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Start-up tests of OMS's minipilot beneficiation plant and its applicability on the research use

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ABSTRACT

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Abstract

The continuous minipilot beneficiation plant is situated in the Department of Process and Environment Engineering of the University of Oulu. The planning basic for the minipilot plant was the ore beneficiation process of the Pyhäsalmi mine in scale of 1:5000. Outotec Oyj and Schneider Electric together with the experts from the Geological Survey of Finland and Pyhäsalmi mine have been the main planners and equipment suppliers.

The main target of this work was to produce a document giving guidelines for the use of the minipilot beneficiation plant and collect data from the process during various trial runs. The collected data was later used to investigate the dynamics and time delays of the process. All the occurred problems and observations done during the trial runs were note, and are introduced in this thesis. Performed investigations give important knowledge about how to improve the repeatability and continuity of the process. Also the measuring devices and improvements needed for the process are discussed based on the data and knowledge received from the trial runs.

During the trial runs it was occurred that to achieve good concentrate grades and recoveries, all the unit processes including grinding, conditioning and flotation must be working flawlessly. As expected, the results obtained throughout the three trial runs of the minipilot experiments performed in this thesis work highlighted the fact that even in pilot scale ore beneficiation process the individual runs are extremely hard to be replicated. Also the chosen process area, from the grinding to the third cleaning flotation, was too wide. For the future runs, the rougher flotation should be first studied individually in the open loop system by closing the circulating flows from the flotation cells to the conditioner. From there the effects of the changes of different variables could be measured and controlled better. When the rougher unit is analysed, flotation units can be added to the analysis one at a time, until the whole flotation train is investigated and the problematic sections are found.

The results and data collected during the trial runs introduced in this work can be used as a basic data for developing the simulator. Further reliable trial runs are needed to achieve and identify a reliable simulator of the minipilot.

Additional Information

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Oulun yliopisto Teknillinen tiedekunta

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Oulu Mining Schoolin minipilot – rikastuslaitteiston käyttöönotto ja sen soveltuvuus tutkimuskäyttöön

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Tiivistelmä

Jatkuvatoiminen minipilot kokoluokan rikastamo otettiin käyttöön keväällä 2013. Minipilot-rikastamo sijaitsee Oulun Yliopiston prosessi- ja ympäristötekniikan osaston tiloissa. Kokoluokaltaan prosessi on skaalattu alaspäin niin, että se on 1:5000 alkuperäisestä Pyhäsalmen kaivoksen rikastusprosessista. Minipilot-rikastamon Oulun Yliopistolle on toimittanut Outotec Oyj ja automaatiojärjestelmän Schneider Electric. Suunnittelussa ovat lisäksi olleet mukana Geologian tutkimuskeskus sekä Pyhäsalmen kaivos.

Työn tavoitteena oli esitellä ja tuottaa ohjeistus minipilot-rikastamon käyttöön. Työn kokeellinen osio sisältää koeajojen sarjan, jonka aikana pyrittiin keräämään mahdollisimman paljon tietoa prosessista. Kerätyn datan avulla pohdittiin prosessin toimintaa sekä määritettiin tärkeitä prosessiparametreja, kuten aikavakiot eri yksikköprosessien välillä.

Koeajojen aikana ilmeni prosessissa useita ongelmia. Ongelmat johtuivat lähinnä suunnittelu ja mitoitus virheistä. Lisäksi mekaanisia laiterikkoja tapahtui ajojen aikana. Työ sisältää luettelon ongelmista, joita ilmeni ajojen aikana sekä parannusehdotuksia tulevia ajoja varten. Koeajojen tuloksia tarkasteltaessa kävi selväksi, että jopa minipilot kokoluokan rikastusprosessia tarkasteltaessa, on koeajojen toistaminen erittäin haastavaa. Koeajon toistettavuus ja yhtäläisten prosessiolosuhteiden saavuttaminen vaatii jokaisen osaprosessin täydellisen toiminnan.

Tulevaisuudessa palautusvirrat vaahdotuskennoista valmentimeen tulisi katkaista, jotta päästää tarkastelemaan avointa piiriä. Avoimessa piirissä muutokset tapahtuvat nopeammin ja häiriöiden määrä vähenee. Prosessin tutkinta kannattaa aloittaa tarkastelemalla vain esivaahdotuskennon toimintaa. Tämän jälkeen voidaan prosessin tarkastelua laajentaa vaahdotusyksikkö kerrallaan, kunnes lopulta saavutetaan koko prosessin laajuinen tarkastelualue. Näin löydämme prosessin pullonkaulat ja ongelmakohdat, sekä saamme huomattavasti paremman käsityksen yksikköprosessien toiminnasta.

Koeajojen aikana prosessista kerätty data tullaan käyttämään prosessisimulaattorin perusdatana. Tulevaisuudessa täytyy suorittaa useita luotettavia koeajoja prosessisimulaattorin kehittämiseksi.

Muita tietoja

FOREWORD

This thesis was carried out at the University of Oulu from April 2013 to December 2013. It is part of the Oulu Mining School minipilot project that aims to research and develop the usage of the continuous minipilot beneficiation plant.

I would like to express my gratitude to Professor Hannu Kuopanportti from University of Oulu, for his supervision and guidance during my work. I also want to thank my advisors, Jari Ruuska, D.Sc. (Tech.), and Aki Sorsa, D.Sc. (Tech.) for their support and instructions. I would also like to acknowledge my advisor, project manager Ilkka Hynynen, for giving me a chance to work in this interesting project as well as for his invaluable directions and support during my work.

I would also like to thank the staff of OMS’s minipilot, for the help in executing the minipilot and laboratory experiments. Finally, I want to thank my friends and family for supporting me throughout this project.

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1 Introduction

A raw ore have to be treated before it can be used as a final product for industrial or commercial uses. The process chain that transforms the raw ore to the final metal is technically a coherent sequence of processes. Starting from underground where the rock is collected and ending to a metal refining plant where the metal is refined. (Hodouin *et al.* 2001.) In this work the study is limited to cover only a part of the mineral processing including the grinding and flotation stages of the process.

The continuous minipilot beneficiation plant is situated in the Department of Process and Environment Engineering of the University of Oulu. The first exercise runs were completed during the spring and the summer of 2013. The planning basic for the minipilot plant was the ore beneficiation process of the Pyhäsalmi mine in scale of 1:5000. Outotec Oyj and Schneider Electric together with the experts from the Geological Survey of Finland and Pyhäsalmi mine have been the main planners and equipment suppliers.

In the theoretical part of this thesis, the effects of different variables to the subprocesses (grinding, conditioning and flotation) of the ore beneficiation process are introduced. Also the measuring devices used to measure these variables are discussed. Introduction to the geology, the mineralogy and the beneficiation process of the Pyhäsalmi mine is also included.

The main target of this work was to produce a document giving guidelines for the use of the minipilot ore beneficiation plant and collect data from the process during various trial runs. The collected data was later used to investigate the dynamics and time delays of the process. All the occurred problems and observations done during the trial runs were note, and are introduced in this thesis. One target of this work was to introduce, based on the trial runs, which measurement devices would be practical and improve the repeatability and continuity of the process.

2 Theoretical framework

This study bases its theoretical foundation on four areas: the geological and mineralogical aspects of Pyhäsalmi ore, description of the Pyhäsalmi ore beneficiation process, description of continuous minipilot size beneficiation process in the University of Oulu and used measurements in sulphide ore beneficiation. These theories are applied with the objective of better understanding of needed measurements in minipilot size ore beneficiation.

In order to understand, which measurements are needed in the continuous minipilot size sulphide ore beneficiation plant, first it is necessary to understand the background and properties of Pyhäsalmi ore, as well as to understand the main principles of the ore beneficiation process used in the Pyhäsalmi mine. Second, the minipilot process is shortly described and similarities between the Pyhäsalmi ore beneficiation process and the minipilot process are highlighted. Third, some widely used methods for measuring different variables from the sulphide ore beneficiation processes are introduced. Only those measuring devices, which are applicable to the minipilot process, are introduced. The third part is divided in three chapters where grinding, conditioning and flotation are observed separately to point out the complexity of the beneficiation process.

2.1 Geology

The Pyhäsalmi mine is one of the deepest mines in Europe and at the moment mining is done at levels, which are 1050 to 1440 meters below ground (Huuskonen *et al.* 2008). The Pyhäsalmi massive Zn-Cu-pyrite deposit is located in central Finland within the 1.88-1.93 Ga old volcano-sedimentary Vihanti-Pyhäsalmi VMS domain regarded as a part of a Paleoproterozoic primitive arc complex at the margin of an Archean basement complex. The ore in Ruotanen formation is hosted by altered felsic tuffs, breccias and quartz-porphyries except in the deep ore zone where unaltered mixed felsic volcanics host the ore. (Balint *et al.* 2002.)

The enveloping alteration zone has a length of 4 kilometers and is 1 kilometer at its widest. At the surface the S-shaped ore body is 650 meters wide becoming narrower to the south and north ends. The ore continues down vertically simultaneously becoming shorter at the same time. At the +1000 level the ore almost disappears being only 150 meters long and 20 meters wide. Below the +1000 level the ore body turns strongly to the east and then to the south forming a tight fold shaped as a horseshoe. The plunge of the southern end of the ore also turns from vertical to 45 degree southwest. (Balint *et al.* 2002.) A geological map of the Pyhäsalmi mine area is shown in Figure 1.

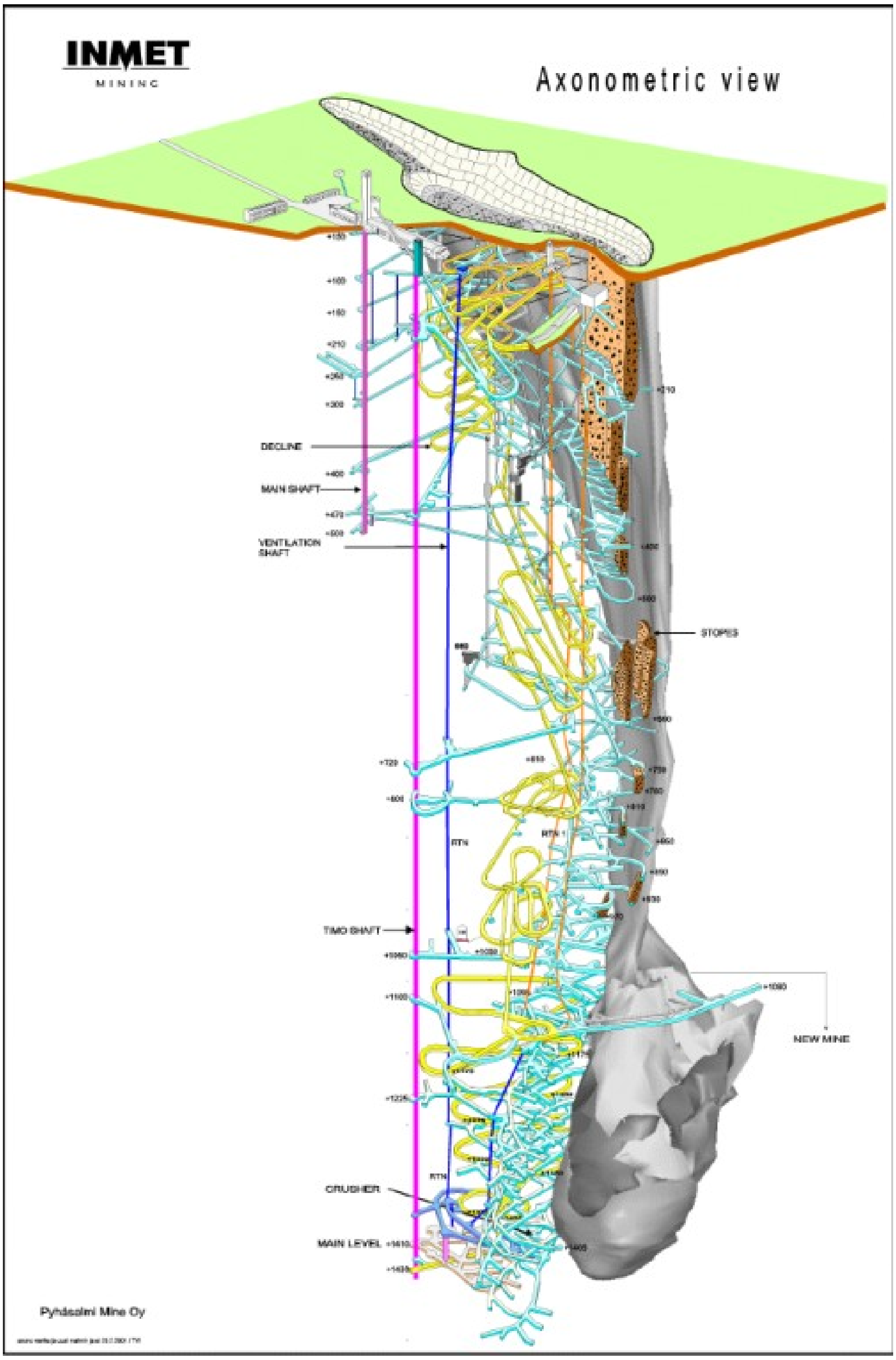

Figure 1. A geological map of the Pyhäsalmi mine area. (Balint *et al.* 2002.)

2.2 Mineralogy

The Pyhäsalmi ore contains about 75% sulphides and is classified as massive sulphide ore. Main minerals of the Pyhäsalmi ore are pyrite (65%), chalcopyrite (3%), sphalerite (4%) and pyrrhotite (3%). The main accessory minerals are galena, magnetite, arsenopyrite and sulphosalts. The Pyhäsalmi ore is very coarse grained. (Balint *et al.* 2002.)

Following ore types can be recognized (Balint *et al.* 2002):

- massive pyrite,
- massive chalcopyrite-pyrite,
- massive chalcopyrite-sphalerite-pyrite,
- massive banded sphalerite-pyrite,
- semi-massive breccia sphalerite-barite-carbonate-pyrite,
- host rock inclusion with breccia chalcopyrite and
- pyrrhotite bearing varieties.

In the following chapters the commercial names of valuables are used, even though the mineral and the metal names have different meanings. This simplify is done with the objective to make the text easier to understand and, because the metal names are widely used in descriptions of beneficiation processes. In this thesis, chalcopyrite is copper and sphalerite is zinc.

