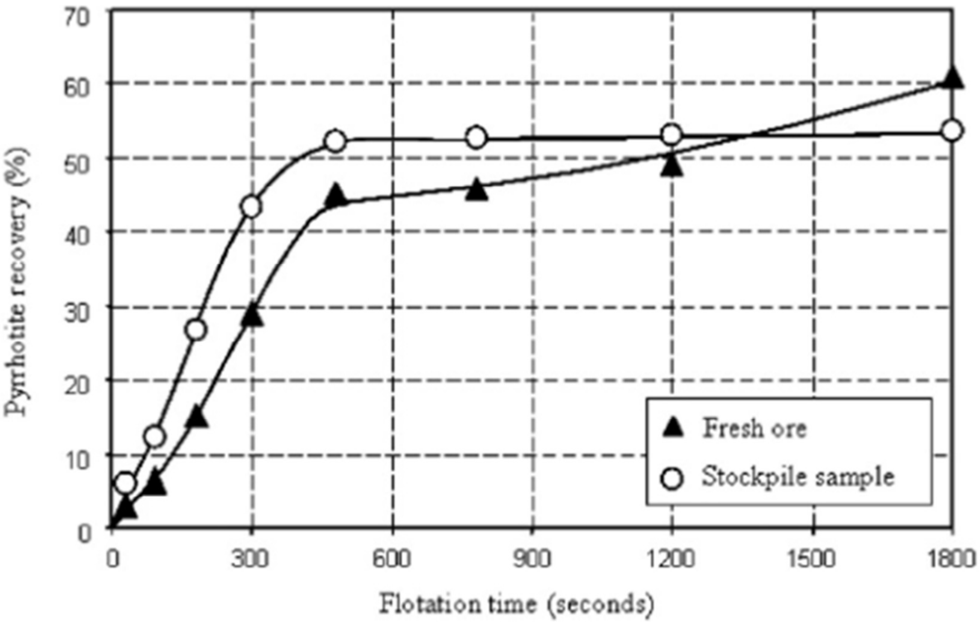


Figure 10: Pyrrhotite recovery-time for fresh ore and stockpile sample - On a ping area of Falconbridge Ltd (Kelebek & Nanthakumar, 2007).

Woods (2003) summarises collectorless flotation as follows: "A few sulphide minerals are naturally floatable as a result of preferential cleavage along planes of sulphur atoms that have all chemical bonds satisfied. Other sulphides have been found to float under oxidizing but not reducing conditions. As with collector-induced flotation, the process responsible for rendering the mineral floatable is electrochemical in nature. In the self-induced flotation case, the anodic reaction is oxidation of the surface to a sulphur species." (Woods, 2003).

Woods (2003) further comments that "self-induced flotation is sensitive to inhibition from metal hydroxides. The development of metal hydroxides will depend on the grinding media and will vary with ore type and water source." (Woods, 2003)

3.7.2. Effect of stiogemetry on the flotation of pyrrhotite

Pyrrhotite ($Fe_{(1-x)}S$) is non-stoichiometric sulphide mineral and occurs in different crystallographic forms and compositions. Becker et al. (2010) studied the flotation recovery of four pyrrhotite samples (Canada, South Africa and Botswana) and concluded that : “Pyrrhotite behaviour cannot simply be explained by a single mineralogical factor, but that it is a combination of mineralogical characteristics governing its reactivity and flotation performance. These key mineralogical factors are the crystallography (number of vacancies), mineral chemistry (ferric iron and nickel content) and mineral association (composite particles).” (Becker, et al., 2010). The effect of this on flotation recovery is shown in **Figure 11** and **Figure 12**. The collectorless recovery and recovery with xanthate for different pyrrhotite types vary significantly, in this study as much as 50%. Not only does its recovery vary for different pyrrhotite samples, but the effect of pH on recovery varies as well. For the magnetic pyrrhotite from Sudbery and the pyrrhotite from Nkomati and Tati Nickel in Botswana, increasing the pH to 10 depresses pyrrhotite recovery significantly. The same effect is not seen for the nonmagnetic pyrrhotite from Sudbury.

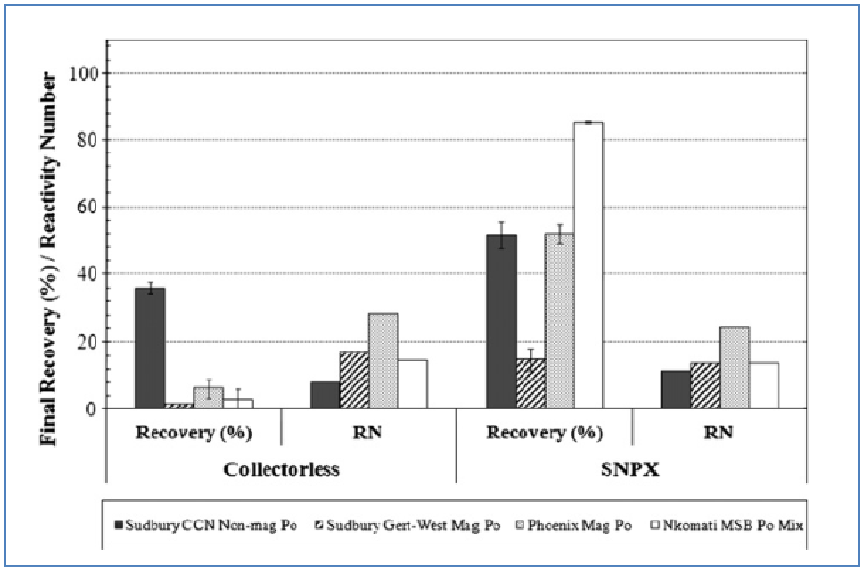


Figure 11: Comparison of the final flotation mass recovery and reactivity number (RN) for the different Pyrrhotite samples at pH 7. (Becker, et al., 2010)

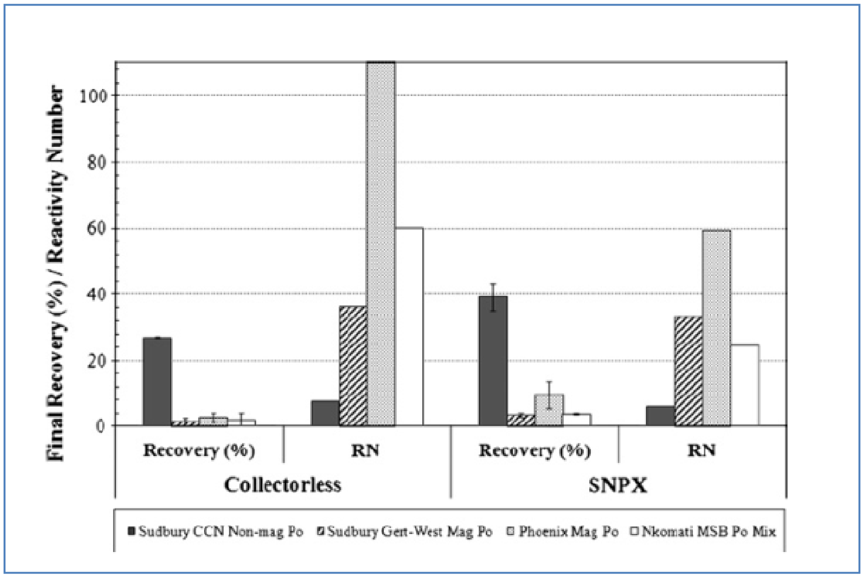


Figure 12: Comparison of the final flotation mass recovery and reactivity number (RN) for the different Pyrrhotite samples at pH 7 (Becker, et al., 2010)

3.7.3. Flotation of chalcopyrite, pentlandite and pyrrhotite with a mixture of xanthates or mixture with xanthate and a co-collector

In flotation practice of chalcopyrite, pentlandite and pyrrhotite, it is common that the flotation plant uses a combination of xanthates or a combination between a xanthate and a co-collector like a dithiophosphate. Lotter et al. (2009) describe the proposed mechanism for the synergy between two or more collectors (Lotter & Bradshaw, 2009). The mechanism of the proposed synergy is outside the scope of this study, but it is interesting to note that the author summarises the proposed mechanisms driving the synergy between collectors as follows:

- "improved adsorption characteristics of the mixed collectors on the mineral surface"
- "better orientation of the different collectors forming a more evenly dispersed surface film than that of pure collectors".
- "a mixture of collectors may result in a balance of better distributed and more strongly held chemisorbed species and more hydrophobic neutral physisorbed

dithiolates, and this could result in the formation of a multilayer surface product that is more strongly attached to the mineral surface."

- "chemisorbed collector provides sites on the mineral surface for the subsequent adsorption of the more hydrophobic, neutral molecules (dixanthogen). The overall hydrophobic properties of the mineral are thereby increased."
- "The lower dosage recommended by several authors with mixtures of collectors may result from better distribution of the collectors to create the necessary 'hydrophobicity.' "
- "mixed collector system produced a more stable froth because of the increased hydrophobicity and wider range of particle sizes held in that froth."
- "mechanism for the synergy of the mixed system was by means of the production of extra dixanthogen driven by the metal thiolate formation of the DTC."

Analysis of the proposed mechanism is outside the scope of this study, but it is important to note that synergy between collector combinations is recognised by the industry and is widely used.

Bulatovic (2007) reports that a mixture of xanthates has been used to increase the selectivity and recovery of chalcopyrite copper ore (Bulatovic, 2007).

3.8. Plant testing of reagents

Irrespective of how carefully and vigorously a collector combination has been tested on laboratory scale, the commercial acceptance of the collector combination, in most instances, will be based on the plant data. Napier-Munn (1995) describes the problem with testing a collector combination on industrial scale well (Napier-Munn, 1995): "mineral processing engineers often conduct trials to improve the performance of their plants. A common problem in such trials is detecting real but relatively small improvements or changes in process performance against a background of very noisy data. This large data variance is frequently caused by (among other things) long-term time trends in performance, which can be a result of systematic changes in feed conditions."

Lotter et al. (2010) comments on the commercial opportunity of plant testing as follows (Lotter, et al., 2010): "small but economically significant performance gains across a concentrator operation present the problem of measurement and proof. This problem is generic for sulphide ore concentrators across the world. Historically the mineral processing industry has left these small gains to the realm of the unprovable, and has preferred to pursue larger performance gains because of their easier proof.

The cost of this approach has left a much larger total business opportunity on the table, since there are many more small performance gain opportunities than there are large ones. The leverage of statistics into mineral processing re-opens the discussion of these small recovery gains, because the test designs can deal with the central problem, viz. small performance changes within noisy data and associated with autocorrelation."

Napier-Munn (1995) gives the following advice (Napier-Munn, 1995): "In the ideal situation, the experiments should be designed with proper statistical principles in mind, and the data analysed accordingly. In reality, however, this tends to be the exception rather than the rule; the data are collected without much forethought and then presented for analysis, or production constraints make it very difficult to utilise classical experimental designs. In such cases, as will be seen, some compromise in the analysis may be necessary."

When planning a plant test the metallurgist needs to decide which method will be used to analyse and over what length of time should the test be conducted.

3.8.1. Alternating ON-OFF testing

One common method to test a collector combination on plant scale is to alternate a collector combination with the baseline collector suite for short periods. This eliminates any long term effect one parameter, for instance plant feed, may have on plant recovery (Napier-Munn & Meyer, 1999), (Napier-Munn, 2010). The practical disadvantage of this test method is that plant inventory carryover and synergistic effects between the collectors in the two test conditions are likely to mask the true effect of the test collector and for this reason Lotter et al. (2010) do not recommend this method (Lotter, et al., 2010).

For a simple ON-OFF test, two accepted statistical methods can be used to test if there is a significant difference between the mean of the measured plant data. One method is to pair the alternating data and calculate the difference between the pairs. The "t-test: paired sample for means method", which can be found in most statistics textbooks (Miller, et al., 1990).

A second method is to group all the data together where the baseline collector was used and all the data where the test collector was used and calculate the mean for these two groups. The "t-test for small samples" method (Miller, et al., 1990) can then be used to motivate if a significant change in plant data was measured.

Napier-Munn (1995) advises that although a 95% confidence level is preferred, 90% confidence is acceptable for a plant reagent test which means there is a 1 in 10 change that the recommendation made will not be correct (Napier-Munn, 1995).

3.8.2. Before, during and after testing of collector combinations on plant scale

Inventory carryover and collector interactions limit the use of the simple on-off test described in Section 3.8.1. As an alternative, a plant test is used where the test collector is tested over an extended, continuous period and the results are compared with the baseline condition before the test and the baseline condition after the test. There is both cost and risk associated with this type of test.

The disadvantage of this test is that the plant data can be influenced by an external factor, of which there are many, and it is also difficult to attribute only measured results to the collector being tested. A change in plant feedstock, ambient temperature, operator style and ability, water quality and flotation and mill wear are a few examples (Napier-Munn, 1995).

The period before the test can be compared with the period after the test to comment if any external process change has occurred.

3.8.3. Analysis of Variance (ANOVA)

Lotter and co-workers (Lotter, et al., 2010) have illustrated how the Analysis of Variance (ANOVA) method can be used to analyse a plant test where two collectors were compared.

The ANOVA single factor method used by Lotter et al. (2010) is explained in most statistics textbooks (Miller, et al., 1990).

Lotter et al. (2010) solve the problem of inventory carryover and collector synergy by discarding the first couple of tests when a collector change is made.

3.8.4. Analysing plant data by comparing confidence intervals

It is statistically correct to test multiple ON-OFF treatments to make it possible to measure the small recovery advantages locked in collector optimisation as stated by Lotter et al. (2010). The ANOVA method is well suited to cope with the potential complexities when trying to use the t-test for multiple treatments.

Even when a test has been well planned and executed with care and dedication, in some cases plant variation makes statistical testing and analysis of data difficult. In these circumstances the metallurgist has a choice to abandon the test data, or he can use it to form an opinion. This opinion is often not only based on the test data, but also on experience and "engineering ingenuity" (Sandenberg, 1999). To help the metallurgist to form an opinion, constructing and plotting confidence intervals can be used, in combination with ANOVA or t-test methods.

Confidence intervals for small sample methods can be used (Miller, et al., 1990) to construct confidence intervals for individual treatments and treatments replicas when conducting a multiple ON-OFF collector tests. Confidence intervals calculate an upper and lower confidence range. The average of a data set has 90% (if 90% confidence interval is used) probability of lying between the upper and lower limit. If the confidence interval for the plant tails values for two test periods overlap, then (at the 90% confidence interval) the averages of the two sets can be identical and the difference in means is not significant. If the two confidence intervals do not overlap, the means cannot be similar and the difference measured is significant. The advantage of this method is that it can be represented graphically and the metallurgist can use “engineering ingenuity” to form an opinion of the effect of the test collector.

3.8.5. Test size and plant test duration

Before a plant test is started, it is necessary to have an idea of how many tests need to be performed to be able to have a reasonable chance of proving the expected difference statistically. If a t-test is used, a method to calculate this sample size can be found in most statistics handbooks (Miller, et al., 1990), (Napier-Munn, 2010). To calculate the sample size the following is required:

- Data of the variability in plant data. This can be calculated from plant historical data.
- An estimate of what change in the data is expected when the collector combination is tested. This is a guess based on the laboratory data, previous plant tests or an opinion.
- The confidence level needed. In the examples a 90% confidence was selected.

The sample size needed can be obtained from **Figure 13**, if an approximate value for the above mentioned variables are known.

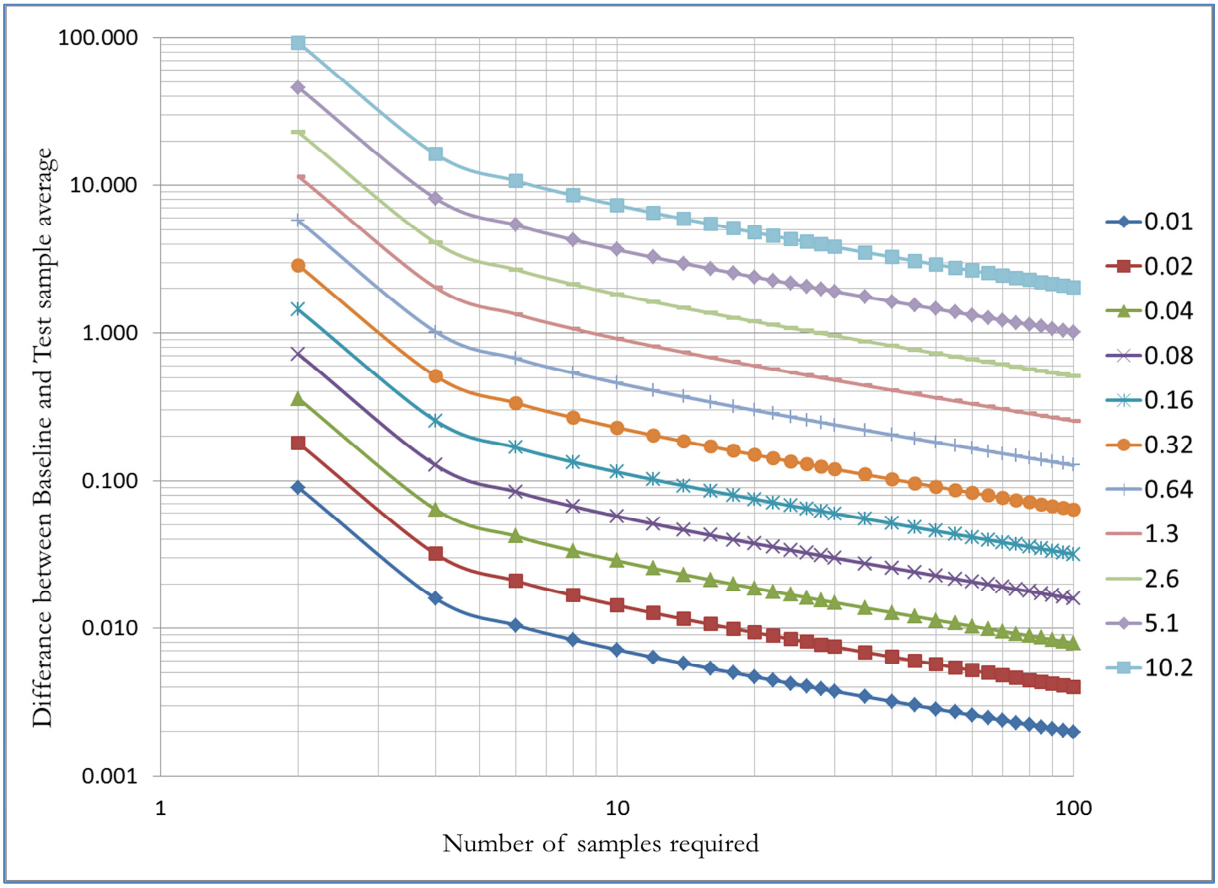


Figure 13: The number of samples needs to be taken (x-axis) can be read if the approximate standard deviation of the data is known (legend) and required error is known (y-axis) for a 90% confidence interval.

***($E = t_\alpha \cdot \frac{s}{\sqrt{n}}$). (E= difference between test and baseline sample mean, s= standard deviation of baseline sample set, n= number of samples t_α = value for t for confidence interval alpha)*

Napier-Munn (1995) gives the following advice to calculate the number of samples if a block design is planned (Napier-Munn, 1995): "The length of time represented by each test and each block will depend mainly on practical considerations, such as the ease of switching the mill, the impact of changeover on plant performance, and the time taken for the circuit to reach steady state again after the change. The number of blocks required will depend on the magnitude of the change being sought relative to the inherent noise (error) in the data.

To minimise the length of the whole period over which the experiment is to be conducted, the duration of each test (mill in or out) should be minimised, to say 24 hours, with another 24 hours following to allow the circuit to settle down after switching the mill. Therefore each block will last a maximum of 8 days.

The ANOVA should be calculated after each block and the experiment terminated after a firm conclusion is reached."

For practical reasons Betachem uses the following procedure (Shaw, 1998):

- Step 1: Crash test. Compare the test collector against the baseline for one shift off-one shift on and again one shift off and one shift on. If t-test data does not indicate a DECREASE in plant performance proceed to Step 2:
- Step 2: Short term test. Test the test collector for one week on and compare data with the week before and also with the week after. If t-test or ANOVA or confidence interval data does not indicate a DECREASE in plant performance, proceed to Step 3A or Step 3B :
- Step 3 A: Block long term test. Run one week OFF followed by one week ON. Repeat 8 times. The test duration is a total of 2 months. A decision should be taken on implementation based on t-test, ANOVA or confidence intervals indicating an INCREASE in plant performance. If the data is promising but an increase cannot be statistically shown, the test can continue for another 2 months. This will add data and would increase the confidence.
- Step 3 B: Long term test. Perform a test campaign for one month with the test collector and compare this data with the month before and month after using t-test, ANOVA method or confidence intervals. A decision should be taken on implementation based data indicating an INCREASE in plant performance. If the results are promising but not conclusive, another test of 1 month ON can be run followed by a month OFF. This will add data points and would increase confidence

4. Experimental procedure

4.1. Introduction

This section describes the methods used to generate data for this study. Some equipment and methods used were developed by the author to assist in the testing and comparison of collectors and other flotation reagents. The design criteria and basis for any method modification is described.

4.2. Bench flotation test

4.2.1. Description of experimental procedure

All laboratory flotation tests in this study were conducted using a Denver D12 laboratory scale flotation unit. It has been shown that by controlling the air flow rate and interface level in a batch flotation test, the reproducibility of the test data can be improved (Lindell & Dunne, 1984) (Luttrell & Yoon, 1983). The following modifications were made to the standard Denver D12 flotation equipment to accommodate these improvements:

- A Dwyer 2-10 ml/min air rotameter was installed on the air intake and air was supplied from an air compressor regulated to 200 KPa. (The rotameter was calibrated with an inverted measuring cylinder in water).
- A window was cut into the side of the stainless steel flotation cell and covered with transparent Perspex. The cell volume during a test is kept constant by adding water to the flotation cell (maintaining a constant level).

The test method followed is the same as the accepted method used by most laboratories (Flemming, 2005), (Lotter, 1993). Reaction between oxygen, steel grinding media, reagents and mineral surfaces affects flotation performance (Leppinen, et al., 2000). The following two changes were made to increase test reproducibility:

- The timed sequence for the test is started when the ore is placed in the mill and not when the flotation test starts. An attempt is therefore made to keep the milling, conditioning and flotation steps identical for all tests.
- An attempt is made to *not* wet mill for longer than 10 minutes or shorter than 4 minutes when a test program is undertaken. The ore sample is split into 10 kg subsamples after crushing and dry milled to a size where the required grind will be achieved with wet milling a 1 kg subsample for 5 minutes.

The laboratory flotation procedure used was as follows:

Ore preparation:

A minimum of 100 kg ore sample is taken from the mill feed belt, using a belt cut. The ore sample is air dried and then crushed to smaller than 20 mm in a jaw crusher.

The ore sample is split into approximately 10 kg subsamples by a cone-and-quarter method. (This calculated to a Gy relative sampling error of 3% if 100 kg sample is split into 10 kg subsamples at 20 mm top size).

One 10 kg subsample is dry milled to 100% passing 300 micron. To prevent over milling, the sample is dry milled for 1 hour, removed and sieved at 300 micron. The particles larger than 300 micron are returned to the mill and milled for another 1 hour. This procedure is repeated until all the particles are smaller than 300 micron.

A 1 kg subsample is split out and wet milled with 400 ml water. The mill is stopped at 4, 6, 8 and 10 minutes, and at each stop a 20 gram subsample is extracted, dried, weighed and wet sieved using a 75 micron sieve. This milling calibration curve is used to judge if the required wet milling particle size distribution is obtainable with wet milling duration of between 4 to 10 minutes. If not, the dry milling top size is either increased or reduced and another 10 kg subsample is milled, and the procedure is repeated until a dry milling top size is found were the required flotation size can be achieved with 4 to 10 minutes of wet milling.

If more than 10 kg of ore is needed for the flotation test program, another batch (s) of 10 kg is dry milled. All the batches are blended and split into 1.00 kg subsamples. (This is a Gy relative sampling error of approximately 0.03%). A cone-and-quarter method is used to approximate a 1 kg subsample. Ore is added to the sample to make up 1.00 kg by adding 1 gram random scoops from one of the subsamples. This is done by spreading the ore out and taking random scoop samples.

The portion of the original ore sample not dry milled is bagged, labeled and stored. The 1 kg dry milled subsamples are stored in a fridge to limit oxidation and used within 4 weeks of dry milling. Samples stored for longer than 4 weeks are not used for flotation tests.

To confirm the milling calibration step, one 1 kg subsample is wet milled, with 400 ml of water (for the milling time determined during the milling calibration step) and subjected to full screening analysis. The particle size distribution is recorded and assumed to be similar for each test.

Preparation of reagents:

If xanthates are being tested and compared to each other, the xanthates are purified (Section 8.1) the day before use. This prevents activity of the xanthate powder being a contributing factor when data is analysed. If a single xanthate is used in the test program, the commercial grade xanthate is used. The activity of the xanthate is measured and recorded (Section 8.2).

A fresh batch of xanthate powder is diluted to a 1% solution with municipal quality water each morning before testing. Loss in xanthate activity when made up with municipal water at 25 degrees was tested to be 6% in 48 hours (Section 8.2). It therefore seems practical to use a xanthate solution within 12 hours, but to discard it after 12 hours.

All reagents, except depressants, are diluted to a 1% solution in the morning of the day of the test. Depressants, which require hydration time, are made up fresh the morning of the day before the test.

Reagents not soluble in water are used undiluted and added with micro syringe or micropipette. The micro syringe is preferred since it has been found that for reagents with high viscosities, the volume dosed with the micropipette can vary unpredictably. The mass of the syringe before and after dosing is recorded to verify if the correct amount of reagent has been added.

Flotation test:

As described in Section 3.2 the flotation test needs to measure the rate of flotation as well as the terminal or maximum recovery possible when collectors are compared. For this reason concentrates at various time intervals are collected.

The time intervals need to be selected so that the rate of flotation can be compared. Curve fitting to an appropriate model can be used (Hay, 2005). This unfortunately adds to the complexity of interpreting flotation data and requires 4 or more points on the time recovery curve to fit a simple first order Klimpel equation with a minimum of one degree of freedom. It also adds to assay complexity as more samples taken add to the relative assay variance and thus reduces test accuracy, especially if the errors are cumulative. A practical alternative suggested by Pistorius (2009) is to use the recovery measured at 5 minutes (R_5) as an approximation of the rate of flotation. At 0-5 minutes the rate of flotation (recovery per unit time) is still proximately linear and a practical measure to compare the rate of flotation (Pistorius, 2009). The practical advantage of this method is that only one concentrate needs to be taken.

As a practical alternative to calculated terminal recovery data from curve fitting, the recovery at forty minutes is assumed to be the terminal recovery. This assumption is tested by measuring the recovery at 20 minutes and comparing this with the recovery at

40 minutes. The assumption is valid if the recovery at 40 minutes does not vary much from the recovery at 20 minutes. The R_{40} is taken as the terminal recovery if $R_{20} \approx R_{40}$.

A typical timed sequence flotation test where 5 minutes wet milling is required and only xanthate is added with 1 minute conditioning time is shown in **Table 1** and serves as an example of the flotation test sequence followed.

Table 1: Example of the flotation sequence where 5 minutes wet milling is required and xanthate is added after milling followed by 1 minute conditioning time.

Time flag (minutes)	**Activity**
0	Add 1 kg dry milled sample to wet mill, with 400 mℓ of water and reagents if required. Close mill and put on the rollers. Wait for next time flag
1 minute	Start mill.
6 minutes	Stop mill Open the lid and remove the rods while washing with water. Transfer the content of mill into the flotation cell. Put flotation cell in position and lower the flotation mechanism. Add water to a position below the "interface" mark. Wait for next time flag
9 minutes.	Start flotation mechanism. Add flotation reagents. Put a concentrate pan under the overflow lip (labeled 5 min concentrate) Wait for next time flag
10 minutes	Open the air and adjust to 4 ℓ/min. Fill the cell to the "interface" mark Scrape froth every 30 seconds. Continue to keep pulp level on the "interface" mark by adding water. Continue until the next time flag.
15 minutes	Remove the concentrate pan and replace with an empty pan. (labeled 5 to 30 min concentrate) Fill the cell to the "interface" mark Scrape froth every 1 minute. Continue to keep pulp level on the "interface" mark by adding water. Continue until the next time flag.
30 min	Remove the concentrate pan and replace with an empty pan. (labeled 30 to 40 min) Fill the cell to the "interface" mark

	Scrape froth every 2 minutes. Continue to keep pulp level on the "interface" mark by adding water. Continue until the next time flag.
50 min	Close air Stop flotation mechanism
Untimed	Transfer the cell content to a pressure filter and filter to a moist cake. Put in oven at a temperature not higher than 50 degrees until dry. Record weight of dry tails sample. Assay for value element and/or mineral.
Untimed	Record weights of the three concentrate pans plus the concentrate. Put pans in oven at a temperature not higher than 50 degrees until dry. Record the weight of the pans plus dry weight. Transfer the content of the pans into appropriate container and record weight of dry pan. Assay for value element and/or mineral.

4.2.2. Verification of method

4.2.2.1. Reproducibility of results

Four repeat bench flotation tests were conducted using the nickel ore described in Section 4.9.5. Percentage standard deviation values (standard deviation / average x 100) are shown in **Table 2**, **Table 3** and **Table 4**. The copper and nickel percentage standard deviation for recovery and grade data were between 1.5 and 15%, which is higher than the target 3% suggested by Lotter et al. (2009). A single repeat would thus not be satisfactory, and at least 3 repeats were done. To indicate repeatability the standard error is used to plot error bars (Standard error = standard deviation / square root of number of data points).

Table 2: Four repeat bench flotation results conducted using a nickel ore containing pentlandite, chalcopyrite and pyrrhotite**.

	Concentrate 1			Concentrate 2			Concentrate 3			Tails			Feed			Call factor	
	0 to 5 min			5 to 20 min			20 to 40 min										
	Dry Mass g	Cu %	Ni %	Dry Mass g	Cu %	Ni %	Dry Mass g	Cu %	Ni %	Dry Mass g	Cu %	Ni %	Dry Mas g	Cu %	Ni %	Cu %	Ni %
Repeat 1	44	1.78	0.99	76	0.51	0.69	58	0.13	2.13	821	0.03	0.25	1000	0.14	0.43	0.98	1.01
Repeat 2	39	2.20	1.07	70	0.41	0.87	52	0.15	1.93	839	0.03	0.25	1000	0.14	0.43	0.97	1.04
Repeat 3	33	2.56	1.04	58	0.50	0.81	65	0.13	2.14	844	0.03	0.24	1000	0.14	0.43	0.97	1.01
Repeat 4	37	2.21	1.03	53	0.61	0.73	79	0.13	1.94	830	0.03	0.24	1000	0.14	0.43	0.96	1.00
% Stdev	12.5	14.7	3.1	16.7	16.7	10.4	18.2	6.7	5.6	1.2	6.6	2.5					

***Call factor : Measured metal content in feed based on feed assay and mass/calculated metal content in feed based on concentrate and tails assay and mass.*

*** Frother used, Betafroth 436a at 30 g/t, depressant used, Betamin 544 at 250 g/t and collector used, Betacol 380AB at 250 g/t. Mill grind, 70% passing 75 micron. Conditioning time 1 minute.*

Table 3: Four repeat bench flotation results conducted using a nickel ore containing pentlandite, chalcopyrite and pyrrhotite**.

	Copper						Nickel					
	Recovery (%)			Grade (%)			Recovery (%)			Grade (%)		
	0-5 min	0-20 min	0-40 min	0-5 min	0-20 min	0-40 min	0-5 min	0-20 min	0-40 min	0-5 min	0-20 min	0-40 min
Repeat 1	54	80	85	1.8	1.0	0.7	10	23	52	1.0	0.8	1.2
Repeat 2	58	78	83	2.2	1.0	0.8	10	25	49	1.1	0.9	1.3
Repeat 3	57	77	83	2.6	1.2	0.8	8	19	52	1.0	0.9	1.4
Repeat 4	55	77	84	2.2	1.3	0.7	9	18	54	1.0	0.9	1.4
% Stdev	3.58	2.08	1.46	14.70	12.95	4.53	11.33	14.86	3.70	3.06	6.78	6.44

Table 4: Four repeat bench flotation results conducted using a nickel ore containing pentlandite, chalcopyrite and pyrrhotite **.

	Nickel					Copper				
	R40 %	G40 %	K5 %/min	G5 %	Call Factor %	R40 %	G40 %	K5 %/min	G5 %	Call Factor %
Repeat 1	52	1.23	2.1	1.0	1.01	85	0.70	10.8	1.8	0.98
Repeat 2	49	1.26	2.0	1.1	1.04	83	0.76	11.7	2.2	0.97
Repeat 3	52	1.42	1.6	1.0	1.01	83	0.78	11.4	2.6	0.97
Repeat 4	54	1.36	1.8	1.0	1.00	84	0.74	11.1	2.2	0.96
	3.70	6.44	11.33	3.06		1.46	4.53	3.58	14.70	

R40 : Recovery of value element after 40 minutes of flotation
G40 : Grade of value element alter 40 minutes of flotation
K5 : Recovery per minute measured after 5 minutes of flotation
G5 : Grade of value element alter 40 minutes of flotation

4.2.2.2. Comparison with plant and published data

The copper value mineral in this ore is chalcopyrite, which as described in Section 4.9.5 floats fast and high recoveries can be achieved, providing the chalcopyrite is liberated and in the flotation range of 10 to 100 microns. From the scanning electron microscope study described in Section 4.9.5 it can be assumed the chalcopyrite is liberated at the grind used for this test (70% passing 75 micron). For this test the copper recovery (chalcopyrite) was measured to be fast and approached 90% at 40 minutes (**Figure 14**).

As expected (Section 3.7) the rate of nickel recovery (pentlandite) is slower than the rate of copper (chalcopyrite) recovery (**Figure 15**).

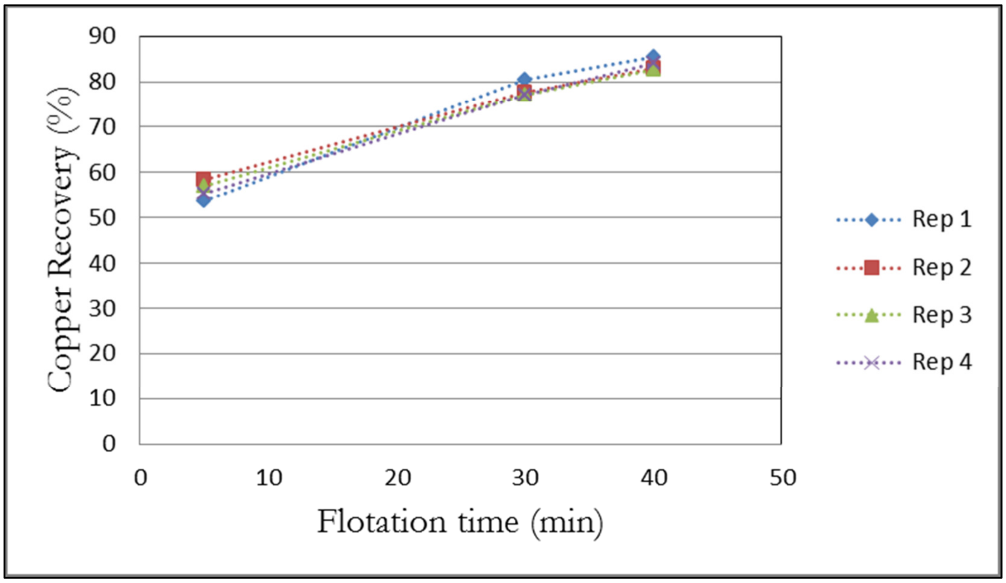


Figure 14: Copper recovery results for 4 repeat bench flotation tests using a nickel ore described in Section 4.9.5

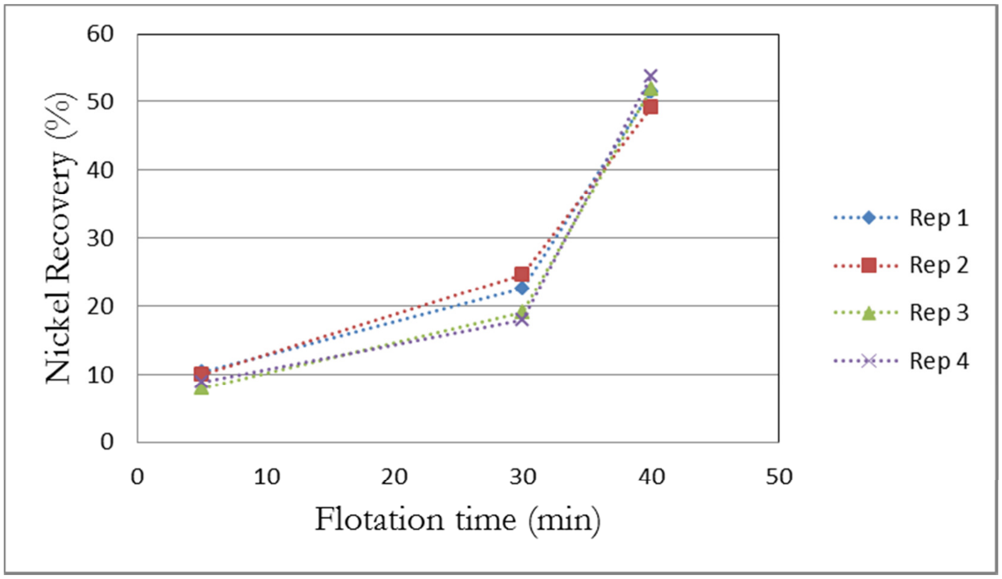


Figure 15: Nickel recovery results for 4 repeat bench flotation tests using a nickel ore described in Section 4.9.5

A histogram for the copper and nickel recoveries measured using the bench flotation test method (**Table 3**) is plotted against plant data in **Figure 16** and **Figure 17**. The bench-scale recovery data corresponds well with the plant data and it is concluded that the bench flotation test is a reasonable indication of recoveries expected on plant scale.

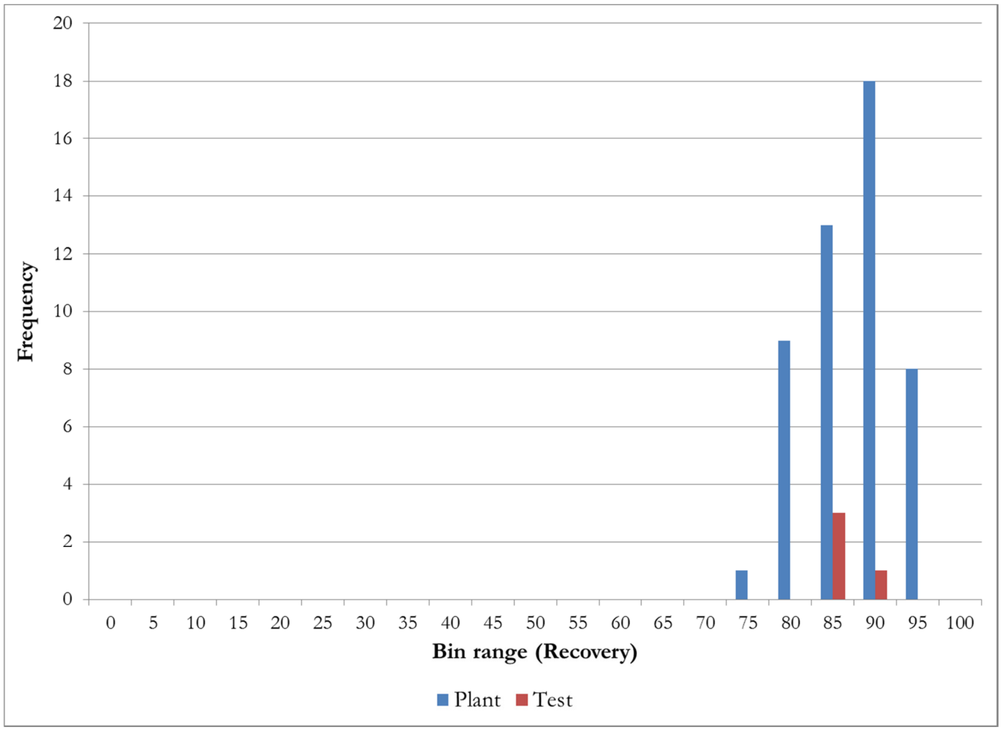


Figure 16: Histogram of copper recovery. Plant shift production data and laboratory – scale replica test for a nickel ore described in Section 4.9.5

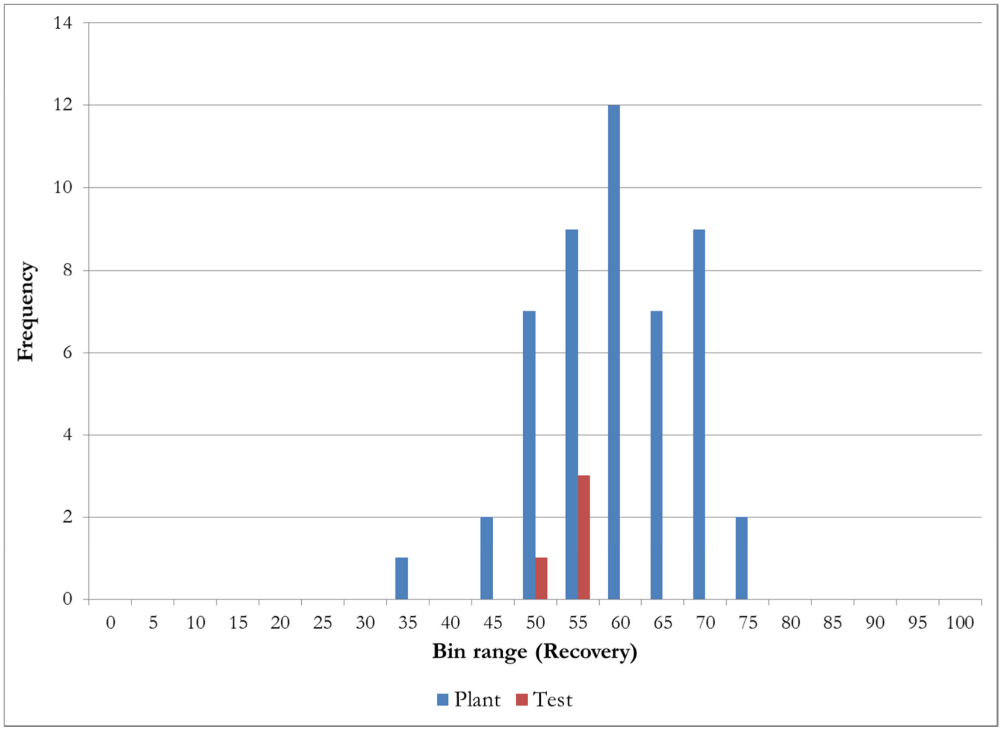


Figure 17: Histogram of nickel recovery. Plant shift production data and laboratory – scale replica test for a nickel ore described in Section 4.9.5

4.3. Bikerman froth stability column.

4.3.1. Design of Bikerman froth stability column

The Bikerman froth stability column is a batch flotation column with an air only sparger system based on the air sparger system used by the Microcell from Metso Minerals (Metso Minerals, 2010). This unit can be used to test froth characteristics in a three phase system (solids, liquid and gas) similar to the test proposed by Bikerman to test froth characteristics of detergents in a two phase system (liquid, gas) (Bikerman, 1973).

The design criteria were:

- A unit that simulated the conditions in an industrial flotation cell where the effect of a reagent (collector, frother and depressant) on froth characteristics could be measured.
- The test unit had to be practical to use.

- One kg milled ore per test was set as a desirable size for the unit. (This conforms to the sampling accuracy method used for batch flotation tests (Section 3.3.)

The Bikerman froth stability design consists of the following parts (**Figure 21**):

Recirculation pump

The highest cost part of the unit was recognised as the pump that recirculates the pulp and prevents the settling of solids. The cost of conventional slurry pump was outside the budget for this study, and a submergible pump, capable of handling solids, was selected as an alternative. A QDN 1.5-4.5-0.08 kw submersible pump from Pump Solutions was selected as a reasonable compromise between cost and the ability to pump pulp. This selection limited the percentage solids of the pulp to 10%. This pump delivers a flow rate of approximately 30 l/min (1.6 m^3/h) at a head of approximately 4 m (see pump curve in **Figure 18**). With the flow reduced by the static mixers and inlet pipework, the flow is reduced to 30 l/min and no additional flow restriction mechanism was needed.

To effectively disperse air into liquid, an air to liquid ratio of 1:3 or higher needs to be maintained (Terblanche, 1999). For 30 l/min pulp recirculation, the air flow rate is limited to approximately 10 l/min.

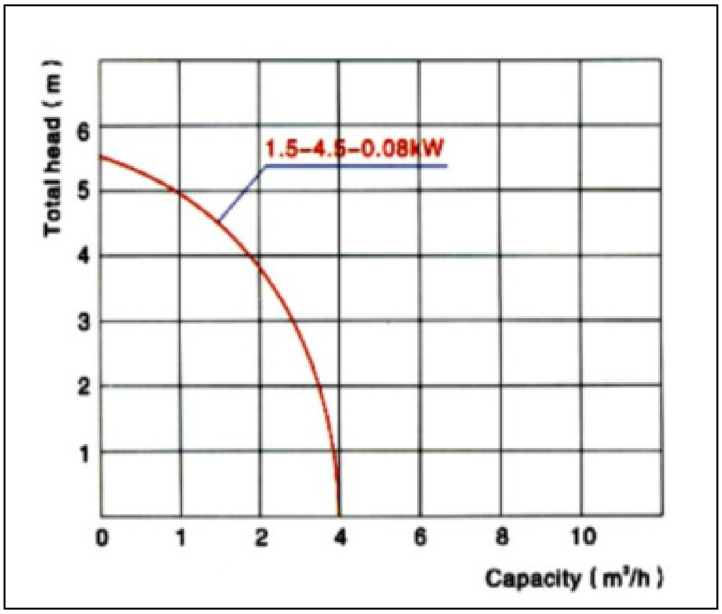


Figure 18: Pump curve for QDN 1.5-4.5-0.08 kw submersible pump

Pulp container

The QDN 1.5-4.5 pump has an outside diameter of 112mm, making it suitable to be installed inside a 160 mm PVC pipe section. To reduce the settling of solids in the pulp container, the inside diameter of the pulp container was selected to be just larger than the outside diameter of the pump.

Approximately 10 litres of pulp at 10% solids (by mass) will contain 1 kg of dry solids. The total volume of the pulp container must as a result be at least 10 litres.

Froth stability column

In the lower section of the froth stability column, pulp is re-circulated. This flow must be high enough to achieve air bubble dispersion, but low enough to allow air bubbles to rise and not be dragged to the outlet of the column in the recirculation stream. Bubble size in a flotation cell has been measured as being typically between 0.6 and 0.7 mm in diameter. (Chen, et al., 2001). Talaia (2007) measured the rise velocity of a 0.7 mm bubble in water as approximately 18 cm/s (**Figure 19**). For more viscous fluids the rise velocity decreases.

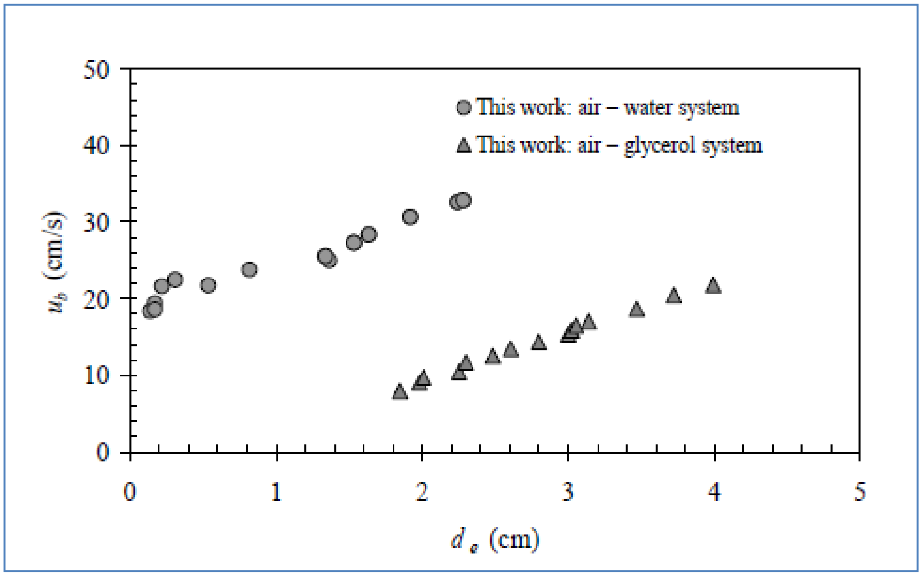


Figure 19: Rise velocity for a single air bubble measured in water and in glycerol (Talaia, 2007).

Pulp recirculation flow of 90 l/min is equal to a superficial liquid velocity of around 18 cm/s (assuming perfect plug flow) in a 100 mm diameter column. 90 l/min is therefore the limit in flow where an air bubble of 0.7 mm will rise in a 100 mm diameter column. At a safety factor of 3 the maximum superficial liquid velocity should be 4.6 cm/s and the maximum liquid flow for a 100 mm column should be 30 l/min. A 100 mm diameter column is therefore suitable for a pump capacity of 30 l/min as delivered by the QDN 1.5-4.5-0.08 kw 220v pump from Pump Solutions.

A 100 mm diameter Perspex tube (3 mm wall thickness) was selected as the material of construction for the froth stability column. This selection is a compromise between the wall effects for froth bubbles larger than 100 mm and practical considerations, for instance the amount of ore used per test and pump capacity.

If the submersible pump cavitates, air will be introduced into the column. A minimum height of 150 mm of pulp needs to be kept in the pulp container at all times to prevent cavitation. A minimum liquid volume of 2 litres therefore needs to be kept in the pulp container when the unit is in operation. Eight litres of pulp is consequently available in the pulp zone of the column which is approximately 1 meter pulp in a 100 diameter column. To measure a maximum of 700 mm froth, total column length therefore needs to be 1.7 meters or higher.

Static mixer

A 50 mm x 50 mm x 400 mm static mixer was selected based on practical construction considerations. No static mixers of this size were commercially available and one was designed and manufactured from 3 mm Perspex. The design of this mixer is outside the scope of this study and will not be discussed further.

Rotameter

To maintain an air superficial velocity of 1 cm/s (Terblanche, 1999), a 6 l/min air flow rate is required for a 100 mm column. This is also within the maximum air limit based on the pump selection of 10 l/min. A Dwyer 2 to 10 l/min rotameter was selected. The air supply is piped through an air dryer pressure controller to remove any moisture from the compressed air, which might affect the rotameter. The air pressure is controlled to 200 kPa to prevent pressure fluctuations affecting the air flow rate measurement in normal l/min.

Level overflow arrangement (level control)

An overflow pipe with a siphon breaker connects the pulp container with the column as indicated in the schematic in **Figure 20**. This arrangement is used to maintain a constant level in the column.

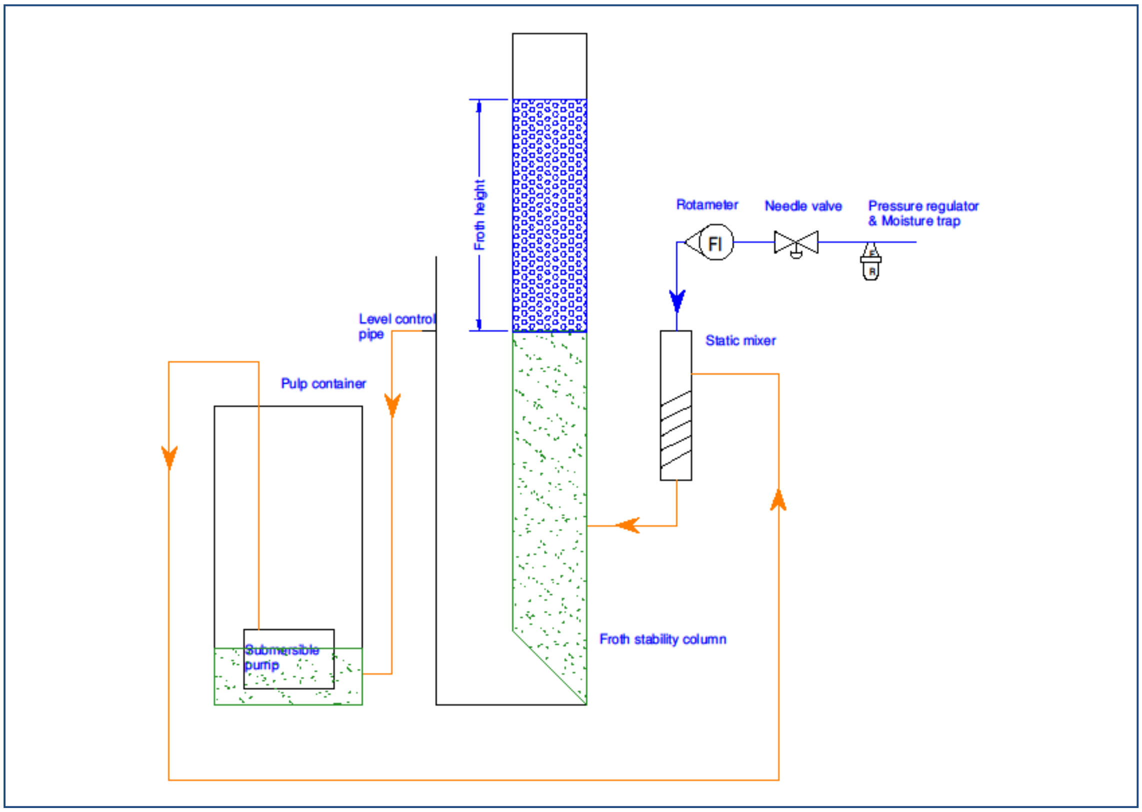


Figure 20: Flow diagram for the Bikerman froth stability column

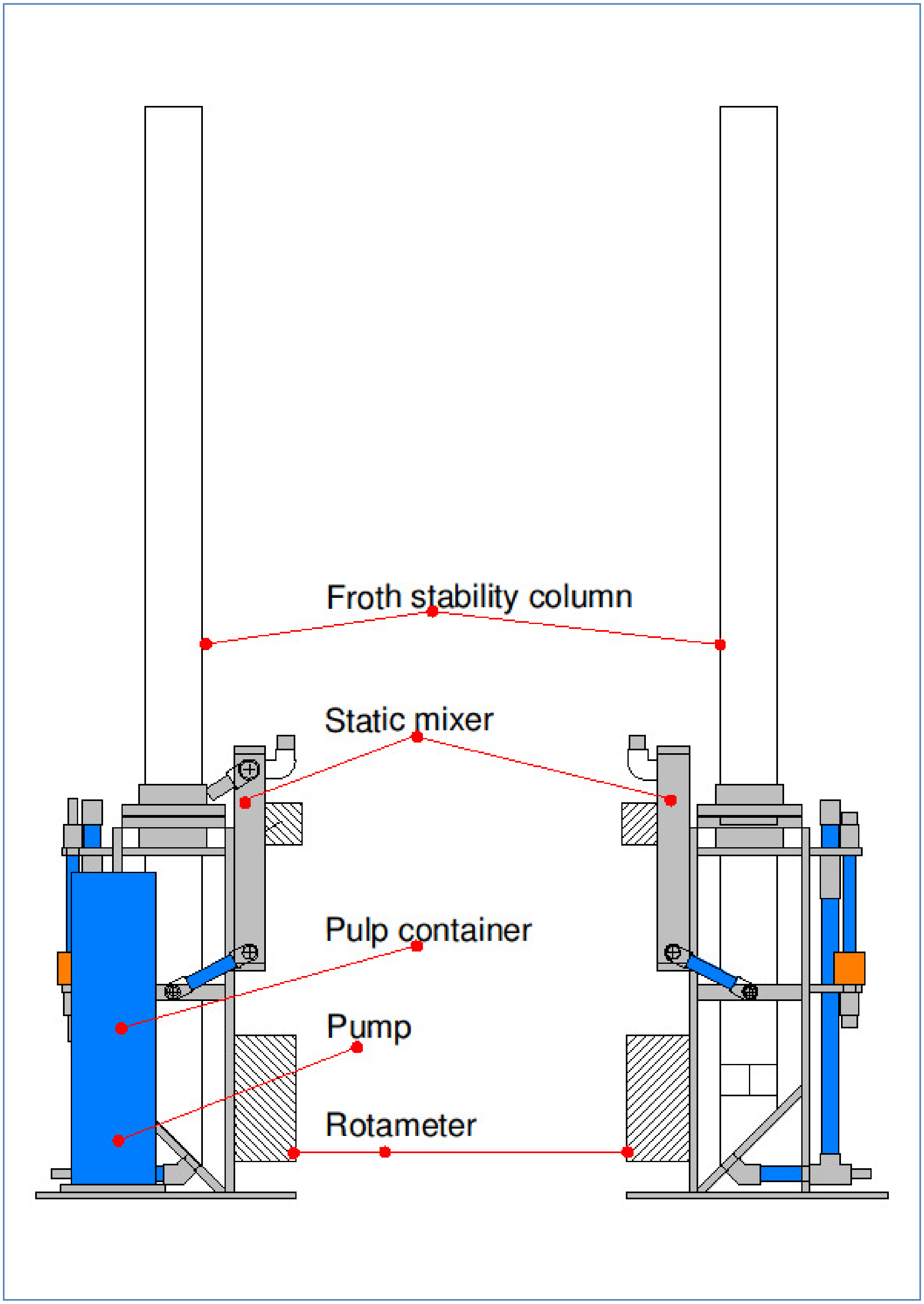


Figure 21: GA drawing of the Bikerman froth stability column manufactured by Betachem (Pty) Ltd (left and right view).

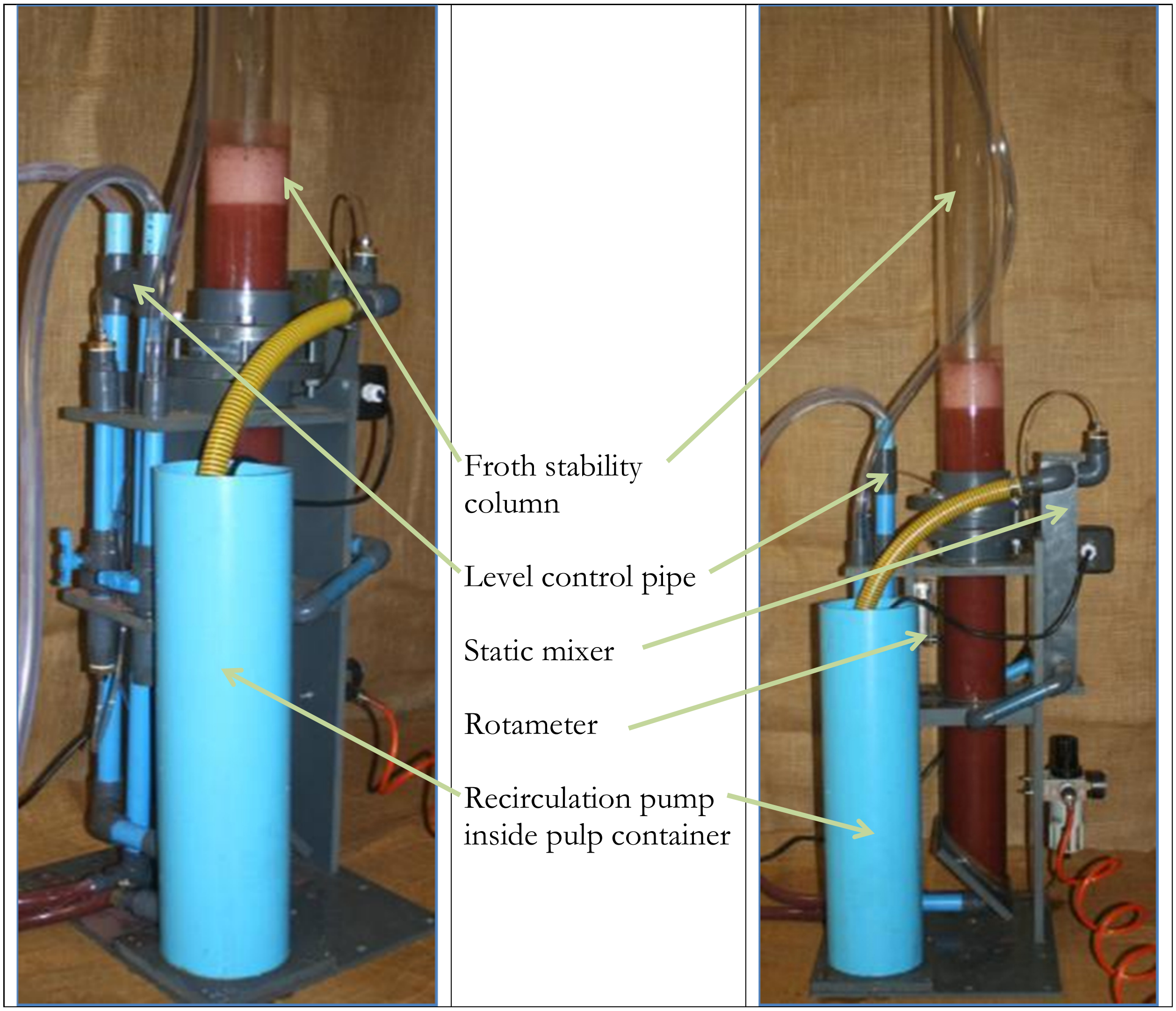


Figure 22: Photos of the Bikerman froth stability column (manufactured by Betachem (Pty) Ltd) in operation.

4.3.2. Description of experimental procedure

The ore preparation and reagent preparation method is similar to the method described in Section 4.2.1. Reagents are made up as discussed under 4.2.1

With the reagents and ore sample prepared, the method to test the effect of a collector, depressant or frother on froth stability is as follows:

A 1 kg ore sample is wet milled with 400 ml of water for a period predetermined to achieve the required particle size distribution (see Section 4.2.1.). This pulp is transferred into the pulp container of the Bikerman froth stability column, 9 litres of water is added and the recirculation pump switched on.

With the pump running, add all the required reagents necessary to simulate the plant reagent suite, except the reagent being tested. All reagents are added in the pulp for sufficient mixing and distribution.

Next, open the air valve and set air flow rate to 6 l/min and the air pressure to 200kPa. Froth will form and slowly build up in the column. Wait until a stable froth has formed and the froth height has stabilised. At this height the froth breakdown rate is equal to the froth formation rate. Measure the froth height (from the froth surface to the pulp froth interface) with a suitable instrument like a tape measure and record this height. Close the air valve and record the time the froth takes to break down.

Decide what the maximum dosage of the test reagent should be. Divide this maximum dosage by 10 (if ten tests are planned,) and add this amount to the pulp container. All reagents need to be added below the pulp surface in a turbulent area. The air valve is opened again and the test is repeated.

4.3.3. Verification of method

4.3.3.1. Reproducibility of results

Five repeat tests were performed using a typical South African Merensky platinum ore. This ore was dry milled to 100% passing 212 by milling for 1 hour, sieving at 212 micron and returning only the plus 212 micron fraction to the dry mill. The minus 212 micron sample was split into 1kg subsamples. SIBX was added at 100 g/t and Betafroth 436 (ethylene glycol ether) was used as frother. The frother dosage in this test was increased from 0 to 0.03 gram per litre (100 gram per ton equivalent). The stable froth height measured is listed in **Table 5** and shown in **Figure 23**. The average percentage standard error (% standard deviation / average) is 4.5%, and indicates acceptable reproducibility if the criteria proposed by Lotter are used (Lotter & Fragomeni, 2010).

The decrease in froth stability at a higher dosage is noted. This was also observed by Tan et al (2005), and ascribed to antifoam properties developing when the solubility limit of a frother is reached (Tan, et al., 2005). It is also possible that this decrease is related to the reduction in bubble size with a frother dosage increase and a decrease in bubble rise velocity. A size is reached where the bubbles do not rise to form froth, but reports to the column tails (Dobby & Finch, 1990). Further work can be done to confirm this, but is outside the scope of this study.

Table 5: Stable froth height measured with Bikerman froth stability column for 5 repeats**.

Frother g/l	Stable froth height - mm (Bikerman method)					% Std Error
	Repeat 1	Repeat 2	Repeat 3	Repeat 4	Repeat 5	
0.000	60	50	50	50	50	8.6
0.005	90	90	90	90	100	4.9
0.010	160	180	180	190	180	6.2
0.015	230	230	250	230	240	3.8
0.020	300	300	300	280	280	3.8
0.025	290	300	310	290	300	2.8
0.030	230	230	230	240	240	2.3

*** Merensky ore milled to 100% passing 212 micron, 100 g/t SIBX, Betafroth 436 as indicated.*

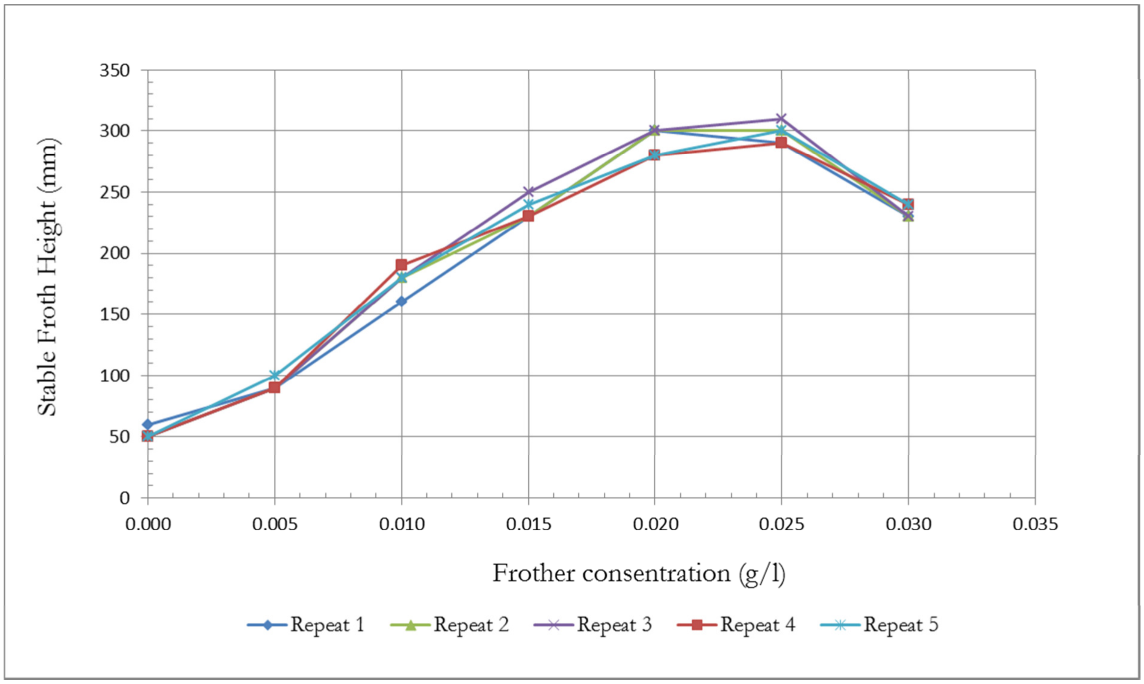


Figure 23: Stable froth height measured with Bikerman froth stability column for 5 repeats**

***Merensky ore milled to 100% passing 212 micron, 100 g/t SIBX, Betafroth 436 as indicated.*

Five repeat tests were performed using a typical Zimbabwe Great Dyke platinum ore dry milled to 100% passing 212. SIBX was added at 400 g/t and Betafroth 436 (ethylene glycol ether) was used as frother at a concentration of 0.01 g/l. Betamin 537 (a high purity CMC -carboxymethyl cellulose) depressant was used as gangue depressant to depress talc. The CMC depressant dosage was increased from 0 to 250 g/t in 50 g/t increments. The stable froth height measured is listed in **Table 5** and shown in **Figure 23**. The average percentage standard error (% standard deviation / average) is 4.6% and indicates acceptable reproducibility (Lotter & Fragomeni, 2010).

Table 6: Stable froth height measured with the Bikerman froth stability column for 5 repeats**.

Depressant g/ton	Stable froth height (Bikerman method)					% Std Error
	Repeat 1	Repeat 2	Repeat 3	Repeat 4	Repeat 5	
0	810	870	840	870	820	3.3
50	530	530	550	560	540	2.4
100	300	310	320	300	300	2.9
150	140	150	170	140	160	8.6
200	70	80	80	80	80	5.7
250	60	60	55	60	60	3.8

***Great Dyke ore milled to 100% passing 212 micron, 400 g/t SIBX, 0.01 g/l Betafroth 436 and Betamin 537 talc depressant as indicated.*

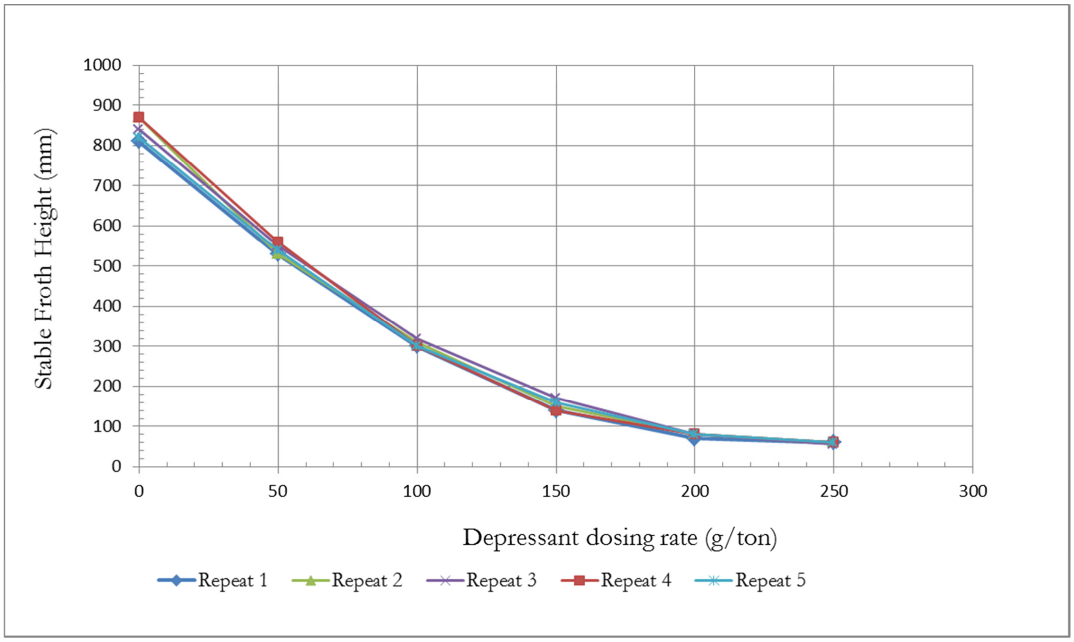


Figure 24: Stable froth height measured with Bikerman froth stability column for 5 repeats**

***Great Dyke platinum ore milled to 100% passing 212 micron, 400 g/t SIBX, 0.01 g/l Betafroth 436 and Betamin 537 talc depressant as indicated.*

4.3.3.2. Comparison with published data

It is well know that an increase in frother concentration results in an increase in froth stability. Tan et al. (2005) has indicated that this is true up to a critical concentration, after which froth stability is not increased further and may even decrease (**Figure 25**) (Tan, et al., 2005). In this study Tan et al. (2005) also indicated that PPG 400 produces a more stable froth (higher stable froth height) than the weaker frother MIBC, and PPG 2000, (which is commonly used as an antifoam in two phase systems) produces the least stable froth (**Figure 26**).

The stable froth height for a range of frothers were tested, using the Bikerman froth stability column with an ore and reagent suite for a South African Merensky operation (**Figure 27**). The stable froth height increases at the same frother concentration for frothers with increasing strengths, Dowfroth 200<Dowfroth 250<Dowfroth 400.

Talc is a natural hydrophobic mineral and as a result of this and its fine particle size it forms phenomenally stable froths in flotation circuits (Lotter, et al., 2008). A gangue depressant that can depress the natural floatability of talc would thus show a froth destabilisation effect as seen in **Figure 28**. The results in **Figure 28** indicate that all the Betamin depressants tested for this ore depress talc, and a dosage of 150 g/t would be required for threshold talc depression (at this dosage the froth height remains constant indicating more talc is not depressed at higher depressant dosages). This data conforms to the threshold depressant dosage tested on this plant.

These results support the hypothesis that the Bikerman froth stability column can be used to test the frothing characteristics of a collector combination. The test method does not only measure the frothing characteristics (analogy to frothers), but also any froth stability increase as a result of gangue activation. Both these mechanisms can result in an increase in froth stability, higher mass recovery and thus higher value mineral recovery which may not be related to higher value mineral floatability as described in Section 3.2.

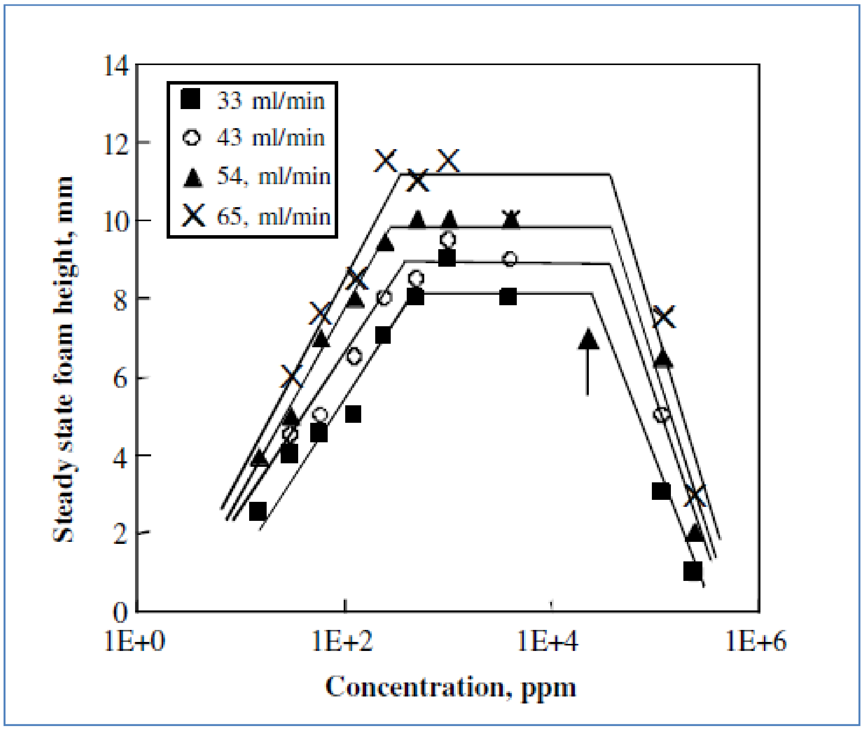


Figure 25: Foamability versus concentration profile of MIBC at a range of gas flow rates (Tan, et al., 2005)

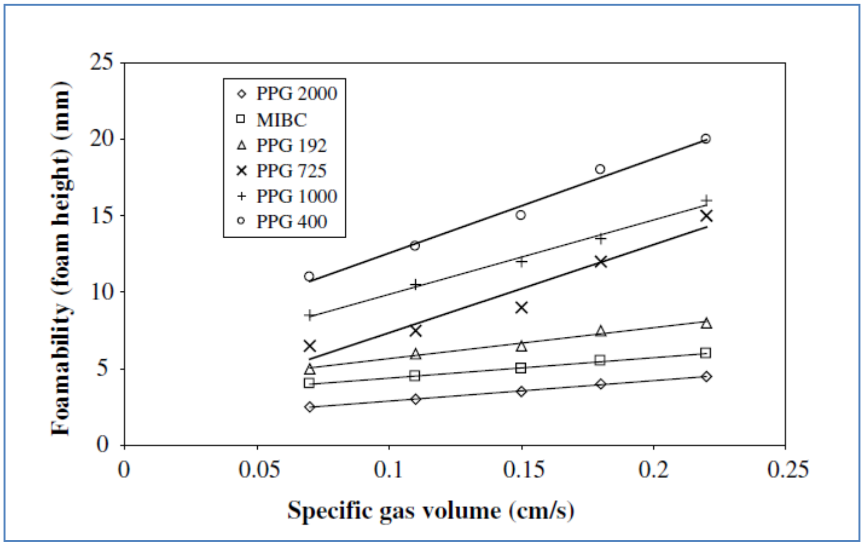


Figure 26: The steady state foam height of the PPG frothers (measured with a froth stability column) compared to MIBC as a function of the specific gas flow rate (concentration 20 ppm) at a range of gas flow rates. (Tan, et al., 2005)

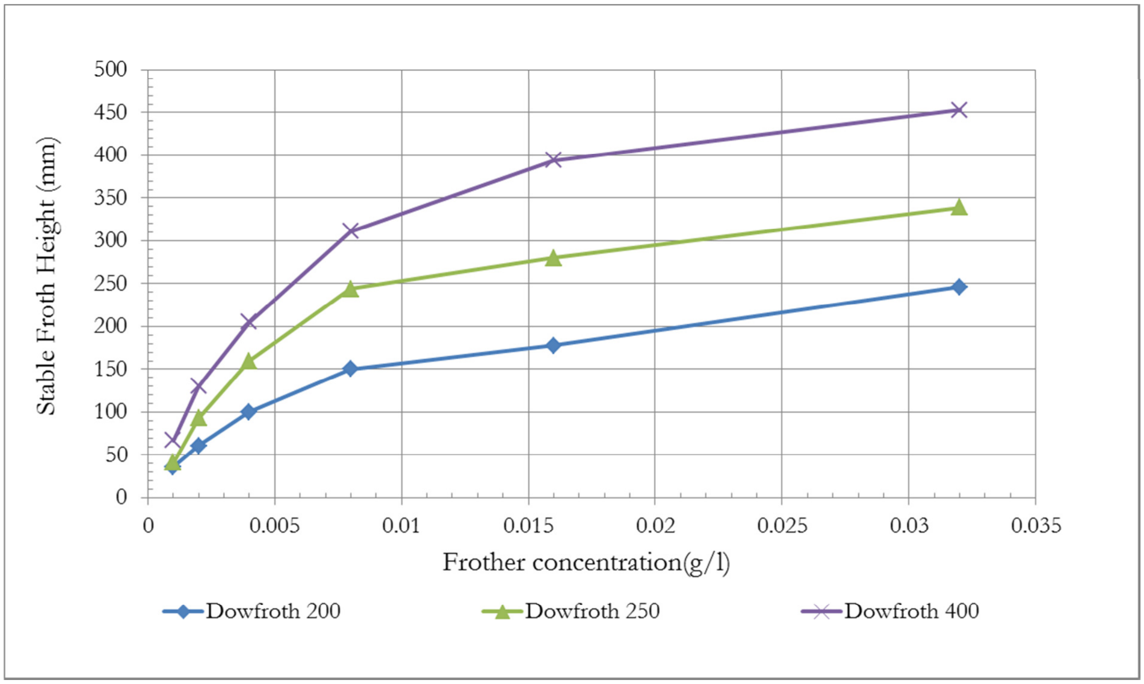


Figure 27: Stable froth height measured for frothers consisting of Dowfroth 200 (medium strong frother), Dowfroth 250 (strong frother) to Dowfroth 400 (very strong frother) using the Bikerman froth stability column**. .

***Tests were done using a South African Platinum mine ore, milled to 70 % passing 75 micron. 100 g/t SIBX*

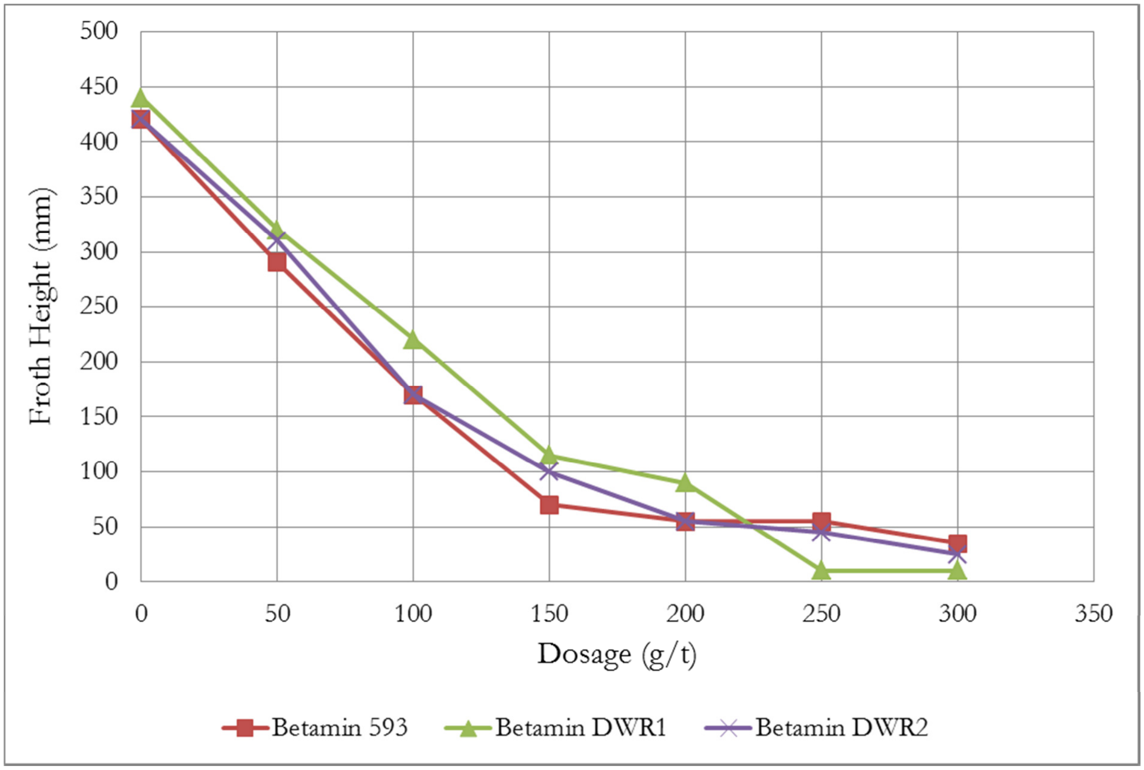


Figure 28: Stable froth height measured for three talc depressants**

***Tests were performed using a South African platinum ore, milled to 70 % passing 75 micron. Collector concentration: 100 g/t SIBX.*

4.4. Micro flotation cell

4.4.1. Design of the Bubble-Flux micro flotation cell

The use of a small scale flotation unit to study the flotation recovery of mineral specimens is well documented (Rao & Leya, 2004).

The micro flotation cell described by Bradshaw et al. (2006) was specifically designed to study the effect of particle loading on a single bubble (Bradshaw & O'Conner, 2006). An attempt was made to modify this design to resemble an industrial flotation cell by changing the air sparging system from a single to multiple air bubbles. The aim of this design change is to ensure particle floatability is the rate limiting step and not bubble surface flux, as will be described in the following section.

The flotation process can be subdivided into four sequential processes. The first process is the reaction of a surfactant (collector and/ or co-collector) with the value mineral to render the surface of the value mineral hydrophobic. The second sequential process is the successful contact between the hydrophobic particle and an air bubble which results in the particle being attached to the air bubble. The third process is the transportation of the air bubble and particle to the froth phase. The fourth process is the successful transportation of the particle to the froth overflow lip. The ideal micro flotation cell to study the effect of collectors is a unit where process 2, 3 and 4 remain equal so that a difference in recovery can be attributed to the first process only, which is the reaction of a collector and/ or co-collector with the value mineral to make its surface hydrophobic. The ideal micro flotation cell to test collectors should therefore measure hydrophobicity or floatability.

The rate of flotation has been discussed in Section 3.2 and can be expressed as:

$$k = P.6.\frac{J_g}{D_b}.R_f \qquad (7)$$

Where k is the rate of flotation (mi n^{-1}), P is the floatability of the value mineral, J_g is the superficial gas velocity (cm/s), D_b is the bubble diameter (m) and R_f is the recovery of the value mineral in the froth phase.

If in a micro flotation cell design, J_g, D_b, and R_f are kept constant, then a change in the rate of flotation (k) would indicate a change in floatability (P). Particle floatability (P) can thus be measured in a micro flotation cell by measuring the rate of mass recovery of a value mineral specimen. In the discussion that follows it will be explained how the bubble flux micro-cell was designed to keep J_g, D_b, and R_f constant.

The term floatability in the equation above is used as a collective noun for a number of properties of which surface hydrophobicity is only one. Attachment and detachment of the value mineral to the air bubble in the pulp phase are properties also grouped as mineral floatability, and are related to the particles size, bubble size and competition for available bubble surface. The fundamental modification of the cell used in this study is aimed at producing a large number of air bubbles and establishing the environment for efficient bubble particle contact. A change in floatability (measured as a change in the rate of recovery) has a good chance of being related to a change in hydrophobicity only. This bubble flux microcell design is therefore aimed at measuring hydrophobicity or being a hydrophobicity meter.

A process flow diagram of the microcell used is shown in **Figure 29** and a photograph in **Figure 30**. The unit consists of the following parts:

Recirculation pump

Similar to the Bikerman froth stability column described in Section 4.3.1, a recirculation pump is needed to keep the particles in suspension and to pump it through the static mixer where particle air contact is established. The most practical pump for this low flow purpose is a peristaltic pump. As discussed below, the air flow rate to the cell was selected as 0.1-0.2 l/min. Practical design criteria to disperse air in liquid call for at least a 1:3 air to liquid flow ratio (Terblanche, 1999). A minimum liquid flow of 0.6 l/min would thus be required. A Watson Marlow pump with a 3.2 mm outside diameter and 2.4 mm wall thickness gives a flow of 0.6 litre per minute at a 30% output setting and was selected for this duty.

Some modifications were made to accommodate the 3.2 mm Marprene tube in the Watson Marlow 604 pump available.

Static mixers and pulse damper.

One of the stated advantages of the micro flotation column as marketed by Svedala and designed by Control International, is the efficient bubble particle contact and uniform bubble size possible with a static mixer as sparging system (Metso Minerals, 2010). This system was used with success in the design of the Bikerman froth stability column and was the natural choice for a sparging system for the bubble flux micro flotation cell. It was not possible to achieve bubble dispersion in a single static mixer, and a double stage static mixer system was selected. This made it possible for the first static mixer to be modified to be a pulse damper and a first stage air bubble disperser. The air bubble and liquid mixture was pumped through a second stage static mixer where micro bubbles could be formed and bubble particle contact increased.

Two static mixers were used for the second stage, which made it possible to reduce the size of the second stage static mixer and hence increase shear force and decrease bubble size. It also allowed for dual entry into the cell similar to the Bikerman froth stability column. The static mixers were obtained from REAC Chemicals. This dual collision entry allowed for third stage micro-bubble formation and bubble particle contact.

Collection or column section

For a recirculation flow rate of 0.6 l/min and a collection or column section of 30 mm diameter, the downward superficial liquid velocity is calculated as 1.5 cm/sec. This flow is lower than the flow selected for the Bikerman froth stability column, since the bubble size is expected to be smaller than 0.7 mm.

The length of the column was selected to be 200 mm with a volume capacity of 140 ml. At 10% solids by mass a maximum of 14 grams solids can be tested per test.

Overflow lip

The overflow lip was ground to 60 degrees edge to present a thin overflow surface. The aim of this is to prevent drop back of particles back into the cell and increase the froth recovery factor.

Air rotameter

A Dwyer 0.1 to 1 l/min air rotameter was the smallest air rotameter available. The air pressure to this flow meter was controlled at 200kPa.

The air flow rate for all tests was kept constant at 0.1 l/min at a supply pressure of 200 kPa. Since the diameter of the cell is fixed, the J_g value for the test is constant, which is a stated design requirement for the unit.

Frother addition

At a constant liquid flow rate and constant air flow rate through the static mixer sparging system, the bubble size for all tests would be similar, providing the collector tested does not have frothing properties which can reduce the bubble size. To prevent a collector having an effect on bubble size, 0.03 g/litre of Betafroth 190 is added to the liquid used in the test. Betafroth 190 mainly consists of di-propylene mono methyl glycol ether, is water soluble and is recommended by the manufacturer to reduce bubble size in a flotation cell.

Water quality

The water used in the test was kept constant. The water was made up by adding the following to 25 litres of distilled water: 8.5 g NaCl, 15.0 g $MgSO_4$, 10.0 g $Ca(NO_3)_2$ and 0.750 gram Betafroth 190. The water temperature added to the cell was kept at 25 degrees by keeping the water supply in a water bath.

Water container and "froth crowder"

To force the froth recovery to approximate 100%, the flotation cell needs to have a constant liquid overflow, thus no froth level. A small amount of water (similar as used in the test and described above) is constantly added to the cell. To reduce entrainment, this amount of water must be as low as possible, but must be higher than the water lost as a result of water recovery. At an air flow rate of 0.1 l/min, 35 ml/min of water was sufficient to maintain a constant liquid overflow.

It is known that if the froth surface area is reduced with a froth crowder, the froth recovery is increased (Grobler, 1992). This principle is used in most modern flotation cells. A glass funnel with a micropipette glued to the outlet was used as froth crowder and doubled as container to add water to the cell. The water level in the glass funnel was topped up to a marked level every minute. This ensured repeatable water flow.

Particle size of ore tested

The particle size of the solids tested was kept between 75 and 106 micron. This was done to keep entrainment constant as will be discussed in Section 4.4.3.3. This narrow size distribution also serves to reduce the variation of attachment and detachment of particles to the air bubble, which is known to be influenced by particle size.

Mass of ore tested

Two grams of ground ore between 75 and 106 micron was used in each test. In theory the bubble flux microcell would be able to operate with 14 grams of ore. The selection of 2 grams was based on the method described by Bradshaw et al. (2006) and practical consideration of the mass of specimen available (Bradshaw & O’Conner, 2006).

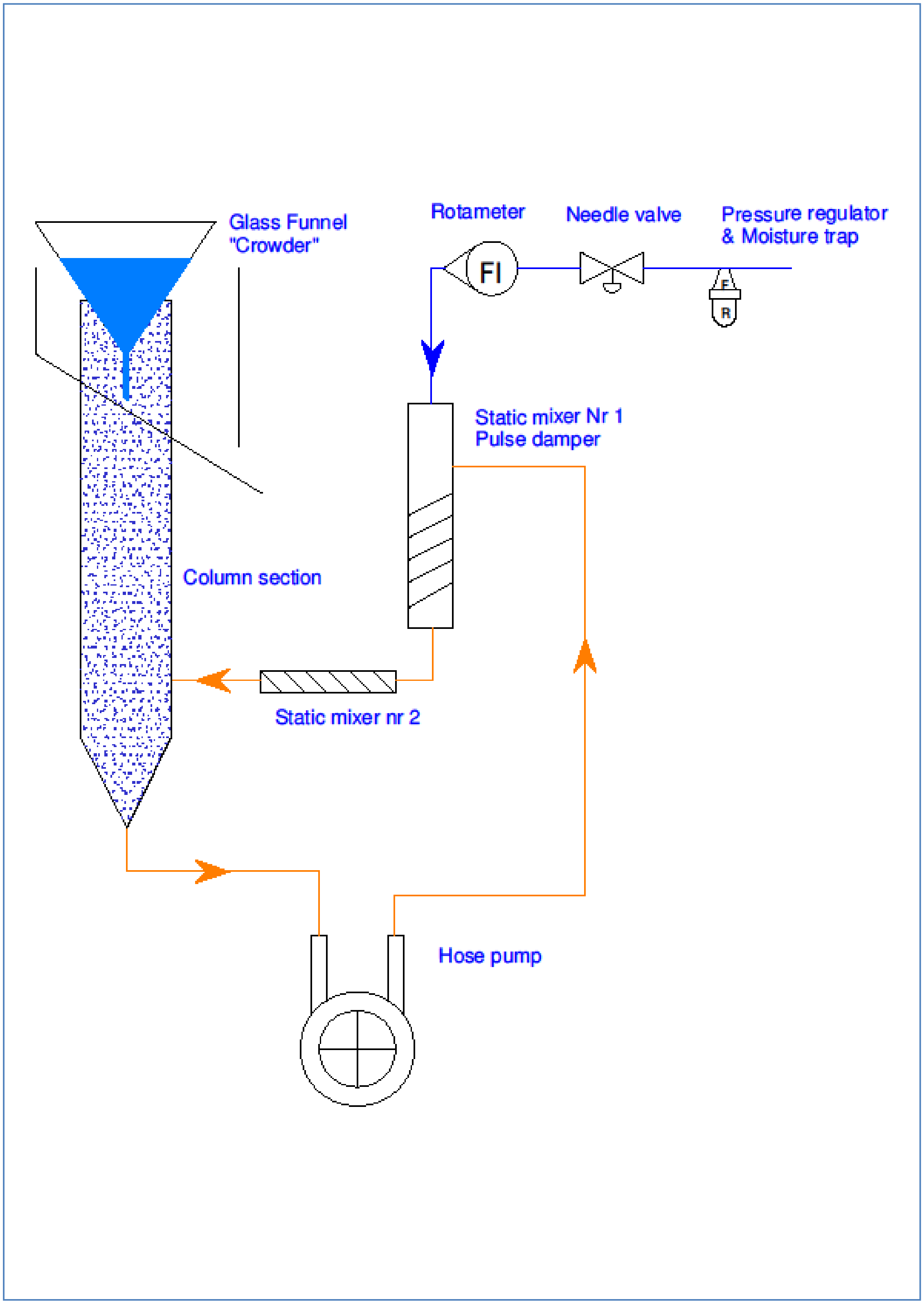


Figure 29: Flow diagram for the bubble flux micro flotation unit

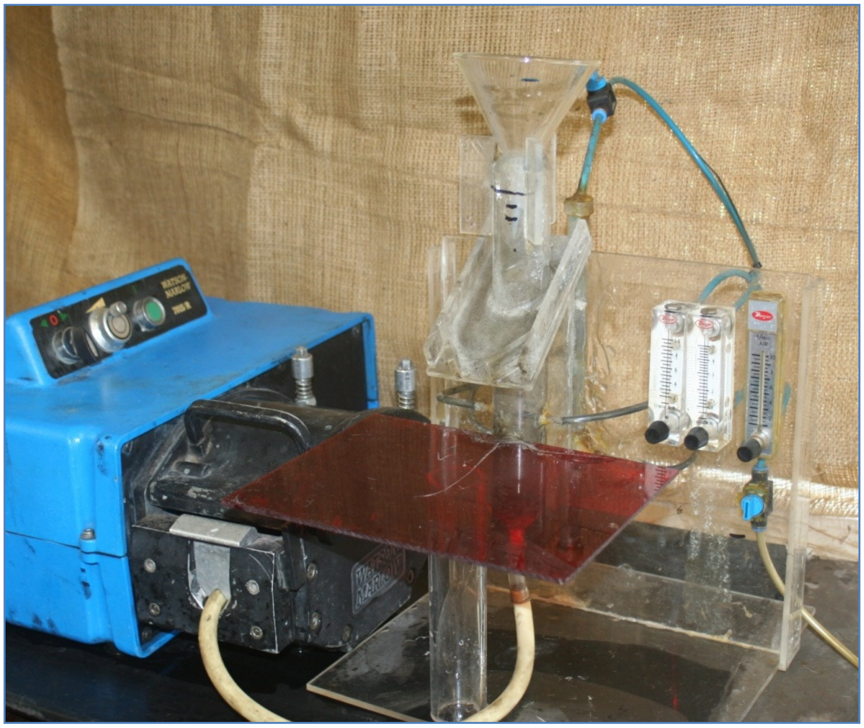


Figure 30: Photograph of the micro flotation cell used in this study.

4.4.2. Description of experimental procedure

The test sequence to test the effect a collector has on the floatability (hydrophobicity) of a mineral is as follows:

Synthetic plant water is prepared by adding the following to 25 ℓ of distilled water: 8.5 g NaCl, 15.0 g $MgSO_4$, 10.0 g $Ca(No_3)_2$ and 0.750 gram Betafroth 190. The temperature of this water is controlled at 25 degrees by placing the water container in a water bath set to 25 degrees.

A mineral specimen is prepared by crushing the mineral specimen to smaller than 1mm in a jaw crusher. This crushed specimen is subdivided into approximately 4 gram subsamples by cone and quarter.

For each test one 4 gram subsample is crushed in a porcelain pestle and mortar and dry sieved at 75 micron. The minus 75 micron fraction is discarded. The plus 75 micron fraction is dry sieved at 106 micron and the plus 106 micron fraction returned to the grinding step with the pestle and mortar. This process is continued until no plus 106 micron particles remains. A 2 gram sample of the plus 75 micron and minus 106 micron fraction is removed and the remainder discarded.

100 mℓ of synthetic plant water is added to the microcell and the recirculation pump switched on. The 2 gram crushed specimen (plus 75 micron minus 106 micron fraction) is added to the water. The reagents required for the test are added and conditioned for the required time. (1 minute). All reagents are added below the surface of the water.

After the conditioning step has been completed, water is added to the cell overflow, the container is placed under the overflow lip (labeled 0-0.5 min concentrate), and the froth crowder is placed into position and is filled with water to the full line. Finally the air valve is opened.

After 30 seconds, replace the container with a container labeled 0.5-1 minutes concentrate and water is added to fill the froth crowder up to the full line.

After 1 minute, replace the container with one labeled 1-2 minutes concentrate and add water to the froth crowder up to the full line.

After 2 minutes, replace the container with one labeled 2-4 minutes concentrate and add water to the froth crowder up to the full line. Refill the froth crowder with water after each minute.

After 4 minutes, replace the container with one labeled 4-8 minutes concentrate and add water to the froth crowder up to the full line. Refill the froth crowder with water after each minute.

Place the five containers in an oven to dry at a maximum temperature of 50 degrees. When dry, weigh the mass in each container and record. (It is more practical to weigh the container plus the dry sample, then remove the dry sample with a brush and reweigh the empty container).

Reagents are made up as discussed under 4.2.1

4.4.3. Verification of method

4.4.3.1. Reproducibility of results

Percentage standard deviation (standard deviation / average x 100) for repeat tests performed on a chalcopyrite, pyrrhotite and quartz specimens is shown in **Table 7**.

The percentage standard deviation for all tests where recoveries of 50% and higher were measured, was repeatable with a percentage standard deviation below the target 3%. For tests where lower than 50% recoveries were measured, repeatability was not as good. It is speculated that this test method is repeatable when true flotation is the recovery mechanism, but not repeatable when entrainment is the main mechanism for recovery.

It is recommended that tests be conducted in triplicate where possible. Only when high recoveries are measured, and entrainment is not expected to be the main recovery mechanism, can data from a single test be used. Error bars based on standard error (standard deviation / square root of number of data points) should be used to indicate repeatability.

Table 7: Percentage standard deviation for repeat tests done using the micro flotation cell. Purified sodium isopropyl xanthate (P-SIPX) at 3.3x 10^{-6} mol/l (50 g/t solids). Minerals used and treatment are as indicated.

Chalcopyrite: Aged in air 4 days					
time (min)	0.5	1	2	4	8
P SIPX	72.6	84.9	88.6	89.8	91.0
P SIPX	69.4	84.4	89.4	91.2	92.8
P SIPX	68.4	82.4	87.4	88.7	89.9
% stdev	3.2	1.6	1.2	1.4	1.6
Chalcopyrite: Fresh					
time (min)	0.5	1	2	4	8
P SIPX	14.4	34.7	55.3	74.4	79.4
P SIPX	14.5	32.2	56.2	75.1	84.1
P SIPX	18.0	39.5	61.0	77.2	84.6
% stdev	13.2	10.5	5.3	1.9	3.5
Pyrrhotite: Aged in Air for 4 days					
time (min)	0.5	1	2	4	8
P SIPX	9.5	16.6	25.6	40.1	59.3
P SIPX	7.3	14.6	22.7	37.3	57.9
P SIPX	7.4	15.0	24.3	38.7	56.6
% stdev	15.7	6.8	6.1	3.7	2.3
Pyrrhotite: Fresh					
time (min)	0.5	1	2	4	8
P SIPX -	3.3	7.0	11.3	17.9	25.1
P SIPX -	3.3	5.4	9.3	13.5	28.2
P SIPX -	3.9	6.6	10.6	15.4	25.4
% stdev	11.4	13.6	9.9	14.2	6.4
Quartz aged in air for 4 days					
time (min)	0.5	1	2	4	8
P SIPX -	1.4	3.2	5.8	9.4	17.9
P SIPX -	1.8	3.5	5.9	10.7	17.1
P SIPX -	3.0	4.3	6.1	9.7	15.9
% stdev	41.8	15.5	2.8	6.6	6.1

4.4.3.2. Comparison with published data

Recovery of fresh chalcopyrite and pyrrhotite specimens (crushed and screened to +75 – 106 micron and floated immediately after size reduction) is shown in **Figure 31**. The measured values compared well with published data (Newell, et al., 2006) where the collectorless recovery of chalcopyrite and pyrrhotite in a South African Merensky ore was studied (**Figure 32**).

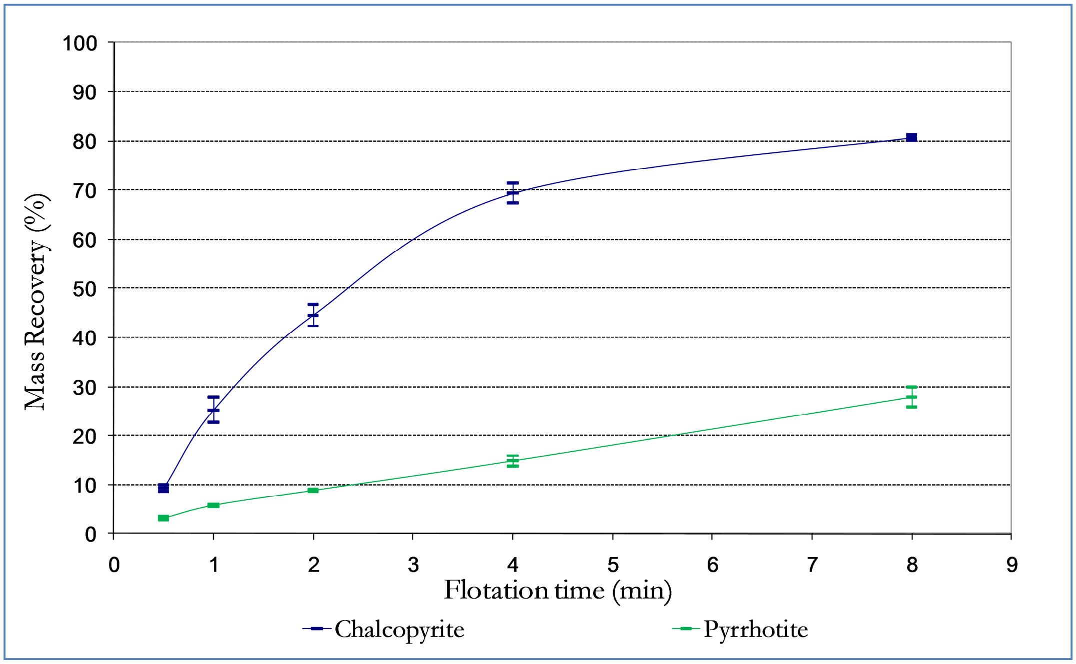


Figure 31: micro flotation test with chalcopyrite and pyrrhotite mineral specimens crushed and screened to +75 and -106 micron and floated directly after size reduction**.

***Frother concentration 0.03 g/l (Betafroth 190). Air flow rate 0.1 l/min. Method used is described in Section 4.4.2. Error bars are based on standard error*

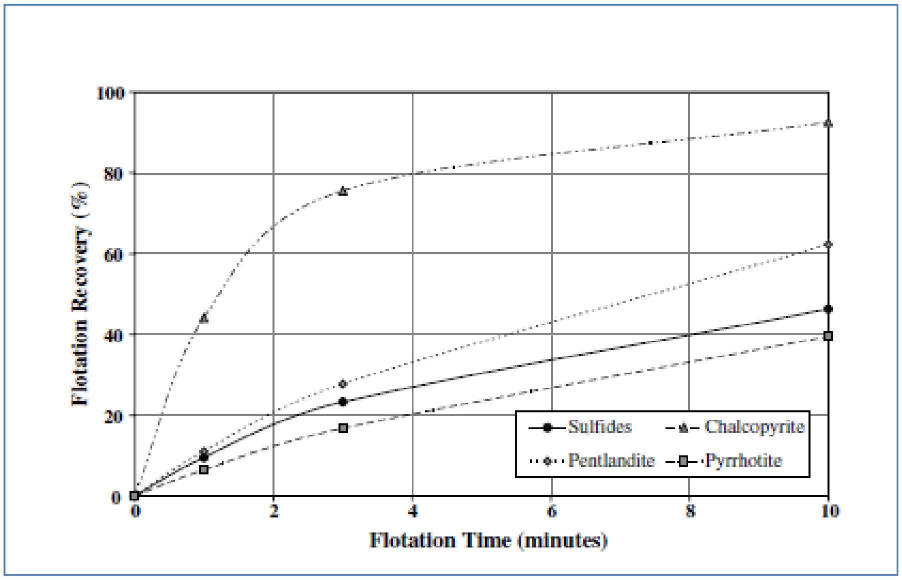


Figure 32: Flotation recovery of a typical Merensky ore without the addition of collector (Newell, et al., 2006)

4.4.3.3. Effect of entrainment

A set of tests was conducted using quartz at three size fractions, being 53 to75 micron, 75 to 106 micron and 106 to 150 micron. The tests were performed with a frother concentration (Betafroth 190) of 0.03 g/l (**Table 8**) and repeated at 0.06 g/t (**Table 9**).

Quartz is not naturally hydrophobic and any recovery measured is as a result of entrainment and not true flotation. As expected, the finer the particle size, the more quartz is recovered by entrainment (**Figure 33**). Almost no entrainment was measured for the 106 to 150 micron size fraction, which conforms to the understanding that entrainment is a result of particles suspended in water being recovered. Entrainment of the 53 to75 micron fraction was measured to be significant, and micro flotation tests with this size fraction are not recommended. The size fraction 75 to 106 micron was selected as the standard size fraction for micro flotation tests. This fraction is within the 100 micron top size which is accepted as the limit for flotation, and entrainment for this fraction is marginal.

Froth stability increases as frother concentration is increased, which leads to higher water recovery and consequently entrainment. Entrainment of all three size fractions increased significantly with an increase in frother concentration (**Figure 34** and **Figure 35**). When a collector with frothing characteristics is tested, it is likely that entrainment will increase and results should be analysed with caution. It is recommended that the frothing characteristics of a collector combination be tested in the Bikerman froth stability column (see Section 4.3) and that this data be used to assist micro flotation data.

The increase in entrainment measured with an increase in frother concentration demonstrates that the bubble flux micro flotation test apparatus is not successful in eliminating the effect of froth recovery on the measured rate of recovery. This should be taken into account when data is interpreted.

Table 8: Entrainment quantified with the micro flotation cell using quartz with particles size as indicated and Betafroth 190 concentration of 0.03 g/l.

Test	Air flow l/min	Mass Quartz (gram)	Grind		Mass (grams)					Cumalative mass recovery (%)				
			min µm	max µm	Flotation time (min)					Flotation time (min)				
					0.5	1	2	4	8	0.5	1	2	4	8
1	0.2	2	75	53	0.085	0.068	0.113	0.153	0.245	4.3	7.7	13.3	20.9	33.2
2	0.2	2	75	53	0.086	0.075	0.120	0.149	0.254	4.3	8.1	14.1	21.5	34.2
3	0.2	2	106	75	0.047	0.024	0.051	0.098	0.043	2.4	3.6	6.1	11.0	13.2
4	0.2	2	106	75	0.031	0.021	0.073	0.073	0.111	1.5	2.6	6.2	9.9	15.4
5	0.2	2	150	106	0.019	0.015	0.025	0.017	0.022	0.9	1.7	2.9	3.8	4.9
6	0.2	2	150	106	0.009	0.011	0.017	0.010	0.024	0.5	1.0	1.9	2.4	3.6

Table 9: Entrainment quantified with the micro flotation cell using quartz with particles size as indicated and Betafroth 190 concentration of 0.06 g/l.

Test	Air flow l/min	Mass Quartz (gram)	Grind		Mass (grams)					Cumalative mass recovery (%)				
			min µm	max µm	Flotation time (min)					Flotation time (min)				
					0.5	1	2	4	8	0.5	1	2	4	8
1	0.2	2	75	53	0.087	0.146	0.214	0.265	0.322	4.3	11.6	22.3	35.6	51.7
2	0.2	2	75	53	0.146	0.122	0.215	0.258	0.372	7.3	13.4	24.1	37.0	55.7
3	0.2	2	106	75	0.086	0.045	0.108	0.143	0.192	4.3	6.6	12.0	19.1	28.7
4	0.2	2	106	75	0.042	0.071	0.032	0.139	0.189	2.1	5.7	7.3	14.2	23.7
5	0.2	2	150	106	0.057	0.036	0.031	0.072	0.084	2.8	4.6	6.2	9.8	14.0
6	0.2	2	150	106	0.068	0.056	0.045	0.074	0.064	3.4	6.2	8.4	12.1	15.3

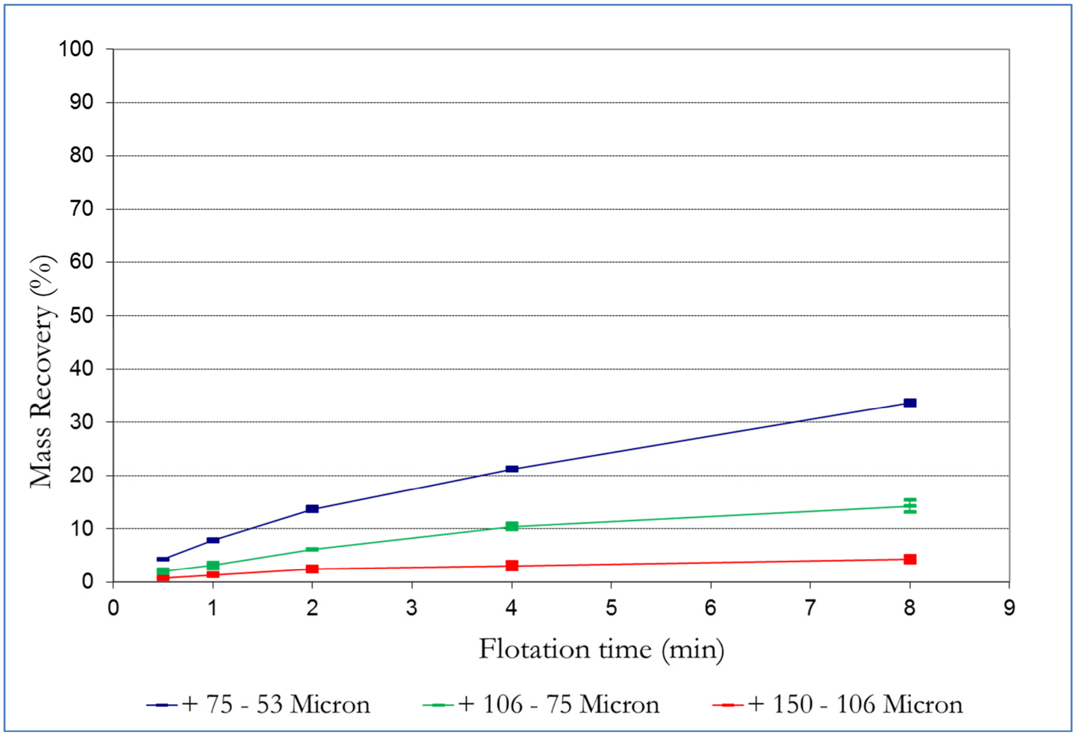


Figure 33: Micro flotation test with quartz and particle size limits as indicated**

*** Frother concentration 0.03 g/l (Betafroth 190). Air flow rate 0.2 l/min. Method used is described in Section 4.4.2. Error bars are based on standard error.*

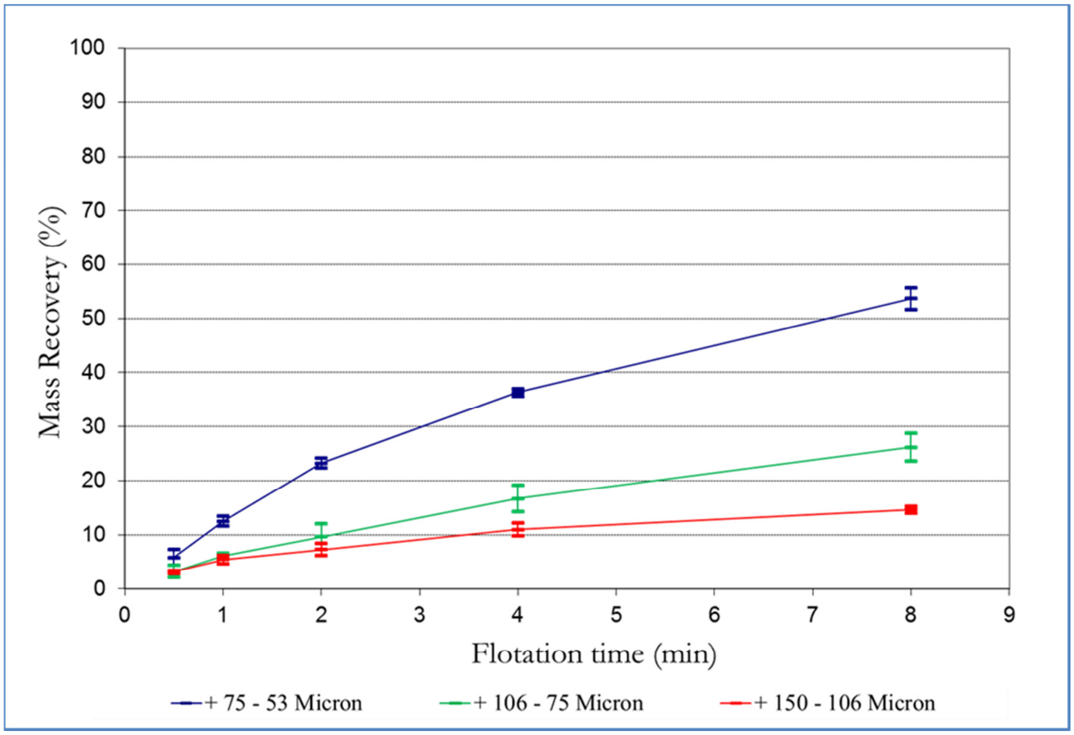


Figure 34: Micro flotation test with quartz and particle size limits as indicated**

***Frother concentration 0.06 g/l (Betafroth 190). Air flow rate 0.2 l/min. Method used is described in Section 4.4.2. Error bars are based on standard error.*

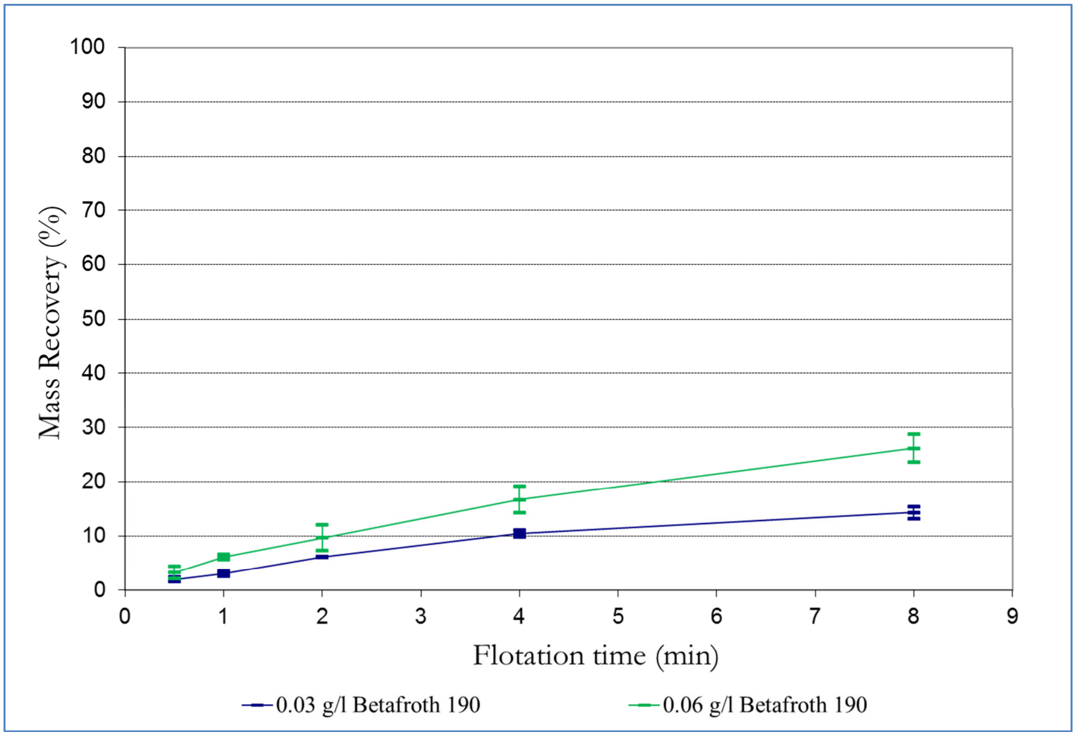


Figure 35: Micro flotation test with Quartz at frother concentration as indicated**

***Quartz screened to + 75 and – 106 micron. Air flow rate 0.2 l/min. Method used is described in Section 4.4.2. Error bars are based on standard error.*

4.5. Pilot plant test of fatty acid based co-collector (Betacol 364). Description of method used.

A 75 litre (4 cell) mobile pilot plant was used to test the use of Betacol 364 (a fatty acid co-collector) against the baseline test condition, being no co-collector. This test was done on pulp stream from a South African nickel flotation plant. The ore contains mainly pentlandite, chalcopyrite and pyrrhotite as described in Section 4.9.5. The pilot plant was transported to the flotation plant. A pulp stream was pumped from the cyclone overflow sump at a flowrate of 5 ℓ/m. Collector (SIBX), frother (Betafroth 436), gangue depressant (Betamin 544) were added to the pulp stream. For the test period a co-collector (Betacol 364) was added. This co-collector was not added for the baseline condition. The reagents were added to the pipe transporting the pulp from the sampling point to the feed of the pilot plant. Co-collector was added first, then xanthate, followed by frother and finally depressant.