2.3 Ore beneficiation process in the Pyhäsalmi mine

The ore beneficiation process starts underground where the ore is crushed. For primary crushing single-stage underground jaw crusher is being used. After primary crushing the ore is hoisted to the fine crushing plant, which is of the vertical type; in practice little more than screening is done at the fine crushing plant. The fine crushing of ore is done on the surface. (Balint *et al.* 2002.) In this work, the object is to observe the ore beneficiation process from grinding to zinc flotation. Thus, mining, crushing and pyrite flotation are not introduced in the following chapters.

In the following chapters, the theoretical perspective of each stage, between grinding to zinc flotation, are first studied. After each theoretical part the method used in the Pyhäsalmi mine is introduced. Grinding is studied first and is followed by investigations of copper conditioning, flotation and chemical reagents used in the copper circuit. Next zinc conditioning, flotation and chemical reagents used in the zinc circuit are observed. Theoretical aspects are explained so that a reader can understand what happens to the particles in a mineral scale.

2.3.1 Grinding

Most minerals are finely disseminated and intimately associated with the gangue, therefore they must be initially “unlocked” or “liberated” before a separation can be undertaken (Wills *et al.* 2006). In most cases this is achieved by grinding. The grinding process is a size reduction operation and is used in the mineral industry to liberate the valuable minerals from the gangue (Pomerleau *et al.* 2000). Coarse material will decrease a recovery of valuable minerals in the flotation due the attached gangue. Fine material increases the energy consumption in the grinding mills and it also has negative effects on flotation (Huuskonen *et al.* 2008). The size limits are not exact but for many mineral systems, the fines are particles that are below 5–10 µm, and the coarse sizes are above 100 µm approximately (Tabosa & Rubio 2010).

In Pyhäsalmi, the ore is ground in three stages. A lump mill is use for primary grinding and Secondary and tertiary grindings are done at ball mills. All the grinding mills have rubber lines and secondary and tertiary mills are in a closed circuit with cyclone classifiers. After grinding the ore fineness is approximately 3% +210 µm and 65% -74 µm. (Balint *et al.* 2002.)

2.3.2 Copper conditioning

Conditioning refers, in conventional meaning, the mechanical pre-treatments of mineral slurry in the presence of specific chemical reagents. The function of these chemical reagents is that minerals would have as beneficial conditions as possible and enough time to adjust to those reactions that are desirable in flotation. (Hukki 1964.)

In the Pyhäsalmi ore beneficiation process there are no actual conditioning units before the copper flotation, because reagent chemicals are fed to the grinding mills. This ensures decent conditioning times for each reagent. Reagents used in the copper flotation are SylvaPine as a frother, isobutyl xanthate (IBX) as a collector, zinc sulphate as a depressant and milk of lime for pH control. (Huuskonen *et al.* 2008.)

2.3.3 Flotation

Flotation is a physico-chemical separation process where the difference in the surface properties of the valuable minerals and the unwanted gangue minerals are utilized. Flotation process where material is being recovered from the pulp comprises three mechanisms: selective attachment to air bubbles, entrainment in the water which passes through the froth and physical entrapment between particles in the froth attached to air bubbles. The attachment of valuable minerals to air bubbles is the most important mechanism and represents the majority of particles that are recovered from the flotation circuit as concentrate. In industrial flotation plant practice, entrainment of gangue is normal and hence a use of only a single flotation is uncommon. Often at least rougher and cleaner flotations are required to reach an economically acceptable quality of valuable minerals in the final product. (Wills *et al.* 2006.)

After conditioning the ore sludge goes to the flotation where valuable minerals are separated from the gangue (Huuskonen *et al.* 2008). The flotation process in the Pyhäsalmi mine contains three sequential flotation circuits. Copper is separated first and followed by the zinc flotation circuit. Finally, pyrite is separated from the unwanted gangue. (Balint *et al.* 2002.) The pyrite flotation is not studied in the following chapters.

2.3.4 Copper flotation

The main mineral of copper in Pyhäsalmi ore is chalcopyrite, which can be easily separated from gangue in the flotation. Small amounts of gold and silver are also collected in the copper flotation. (Huuskonen *et al.* 2008.)

A flow sheet of the Pyhäsalmi mine copper flotation circuit is shown in Figure 2. Copper flotation includes a rougher flotation followed by a four-step cleaning, where the grade of the copper concentrate from the rougher is increased. Tailings from the first three cleaning units are recycled back to the rougher flotation and the final tailing is fed to the previous cleaner. The final copper concentrate is collected after the fourth cleaning flotation. The rougher tailing is fed to the scavenger flotation. In the scavenger flotation recovery of copper is increased and copper concentrate is recycled back to the cleaning. The scavenger tailing (copper waste) is pumped to a zinc conditioner and prepared for the zinc flotation. (Huuskonen *et al.* 2008.)

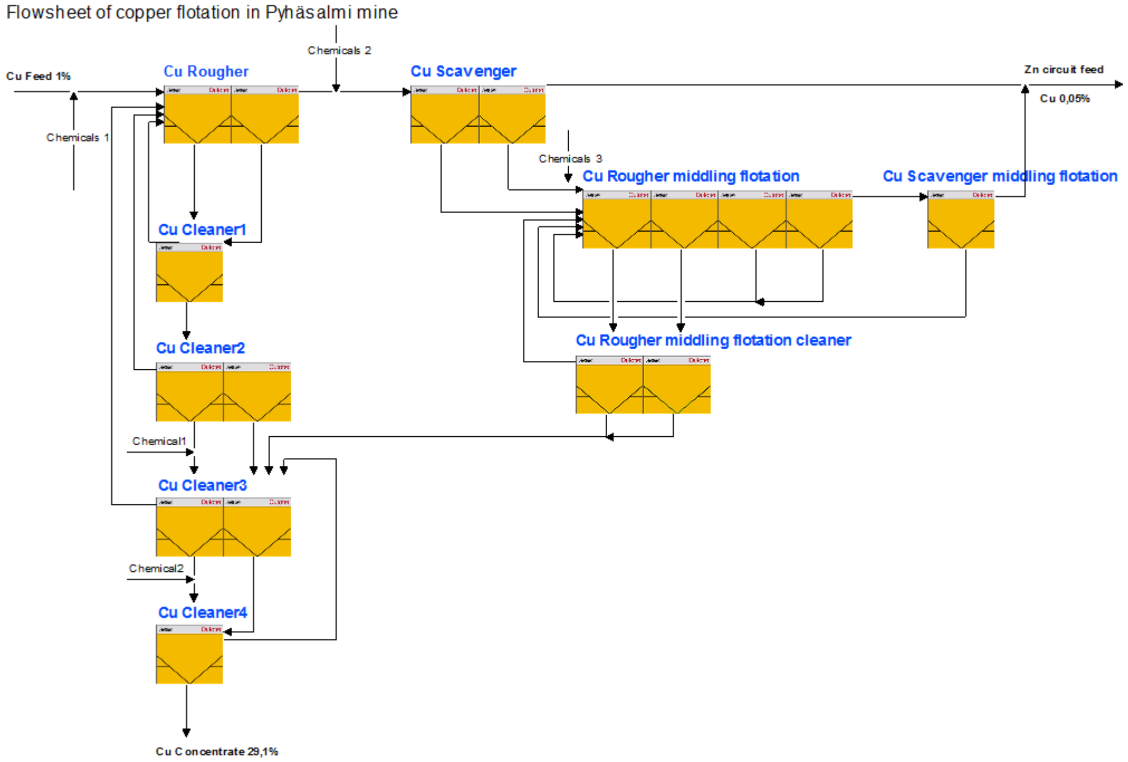

Figure 2. Copper flotation circuit of the Pyhäsalmi mine.

2.3.5 Used reagents in the copper flotation

Hydrophobicity has to be imparted to the surfaces of most minerals in order to float them. This is achieved with a use of surfactants known as collectors. Collectors are added to the slurry in different parts of the flotation process. (Wills *et al.* 2006.) The most important factor affecting the floatability of sulphide minerals is the adsorption of the collector on the solid particles (Hadler *et al.* 2005). Xanthates are normally used as collectors of sulphide minerals (Kuopanportti *et al.* 1997).

A used collector in the copper flotation in Pyhäsalmi is isobutyl xanthate (IBX). Isobutyl xanthate will attached to the surface of the copper particles, making them hydrophobic. The hydrophobic copper particles attach to air bubbles, which rise to the surface. (Huuskonen *et al.* 2008.)

Frothers are added to stabilize a bubble formation in the pulp phase, to create a reasonably stable froth to allow selective drainage from the froth of entrained gangue, and to increase flotation kinetics (Wills *et al.* 2006). Sylva pine is used as a frother in the Pyhäsalmi mine (Huuskonen *et al.* 2008).

Depression is used to increase the selectivity of flotation by rendering certain minerals hydrophilic (water-avid), thus preventing their flotation (Wills *et al.* 2006). In Pyhäsalmi zinc sulphate is used as a depressant for zinc particles and cyanide for pyrite (Huuskonen *et al.* 2008).

Lime is very widely used in mineral processes to regulate slurry alkalinity. Lime is used in the form of milk of lime, which is suspension of calcium hydroxide particles in a saturated aqueous solution. Milk of lime is normally added to the slurry prior to flotation to precipitate heavy metal ions from solution. In this sense, the milk of lime is acting as a deactivator, as these heavy metal ions can activate sphalerite and pyrite and prevent their selective flotation from copper minerals. When xanthates are used as collectors, sufficient alkali will depress almost any sulphide mineral. There is a certain pH value for different concentrations of xanthate where below any given mineral will float, and above where it will not float. (Wills *et al.* 2006.) Milk of lime is used to control pH and depress pyrite in the copper flotation at the Pyhäsalmi mine. The optimal pH value for the copper flotation in Pyhäsalmi is 12. (Huuskonen *et al.* 2008.)

2.3.6 Zinc conditioning

A flow sheet of the Pyhäsalmi mine zinc flotation process is shown in Figure 3. The output (copper waste) from the copper flotation is taken to the zinc flotation. The zinc flotation starts with three separate conditioning units as seen from Figure 3. This ensures decent conditioning times for reagents. Especially copper sulphate adsorption is quite time consuming hence it is added already at a sump of the copper flotation. Collectors and milk of lime are added in conditioner units. (Huuskonen *et al.* 2008.)

2.3.7 Zinc flotation

A flow sheet of the Pyhäsalmi mine zinc flotation circuit is shown in Figure 3. Zinc flotation includes a rougher flotation followed by a three-step cleaning flotation, where the grade of zinc concentrate from the rougher flotation in increased. Tailings from the first two cleaning stages are fed to a cyclone. Also concentrates from scavenger units, concentrates from last two rougher middling flotation units and concentrates from a scavenger middling flotation are pumped to the cyclone. Coarse material from the cyclone is taken to regrinding, where a ball mill is used to clean surfaces of valuable minerals and reduce the particle size of large particles. Discharge from the regrinding, as well as overflow from the cyclone, is taken to the rougher middling flotation. Tailings from the middling flotation and scavenger middling flotation are fed, together with the scavenger tailing, out from the zinc flotation as a zinc waste. (Huuskonen *et al.* 2008.)

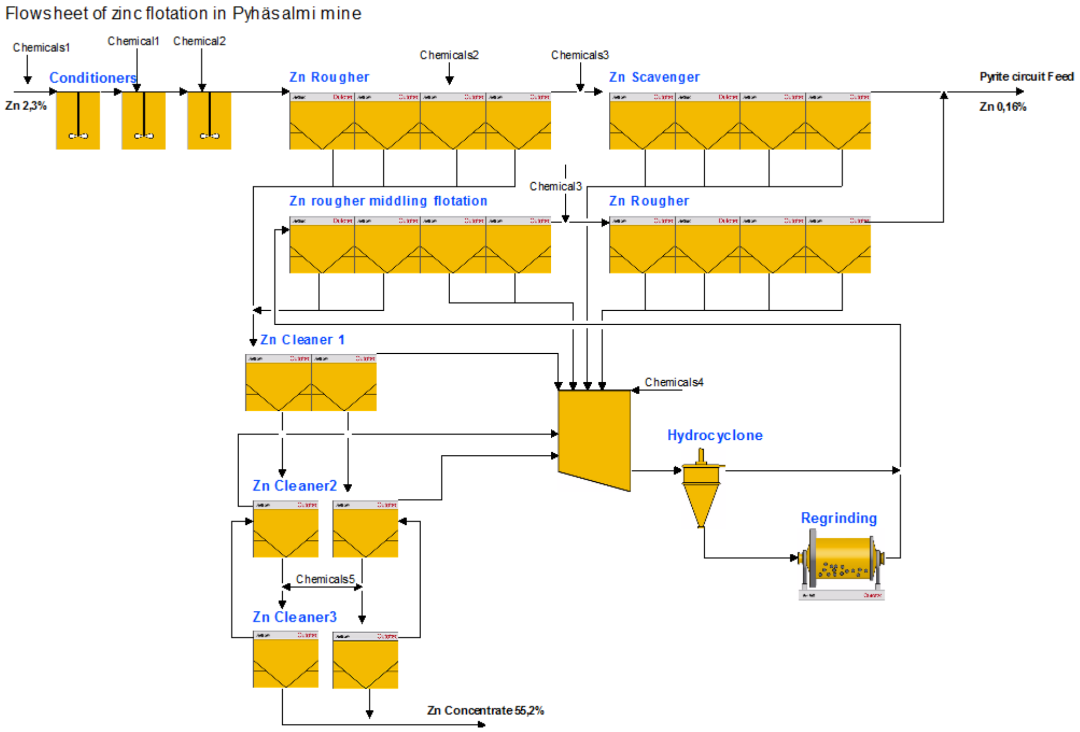

Figure 3. Zinc flotation circuit of the Pyhäsalmi mine.

2.3.8 Used reagents in the zinc flotation

In the zinc flotation isobutyl xanthate (IBX) is used as a collector, SylvaPine as a frother and copper sulphide as an activator. Milk of lime is used to control pH and depress pyrite in the zinc flotation. The optimal pH value for the zinc flotation in Pyhäsalmi is 12. Also a small quantity of cyanide is used as pyrite depressant. (Huuskonen *et al.* 2008.)

Xanthate collectors do not float zinc particles satisfactorily, since the collector products formed, such as zinc xanthate, are comparatively soluble in water. Hence xanthate does not provide a hydrophobic film around the mineral. Large quantities of long-chain xanthates can be used to improve the floatability of minerals, but more satisfactory method is to use an activator, which is readily soluble and dissociates into copper ions in solution. The activator forms a film of copper sulphide molecules on the mineral surface. Xanthate collectors can then attach to the mineral surface and make the particles hydrophobic. (Wills *et al.* 2006.) Copper sulphate is used as an activator in the Pyhäsalmi mine zinc flotation (Huuskonen *et al.* 2008).