The test procedure was as follows:

1. Prepare the test reagents.
2. Fill the pilot plant with water.
3. Start the pulp flow and also the reagent addition.
4. Measure the pulp flow by diverting the pulp into a 20 litre bucked and measure the time for the bucket to fill.
5. Measure the reagent flow and adjust to the required dosage rate.
6. Allow 60 minutes to stabalise.
7. Sample the four concentrate streams by diverting the full flow to a sample bucket. Measure and record the time for the bucket to fill. Seal and label the bucket.
8. Sample the tails by diverting the full flow to a sample bucket. Measure and record the time for the bucket to fill. Seal and label the bucket.
9. Samples were filtered on site and filter cake transported to the laboratory for drying. The dry weight was measured and recorded.
10. A sub sample from the dry filter cake was assayed for Ni and Cu using the metode described in Section 7.1

4.6. Short plant scale test (crash test) of fatty acid co-collector (Betacol 364). Description of method used.

The first step in the testing of a flotation reagents combination is described in Section 3.8 as the short term test or "crash test". The addition of a fatty acid co-collector (Betacol 364) was tested against the baseline condition of no co-collector on a South African UG2 platinum concentrator. The sulphide mineral in this ore is mainly chalcopyrite, pentlandite and pyrrhotite as described in Section 4.9.6.

The flotation plant uses xanthate (SIBX) as collector at 100 g/t added at various dosing points in the flotation circuit. A CMC based gangue depressant is also added to reduce gangue recovery. The plant operators have flexibility to change the depressant dosage rate to achieve the target concentrate grade. Betafroth 436 is used as frother.

Betacol 364 (fatty acid) was added, at 50 g/t to the primary rougher feed (primary cyclone overflow) and all other conditions were kept constant. The operators were requested to manage the plant as they would normally do.

Variation in plant performance was identified as the largest challenge for this plant test. The variation was assumed to be related to the variance in ore from the open cast mine and variation from the changing operating strategies that the various operators employ to manage the variations from the mine.

For this set of conditions, it was clear that the plant test needed to produce as much data as possible in multiple short test periods. The following test procedure was used as a compromise between generating sufficient data and the production risk inherent in a plant test:

- Betacol 364 at 50 g/t was tested over two days in two test blocks.
- Operators will be allowed to manage the plant as they normally do.
- Each of the 2 test days will be divided into three test blocks.

- During the first block (4 hours), the plant will run with the baseline reagent suite and will be sampled every 30 minutes. The purpose of this block is to generate a large number of data points as the baseline condition.
- For the next block (3 hours), Betacol 364 will be added at 50 g/t to the primary flotation feed. No samples will be taken. The purpose of this period is to achieve steady state after the reagent change.
- The last block (4 hours) will continue with Betacol 364 at 50 g/t and the plant will be sampled every 30 minutes. The purpose of this block is to generate a large number of data points as the test condition.

Plant tests are very seldom executed as planned. This test was no exception. A plant maintenance problem occurred during day 2 and the baseline block was reduced to 3 hours and the test block to 2 hours.

The samples were filtered on site, dried and submitted for PGM assay by fire assay. The flotation concentrate were also submitted for sulphur assay by Leco.

4.7. Analytical techniques

The analytical techniques used in this this study is described in Appendix A. The methods described are:

- Sodium peroxide digestion with AA finish. Section 7.1.
- Leco sulphur analyser. Section 7.2.
- Scanning electron microscope with EDS. Section 7.3.
- XRD identification and quantification. Section 7.4.

4.8. Description of flotation reagents used

4.8.1. Xanthate

All xanthate samples used were sourced from Nasaco International. The method used to purify the commercial xanthate is described in Appendix B (Section 8.1). The method used to measure the activity of the xanthate used is described in Appendix B (Section 8.2 and Section 8.3)

4.8.2. Copper Sulphate

Copper sulphate used was sourced from Kimleigh. The Copper sulphate is of an industrial grade and is used extensively in South African flotation plants.

4.8.3. Betacol 364

Betacol 364 is a fatty acid collector supplied by Betachem (Pty) Ltd.

4.8.4. Betacol 316

Betacol 316 is a nonionic dithiophosphate collector supplied by Betachem (Pty) Ltd.

4.8.5. Betacol 44A

Betacol 44A is an ionic dithiophosphate collector supplied by Betachem (Pty) Ltd.

4.8.6. Betacol 25

Betacol 25 is a paraffin based collector supplied by Betachem (Pty) Ltd.

4.9. Characterisation of ore samples used

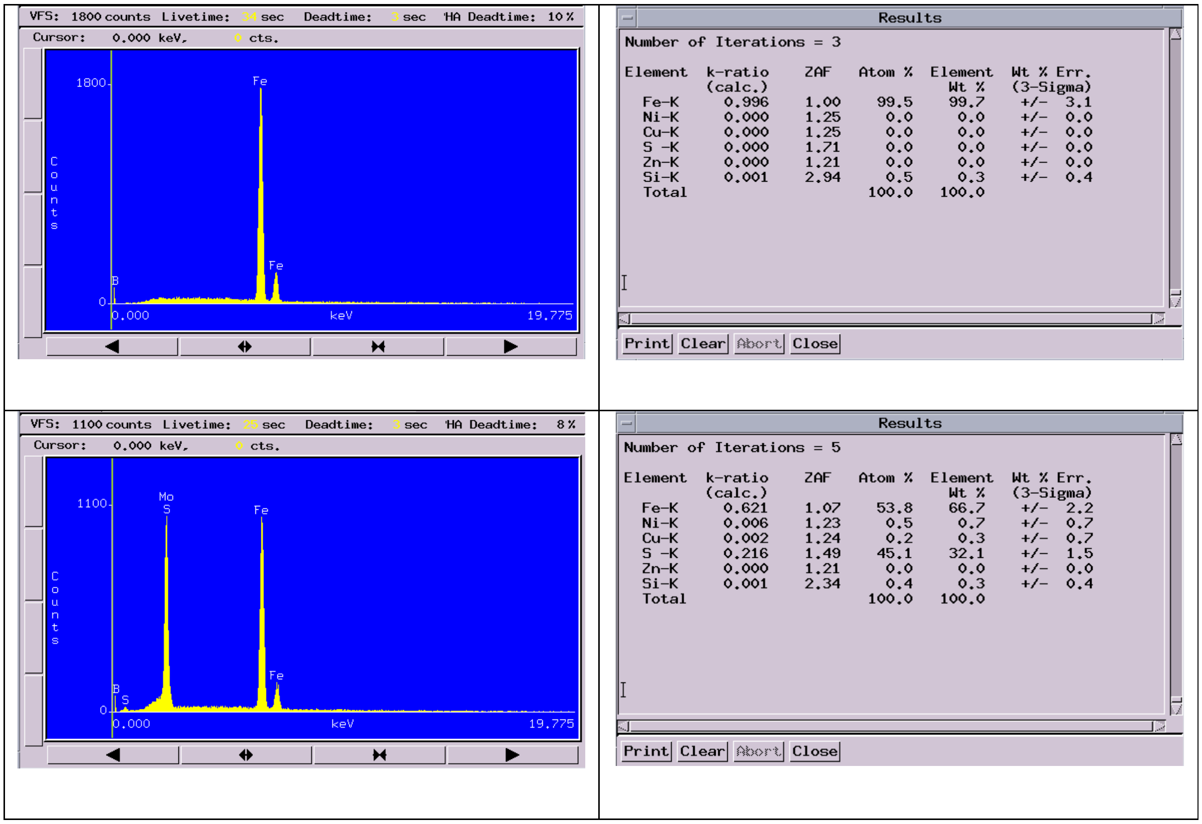
4.9.1. Pyrrhotite

A pyrrhotite specimen was obtained from the Munali mountain deposit in Zambia. The pyrrhotite specimen was crushed and dry milled to 100% passing 212 micron, and the magnetic fraction separated with a hand held rare earth magnet (Eriez Magnetics). A 1 gram subsample was pulverised and sent to XRD Analytical and Consulting CC for XRD identification and quantification as described in Section7.4. XRD results (**Table 10**) indicate that the sample contains 64% pyrrhotite. The XRD results did not identify if the pyrrhotite is monoclinic or a combination of hexagonal and monoclinic pyrrhotite.

Scanning electron microscope images of the pyrrhotite sample milled to 100% passing 212 micron (by iterative milling and sieving) is shown in **Figure 37** and indicates that the majority of the pyrrhotite is fully liberated at 212 micron. The Electron Diffraction Spectroscopy (EDS) display and quantification results for a pyrrhotite and magnetite particle are shown in **Figure 36**. It should be noted that the EDS used does not detect light elements like oxygen.

Table 10: Mineral identification and quantification by XRD for a pyrrhotite specimen from the Munali Mountains in Zambia

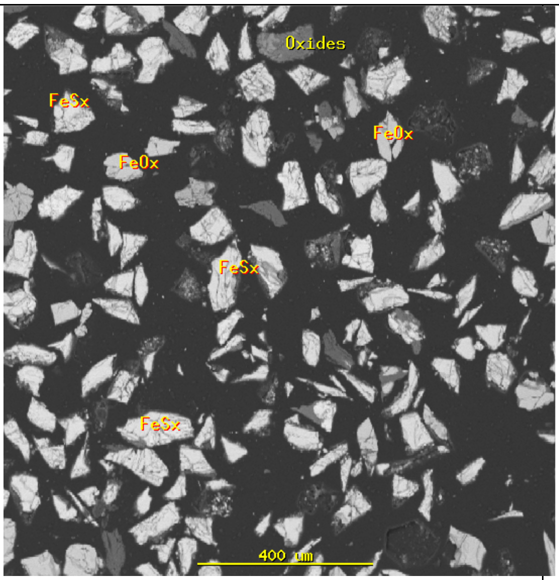
Mineral	Relative phase amounts (% weight)	Error -3 sigma (% weight)
Chlorite	10.8	2.58
Magnetite	8.5	0.96
Pentlandite	5.6	0.6
Pyrrhotite	63.9	3.3
Talc	11.2	5.4

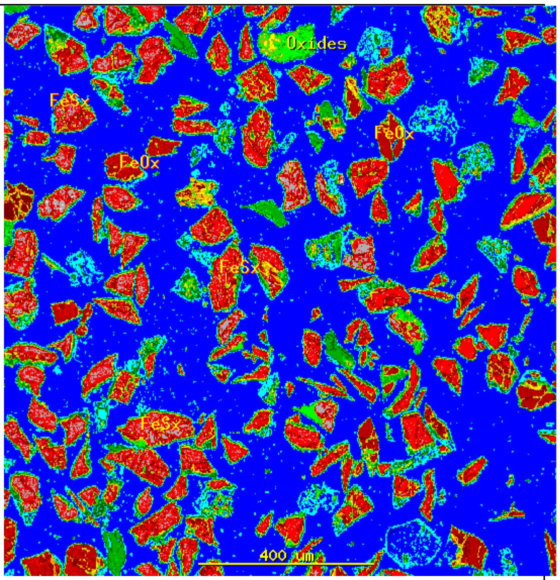


```
Results
Number of Iterations = 3

Element   k-ratio     ZAF    Atom %  Element  Wt % Err.
          (calc.)                      Wt %   (3-Sigma)
  Fe-K     0.996     1.00     99.5    99.7    +/-  3.1
  Ni-K     0.000     1.25      0.0     0.0    +/-  0.0
  Cu-K     0.000     1.25      0.0     0.0    +/-  0.0
  S -K     0.000     1.71      0.0     0.0    +/-  0.0
  Zn-K     0.000     1.21      0.0     0.0    +/-  0.0
  Si-K     0.001     2.94      0.5     0.3    +/-  0.4
  Total                      100.0   100.0
```

```
Results
Number of Iterations = 5

Element   k-ratio     ZAF    Atom %  Element  Wt % Err.
          (calc.)                      Wt %   (3-Sigma)
  Fe-K     0.621     1.07     53.8    66.7    +/-  2.2
  Ni-K     0.006     1.23      0.5     0.7    +/-  0.7
  Cu-K     0.002     1.24      0.2     0.3    +/-  0.7
  S -K     0.216     1.49     45.1    32.1    +/-  1.5
  Zn-K     0.000     1.21      0.0     0.0    +/-  0.0
  Si-K     0.001     2.34      0.4     0.3    +/-  0.4
  Total                      100.0   100.0
```

Figure 36: Analysis of pyrrhotite and magnetite particle by EDS. Pyrrhotite specimen from Munali Mountains. (The EXD does not detect light elements like Oxygen).





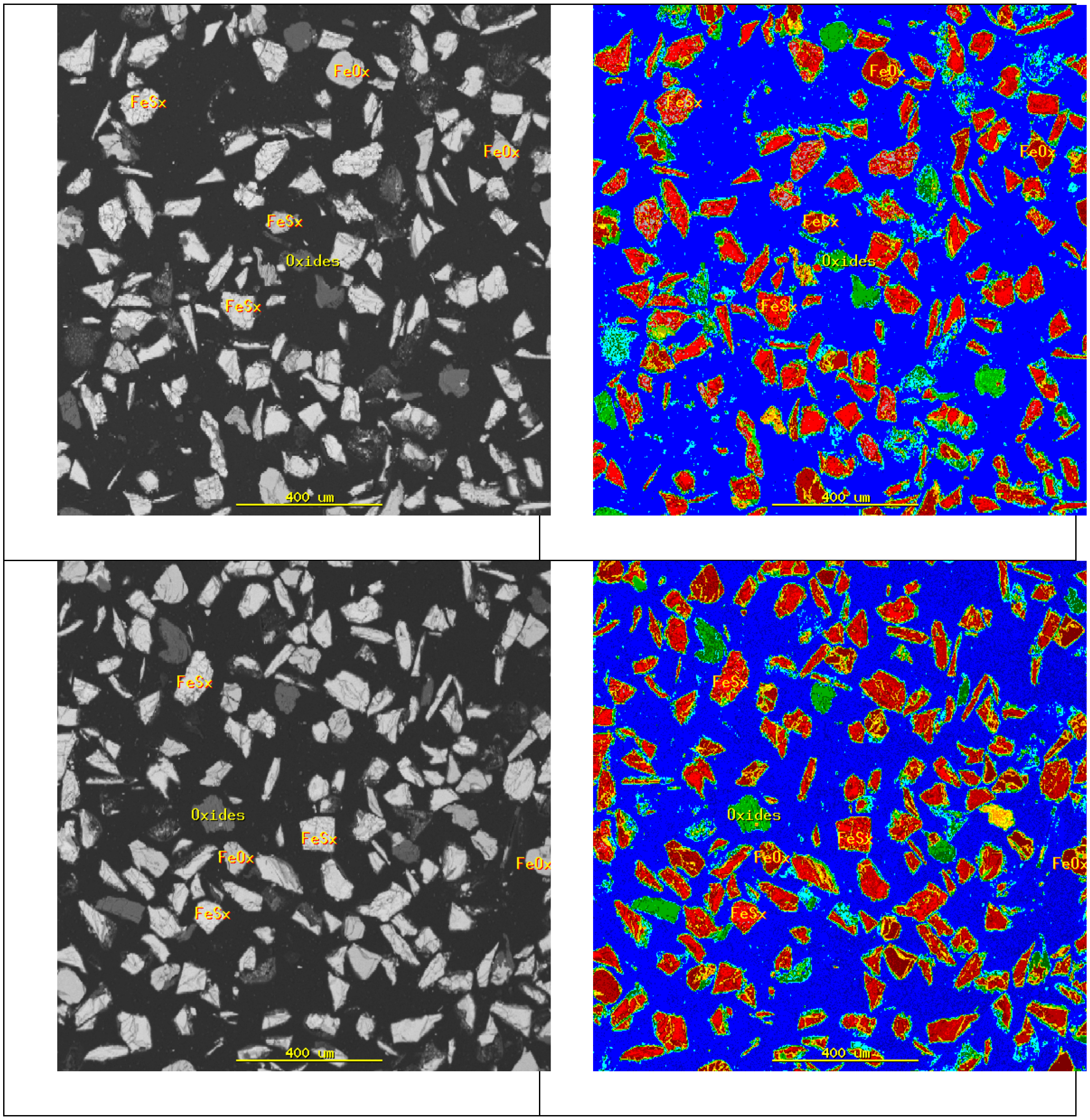


Figure 37: SEM images for pyrrhotite specimen from the Munali Mountains in Zambia after dry milling to 212 micron and magnetic fraction separate with rare earth magnet**.

***The SEM images on the left are original GREY SCALE images and the images on the right are chromatic colour interpretation of the images. Pyrrhotite particles are indicated as FeSx and are the light red particles in the chromatic colour images. Magnetite is indicated by FeOx and is the dark red particles. The oxides are indicated and are the green particles.*

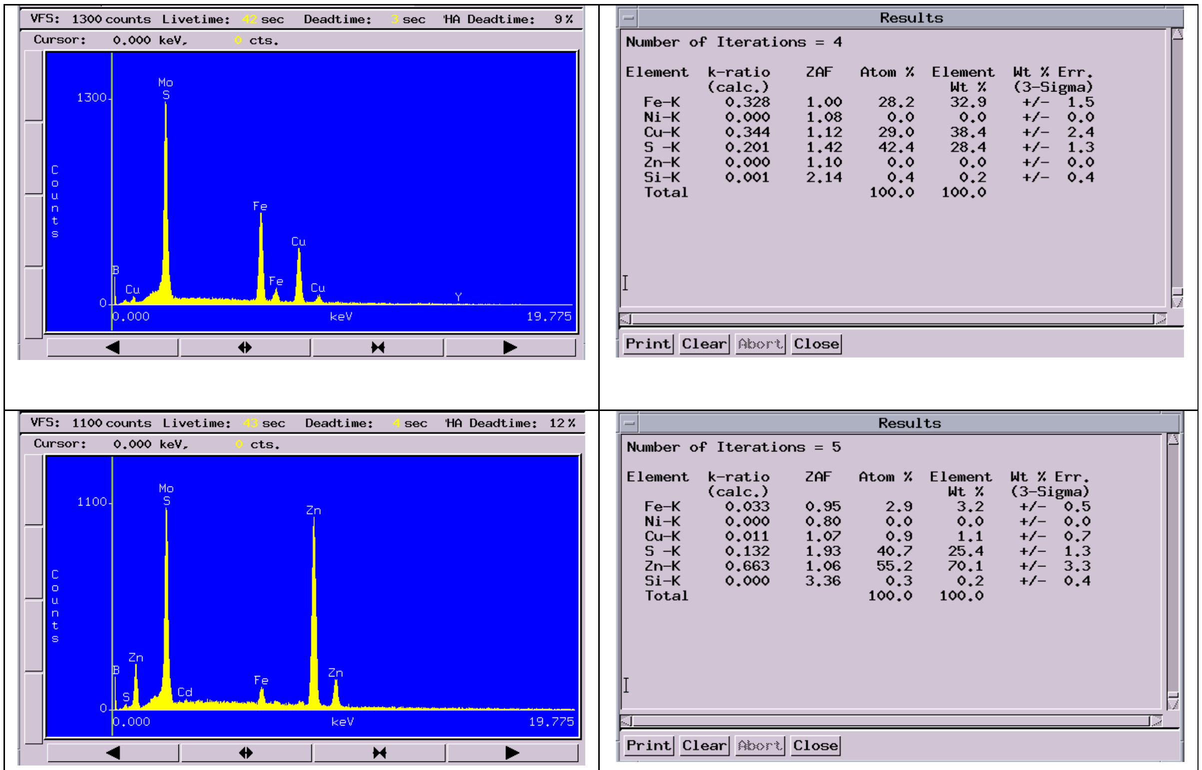
4.9.2. Chalcopyrite

A chalcopyrite specimen was obtained from Ward's Natural Science. The specimen was crushed to 100% passing 1 mm, a 1 gram pulverised and sent to XRD Analytical and consulting for XRD identification and quantification as described in Section 7.4. XRD results indicate that the sample contains both chalcopyrite and sphalerite with oxides (**Table 11**). The calcite and quartz were coarse and an attempt was made to upgrade the chalcopyrite content by hand picking and removing the oxides from the sample.

Scanning electron microscope images of the chalcopyrite sample milled to 100% passing 212 micron (by iterative milling and sieving) are shown in **Figure 39** and indicate that the majority of the chalcopyrite and sphalerite is fully liberated at 212 micron. The EDS spectral display and quantification results for a chalcopyrite and sphalerite particle are shown in **Figure 38**.

Table 11: Mineral identification and quantification by XRD for a chalcopyrite sample obtained from Ward's Natural Science

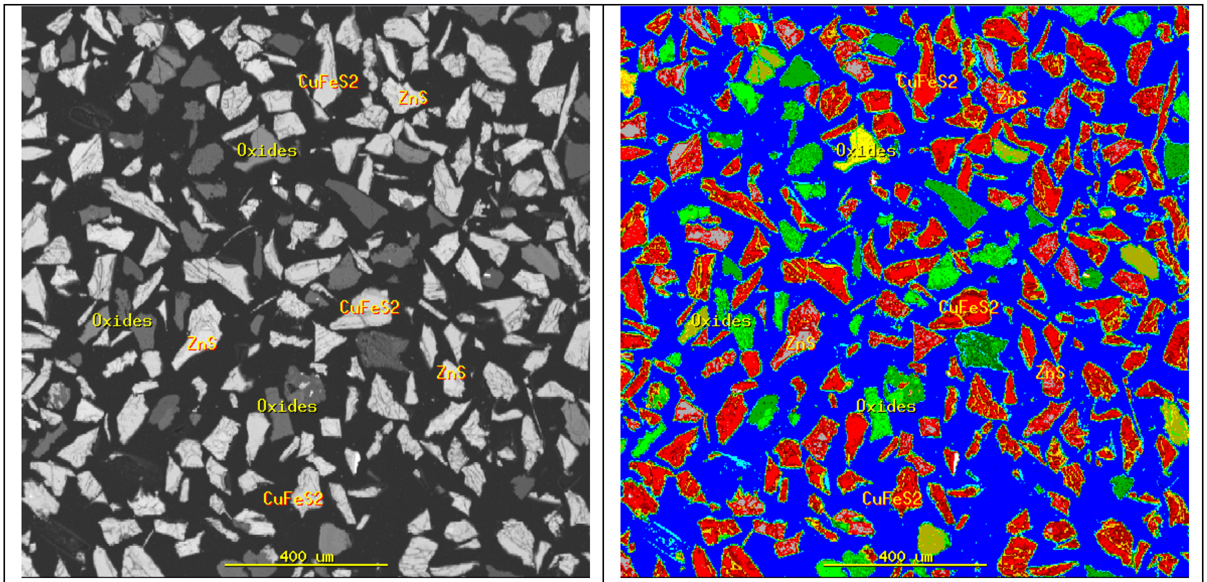
Mineral	Relative phase amounts (% weight)	Error -3 sigma (% weight)
Calcite	11.3	1.65
Chalcopyrite	55.1	1.35
Chlorite	2.1	1.05
Quartz	9.8	0.54
Sphalerite	21.9	0.66



```
Number of Iterations = 4

Element  k-ratio    ZAF    Atom %  Element  Wt % Err.
         (calc.)                     Wt %   (3-Sigma)
  Fe-K    0.328     1.00    28.2     32.9    +/-  1.5
  Ni-K    0.000     1.08     0.0      0.0    +/-  0.0
  Cu-K    0.344     1.12    29.0     38.4    +/-  2.4
  S -K    0.201     1.42    42.4     28.4    +/-  1.3
  Zn-K    0.000     1.10     0.0      0.0    +/-  0.0
  Si-K    0.001     2.14     0.4      0.2    +/-  0.4
  Total                    100.0    100.0
```

```
Number of Iterations = 5

Element  k-ratio    ZAF    Atom %  Element  Wt % Err.
         (calc.)                     Wt %   (3-Sigma)
  Fe-K    0.033     0.95     2.9      3.2    +/-  0.5
  Ni-K    0.000     0.80     0.0      0.0    +/-  0.0
  Cu-K    0.011     1.07     0.9      1.1    +/-  0.7
  S -K    0.132     1.93    40.7     25.4    +/-  1.3
  Zn-K    0.663     1.06    55.2     70.1    +/-  3.3
  Si-K    0.000     3.36     0.3      0.2    +/-  0.4
  Total                    100.0    100.0
```

Figure 38: Analysis of chalcopyrite and sphalerite particles by EDS.



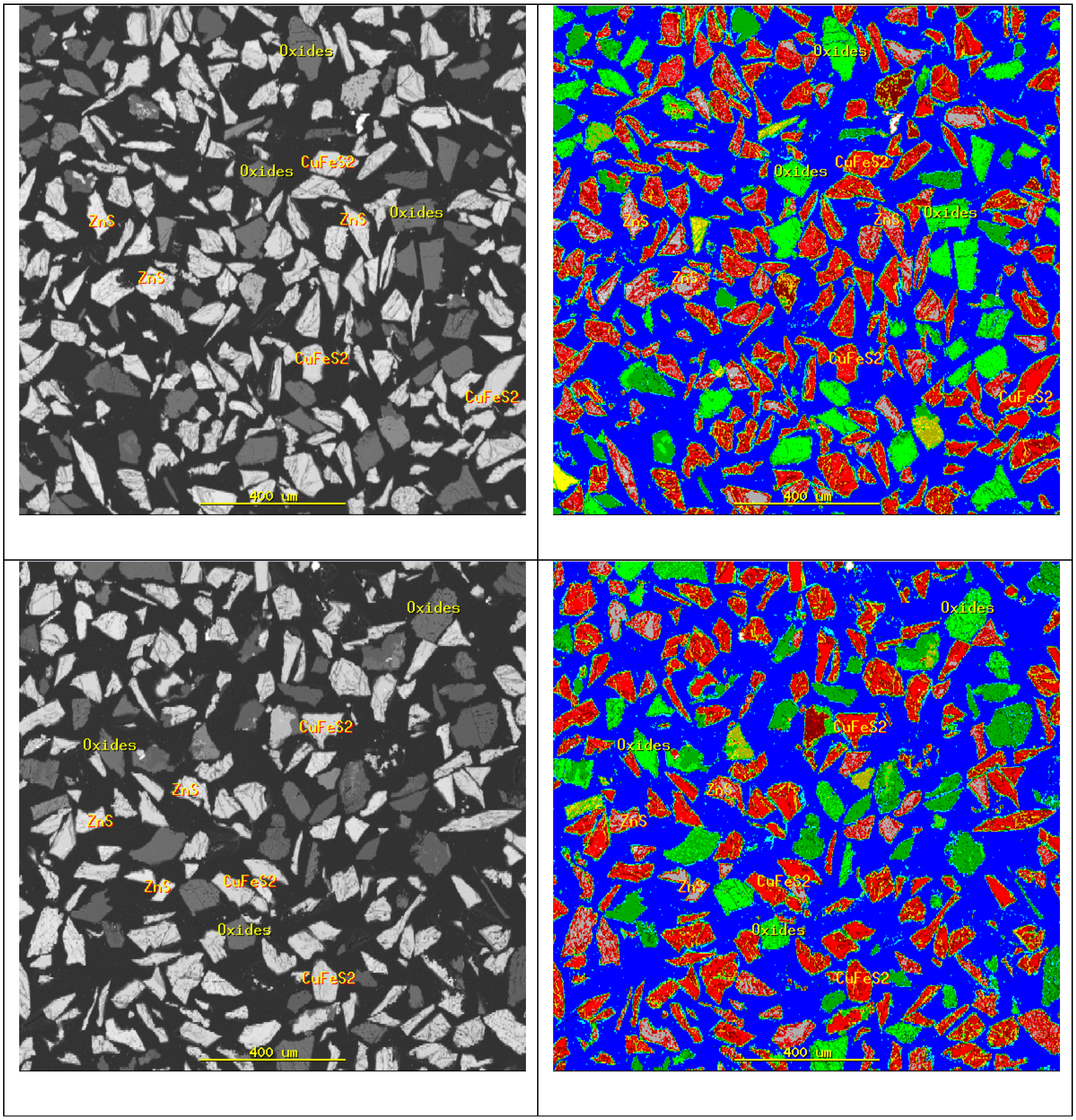


Figure 39: SEM images for chalcopyrite specimen from Ward's natural Science after dry milling to 212 micron **.

***The SEM images on the left are original GREY SCALE images and the images on the right are chromatic colour interpretation of the images. Chalcopyrite particles are indicated as CuFeS2 and are the light red particles in the chromatic colour images. Sphalerite is indicated by ZnS and is the red/grey particles. The oxides are indicated and are the green particles.*

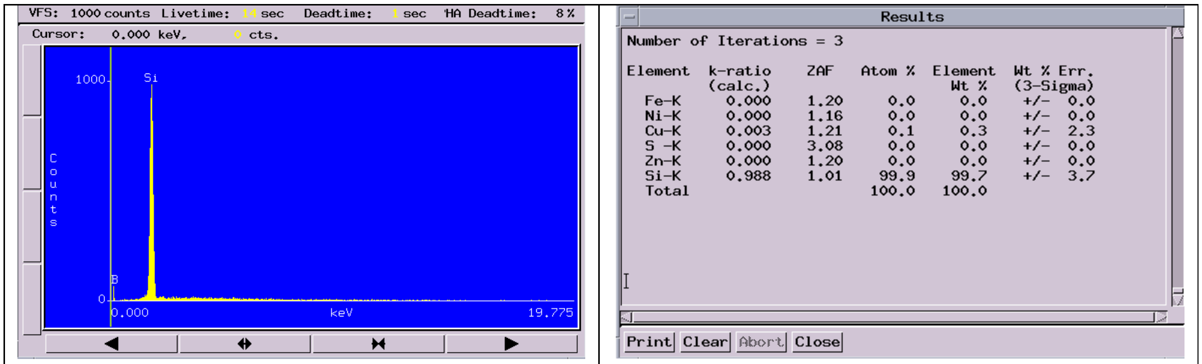
4.9.3. Quartz

A bag of quartz was purchased. The quartz was already crushed and sized into +500 minus - 1 mm. The sample contained some iron particles, which were removed by magnetic separation. A 1 gram subsample was pulverised and sent to XRD Analytical and Consulting CC for XRD identification and quantification as described in Section 7.4. Results in **Table 12** indicate a pure quartz sample with traces of hematite.

Scanning electron microscope images of the quartz sample milled to 100% passing 212 micron is shown in **Figure 41** and indicate that the quartz is fully liberated at 212. The EDS spectral display and quantification results for a quartz particle are shown **Figure 40**. It should be noted that the EXD used does not detect light elements like oxygen. The analysis for quartz therefore only shows silicon.

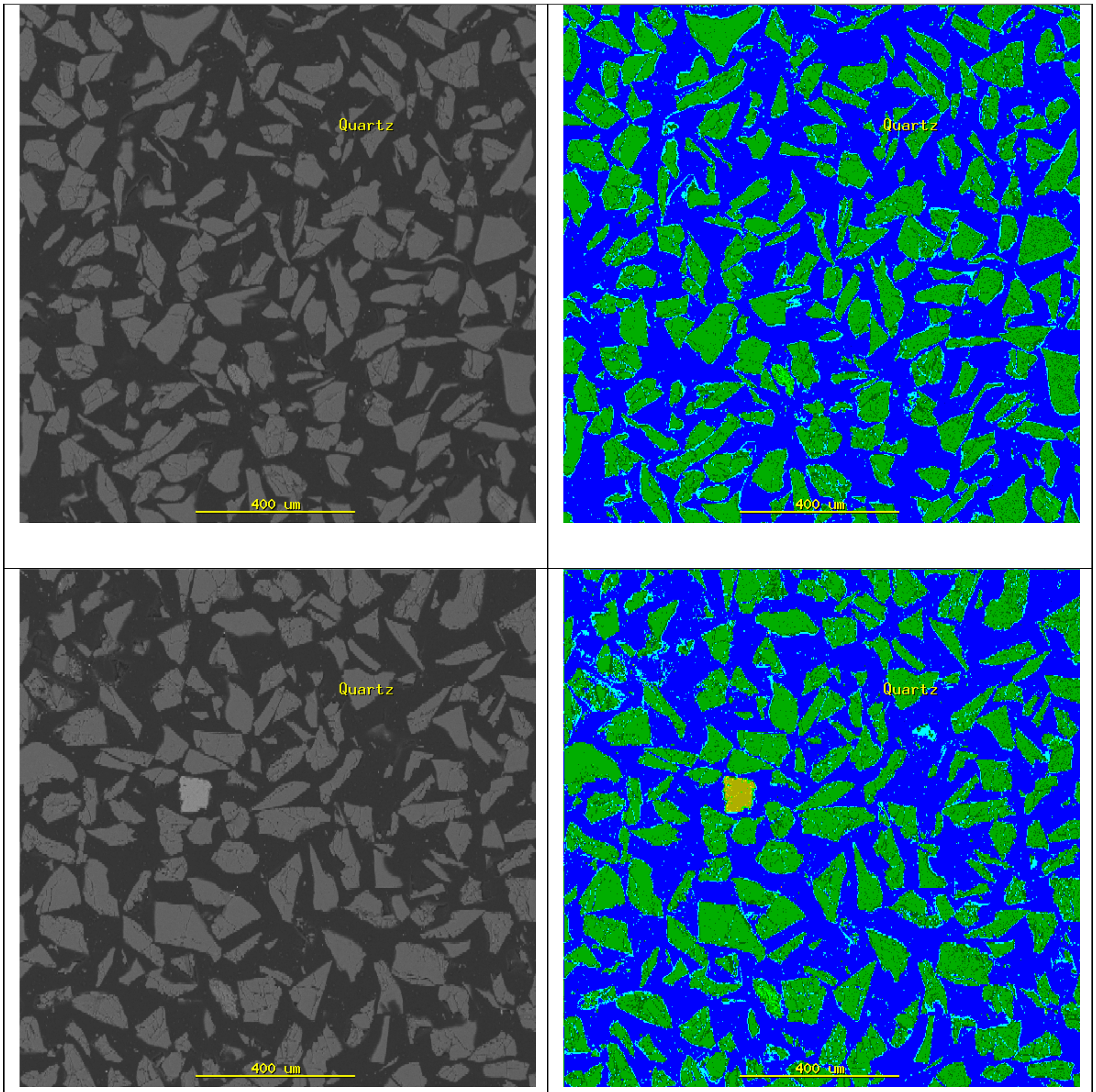
Table 12: Mineral identification and quantification by XRD for a quartz sample after iron removal by magnetic separation

Mineral	Relative phase amounts (% weight)	Error -3 sigma (% weight)
Hematite	0.18	0.15
Quartz	99.82	0.15



```
Number of Iterations = 3

Element  k-ratio     ZAF    Atom %  Element   Wt % Err.
         (calc.)                      Wt %   (3-Sigma)
  Fe-K    0.000     1.20     0.0      0.0    +/-  0.0
  Ni-K    0.000     1.16     0.0      0.0    +/-  0.0
  Cu-K    0.003     1.21     0.1      0.3    +/-  2.3
  S -K    0.000     3.08     0.0      0.0    +/-  0.0
  Zn-K    0.000     1.20     0.0      0.0    +/-  0.0
  Si-K    0.988     1.01    99.9     99.7    +/-  3.7
  Total                    100.0    100.0
```

Figure 40: Analysis of quartz particle by EDS. (The EXD does not detect light elements like Oxygen).



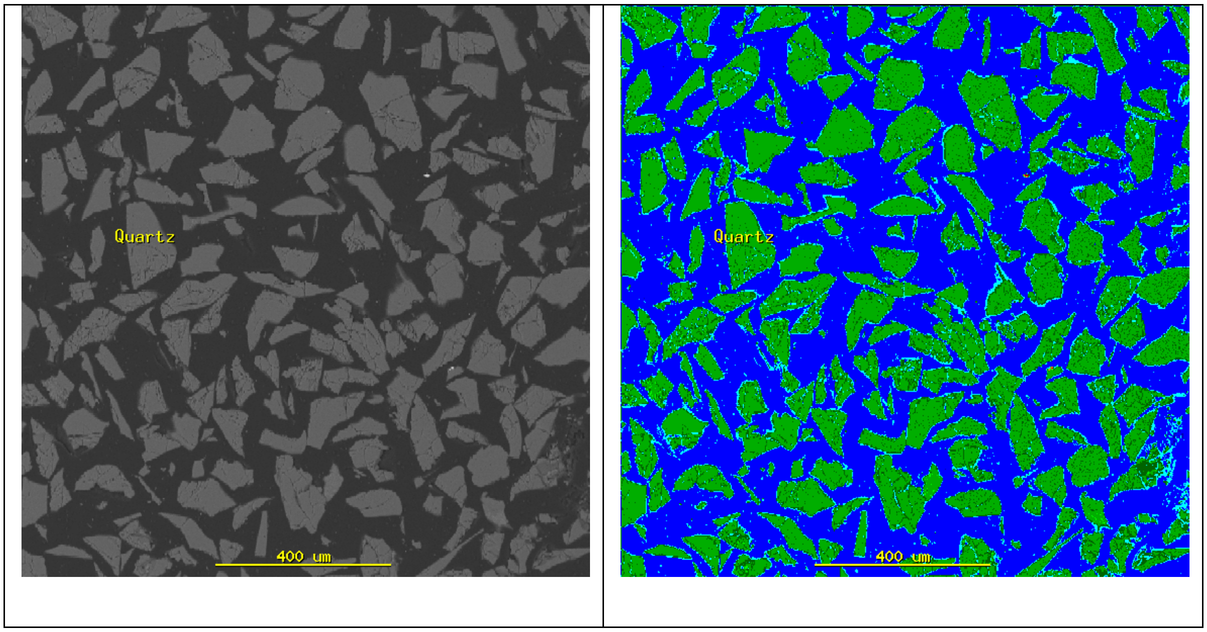


Figure 41: SEM images for quartz specimen after dry milling to 212 micron and magnetic fraction separate with rare earth magnet**.

*** The SEM images on the left are original GREY SCALE images and the images on the right are chromatic colour interpretation of the images. Quartz particles are indicated as quartz and are the green particles in the chromatic colour images.*

4.9.4. Laboratory prepared mixed ore

Two synthetic ores were prepared. The first was a blend with 10% of the chalcopyrite specimen described in Section 4.9.2 and 90% of the quartz described in Section 4.9.3. The second synthetic ore was a blend with 10% of the pyrrhotite specimen described in Section 4.9.1 and 90% quartz described in Section 4.9.3.

4.9.5. Nickel ore

A natural ore containing pentlandite, chalcopyrite and pyrrhotite (traces of pyrite) was received from a nickel mine in South Africa. The ore was crushed to 100% minus 20 mm and dry milled to 100% passing 300 micron. A 1 gram subsample was pulverised and send to XRD Analytical and Consulting CC for XRD identification and quantification as described in Section 7.4.

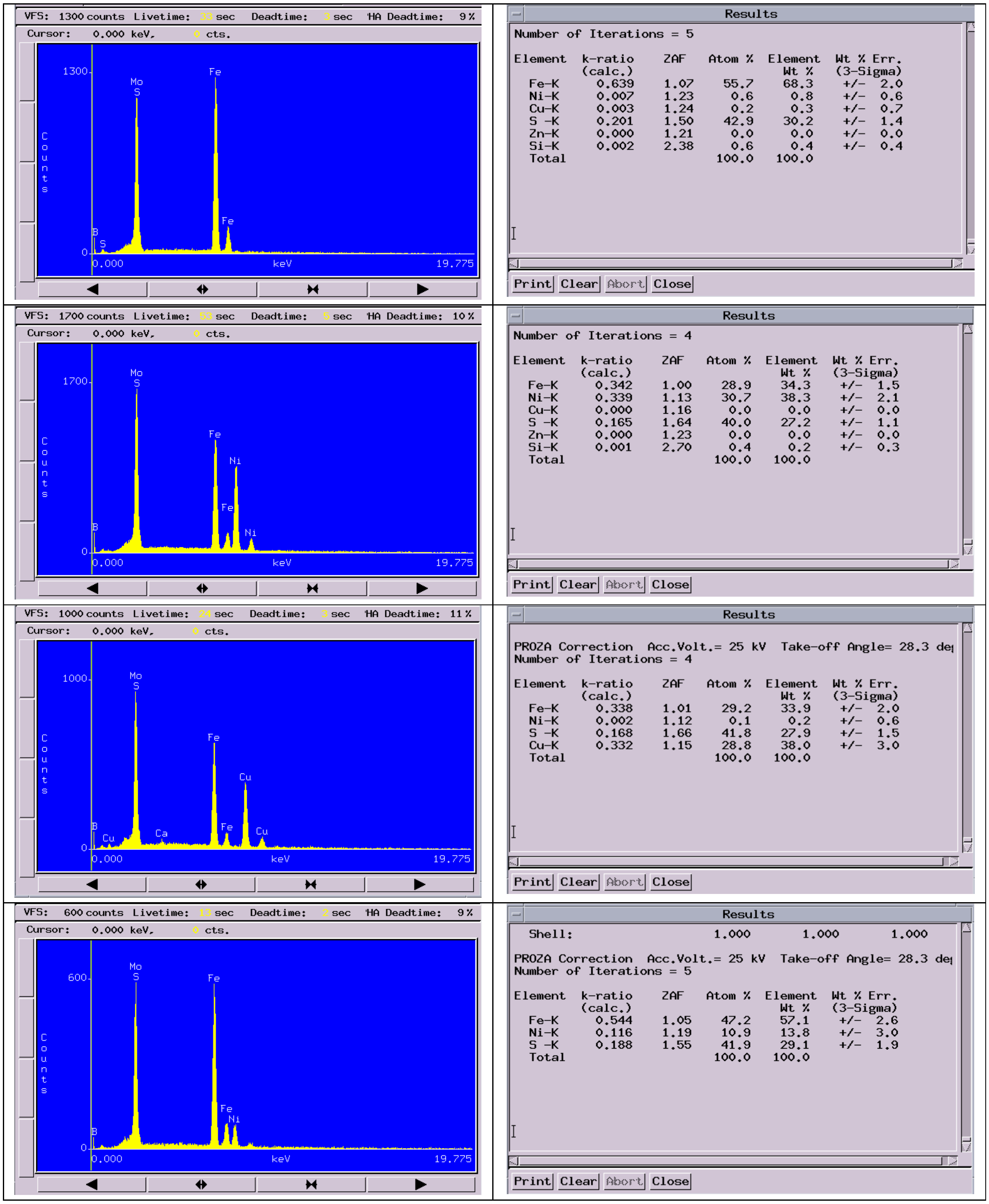
Table 13: Mineral identification and quantification by XRD for natural ore sample for a South African nickel mine.

Mineral	Relative phase amounts (% weight)	Error -3 sigma (% weight)
Biotite	6.87	2.40
Chalcopyrite	0.97	0.45
Chlorite	9.55	2.49
Diopside	6.81	1.8
Enstatite	7.65	2.22
Fosterite	4.94	1.62
Hornblende	17.13	2.22
Lizardite	14.79	2.01
Magnetite	3.46	1.23
Pentlandite	0.76	0.33
Plagioclase	14.2	3.30
Pyrrhotite	5.65	1.02
Quartz	1.42	0.75
Talc	5.7	8.7

This ore does not only contain sulphides (chalcopyrite, pentlandite and pyrrhotite) but also gangue which often consists of positive floaters like talc, diopside, enstatite and plagioclase (Becker, et al., 2009).

Scanning electron microscopy of this ore milled to 70% passing 75 microns, suggests little intergrowth between the sulphides with some intergrowth between gangue and sulphides (**Figure 43**).

The EDS spectral display and quantification results for pyrrhotite, chalcopyrite and pentlandite particles are shown in **Figure 42**. The nickel content in pentlandite is variable. The majority of the pentlandite contained around 31 atom % Ni. Nickel content as low as 10% (atom %) was detected and the spectral display and quantification results for this particle are also shown in **Figure 42**.



```
VFS:  1300 counts  Livetime:  33 sec   Deadtime:   3 sec   'HA Deadtime:   9 %
 Cursor:     0.000 keV,        0 cts.
```

```
Number of Iterations = 5

Element  k-ratio     ZAF    Atom %  Element  Wt % Err.
         (calc.)                     Wt %    (3-Sigma)
  Fe-K     0.639    1.07     55.7    68.3     +/-  2.0
  Ni-K     0.007    1.23      0.6     0.8     +/-  0.6
  Cu-K     0.003    1.24      0.2     0.3     +/-  0.7
  S -K     0.201    1.50     42.9    30.2     +/-  1.4
  Zn-K     0.000    1.21      0.0     0.0     +/-  0.0
  Si-K     0.002    2.38      0.6     0.4     +/-  0.4
  Total                     100.0   100.0
```

```
VFS:  1700 counts  Livetime:  53 sec   Deadtime:   5 sec   'HA Deadtime:  10 %
 Cursor:     0.000 keV,        0 cts.
```

```
Number of Iterations = 4

Element  k-ratio     ZAF    Atom %  Element  Wt % Err.
         (calc.)                     Wt %    (3-Sigma)
  Fe-K     0.342    1.00     28.9    34.3     +/-  1.5
  Ni-K     0.339    1.13     30.7    38.3     +/-  2.1
  Cu-K     0.000    1.16      0.0     0.0     +/-  0.0
  S -K     0.165    1.64     40.0    27.2     +/-  1.1
  Zn-K     0.000    1.23      0.0     0.0     +/-  0.0
  Si-K     0.001    2.70      0.4     0.2     +/-  0.3
  Total                     100.0   100.0
```

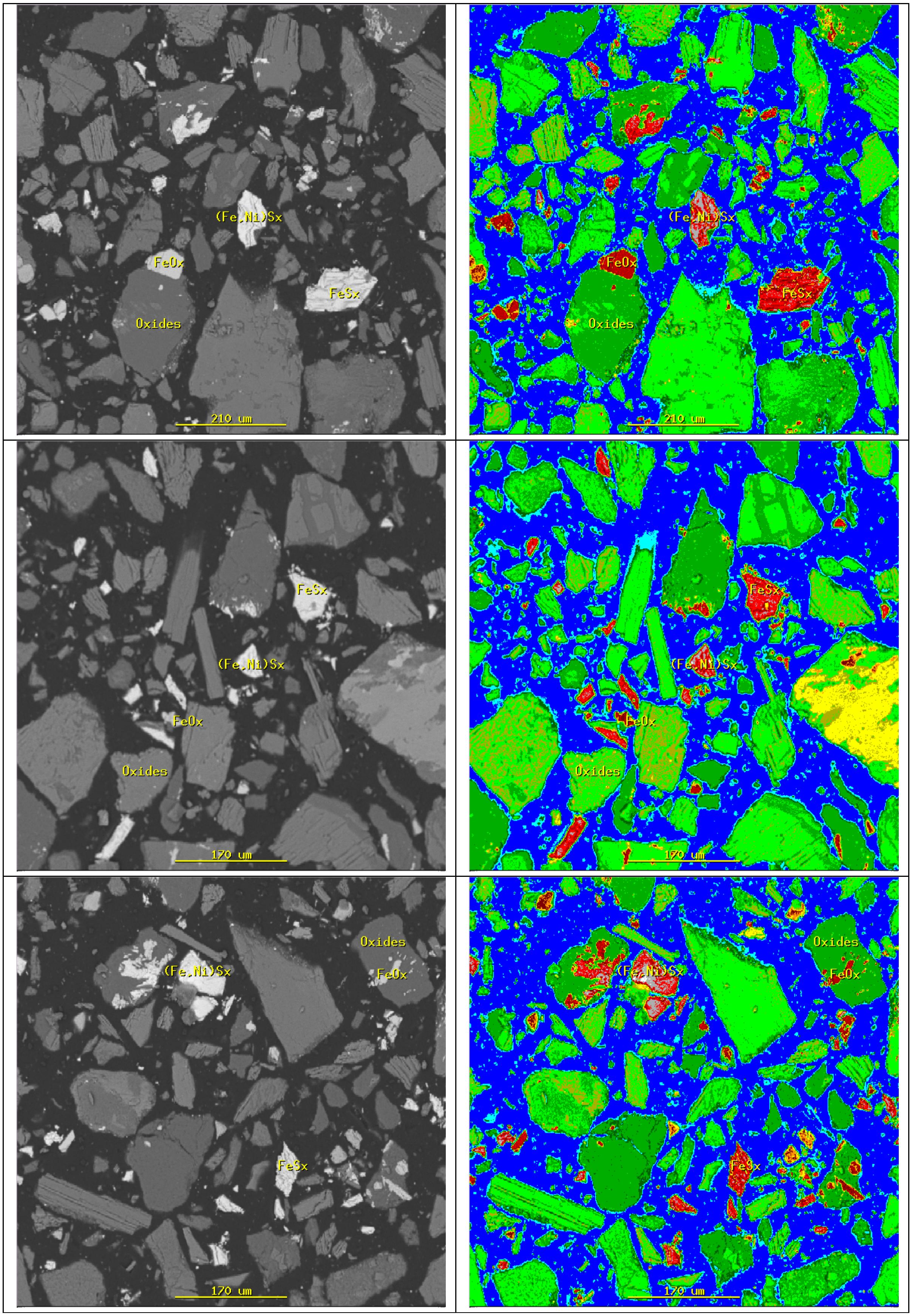
```
VFS:  1000 counts  Livetime:  24 sec   Deadtime:   3 sec   'HA Deadtime:  11 %
 Cursor:     0.000 keV,        0 cts.
```

```
PROZA Correction  Acc.Volt.= 25 kV  Take-off Angle= 28.3 de
Number of Iterations = 4

Element  k-ratio     ZAF    Atom %  Element  Wt % Err.
         (calc.)                     Wt %    (3-Sigma)
  Fe-K     0.338    1.01     29.2    33.9     +/-  2.0
  Ni-K     0.002    1.12      0.1     0.2     +/-  0.6
  S -K     0.168    1.66     41.8    27.9     +/-  1.5
  Cu-K     0.332    1.15     28.8    38.0     +/-  3.0
  Total                     100.0   100.0
```

```
VFS:   600 counts  Livetime:  13 sec   Deadtime:   2 sec   'HA Deadtime:   9 %
 Cursor:     0.000 keV,        0 cts.
```

```
  Shell:                 1.000         1.000         1.000

PROZA Correction  Acc.Volt.= 25 kV  Take-off Angle= 28.3 de
Number of Iterations = 5

Element  k-ratio     ZAF    Atom %  Element  Wt % Err.
         (calc.)                     Wt %    (3-Sigma)
  Fe-K     0.544    1.05     47.2    57.1     +/-  2.6
  Ni-K     0.116    1.19     10.9    13.8     +/-  3.0
  S -K     0.188    1.55     41.9    29.1     +/-  1.9
  Total                     100.0   100.0
```

Figure 42: Analysis of pyrrhotite, chalcopyrite and pentlandite particle by EDS. Natural ore specimen from South African nickel mine.


(Fe,Ni)Sx
FeOx
FeSx
Oxides
210 um
(Fe,Ni)Sx
FeOx
FeSx
Oxides
210 um
FeSx
(Fe,Ni)Sx
FeOx
Oxides
170 um
FeSx
(Fe,Ni)Sx
FeOx
Oxides
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(Fe,Ni)Sx
FeOx
FeSx
170 um

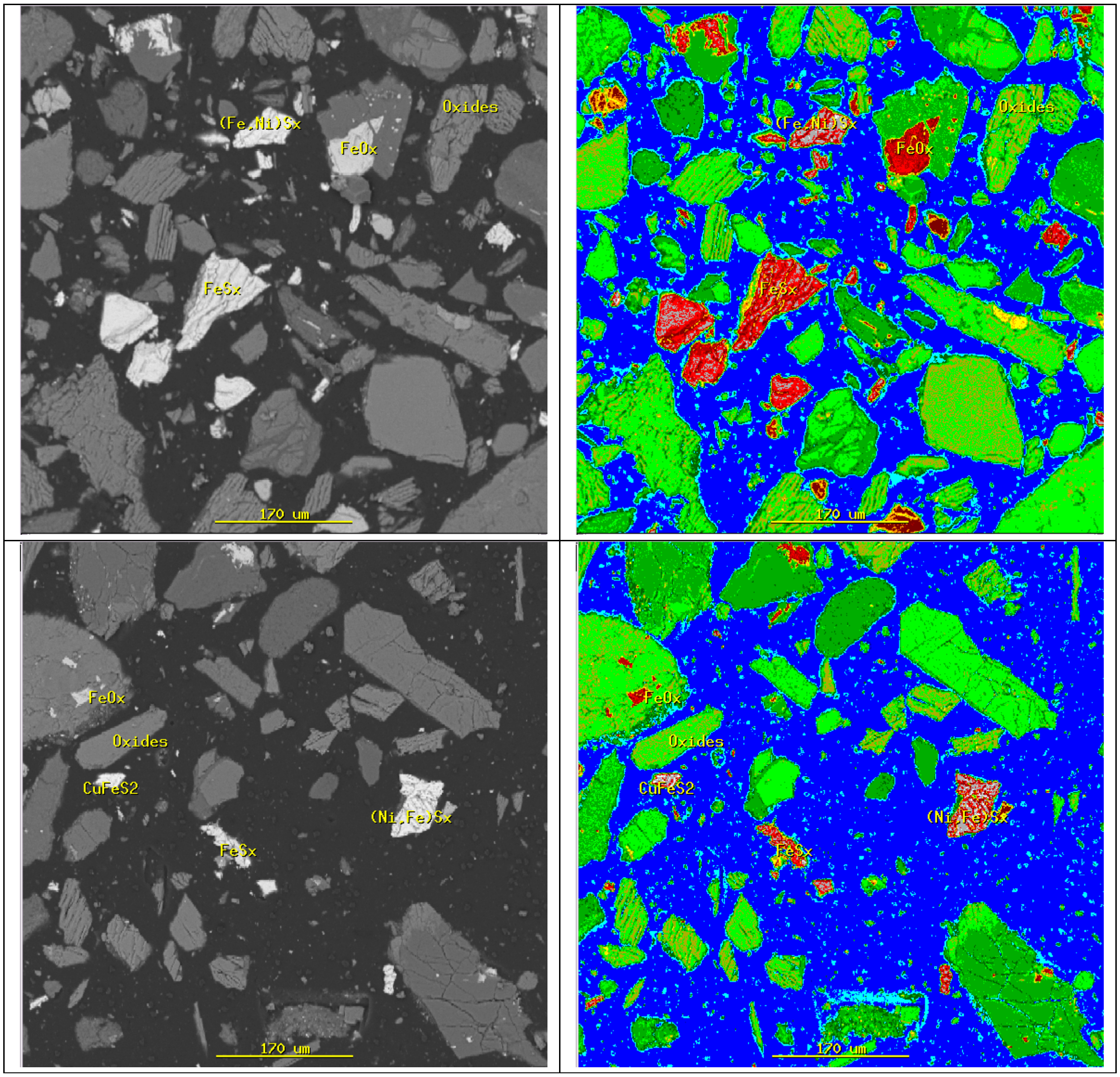


Figure 43: Scanning electron microscope images of nickel ore ROM sample milled in laboratory to 75% passing 75 micron**.

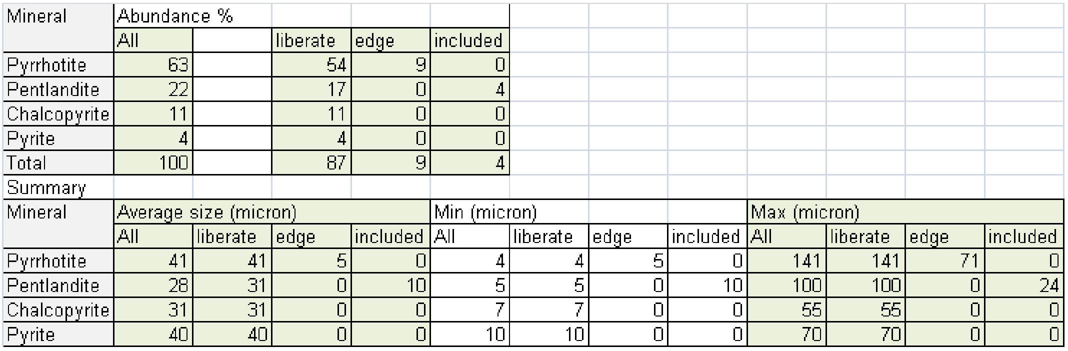
*** The SEM images on the left are original GREY SCALE images and the images on the right are chromatic colour interpretation of the images. Pyrrhotite is indicated as FeSx and is the light Red particles. Pentlandite is indicated as (Ni,Fe)Sx and is the Red particles with grey alteration. Chalcopyrite is indicated as CuFeS2 and is the particles with more grey than red.*

50 particles were randomly selected and classified as pyrrhotite, pentlandite, chalcopyrite and pyrite as well as liberated, edge on gangue and fully included in gangue. This data suggests that at a grind of 70% passing 75 micron, most of the sulphides are liberated (87%) with 9% edge particle and 4% included in gangue. It further suggests that the average size of the sulphides is between 25 and 45 micron and therefore well suited for flotation, which is considered to be efficient between 10 and 100 microns.

The metal content of a target spot of particle can be measured with the EDS function of the SEM used for this study. The nickel content in 30 randomly selected pentlandite particles was analysed. The majority of the pentlandite had a nickel content of between 30 and 40 atom% (38% by mass). The average atom nickel content is 30.9% (standard deviation 4.6). The average atom Fe and S contents are 30% and 39% respectively (standard deviation 3.8 and 3.5).

It should be noted that the study of 50 randomly selected particles is not intended to be a statistically accurate representation of the bulk assay or particle occurrence and serves only as indicative data.

Table 14: Characteristics of 50 sulphide particles randomly selected from a nickel ore Run of mine (ROM) sample milled in laboratory to 70% passing 75 micron



Mineral	Abundance %				
	All		liberate	edge	included
Pyrrhotite	63		54	9	0
Pentlandite	22		17	0	4
Chalcopyrite	11		11	0	0
Pyrite	4		4	0	0
Total	100		87	9	4

Summary

Mineral	Average size (micron)				Min (micron)				Max (micron)			
	All	liberate	edge	included	All	liberate	edge	included	All	liberate	edge	included
Pyrrhotite	41	41	5	0	4	4	5	0	141	141	71	0
Pentlandite	28	31	0	10	5	5	0	10	100	100	0	24
Chalcopyrite	31	31	0	0	7	7	0	0	55	55	0	0
Pyrite	40	40	0	0	10	10	0	0	70	70	0	0

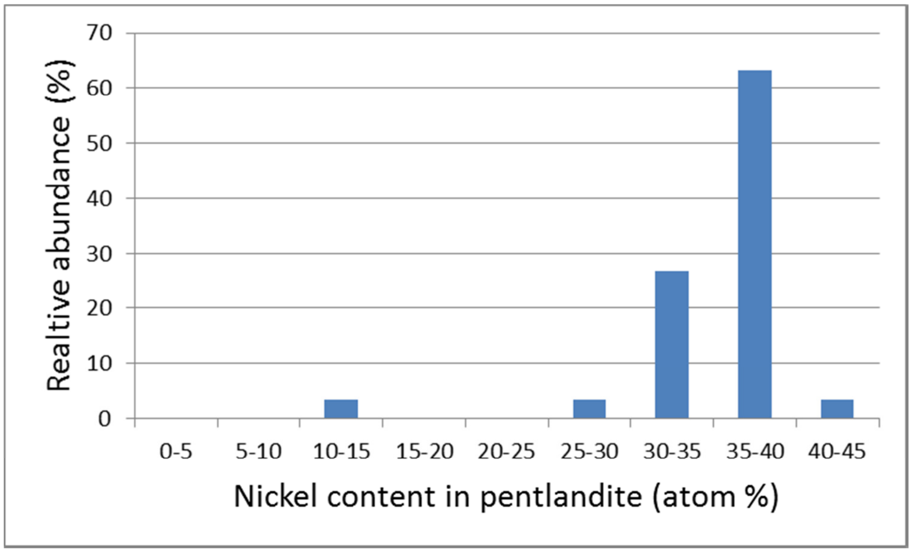


Figure 44: Histogram of the nickel content in pentlandite (atom %) as measured by the EDS attached to the Jeol 6400 SEM used. Results for 30 randomly selected pentlandite particles.

4.9.6. Description of ore sample from of a South African UG2 concentrator.

A final concentrate and final tails sample from the South African platinum UG2 concentrator were studied by XRD and SEM to form an opinion of platinum and sulphide mineralogy.

XRD interpretation and quantification of the final concentrate sample (**Table 15**) indicates that chalcopyrite, pentlandite and pyrrhotite are the major sulphides in the final flotation concentrate. It is assumed that these sulphides contains PGM and Au as nano-inclusions or solid solution and is the target value minerals for flotation. This was not studied further.

Table 15: Quantitative XRD results for UG2 final concentrate

Minerals	Relative abundance %
Chalcopyrite	1.8
Pentlandite	2.1
Pyrrhotite	2.7
Chlorite	10.6
Diopside	0.7
Enstatite	11.5
Plagioclase	3.9
Talc	52.8
Forsterite	1.3
Hornblende	1.4
Lizardite	10.7
Magnetite	0.2
Quartz	0.3

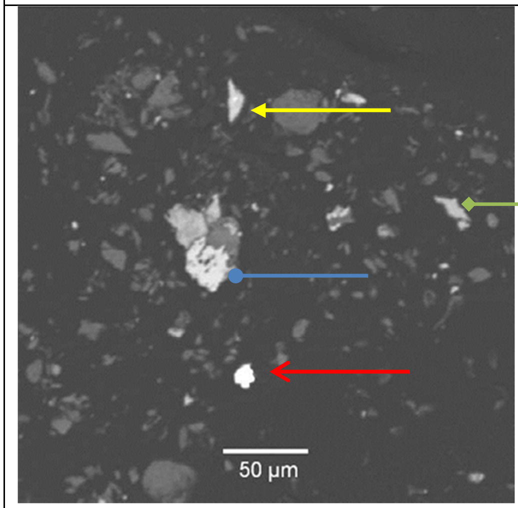
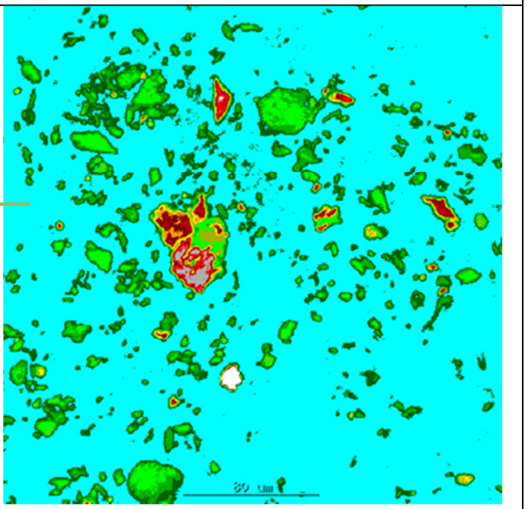
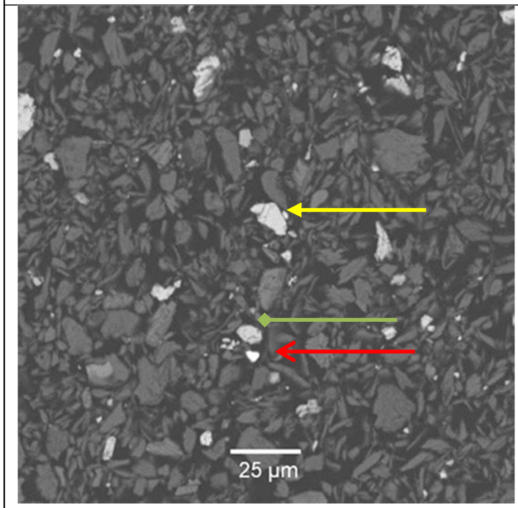
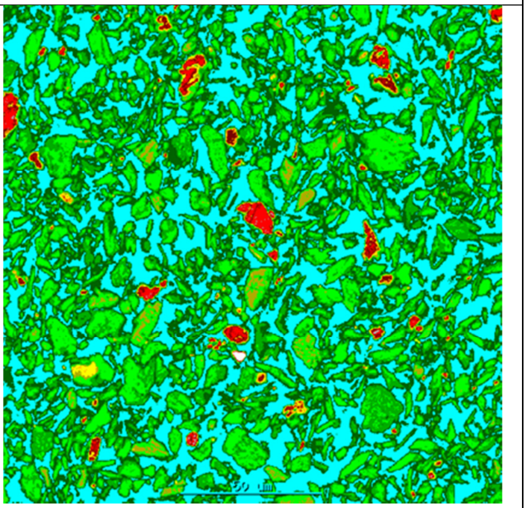
The SEM images in **Table 16** shows platinum alloy particles in the UG2 final concentrate. These particles are abundant and were easily detected.

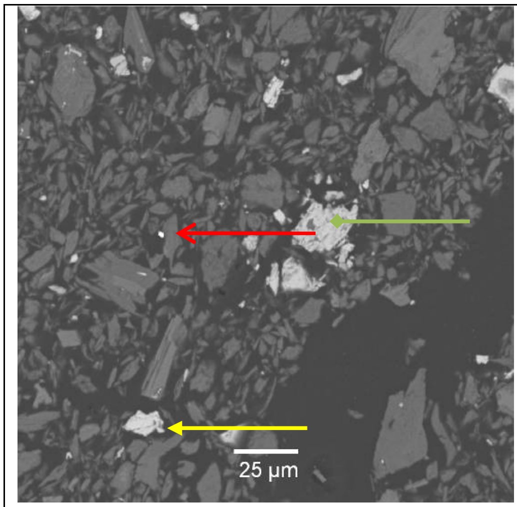
Pyrrhotite had been identified as predominant sulphide mineral in the UG2 concentrate. Both monoclinic and hexagonal pyrrhotite had been detected by XRD.

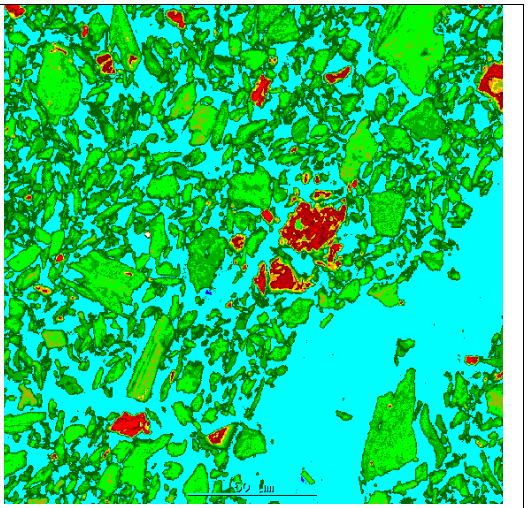
Pentlandite had been identified as the second most abundant sulphide in the UG2 concentrate. Most of the pentlandite detected has a Ni content of 35w%. This would be considered as being high and it is likely that the pentlandite might be slow floating (Chanturiya, et al., 2004). Some liberated fine Millerite (NiS) particles were detected in the final tails (**Table 17**). It is reported that Millerite and pentlandite below 10 micron floats poorly (Smith, et al., 2011)

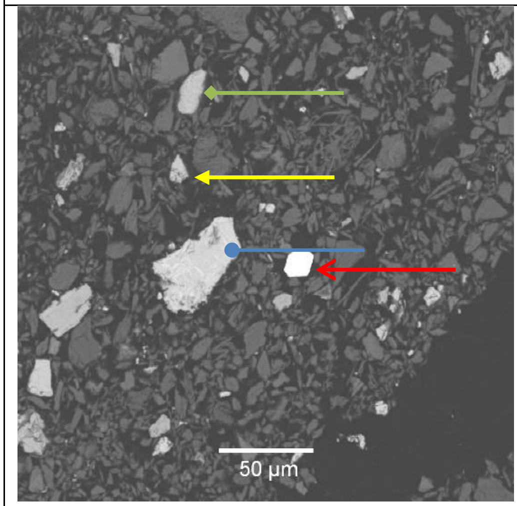
Chalcopyrite has been identified as the least predominant sulphide mineral in the final UG2 concentrate.

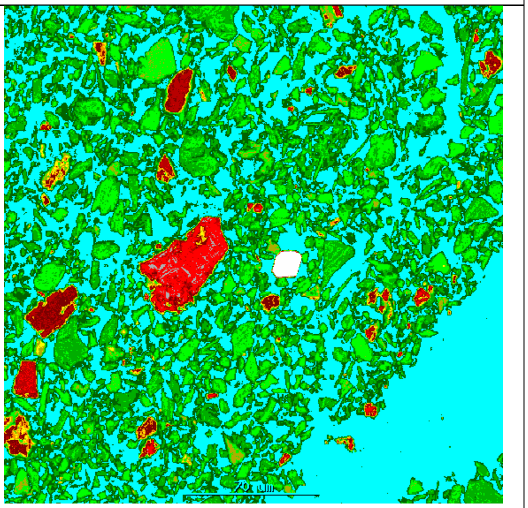
Table 16: Selected SEM images of a South African platinum concentrator final flotation concentrate.

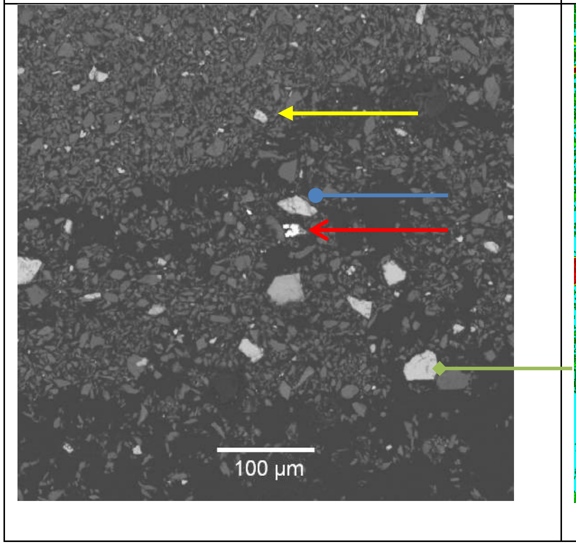
Scanning Electron Microscope images of: UG 2 Final Concentrate	
Pyrrhotite	◆―――
Chalcopyrite	●―――
Platinum Alloy	←―――
Pentlandite	◄―――
	
	


25 µm




50 µm




100 µm

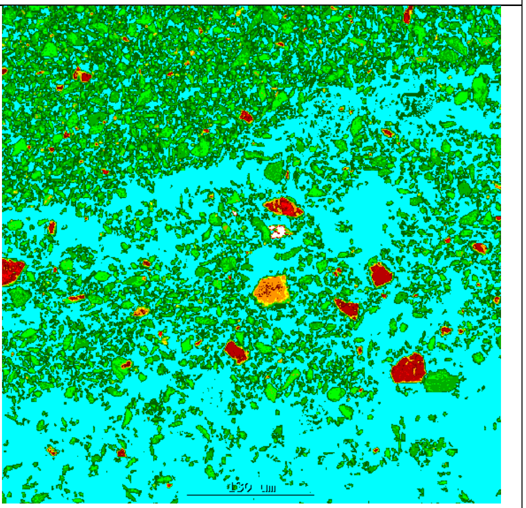
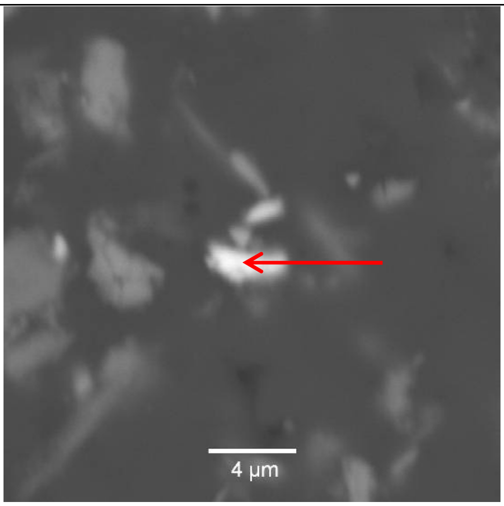
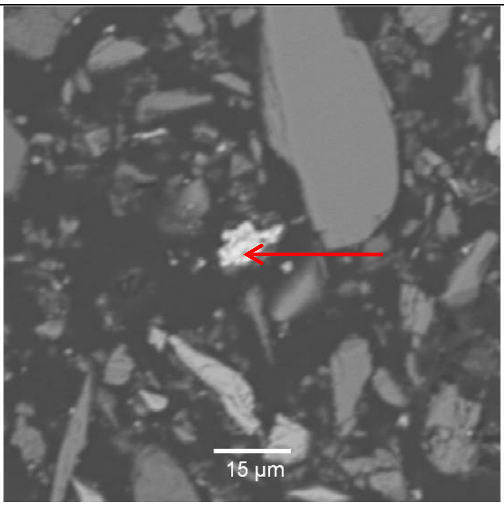
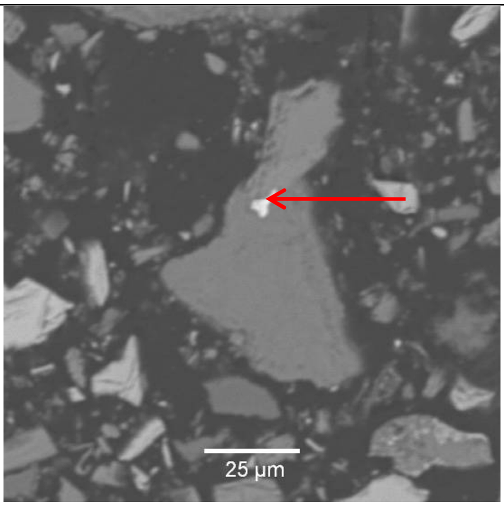


Table 17: Selected SEM images of a South African Platinum concentrator final flotation tails.

	Millerite (Ni_2S)
	Pentlandite (18w% Ni)
	1- Pentlandite (33w% Ni) included in Enstatite

A subsequent scan of a flotation concentrate sample using the Mineral Liberation Analyser (MLA) has identified the PGE mineral as mainly Braggite (PtPdS) and Cooperite (PtS). The sample was stored as wet filtercake and most sulphides was oxidised when scanned. No association of the PGM and BMS could be measured. The assumption that the PGM are associated with the base metal sulphides is thus untested.

5. Results and discussion

5.1. Laboratory scale batch flotation test using a base metal sulphide ore

Laboratory scale batch flotation tests were conducted to test the rate of base metal recovery when a fatty acid collector (Betacol 364) is used in combination with xanthate and compare this with tests where xanthate were used as collector. An ore containing chalcopyrite, pentlandite and pyrrhotite as the main sulphide minerals was used for these tests. This ore is described in Section 4.9.5. A standard laboratory scale batch flotation test method was used which is described in Section 4.2.1.

The fatty acid collector (Betacol 364) and xanthate combination was tested against the baseline condition where only xanthate was used as collector. As further references, two dithiophosphate (DTP) collectors in combination with xanthate were used. The first DTP used was Betacol 44A which is a di-isobutyl dithiophosphate based collector and the second was Betacol 316 which is a zinc dithiophosphate based collector. As an added reference, a combination between xanthate and a hydrocarbon based collector (Betacol 25) was included in the test procedure.

Each test was done in triplicate. The test quality control method used for these tests compared the calculated feed grade with the measured feed grade (call factor), as described by Lotter et al. (Lotter & Fragomeni, 2010). For these tests the call factor acceptance limits were 110% to 90%.

The nickel and copper grade and recovery data for the test is summarised in **Table 18**. The data set is listed in the appendix as **Table 33**. The Cu and Ni elemental grades were measured using a sodium peroxide fusion method followed by Atomic Absorption Spectroscopy (AAS) as described in Section 7.1.

The rate of flotation can be compared by fitting the data to the rate equations and comparing the equation constants (Hay, 2005), or by plotting and comparing time vs. recovery data. The time vs. recovery data for copper and nickel is shown in **Figure 45** and **Figure 47**. A useful additional method is to compare the rate of flotation at the initial stages of the flotation test where it is assumed the rate of flotation is constant (Pistorius, 2009). For this data set the rate of flotation at the 5 minutes interval was used and is listed in **Table 18**.

A low increase in the rate of copper recovery is measured when the fatty acid collector (Betacol 364) is used in combination with xanthate (5 min Cu rate of recovery – 12 %/min), compared to the baseline test where only xanthate was used (5 min Cu rate of recovery – 11.2 %/min), (**Table 18** and **Figure 45**). A higher rate of copper recovery was measured for the two DTP and xanthate combinations (5 min Cu rate of recovery – 11.7 and 12.9 %/min), (**Table 18** and **Figure 45**).

The selectivity of the flotation process can be measured by fitting gangue recovery data to suitable rate equation, plotting gangue recovery as a function of time or plotting the value element grade recovery curve. The copper grade recovery data (**Figure 46**) indicates more selective copper flotation when Betacol 364 is used in combination with xanthate than does the baseline test or the two tests where a DTP is used with xanthate.

When the hydrocarbon collector (Betacol 25) was used in combination with xanthate, the rate of Cu flotation decreased (**Table 18** and **Figure 45**), as well as the selectivity (**Figure 46**). This result was unexpected; as hydrocarbon collectors are used in combination with thiol collectors to recover copper (Rubio, et al., 2007).

The rate of nickel recovery was significantly higher when Betacol 364 was used in combination with xanthate (5 min Ni rate of recovery – 5.6 %/min), compared to the baseline test (5 min Ni rate of recovery – 1.9 %/min). However, when any of the two DTP collectors were used, the rate of Ni recovery was higher (5 min Ni rate of recovery – 6.8 and 6.8 %/min) (**Table 18** and **Figure 47**).

Nickel was floated more selectively when Betacol 364 was used in combination with xanthate than the baseline test and any of the other reference collector combinations (**Figure 48**).

The reference test where the hydrocarbon collector (Betacol 25) was used in combination with xanthate was similar to the baseline test in the rate of Ni recovery and selectivity (**Table 18** , **Figure 47** and **Figure 48**).

Table 18: Summary of bench scale flotation test results**.

	Copper					Nickel				
Co-collector	Recovery	Conc	Rate of	Conc	Call	Recovery	Conc	Rate of	Conc	Call
		Grade	flotation	Grade	Factor		Grade	flotation	Grade	Factor
	40 min	40 min	at 5 min	5 min		40 min	40 min	at 5 min	5 min	
	%	%	%/min	%	%	%	%	%/min	%	%
Nil (Baseline)	85	0.70	10.8	1.8	98	52	1.23	2.1	1.0	101
Nil (Baseline)	83	0.76	11.7	2.2	97	49	1.26	2.0	1.1	104
Nil (Baseline)	83	0.78	11.4	2.6	97	52	1.42	1.6	1.0	101
Nil (Baseline)	84	0.74	11.1	2.2	96	54	1.36	1.8	1.0	100
Average	**84**	**0.74**	**11.2**	**2.2**		**52**	**1.32**	**1.9**	**1.0**	
(Betacol 364)	83	1.05	11.8	3.3	93	76	2.74	6.3	5.0	99
(Betacol 364)	84	1.03	12.2	3.0	100	73	2.65	6.7	5.0	101
(Betacol 364)	83	1.10	12.1	4.4	95	72	2.82	4.0	4.3	97
Average	**83**	**1.06**	**12.0**	**3.6**		**74**	**2.74**	**5.6**	**4.7**	
Betacol 44A	82	0.75	12.4	1.6	102	79	2.29	6.2	2.6	97
Betacol 44A	84	1.04	12.4	2.5	100	78	3.11	5.9	3.8	95
Betacol 44A	88	0.61	13.5	1.4	100	83	1.79	8.3	2.7	97
Average	**85**	**0.80**	**12.7**	**1.8**		**80**	**2.40**	**6.8**	**3.0**	
(Betacol 316)	85	0.77	12.4	1.7	107	78	2.32	6.1	2.7	99
(Betacol 316)	84	0.78	12.2	1.5	105	77	2.29	5.7	2.3	100
(Betacol 316)	88	0.68	14.0	1.2	103	82	1.95	8.4	2.2	100
Average	**85**	**0.74**	**12.9**	**1.5**		**79**	**2.19**	**6.8**	**2.4**	
(Betacol 25)	72	0.71	6.5	1.1	96	42	1.17	1.3	0.6	103
(Betacol 25)	69	0.71	5.8	1.3	98	44	1.24	1.0	0.6	107
(Betacol 25)	69	0.72	4.5	1.3	103	44	1.34	0.7	0.5	107
Average	**70**	**0.71**	**5.6**	**1.2**		**43**	**1.25**	**1.0**	**0.6**	

***Four co-collectors were tested against baseline using a D12 bench flotation machine. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother; Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated added at 20 g/t. Assayed results by sodium peroxide digestion and AAS finish. Recovery data is shown after 40 minutes of flotation as an indication of terminal recovery. Rate of flotation is calculated at 5 minutes as the linear recovery per minute. The concentrate grade at 5 minutes is shown as an indication of selectivity as the interval kinetic data is calculated.*

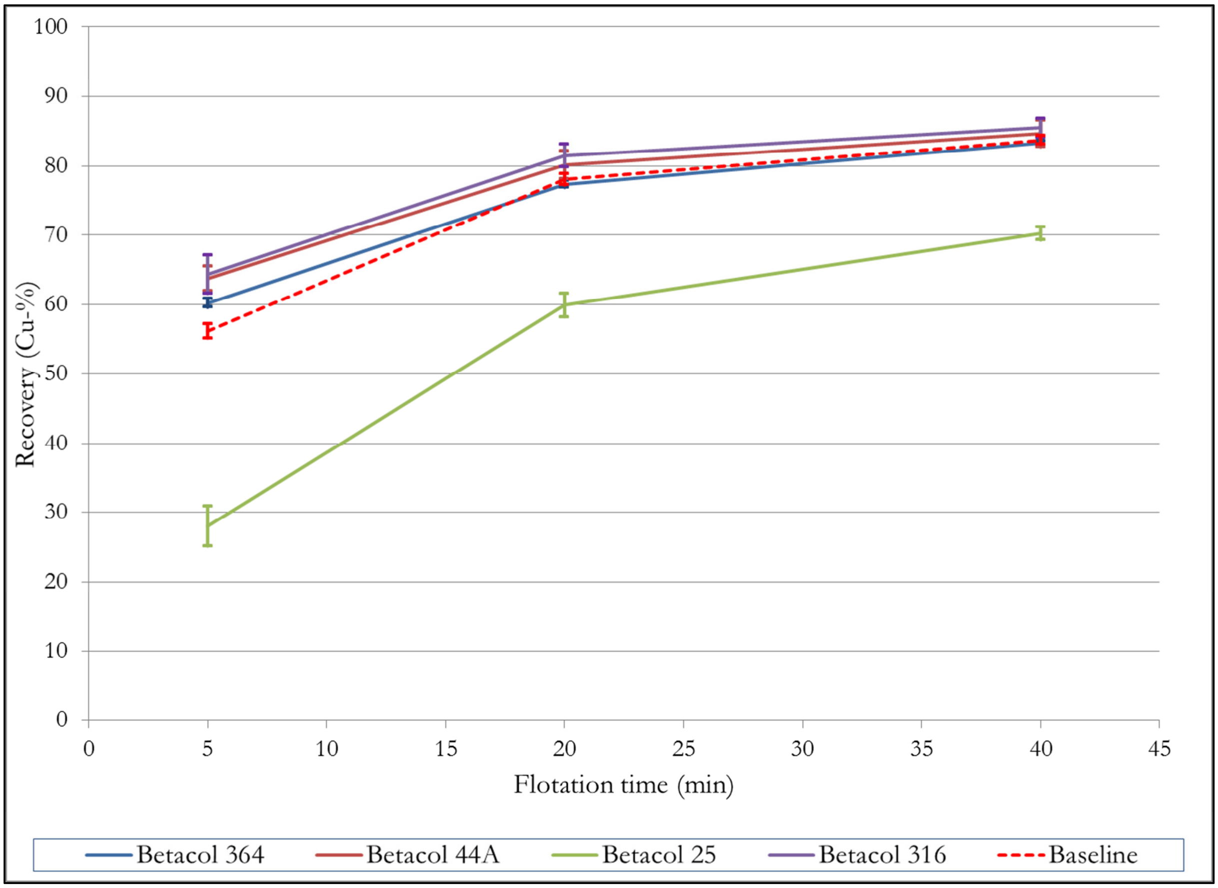


Figure 45: Copper recovery and time plot for bench scale flotation test. Natural ore**.

*** Four co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time: 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), co-collector as indicated at 20 g/t. Assayed by sodium peroxide digestion and AAS finish. Error bars based on standard error.*

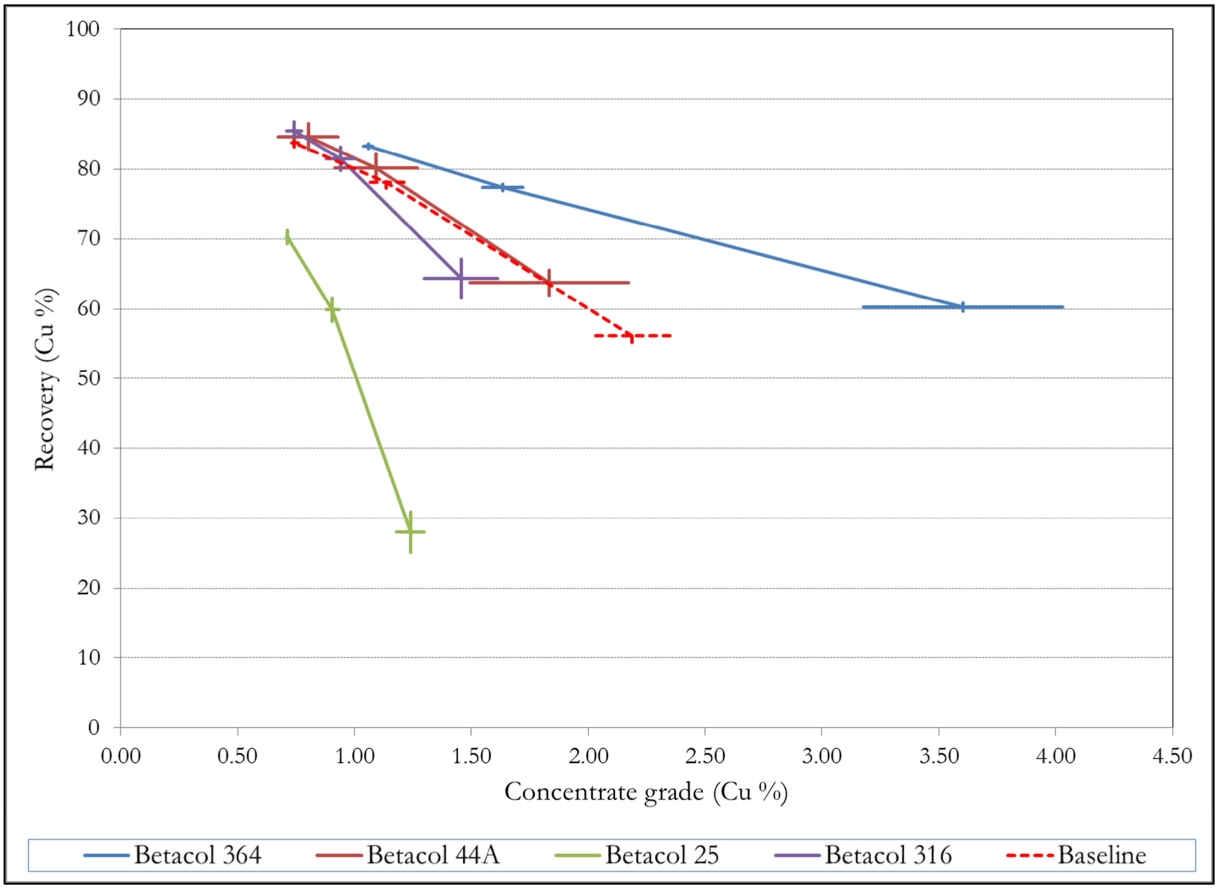


Figure 46: Copper grade and recovery plot for bench scale flotation test. Natural ore**.

***Four co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time: 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Assayed by sodium peroxide digestion and AAS finish. Error bars based on standard error.*

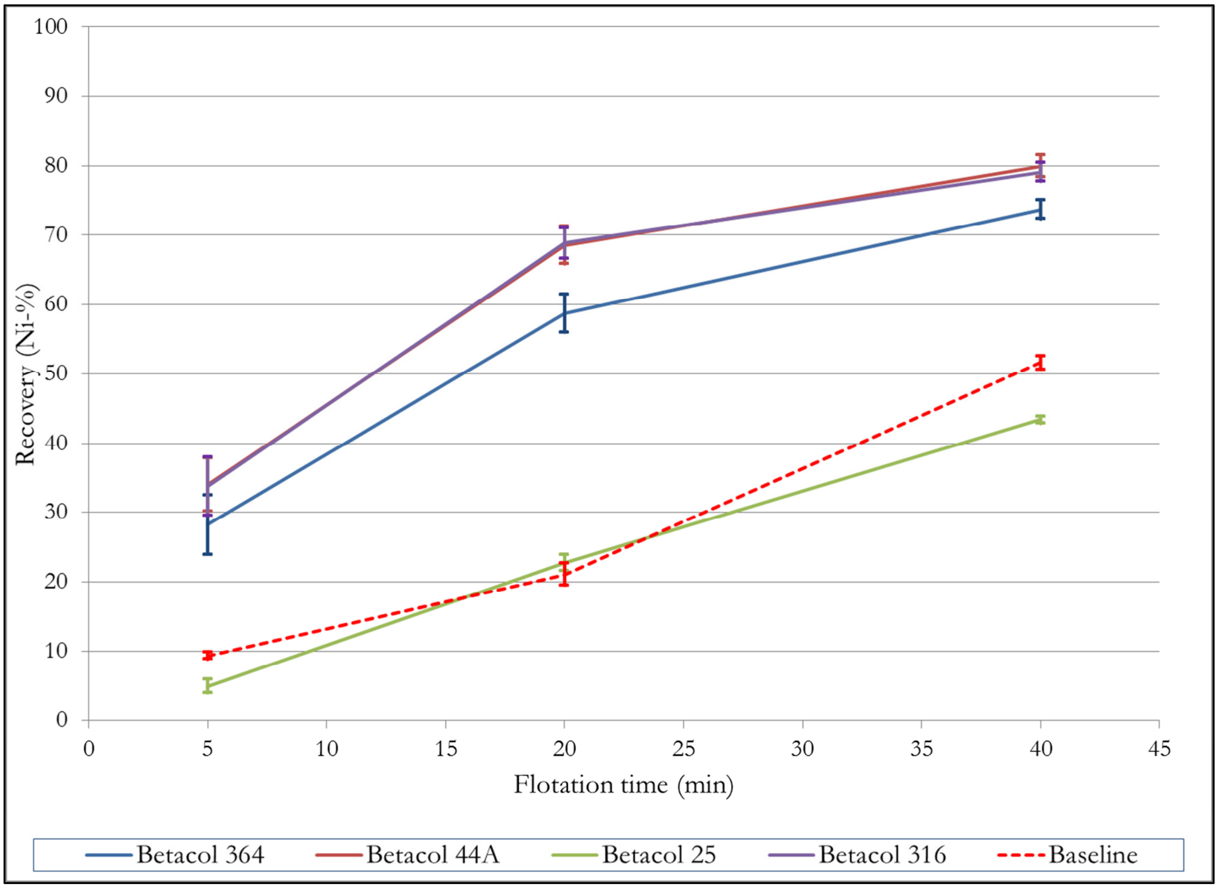


Figure 47: Nickel recovery and time plot for bench scale flotation test. Natural ore**

***Four co-collectors were tested against the baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time: 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Assayed by sodium peroxide digestion and AAS finish. Error bars based on standard error.*

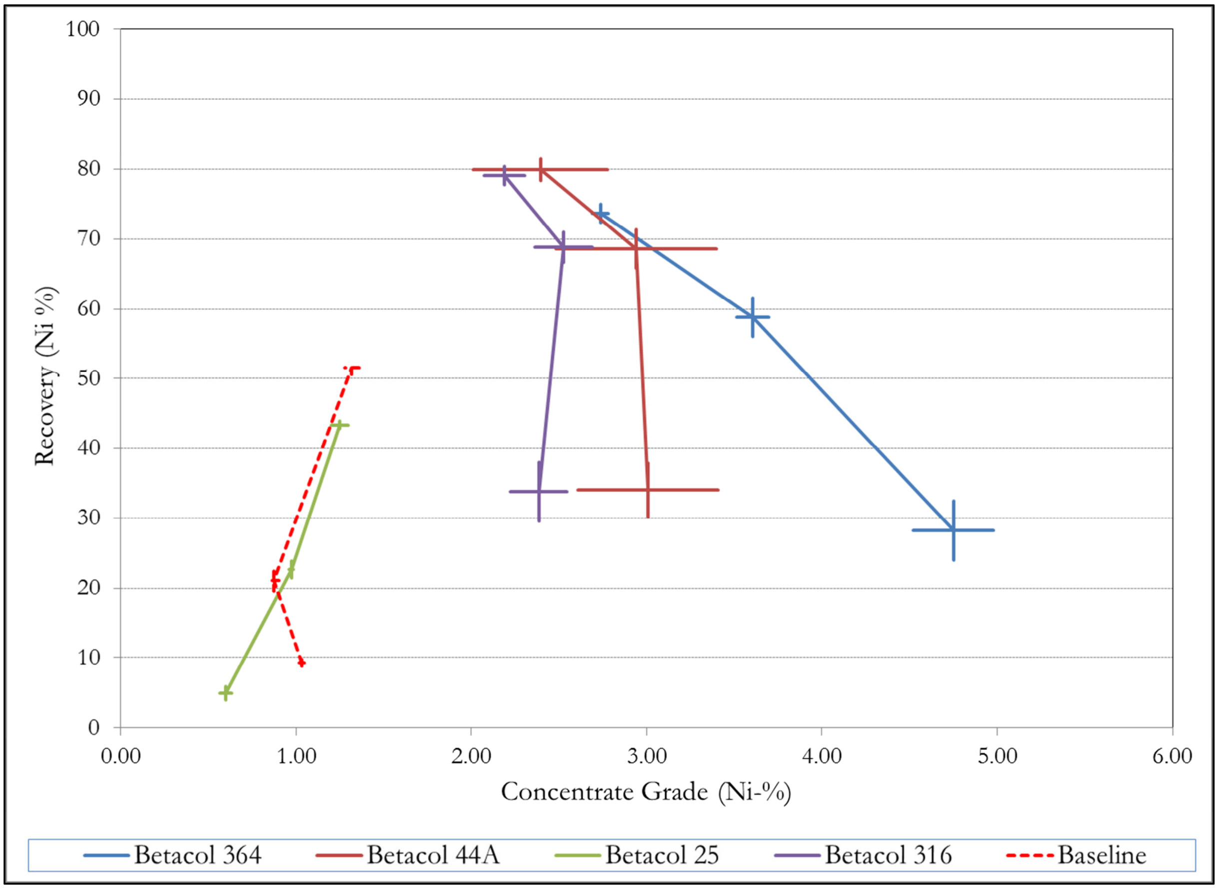


Figure 48: Nickel grade and recovery plot for bench scale flotation test. Natural ore**.

***Four co-collectors were tested against the baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time: 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Assayed by sodium peroxide digestion and AAS finish. Error bars based on standard error.*

One of the three replica flotation test samples for the baseline test, the test where the fatty acid collector (Betacol 364) with xanthate and the test where a DTP with xanthate was used as collector were submitted for sulphide sulphur assay using a LECO sulphur analyser (Section 7.2). The test data is listed in the appendix as **Table 35** and summarised in **Table 19**.

The sulphur recovery vs. time plot (**Figure 49**) indicates that the recovery of sulphur proceeds faster when the fatty acid collector is used in combination with xanthate than the baseline test where only xanthate is used as collector. The recovery of sulphur is, however, not faster than when a DTP (Betacol 316) is used in combination with xanthate. The grade vs. recovery curve (**Figure 50**) indicates that the sulphur is recovered more selectively when the fatty acid collector (Betacol 364) is used with xanthate compared to the baseline test and the test where a DTP (Betacol 316) was used with xanthate.

Table 19: Sulphur assay (by LECO) for concentrate samples from bench flotation test conducted with two co-collectors and the baseline**.

		Mass	Sulphur Assay Leco (%)	Sulphur			
				Recovery %	Recovery Cumulative %	Grade %	Grade Cumulative %
Feed		1000.0	3.28				
Betacol 364	Tails	878.1	1.32				
	Call		99%				
	Conc 0-5 min	27.4	17.2	14.5	14.5	17.2	17.2
	Conc 5-20 min	49.8	16.8	25.8	40.3	16.8	16.9
	Conc 20-40 min	44.7	17.4	24.0	64.3	17.4	17.1
Betacol 316	Tails	853.3	1.22				
	Call		99%				
	Conc 0-5 min	49.1	10.0	15.1	15.1	10.0	10.0
	Conc 5-20 min	58.7	18.8	33.9	49.0	18.8	14.8
	Conc 20-40 min	38.9	15.9	19.0	68.0	15.9	15.1
Baseline	Tails	838.6	2.60				
	Call		103%				
	Conc 0-5 min	39.0	5.57	6.4	6.4	5.6	5.6
	Conc 5-20 min	70.0	5.93	12.3	18.7	5.9	5.8
	Conc 20-40 min	52.4	10.8	16.8	35.5	10.8	7.4

*** South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Sulphide assay by Leco.*

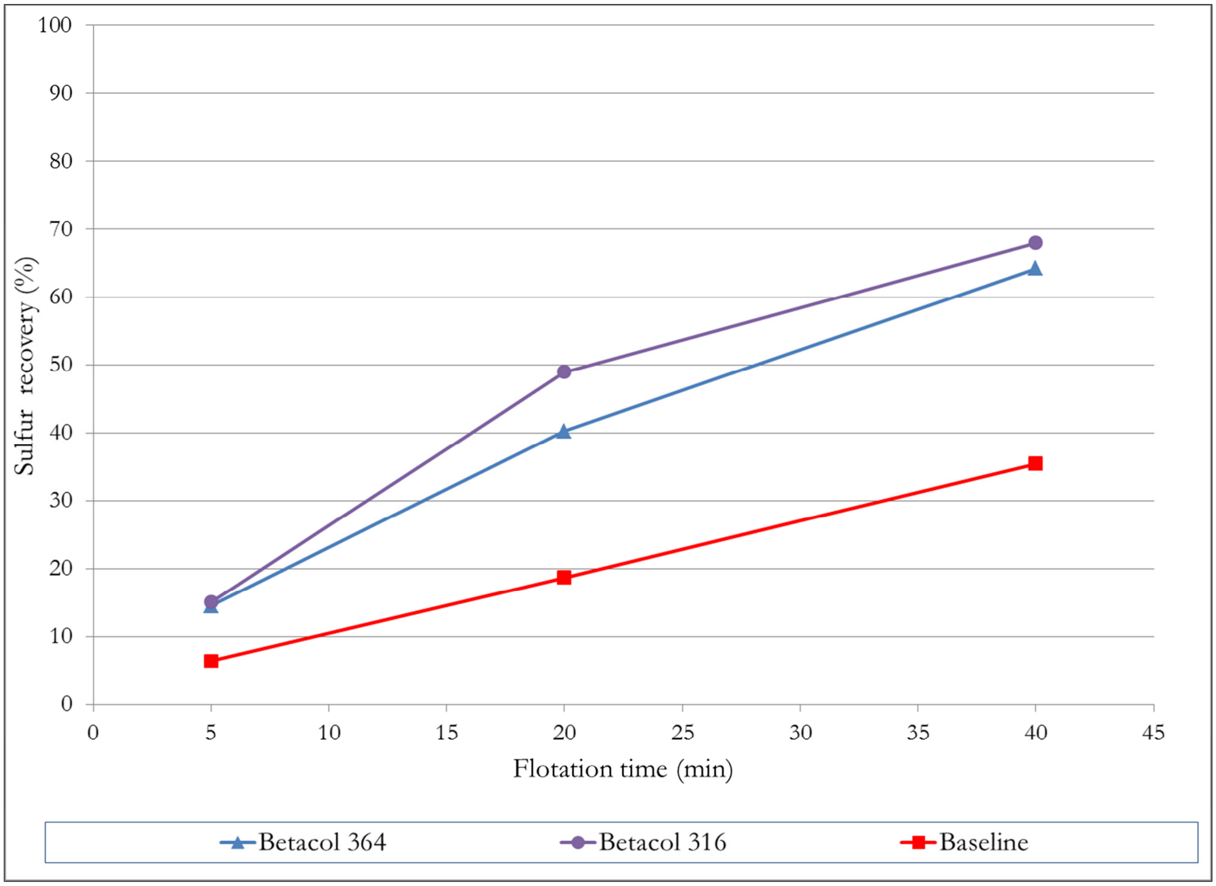


Figure 49: Sulphide sulphur recovery and time plot for bench scale flotation test**

*********Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; (SEX, 50 g/t) SIPX (50 g/t), Frother Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Sulphide assay by Leco.*

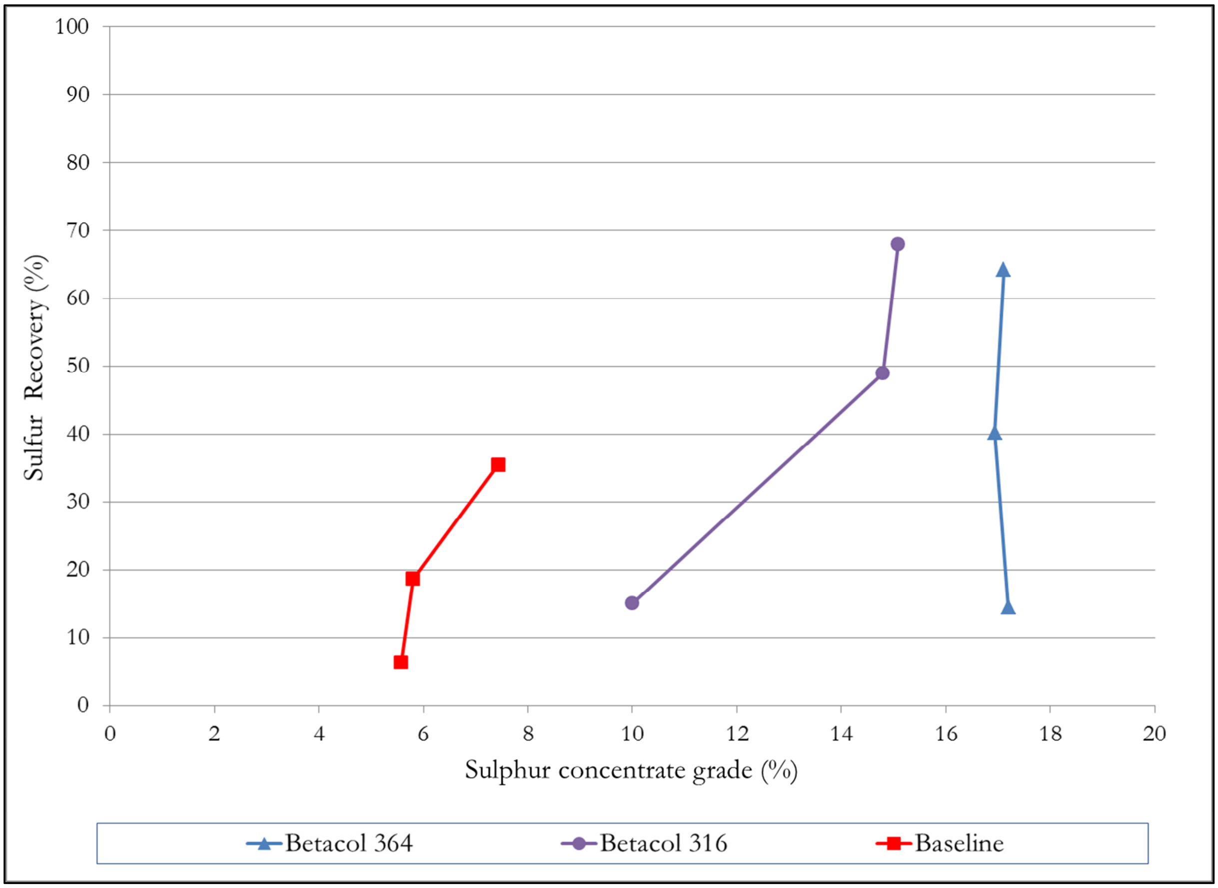


Figure 50: Sulphide grade and recovery plot for bench scale flotation test**

*** Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; (SEX, 50 g/t) SIPX (50 g/t), Frother Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Sulphide assay by Leco.*

In this ore sample the three main sulphide minerals are chalcopyrite, pentlandite and pyrrhotite. Chalcopyrite flotation behavior can be studied using the Cu assay data, pentlandite using the Ni assay data and the sum of the three sulphide minerals using the sulphide sulphur data.

Minerals grade and recovery data for the tests was calculated using two methods. The first method used was to do XRD scans (Rietveld refinement technique), to quantify the relative mineral content in the samples. This work was done by Dr Verryn from XRD Analytical and Consulting (Verryn, 2009). The analytical data is listed in the appendix as **Table 34** and summarised in **Table 20**.

The second method used was to calculate the mineral content in the ore samples from the base metal element and sulphur assay data. For this ore the only significant nickel mineral is pentlandite with a narrow range Ni content. The only significant copper mineral is chalcopyrite. Pyrrhotite is the only other sulphide present in significant quantities, and is assumed to have a constant F to S ratio. This has been described in Section 4.9.5. It is thus possible to calculate the chalcopyrite content from the Cu assay value, and the pentlandite from the Ni assay value. The pyrrhotite content can be calculated using the sulphur assay value by deducting the sulphur present in chalcopyrite and pentlandite from the total sulphur content. The mineral content in the flotation test samples that was calculated using this method is listed in the appendix as **Table 35** and summarised in **Table 20**.

A comparison between the mineral grades calculated using the two methods to calculate mineral content is shown in **Figure 51** with linear data fit (x-coefficient and error R^2) shown on the data plot. From this data it is concluded that the two methods used do not calculate the mineral content in the samples to be the same. The accuracy of the XRD quantification method and the assumptions made when calculating mineral content from assay data are the reasons for this variance. The value of using the mineral data to help interpret flotation performance is in this instance the balance between additional clarity to interpret flotation data, and the loss of data accuracy. For this study, mineral flotation data for both methods will be considered simultaneously and used only to support the assay data where possible.

Table 20: Mineral identification and quantification by XRD and calculated mineral content from Cu, Ni assay (by AAS) and S (by Leco) for bench flotation test**. .

		Pentlandite		Chalcopyrite		Pyrrhotite		Oxide gangue	
		Calc mass % Ni assay	XRD Mass %	Calc mass % Cu assay	XRD Mass %	Calc mass % S assay	XRD Mass %	Calc mass % S assay	XRD Mass %
Feed		1.3	1.0	0.4	0.5	7.1	5.7	91.1	92.8
Betacol 364	Tails	0.4		0.1		3.1		96.4	
	Conc 1	15.5	11.3	9.6	11.3	23.2	9.4	51.7	68.0
	Conc 2	8.7	6.0	1.7	1.4	35.4	20.1	54.2	72.5
	Conc 3	3.9	2.0	0.5	0.7	42.2	24.0	53.3	73.3
Betacol 316	Tails	0.3		0.1		2.9		96.7	
	Conc 1	8.4	5.1	4.9	5.6	14.7	5.9	72.1	83.4
	Conc 2	8.5	7.5	1.2	1.2	41.4	27.5	49.0	63.8
	Conc 3	3.8	2.7	0.4	0.7	38.5	25.9	57.3	70.7
Baseline	Tails	0.8		0.1		6.1		93.0	
	Conc 1	3.3	1.8	6.4	5.1	6.0	5.1	84.3	88.1
	Conc 2	2.7	1.7	1.2	1.4	12.3	6.7	83.8	90.2
	Conc 3	6.0	4.0	0.4	0.9	23.0	10.2	70.6	84.9

***Two co-collectors were tested ((Betacol 364) at 20- g/t plus 100 g/t Xanthate and (Betacol 316) at 20 g/t plus 100 g/t xanthate) using D12 bench flotation equipment . South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; (SEX, 50 g/t) SIPX (50 g/t), Frother; Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated added at 20 g/t. Minerals recoveries calculated assuming: Pentlandite 34.9 % Ni and 35% S, chalcopyrite 34.6% Cu and 35% S, pyrrhotite 37.7% S*

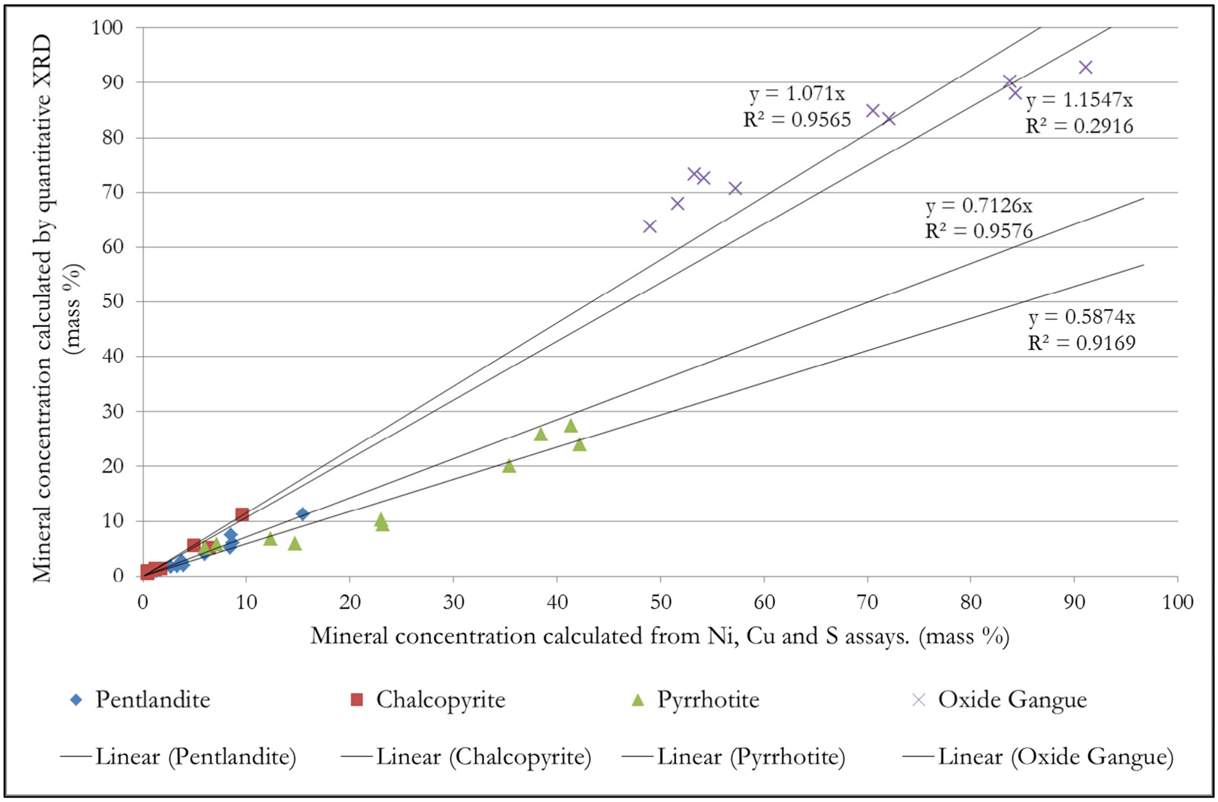


Figure 51: Mineral concentration measured by quantitative XRD (y axis) compared to calculate mineral concentration from Cu and Ni assay (x axis).