2.4 Continuous minipilot beneficiation plant

The continuous minipilot beneficiation plant, shown in Figure 4, is situated in the Department of Process and Environment Engineering of the University of Oulu. The planning basic for the minipilot plant was the ore beneficiation process of the Pyhäsalmi mine in scale of 1:5000. Outotec Oyj and Schneider Electric together with the experts from the Geological Survey of Finland and Pyhäsalmi mine have been the main planners and equipment suppliers. Later in this work the continuous minipilot beneficiation plant is called the minipilot.

Figure 4. Continuous minipilot beneficiation plant.

Even though the minipilot is much smaller than full-scale beneficiation plant, particle sizes and phenomenon are the same. For this reason many different aspects can be studied by the minipilot process. Some of the research areas carried out with the minipilot beneficiation plant are listed below *(Poster, Innovation environment for mineral processing)*:

- process basic research including; chemical and physical phenomenon research,
- process development including
  - effectiveness of industrial management,
  - energy,
  - environment,
  - utilization of waste rock and
  - occupational safety
- instrumentation, process automation,
- mechanical engineering and diagnostics and
- process management

The minipilot has a maximum capacity of 30 kg/h, for Pyhäsalmi ore, when the grinding circuit and flotation circuit are used continuously. At present the ore from the

Pyhäsalmi mine is used in the process and the objective is to separate copper and zinc from the gangue. For the Pyhäsalmi ore, the objective is to reach 26% copper concentrate and 53% zinc concentrate.

2.4.1 Process description

The continuous minipilot beneficiation plant contains both the grinding circuit and the flotation circuit as shown in Figure 4. The grinding circuit has two stages of grinding: a rod mill in an open circuit and a ball mill in a closed circuit with a screw classifier. The flotation flow sheet consists of two flotation circuits, one for copper and another for zinc flotation.

Before ore can be used in the minipilot, it has to be primary crushed in Pyhäsalmi and carried to the minipilot in containers. Ore is then sieved to particle size -3.6mm and fed to the silo. From the silo, ore is fed continuously to the rod mill by a screw conveyer. Additional water and lime are also fed to the rod mill at the same time. A continuous flow of the slurry from the rod mill is discharged to the screw classifier. In the screw classifier, the slurry is separated into an underflow stream (the coarser particle) and an overflow stream (the finer particles). The objective of the grinding circuit is to reduce particle size to D80 -80µm; the coarser particles go to the ball mill for regrinding and the finer particles are pumped into the first conditioner.

In the first conditioner, the slurry is mixed and all reagents for the copper flotation are fed. Reagents used in the first conditioner are Dowfroth as a frother, isobutyl xanthate (IBX) as a collector, zinc sulphate as a depressant and milk of lime for pH control. The first conditioner is operated at pH 12.

The flow sheet of the minipilot plant copper flotation is shown in Figure 5. Copper flotation includes a rougher flotation followed by a three-step cleaning in a closed circuit. Each cleaning tailing is returned to previous cleaner step except tailing from the first cleaning which is recycled back to the first conditioner. The final copper concentrate is collected after the third cleaning flotation. The tailing from the rougher is fed to a scavenger flotation. The scavenger concentrate is split to high copper concentrate and low copper concentrate. The high copper concentrate is fed to the primary cleaning flotation and low copper concentrate is recycled back to the first conditioner. The scavenger tailing is pumped to a second conditioner as a copper tailing.

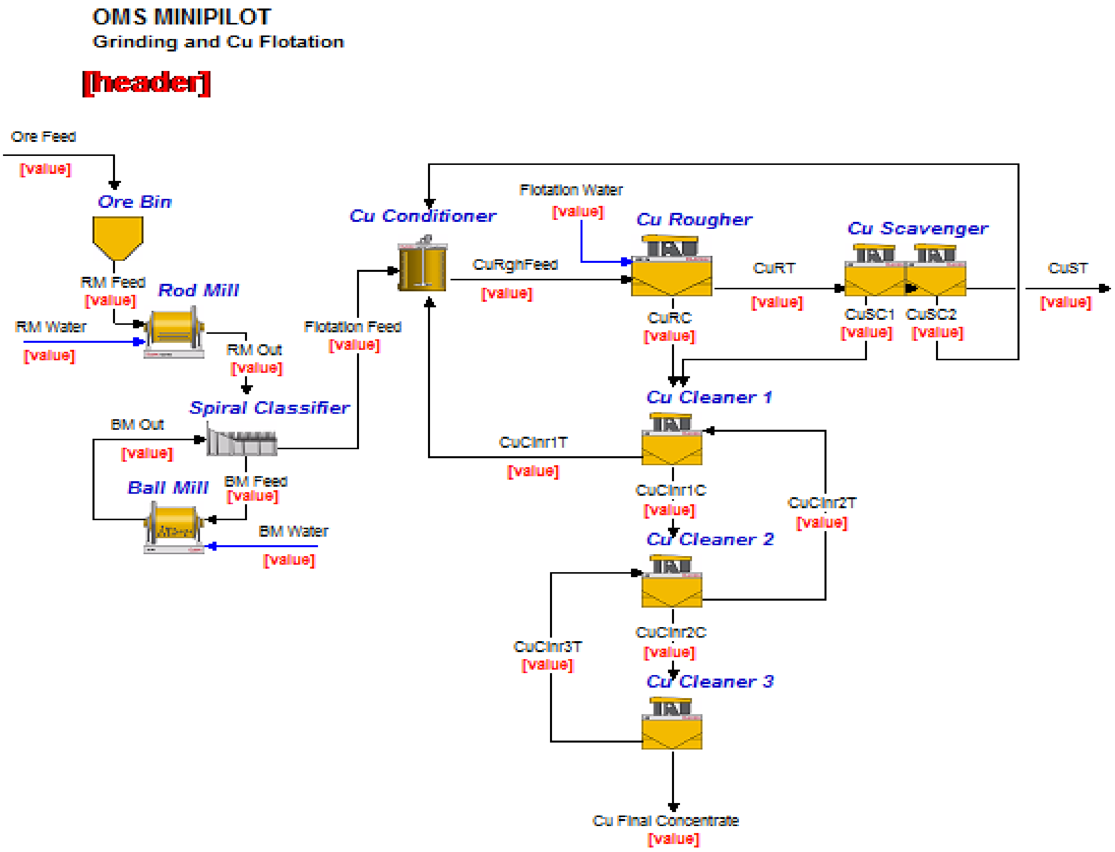

Figure 5. The flow sheet of the minipilot plant grinding circuit and copper flotation.

In the second conditioner copper tailing is mixed and additional reagents are added. Reagents used in the second conditioner are Dowfroth as a frother, isobutyl xanthate (IBX) as a collector, copper sulphate as an activator and milk of lime for pH control. The second conditioner is operated at pH 12.

The zinc flotation is consisted of rougher flotation followed by a four-step cleaning flotation as shown in Figure 6. The rougher tailing is fed to the scavenger flotation where the remaining zinc is separated from gangue. Concentrate from the scavenger is fed to regrinding where a small ball mill is used. Regrinding discharge is pumped to the middlings flotation. The middlings flotation concentrate is recycled back to the zinc conditioner. Tailing from the middlings flotation together with tailing from the scavenger is zinc tailing, which is the input of the pyrite flotation in the Pyhäsalmi beneficiation process, but in the OMS minipilot threated as a final tailing. The final zinc concentrate is collected from the fourth cleaning flotation.

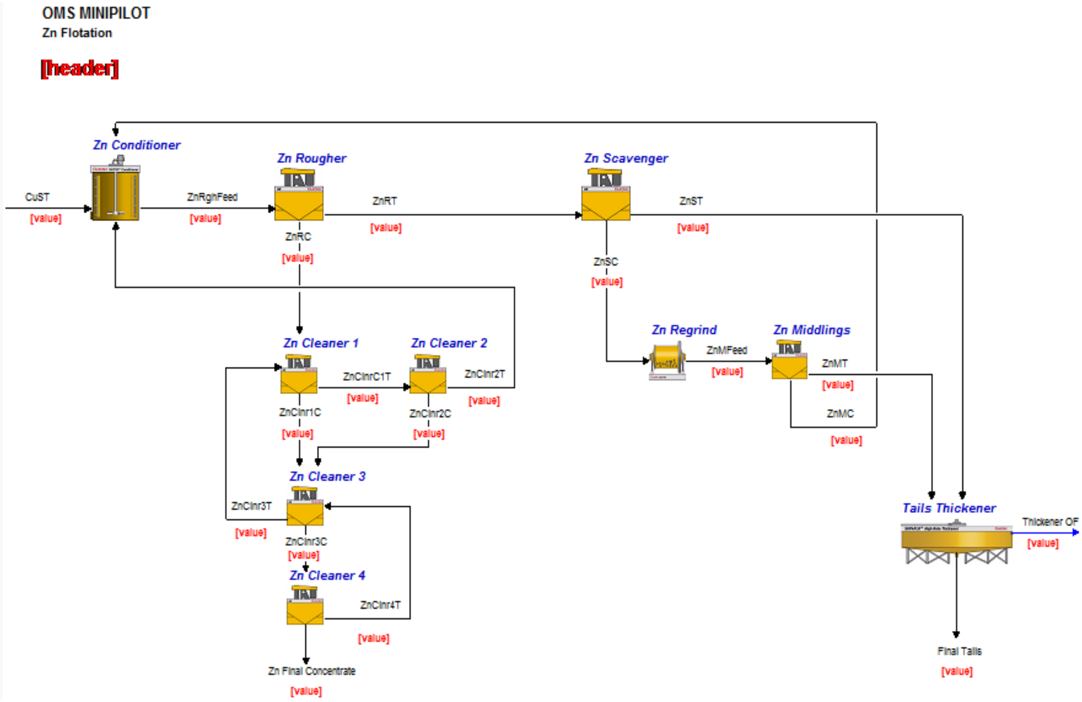

Figure 6. The flow sheet of the OMS minipilot plant zinc flotation.

2.5 Measurements in sulphide ore beneficiation processing (from grinding to zinc flotation)

The general practice employed in the beneficiation of copper sulphide ores is a combination of wet milling and flotation. The used grinding conditions have influence on the flotation of minerals, including an interaction of the minerals with grinding media, the gaseous atmosphere used in the milling, the generation of presence of oxyhydroxide species in the slurry, the effect of any chemical reagent added in the slurry, and the type of grinding method used. (Bruckard *et al.* 2011.)

To achieve the optimization and control of the mineral process a minimum amount of data from the input disturbances, the process states, and the final product quality must be available. The Input disturbances are usually related to material properties. The central measurements needed in mineral processing operations are particle size and composition, but less specific instruments such as level meters, flowmeters, density gauges and pH meters are also required. With these measurements and instruments the control of the mineral process can be achieved. (Hodouin *et al.* 2001 and Bergh & Yianatos 2011.) The environment and process conditions of mineral processing are typically harsh. Thus special consideration to the maintainability, reliability and ruggedness of the measuring devices must be given (Joseph & Sienkiewicz 2002).

In this chapter, for a systematic presentation, the following status and notations of each input and output process variables are used: u is used for manipulated variables (control variables), y for the controlled variables, d for the disturbances, and x for output variables other than the controlled ones. The variables denoted with x are process states that are dependent on control and disturbance inputs. (Hodouin 2011.) The generic names of input-output process variables are shown in Figure 7.

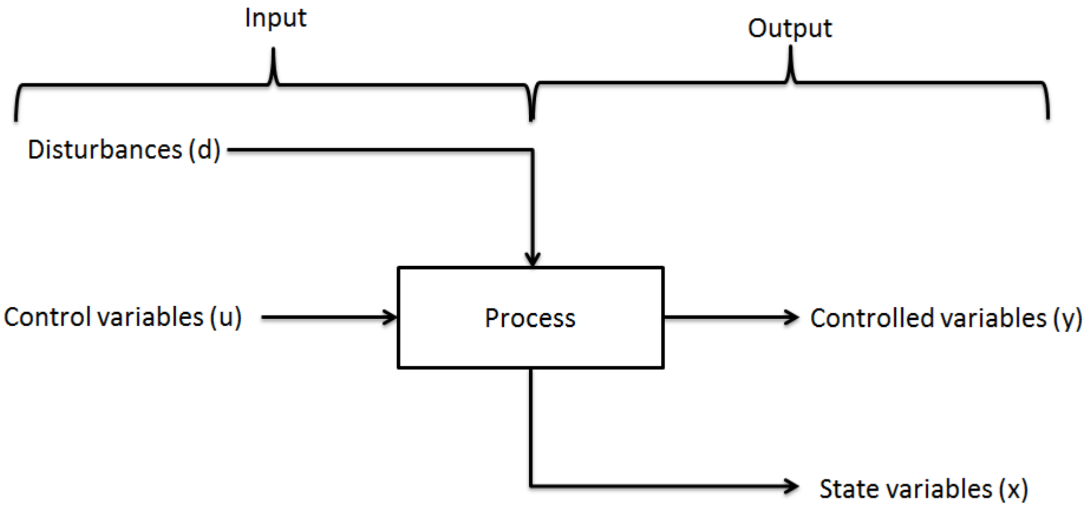

Figure 7. Generic names of input-output process variables. (Hodouin 2011)

Three basic steps are normally used in the design of instrumentation for a mineral processing plant: First, selection of the variables to be estimated by the specific subsequent usage is made; second, evaluation of the measuring devices availability is checked; third, sensor selection and placement plan to obtain the information required by the controller and optimizer is designed (Hodouin 2011). In the following chapters, the objective is to study the first two aspects, mentioned above, from each stage of the beneficiation process. First, the variables and measuring devices for a conventional grinding circuit are introduced. Second, the same observation is done for the sulphide ore conditioning and flotation. The third aspect, sensor selection and placement, is studied more closely later in this work.

2.5.1 Grinding

Grinding is a complex machining process where a large number of parameters are influencing each other. It can be considered as a process where grinding wheel engage with the workpiece at a high speed. A conventional grinding circuit is essentially a two-input-two-output system, consisting of an open-loop rod mill and a closed-loop ball mill. It is assumed here that a classifier pump box is controlled by a local loop. The ore and water rates are the inputs and product fineness and the circulating load are the outputs. (Pomerlau *et al.* 2000.)

The objective, regarding grinding circuit, is to reduce material to a size that is suitable for treatment in later separation and recovery processes. Usually the grinding process is the most expensive and energy intensive part of the ore beneficiation process and therefore the study of the control systems for grinding circuits remain important. (Edwards *et al.* 2002 and Wei & Craig 2008.)