When considering both sets of mineral data (**Figure 52** and **Figure 53**), there is no evidence to support any conclusion that the use of the fatty acid collector (Betacol 364) with xanthate has any effect on the recovery of chalcopyrite.

The rate of pentlandite recovery increased when Betacol 364 was used in combination with xanthate, compared to the baseline test where only xanthate was used as collector (**Figure 54** and **Figure 55**). The data does not support a conclusion that the use of the fatty acid collector (Betacol 364) with xanthate floated pentlandite faster than the test where DTP (Betacol 316) was used in combination with xanthate.

The use of Betacol 364 with xanthate resulted in an increase in the rate of pyrrhotite recovery in comparison to the baseline test where only xanthate was used as collector. The use of a DTP (Betacol 316) resulted in faster pyrrhotite flotation compared to the fatty acid xanthate combination (**Figure 56** and **Figure 57**).

From both mineral recovery data sets it can be concluded that less oxide gangue is recovered when either the fatty acid or the DTP collector is used in combination with xanthate than the baseline test, where xanthate is used as sole collector (**Figure 58** and **Figure 59**).

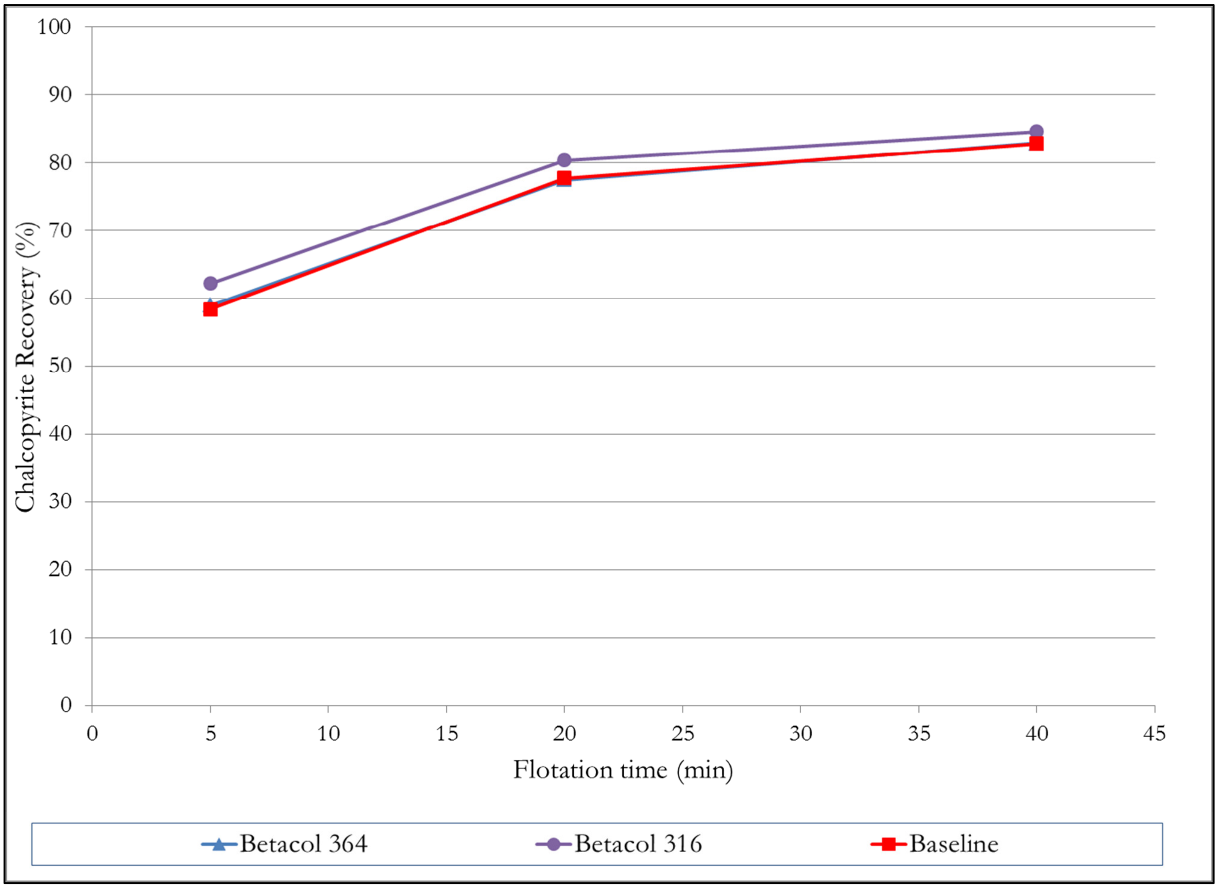


Figure 52: Chalcopyrite recovery and time plot for bench scale flotation test calculated from Cu, Ni and S assay values**

*** Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; (SEX, 50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t.*

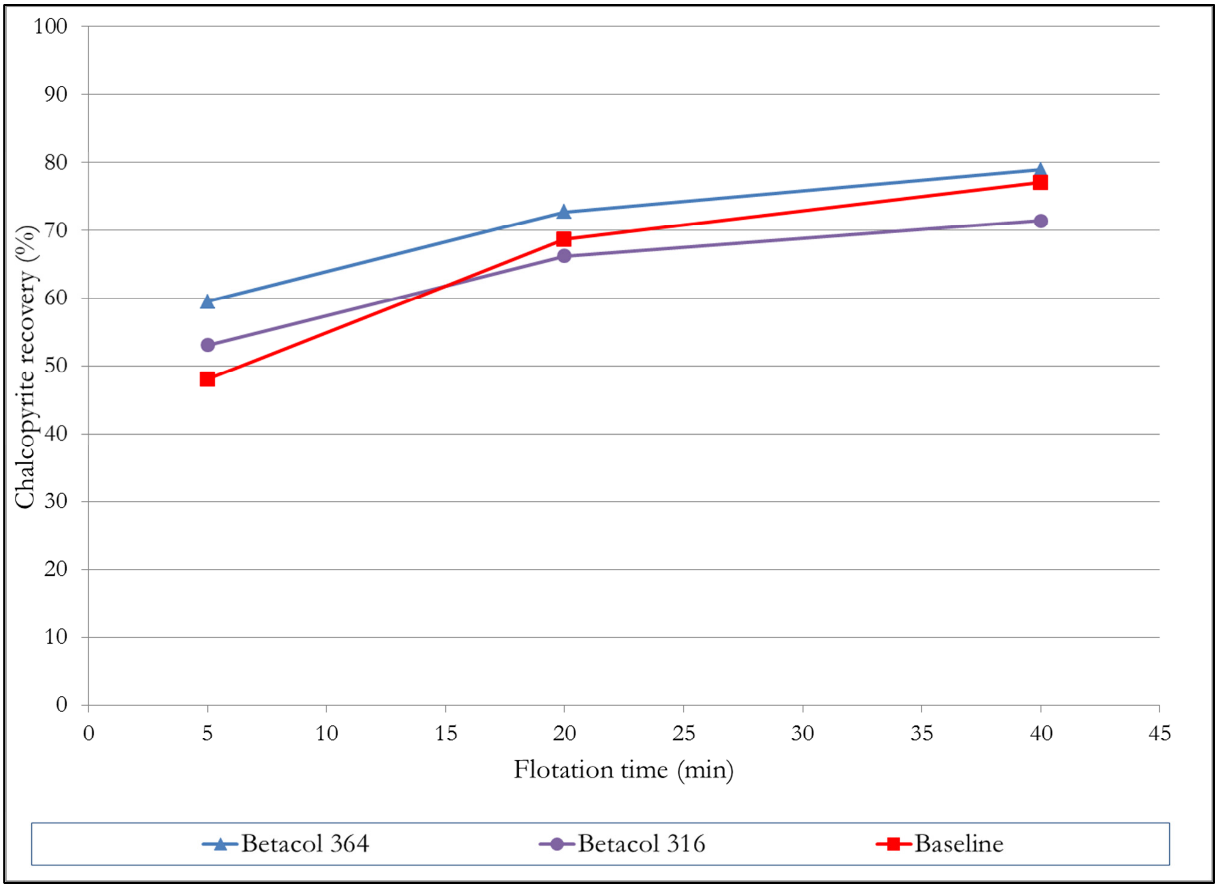


Figure 53: Chalcopyrite recovery and time plot for bench scale flotation test**

***Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Mineral identification and quantification by XRD.*

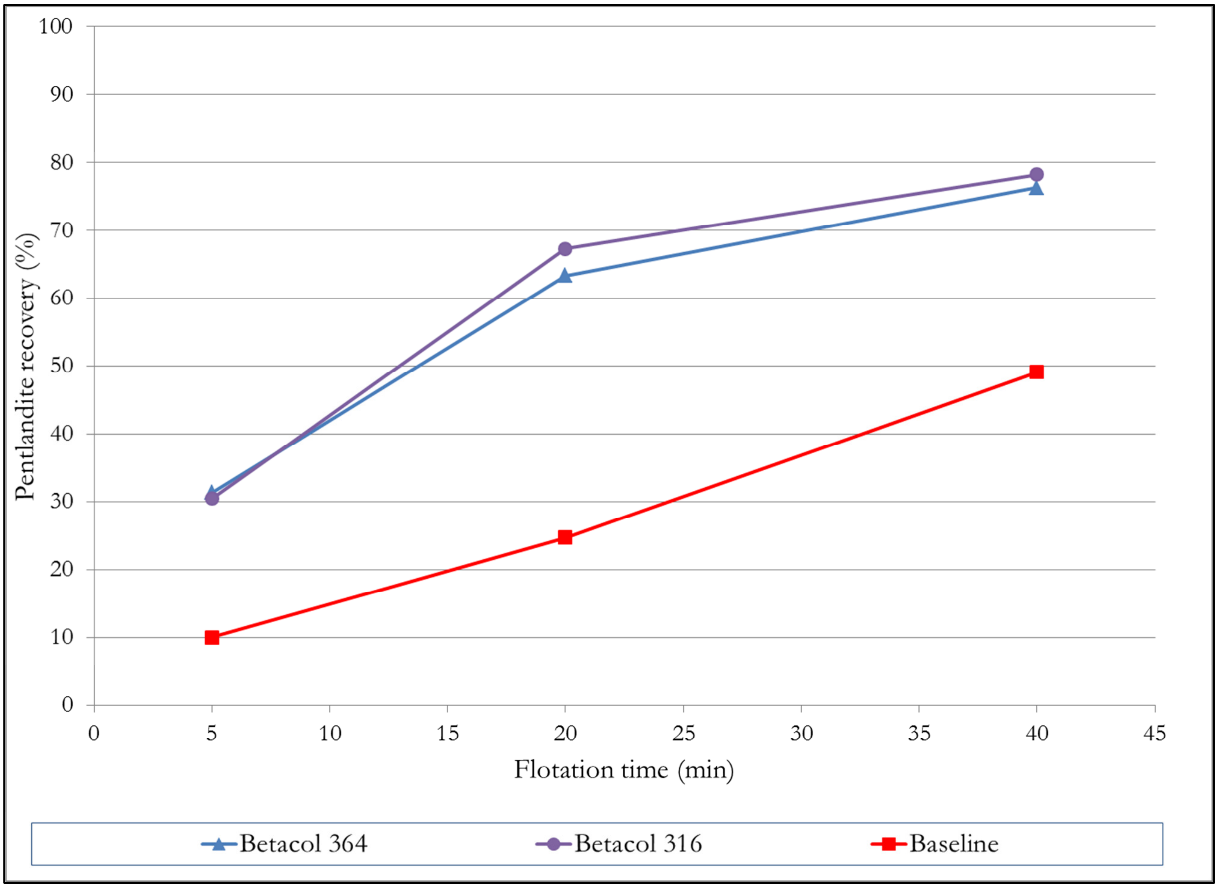


Figure 54: Pentlandite recovery and time plot for bench scale flotation test calculated from Cu, Ni and S assay values**

***Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; (SEX, 50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t.*

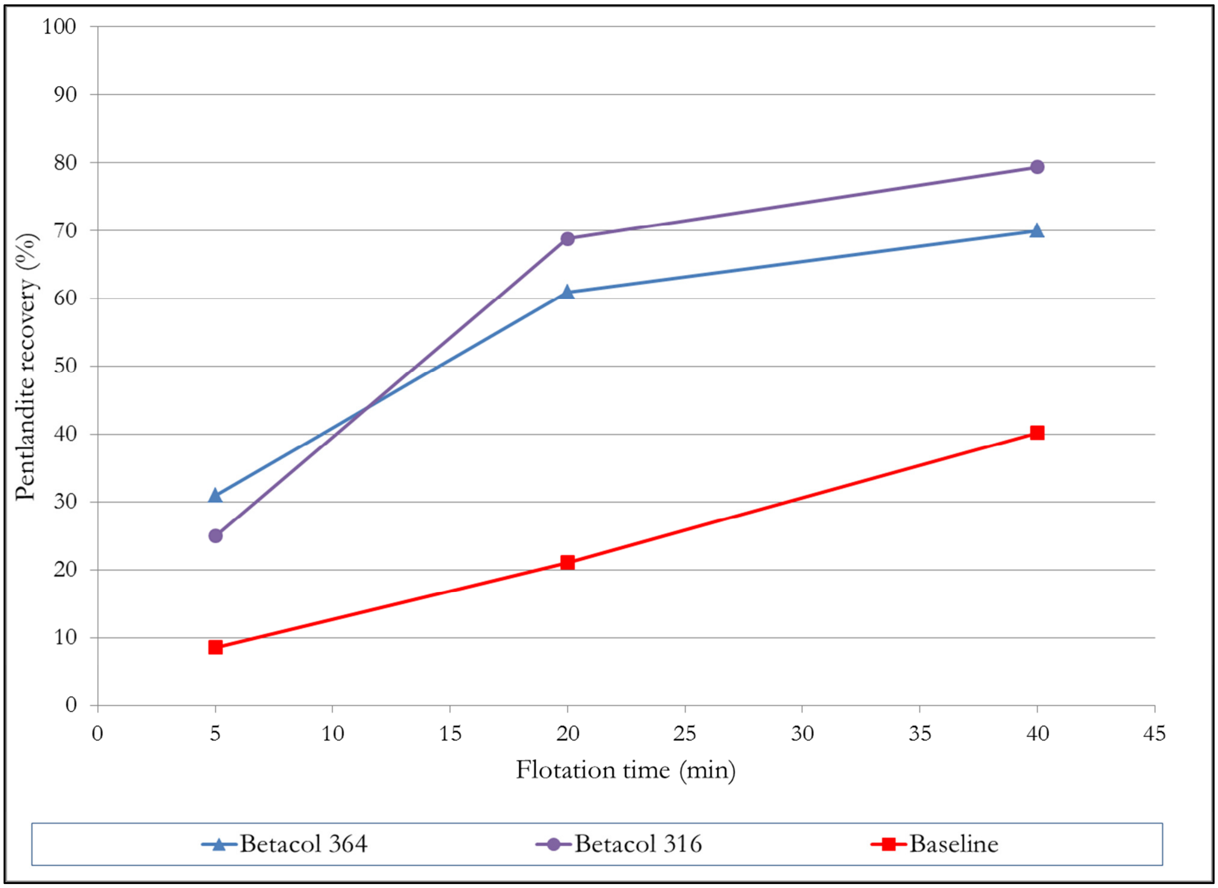


Figure 55: Pentlandite recovery and time plot for bench scale flotation test**

***Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Mineral identification and quantification by XRD.*

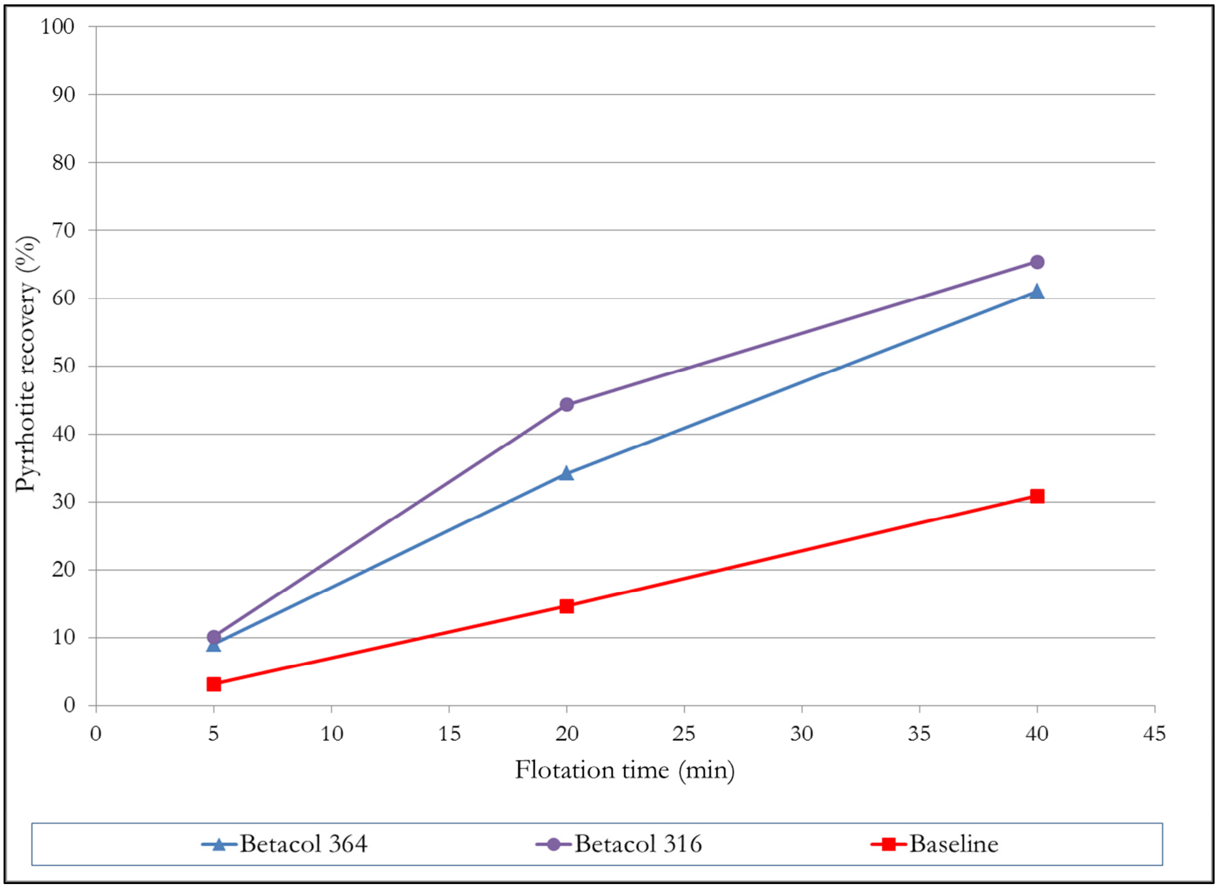


Figure 56: Pyrrhotite recovery and time plot for bench scale flotation test calculated from Cu, Ni and S assay values**

*** Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; (SEX, 50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t.*

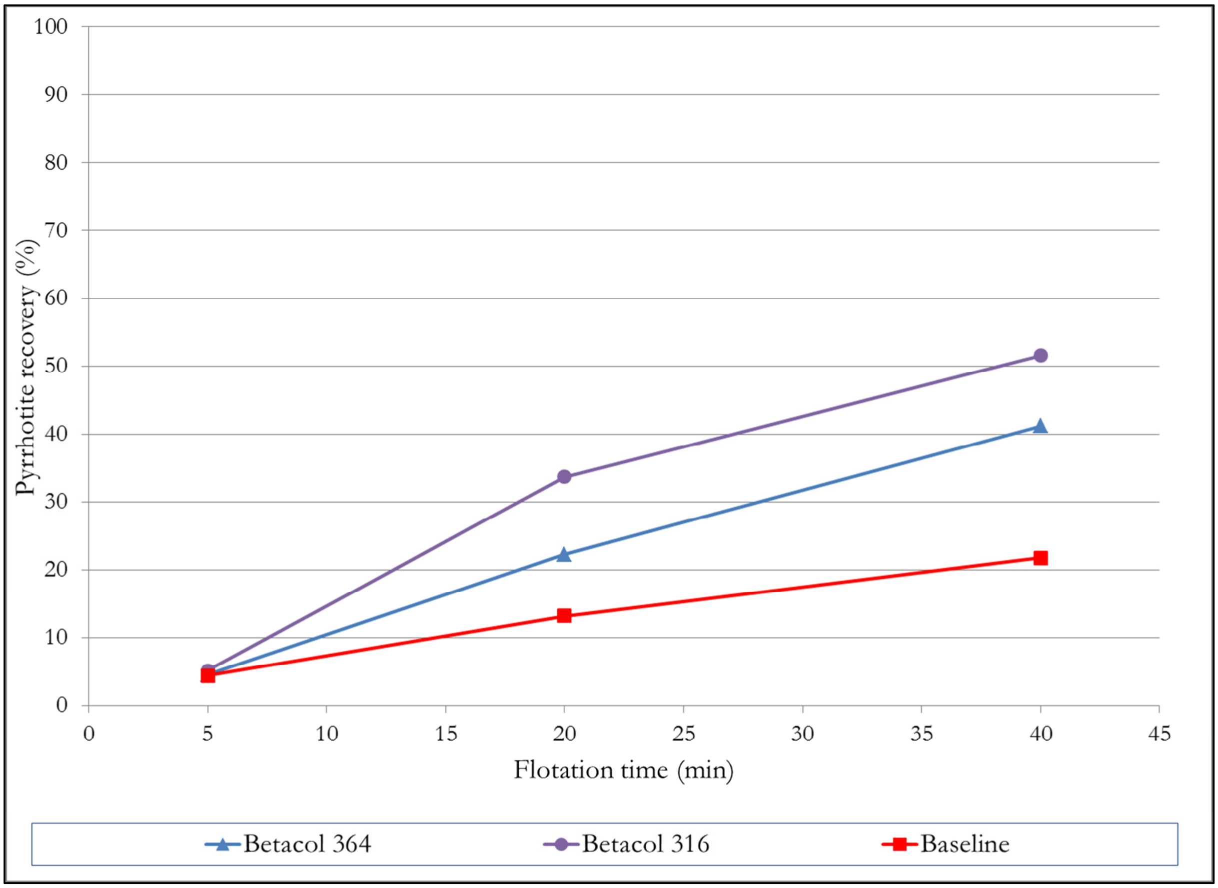


Figure 57: Pyrrhotite recovery and time plot for bench scale flotation test**

*** Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Mineral identification and quantification by XRD.*

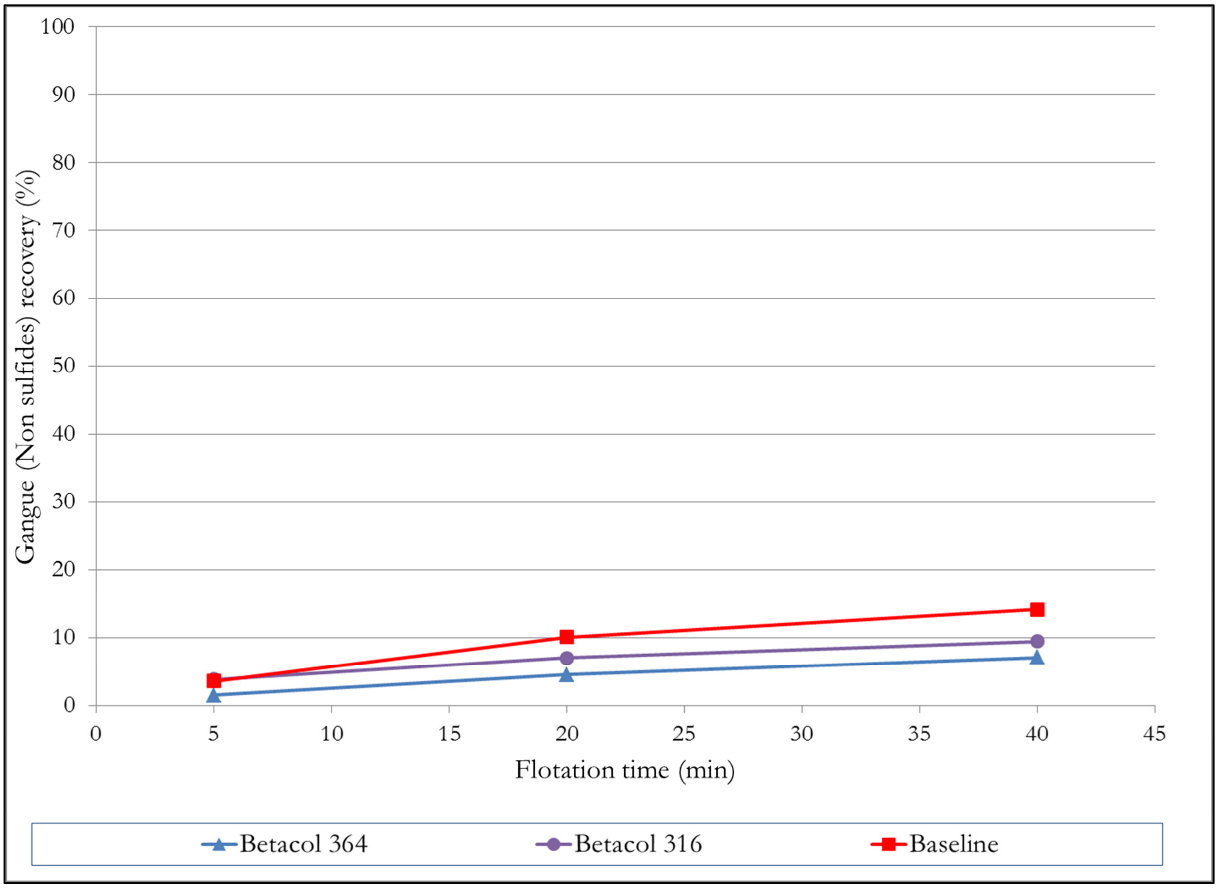


Figure 58: Gangue (non sulphides) recovery and time plot for bench scale flotation test calculated from Cu, Ni and S assay values**

*** Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; (SEX, 50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t.*

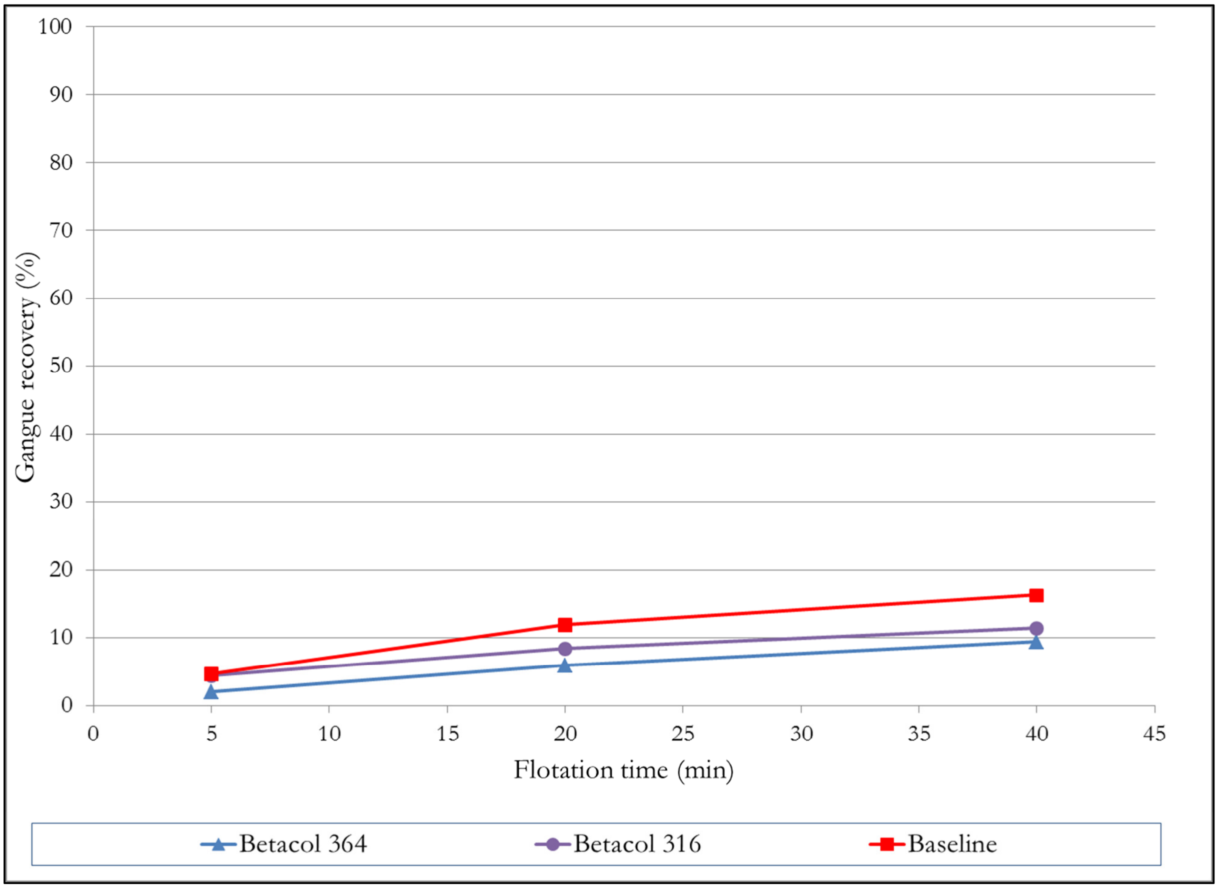


Figure 59: Gangue (non-sulphide) recovery and time plot for bench scale flotation test**

*** Two co-collectors were tested against baseline using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated at 20 g/t. Mineral identification and quantification by XRD.*

The laboratory scale batch flotation test done on an ore sample containing chalcopyrite, pentlandite and pyrrhotite as the predominant sulphide minerals supports the following conclusions:

- The use of the fatty acid based collector (Betacol 364) with xanthate had no effect on the rate of chalcopyrite recovery when compared to the baseline test where xanthate was used as sole collector.
- The use of the fatty acid based collector (Betacol 364) with xanthate increased the rate of pentlandite recovery when compared to the baseline test where xanthate was used as sole collector. This increase in the rate of flotation

recovery was not different to what was measured when a DTP and xanthate combination was tested.

- The use of the fatty acid based collector (Betacol 364) with xanthate increased the rate of pyrrhotite recovery when compared to the baseline test where xanthate was used as sole collector. This increase in rate of flotation recovery was lower than the rate of pyrrhotite recovery increase measured when a DTP and xanthate combination was tested.
- The use of the hydrocarbon based collector (Betacol 25) with xanthate decreased the rate of chalcopyrite recovery and had no effect on the pentlandite rate of recovery when compared to the baseline test where xanthate was used as sole collector.
- The rate of oxide gangue reduced when the fatty acid based collector (Betacol 364) was used in combination with xanthate compared to the baseline test where xanthate was used as sole collector.

The rate of mineral recovery can be expressed by Equation 5 and 6 (Gorain, et al., 1999):

$$k = P.S_b.R_f \tag{5}$$

$$S_b = \frac{6.J_g}{D_b} \tag{6}$$

Or as:

$$k = P.6.\frac{J_g}{D_b}.R_f \tag{7}$$

where k is the rate constant (min^{-1}), P is a parameter which represents the floatability of an ore, R_f is the froth recovery factor, S_b is the bubble surface area flux, J_g is the superficial gas velocity (m/sec) and D_b is the Sauter mean bubble size (m). In the laboratory scale batch flotation test care was taken to keep the superficial gas velocity (J_g) in all tests the same. An incorrect assumption can be made that if a change in the rate of mineral recovery is measured when a collector change is made during a laboratory scale test, then this will be as a result of a change in the particle floatability (P). Equation 7 suggests that this assumption cannot be made. If the collector tested has frothing characteristics, then the collector change could change the froth recovery (R_f) or the air bubble size in the pulp (S_b), and in this instance an increase in the rate of mineral recovery might be measured without the collector changing particle floatability at all.

If the rate of mineral recovery increase measured when a collector change is made is as a result of a change of the froth recovery and air bubble size, then an argument can be made that this change may be done more effectively or economically by changing the frother. The laboratory scale flotation test, in this instance, is not a reliable method to predict plant scale results. It can be concluded that if a change in the rate of mineral flotation is measured when a collector change is made, then the desired reason for this change is a change in particle floatability. In this instance the laboratory scale flotation test is a reliable method to predict plant scale results.

The conclusion is valid that Betacol 364 and Betacol 316, when used with xanthate, increase the rate of pentlandite and pyrrhotite recovery, but the data cannot conclude that this increase in the rate of recovery is a result of increasing pentlandite and pyrrhotite floatability. A further set of tests is therefore needed to test and confirm this.

5.2. Investigation into the effect a collector and co-collector combination has on froth characteristics

The rate of mineral recovery can be expressed by Equation 7 (Gorain, et al., 1999):

$$k = P.6.\frac{J_g}{D_b}.R_f \tag{7}$$

where k is the rate constant (min^{-1}), P is a parameter which represents the floatability of an ore, R_f is the froth recovery factor, J_g is the superficial gas velocity (m/sec) and D_b is the Sauter mean bubble size (m).

It is argued that if a laboratory scale batch flotation test indicates that a collector change results in a change in the rate of mineral recovery, then this change can be as a result of a change the collector has on the mineral floatability (P), or a change in froth recovery (R_f)or bubble size (D_b), or a combination of these factors. If a collector has froth stabilising or destabilising characteristics, then there is a higher probability that the collector has affected froth recovery (R_f) or bubble size (D_b) than if the collector has no effect on froth characteristics.

It can then be argued that if the effect a collector change has on froth stability could be measured, and it is measured that the collector change *does not change* froth stability, then a batch flotation test is sufficient to conclude that the collector change affects mineral floatability. An increase in the measured rate of mineral recovery resulting from a collector change, which can be attributed to a change in mineral floatability, has a high probability to be reproduced on plant scale, and can then be recommended for commercial testing.

It can also be argued that if the effect a collector change has on froth stability can be measured and it is measured that the collector change *does change* froth stability, then a batch flotation test is not sufficient to conclude that the collector change affects mineral floatability only. In this instance, the collector change may also affect froth recovery (R_f) and bubble size (D_b). A change in the rate of mineral recovery when a collector is changed may in this instance be done more effectively or economically with a change in frother. In this instance the collector change has a low probability of being reproduced on plant scale, where operation algorithms often control froth stability with frother dosage, pulp interface level or air flow rate.

A test where the effect a collector change has on froth stability therefore has value together with the batch flotation test in a collector test program. A test unit was designed for this purpose based on the Bikerman test used to measure froth stability of detergents (Bikerman, 1973). The design is discussed in Section 4.3.

The equilibrium froth high measured at increasing dosing rates of the fatty acid based collector (Betacol 364) is shown in **Figure 60**, using the same ore and reagent combination tested in the preceding section (Section 5.1). The fatty acid based collector (Betacol 364) has froth stabilising characteristics in the dosing range tested in the preceding section. The increase in the rate of flotation measured for pentlandite and pyrrhotite can therefore not be concluded to be only as a result of a change in pentlandite or pyrrhotite floatability.

The two DTP based collectors Betacol 316 and 44A also have froth stabilising characteristics. These products are frothier than the fatty acid based collector (Betacol 364). The increase in the rate of flotation measured for pentlandite and pyrrhotite can thus not be concluded to be as result of a change in pentlandite or pyrrhotite floatability only.

The hydrocarbon collector (Betacol 25) tested has a froth destabilising effect on the flotation froth. It may therefore be possible that the use of Betacol 25 with xanthate may reduce the froth recovery (R_f) and bubble size (D_b), and may result in a decrease in the measured rate of recovery in a batch flotation test. The net effect of an increase in particle floatability with a decrease in froth recovery and bubble size may be the reason a decrease in the chalcopyrite rate of recovery is measured and has no effect on the pentlandite rate of recovery.

The conclusions drawn from the froth stability tests are that Betacol 364, Betacol 316 and Betacol 44A have froth *stabilising* characteristics and Betacol 25 has froth *destabilising* characteristics. The rate of flotation increase or decrease measured with the batch flotation test cannot be attributed to a change in mineral floatability only. It can be argued that another set of tests needs to be performed to confirm if the collector change affects particle floatability. This should be done before the commercial use of any of these collectors can be recommended.

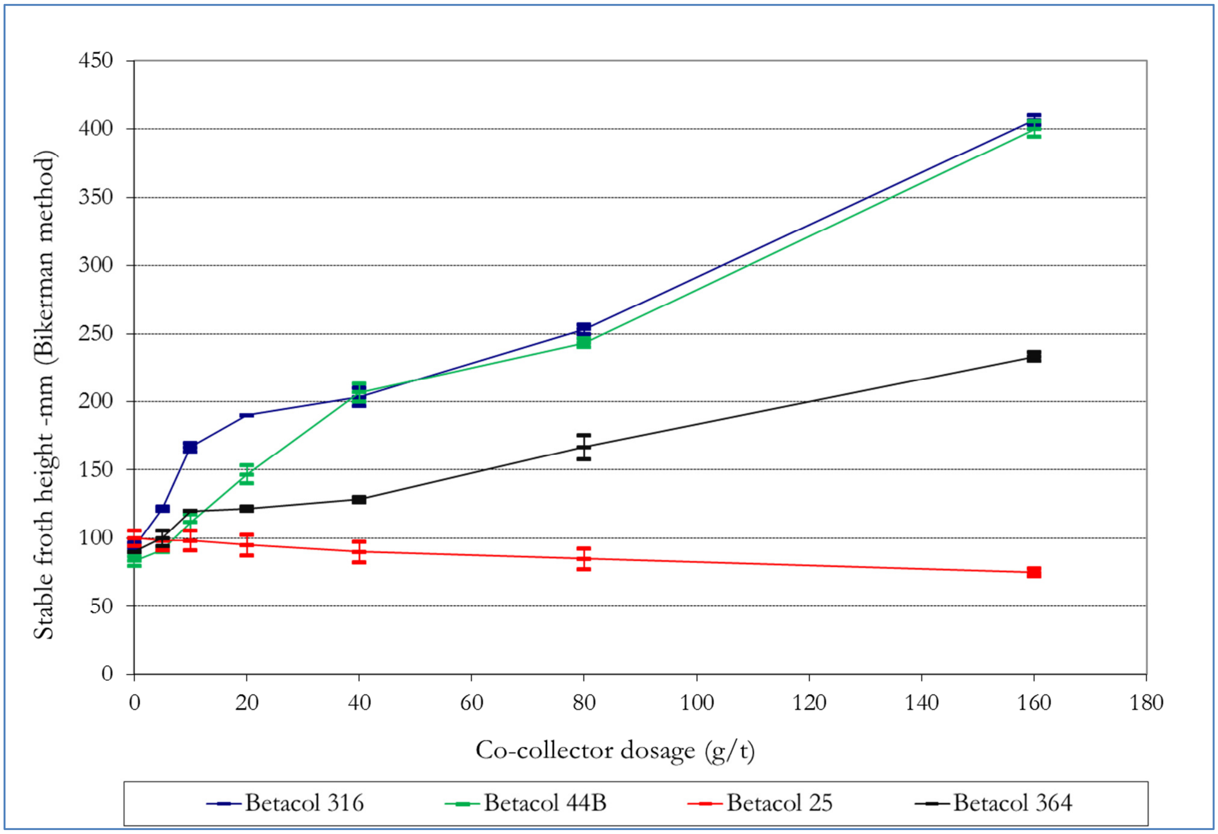


Figure 60: Stable froth height measured using the Bikerman froth stability column**

*** South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother: Betafroth 436B (90 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated. Error bars represent standard error.*

Although not directly related to the research problem statement, work was done to test the effect a number of commercial xanthates has on froth stability. Four commercial xanthates from laboratory stock were selected and the effect an increase in dosage has on froth stability was measured using the Bikerman froth stability column (**Figure 61**). This set of tests was repeated on the same xanthate samples, but only after it had been purified by the re-precipitation method described in appendix B Section 8.1 (**Figure 62**). The activity of the xanthate samples used was measured before and after purification, using the phenolphthalein titration method described in appendix B Section 8.2. This data is listed in **Table 21**.

The type of xanthate used and its activity has a significant influence on the froth stability for the ore sample used in this test set. If a laboratory scale batch test was conducted and it was found that a change to potassium amyl xanthate (PAX) from sodium ethyl xanthate (SEX) resulted in an increase in the rate of mineral recovery, care should be taken not to attribute this to a change in mineral floatability only. An additional set of tests would be recommended to confirm if the increase in the rate of recovery is as a result of an increase in mineral floatability before commercial testing is recommended.

Table 21: Xanthate activity measured using the phenolphthalein titration method (Section 8) for the four xanthates used for Bikerman froth stability column test work before and after purification by re-precipitation (Section 8).

Commercial Xanthate	% Activity	Purified Xanthate	% Activity
SEX	72	P-SEX	86
SIPX	53	P-SIPX	93
SIBX	56	P-SIBX	86
PAX	49	P-PAX	86

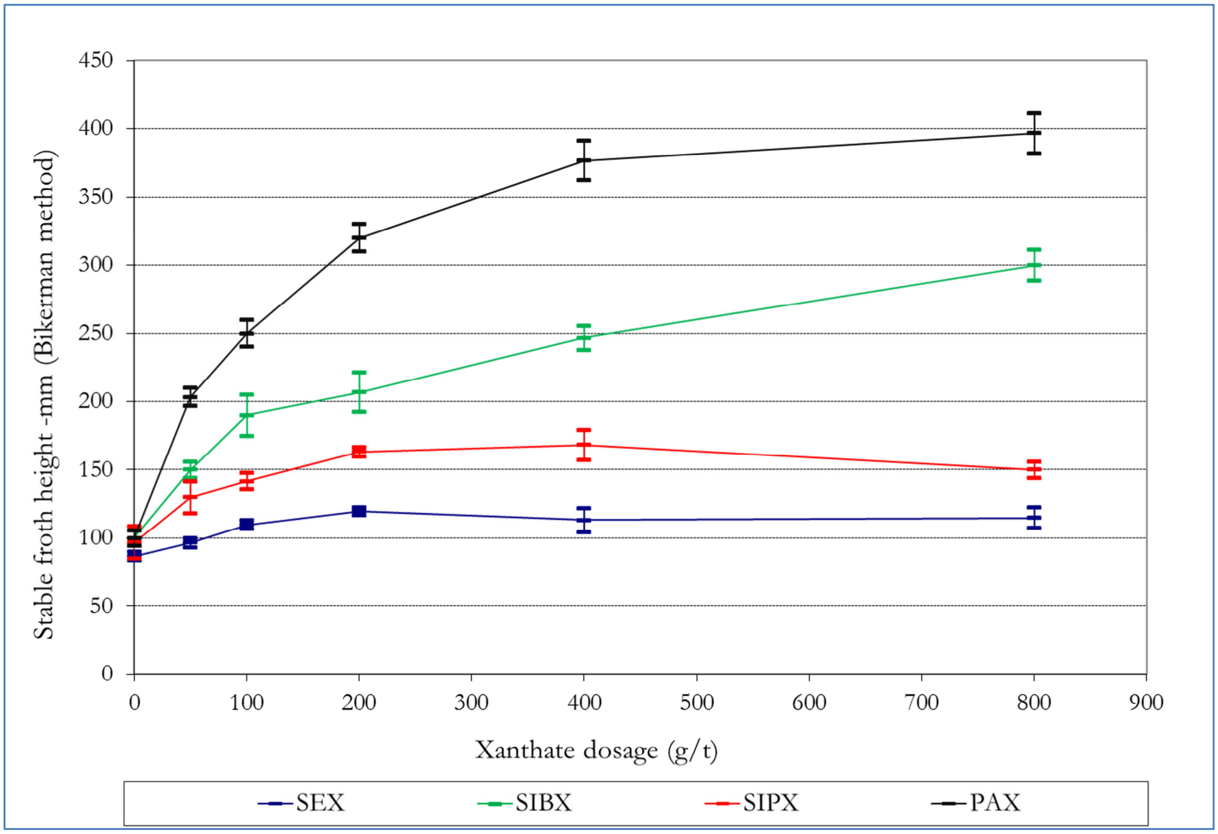


Figure 61: Stable froth height measured using the Bikerman froth stability column**

*** South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Baseline conditions: Betafroth 436B (90 g/t), Depressant; Betamin 544 (250 g/t). Commercial grade xanthates as indicated. Error bars represent standard error.*

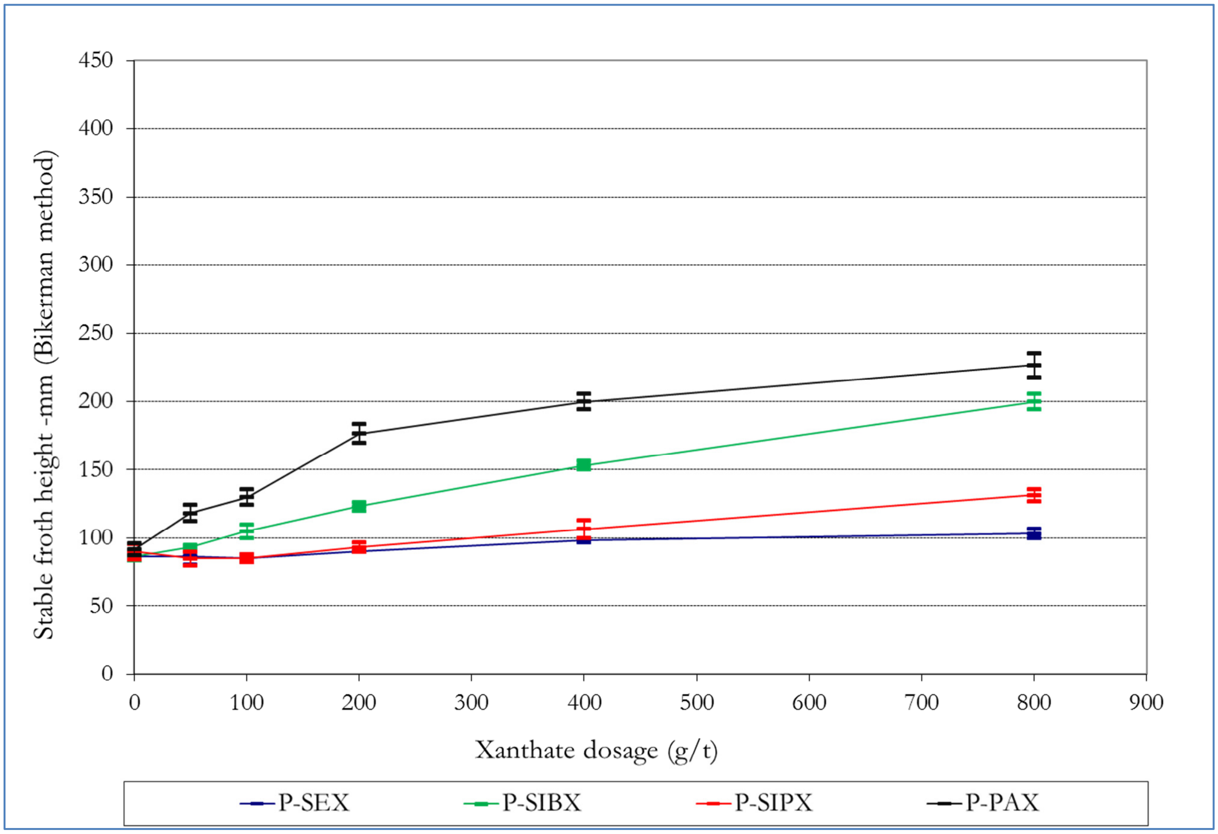


Figure 62: Stable froth height measured using the Bikerman froth stability column**

***South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Baseline conditions: Betafroth 436B (90 g/t), Depressant; Betamin 544 (250 g/t). Xanthates purified by precipitation. Dosage and type as indicated. Error bars represent standard error*

5.3. Laboratory scale batch flotation test using a synthetic base metal sulphide ore consisting of quartz and chalcopyrite or pyrrhotite

The rate of mineral recovery in a flotation process is dependent on the floatability of a mineral (P), the froth recovery (R_f), the superficial gas velocity (J_g)and on the Sauter mean bubble size (D_b) according to Equation 7 (Gorain, et al., 1999):

$$k = P.6.\frac{J_g}{D_b}.R_f \qquad (7)$$

It has been argued that if a collector change has no effect on froth characteristics, then it can be assumed that the froth recovery and bubble size in a batch flotation test will be unaffected by the collector change. If the air flow rate is kept constant and the same test equipment is used (constant superficial gas velocity), and a change is then measured in the rate of mineral recovery in a laboratory scale batch flotation test, it can be concluded that the collector change changed the mineral floatability.

If the collector change changes the flotation froth characteristics, then the laboratory scale batch flotation test cannot be used to conclude that the rate of recovery is as a result of a change in mineral floatability only. It may be that the rate of flotation is as a result of a change in froth recovery or bubble size. In this instance another set of tests is recommended to measure the effect a collector change has on mineral floatability.

A set of laboratory scale batch flotation tests were done using two synthetic ore types. The first ore type was a mixture of 90% quartz and 10% chalcopyrite. The second was a mixture of 90% quartz and 10% pyrrhotite. The main advantage of using a synthetic ore, is that this ore does not contain froth stabilising gangue. The batch flotation test can be performed with a very shallow froth depth and with a high frother concentration. The aim of this method is to reduce the effect a change in collector has on the froth recovery and bubble size. The test method is described in Section 4.2.1.

The rate of chalcopyrite recovery increased marginally when the fatty acid collector was used in combination with xanthate, compared to the baseline test where xanthate was used as sole collector (**Figure 63**). A similar increase in the rate of recovery was measured when Betacol 44A (DTP) and Betacol 25 (hydrocarbon) were used in combination with xanthate.

For all these tests, the rate of quartz recovery remained unchanged (**Figure 64**).

The rate of pyrrhotite recovery increased significantly when the fatty acid collector was used in combination with xanthate, compared to the baseline test where xanthate was used as only collector (**Figure 65**). A similar increase in the rate of recovery was measured when Betacol 25 (hydrocarbon) was used in combination with xanthate. For the test where Betacol 316 (DTP) and Betacol 44A (DTP) were used in combination with xanthate an increase in the rate of pyrrhotite recovery was also measured compared to the baseline test, but this rate was lower than with (Betacol 364) (fatty acid) and (Betacol 25) (hydrocarbon).

For all these tests, the rate of quartz recovery remained unchanged (**Figure 66**).

The rate of recovery change when Betacol 25 (hydrocarbon) was used in combination with xanthate produced interesting, but not unexpected results. In this set of tests, the use of Betacol 25 with xanthate resulted in a marginal increase in the rate of chalcopyrite recovery. When the same set of tests was performed on the base metal sulphide ore, (Section 5.1), the rate of recovery for chalcopyrite decreased significantly. A possible explanation for these anomalous results is that Betacol 25 has been shown to decrease froth stability in Section 5.2. The rate of recovery for laboratory scale batch flotation tests can be expected to be influenced by particle floatability, froth recovery and bubble size according to Equation 7. If the assumption is valid that with the synthetic ore, the influence of froth recovery and bubble size is reduced by the shallow froth depth and high frother concentration, then it would be expected that for this test a decrease in the rate of recovery would not be measured. This was indeed the outcome of the test. It can then tentatively be concluded that the synthetic ore test assumption that the effect of froth recovery and bubble size was reduced or eliminated, is valid. The use of a synthetic ore may therefore be a practical test to supplement a test program when a collector with a frothy nature is tested.

If it is assumed that this test method reduces the effect of a frothy collector sufficiently to make the rate of recovery measurement independent of froth recovery and bubble size, then it can be concluded that the fatty acid based collector (Betacol 364), in combination with xanthate, increases the rate of pyrrhotite flotation significantly, but only marginally increases the rate of chalcopyrite recovery. There is a high probability that the increase in the rate of pyrrhotite recovery is as a result of an increase in the mineral floatability, and commercial testing can be recommended.

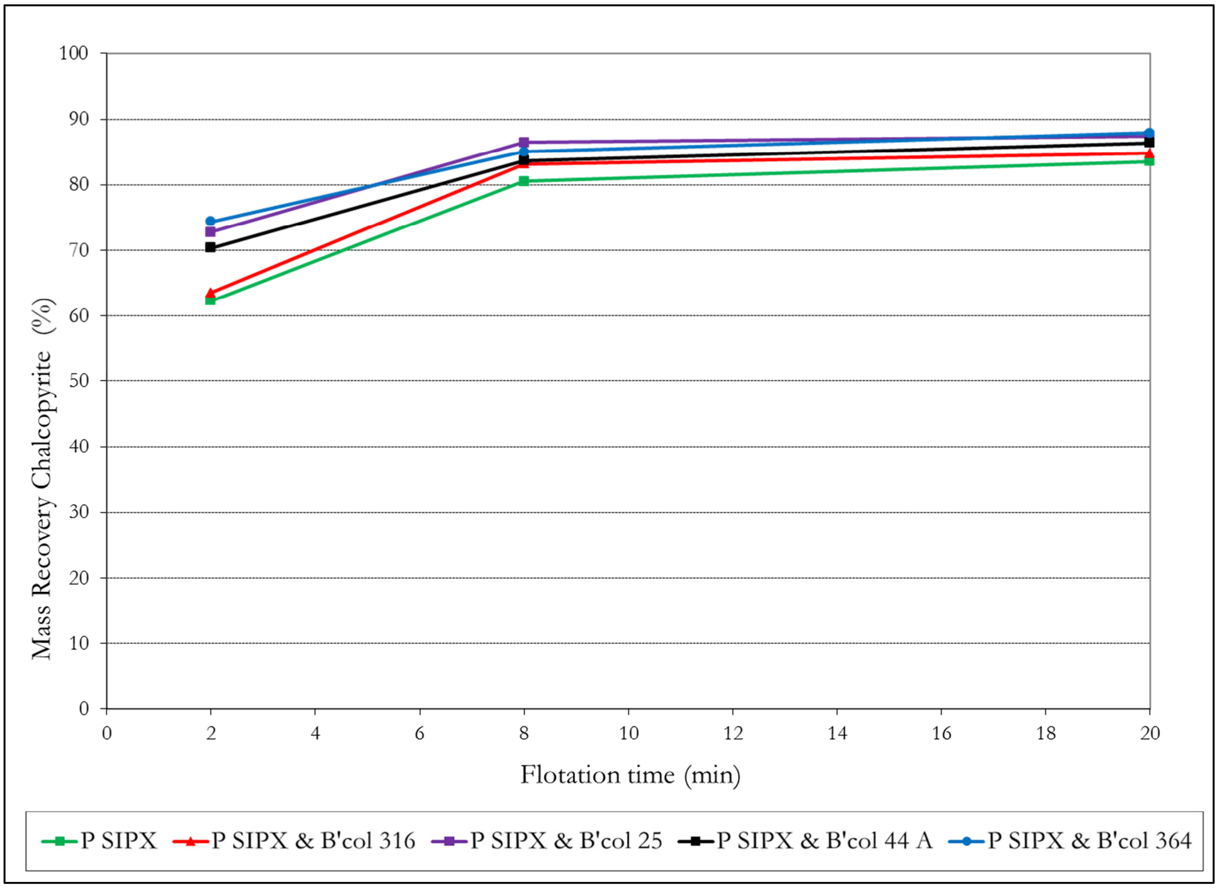


Figure 63: Bench flotation test using D12 equipment. Ore tested is a lab mixture of 90% quartz and 10% chalcopyrite**.

***Lab milled to 70% passing 100 micron. SIPX (purified by re-crystallisation) added at 50 g/t. Co-collectors added at 50 g/t (type as indicated). Frother: Betafroth 466 at 100 g/t. Mineral recovery data calculated from Cu assay by AAS.*

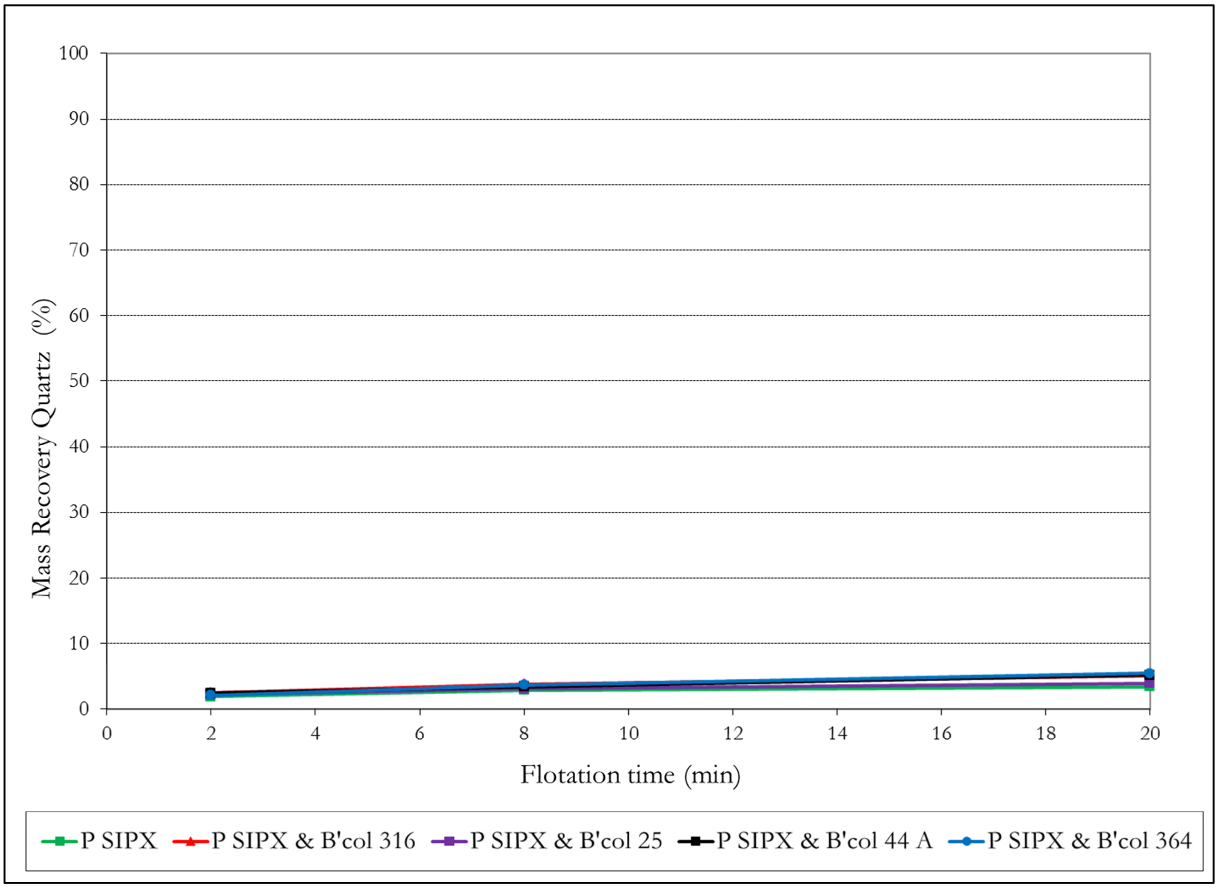


Figure 64: Bench flotation test using D12 equipment. Ore tested is a lab mixture of 90% quartz and 10% chalcopyrite**.

***Lab milled to 70% passing 100 micron. SIPX (purified by re-crystallisation) added at 50 g/t. Co-collectors added at 50 g/t (type as indicated). Frother: Betafroth 466 at 100 g/t. Mineral recovery data calculated from Cu assay by AAS.*

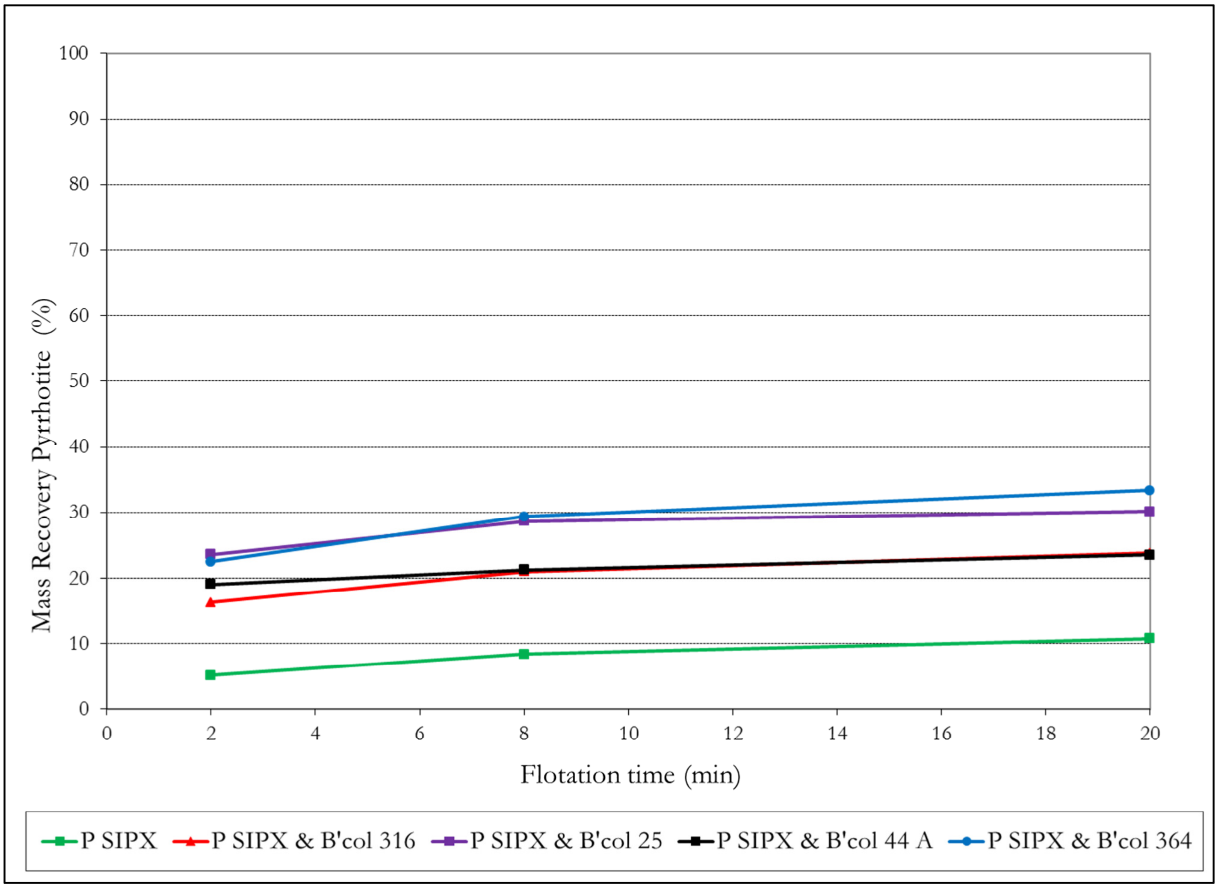


Figure 65: Bench flotation test using D12 equipment. Ore tested is a lab mixture of 90% quartz and 10% pyrrhotite**.

*** Lab milled to 70% passing 100 micron. SIPX (purified by re-crystallisation) added at 50 g/t. Co-collectors added at 50 g/t (type as indicated). Frother: Betafroth 466 at 100 g/t. Mineral recovery data calculated from Fe assay by AAS.*

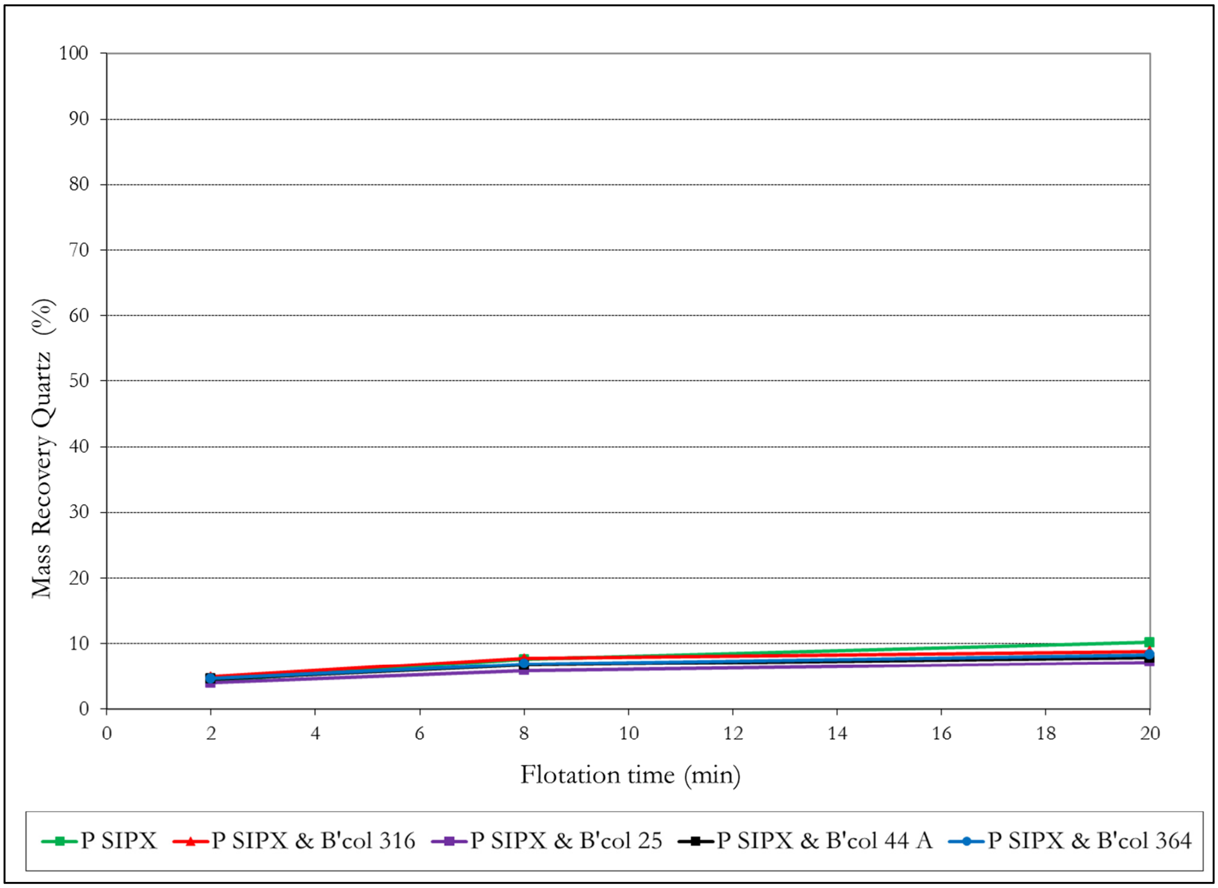


Figure 66: Bench flotation test using D12 equipment. Ore tested is a lab mixture of 90% quartz and 10% pyrrhotite**.

***Lab milled to 70% passing 100 micron. SIPX (purified by re-crystallisation) added at 50 g/t. Co-collectors added at 50 g/t (type as indicated). Frother: Betafroth 466 at 100 g/t. Mineral recovery data calculated from Fe assay by AAS.*

5.4. Investigation into the floatability of chalcopyrite and pyrrhotite with xanthate and co-collectors as flotation collectors using a micro flotation technique

The rate of mineral recovery in a flotation process is dependent on the floatability of a mineral (P), the froth recovery (R_f), the superficial gas velocity (J_g) and on the Sauter mean bubble size (D_b), according to Equation 7 (Gorain, et al., 1999):

$$k = P.6.\frac{J_g}{D_b}.R_f \tag{7}$$

It has been argued that if a collector change has no effect on froth characteristics, then it can be assumed that the froth recovery and bubble size in a batch flotation test will be unaffected by the collector change. If the air flow rate is kept constant and the same test equipment is used (constant superficial gas velocity), and a change is then measured in the rate of a mineral recovery in a laboratory scale batch flotation test, it can be concluded that the collector change changed the mineral floatability.

If the collector change changes the flotation froth characteristics, then the laboratory scale batch flotation test cannot be used to conclude that the rate of recovery is as a result of a change in mineral floatability only. It may be that the rate of flotation is as a result of a change in froth recovery or bubble size. In this instance another set of tests is recommended to measure the effect a collector change has on mineral floatability.

A micro flotation test has been used to measure the rate of mineral flotation (Venter, 2009) (Vermaak, 2005) and (Bradshaw & O'Conner, 2006). A micro flotation test unit was designed and used to test the rate of flotation of chalcopyrite and pyrrhotite mineral particles. In the design of the micro flotation test unit, the aim was to simulate the natural flotation process as closely as possible, and reduce the effect of froth recovery and bubble size in the measured rate of flotation (Section 4.4.1). If the test unit design achieved these criteria, a change in the measured rate of flotation would indicate a change in the particle floatability.

To eliminate or reduce the effect froth recovery has on the micro flotation test results, the unit was designed to operate with a constant liquid overflow, thus no froth interface. The disadvantage of this is that particles are recovered via an entrainment mechanism. The rate of quartz recovery tested using the bubble flux micro flotation unit is shown in **Figure 67**. Recovery via an entrainment mechanism is significant for this test method, and has to be considered when interpreting the rate of flotation data.

Chalcopyrite milled before the test, (fresh surface,) floats fast and effectively in the test unit (**Figure 68**). The addition of xanthate, irrespective of the type, results in an increase in the rate of recovery and it is concluded that xanthate increases the floatability of chalcopyrite. It is interesting to note that when the same set of tests is performed on chalcopyrite milled and aged for 14 days in air, the rate of flotation increases significantly, compared to the same test performed on a freshly milled sample that is unaffected by the addition of xanthate. This is an indication of collectorless flotation. Rao attributes the collectorless flotation of chalcopyrite to the formation of a hydrophobic sulphur film on the chalcopyrite surface by surface oxidation (Rao, 2004). This was not studied further.

The rate of pyrrhotite flotation is low with xanthate as sole collector, irrespective of the xanthate type (**Figure 70**). When the test was repeated for pyrrhotite particles milled and aged in air for 14 days, the rate of flotation increased significantly (**Figure 71**), but remained unaffected by the addition of xanthate.

It is concluded that xanthate increases the floatability of chalcopyrite, but not the floatability of pyrrhotite under the conditions measured.

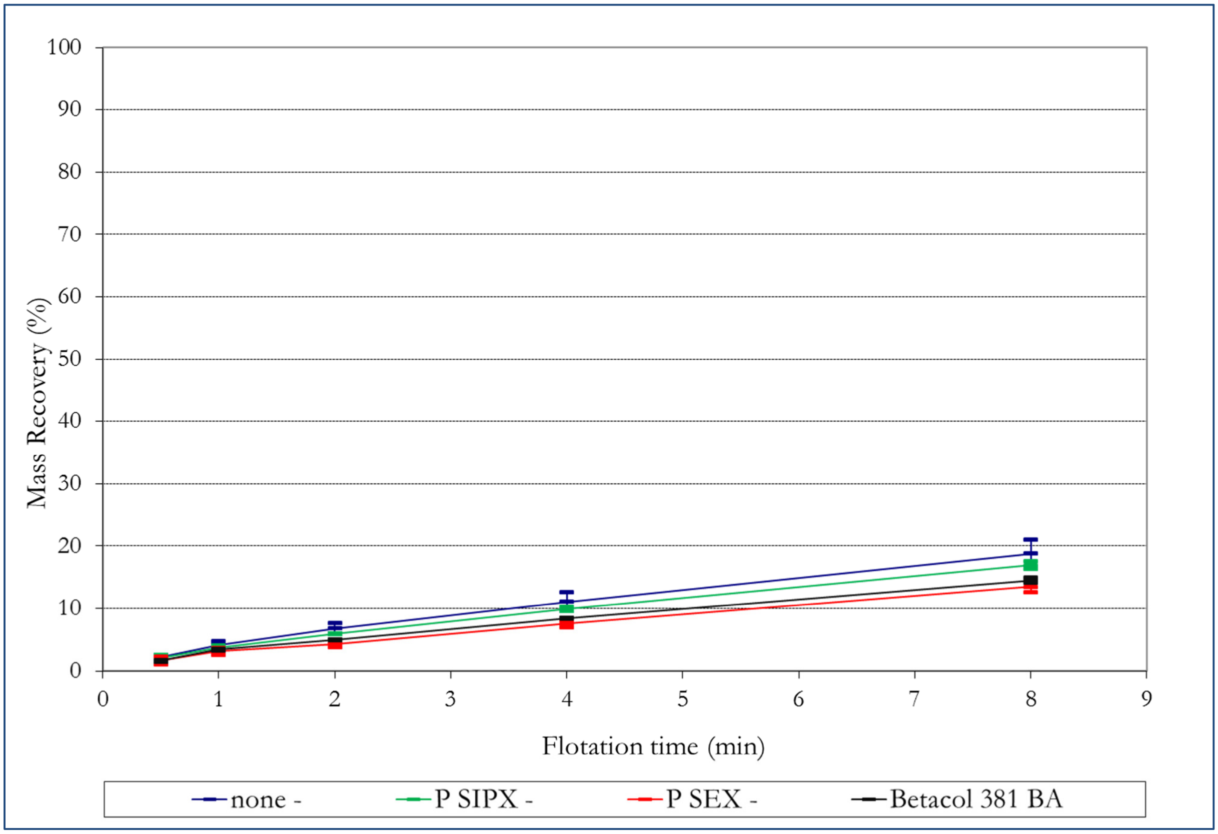


Figure 67: Micro flotation test results using micro-bubble flux micro flotation equipment. Quartz ore specimen dry milled to -106 ,+ 75 micron and left in air for 14 days before flotation (aged surface)**.

*** Distilled water with added ions to simulate plant water used. Air flow rate equivalent to a superficial gas velocity of Jg=0.24 cm/s (0.1 l/min). Xanthate added at 50 g/t and type as indicated. Error bars represent standard error calculated for replica tests.*

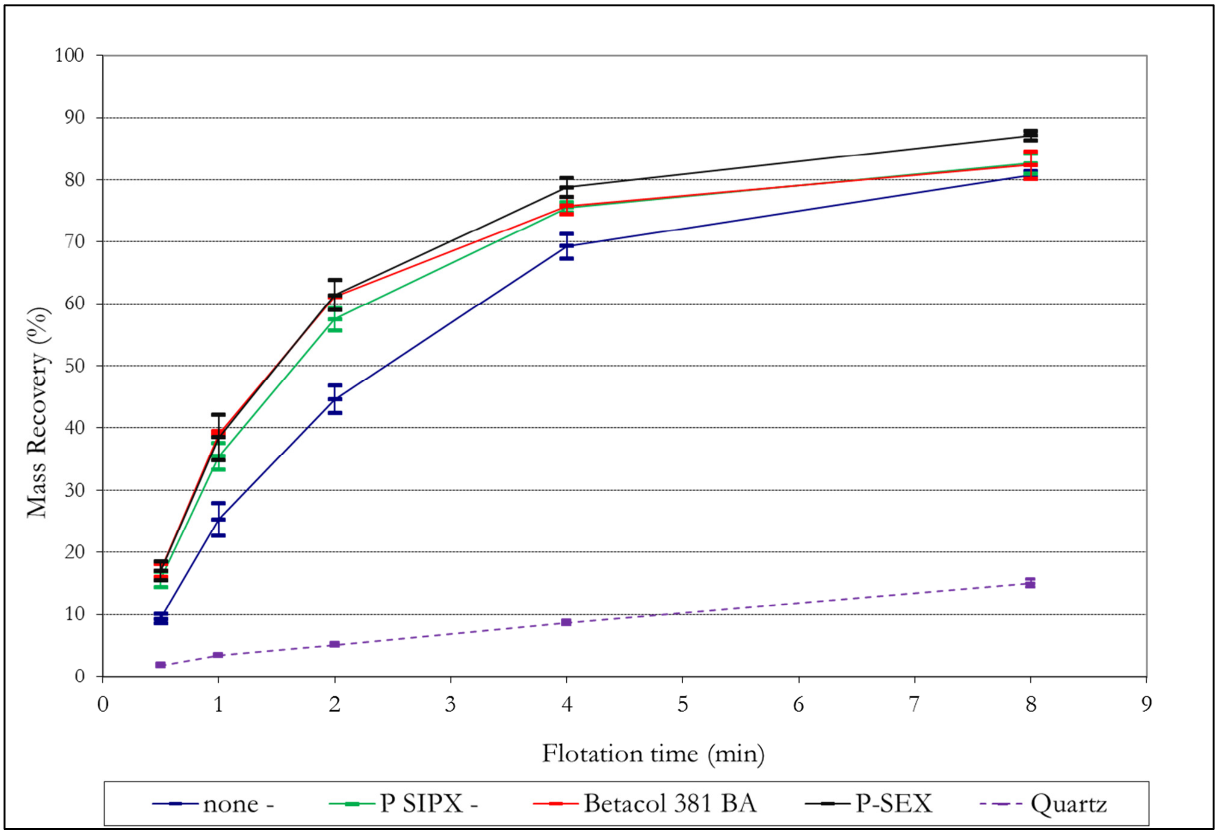


Figure 68: Micro flotation test results using micro-bubble flux micro flotation equipment. Chalcopyrite ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**.

***Distilled water with added ions to simulate plant water used. Air flow rate equivalent to a superficial gas velocity of Jg=0.24 cm/s (0.1 l/min). Xanthate added at 50 g/t and type as indicated. Error bars represent standard error calculated for replica tests.*

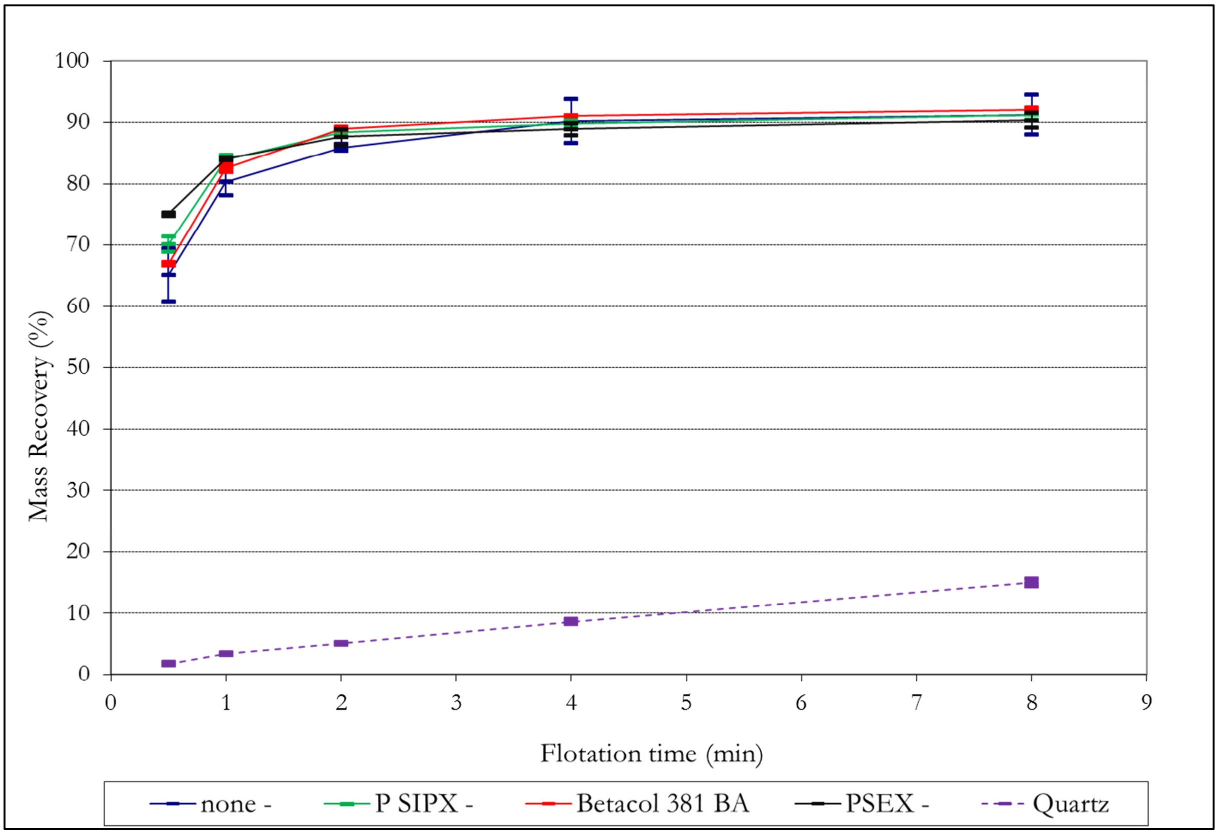


Figure 69: Micro flotation test results using micro-bubble flux micro flotation equipment. Chalcopyrite ore specimen dry milled to -106 ,+ 75 micron and left in air for 14 days before flotation (aged surface)**.

*** Distilled water with added ions to simulate plant water used. Air flow rate equivalent to a superficial gas velocity of Jg=0.24 cm/s (0.1 l/min). Xanthate added at 50 g/t and type as indicated. Error bars represent standard error calculated for replica tests.*

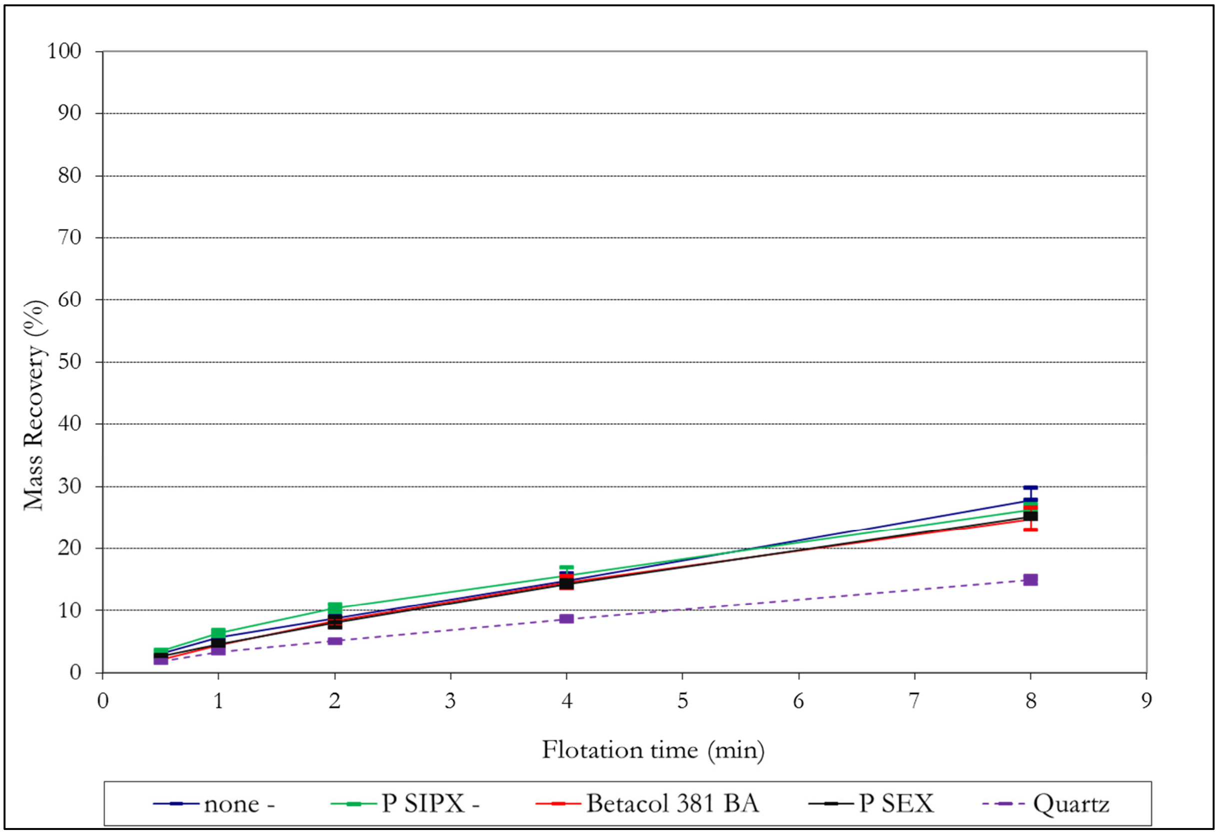


Figure 70: Micro flotation test results using micro-bubble flux micro flotation equipment. Pyrrhotite ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**.

*** Distilled water with added ions to simulate plant water used. Air flow rate equivalent to a superficial gas velocity of Jg=0.24 cm/s (0.1 l/min). Xanthate added at 50 g/t and type as indicated. Error bars represent standard error calculated for replica tests.*

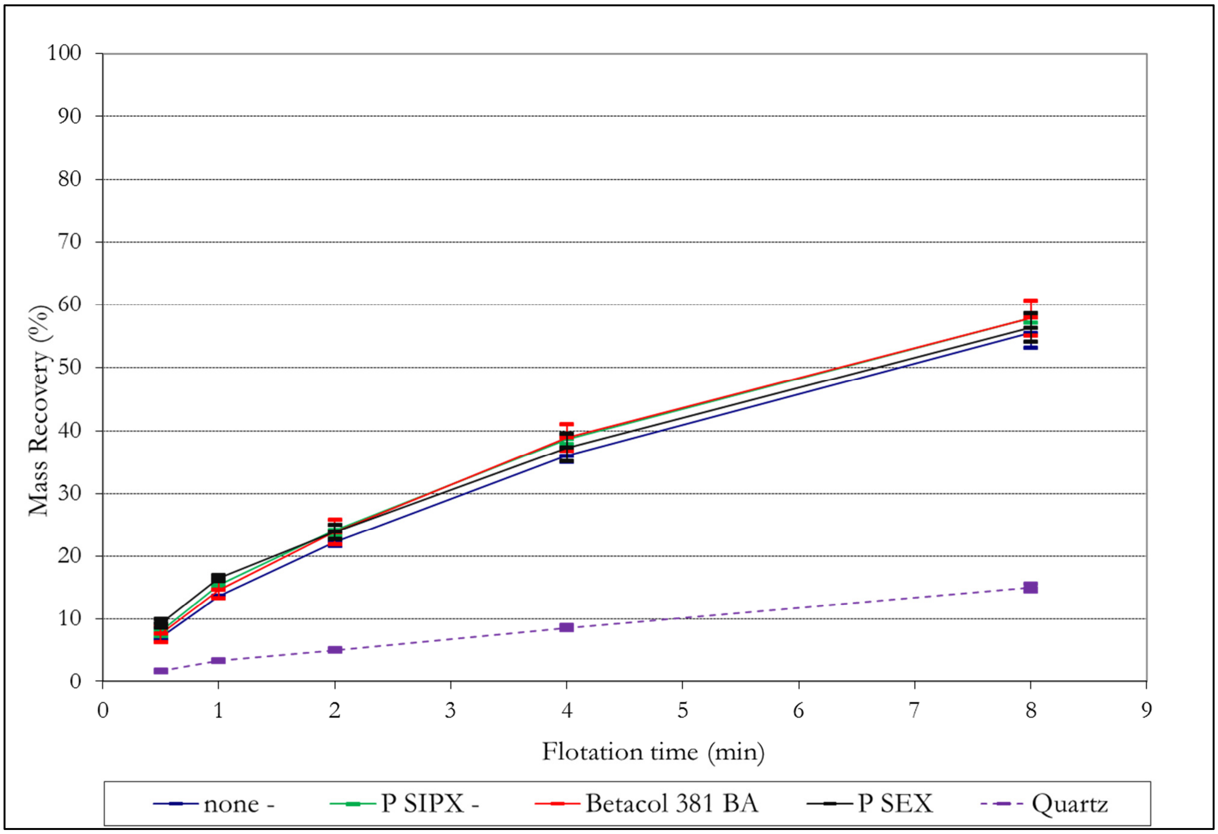


Figure 71: Micro flotation test results using micro-bubble flux micro flotation equipment. Pyrrhotite ore specimen dry milled to -106 ,+ 75 micron and left in air for 14 days before flotation (aged surface)**.

*** Distilled water with added ions to simulate plant water used. Air flow rate equivalent to a superficial gas velocity of Jg=0.24 cm/s (0.1 l/min). Xanthate added at 50 g/t and type as indicated. Error bars represent standard error calculated for replica tests.*

The rate of pyrrhotite recovery when the fatty acid based collector (Betacol 364) was used in combination with xanthate was measured to be significantly higher than the baseline test where xanthate was used as sole collector (**Figure 72**). The rate of pyrrhotite recovery when Betacol 364 was used with xanthate was not significantly different than when either of the DTP collectors Betacol 316 and Betacol 44A was used in combination with xanthate.

The rate of quartz recovery (**Figure 73**) was lower when Betacol 364 was used in combination with xanthate compared to the baseline test.

If the assumption is valid that the micro flotation test equipment and procedure used reduces or eliminates the effect froth recovery and bubble size has on the rate of mineral recovery, then it can be concluded that the use of Betacol 364 in combination with xanthate increases the floatability of pyrrhotite and it can therefore be recommended for commercial testing.

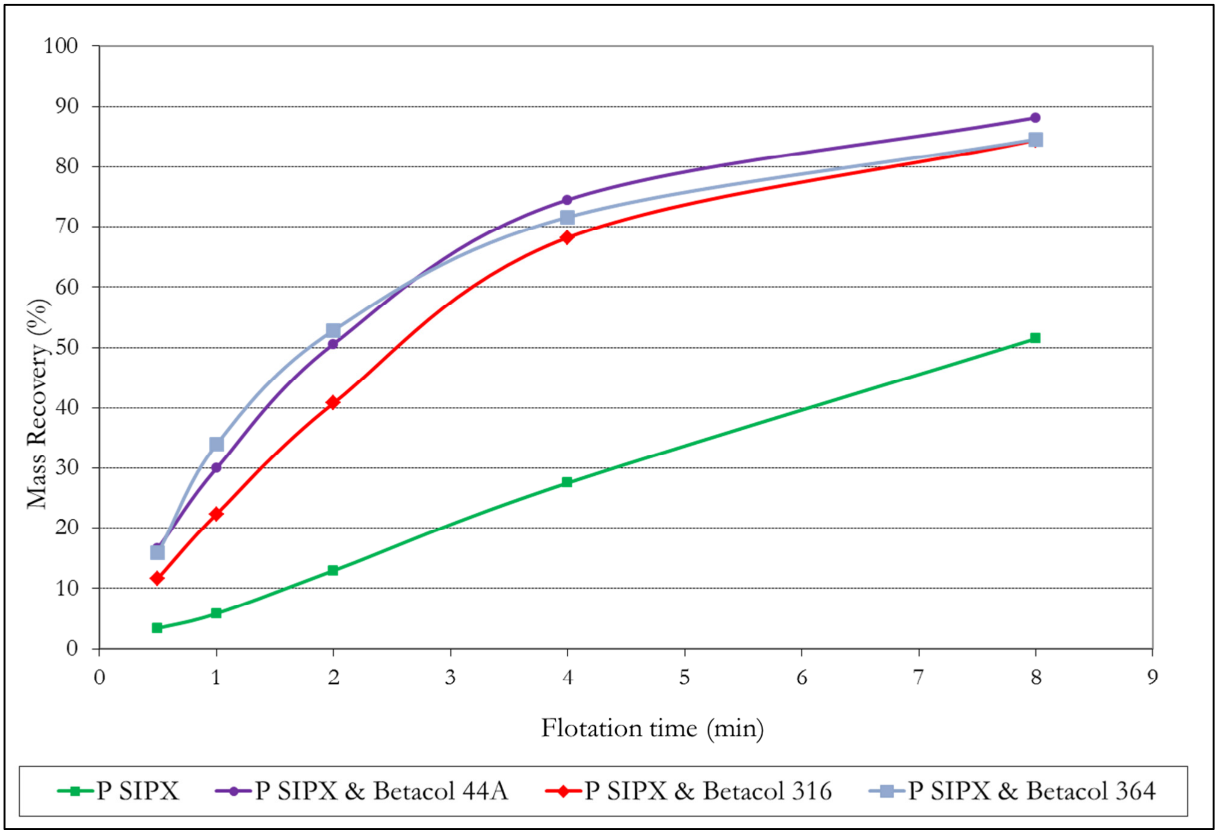


Figure 72: Micro flotation test results using micro-bubble flux micro flotation equipment. Pyrrhotite ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**.

***Distilled water with added ions to simulate plant water used. Air flow rate equivalent to a superficial gas velocity of Jg=0.48 cm/s (0.2 l/min). Xanthate added at 50 g/t and type as indicated. Betacol 364, 44A and 316 at 50 g/t. Data for individual tests.*

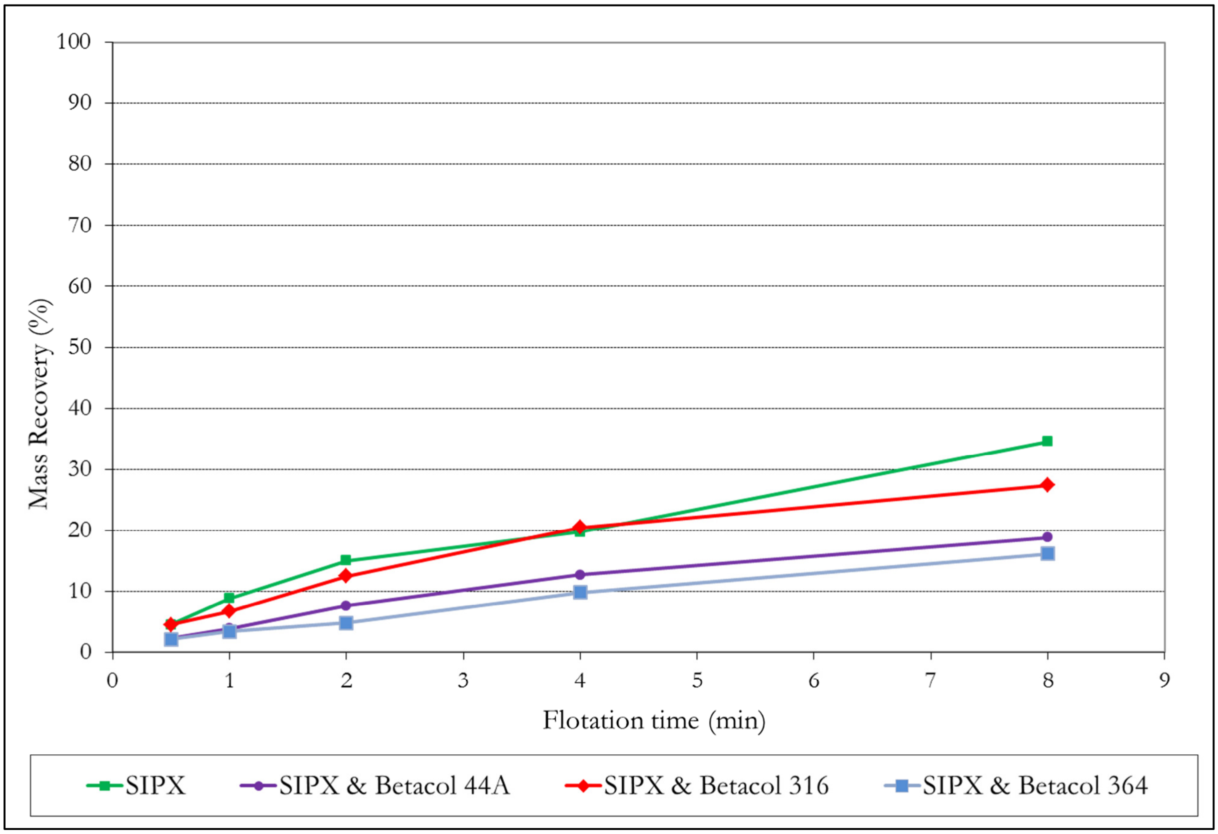


Figure 73: Micro flotation test results using micro-bubble flux micro flotation equipment. Quartz ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**. .

***Distilled water with added ions to simulate plant water used. Air flow rate equivalent to a superficial gas velocity of Jg= 0.48 cm/s (0.2 l/min). Xanthate added at 50 g/t and type as indicated. Betacol 364, 44A and 316 at 50 g/t. Data for individual tests.*

Fatty acid collectors are commercially used in oxide flotation circuits. Oleic acid is an example of a fatty acid used commercially (Rao, 2004). The rate of pyrrhotite flotation where oleic acid was used in combination with xanthate was tested against a Betacol 364 / xanthate combination. The rate of pyrrhotite recovery when oleic acid was used in combination with xanthate, (and thus particle floatability), was faster than the Betacol 364/ xanthate combination (**Figure 74**). Oleic acid was, however, not selective, and also increased the rate of quartz recovery (**Figure 75**). Oleic acid can therefore not be considered in a base metal sulphide flotation plant.

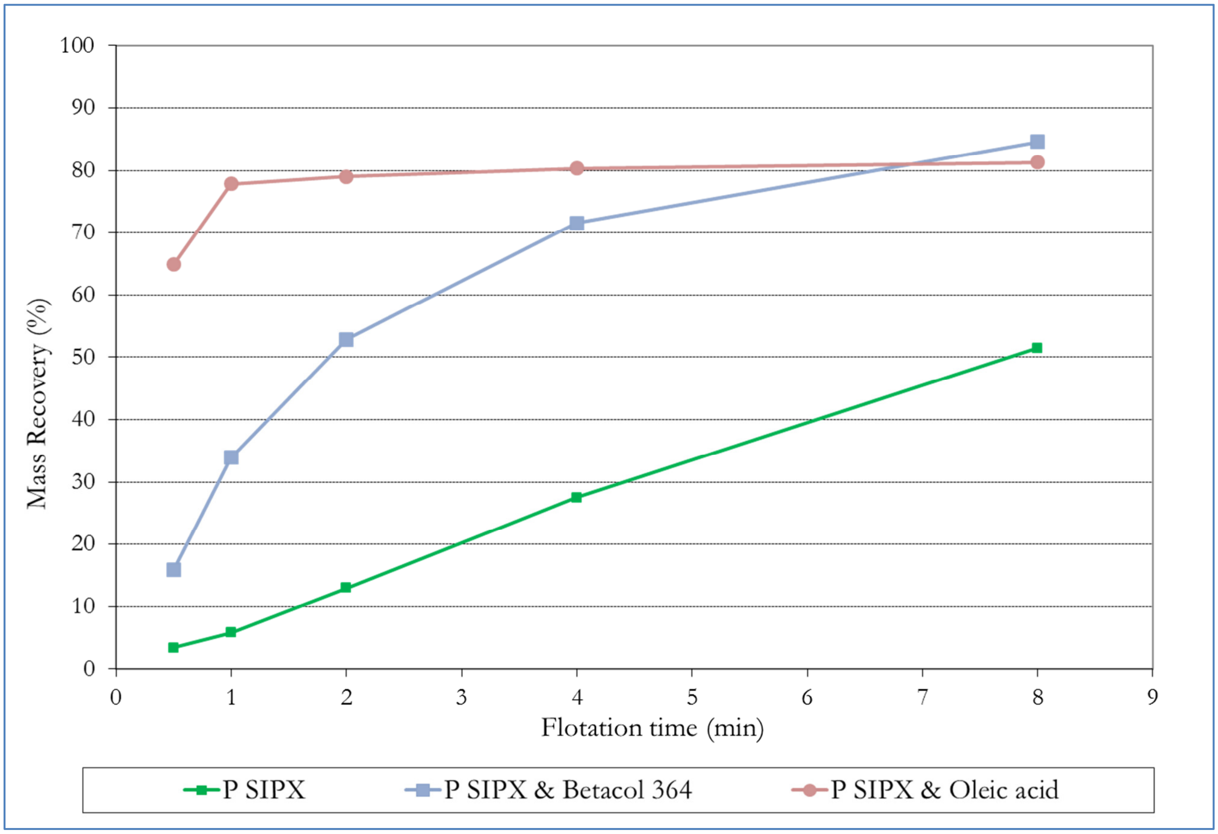


Figure 74: Micro flotation test results using micro-bubble flux micro flotation equipment. Pyrrhotite ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**.

***Distilled water with added ions to simulate plant water used. Air flow rate equivalent to a superficial gas velocity of Jg=0.48 cm/s (0.2 l/min). Xanthate added at 50 g/t and type as indicated. Betacol 364 and Oleic acid at 50 g/t. Data for individual tests.*

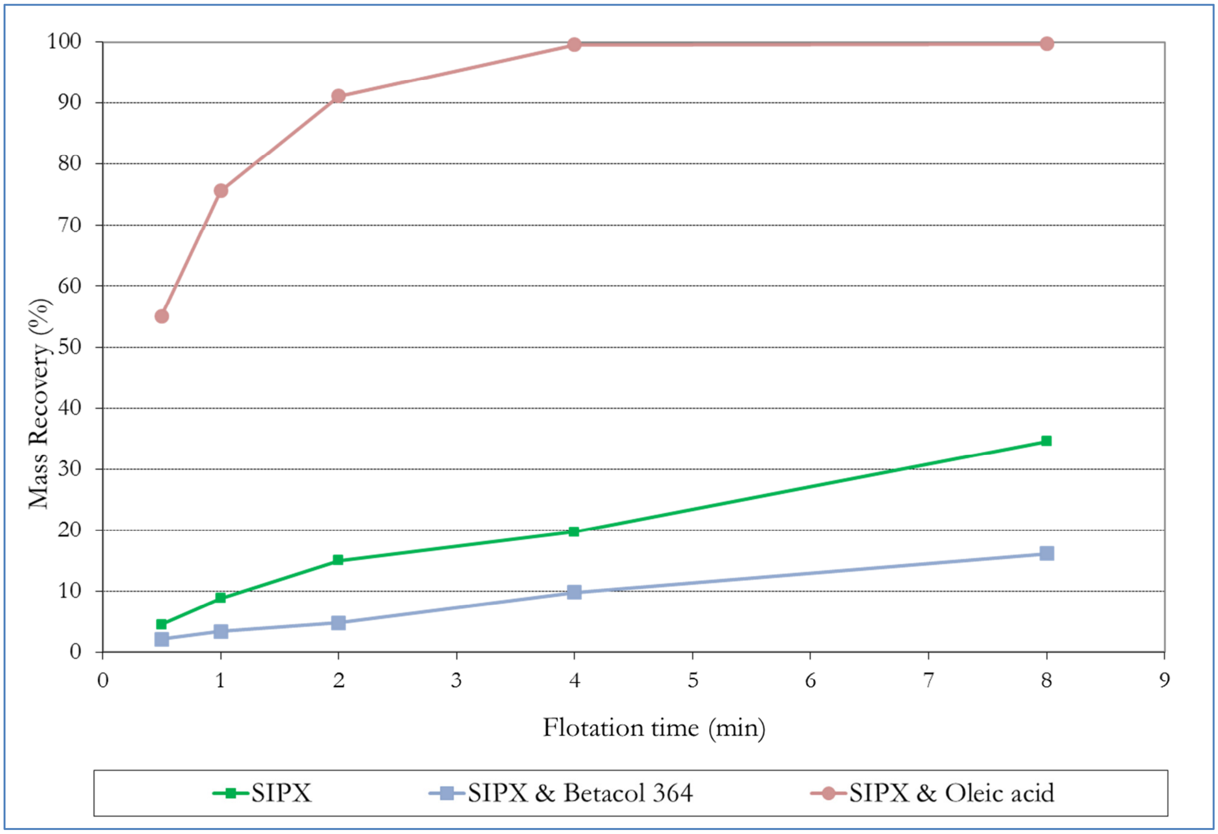


Figure 75: Micro flotation test results using micro-bubble flux micro flotation equipment. Quartz ore specimen dry milled to -106 ,+ 75 micron and tested directly after milling (fresh surface)**.

***Distilled water with added ions to simulate plant water used. Air flow rate equivalent to a superficial gas velocity of Jg=0.48 cm/s (0.2 l/min). Xanthate added at 50 g/t and type as indicated. Betacol 364 and Oleic acid at 50 g/t. Data for individual tests.*

5.5. Short pilot plant test of a fatty acid co-collector (Betacol 364) against the baseline condition being; no co-collector.

Pilot scale flotation tests were done to test the use of a fatty acid co-collector (Betacol 364) against the baseline of no co-collector. The test metode is described in Section 4.5 and the raw data appended in Section 9.5.

More copper and nickel were recovered in the first 4 minutes of flotation when the fatty acid co-collector was used in combination with xanthate, compared to the baseline of xanthate only. At 15 minutes flotation time there is little difference in copper recovery between the test (fatty acid co-collector) and baseline test. The nickel recovery at 15 minutes flotation time for the test condition (fatty acid co-collector) is higher than the baseline tests, but not as significant as at 4 minutes (**Figure 76** & **Figure 77**).

The copper concentrate grade for the test condition is not significantly different than the baseline condition. The nickel concentrate grade is lower for the test condition compared to the baseline condition (**Figure 78** & **Figure 79**).

The pilot plant tests confirms the observation that the use of Betacol 364 (fatty acid co-collector) together with xanthate increases the rate of pentlandite recovery but only the initial rate of chalcopyrite recovery, compared to the baseline condition (xanthate sole collector) .

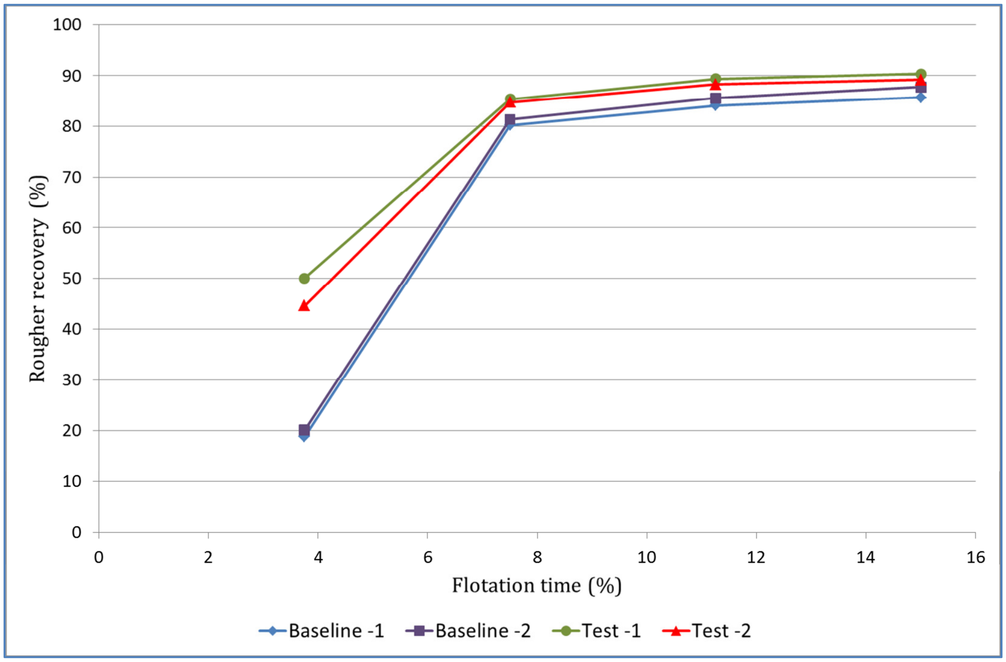


Figure 76: Copper assay data from pilot plant flotation test. Test -1 and test -2 are replica tests of baseline condition plus 50 g/t Betacol 346 **.

***Test done on continuous pulp stream extracted from cyclone overflow sump from a South African Nickel plant. Baseline conditions: 100 g/t SIBX, 30 g/t, Betafroth 436 and 250 g/t Betamin 544 .*

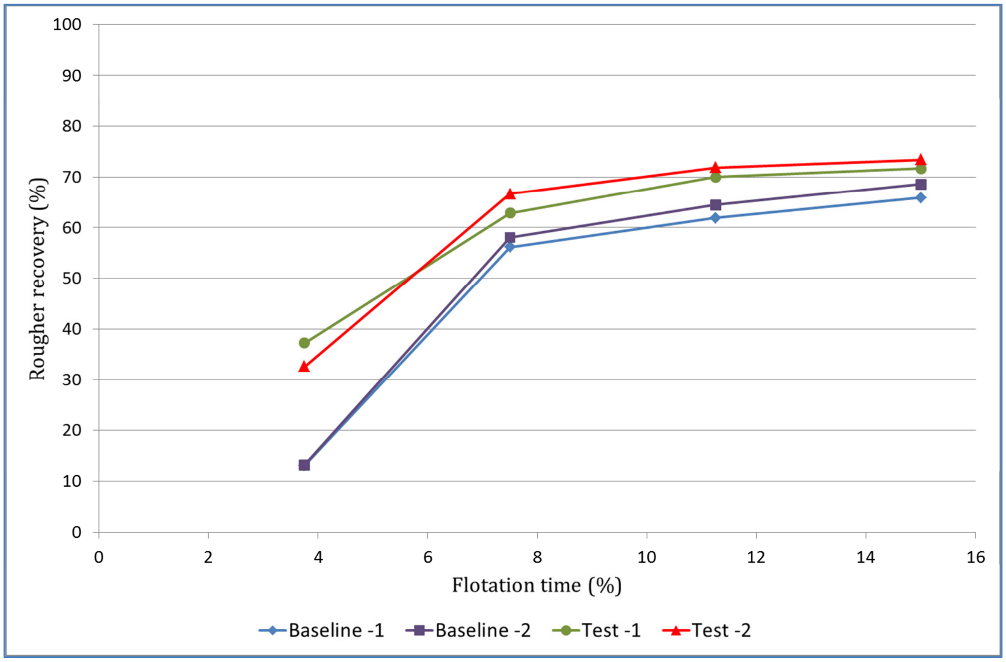


Figure 77: Nickel assay data from pilot plant flotation test. Test -1 and test -2 are replica tests of baseline condition plus 50 g/t Betacol 346 **.

***Test done on continuous pulp stream extracted from cyclone overflow sump from a South African Nickel plant. Baseline conditions: 100 g/t SIBX, 30 g/t, Betafroth 436 and 250 g/t Betamin 544 .*

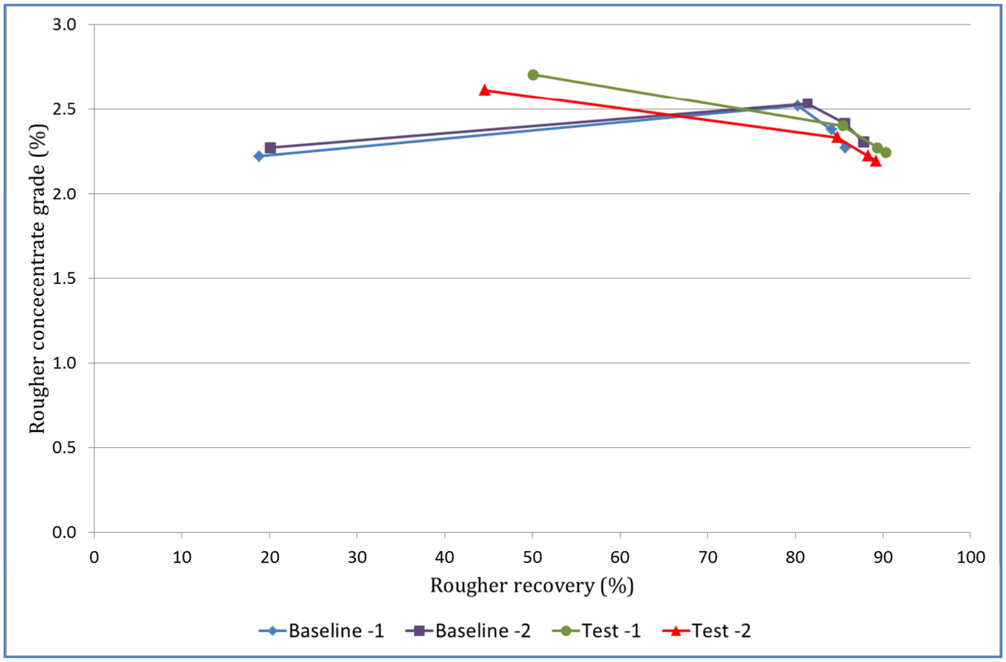


Figure 78: Copper assay data from pilot plant flotation test. Test -1 and test -2 are replica tests of baseline condition plus 50 g/t Betacol 346 **.

***Test done on continuous pulp stream extracted from cyclone overflow sump from a South African Nickel plant. Baseline conditions: 100 g/t SIBX, 30 g/t, Betafroth 436 and 250 g/t Betamin 544 .*

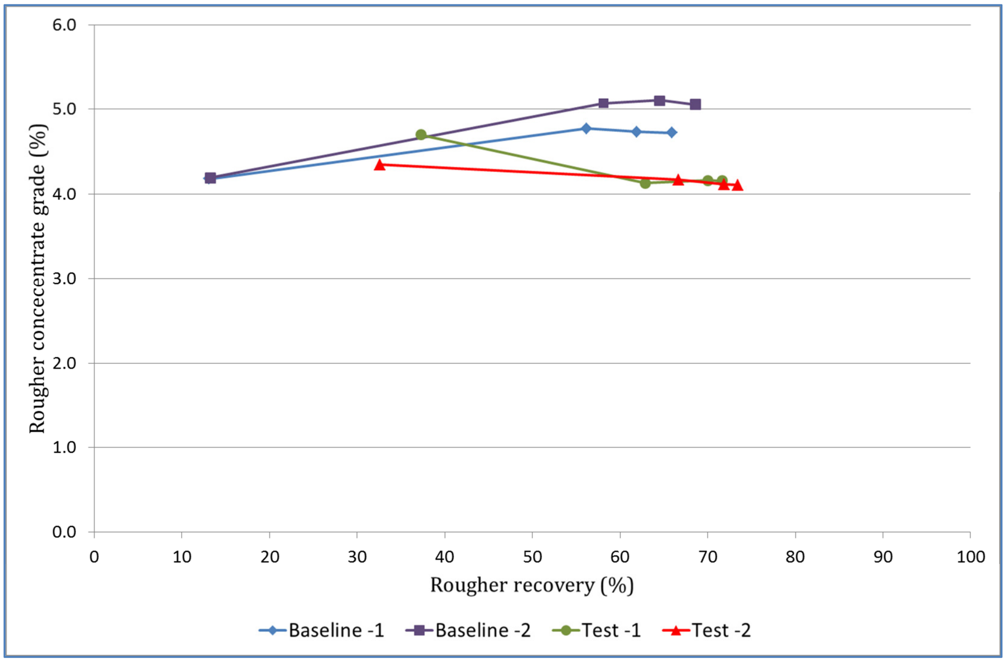


Figure 79: Nickel assay data from pilot plant flotation test. Test -1 and test -2 are replica tests of baseline condition plus 50 g/t Betacol 346 **.