2.5.2 Variables

There are many variables which have an influence on the reduction of particle sizes, which can be considered to be ether equipment variables or manipulated variables. Equipment variables such as the mill size and the ball or rod charge can be fixed and are dependable of the used equipment. Manipulated variables such as the distribution of media size presented in the mill, the particle size distribution of the feed material and the solid content and viscosity of the slip during the mill operation can be changed continuously. (Edwards *et al.* 2002.) Also some material characteristics needs to be considered to achieve optimal result from the grinding process, such as ore hardness, reduction ration, the new surface area generated, degree of agglomeration etc. (Juhasz & Opoczky 1990).

There are multiple principle variables that can affect the grinding circuit control. In this chapter only the closed-loop circuit consisting of a ball mill, a sump and a classifier is discussed, as the variables in open-loop rod mill are very similar to variables in the ball mill.

From a control point of view, a ball mill grinding circuit is a multivariable system with strong interaction among the process variable. Typically, a closed-loop circuit for wet grinding has a structure of the ball mill, the sump and the classifier. (Monov *et al.* 2012 and Edwards *et al.* 2002.) Closed-loop grinding is schematically shown in Figure 8.

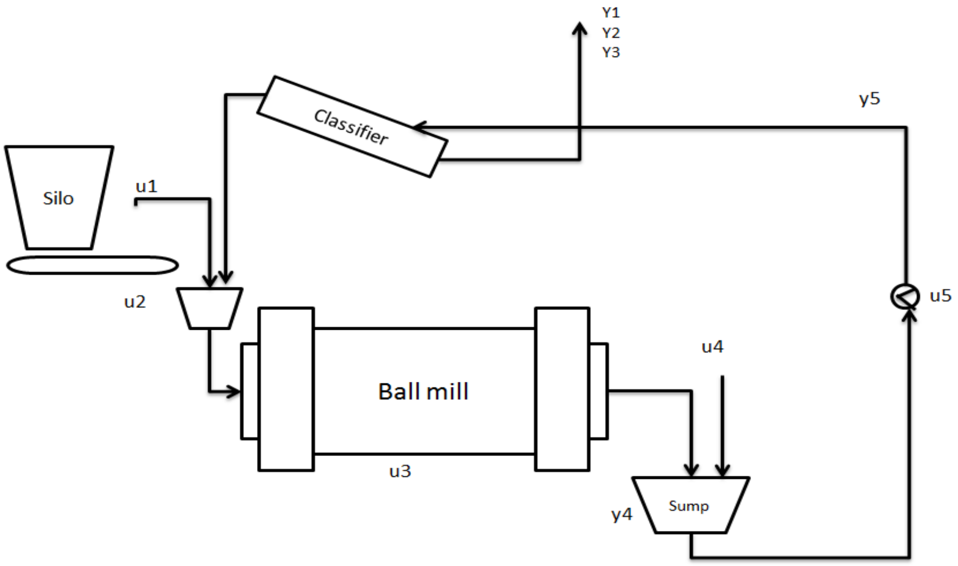

Figure 8. Closed-loop grinding process. (Monov *et al.* 2012)

The process input variables in Figure 8 are: u_1 – mill feed water flow rate, u_2 – fresh ore feed rate, u_3 – mill critical speed fraction, u_4 – sump dilution water flow rate and u_5 – sump discharge flow rate. These variables are manipulated variables and can be manipulated in order to control the output variables. The controlled output variables are: y_1 – product mass fraction with size of particles less than a given value, y_2 – product solids concentration, y_3 – product flow rate, y_4 – slurry level in the sump, y_5 – sump solid concentration. Ore hardness changes and feed size variations are the most important disturbances to the process. (Monov *et al.* 2012.)

There are severe difficulties in the process control of a ball mill grinding circuit due the following characteristics: The process circuit is nonlinear and contains immeasurable disturbances and unmodelled dynamics, there are strong interconnections among variables, which means that each input variable interacts with multiple output variables, the time constants of the process have values in a wide range and the time delays of some input-output pairs are significantly long, there are technological constraints on the manipulated (*u*) and controlled variables (*y*) and the measurements are unreliable and noisy. Due to these difficulties it is necessary at first to maintain a stable operation at fixed set-points of the output variables. The most important variable to maintain in a ball mill grinding circuit is a stable product size distribution. (Monov *et al.* 2012). The most important variables associated with the conventional grinding circuit are shown in Figure 9.

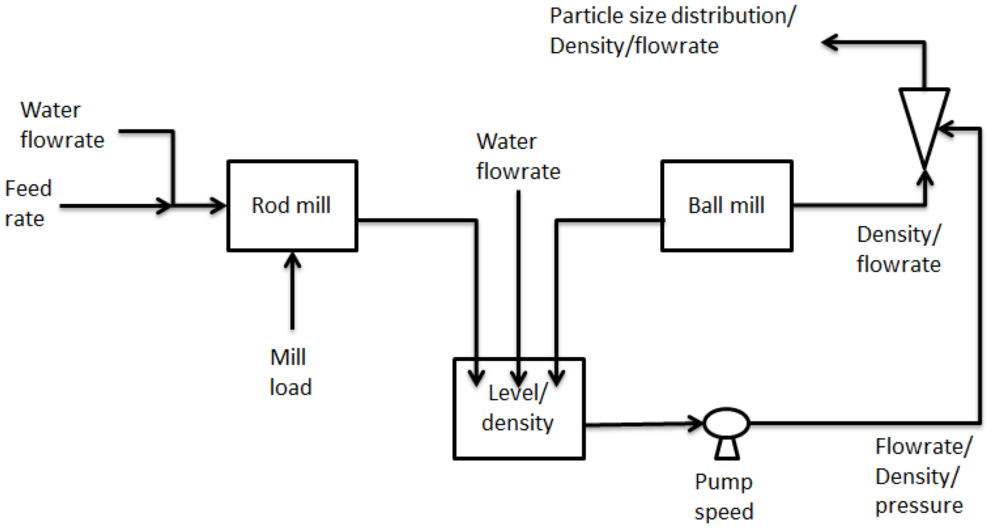

Figure 9. The most important variables in grinding circuit. (Wills *et al.* 2006)

2.5.3 Measurements

Reliable, accurate and precise measurements from the grinding process are critical to accomplish a good control of the process. The quality of the measurement must be known to ensure it is used correctly when applying the control strategy. (Edwards *et al.* 2002.) To achieve the control of mineral processing operations certain measurements are needed, such as ore composition, particle size distribution, flow rates, mill power, mill load mass, mill load volume, feed density of the sump discharge slurry and sump level (Hodouin 2011 and Wei D & Ian K. Craig 2008). Some critical properties such as ore grindability, mineral texture, liberation degree, surface activity, grinding media size distribution, bubble size distribution and air load are important variables in the control of grinding circuit, but extremely challenging to measure on-line and even to infer from other measurements (Hodouin 2011).

Measured variables and instruments, which are used to measure them in a conventional grinding circuit, are shown in Table 1. Table 2 shows the problems with the instruments introduced in Table 1.

Table 1. Instruments for measuring different variables. (Wei & Craig 2008 and Edwards *et al.* 2002)

Measured variables	Instruments
Product particle size	Sieve analysis, sedimentation methods, elutriation techniques, microscopic sizing and image analysis, Laser diffraction and on-line particle size analysis. (Wills et al. 2008)
Mill feed particle size	Digital camera, laboratory wet screening, laboratory sieve, manual screen analysis, PSI, visual estimate
Flow rates of water and slurry	Magnetic flow meter, pressure based meters
Mill motor speed	Variable frequency drive (VFD) motor
Mill load mass	Load cells, derived from bearing pressure, mill vibration monitors
Mill load volume	Dual microphones, tape measure, soft sensors
Mill density	Nuclear density gauges
Mill power	Current transformers, Enermax, KWH meter, mill motor KW output
Sump level	Ultrasonic, electrical, pressure and mechanical techniques
Sump discharge density	Nuclear density gauge, Marcy density scale, specific gravity (Sg) scale and Coriolis mass flow meter
Mill solids feed	Belt weightometer, load cell weightometer, weightometer coupled with tachometer
Sound	Acoustic microphone, FFE minerals SAG analyzer
Image	Vision systems to characterize the feed materials
Mill volumetric filling	Impact meter to determine the location of impact on the shell
Steel ball consumption	Ball addition equipment

Table 2. Troublesome instruments and reasons. *(Wei & Craig 2008)*

Instruments	Reasons
Flow meters	Poor quality; continually drift and require recalibration
Level sensor	Steam and slurry build-up in front of measuring device
Load cells below mill	Poor reliability
Densitometers	Continually drift and require recalibration
Pressure transducers	Cementation on sensing parts, choking of offtake pipe
Particle size distribution monitors	High maintenance cost
Particle size instrument	Outdated software, expensive to replace
Valves, flow meters	Poor quality
Vibrating sensors	Poor quality

The raw measurement values delivered by the measuring devices can be directly used in the numerous tools of the generalized control loop. However, usually the measurements are processed through model-based observers that filter measurement noise and estimate important unmeasured state variables. (Hodouin 2011.)

2.6 Conditioning

Sulphide ore conditioning is a highly complex process where the ore undergoes a number of physical and chemical treatments steps. The conditioning of sulphide ore flotation is considered to begin from the grinding process, where part of chemical reagents are added, and to end in the flotation cell at the moment a gas bubble catches a mineral grain. Hence the conditioning affects not only to particle size distribution, but also the chemical properties of the mineral surface and the suspension. Chemical properties of the mineral surface and the suspension control, among other things, the degree of oxidation of particle surface, pH, conductivity, concentration of dissolved oxygen and the temperature of the ore suspension. For example, electrochemical activity of the sulphide mineral, the amount of oxygen available and the relative size of reductive mild steel surface in the grinding stage have a great effect on the oxidation of a sulphide surface. Therefore, conditioning ultimately determines the path and rate of reactions at the surface of the mineral and in the bulk phase. Hence, it can be said that conditioning essentially determines the floatability of the mineral. (Kuopanportti *et al.* 1997.)

2.6.1 Variables

Process variables for conditioning are shown in Figure 10. Manipulated variables, marked with *u* in Figure 10, for conditioning are flow rates of additional chemical reagents, air feed rate, rotor speed, the temperature in the slurry, conditioning time, slurry feed rate and water feed rate. Controlled variables, marked with *y* in Figure 10, are chemical properties on mineral surface and slurry density. Process disturbances, marked with *d* in Figure 10, are particle surface and composition of ore. Output variables other than the controlled ones, marked with *x* in Figure 10, are slurry density, conductivity, temperature, concentrate of dissolved oxygen, electrochemical activity of the sulphide mineral and mechanical work added to pulp.

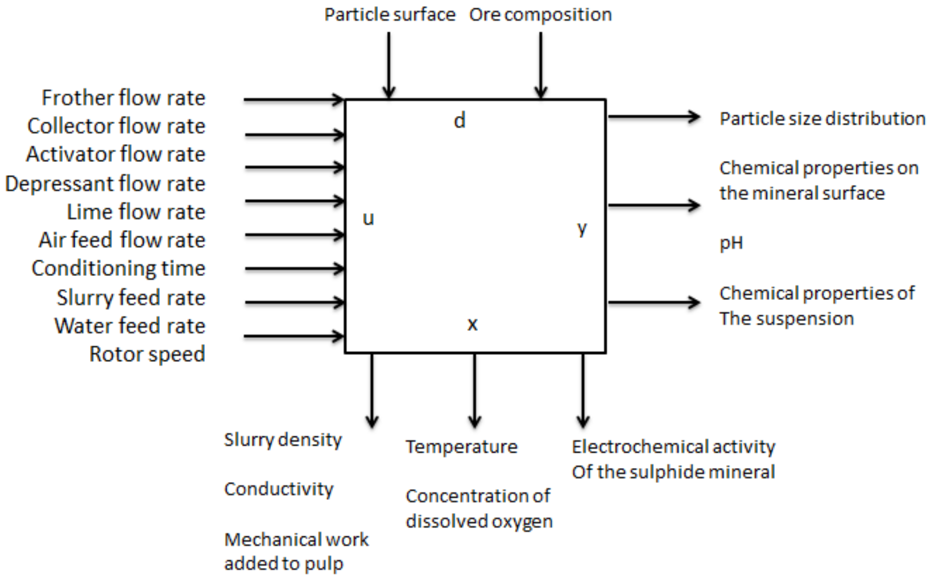

Figure 10. Process variables for a conditioner.

2.6.2 Measurements

Measured variables and instruments, which are used to measure them in a conditioner, are shown in Table 3.

Table 3. Instruments for measuring different variable.

Measured variables	**Instruments**
Mechanical work added to the pulp	Torque measuring device (Engel *et al.* 1996)
Chemical reagent addition	Metering pumps, a simple on-off type dosing system (Shean & Cilliers 2011)
Air flow rate	Flow meter (Tabosa & Rubio 2010)
Slurry flow rate	Magnetic flow meters, fixed speed pumps
Water flow rate	Magnetic flow meters
Temperature	Sensor
Slurry density	Nuclear density meters, XRF analysers
pH	pH meter
Conductivity	Electrodes, conductivity meter
Eh	Electrode probes
Particle size distribution	Sieve analysis, sedimentation methods, elutriation techniques, microscopic sizing and image analysis, Laser diffraction and on-line particle size analysis. (Wills *et al.* 2008)
The xanthate concentration	UV spectrometer (Kuopanportti *et al.* 1997)

Measuring each of these variables simultaneously may well be unnecessary to achieve a good control result. However, each of these variables and their effect on the conditioning process should be considered.

Conditioning time is the primary object to be controlled in the conditioner. Conditioning time, often expressed as energy transferred in the conditioning stage, has a great effect on the concentrate recovery, grade and flotation rate. (Tabosa & Rubio 2010.)

2.7 Flotation

Flotation is nowadays a one of the most widely used separation process in the mineral industry. However, it is quite an inefficient process and also the most difficult stage of the ore beneficiation process. The difficulties come from the complexity and nonlinearity of the flotation process itself. (Laurila *et al.* 2002.) Flotation is a three stage separation process for complex ores. It is based on the manipulation of the difference in hydrophobicity of the particle surfaces. Hydrophobic mineral particles are contacted with air bubbles in the flotation cell, whilst the more hydrophilic gangue particles sink and recover to the tail stream. The bubbles, loaded with valuable-mineral, rise to a froth phase and overflow into a launder, before being recovered to the concentrate stream. Although the process may sound quite simple, there are roughly 100 variables that affect the flotation due occurring simultaneous sub-processes. (Shean *et al.* 2011.) For example froth depth, air and wash water flow rates have significant interactions with the other variables (Bergh & Yianatos 2002).

2.7.1 Variables

There are only a very few numbers of output variables defining the operation performance of a flotation plant. Normally grade and recovery are the two degree of freedom from the process outputs that are used to describe the targets, even though recovery is essentially a steady state concept. Although the flotation plant has only the two-degree of freedom output, the number of variables to be measured and to be manipulated is extraordinarily large. Thus, the variety of different instruments used in the flotation process is extensive. Moreover, there are multiple circulating streams in a flotation circuit that create strong interactions between the variables. (Hodouin *et al.* 2001 and Laurila *et al.* 2002.)

The process input variables, marked with u in Figure 11, are: flow rates of additional chemical reagents (frother, collector and activator), air feed rate, froth levels in flotation units, slurry level and wash water feed rate. These variables are manipulated variables and can be manipulated in order to control the output variables. The controlled output variables, marked with y in Figure 11, are: concentrate grade and flowrate, tail grade and flowrate and circuit recovery. The disturbances, marked with d in Figure 11, to the process are ore particle size and surface composition distribution, slurry properties (density, solids content) and slurry flow rate. Output variables other than the controlled ones are process states that are dependent on control and disturbance inputs. The output variables other than the controlled ones, marked with x in Figure 11, are distribution of particle hydrophobicity, slurry to froth entrainment, stream composition, stream flowrates, percentage of solids stream, cell loads, froth loads and froth appearance. (Hodouin 2011 and Laurila *et al.* 2002.)

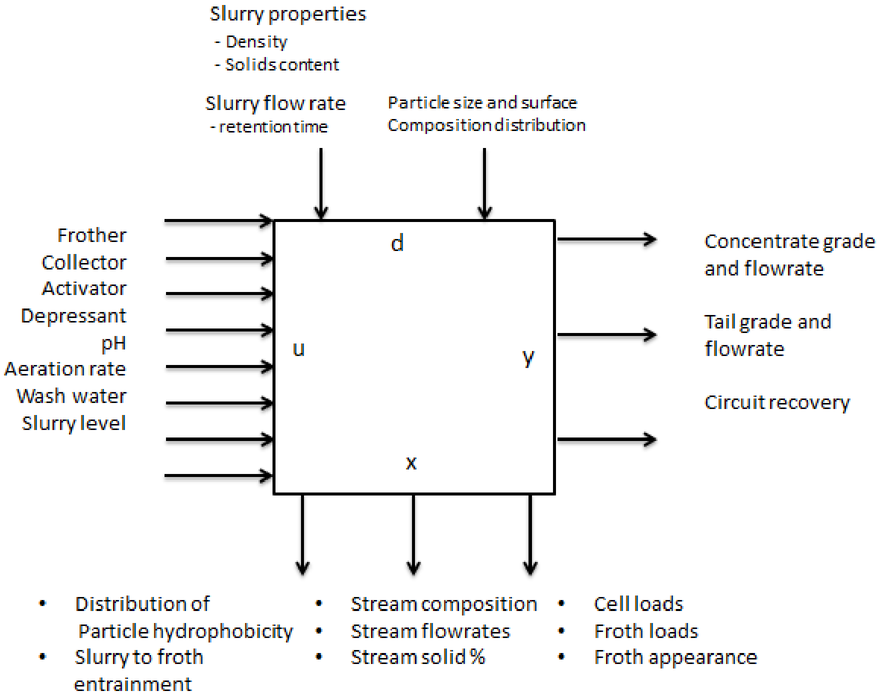

Figure 11. Process variables for a flotation plant. (Hodouin 2011 and Laurila *et al.* 2002)

Column recovery and concentrate grade are the primary objectives in the flotation circuit, these objectives represent the indices of process productivity and product quality. It usually requires a significant amount of work to apply an on-line estimation, in order to maintain good accuracy and high availability, of these indices. Therefore, secondary objectives such as pH at the feed, froth depth, airflow and wash water flow rate are controlled. The airflow rate to the flotation column is one of the most sensitive variables, which directly affects the air holdup, product grade and mineral recovery. (Bergh & Yianatos 2002.)

2.7.2 Measurements

Even though flotation processes have more than a hundred years of history, there are just few reports on application of new technologies for the monitoring and control of flotation processes. Reason for this is that the process is very complex and the process responses are unpredictable. Usually, the controls that can be achieved in flotation plants are pulp level control, precise chemical reagent addition, air feed rate and water flow rate. Devices for measuring these variables are available and they work with reasonable performance. (Bergh & Yianatos 2011.) Some critical properties such as liberation degree, bubble size distribution, surface chemistry and bubble loading are still difficult to measure and infer (Shean & Cilliers 2011).

Measured variables and instruments, which are used to measure them in a flotation circuit, are shown in Table 4. Table 5 shows the problems with the instruments introduced in Table 4.

Table 4. Instruments for measuring different variable. (Shean & Cilliers 2011 and Laurila et al. 2002)

Measured variables	Instruments
Pulp level	Float with a target plate and ultrasonic transmitter, Float with angle arms and capacitive angle transmitter, Reflex radar
Chemical reagent addition	Metering pumps, a simple on-off type dosing system
Air flow rate	Thermal gas mass flow sensor
Slurry flow rate	Magnetic flow meters, fixed speed pumps
Water flow rate	Magnetic flow meters
Mineral concentrations in the feed, concentrate and tailings (recovery, grade)	XRF analyser (on-line or off-line), XRD
Froth stability	Machine vision
Gas hold up	Requires two measurements in separate vessels: one vessel measures the conductivity of the aerated pulp, the other measures the conductivity of the air free pulp. The ration of these measurements is used to solve Maxwell's model and estimate the gas hold up.
Feed solid percentage	
Slurry density	Nuclear density meters, XRF analysers
Slurry solid content	
pH	Ion selective electrodes
Conductivity	Electrodes, conductivity meter
Bubble size distribution	Image analysis (McGill bubble sizing device), machine vision
Froth stability	Machine vision
Particle size distribution	Sieve analysis, sedimentation methods, elutriation techniques, microscopic sizing and image analysis, Laser diffraction and on-line particle size analysis. (Wills *et al.* 2008)
Eh	Electrode probes

To achieve a good control result, it may be unnecessary to measure each of these variables simultaneously. However, the effect of the mentioned variable to the flotation process should be considered. (Shean & Cilliers 2011.)

Table 5. Troublesome instruments and reasons. (Shean & Cilliers 2011)

Instruments	Reasons
Flow meters	Poor quality; continually drift and require recalibration
Pulp level measurements	Slurry-froth transition is not sharp and variations in slurry density and/or very dense layers often exist
XRF	Reliability problems when measured on-line
Densitometers	Continually drift and require recalibration
On-off type dosing system	Regular checks required, inaccurate
Electrodes	Easily contaminated by active substances in the slurry
Gas holdup measurement sensor	Inaccurate when bubbles enter the air free vessel
Machine vision	Lower layers can’t be seen

Analogue signal technology is mostly used in instrumentation on flotation plants. Analogue signals need to be converted into a digital format before interfacing with automation system. As such, it is thought that analogue technology, in the flotation plants, is to be replaced by digital fieldbus technology. Digital fieldbus technology will result in fully digital communication between measuring instruments and base level control at source. Digital communication has already enabled the decentralization of pulp level and air flowrate base level control systems in flotation plants. (Shean & Cilliers 2011.)

3 Experimental part

3.1 Targets of the study

The main object of the experimental part of this work was to collect data from the minipilot process under different operating conditions and subjected to step changes of certain variables. The collected data was later used to investigate the process dynamics and time delays. Performed investigations give important knowledge about how to improve the repeatability and continuity of the process. Also the measuring devices and improvements needed for the process are discussed based on the data and knowledge received from the trial runs.

The aim of this chapter is to introduce all the actions made before, during and after the runs. First, ore handling and chemical reagent preparations for the runs are presented. Second, samplings and measurements executed during the runs are introduced. Third, the laboratory analyses made after each of the runs are studied. Finally, all minipilot trial runs for the thesis are presented.

3.2 Ore handling and chemical reagent preparations

As explained in Chapter 2.4.1 the grain size of ore must be below 3.6 mm, before it can be fed to the silo. To accomplish systematic runs and comparable results from different runs, the pre-crushed ore from the container has to be sieved and sampled before it is fed to the silo. The idea of the sampling is to split the sieved ore to the piles and then systematically collect ore from the piles to achieve random and representative samples.

1000 kg of -3.6 mm sieved ore was split to 5 different piles, 200 kg each. From each of these piles ore was collected from different locations around piles, one from the top and 4 around the sides, as shown in Figure 10. From each of the locations a scoop of ore was taken. Ore was then collected the same way from the other piles as well and stored to 25 kg airtight buckets.

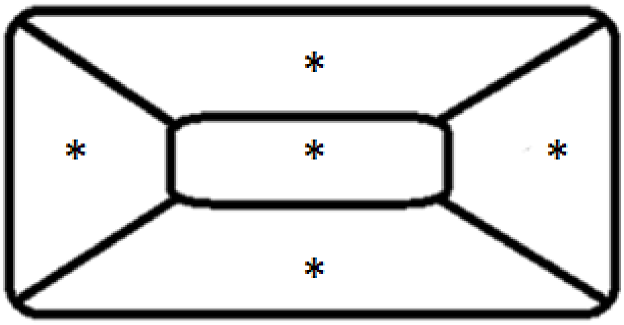

Figure 12. Collected samples from the pile.

The chemical reagents were prepared for all of the runs, before the first run was started. Dowfroth (liquid, purity of 98%), xanthate (solid, purity 90–95%), zinc sulphate (solid, purity 98%) and burned lime (solid, purity 92%) were purchased. Working solutions of individual compounds were prepared in a mixture of water. Used working solutions were: Dowfroth 0.1%, xanthate 1.5%, zinc sulphate 1.5% and the milk of lime 12%.

3.3 Sampling

During the runs process data from motors, pumps, pH meters, chemical reagents flow rates and water flow rates were measured online and stored to the database. Some critical variables, such as slurry density, particle size distribution and element concentrations were measured off-line. The most interesting variables for this thesis are pH and element concentrations in the slurry. Also some particle size samples were taken. The sampling interval was modified for each of the runs and changed during the runs. During the changes the sampling interval was smaller in comparison with steady state conditions.

The pH was measured from the conditioner and from the different cells of copper flotation. The flowsheet of the copper flotation of the minipilot process is given in Figure 5 in Chapter 2.4.1. The pH measurements from the conditioner were executed online by using Endress+Hauser pH measurement device shown left on Figure 13. Data from the device was collected into the Historian database. From the flotation cells pH measurements were made manually by using VWR pH10 pen device. The pH10 pen is presented on the right side on Figure 13.

Figure 13. pH measuring devices used during the trial runs. (Edress+Hauser 2013 and VWR 2012)

3.4 Laboratory analyses

The slurry samples to be studied in the XRF analyser were taken from different process streams. Four different sampling locations were chosen as shown in Figure 14. Sample 00 was taken from the raw material coming from the silo, sample 01 from the stream coming from the conditioner, sample 02 from the copper concentrate and sample 03 from the copper tailing. From each of the runs, different amount of samples from the streams were collected.

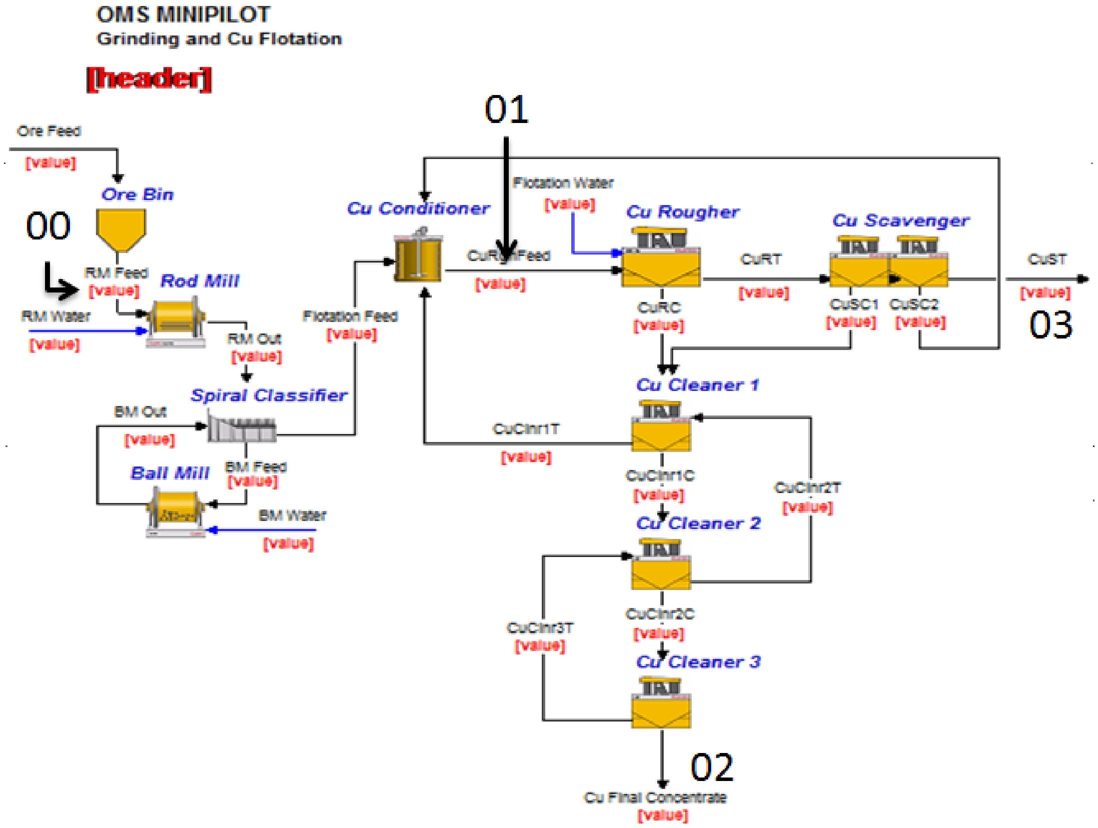

Figure 14. Sampling locations from the process.

The slurry samples taken from the process were first dried in the oven. Dry material was then ground to very fine size and pressed to pellets. Element analyses for the pellets were made with the XRF analyser (X-ray fluorescence). The data captured with the analyser was send to the database for identifying the concentration of major elements (Cu, Zn, S, Fe, Pb) present in the samples. The XRF analyser is shown in Figure 15.