***Test done on continuous pulp stream extracted from cyclone overflow sump from a South African Nickel plant. Baseline conditions: 100 g/t SIBX, 30 g/t, Betafroth 436 and 250 g/t Betamin 544 .*

5.6. Short term (crash test) plant test of a fatty acid co-collector (Betacol 364) against the baseline condition being; no co-collector.

The first step to test a reagent suite is a short term plant test (crash test) as described in Section 3.8. The test procedure to test the use of a fatty acid co-collector (Betacol 364) against the baseline of no co-collector is described in Section 4.6.

The assumption made is that the PGE minerals (mainly braggite and cooperite) is associated or included in the base metal sulphides, chalcopyrite, pentlandite and pyrrhotite. If this assumption is valid, then PGM fire assay can be used to evaluate the effect the co-collector has on BMS recovery. This assumption is untested and may not be valid. The nickel, copper and sulphur assays in the final tails were below the detection limit of the laboratory used. These values would have been the preferred assay results to use.

5.6.1. Interpretation of plant results using the t-test.

The t-test was used to test the hypothesis that the plant results during the test block were similar to the results for the baseline block at 80% confidence. If this hypothesis is confirmed, the following terminology will be used: there is NO SIGNIFICANT difference between the test and baseline blocks. If the hypothesis can not be confirmed, the following terminology will be used: there is a SIGNIFICANT difference between the test and baseline blocks.

One approach is to view each test day as a separate test and apply the t-test to each day's data individually. The test data and evaluation is shown in **Table 22** and **Table 23**.

In summary it can be shown:

- The average PGM (4E) g/t feed to the flotation plant for both test days were not significantly different.
- The average PGM (4E) g/t in the final tails for both test days was significantly lower during the test period compared to the baseline.
- The average PGM (4E) g/t in the final concentrate for day one was not significantly lower, but for day two was significantly lower for the test block compared to the baseline.
- The average sulphur percentage (%) in the final concentrate for both test days were significantly lower for the test block compared to the baseline.
- The average PGM (4E) g/t flotation recovery for day one was significantly higher, but for day two was not significantly higher for the test block compared to the baseline block.

A second approach would be to group all the test block data for both days together and compare this to the baseline data for both days. The test data and evaluation is shown in **Table 24**. In summary it can be shown:

- The average PGM (4E) g/t feed to the flotation plant for the baseline blocks were not significantly different than the test blocks.
- The average PGM (4E) g/t in the final tails for the test blocks were significantly lower compared to the baseline blocks.
- The average PGM (4E) g/t in the final concentrate for the test blocks was not significantly lower than the baseline blocks.
- The averages sulphur (%) in the final concentrate for the test blocks were significantly lower than the baseline blocks.
- The average PGM (4E) g/t recovery for the test blocks were significantly higher compared to the baseline blocks.

From this analysis it is concluded that the addition of 50 g/t Betacol 364 (fatty acid) to the normal reagent suite, results in higher PGM recovery without a decreased in PGM concentrate grade (sulphide concentrate is lower).

Table 22: PGM (4E) g/t and sulphide (by Leco) measured during a two day plant test on a South African opencast Platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay and sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. T-test used to calculate if the data during the test period is significantly (at 80% confidence level) different than the baseline period. For this calculation each of the two test days are analysed separately.
Day 1

			4E PGM												Sulfur		
Treatment	Day	Shift	Feed			Conc			Tails			Recovery			Conc		
			OFF	ON		OFF	ON		OFF	ON		OFF	ON		OFF	ON	
OFF	15-Jun-10	08:00	2.81			1045			1.39			51			4.80%		
OFF	15-Jun-10	08:30	2.81			1047			1.37			51			4.86%		
OFF	15-Jun-10	09:00	3.04			760			1.59			48			3.71%		
OFF	15-Jun-10	09:30	2.97			410			1.29			57			2.50%		
OFF	15-Jun-10	10:00	3.16			358			1.29			59			2.12%		
OFF	15-Jun-10	10:30	3.09			356			1.37			56			2.01%		
OFF	15-Jun-10	11:00	2.96			259			1.56			48			1.79%		
OFF	15-Jun-10	11:30	2.74			208			1.50			46			1.49%		
ON	15-Jun-10	15:00		3.41			246			1.41			59			1.09%	
ON	15-Jun-10	15:30		2.87			260			1.44			50			1.51%	
ON	15-Jun-10	16:00		2.61			49			1.41			47			0.29%	
ON	15-Jun-10	16:30		2.62			250			1.46			45			1.98%	
ON	15-Jun-10	17:00		2.73			321			1.12			59			2.18%	
ON	15-Jun-10	17:30		2.88			1461			1.16			60			4.53%	
ON	15-Jun-10	18:00		2.96			644			0.99			67			3.08%	
ON	15-Jun-10	18:30		3.00			261			0.68			78			1.40%	
Null hypothesis (μ=?)					0			0			0			0			0
Confidence					80			80			80			80			80
Average			2.95	2.89		555	437		1.42	1.21		51.9	58.0		2.91%	2.01%	
Standard Deviation			0.15	0.26		345	445		0.12	0.28		5.0	10.8		1.4%	1.3%	
Level of Significance (α)					0.1			0.1			0.1			0.1			0.1
Number of test			8	8	16	8	8	16	8	8	16	8	8	16	8	8	16
t Critical one-tail					1.35			1.35			1.35			1.35			1.35
t-Test: Two-Sample Assuming Equal Variances																	
t stat (small sample)					0.59			0.60			1.99			-1.46			1.36
(returns positive value for t stat)					0.59			0.60			1.99			1.46			1.36
			not significant			not significant			significant			significant			significant		

Table 23: PGM (4E) g/t and sulphide (by Leco) measured during a two day plant test on a South African opencast Platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay and sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. T-test used to calculate if the data during the test period is significantly (at 80% confidence level) different than the baseline period. For this calculation each of the two test days are analysed separately. Day 2

			4E PGM												Sulfur		
Treatment	Day	Shift	Feed			Conc			Tails			Recovery			Conc		
			OFF	ON		OFF	ON		OFF	ON		OFF	ON		OFF	ON	
OFF	16-Jun-10	07:30	2.41			214			1.37			43			1.56%		
OFF	16-Jun-10	08:00	2.60			286			1.77			32			2.28%		
OFF	16-Jun-10	08:30	3.06			492			1.51			51			2.93%		
OFF	16-Jun-10	09:00	2.53			335			1.57			38			2.48%		
OFF	16-Jun-10	09:30	2.59			383			1.72			34			2.29%		
OFF	16-Jun-10	10:00	2.75			207			1.72			38			1.35%		
ON	16-Jun-10	13:30		2.58			206			1.32			49			1.28%	
ON	16-Jun-10	14:00		2.63			140			1.53			42			0.90%	
ON	16-Jun-10	14:30		2.21			36			1.31			42			0.44%	
Null hypothesis (μ=?)					0			0			0			0			0
Confidence					80			80			80			80			80
Average			2.66	2.47		320	127		1.61	1.39		39.3	44.6		2.15%	0.87%	
Standard Deviation			0.23	0.23		108.6	85.8		0.15	0.12		6.9	4.0		0.59%	0.42%	
Level of Significance (α)					0.1			0.1			0.1			0.1			0.1
Number of test			6	3	9	6	3	9	6	3	9	6	3	9	6	3	9
t Critical one-tail					1.41			1.41			1.41			1.41			1.41
t-Test: Two-Sample Assuming Equal Variances																	
t stat (small sample)					1.14			2.65			2.16			-1.20			3.30
(returns positive value for t stat)					1.14			2.65			2.16			1.20			3.30
			not significant			significant			significant			not significant			significant		

Table 24: PGM (4E) g/t and sulphide (by Leco) measured during a two day plant test on a South African opencast Platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay and sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. T-test used to calculate if the data during the test period is significantly (at 80% confidence level) different than the baseline period. For this calculation data for both test periods are combined.

			4E PGM												Sulfur		
Treatmer	Day	Shift	Feed			Conc			Tails			Recovery			Conc		
			OFF	ON		OFF	ON		OFF	ON		OFF	ON		OFF	ON	
OFF	15-Jun-10	08:00	2.81			1045			1.39			51			4.80%		
OFF	15-Jun-10	08:30	2.81			1047			1.37			51			4.86%		
OFF	15-Jun-10	09:00	3.04			760			1.59			48			3.71%		
OFF	15-Jun-10	09:30	2.97			410			1.29			57			2.50%		
OFF	15-Jun-10	10:00	3.16			358			1.29			59			2.12%		
OFF	15-Jun-10	10:30	3.09			356			1.37			56			2.01%		
OFF	15-Jun-10	11:00	2.96			259			1.56			48			1.79%		
OFF	15-Jun-10	11:30	2.74			208			1.50			46			1.49%		
ON	15-Jun-10	15:00		3.41			246			1.41			59			1.09%	
ON	15-Jun-10	15:30		2.87			260			1.44			50			1.51%	
ON	15-Jun-10	16:00		2.61			49			1.41			47			0.29%	
ON	15-Jun-10	16:30		2.62			250			1.46			45			1.98%	
ON	15-Jun-10	17:00		2.73			321			1.12			59			2.18%	
ON	15-Jun-10	17:30		2.88			1461			1.16			60			4.53%	
ON	15-Jun-10	18:00		2.96			644			0.99			67			3.08%	
ON	15-Jun-10	18:30		3.00			261			0.68			78			1.40%	
OFF	16-Jun-10	07:30	2.41			214			1.37			43			1.56%		
OFF	16-Jun-10	08:00	2.60			286			1.77			32			2.28%		
OFF	16-Jun-10	08:30	3.06			492			1.51			51			2.93%		
OFF	16-Jun-10	09:00	2.53			335			1.57			38			2.48%		
OFF	16-Jun-10	09:30	2.59			383			1.72			34			2.29%		
OFF	16-Jun-10	10:00	2.75			207			1.72			38			1.35%		
ON	16-Jun-10	13:30		2.58			206			1.32			49			1.28%	
ON	16-Jun-10	14:00		2.63			140			1.53			42			0.90%	
ON	16-Jun-10	14:30		2.21			36			1.31			42			0.44%	
Null hypothesis (µ=?)					0			0			0			0			0
Confidence					80			80			80			80			80
Average			2.82	2.77		454	352		1.50	1.26		46.5	54.3		2.58%	1.70%	
Standard Deviation			0.23	0.31		288	402		0.16	0.25		8.5	11.2		1.13%	1.23%	
Level of Significance (α)					0.1			0.1			0.1			0.1			0.1
Number of test			14	11	25	14	11	25	14	11	25	14	11	25	14	11	25
t Critical one-tail					1.32			1.32			1.32			1.32			1.32
t-Test: Two-Sample Assuming Equal Variances																	
t stat (small sample)					0.47			0.74			2.95			-2.00			1.87
(returns positive value for t stat)					0.47			0.74			2.95			2.00			1.87
			not significant			not significant			significant			significant			significant		

5.6.2. Interpretation of plant results by ANOVA.

ANOVA was used to test the hypothesis that the plant results during the 4 blocks (two test blocks and 2 baseline blocks) were similar at 90% confidence. If this hypothesis is confirmed the following terminology will be used: there is NO SIGNIFICANT difference between the blocks. If the hypothesis can not be confirmed, the following terminology will be used: there is a SIGNIFICANT difference between the blocks.

The test data and evaluation is shown in **Table 25**. In summary it can be shown:

- The average PGM (4E) g/t feed to the flotation plant for the 4 blocks were not significantly different.
- The average PGM (4E) g/t in the final tails for the 4 blocks was significantly different.
- The average PGM (4E) g/t in the final concentrate for the 4 blocks was not significantly different.
- The averages sulphur (%) in the final concentrate for the 4 blocks was not significantly different.
- The average PGM (4E) g/t recovery for the 4 blocks was significantly different.

From this analysis it is concluded that the addition of 50 g/t Betacol 364 (fatty acid) to the normal reagent suite, results in higher PGM recovery without a decreased in PGM concentrate grade.

Table 25: PGM (4E) g/t and Sulphide (by Leco) measured during a two day plant test on a South African opencast Platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by Fire assay and Sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. ANOVA used to calculate if the data during the test period is significantly (at 80% confidence level) different than the baseline period. Each test bloc treated as a separate treatment

			4E PGM																Sulfur			
Treatment	Day	Shift	Feed				Conc				Tails				Recovery				Conc			
			Off	On	Off	On	Off	On	Off	On	Off	On	Off	On	Off	On	Off	On	Off	On	Off	On
OFF	15-Jun-10	08:00	2.81				1045				1.39				51				4.80%			
OFF	15-Jun-10	08:30	2.81				1047				1.37				51				4.86%			
OFF	15-Jun-10	09:00	3.04				760				1.59				48				3.71%			
OFF	15-Jun-10	09:30	2.97				410				1.29				57				2.50%			
OFF	15-Jun-10	10:00	3.16				358				1.29				59				2.12%			
OFF	15-Jun-10	10:30	3.09				356				1.37				56				2.01%			
OFF	15-Jun-10	11:00	2.96				259				1.56				48				1.79%			
OFF	15-Jun-10	11:30	2.74				208				1.50				46				1.49%			
ON	15-Jun-10	15:00		3.41				246				1.41				59				1.09%		
ON	15-Jun-10	15:30		2.87				260				1.44				50				1.51%		
ON	15-Jun-10	16:00		2.61				49				1.41				47				0.29%		
ON	15-Jun-10	16:30		2.62				250				1.46				45				1.98%		
ON	15-Jun-10	17:00		2.73				321				1.12				59				2.18%		
ON	15-Jun-10	17:30		2.88				1461				1.16				60				4.53%		
ON	15-Jun-10	18:00		2.96				644				0.99				67				3.08%		
ON	15-Jun-10	18:30		3.00				261				0.68				78				1.40%		
OFF	16-Jun-10	07:30			2.41				214				1.37				43				1.56%	
OFF	16-Jun-10	08:00			2.60				286				1.77				32				2.28%	
OFF	16-Jun-10	08:30			3.06				492				1.51				51				2.93%	
OFF	16-Jun-10	09:00			2.53				335				1.57				38				2.48%	
OFF	16-Jun-10	09:30			2.59				383				1.72				34				2.29%	
OFF	16-Jun-10	10:00			2.75				207				1.72				38				1.35%	
ON	16-Jun-10	13:30				2.58				206				1.32				49				1.28%
ON	16-Jun-10	14:00				2.63				140				1.53				42				0.90%
ON	16-Jun-10	14:30				2.21				36				1.31				42				0.44%
Null hypothesis (μ=?)						0				0				0				0				0
Confidence						90				90				90				90				90
Average			2.95	2.89	2.66	2.47	555.38	436.51	319.50	127.30	1.42	1.21	1.61	1.39	51.9	58.0	39.3	44.6	2.91%	2.01%	2.15%	0.87%
Sum			23.58	23.08	15.94	7.42	4443.0	3492.1	1917.0	381.90	11.36	9.67	9.66	4.16	414.9	464.1	236.0	133.7	0.23	0.16	0.13	0.03
Standard Deviation			0.15	0.26	0.23	0.23	344.68	445.47	108.57	85.76	0.12	0.28	0.15	0.12	4.97	10.82	6.87	3.97	0.01	0.01	0.01	0.00
Level of Significance (α)						0.05				0.05				0.05				0.05				0.05
Number of Teatments						4				3				3				3				3
Number of test			8	8	6	3	8	8	6	3	8	8	6	3	8	8	6	3	8	8	6	3
ANOVA: Single factor																						
Source of variation			Degrees of freedom		Sum o Squer	Mean Squar	Degrees of freedom		Sum of Squere	Mean Square	Degrees of freedom		Sum o Squer	Mean Squar	Degrees of freedom		Sum o Squer	Mean Squar	Degrees of freedom		Sum o Squere	Mean Square
Between treatments			3	0.68		0.225	2	463584		231792	2	0.56		0.280	2	1313		656.3	2	0.10%		0.0
within treatments			13	0.98		0.075	14	2294395		163885	14	0.78		0.056	14	1259		90.0	14	0.27%		0.0
Total			16	1.66			16	2757978			16	1.34			16	2572			16	0.37%		
F stat						2.98				1.41				5.03				7.30				2.50
F Critical (Single factor)						3.41				3.74				3.74				3.74				3.74
			not significant				not significant				significant				significant				not significant			

5.6.3. Interpretation of plant results by confidence intervals.

Both the t-test and ANOVA are useful statistical tools to help evaluate data from a plant test. A third method which can be used is evaluate plant test data is to plot confidence intervals. The test data and data analysis is shown in **Table 26**. The statistical interpretation of the confidence interval calculated in this analysis is that the average of the test block data is, with 60% confidence, are between the upper and lower confidence limits. If the upper limit of one block is higher than the lower limit of a second block, then with 60% confidence the first average is higher than the second average and this can help with the interpretation of test data. This method is useful if multiple blocks need to be evaluated. It is helpful to plot the plant data and confidence limits.

The convention that will be used is that if the confidence intervals overlap it will be noted that the average of the two blocks is NOT SIGNIFICANTLY different. If the two limits do not overlap, it will be noted that the average of the two blocks is SIGNIFICANTLY different.

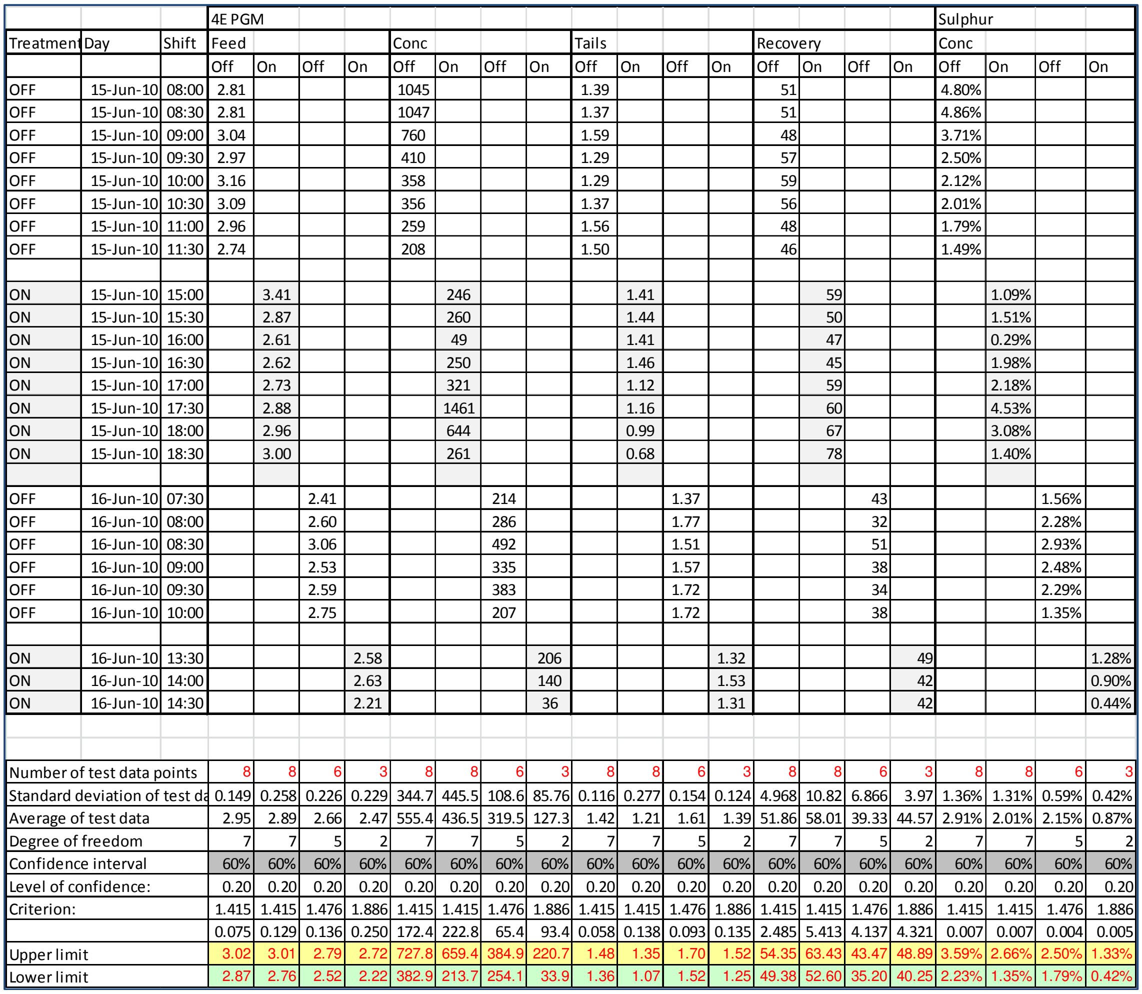
The test data and evaluation is shown **Table 26**. In summary it can be shown:

- The PGM (4E) g/t feed to the flotation plant decreased over time from block 1 on day 1 to block 2 on day 2 (**Figure 80**). The average feed for the test and baseline block for both days are not significantly different.
- The PGM (4E) g/t plant final concentrate decreased over time from block 1 on day 1 to block 2 on day 2 (**Figure 81**). The PGM concentrate grade for the first day's test and baseline block s in not significantly different. On the second day the PGM grade for the test block is lower than the baseline block.
- The PGM (4E) g/t flotation tails for both days are lower during the test block than during the baseline block (**Figure 82).**
- The PGM recovery measured during the test block appears to be lower for the test block compared to the baseline block (**Figure 83**). The confidence intervals do overlap and it thus has to be concluded that higher recovery is probably but can not be confirmed.

- The sulphide concentration in the final concentrate appears to be lower for the test block compared to the baseline block (**Figure 84**), but this can only be confirmed with the confidence interval method for Day 2.

From this data it can be concluded that the final flotation tails were lower when 50 g/t Betacol 364 (fatty acid) were added to the normal flotation reagent suite. It appears that the concentrate grade is lower when the fatty acid is used as co-collector.

Table 26: PGM (4E) g/t and sulphide (by Leco) measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay and sulphur by Leco. Plant feed, final flotation concentrate and final flotation tails were sampled. Confidence intervals calculated based on a 60% confidence interval.



			4E PGM																Sulphur			
Treatment	Day	Shift	Feed				Conc				Tails				Recovery				Conc			
			Off	On	Off	On	Off	On	Off	On	Off	On	Off	On	Off	On	Off	On	Off	On	Off	On
OFF	15-Jun-10	08:00	2.81				1045				1.39				51				4.80%			
OFF	15-Jun-10	08:30	2.81				1047				1.37				51				4.86%			
OFF	15-Jun-10	09:00	3.04				760				1.59				48				3.71%			
OFF	15-Jun-10	09:30	2.97				410				1.29				57				2.50%			
OFF	15-Jun-10	10:00	3.16				358				1.29				59				2.12%			
OFF	15-Jun-10	10:30	3.09				356				1.37				56				2.01%			
OFF	15-Jun-10	11:00	2.96				259				1.56				48				1.79%			
OFF	15-Jun-10	11:30	2.74				208				1.50				46				1.49%			
ON	15-Jun-10	15:00		3.41				246				1.41				59				1.09%		
ON	15-Jun-10	15:30		2.87				260				1.44				50				1.51%		
ON	15-Jun-10	16:00		2.61				49				1.41				47				0.29%		
ON	15-Jun-10	16:30		2.62				250				1.46				45				1.98%		
ON	15-Jun-10	17:00		2.73				321				1.12				59				2.18%		
ON	15-Jun-10	17:30		2.88				1461				1.16				60				4.53%		
ON	15-Jun-10	18:00		2.96				644				0.99				67				3.08%		
ON	15-Jun-10	18:30		3.00				261				0.68				78				1.40%		
OFF	16-Jun-10	07:30			2.41				214				1.37				43				1.56%	
OFF	16-Jun-10	08:00			2.60				286				1.77				32				2.28%	
OFF	16-Jun-10	08:30			3.06				492				1.51				51				2.93%	
OFF	16-Jun-10	09:00			2.53				335				1.57				38				2.48%	
OFF	16-Jun-10	09:30			2.59				383				1.72				34				2.29%	
OFF	16-Jun-10	10:00			2.75				207				1.72				38				1.35%	
ON	16-Jun-10	13:30				2.58				206				1.32				49				1.28%
ON	16-Jun-10	14:00				2.63				140				1.53				42				0.90%
ON	16-Jun-10	14:30				2.21				36				1.31				42				0.44%
Number of test data points			8	8	6	3	8	8	6	3	8	8	6	3	8	8	6	3	8	8	6	3
Standard deviation of test da			0.149	0.258	0.226	0.229	344.7	445.5	108.6	85.76	0.116	0.277	0.154	0.124	4.968	10.82	6.866	3.97	1.36%	1.31%	0.59%	0.42%
Average of test data			2.95	2.89	2.66	2.47	555.4	436.5	319.5	127.3	1.42	1.21	1.61	1.39	51.86	58.01	39.33	44.57	2.91%	2.01%	2.15%	0.87%
Degree of freedom			7	7	5	2	7	7	5	2	7	7	5	2	7	7	5	2	7	7	5	2
Confidence interval			60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%	60%
Level of confidence:			0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
Criterion:			1.415	1.415	1.476	1.886	1.415	1.415	1.476	1.886	1.415	1.415	1.476	1.886	1.415	1.415	1.476	1.886	1.415	1.415	1.476	1.886
			0.075	0.129	0.136	0.250	172.4	222.8	65.4	93.4	0.058	0.138	0.093	0.135	2.485	5.413	4.137	4.321	0.007	0.007	0.004	0.005
Upper limit			3.02	3.01	2.79	2.72	727.8	659.4	384.9	220.7	1.48	1.35	1.70	1.52	54.35	63.43	43.47	48.89	3.59%	2.66%	2.50%	1.33%
Lower limit			2.87	2.76	2.52	2.22	382.9	213.7	254.1	33.9	1.36	1.07	1.52	1.25	49.38	52.60	35.20	40.25	2.23%	1.35%	1.79%	0.42%

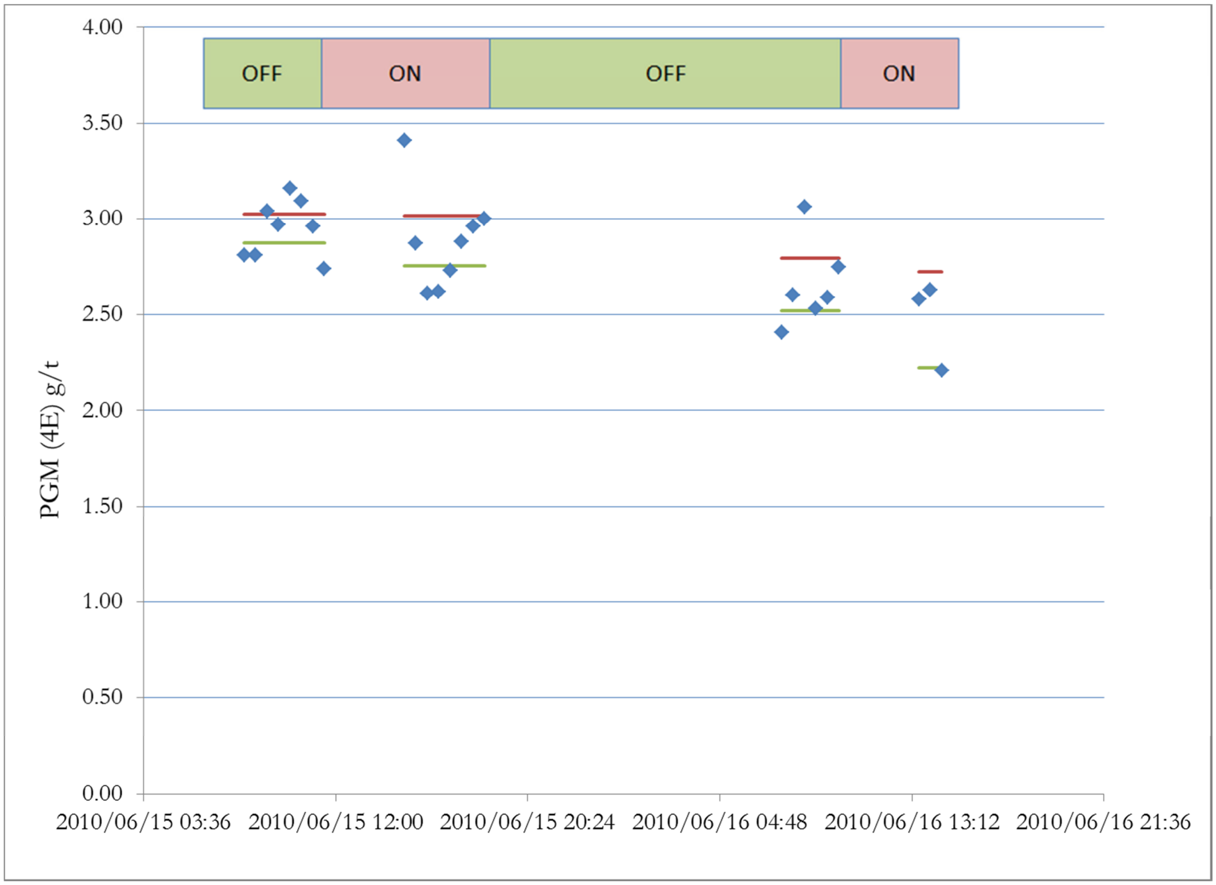


Figure 80: PGM (4E) g/t feed grade measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period.

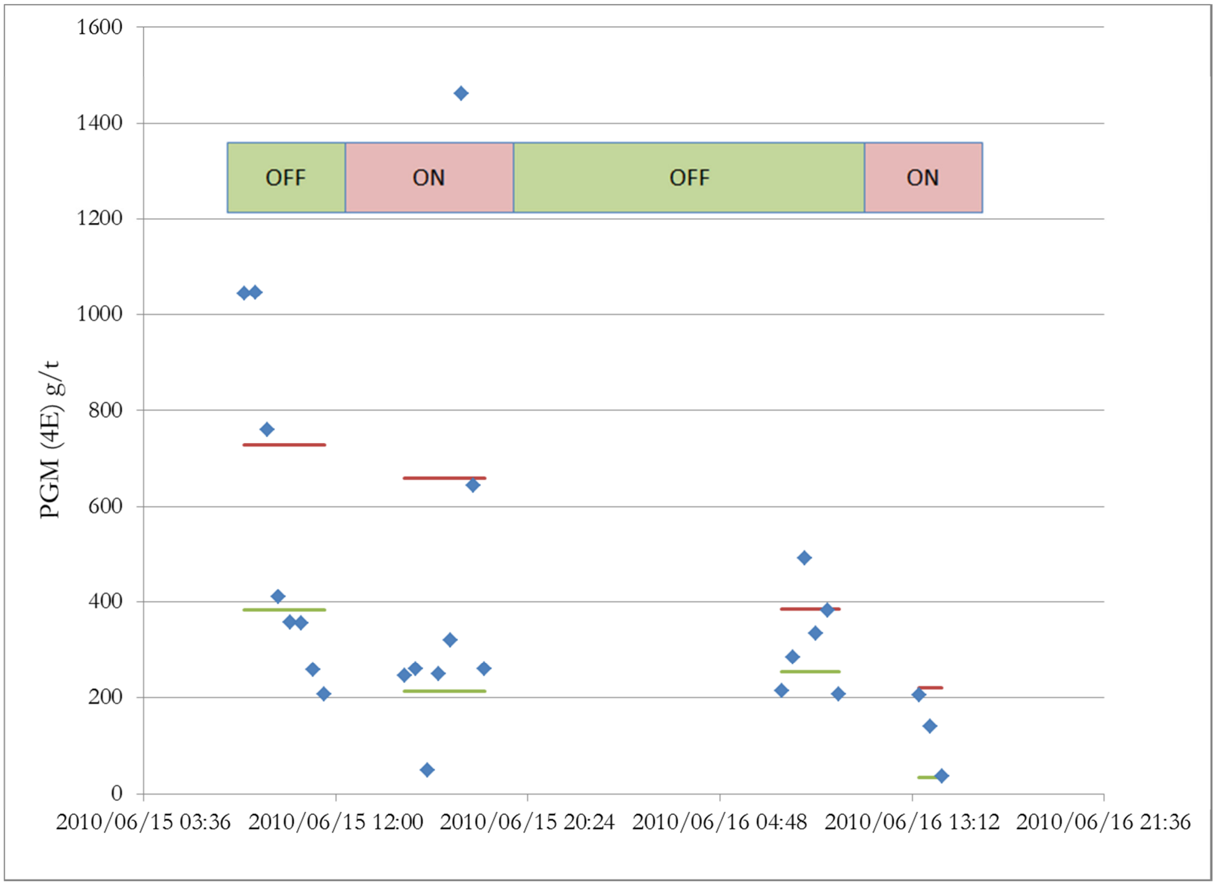


Figure 81: PGM (4E) g/t final concentrate grade measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period.

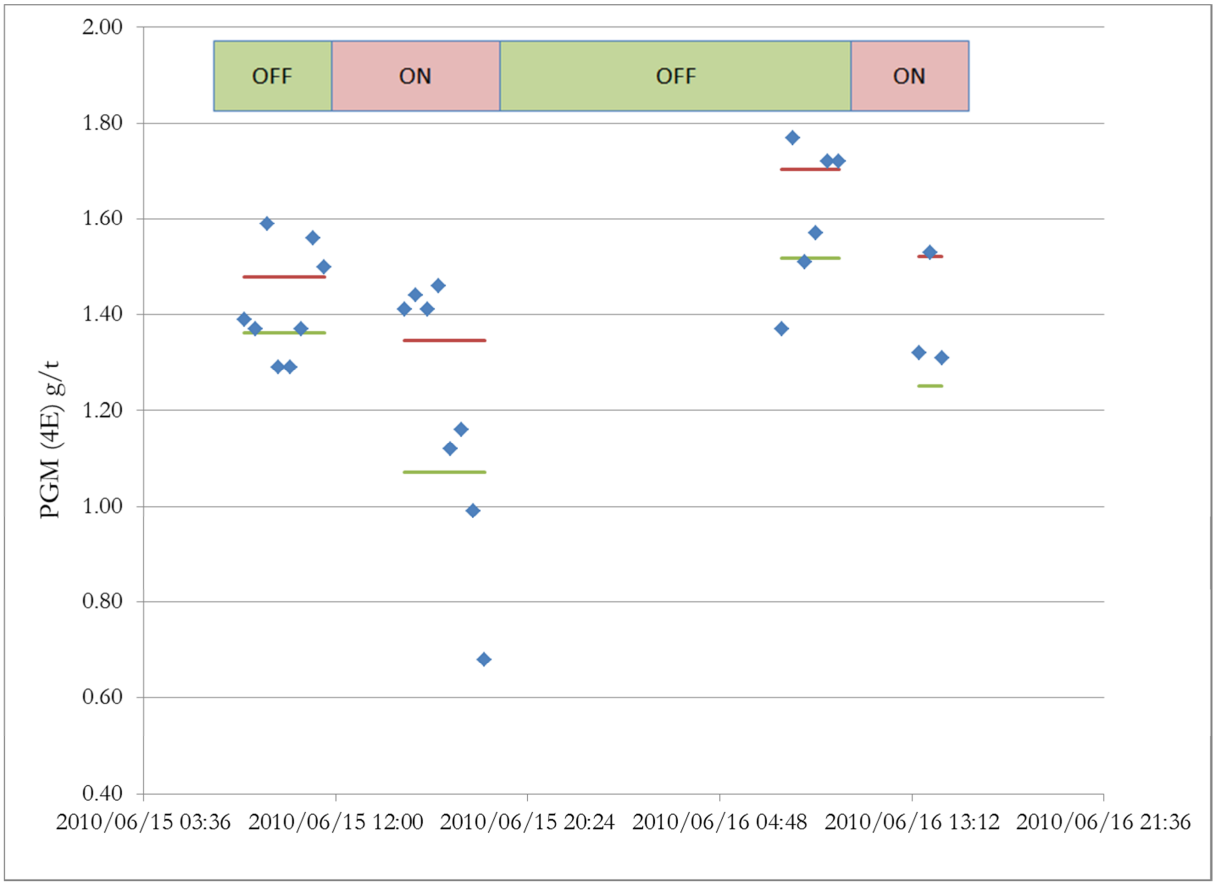


Figure 82: PGM (4E) g/t final flotation tails grade measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period.

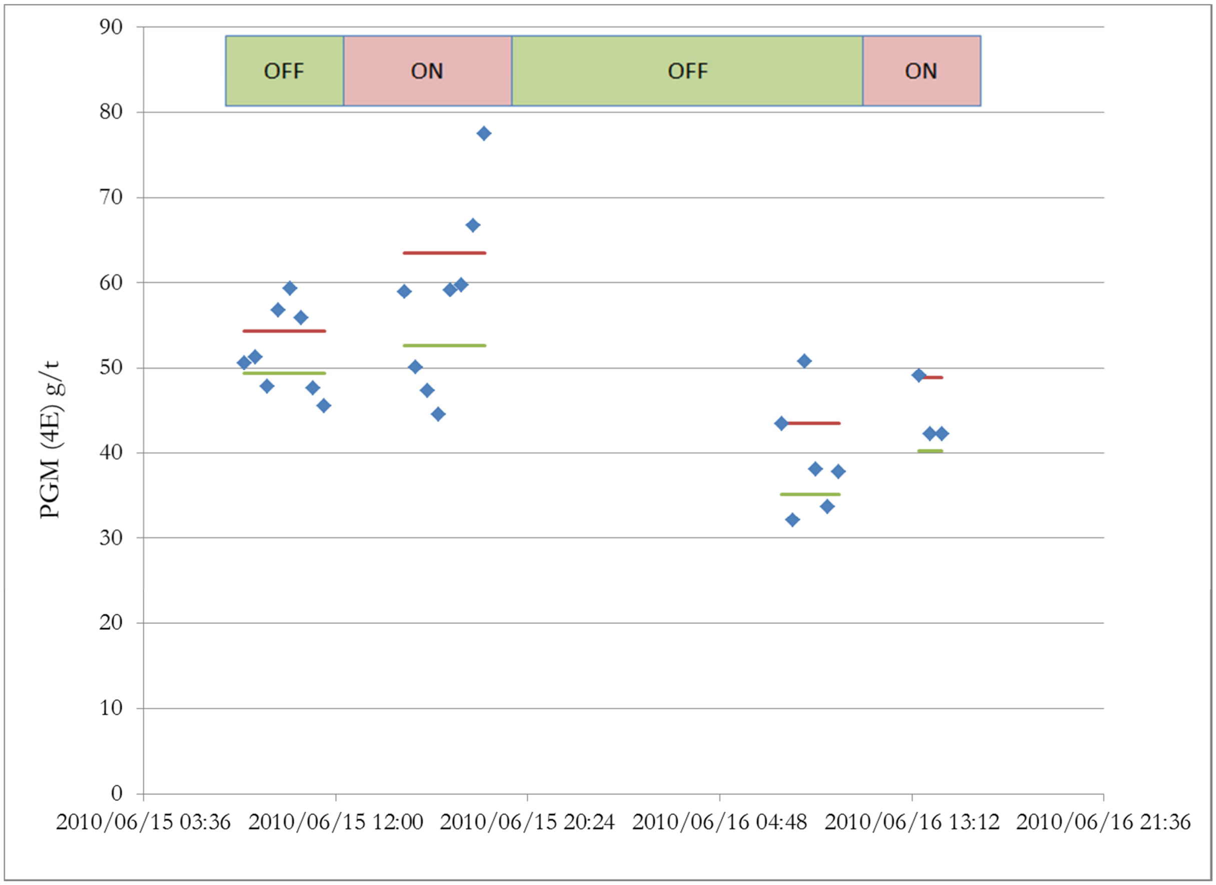


Figure 83: PGM (4E) g/t plant recovery measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for PGM-4E by fire assay. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period.

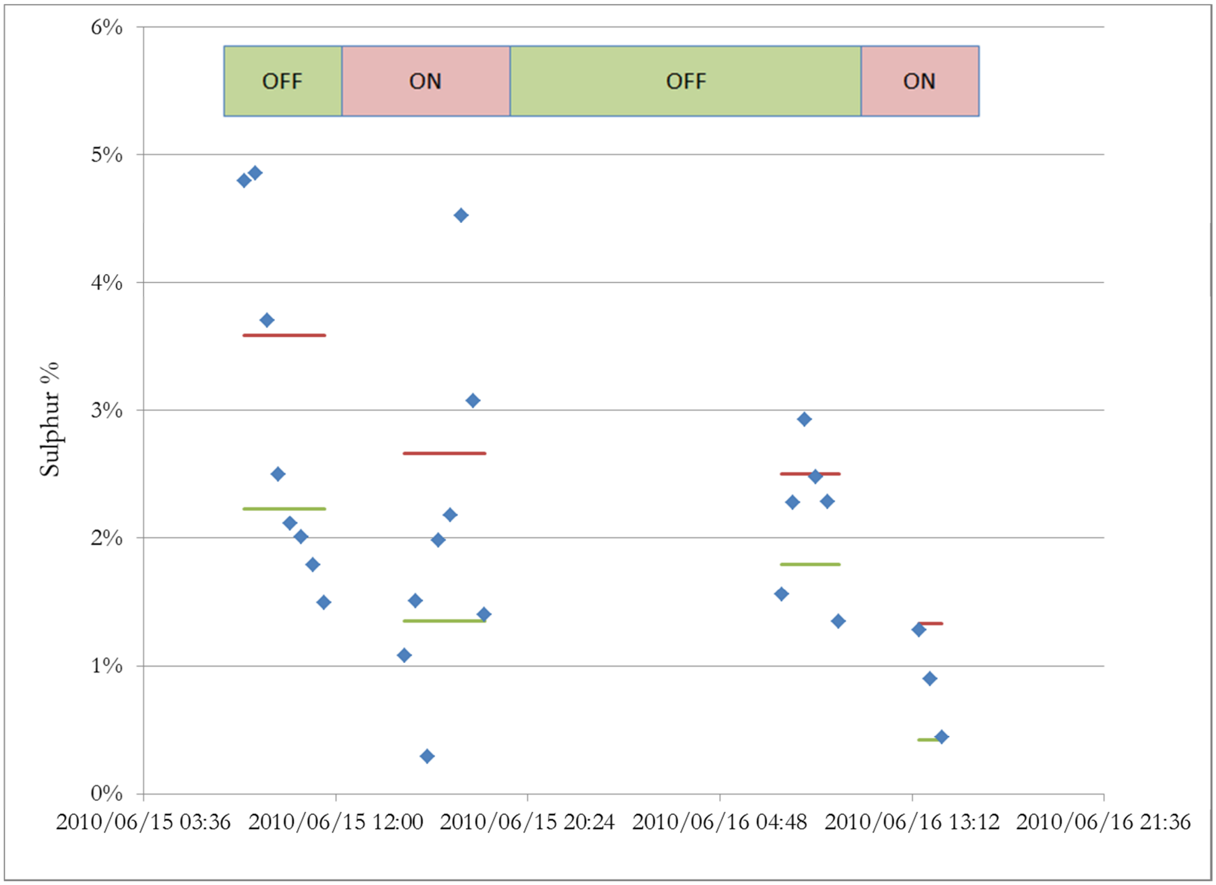


Figure 84: Sulphur (by Leco) final concentrate grade measured during a two day plant test on a South African opencast platinum concentrator. Betacol 364 at 50 g/t was added to primary rougher feed for two 4 hour blocks indicated as ON. Test results are compared to the 4 hours before the test period indicated as OFF. 30 minute composite samples were taken and assayed for sulphur by Leco. Confidence intervals calculated based on a 60% confidence interval and shown as horizontal lines for each test period.

5.6.4. Summary of results and conclusions drawn from this set of tests.

Analysis of the plant data for a two day (4 hours on and 4 hour off) test with Betacol 346 confirms that lower PGM flotation tails were lower when 50 g/t of Betacol 364 was added as co-collector to the normal flotation reagent suite. It is probable that the concentrate grade will be lower. Based on this conclusion, a long term plant test is recommended.

6. Conclusions and recommendations

Xanthate is not an effective pyrrhotite collector. When Betacol 364, a fatty acid based collector, was used in combination with xanthate, the rate of pyrrhotite and pentlandite recovery was increased although it had little effect on the rate of chalcopyrite recovery.

Betacol 364 is a collector with froth stabilising characteristics. Care should be taken when interpreting laboratory scale batch flotation tests when a collector has frothing characteristics. If it is accepted that the rate of mineral recovery in a flotation process is dependent on the floatability of a mineral (P), the froth recovery (R_f), the superficial gas velocity (J_g) and on the Sauter mean bubble size (D_b) according to Equation 7 (Gorain, et al., 1999), it is necessary to answer the question of whether the rate of recovery increase measured is as a result of an increase in mineral floatability, or as a result of an increase in the frothiness of a test, and thus a change in froth recovery or bubble size.

$$k = P.6.\frac{J_g}{D_b}.R_f \qquad (7)$$

If the change in mineral floatability is as a result of a change in the froth structure or bubble size, then this froth structure and bubble size change may be more effectively achieved with a change in the frother dosage rate or frother type. If the rate of mineral recovery is as a result of a change in mineral floatability, the collector combination has a higher probability of finding commercial application.

It is concluded that the laboratory scale batch flotation test is not sufficient to answer these questions. Additional to the laboratory batch flotation test, it is recommended that a test be done to answer the question of whether a collector change also changes the froth characteristics of a flotation system.

The Bikerman froth stability column, designed for this study, can be used to measure if a collector has froth stabilising or destabilising characteristics. It is recommended that additional to a laboratory batch flotation test, the Bikerman froth stability column should be used to test if a collector has froth stabilising or destabilising characteristics.

If a collector change has no influence on froth stability, then the laboratory scale batch flotation test is sufficient to conclude that if a collector changes, then the mineral floatability also changes. If the test collector has froth stabilising or destabilising characteristics, then an additional test is recommended to conclude if the change in the rate of mineral recovery is at least in part as a result of a change in mineral floatability.

Betacol 364 has froth stabilising characteristics (as measured with the Bikerman froth stability column), and the increase in the rate of pentlandite and pyrrhotite recovery measured when Betacol 364 was used in combination with xanthate against the baseline, on xanthate as sole collector with the laboratory scale batch flotation test, is not sufficient to recommend further commercial testing.

It has been demonstrated that a laboratory scale batch flotation test using a synthetic ore can help to answer the question of whether the rate of pyrrhotite recovery measured when Betacol 364 in combination with xanthate was used as collector is as a result of a change in mineral floatability.

The bubble flux micro flotation cell designed for this study can also be used to test if a change in the rate mineral recovery is as a result of a change in mineral floatability. It has been demonstrated with the bubble flux micro flotation cell that Betacol 364 in combination with xanthate increases the floatability of pyrrhotite.

It is concluded that the increase in the rate of pyrrhotite recovery when Betacol 364 is used in combination with xanthate is as a result of an increase in pyrrhotite floatability and further commercial testing is recommended.

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7. Appendix A: Analytical techniques

7.1. Sodium peroxide digestion with AAS finish

7.1.1. Description of method

A Varian Spectra AA 600 Flame Atomic Absorption Spectrometer was used to assay for Cu, Ni and Fe. For these elements acetylene burner is recommended by the manufacturer.

Sample digestion by sodium peroxide fusion

The sample was prepared for AAS by digesting with sodium peroxide. The method followed is as follows:

- Add 0.5 grams of the ore sample into a porcelain pestle and mortar.
- Grind sample until fine.
- Add 3 grams of sodium peroxide (AR grade) and 1.5 grams of sodium carbonate (AR grade).
- Mix well.
- Transfer to 50 ml Zirconium crucible.
- Melt contents by heating crucible with a Bunsen burner.
- Remove and let melt cool down.
- Add 100.0 ml of distilled water and 20.0 ml of 30% AR grade hydrochloric acid to a 250 ml Erlenmeyer flask. (Total volume 120 ml)
- Put crucible in the liquid (be careful not to spill) and wait for melt to dissolve.
- Remove crucible.
- Transfer solution to sample container for assay with AAS. The concentration value determined by AAS should be back calculated to element mass in the ore sample. For this use 0.5 gram ore digested to 120 ml liquid.

Standard liquids for AA

Liquid standard are purchased from Anacon Analytical.

Reference sample

An ore containing Ni, Cu and Fe are used as standard reference. The sample is digested in duplicate and analysed after every 20 readings.

Flame Atomic Absorption Spectrometer

The settings used to assay for Cu. Ni and Fe using the Varian Spectra AA 600 AA is listed in **Table 27**. A direct acetylene flame and burner was used.

Table 27: Settings used to Assay for Cu, Ni and Fe the Spectra 600 AA.

Metal	Cathode lamp current (mA)	Wavelength (nm)	Slit Width (nm)	Liquid concentration range (mg/l)	Calculated solid concentration in % (0.5 grams digested to 120 mℓ)
Copper	4	324,8	0,5	0.2 – 6.3	0.005 - 0.15
Nickel	4	352.5	0,2	0.3 – 8.3	0.008 - 0.2
Iron	5	372.0	0,2	0.4 – 10	0.01- 0.25

***Also shown is the liquid concentration range and calculated concentration range (in %) for a 0.5 gram ore sample digested to 120 mℓ liquid.*

After calibration of the AAS with standard solution, each sample is analysed and element concentration in ml/l recorded. The minimum and maximum concentration (ml/l) for each element is shown in **Table 27**. If the concentration of the element is lower than the minimum, the AAS is not able to assay the ore sample and a pre concentration method is needed. For this study all assays are higher than the listed minimum values and no pre-concentration was needed. If the element concentration is higher than the limits, the liquid needs to be diluted until the concentration is within this range. Dilution is performed with distilled water and the dilution is taken into account when the value element concentration is calculated from the AA concentration reading.

7.1.2. Validation of method

An ore sample (from a South African nickel flotation plant) was split into nine subsamples by cone and quarter. Three subsamples were submitted to Setpoint laboratories for copper and nickel assay. The remaining six samples were assayed for copper and nickel using the sodium peroxide digestion method and AAS method described above. Each digested ore sample was assayed five times. This data is shown in **Table 39** and **Table 40**.

The relative standard error for copper between digestions is 1.64% and between AAS readings 1.24%. The average copper value assayed was calculated as 0.176% and the average copper value reported by the referee laboratory was 0.178%.

The relative standard error for nickel between digestions is 1.09% and between AAS readings 1.51%. The average nickel value assayed was calculated as 0.386% and the average nickel value reported by the referee laboratory was 0.387%.

It is concluded that the sodium peroxide with AAS finish is suitable to assay ore samples for copper and nickel.

Table 28: Repeat Copper reading using Spectra 600 AAS using the same digested ore sample and repeat AAS reading from six ore digestions by sodium peroxide fusion compared to three referee laboratory assay values.

Copper	Sample digestion repeats (sodium peroxide fusion)							Referee lab. Repeats			
Repeats by AAS	1	2	3	4	5	6	Average	1	2	3	Average
1	0.183	0.171	0.181	0.19	0.18	0.179		0.180	0.177	0.176	
2	0.17	0.16	0.181	0.178	0.18	0.18					
3	0.172	0.156	0.178	0.176	0.183	0.174					
4	0.167	0.171	0.182	0.188	0.181	0.174					
5	0.166	0.16	0.179	0.182	0.172	0.181					
Average	0.172	0.164	0.180	0.183	0.179	0.178	0.176	0.180	0.177	0.176	0.178
Between repeat readings by AAS using the same digested ore sample											
Relative std. error	1.77%	1.90%	0.41%	1.49%	1.05%	0.85%	1.24%				
Between average AAS reading of different digested ore samples											
Relative std. error							1.64%				0.56%

Table 29: Repeat Nickel reading using Spectra 600 AAS using the same digested ore sample and repeat AAS reading from six ore digestions by sodium peroxide fusion compared to three referee laboratory assay values.

Nickel	Sample digestion repeats (sodium peroxide fusion)							Referee lab. Repeats			
Repeats by AAS	1	2	3	4	5	6	Average	1	2	3	Average
1	0.393	0.398	0.386	0.41	0.378	0.364		0.381	0.393	0.388	
2	0.418	0.384	0.391	0.412	0.38	0.378					
3	0.412	0.363	0.389	0.365	0.394	0.385					
4	0.411	0.38	0.365	0.359	0.394	0.367					
5	0.396	0.397	0.379	0.374	0.388	0.384					
Average	0.406	0.384	0.382	0.384	0.387	0.376	0.386	0.381	0.393	0.388	0.387
Between repeat readings by AAS using the same digested ore sample											
Relative std. error	1.20%	1.67%	1.23%	2.94%	0.87%	1.15%	1.51%				
Between average AAS reading of different digested ore samples											
Relative std. error							1.09%				0.91%

7.2. Leco sulphur analyser

7.2.1. Description of method

All sulphur values for this study was done by Setpoint laboratories. A Leco Sulphur analyser was used.

7.2.2. Validation of method

Setpoint laboratories hold an ISO 17025 rating, and are accredited by SANAS. No verification of results was deemed necessary.

7.3. Scanning Electron Microscope with EDS

7.3.1. Preparation of 25 mm polished stub

Milled ore samples are normally cast in resin, polished and coated with gold or carbon for study by SEM (Fandrich, et al., 2007). The Joel JSM 6300 Scanning electron microscope used is equipped to be loaded with 25 mm polished stubs (**Figure 86**).

The stubs are prepared by sub-sampling 0.3 grams of the ore sample and mixing this with approximately 3 ml of cold mounting epoxy resin (from Struers) in a 25 mm polypropylene mounting cup. Approximately 10 ml of resin is cast on top of the sample resin mix. The sample cup is placed in a glass desiccator and a vacuum is applied. The sample is left under vacuum for 30 minutes for air bubbles to dissipate. The sample cup is then placed in a Technomat pressure pot at 400kPa for 12 hours to cure.

The cured sample is removed from the mounting cups and an approximately 1mm section is ground off to expose the cast minerals using Struers grinding equipment (**Figure 85**). The ground epoxy stub is then polished using a sequence of grinding and polishing cloths and stable diamond suspensions from Struers.

The sample is then coated with gold using a gold sputter coater, under vacuum set at 1.4 kV DC for 60 seconds (**Figure 87**).

Figure 85: Photo of Struers grinding and polishing equipment used to prepare 25 mm polished stubs.

Figure 86: Example of 25 mm polished stubs prepared for SEM analysis.

Figure 87: Photo of gold sputter coater used to coat polished stubs with Gold

7.3.2. Description method to study a polished stub using a SEM/EDS.

A 25 mm polished stub prepared as described above, are studied using the Joel 6400 SEM located at Industrial Minerals and Metals Research Institute (IMMRI). The samples were studied using 15-20 kV at a working distance of 25 mm and a magnification of approximately 70 times. Particles and mineral phases were identified with the accessory energy-dispersive X-ray spectroscopy (EDS) systems attached to the SEM. A colour image analyser function (chromatic) of the EDS software was used to intensify the greyscale of the image. The contrast of the image was adjusted to show the background as blue and sulphides as red or white, depending on the molecular weight and thus the nature of the sulphide. All sulphides of the same type will have the same colour. The key or colour of identified sulphides (by EDS) can be used to identify similar sulphides.

Figure 88: Joel JSM -6300 SEM with EXD used. This instrument is located at IMMRI a division of the University of Pretoria

7.4. XRD identification and quantification

7.4.1. Description of method

Crystalline mineral can be identified by XRD (X-ray diffraction). Pyrrhotite, pentlandite and chalcopyrite are crystalline and can be detected and identified by XRD analysis. All XRD work was subcontracted to XRD Analytical and Consulting CC. All samples were analysed with a PAN alytical X'Pert Pro powder diffractometer with X'Celerator detector and variable divergence- and fixed receiving slits with Fe filtered Co-K radiation. The phases were identified using X'Pert Highscore plus software. The relative phase amounts (weight %) was estimated using the Rietveld method (Autoquan program).

8. Appendix B: Methods used to purify xanthate and measure xanthate activity

8.1. Purification of xanthate

8.1.1. Description of method

The method followed to prepare purified xanthate was as follows:

- Weigh approximately 12 grams xanthate powder and dissolve in 20 ml of acetone.
- Filter with filter paper folded in a glass funnel in to a 250 volumetric flask. Discard solids.
- Transfer the filtered liquid into a 250 ml Erlenmeyer flask (flask A).
- Add 20 ml diethyl ether to a 100 ml Erlenmeyer flask (flask B).
- Put both flasks in the refrigerator until liquid has reached approximately 0 degrees.
- Slowly, without mixing, add the diethyl ether (flask B) into the xanthate solution (flask A).
- White xanthate crystal will precipitate and accumulate at the top of the flask.

- Quickly remove the acetone solution on the bottom of the flask by siphoning. Acetone will dissolve the xanthate crystals and must be removed.
- Filter the sample with a Buchner funnel and leave the xanthate crystal to dry.
- Pack the xanthate crystals in a suitable container under a nitrogen jacket.

8.1.2. Validation of method

A batch of SIPX was purified and its activity tested with the acetone and phenolphthalein methods (see Section 8.2) directly after purification (**Table 30**). The activity measured with the two methods compared well and were measured to be 92 % which is reasonable for a single precipitation step.

The activity of a batch of commercial SIPX was tested with both methods and again with the phenolphthalein method 48 hours later (Table 31). The activity of the commercial xanthate was measured to be approximately 76-79% (comparable to manufacturers' claim of 75% when packed) and decreases as expected after 48 hours to 69%.