Figure 15. The XRF analyser. (Panalytica 2013)

Particle size samples from each trial run were collected. The samples were analysed with a mechanical shaker involving a nested column of sieves presented in Figure 16. The particle size samples were taken from the rod mill output, the ball mill output, the overflow from the classifier and the copper concentrate streams. Before particle size analyses were done, samples were dried in the oven. Sieve opening sizes used in the column were 500µm, 250 µm, 125µm, 90µm, 63 µm, 45 µm and 32 µm.

Figure 16. The mechanical shaker involving a nested column of sieves. (Fritsch 2013)

3.5 Minipilot experiments

The minipilot experiments were executed during a three month trial period. The first run was executed on 29th of August, the second on 20th of September and the third on 17th of October. Experiments were performed using the grinding and copper flotation sections of the minipilot beneficiation plant as introduced in Chapter 2.4. Due to the relatively long time delay of the process, the zinc flotation circuit was not investigated. The experimental set up including the grinding and the copper flotation sections is shown in Figure 17. The same operating parameters and reagent chemicals, excluding the milk of lime, were used in all the runs as presented in Table 6. The rpm values given in Table 6 present the motor speeds of the machines.

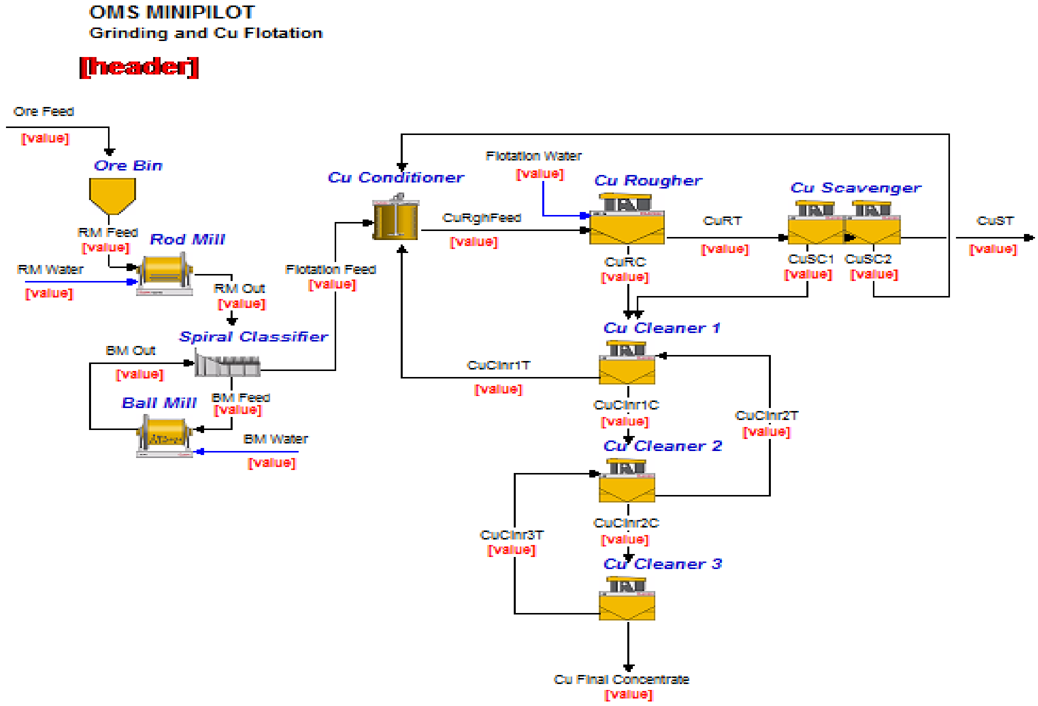

Figure 17. Process set up for the experimental runs.

Table 6. Used process parameters and reagent chemicals feed rates.

PARAMETERS	
Screw conveyor, silo (rpm)	220
-Input (kg/h)	16–17
Rod Mill (rpm)	1000
-Input of Water (l/min)	0.2
Ball Mill (rpm)	1000
-Input of Water (l/min)	0.1
Screw classifier (rpm)	200
PP-01 (rpm)	600
PP-02 (rpm)	600
Conditioner 1 (rpm)	1400
Flotation tank 1 (rpm)	1400
- Input of Air (Nl/min)	5
Flotation tank 2 (rpm)	1400
- Input of Air (Nl/min)	5
Flotation tank 3 (rpm)	1400
- Input of Air (Nl/min)	5
Flotation tank 4 (rpm)	1400
- Input of Air (Nl/min)	5

CHEMICALS' INPUT (ml/min)	
Nasfroth (0.1%)	14
Xanthate (0.5%)	20
Zinc Sulphate (2.5%)	14

Milk of lime feed rate, which is used to control the slurry pH level, was the only parameter changed during different runs. In every case, the change was made as a step change at a specific time instant, either from zero to a high value or from a high value to zero as presented in Table 7.

Table 7. Feed rate of milk of lime (ml/min) before and after the change.

Run	At the beginning (ml/min)	After the change (ml/min)
1	0	300
2	300	0
3	400	0

The adopted parameters and chemical reagents feed rates were based on the test run experiments executed earlier.

3.5.1 First experimental run

The first run was conducted with the objective of finding correct sampling intervals for pH and XRF samples. The objective was to feed the milk of lime to the mills (250 ml/min to the rod mill and 100 ml/min to the ball mill) and after a certain time change it to water (250 ml/min to the rod mill and 100 ml/min to the ball mill). The lime concentration of the milk of lime was 12% and pH of the mixture was from 12.4 to 12.9. The test procedures for the first run are:

1. The ramp-up of the grinding circuit of the process was started on a previous day. 200 kg of handled ore was added to the silo.
2. Constant parameters for the screw conveyor, rod mill, classifier, ball mill and milk of lime feeds were applied as shown in Table 6. The ramp-up of the grinding circuit was stopped when the classifier was fully working and the slurry density was around 1.6 kg/l.
3. During the grinding circuit ramp-up it was realized that there were problems with the consistency and density of the slurry. The slurry was too thick and heavy for the spiral classifier to work properly and also the pumps were jammed. After many hours without the change, the milk of lime feed was changed to water. Due to the problems with the mixture of the milk of lime and ore, it was impossible to observe how the changes made in the mills affected to the flotation. Thus the observation of the process was limited to only covering the area between the conditioner and the copper flotation. The procedures 1

and 2 carried out for the grinding circuit ramp-up were followed by the following actions:

4. On a next day, the same parameters for the grinding circuit were used and the flotation was started. The used flotation parameters and chemical reagent feed rates are shown in Table 6. The chemical reagents were fed to the conditioner. As shown in Table 7, it was decided to first run the process without the milk of lime and after a certain time start its feed to the conditioner.
5. When the whole copper flotation was ready and at the steady state condition, the sampling was started. Different samples for the XRF, pH and particle size analyses were taken.
6. The milk of lime feed to the conditioner was started one hour after the first sample was taken. As shown in Table 7, 300 ml/min feed rate was applied.
7. The process was run for two hours after the milk of lime feed was started.

According to the careful analysis of the results obtained from the first run, it was decided to make an opposite step change to the milk of lime feed in the second and third runs. In the second run, the milk of lime feed rate is to be high initially and dropped to zero after a certain time. The sampling intervals for the XRF and pH samples were fixed due to the problems noticed in the analysis. The number of XRF samples taken from the sampling locations 00 and 01 was noticed to be too high, and the number of pH samples taken from the flotation cells was noticed to be too low. Based on these observations the sampling intervals were adjusted for the following trial runs.

3.5.2 Second experimental run

The second run was conducted with the objective of investigating a process response and delay time between the conditioner and the flotation cells after the step change to the milk of lime feed was performed. For the second run, the ramp-up of the grinding mills (procedures 1 and 2) was carried out as in the first run. The only exception was that the milk of lime was not fed to the mills. The test procedures following the ramp-up for the second run are:

3. The used flotation parameters and chemical reagent feed rates are shown in Table 6. The chemical reagents, including the milk of lime, were fed to the conditioner. The feeding rate of the milk of lime was 300 ml/min.
4. After the whole copper flotation was at the steady state condition, the sampling was started. Different samples for the XRF, pH and particle size analyses were taken.
5. The milk of lime feed to the conditioner was stopped 91 min after the first sample was taken.
6. Process was run until the pH was dropped below 7 in each of the flotation cells.
7. According to the analysis of the result obtained from the second run, pH of the slurry was noticed to be too low to accomplish the best results. It was thus decided to increase the milk of lime feed rate for the third run.

3.5.3 Third experimental run

The third run was conducted with the objective of investigating the repeatability of the process by repeating the procedures carried out on the second trial run. Another goal for the third run was to accomplish the desired pH value 12 by increasing the initial milk of lime feed rate from 300 ml/min to 400 ml/min.

During the third run, two impellers from the flotation cells 1 and 2 were detached and could not be used. It was decided to take the final concentrate out already after the first cleaning flotation. Due to the breakage and concern of further damages, the schedule of the run was cut short. The milk of lime feed was stopped 83 min after the first sample was taken. The whole process was run down 1.5 h from that.

4 Results

The results obtained during the minipilot experimental runs are introduced here. The results have been grouped according to these runs. For each trial run, XRF, pH and particle size analyses are presented separately. At the end of each chapter, the observations done during the runs are presented. The complete data from the XRF analyses can be found in Appendices 1 to 10.

4.1 First experimental run

The findings of the measurements and laboratory analyses from the first minipilot test run are introduced here. First, all the results from the XRF analyses are presented and explained. The results from the pH measurements are reported and studied next. Third, the particle size analyses from different streams of the process are presented. Finally, the noteworthy observations made during the run are collected and presented.

4.1.1 XRF analyses

In Figure 18, the contents of copper, zinc and iron in a sample taken from the sampling location 00 (feed to the rod mill) are presented as a function of time. In Figure 19, the same parameters are shown for the sampling location 01 (feed to the flotation). For sampling locations, see Figure 14. In both figures on the left vertical axis the copper and zinc contents of the ore are shown. On the right vertical axis, the iron content of the sample is presented.

As seen from Figure 18, the changes of element contents are quite small. Copper content changes between ca. 0.7% and 1.4%. The average copper content is 0.94%, and the standard deviation is 0.18 % as presented in Appendix 1. The average content of zinc in samples from the location 00 is 3.2% and the standard deviation is 0.42% as shown in Appendix 1. In Appendix 1, also the element contents as a function of time, the average contents and the standard deviations of iron, sulphur and lead are presented.

As shown in Figure 19, the copper content between different samples is quite stable. As presented in Appendix 2, the average content of copper of samples taken from the location 01 is 0.92% and the standard deviation is 0.06%. In Appendix 2, also the element contents as a function of time, the average contents and the standard deviations of zinc, iron, sulphur and lead are presented.

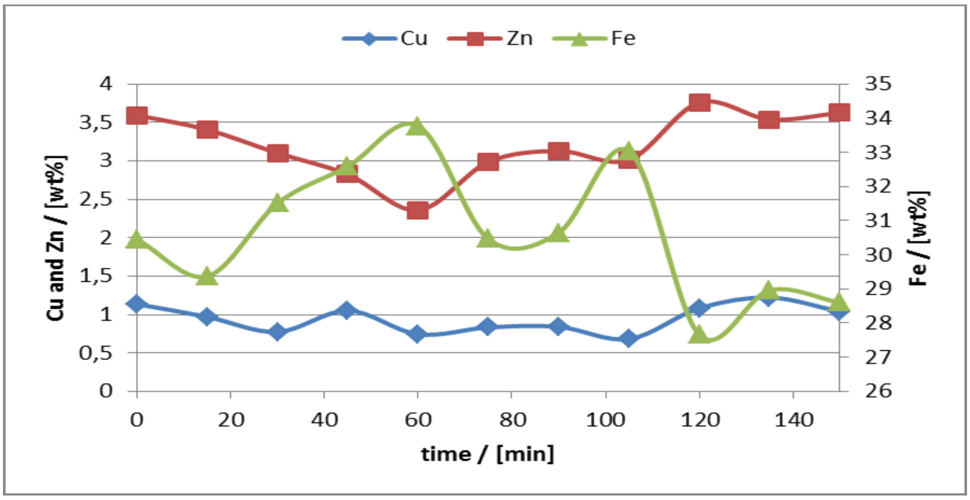

Figure 18. Cu, Zn and Fe contents in XRF samples from the sampling location 00.

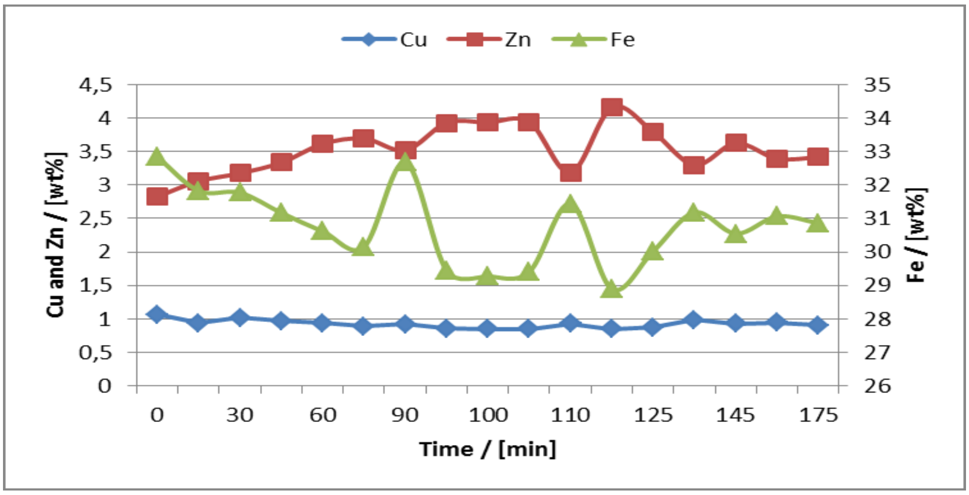

Figure 19. Cu, Zn and Fe contents in XRF samples from the sampling location 01.

In Figures 20 and 21, the copper, zinc and iron contents in samples taken from sampling locations 02 (copper concentrate) and 03 (copper tailing) are presented. As shown in Figure 20 the copper and zinc contents are relatively stable. After the process was run for 100 min some small changes were detected. From Appendix 3, the element contents as a function of time, the content averages and the standard deviation of each element can be found.

As shown in Figure 21 the zinc and the iron contents experience some radical changes during the time. Zinc content rises from 4.1% to 9.1% and iron goes down from 26% to 19 %. The copper content is relatively stable. From Appendix 4, the element contents as a function of time, the content averages and the standard deviation of each element can be found.

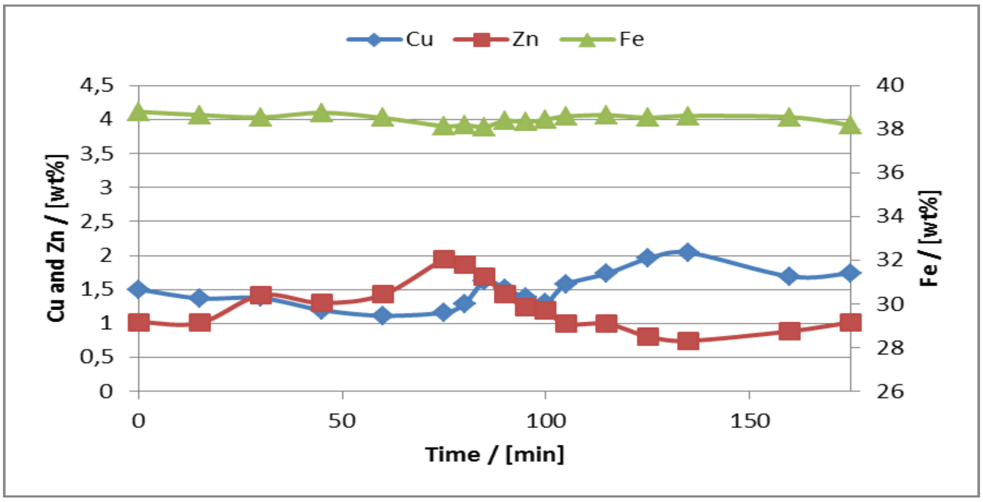

Figure 20. Cu, Zn and Fe contents in XRF samples from the sampling location 02.

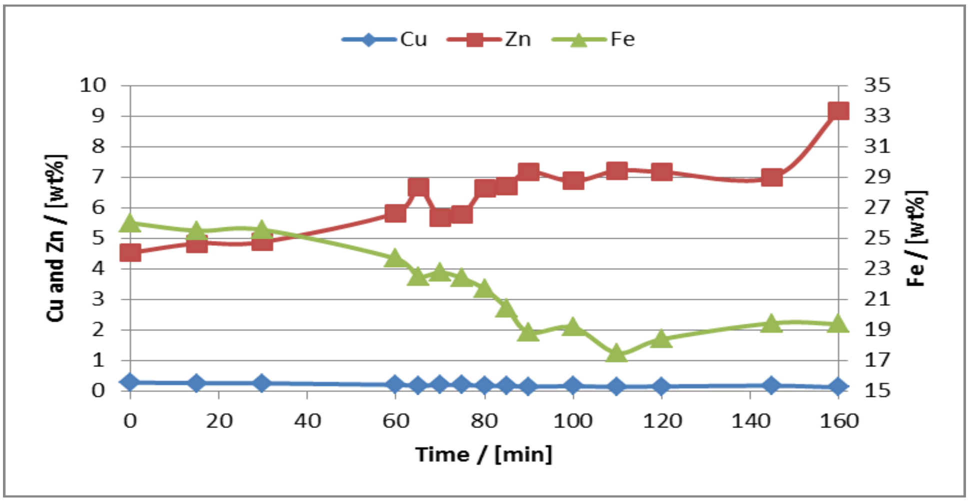

Figure 21. Cu, Zn and Fe contents in XRF samples from the sampling location 03.

4.1.2 pH

The pH was measured online from the conditioner and manually from the flotation cells. In Figure 22, pH from the conditioner is presented. As shown in the figure, pH is ca. 6.5 at the beginning of the run. After the milk of lime feed was started, pH went up to 9. Table 8 shows how pH changed in each cell during the run. As shown in Table 8, the rougher flotation pH was 8.4, the scavenger flotation pH was 7.8 and the third cleaning flotation pH was 7.7 at the end of the trial run. The number of samples taken from the cells is relatively low, and the changes occurred during the run are quite small and hard to detect.

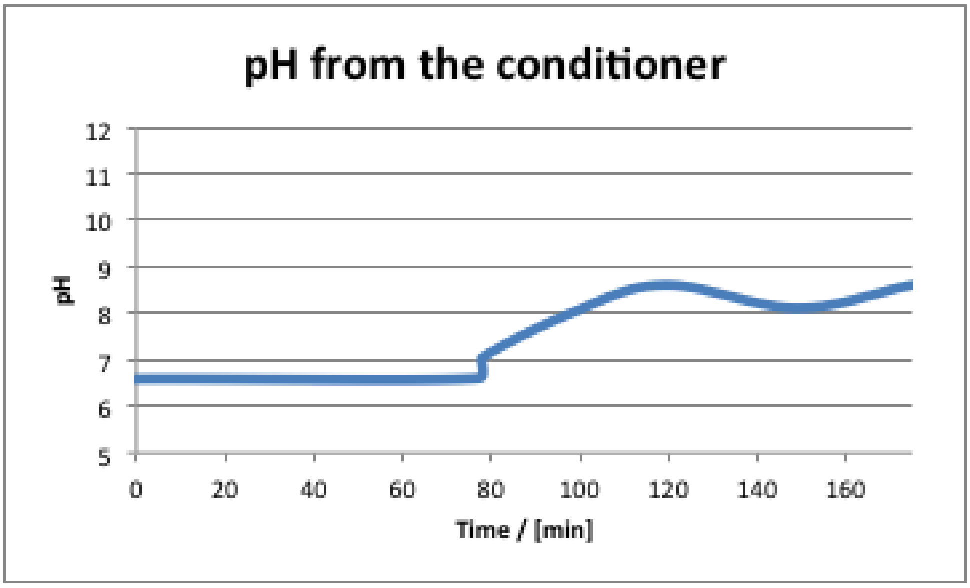

Figure 22. pH from the conditioner.

Table 8. pH from different cells.

Time [min]	Rougher	Scavnger	1st Cleaning	2nd cleaning	3rd cleaning
0	7,04	7,04	7,04	7,04	7,04
85	7,8	7,6	7,6	7,6	7,8
100	8,1	7,8	7,8	7,8	7,8
115	8,4	7,8	7,8	7,8	7,7
165					7,7

4.1.3 Particle size distribution

The particle size analyses were made for the outputs of the rod and ball mills, the classifier overflow, the copper concentrate and the copper tailing before and after the pH change as shown in Figure 23. Sieve opening size is presented as a logarithmic scale and the vertical axis presents the percentage of ore passed the sieves.

As shown in Figure 23, small particles in the flotation are in the copper concentrate and bigger particles in the tailing. 80 percent of the copper concentrate particles are smaller than 62 µm, when in the tailing the d80 is from 90 µm to 100 µm. Figure 24 presents that the rod mill outflow includes bigger particle sizes than the ball mill outflow. 80 percent ore passed value for the rod mill is 80 µm, and 70 µm for the ball mill.

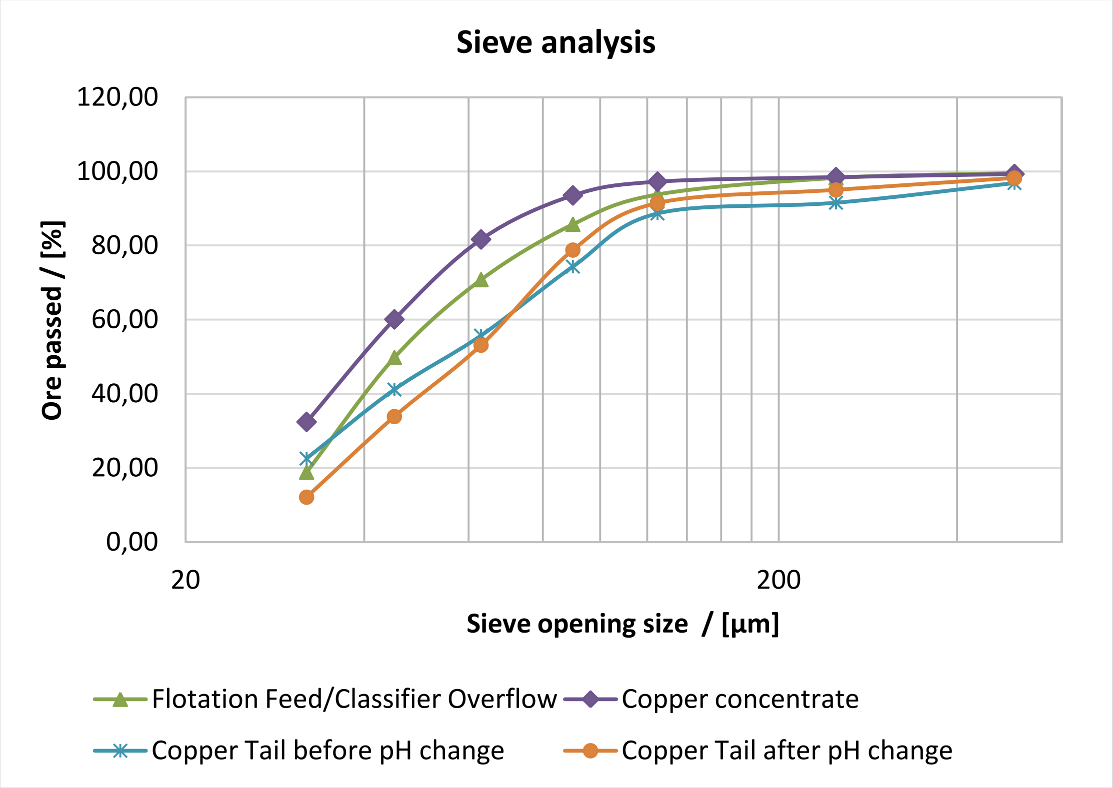

Figure 23. Particle size distribution of the flotation.

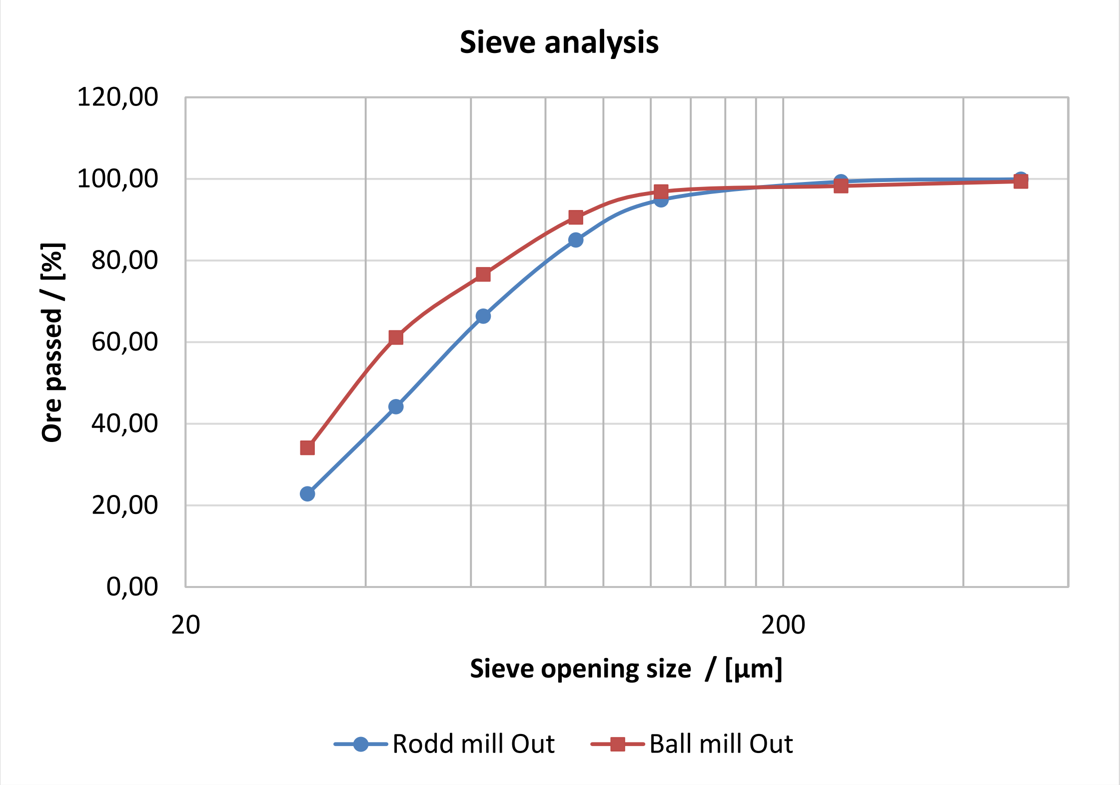

Figure 24. Particle size distribution of the ball and the rod mill.

4.1.4 Observations

The most significant problem observed during the first run was the slurry composition created from ore and the milk of lime when both added to the mills. The mixture of ore and the milk of lime were really dense and it caused problems in further steps of the process. Due to the problem the milk of lime feed was changed from the mills to the conditioner.

From the measurements carried out during the first run, it was noticed that the step change of the milk of lime feed was made in a wrong direction. The delay times in the flotation cells are so long that it takes too much time or the milk of lime feed rate must be really high to observe any changes when the milk of lime feed is started in the middle of the run. Also the used chemical pumps for the milk of lime were too small and powerless to pump it. Thus only the clear part of the mixture from the surface was used and pumped to the process.

The aim of the first run was to find appropriate sampling intervals as stated in Chapter 3.5.1. Thus the sampling intervals were evaluated from the analyses made after the run to determine if they need to be adjusted. It was noticed that the sampling interval for the pH samples must be increased and matched with the samples taken for the XRF. With the increased number of samples, the correlation between pH and element contents of the slurry can be studied. It was also decided to decrease the sampling interval of the sampling location 01 for next runs, due the small changes observed in element contents in XRF analyses.

4.2 Second experimental run

The findings of the measurements and laboratory analyses from the second minipilot trial run are introduced here. First, all the result from the XRF analyses are presented and explained. Second, the result from the pH measurements are reported and studied. Third, the particle size analyses from different streams of the process are presented. Finally, the noteworthy observations made during the run are collected and presented.

4.2.1 XRF analyses

In Figures 25 and 26, the contents of copper, zinc and iron of the samples taken from the sampling location 00 (feed to the rod mill) and 01 (feed to the flotation) are presented as a function of time. In both figures on the left vertical axis the copper and zinc contents of the ore are shown. On the right vertical axis, the iron content of the sample is presented.