Therefore it is concluded that both the acetone or phenolphthalein titration methods are valid methods to measure xanthate activity. Xanthate can be purified to in the order of 90% activity with a single acetone crystallisation method described in Section8.1.

Table 30: Activity of purified SIPX measured with the Acetone and phenolphthalein method directly after preparation.

Xanthate (SIPX)	Acetone		Phenolphthalein	
	Flask 1	Flask 1	Flask 2	Flask 2
Weight of Sample (g) **(a)**	5.00	5.00	5.00	5.00
Volume HCl (ml) **(c)**	35	35	50	50
Volume NaOH (ml) **(f)**	6.5	7	23	23.5
Molatity HCl (mol/l) **(d)**	0.100	0.100	0.100	0.100
Molatity NaOH (mol/l) **(g)**	0.091	0.091	0.091	0.091
Molecular weight (g/mol) **(h)**	158.22	158.22	158.22	158.22
% Activity	92	91	92	91

Table 31: Activity of commercial SIPX measured with the acetone and phenolphthalein method directly after preparation and 48 hours later.

Method	Acetone		Phenolphthalein			
Xanthate (SIPX)	0 hrs	0 hrs	0 hrs	0 hrs	48 hrs	48 hrs
Weight of Sample	5.00	5.00	5.00	5.00	5.00	5.00
Volume HCl (ml)	35	35	50	50	50	50
Volume NaOH (ml)	11.5	11	28.8	28.6	31	31.2
Molatity HCl (mol/l)	0.100	0.100	0.100	0.100	0.100	0.100
Molatity NaOH (mol/l)	0.091	0.091	0.091	0.091	0.091	0.091
Molecular weight (g/mol)	158.2	158.2	158.2	158.2	158.2	158.2
% Activity	78	79	75	76	69	68

8.2. Measuring activity of xanthate by titration Acetone and Phenolphthalein titration methods.

Following is a step by step guide for the Acetone and Phenolphthalein titrations methods.

Step 1: Preparing 0.1 M HCl (0.1 M HCl = 3.65 grams HCl / litre)

- Weight of HCl needed = 3.65 * 100 / Purity of acid.
- For 32 % HCl = 3.65 * 100 / 32 = 11.41 grams.
- Thus 11.41 grams 32 % HCl made up to 1 litre solution.

Step 2: Preparing 0.1 M NaOH (0.1 M NaOH = 4.00 grams NaOH /litre)

- Weight of NaOH needed = 4 * 100 / Purity of base.
- For 90 % NaOH = 4* 100 / 90 = 4.44 grams.
- Thus 4.44 grams 90 % NaOH made up to 1 litre solution.

Step 3: Standardising NaOH solution

- Add 35 ml of 0.1M HCl in Erlenmeyer flask.
- Add 250 ml acetone.
- Add Methyl Red indicator
- Titrate to yellow with solution of NaOH.
- The Normality(N) of the NaOH solution is:
  (Volume of HCl / Volume of NaOH) x 0.1

Acetone titration

- Weigh a 5 gram sample into a 250 ml glass Erlenmeyer flask and record to milligrams. (a)
- Add approximately 100 ml acetone to the sample and swirl vigorously until xanthate is dissolved.
- Allow to stand for 5 minutes.
- Filter with filter paper folded in a glass funnel into a 250 ml volumetric flask. Discard solids.
- Dilute filtrate to 250 ml with acetone and mix thoroughly.
- Add 35 ml (c) of 0.1 M HCl (d) (refer to **Table 32** for required volume of acid) to two 250 ml Erlenmeyer flasks (flask #1 and flask #2).
- Pipette a 25 ml aliquot of sample into both flasks.
- Swirl and leave at room temperature for 20 minutes, with occasional swirling.
- Add methyl Red Indicator and titrate to yellow with 0.1 M NaOH (f) and record volume to ml (g).

$$\% \text{ Xanthate} = \frac{(Va \times Na) - (Vb \times Nb) \times M.Wt}{(\text{Wt of sample})}$$

Where: Va = Volume of acid used (ml) -(c)
Na = Normality of acid used – (d)
Vb = Volume of base titrated (ml) – (f)
Nb = Normality of base used - (g)
M.Wt = Molecular weight of xanthate analysed – (h)
Wt of sample = weight of xanthate sample used - (a)

For an example of this method refer to **Table 30**

Phenolphthalein titration

- Weigh accurately 5 grams xanthate in a 250 ml Erlenmeyer flask and record to milligrams (a).
- Dissolve in 30 ml of water.
- Transfer to a 250 ml volumetric flask and dilute to volume.
- Pipette 25 ml aliquot of xanthate solution into 250 ml Erlenmeyer flask containing 100 ml water and 3 drops of Methyl Red Indicator.
- Add 0.1 M HCl drop wise until Methyl Red Indicator changes.
- Add 50 ml (c) of 0.1 M HCl. (d).
- Warm flask for 15 minutes and cool to room temperature.
- Add a further 4 drops of phenolphthalein indicator.
- Titrate with 0.1 M NaOH(g) until colour changes and record in ml (f).

$$\% \text{ Xanthate} = \frac{(Va \times Na) - (Vb \times Nb) \times M.Wt}{(Wt \text{ of sample})}$$

Where: Va = Volume of acid used (ml) -(c)
Na = Normality of acid used – (d)
Vb = Volume of base titrated (ml) – (f)
Nb = Normality of base used - (g)
M.Wt = Molecular weight of xanthate analysed – (h)
Wt of sample = weight of xanthate sample used - (a)

For an example of this method refer to **Table 30**.

Table 32: Molecular weight for a number of xanthate types and acid volume needed for acetone titration.

Xanthate	Acid Volume (ml) (c)	Molecular Weight (h)
Potassium Ethyl Xanthate	35	160.30
Sodium Ethyl Xanthate	40	144.19
Potassium Amyl Xanthate	30	202.38
Sodium Isopropyl Xanthate	35	158.22
Sodium Sec-butyl Xanthate	35	172.25
Sodium Isobutyl Xanthate	35	172.25

8.3. Method to determine the activity of a xanthate solution

Although not relevant to the main argument of this study, a derivative of the preceding section is a method to measure the xanthate concentration of a solution used in a flotation plant and also the activity of the powder used to make the solution. This might be useful for plant metallurgist and is included.

Equipment needed:

Analytical grade HCl
Analytical grade NaOH
Methyl Red Indicator
Phenolphthalein Indicator
2 x 1 liter volumetric flasks
1 x 250 ml volumetric flask
50 ml pipette
100 ml burette
Scale accurate to measure in milligrams
500 ml glass beaker
250 ml Erlenmeyer flask

Preparation:

(a) <u>0.1 M of HCl solution</u>

Weight of HCl needed = 3.65 * 100 / purity of acid

For a 32% HCl purity = 3.65 * 100 / 32 = 11.41 grams

Thus 11.41 grams of 32% HCl made up to a 1-liter solution

- Add 11.41 grams of HCl to 1-liter volumetric flask
- Fill to mark with distilled water

(b) <u>0.1 M of NaOH solution</u>

Weight of NaOH needed = 4 * 100 / purity of acid

For a 90% NaOH purity = 4 * 100 / 90 = 4.44 grams

Thus 4.44 grams of 90% NaOH made up to a 1-liter solution

- Add 4.44 grams of NaOH to 1-liter volumetric flask
- Fill to mark with distilled water

(c) Titration:

- Add 50 ml of 0.1M HCl solution into a 250 ml Erlenmeyer flask
- Add 3 drops of Methyl Red indicator
- Titrate with 0.1M NaOH until colour changes
- Record the volume of NaOH used to titrate = 49 ml

(d) Calculation:

Molarity of NaOH solution $= 0.1M_{HCl} \text{ x } V_{HCl} / V_{NaOH}$

$= 0.1 \text{ x } 50 / 49$

$= \underline{0.102 \text{ mol}}$

Where: V_{HCL} is the HCL volume in ml = 50

V_{NaOH} is the NaOH volume in ml = 49

Use this standardized value for M_{NaOH}

Xanthate reference solution:

- Weigh accurately 5 grams of xanthate (record to mg) and dissolve in 30 ml of water.
- Transfer to a 250 ml volumetric flask and dilute to mark (Vol A).
- Pipette 25 ml (aliquot) of xanthate solution into 500ml glass beaker containing 100 ml of water and add 3 drops of Methyl Red indicator.
- Add 0.1M HCL drop wise until methyl red indicator changes.
- Add 50 ml of 0.1M HCl.

- Warm the flask for 15 minute, and then cool to room temperature.
- Add 4 drops of phenolphthalein indicator.
- Titrate with 0.1M NaOH until colour changes.

$$\text{Factor (F)} = \frac{((\text{VHCL x MHCl})\text{-}(\text{VNaOH x MnaOH})) \text{ x Vol A}}{\text{Weight of sample x Aliquot taken}}$$

$$= \frac{((53.5 \text{ x } 0.1) - (30.5 \text{ x } 0.102)) \text{ x } 0.250}{4.32 \text{ x } 25}$$

$$= \underline{0.005183}$$

Where: V_{HCl} - is the volume in ml = 53.5
V_{NaOH} - is the volume in ml = 30.5
M_{HCl} - is the molarity = 0.100
M_{NaOH} - is the molarity = 0.102
Vol A - final dilution volume in litres = 0.250
Weight of sample in grams = 4.32
Aliquot taken in ml = 25

Activity of a xanthate plant solution

- Pipette 25 ml (aliquot) of xanthate solution into 250 ml Erlenmeyer flask containing 100 ml of water and add 3 drops of Methyl Red indicator.
- Add 0.1M HCL drop wise until methyl red indicator changes.
- Add 50 ml of 0.1M HCl
- Warm the flask for 15 minute, and then cool to room temperature.
- Add 4 drops of phenolphthalein indicator.
- Titrate with 0.1M NaOH until colour changes.

$$\text{Xanthate solution strength (g/l)} = \frac{((\text{VHCL x MHCl})\text{-}(\text{VNaOH x MnaOH}))}{\text{Aliquot taken x Factor (F)}}$$

$$= \frac{((53.5 \text{ x } 0.1) - (30.5 \text{ x } 0.102))}{25 \text{ x } 0.005183}$$

$$= \underline{17.28 \text{ g/l}}$$

Where: V_{HCl} - is the volume in ml = 53.5
V_{NaOH} - is the volume in ml = 30.5
M_{HCl} - is the molarity = 0.100
M_{NaOH} - is the molarity = 0.102
Factor (F) - xanthate factor of reference solution = 0.005183
Aliquot taken in ml = 25

Activity of a powder xanthate

% Xanthate powder activity = Mw x Factor (F) x 100

= 172.25 x 0.005183 x 100

= <u>89.25 %</u>

Where: Mw – is the molecular weight of SIBX = 172.25

Factor (F) - xanthate factor of reference solution = 0.005183

9. Appendix C: Test data

9.1. Bench flotation test with natural ore containing chalcopyrite, pentlandite and pyrrhotite

Table 33: Bench scale flotation test results. Four co-collectors tested against baseline using a D12 bench flotation machine.

		Conc. 1 @ 5 min			Conc. 2 @ 20 min			Conc. 3 @ 40 min			Tails			Call Factor	
Co-Collector	Dosage	Mass	Cu	Ni	Mass	Cu	Ni	Mass	Cu	Ni	Mass	Cu	Ni	Cu	Ni
	g/t	g	%	%	g	%	%	g	%	%	g	%	%	%	%
Betacol 364	20	27	3.33	5.00	50	0.58	2.81	45	0.19	1.27	878	0.030	0.118	93	99
Betacol 364	20	29	3.04	4.96	44	0.56	2.48	44	0.19	1.33	883	0.026	0.131	100	101
Betacol 364	20	21	4.44	4.29	43	0.55	3.52	50	0.19	1.61	887	0.029	0.141	95	97
Betacol 44A	20	54	1.62	2.56	51	0.42	2.99	48	0.14	1.23	847	0.030	0.111	102	97
Betacol 44A	20	35	2.50	3.81	44	0.56	3.81	35	0.19	1.53	886	0.026	0.113	100	95
Betacol 44A	20	70	1.38	2.67	77	0.31	1.87	60	0.10	0.68	794	0.021	0.095	100	97
Betacol 25	20	43	1.13	0.65	67	0.68	1.14	41	0.32	1.75	848	0.049	0.285	96	103
Betacol 25	20	31	1.33	0.62	61	0.71	1.17	51	0.32	1.71	857	0.052	0.264	98	107
Betacol 25	20	25	1.26	0.53	60	0.81	1.18	48	0.32	1.97	867	0.049	0.262	103	107
Betacol 316	20	49	1.69	2.70	59	0.41	2.73	39	0.15	1.22	853	0.024	0.111	107	99
Betacol 316	20	55	1.52	2.28	53	0.47	3.06	39	0.15	1.26	854	0.026	0.115	105	100
Betacol 316	20	84	1.16	2.18	60	0.35	2.24	37	0.13	1.00	820	0.020	0.096	103	100
Baseline		44	1.78	0.99	76	0.51	0.69	58	0.13	2.13	821	0.026	0.252	98	101
Baseline		39	2.20	1.07	70	0.41	0.87	52	0.15	1.93	839	0.030	0.252	97	104
Baseline		33	2.56	1.04	58	0.50	0.81	65	0.13	2.14	844	0.030	0.243	97	101
Baseline		37	2.21	1.03	53	0.61	0.73	79	0.13	1.94	830	0.029	0.240	96	100

***South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, (50 g/t) SIPX (50 g/t), Frother; Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated added at 20 g/t. Assays results by sodium peroxide digestion and AAS finish.*

9.2. Mineral grade and recovery relationships determined by XRD quantification

Table 34: Mineral identification and quantification by XRD for concentrate samples from bench flotation test conducted with two co-collectors and the baseline.

	Feed	Baseline			(Betacol 316)			(Betacol 364)		
		C1 X1	C2 X2	C3 X3	C1 X4	C2 X5	C3 X6	C1 X7	C2 X8	C3 X9
Biotite	6.87	6.96	8.2	9.73	7.73	5.71	4.98	7.8	10.3	5.33
Chalcopyrite	0.97	5.1	1.43	0.92	5.63	1.16	0.7	11.3	1.38	0.72
Chlorite	9.55	17.97	16.39	21.2	17.59	10	11.1	11.64	18.5	12.77
Diopside	6.81	6.27	6.49	5.11	6.23	0.75	2.94	6.52	0.71	1.04
Enstatite	7.65	6.1	8.44	6.76	6.48	8	7.46	10.1	2.21	8.45
Fosterite	4.94	0	0	0	0	0	0	0	0	0
Fayalite	0	0	1.99		0	0	0	0	0	0
Hornblende	17.13	18.63	14.86	13.56	17.03	9.82	11.53	11.81	11.17	12.29
Lizardite	14.79	6.89	9.3	11.06	8.96	12.47	11.78	6.62	9.82	12.84
Magnetite	3.46	3.79	3.58	3.17	3.44	3.55	5.9	3.05	2.52	2.91
Pentlandite	0.76	1.75	1.67	4.03	5.09	7.47	2.71	11.33	6.03	2.02
Plagioclase	14.2	5.54	5.31	2.81	3.82	2.55	1.49	1.38	5.17	2.38
Pyrrhotite-4C	5.66	5.06	6.67	10.17	5.92	27.53	25.94	9.39	20.1	24.01
Quartz	1.42	0.79	1.02	2.36	0.91	0.33	0.34	0.27	1.04	1.67
Talc	5.7	15.16	14.67	9.5	11.16	10.67	13.1	8.82	11.02	13.58

***South African Nickel ore with Chalcopyrite, Pentlandite and Pyrrhotite as the identified sulphide minerals. Grind 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; SEX, 50 g/t SIPX 50 g/t, Frother- Betafroth 436B-30 g/t, Depressant; Betamin 544-250 g/t, Co-collector as indicated at 20 g/t. C1 is concentrate collected from 0-5 min. C2 is concentrate collected from 5-20 min and C3 is concentrate collected from 20-40 min. Results are indicated as relative abundance (mass %)*

9.3. Mineral grade and recovery calculated from assay results

Table 35: Calculated mineral content from Cu, Ni assay (by AAS) and S (by Leco) for bench flotation test.

		Mass	Sulphur				Sulphur			
			Assay Leco (%)				Recovery %	Cum %	Grade %	Cum %
Feed		1000.0	3.28							
(Betacol 364) 364	Tails	878.1	1.320							
	Call		99%							
	Conc 1	27.4	17.20				14.5	14.5	17.2	17.2
	Conc 2	49.8	16.80				25.8	40.3	16.8	16.9
	Conc 3	44.7	17.40				24.0	64.3	17.4	17.1
(Betacol 316) 316	Tails	853.3	1.220							
	Call		99%							
	Conc 1	49.1	10.00				15.1	15.1	10.0	10.0
	Conc 2	58.7	18.80				33.9	49.0	18.8	14.8
	Conc 3	38.9	15.90				19.0	68.0	15.9	15.1
Baseline	Tails	838.6	2.600							
	Call		103%							
	Conc 1	39.0	5.57				6.4	6.4	5.6	5.6
	Conc 2	70.0	5.93				12.3	18.7	5.9	5.8
	Conc 3	52.4	10.80				16.8	35.5	10.8	7.4

**Two co-collectors were tested ((Betacol 364) at 20- g/t plus 100 g/t Xanthate and (Betacol 316) at 20 g/t plus 100 g/t xanthate) using D12 bench flotation equipment. South African nickel ore with chalcopyrite, pentlandite and pyrrhotite as the identified sulphide minerals. Grind: 70% passing 75 micron. Flotation time 5, 20 and 40 minutes. Baseline conditions: Xanthate; (SEX, 50 g/t) SIPX (50 g/t), Frother; Betafroth 436B (30 g/t), Depressant; Betamin 544 (250 g/t), Co-collector as indicated added at 20 g/t. Minerals recoveries calculated assuming: Pentlandite 34.9 % Ni and 35% S, Chalcopyrite 34.6% Cu and 35% S, Pyrrhotite 37.7% S

		Mass	Pentlandite				Pentlandite			
			Assay Mass % Nickel	Calc mass % NI assay	XRD Mass %	Calc S content %	Recovery %	Cum %	Grade %	Cum %
Feed		1000.0	0.43	1.3	1.0	0.4				
Betacol 364	Tails	878.1	0.118	0.4		0.1				
	Call		101%							
	Conc 1	27.4	5.00	15.5	11.3	5.1	31.3	31.3	15.5	15.5
	Conc 2	49.8	2.81	8.7	6.0	2.9	32.0	63.4	8.7	11.1
	Conc 3	44.7	1.27	3.9	2.0	1.3	12.9	76.3	3.9	8.5
Betacol 316	Tails	853.3	0.111	0.3		0.1				
	Call		101%							
	Conc 1	49.1	2.70	8.4	5.1	2.8	30.5	30.5	8.4	8.4
	Conc 2	58.7	2.73	8.5	7.5	2.8	36.9	67.3	8.5	8.4
	Conc 3	38.9	1.22	3.8	2.7	1.2	10.9	78.2	3.8	7.2
Baseline	Tails	838.6	0.252	0.8		0.3				
	Call		96%							
	Conc 1	39.0	1.07	3.3	1.8	1.1	10.1	10.1	3.3	3.3
	Conc 2	70.0	0.87	2.7	1.7	0.9	14.7	24.7	2.7	2.9
	Conc 3	52.4	1.93	6.0	4.0	2.0	24.4	49.1	6.0	3.9

		Mass	Chalcopyrite				Chalcopyrite			
			Assay Mass % Copper	Calc mass % Cu assay	XRD Mass %	Calc S content %	Recovery %	Cum %	Grade %	Cum %
Feed		1000.0	0.14	0.4	0.5	0.1				
Betacol 364	Tails	878.1	0.030	0.1		0.0				
	Call		108%							
	Conc 1	27.4	3.33	9.6	11.3	3.4	59.0	59.0	9.6	9.6
	Conc 2	49.8	0.58	1.7	1.4	0.6	18.6	77.5	1.7	4.5
	Conc 3	44.7	0.19	0.5	0.7	0.2	5.4	82.9	0.5	3.0
Betacol 316	Tails	853.3	0.024	0.1		0.0				
	Call		93%							
	Conc 1	49.1	1.69	4.9	5.6	1.7	62.2	62.2	4.9	4.9
	Conc 2	58.7	0.41	1.2	1.2	0.4	18.2	80.4	1.2	2.9
	Conc 3	38.9	0.15	0.4	0.7	0.1	4.3	84.7	0.4	2.2
Baseline	Tails	838.6	0.030	0.1		0.0				
	Call		103%							
	Conc 1	39.0	2.20	6.4	5.1	2.2	58.4	58.4	6.4	6.4
	Conc 2	70.0	0.41	1.2	1.4	0.4	19.3	77.7	1.2	3.0
	Conc 3	52.4	0.15	0.4	0.9	0.1	5.2	82.9	0.4	2.2

		Mass	Pyrrhotite				Pyrrhotite			
			Calc S content %	Calc mass % S assay	XRD Mass %		Recovery %	Cum %	Grade %	Cum %
Feed		1000.0	2.69	7.1	5.7					
Betacol 364	Tails	878.1	1.169	3.1						
	Call		98%							
	Conc 1	27.4	8.74	23.2	9.4		9.1	9.1	23.2	23.2
	Conc 2	49.8	13.35	35.4	20.1		25.2	34.2	35.4	31.1
	Conc 3	44.7	15.92	42.2	24.0		26.9	61.1	42.2	35.2
Betacol 316	Tails	853.3	1.082	2.9						
	Call		99%							
	Conc 1	49.1	5.53	14.7	5.9		10.1	10.1	14.7	14.7
	Conc 2	58.7	15.59	41.4	27.5		34.2	44.4	41.4	29.2
	Conc 3	38.9	14.51	38.5	25.9		21.1	65.5	38.5	31.7
Baseline	Tails	838.6	2.312	6.1						
	Call		104%							
	Conc 1	39.0	2.25	6.0	5.1		3.1	3.1	6.0	6.0
	Conc 2	70.0	4.63	12.3	6.7		11.6	14.7	12.3	10.0
	Conc 3	52.4	8.68	23.0	10.2		16.2	30.9	23.0	14.2

		Mass	Gangue (Non Sulphids)				Gangue (Non sulphides)			
				Calc mass % S assay	XRD Mass %		Recovery %	Cum %	Grade %	Cum %
Feed		1000.0		91.1	92.8					
Betacol 364	Tails	878.1		96.4						
	Call			100%						
	Conc 1	27.4		51.7	68.0		1.55	1.6	51.7	51.7
	Conc 2	49.8		54.2	72.5		2.96	4.5	54.2	53.3
	Conc 3	44.7		53.3	73.3		2.61	7.1	53.3	53.3
Betacol 316	Tails	853.3		96.7						
	Call			100%						
	Conc 1	49.1		72.1	83.4		3.88	3.9	72.1	72.1
	Conc 2	58.7		49.0	63.8		3.15	7.0	49.0	59.5
	Conc 3	38.9		57.3	70.7		2.45	9.5	57.3	58.9
Baseline	Tails	838.6		93.0						
	Call			100%						
	Conc 1	39.0		84.3	88.1		3.62	3.6	84.3	84.3
	Conc 2	70.0		83.8	90.2		6.46	10.1	83.8	84.0
	Conc 3	52.4		70.6	84.9		4.07	14.2	70.6	79.7

9.4. Investigation into the recovery of chalcopyrite and pyrrhotite from a synthetic ore consisting of value mineral and quartz

Table 36: Laboratory scale flotation test work done using 1.3 litre Denver cell and 100 gram synthetic ore (90% quartz and 10% chalcopyrite).

Co-collector		2 minute concentrate			8 minute concentrate			20 minute concentrate			Tails			Feed		
Type	Dosage	Mass	Cu		Mass	Cu		Mass	Cu		Mass	Cu		Mass	Cu	
	g/t	g	%			%					g	%		g	%	
Baseline	0	4.44	22.9		2.0	14.8		1.6	3.1		92.0	0.3		100	1.53	
316	50	4.33	22.6		2.4	12.6		1.6	1.5		91.7	0.3		100	1.53	
25	50	4.74	22.6		2.0	10.0		1.0	1.4		92.2	0.2		100	1.53	
44A	50	4.44	23.4		2.0	9.9		1.5	2.7		92.0	0.2		100	1.53	
364	50	4.81	24.0		3.9	4.3		2.5	1.8		88.8	0.2		100	1.53	

Co-collector		2 minute concentrate			8 minute concentrate			20 minute concentrate			Tails			Feed		
Type	Dosage	Mass	Chalco	Quartz	Mass	Chalco	Quartz	Mass	Chalco	Quartz	Mass	Chalco	Quartz	Mass	Chalco	Quartz
	g/t	g	%	%		%	%		%	%	g	%	%	g	%	%
Baseline	0	5.3	66.1	33.9	1.6	42.7	57.3	0.6	9.0	91.0	92.5	0.8	99.2	100	4.4	95.6
316	50	6.67	65.3	34.7	1.9	36.4	63.6	1.3	4.4	95.6	90.2	0.7	99.3	100	4.4	95.6
25	50	5.92	65.2	34.8	1.2	28.9	71.1	0.8	4.1	95.9	92.1	0.6	99.4	100	4.4	95.6
44A	50	6.95	67.7	32.3	1.3	28.5	71.5	1.8	7.7	92.3	90.0	0.6	99.4	100	4.4	95.6
364	50	6.47	69.3	30.7	1.6	12.3	87.7	1.8	5.1	94.9	90.1	0.6	99.4	100	4.4	95.6

Summary of copper results

	Dosage	Feed	2 min		8 min		20 min		Call
	g/t	%	Rec (%	Grade	Rec (%	Grade	Rec (%	Grade	%
Baseline	0	1.5	62	22.9	81	20.4	84	17.0	107
316	50	1.5	64	22.6	83	19.1	85	15.7	101
25	50	1.5	73	22.6	86	18.8	87	16.5	96
44A	50	1.5	70	23.4	84	19.2	86	16.0	97
364	50	1.5	74	24.0	85	15.2	88	12.2	102

Summary of Quartz results

	Dosage	Feed	2 min		8 min		20 min		Call
	g/t	%	Rec (%	Grade	Rec (%	Grade	Rec (%	Grade	%
Baseline	0	95.6	1.9	33.9	2.8	39.2	3.4	43.3	99
316	50	95.6	2.5	34.7	3.7	41.1	5.0	48.1	99
25	50	95.6	2.2	34.8	3.1	40.9	3.9	46.5	100
44A	50	95.6	2.4	32.3	3.4	38.6	5.1	48.0	99
364	50	95.6	2.1	30.7	3.6	42.1	5.4	51.6	99

***Ore milled wet milled to 70% passing 75 micron, xanthate dosage (purified SIPX -50 g/t), frother (Betafroth 466) 100 g/t and co-collector as indicated. Assay results by sodium peroxide fusion and AA finish.*

Table 37: Laboratory scale flotation test work done using 1.3 litre Denver cell and 100 gram synthetic ore (90% quartz and 10% pyrrhotite)..

Co-collector		2 minute concentrate			8 minute concentrate			20 minute concentrate			Tails			Feed		
Type	Dosage	Mass	Fe		Mass	Fe		Mass	Fe		Mass	Fe		Mass	Fe	
Type	g/t	g	%			%			%		g	%		g	%	
Baseline	0	1.44	21.47		2.01	9.801		1.59	9.353		95.0	5.704		100	5.83	
316	50	4.33	21.5		2.4	11.27		1.6	10.04		91.7	4.765		100	5.83	
25	50	4.74	29.12		2.02	14.67		1.04	8.506		92.2	4.439		100	5.83	
DTP	50	5.34	20.73		0.96	12.97		1.54	8.755		92.2	4.814		100	5.83	
364	50	4.81	26.33		3.89	9.952		2.51	9.044		88.8	4.235		100	5.83	

Co-collector		2 minute concentrate			8 minute concentrate			20 minute concentrate			Tails			Feed		
Type	Dosage	Mass	Pyrrhot	Quartz	Mass	Pyrrhot	Quartz	Mass	Pyrrhot	Quartz	Mass	Pyrrhot	Quartz	Mass	Pyrrhot	Quartz
	g/t	g	%	%		%	%		%	%	g	%	%	g	%	%
Baseline	0	5.9	36.9	63.1	3.6	16.8	83.2	2.7	16.1	83.9	87.8	9.8	90.2	100	10.0	90.0
316	50	6.87	36.9	63.1	3.2	19.4	80.6	1.2	17.2	82.8	88.7	8.2	91.8	100	10.0	90.0
25	50	7.04	50.0	50.0	2.3	25.2	74.8	1.3	14.6	85.4	89.4	7.6	92.4	100	10.0	90.0
DTP	50	6.35	35.6	64.4	2.4	22.3	77.7	1.2	15.0	85.0	90.0	8.3	91.7	100	10.0	90.0
364	50	7.48	45.2	54.8	2.5	17.1	82.9	1.5	15.5	84.5	88.5	7.3	92.7	100	10.0	90.0

Summary of Iron results

	Dosage	Feed	2 min		8 min		20 min		Call
	g/t	%	Rec (%)	Grade	Rec (%)	Grade	Rec (%	Grade	%
Baseline	0	5.8	5	21.5	8	14.7	11	13.0	104
316	50	5.8	16	21.5	21	17.9	24	16.4	98
25	50	5.8	24	29.1	29	24.8	30	22.6	100
DTP	50	5.8	19	20.7	21	19.5	24	17.4	100
364	50	5.8	22	26.3	29	19.0	33	16.8	97

Summary of Quartz results

	Dosage	Feed (	2 min		8 min		20 min		Call
	g/t	%	Rec (%)	Grade	Rec (%)	Grade	Rec (%	Grade	%
Baseline	0	90	4.2	63	7.6	71	10	74	98
316	50	90	4.8	63	7.7	69	9	70	99
25	50	90	4.0	50	5.9	56	7	60	99
DTP	50	90	4.6	64	6.7	68	8	70	100
364	50	90	4.6	55	6.8	62	8	65	99

***Ore milled wet milled to 70% passing 75 micron, xanthate dosage (purified SIPX -50 g/t), frother (Betafroth 466) 100 g/t and co-collector as indicated. Assay results by sodium peroxide fusion and AA finish.*

9.5. Short pilot plant test of a fatty acid co-collector (Betacol 364) against the baseline condition being; no co-collector.

	Feed			Conc 1			Tails			Call
Test			Mass			Mass			Mass	Cu
No.	Cu(%)	Ni(%)	g/min	Cu(%)	Ni(%)	g/min	Cu(%)	Ni(%)	g/min	%
Baseline	0.185	0.45	2520	2.224	4.182	40.3	0.029	0.187	2340.9	98
Baseline	0.185	0.45	2520	2.274	4.192	37.5	0.022	0.158	2359.0	110
B'col364 50 g/t	0.185	0.45	2520	2.706	4.7	80.6	0.018	0.123	2344.7	107
B'col364 50 g/t	0.185	0.45	2520	2.616	4.348	87.3	0.024	0.134	2312.0	91

	Conc 2			Conc 3			Conc 4			Call
Test	Mass	Cu	Ni	Mass	Cu	Ni	Mass	Cu	Ni	Ni
No.	g/min	g/min	g/min	g/min	g/min	g/min	g/min	g/min	g/min	%
Baseline	110.9	2.92	5.528	16.7	0.18	0.736	11.2	0.08	0.512	88
Baseline	98.5	2.59	5.322	14.1	0.18	0.760	11.0	0.09	0.486	96
B'col364 50 g/t	74.2	1.54	2.604	16.3	0.17	0.731	4.1	0.04	0.164	111
B'col364 50 g/t	98.8	2.06	3.964	17.1	0.18	0.608	4.8	0.05	0.177	97

10. Additional Notes

10.1. Ore sampling

Lotter and Fragomeni (2009) comments that high-confidence flotation testing "methodology is based on two principles: one, to ensure that the ore sample is representative and has been well blended and subsampled; two, to perform the flotation test in sufficient numbers of replicas with appropriate quality controls, so as to improve reproducibility of test data" (Lotter & Fragomeni, 2010).

The Gy sampling theory is still widely used to obtain an opinion of the possible error (relative variance or relative precision) when a sample is taken from an operating plant for flotation test work or to specify how much sample must be taken for a specified relative variance (Minnitt, et al., 2007). The Equation used to calculate the fundamental sampling error (FE) is given as (Minnitt, et al., 2007):

$$\sigma_{FE}^2 = \left(\frac{1}{M_S} - \frac{1}{M_L}\right).f.g.c.l.d_N^3$$

(6)

where:

σ_{FE}^2 The variance of the fundamental sampling error. Relative precision, which is a term commonly used is: $\left(\sqrt{\sigma_{FE}^2}\right).100$ (7)

M_L Mass of sample measured in grams

M_S Mass of the material from which the sample (M_s) is taken measured in grams

f Shape factor. For most ores a value of 0.5 is used (Minnitt, et al., 2007)

g Grain size distribution factor (0 - 1). Value for g is commonly taken as 0.25 for most ores (Minnitt, et al., 2007). (There are refinements suggested to this assumption, but a constant value of 0.25 will be assumed for this study).

c Is the mineralogical composition factor. This factor is approximated by the following equation. $c = \frac{(1-a)}{a} . \left[(1-a) . \rho_m + a . \rho_g\right]$

(8)

ρ_g Density of the gangue (cm^3).

a The grade of the value mineral in the ore in grams of value mineral per gram of ore.

l Liberation factor (0-1). The liberation factor is taken as:

$$l = \left(\frac{d_l}{d_n}\right)^n$$

(9)

With n = 0.5. Values for n between 0 and 3 have been suggested and 1.5 has been shown to be practical and useful for gold ores (Minnitt, et al., 2007). For this study the original value suggested by Gy of 0.5 will be used since this is seen as the most conservative approach.

d_l Nominal size of fully liberated value metal grains (in cm).

d_n Nominal size of the fragments that needs to be sampled (in cm). This is normally taken as the screen size that retains 5% of the lot being sampled (in cm).

The Gy equation was used to plot the relative sampling precision (%) for a chalcopyrite, pentlandite, pyrrhotite and gold ore containing elemental gold particles (figures 1 to 4). The Relative Error is plotted for a nominal sample fragment size (cm) and the size of the sample taken in kg.

A typical sample size of 100 kg taken from the mill feed belt with a top size of 4 cm will, if taken properly, have a calculated sampling precision of approximately 3% for the chalcopyrite, pentlandite and pyrrhotite examples (figures 1-3). For the example where gold is present in elemental particles (if 1 micron size) a significant larger sample needs to be taken to achieve a sampling precision of 3% (figure 4).

This has led to the adaptation of the Gy equation for gold ores where it has been shown that more realistic and useful sampling errors can be calculated for gold ores if $n = 1.5$ (Equation 9) (Minnitt, et al., 2007). Recalculating the relative sampling error using n=1.5 produces relative error values comparable to the values calculated in the examples as can be seen in figure 5.

From the Guy equations (figures 1 to 5) a reasonable guideline is that a properly sampled sample of 100 kg can be taken for batch flotation test, providing the top size of the ore to be sampled is smaller than 4 mm.

For larger top sizes in the sample, larger sampling errors are introduced. A second guideline is thus to sample the mill feed rather than the crusher feed. For plants with an autogenously milling circuit, where the mill feed top size is considerably larger than 4 mm, it is necessary to calculate the relative sampling error and evaluate if high confidence flotation testing can be conducted on this ore sample.

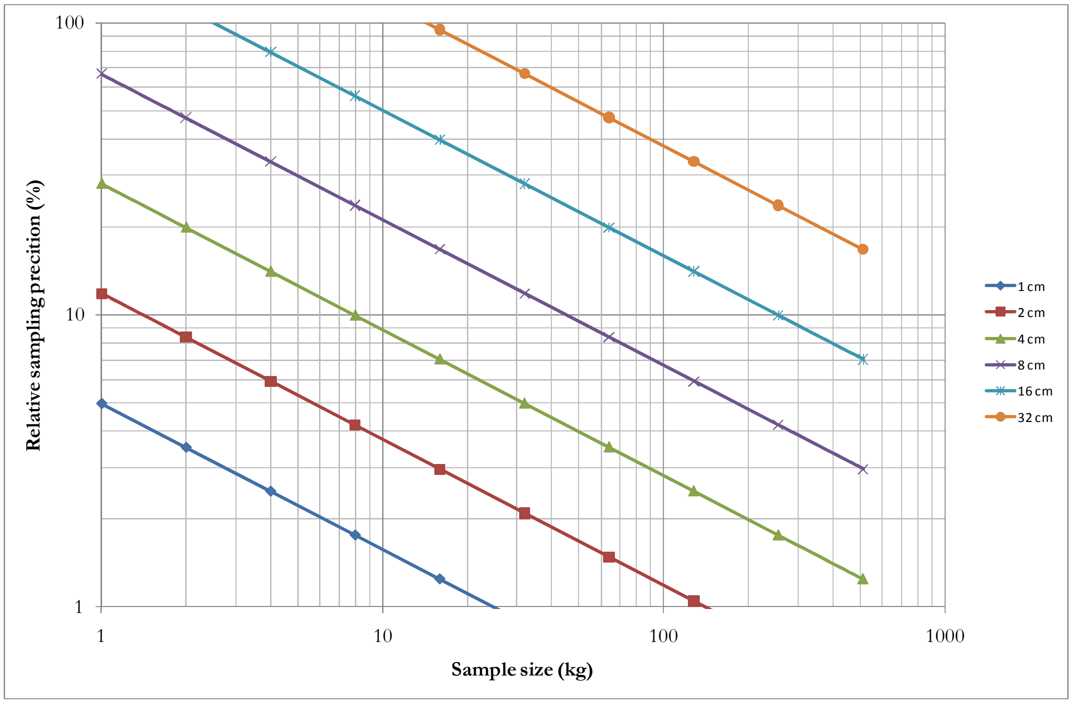

Figure 89: Relative sampling precision calculated using the Gy equation for a chalcopyrite ore example

***(1% Cu, liberation size 50 micron and gangue density 3 g/cm*^{3}*). The figure shows the relative sampling precision (%) for a range of particle top sizes (see legend) and size of sample taken.*

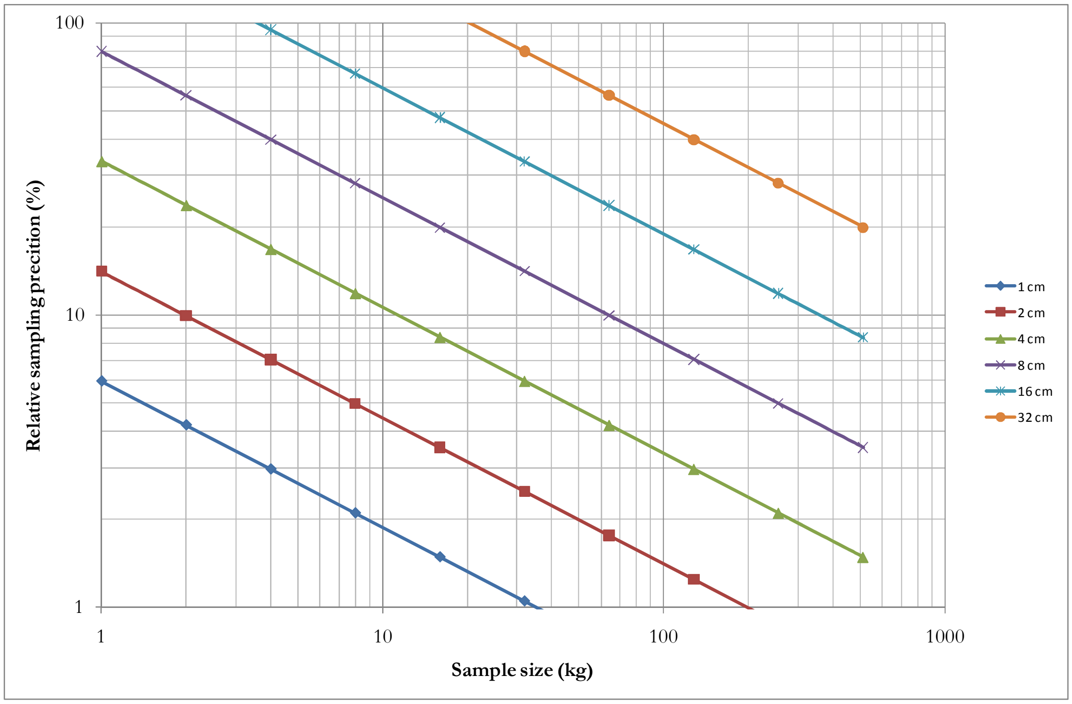

Figure 90: Relative sampling precision calculated using the Gy equation for a pentlandite ore example.

***(0.5% Ni, liberation size 70 micron and gangue density 3 g/cm*^{3}*). The figure shows the relative sampling precision (%) for a range of particle top sizes (see legend) and size of sample taken*

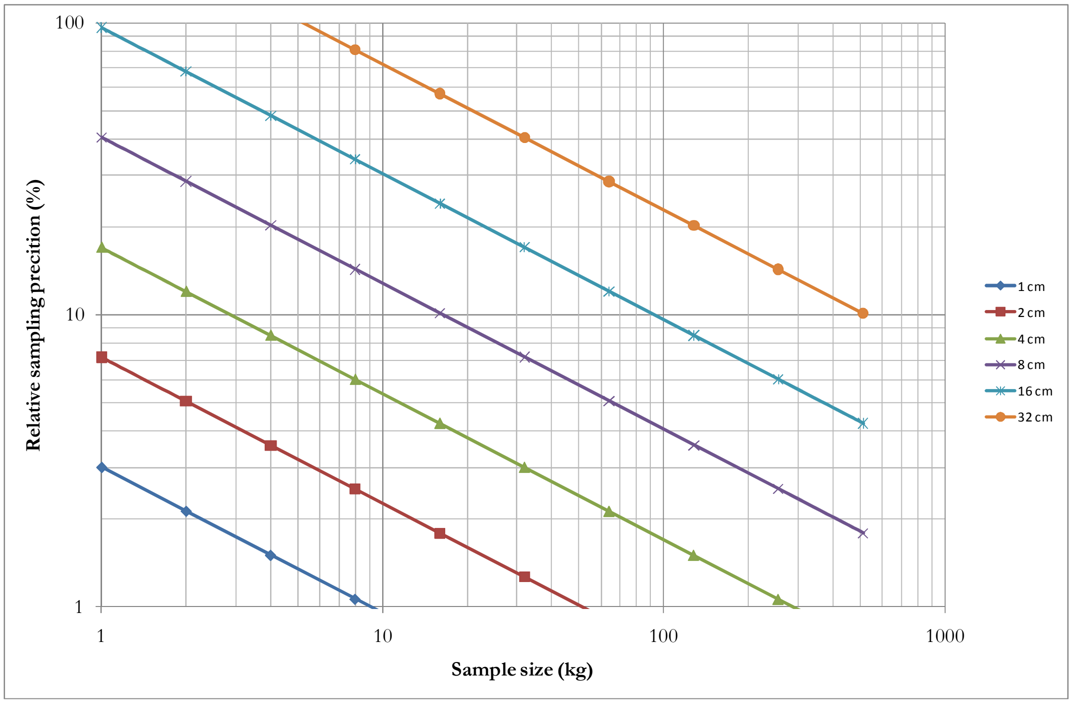

Figure 91: Relative sampling precision calculated using the Gy equation for a pyrrhotite ore example

***(2% S, liberation size 100 micron and gangue density 3 g/cm3). The figure shows the relative sampling precision (%) for a range of particle top sizes (see legend) and size of sample taken.*

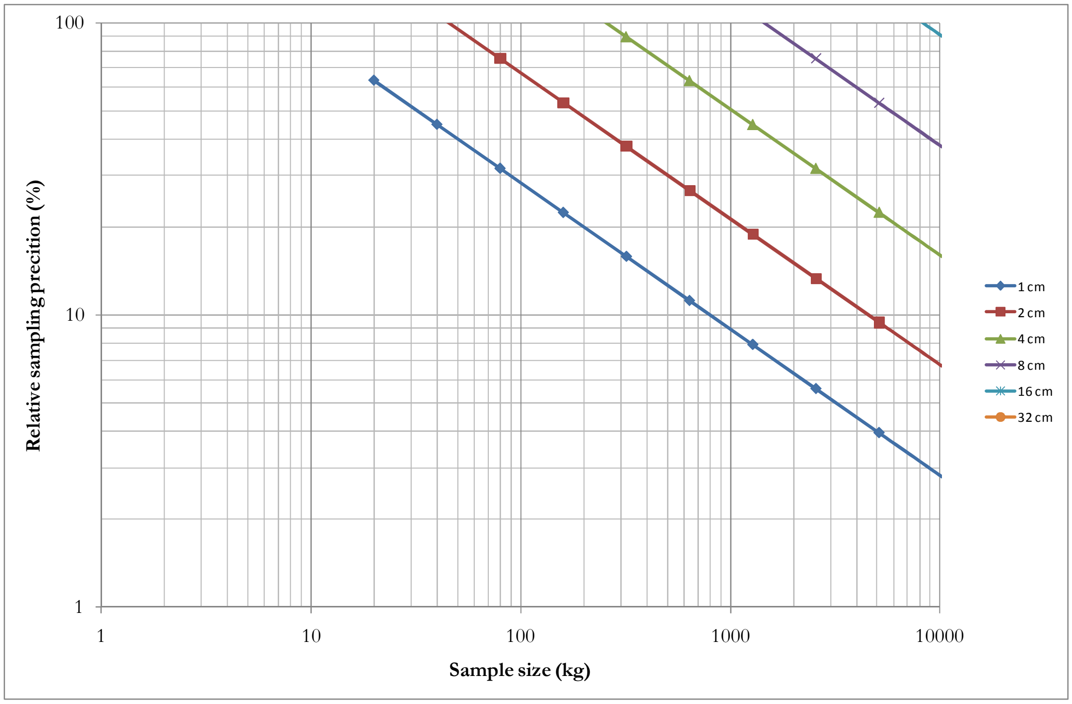

Figure 92: Relative sampling precision calculated using the Gy equation for a gold containing ore sample

***(3 g/t Gold% S, liberation size 1 micron and gangue density 2.7 g/cm*^{3}*). The figure shows the relative sampling precision (%) for a range of particle top sizes (see legend) and size of sample taken.*

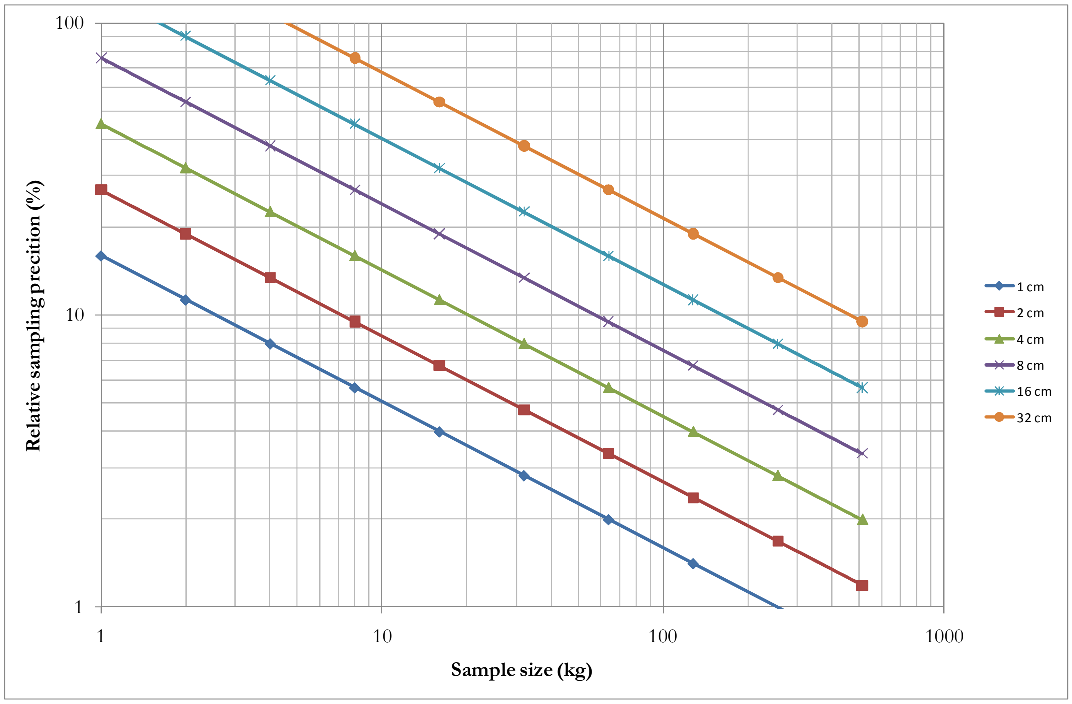

Figure 93: Relative sampling precision calculated using the modified Gy equation (n-=1.5) for a gold containing ore sample

***(3 g/t Gold% S, liberation size 10 micron and gangue density 2.7 g/cm*^{3}*). The figure shows the relative sampling precision (%) for a range of particle top sizes (see legend) and size of sample taken.*

If it is assumed that a batch flotation test is performed on a 1 kg ore sample, milled to 100 microns before flotation, it is apparent that another sampling stage will be involved. The relative sampling error for taking a subsample of variable size from the 100 kg sample (for an ore containing chalcopyrite) is shown in figure 6. The complete 100 kg sample will have to be crushed to smaller than 10 mm and then split into 1 kg subsamples if a relative precision of 3% is targeted (figure 6).

An alternative method is to dry mill a subsample to a size coarser than required, subsample into 1 kg samples and then wet mill prior to flotation (see Section 3.3.5). For this alternative method, the ore sample can be crushed to 20 mm in the primary crushing stage when a subsample of 10 kg is taken for dry milling. For the same chalcopyrite example, assuming the sample is crushed and split into 10 kg subsamples, then milled to 300 microns and split into 1 kg sub-subsamples for wet milling and flotation, a relative sampling error for this sampling stage is given in figure 7.

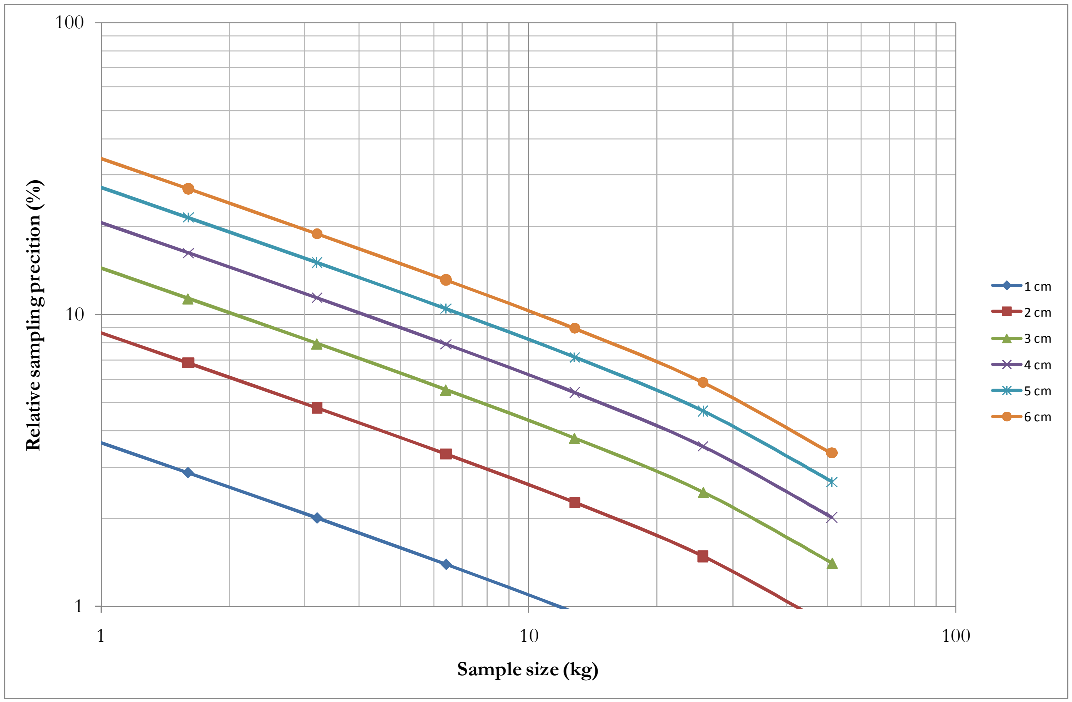

Figure 94: Relative sampling precision calculated for taking a subsample form a 100 kg sample, using the Gy equation for a Chalcopyrite ore example

***(1 % Cu, liberation size 50 micron and gangue density 3 g/cm3). The figure shows the relative sampling precision (%) for a range of particle top size (legend) and size of sample taken.*

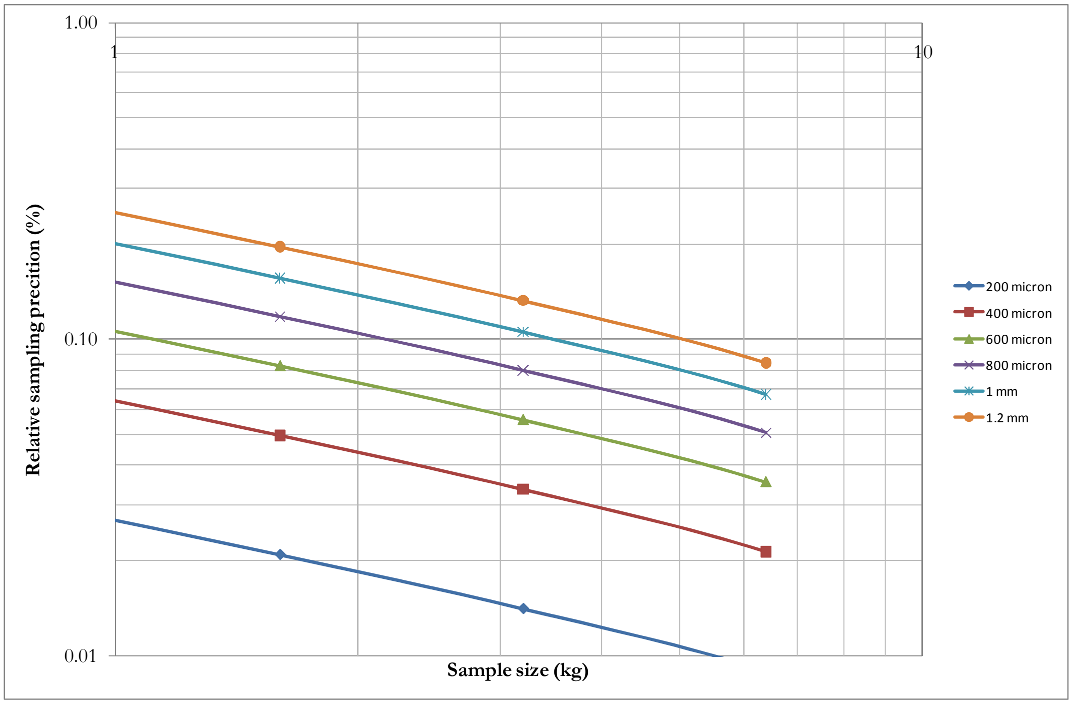

Figure 95 : Relative sampling precision calculated for taking a subsample form a 10 kg sample, using the Gy equation for a Chalcopyrite ore example

***(1 % Cu, liberation size 50 micron and gangue density 3 g/cm*^{3}*). The figure shows the relative sampling precision (%) for a range of particle top size (legend) and size of sample taken.*

The total sampling error is the sum of all the individual sampling errors for each stage. Consider the following two examples for a chalcopyrite ore (1% Cu and liberation size of 50 micron.):

Example 1: A 100 kg sample is taken from the mill feed belt with a top size of 40 mm. This is transported to the laboratory and crushed to 10 mm, and split into 1 kg subsamples ready for batch flotation tests to test a range of collector combinations. The total sampling precision is 3% (figure 1) + 3.5% (figure 6) = 6.5%

Example 2: A 100 sample is taken from the mill feed belt with a top size of 40 mm. This is transported to the laboratory and crushed to 20 mm, and split into 10 kg subsamples. Each 10 kg subsample is dry milled to 300 micron and split into 1 kg subsamples ready for batch flotation tests to test a range of collector combinations. The total sampling precision is 3% (figure 1) + 2.8% (figure 6) + 0.06 (figure 7) = 5.8%

If it's assumed that the sampling method is precise, it seems there is a slight advantage to a three stage sampling method as opposed to a two stage sample method to prepare ore samples for batch flotation tests.

The normal variation of the feed material to the flotation introduces a potentially large sampling error (process variation error) when a sample is prepared for batch flotation tests. The accepted method to reduce this error is to make a composite sample out of a number of small samples taken over a period of time. It is at the least necessary to note the time and date when a sample is taken, if it is not possible to prepare a composite sample.

Equipment and sample taking methods is the last group of errors that is identified by Gy. A belt cut method is normally used to sample the mill feed. A sample splitter or the cone and quarter method is used when a subsample is prepared.

The storage of the sample, before the batch flotation test is performed, has to be considered. Oxidation of the particle surface needs to be reduced or prevented. Some laboratories store ore samples at low temperatures to reduce oxidation (Flemming, 2005). It is necessary to keep the samples dry and away from elevated temperatures.

An alternative method to preserve ore samples is described by Goddard and Brosnahan (1982). According to this technique the ore samples are stored in a container filled with gaseous nitrogen (Goddard & Brosnahan, 1982).