As seen from Figure 25, the changes in the copper, zinc and iron contents are quite small. Copper content changes between ca. 1% and 1.1%. The average copper content is 1.07% and the standard deviation is 0.05% as presented in Appendix 5. The average content of zinc in samples from the location 00 is 3.8% and the standard deviation is 0.1% as shown in Appendix 5. In Appendix 5, the element contents as a function of time, the content averages and the standard deviation of each of the elements for the sampling location 02 are presented.

As shown in Figure 26, the changes in copper content between different samples are quite small. As presented in Appendix 6, the average content of copper of samples taken from the location 01 is 1% while the standard deviation is 0.19%. In Appendix 6, the element contents as a function of time, the content averages and the standard deviation of each of the elements for the sampling location 01 are presented.

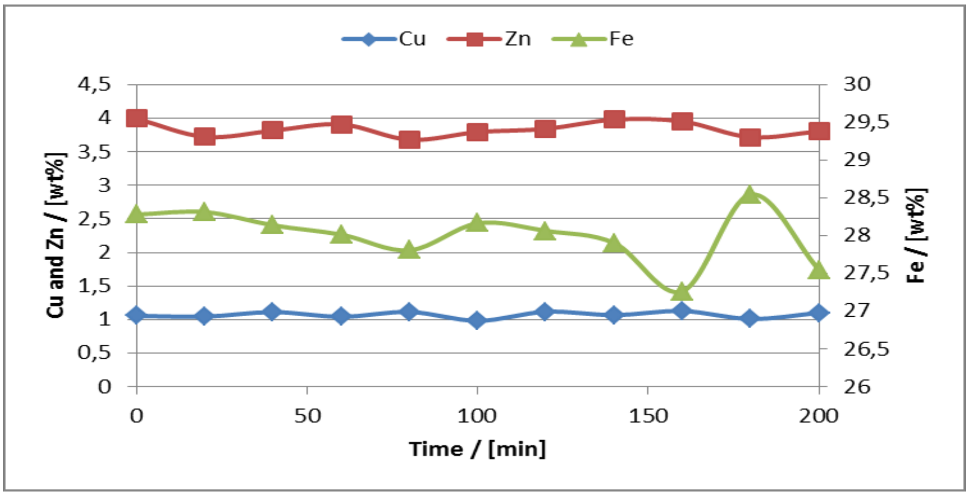

Figure 25. Cu, Zn and Fe contents on XRF samples from the sampling location 00.

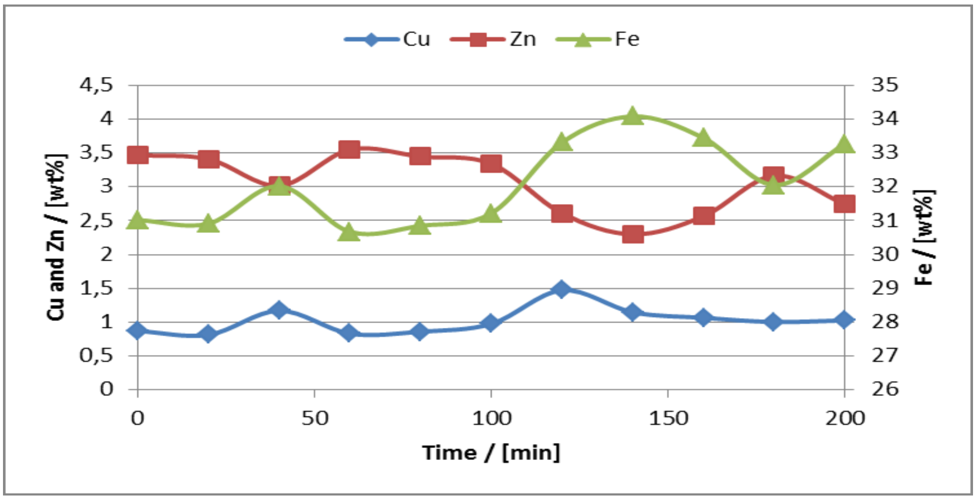

Figure 26. Cu, Zn and Fe contents on XRF samples from the sampling location 01.

In Figures 27 and 28, the copper, zinc and iron contents in samples taken from sampling locations 02 (copper concentrate) and 03 (copper tailing) are presented. As shown in Figure 27, the copper content is dropped during the time due to the change made in the milk of lime feed. The copper content is first declined from 7.2% to under 4% and after 100 min the content is dropped almost linearly until the value is near to 1%. As presented in Figure 27, the iron content seems to correlate with the copper content. When there is a drop in the copper content the iron content increases, and when the copper content increases, the iron content drops. In Appendix 7, the element contents as a function of time, the content averages and the standard deviation of each of the elements for the sampling location 02 are presented.

As shown in Figure 28, the copper and zinc contents in samples taken from sampling location 03 are relatively stable. The iron content rises from 28.5% to 30.5% during the run. In Appendix 8, the element contents, the content averages and the standard deviation of each element for the sampling location 03 are presented.

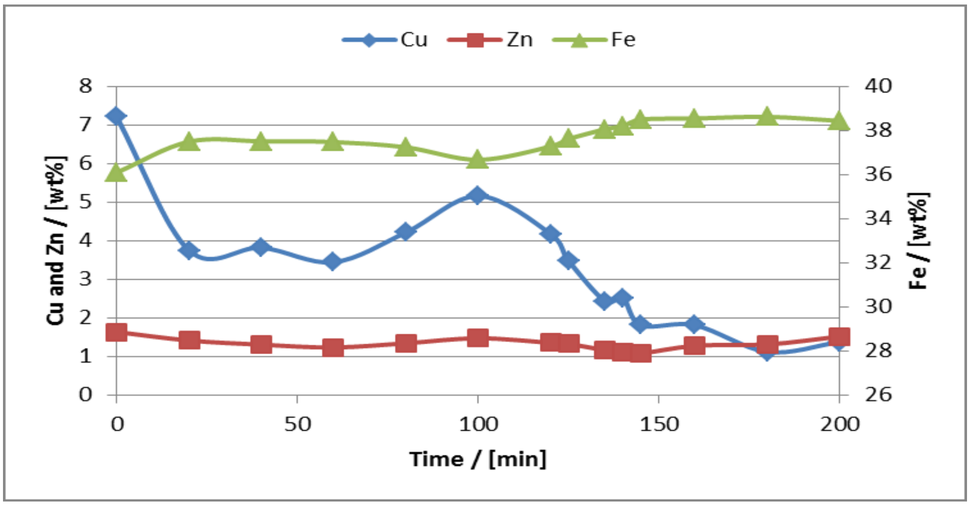

Figure 27. Cu, Zn and Fe contents on XRF samples from the sampling location 02.

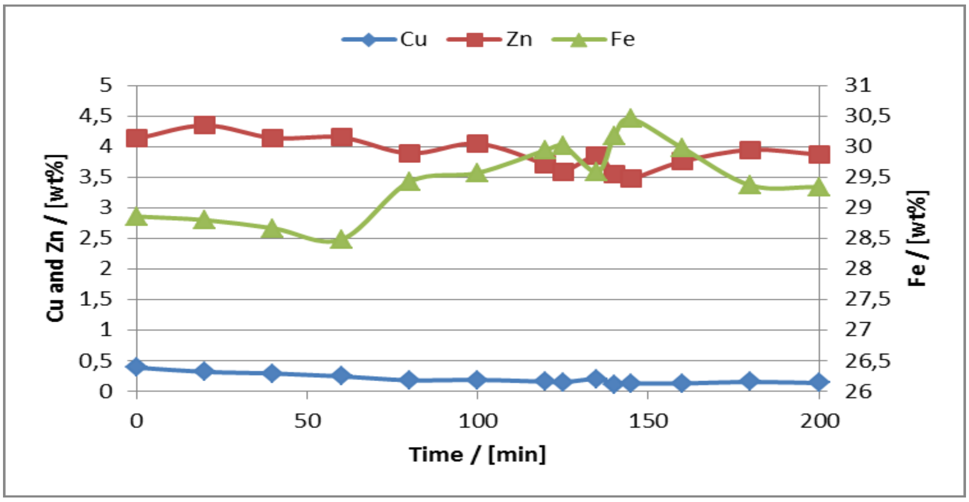

Figure 28. Cu, Zn and Fe contents on XRF samples from the sampling location 03.

4.2.2 pH

The measured pH values from the rougher, scavenger, first cleaner and third cleaner flotation units are shown in Figure 29. As presented in Figure 29, the pH level in the rougher flotation has the highest value during the first 100 min of the run. The pH level in the third cleaner flotation only reaches the value of 9.5 during the run. The scavenger and the third cleaner flotation had the smallest variations during the run. The rougher flotation reacts fastest to changes made to the conditioner and the scavenger has the longest delay time.

From Figure 29, a time constant, a time delay and a slope for the rougher, the first cleaner, the final concentrate and the scavenger flotation were determined. The time constant represents the time it takes the pH to reach 63.2% of its final value. The time constant for the rougher flotation was 26 min, for the first cleaner flotation 18 min and for the final concentrate flotation 21 min. The pH of the scavenger flotation was not stabilised before the sampling was ended. Thus, the time constant for the scavenger flotation was not counted. The milk of lime feed to the conditioner was stopped 91 min after the first sample was taken. The time delay represents the time it takes to notice changes on the pH after the milk of lime feed to the conditioner was stopped. The time delay for the rougher flotation was 7 min, for the first cleaner flotation 25 min, for the final concentrate flotation 37 min and for the scavenger flotation 35 min. From the slopes of the pH curves the rate of change can be defined. The slopes of the pH curves of the flotation cells during the change are: the rougher flotation 66 1/min, the first cleaner flotation 63 1/min, the final concentrate flotation 48 1/min and the scavenger flotation 33 1/min. Figure 30 presents the online measurements of the pH from the conditioner. As shown in Figure 30, the pH values measured from the conditioner and from the rougher flotation are relatively similar.

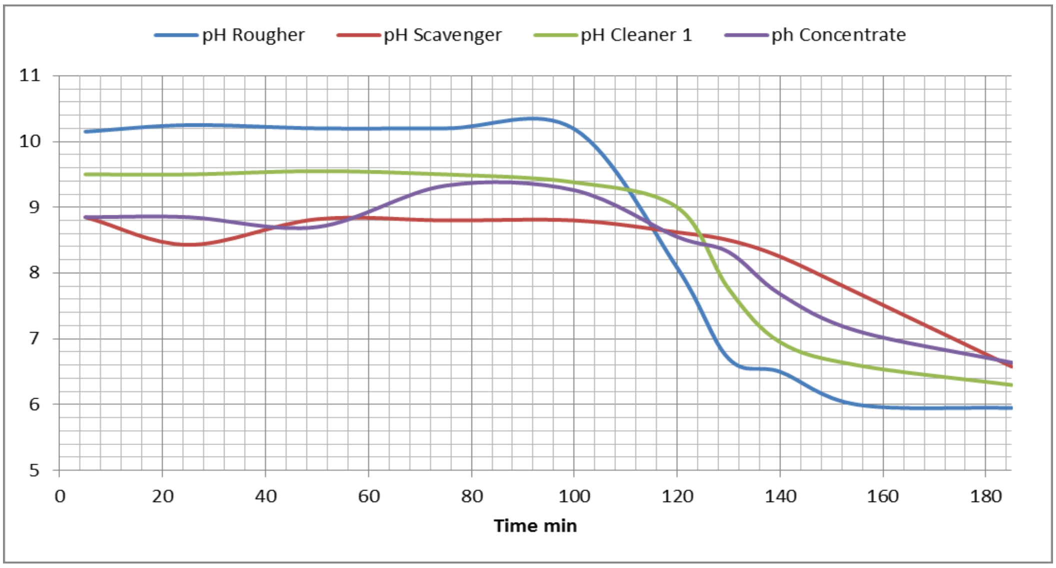

Figure 29. The pH of different cells.

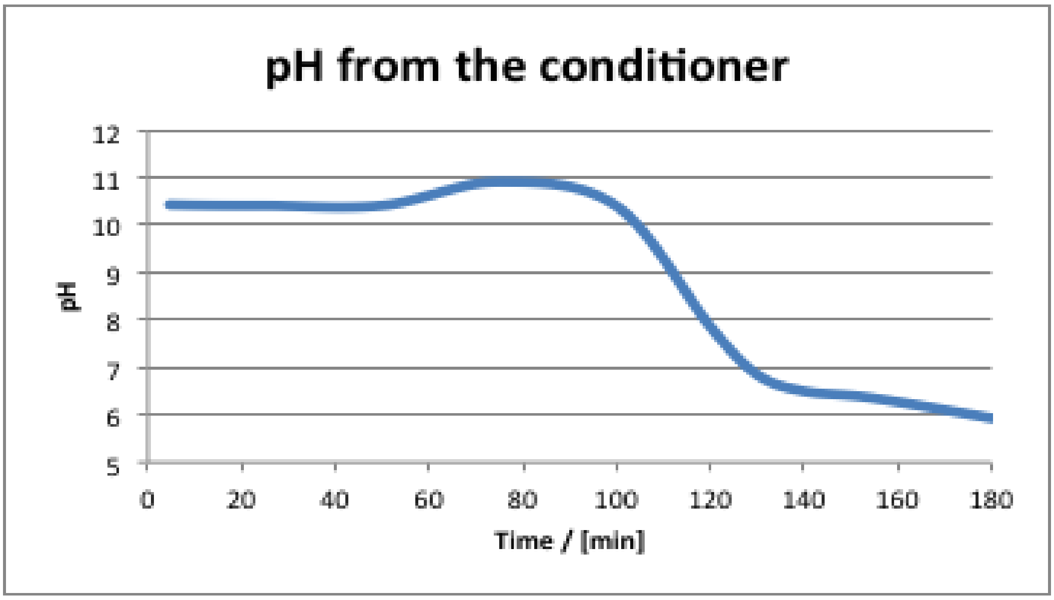

Figure 30. pH of the conditioner.

4.2.3 Particle size distribution

The particle size analyses were made for the outputs of the rod and ball mills, the classifier overflow, the copper concentrate and the copper tailing before and after the pH change as shown in Figure 31 and 32. Sieve opening size is presented as a logarithmic scale and the vertical axis presents the percentage of ore passed the sieves.

As shown in Figure 31, the copper concentrate and copper tailing have the similar particle size distributions. The 80% passed ore value for the copper concentrate and the tailing is 60 µm. Figure 32 presents that the rod mill outflow includes bigger particle sizes than the ball mill outflow. The 80% ore passed value for the rod mill is 85 µm, and 75 µm for the ball mill.

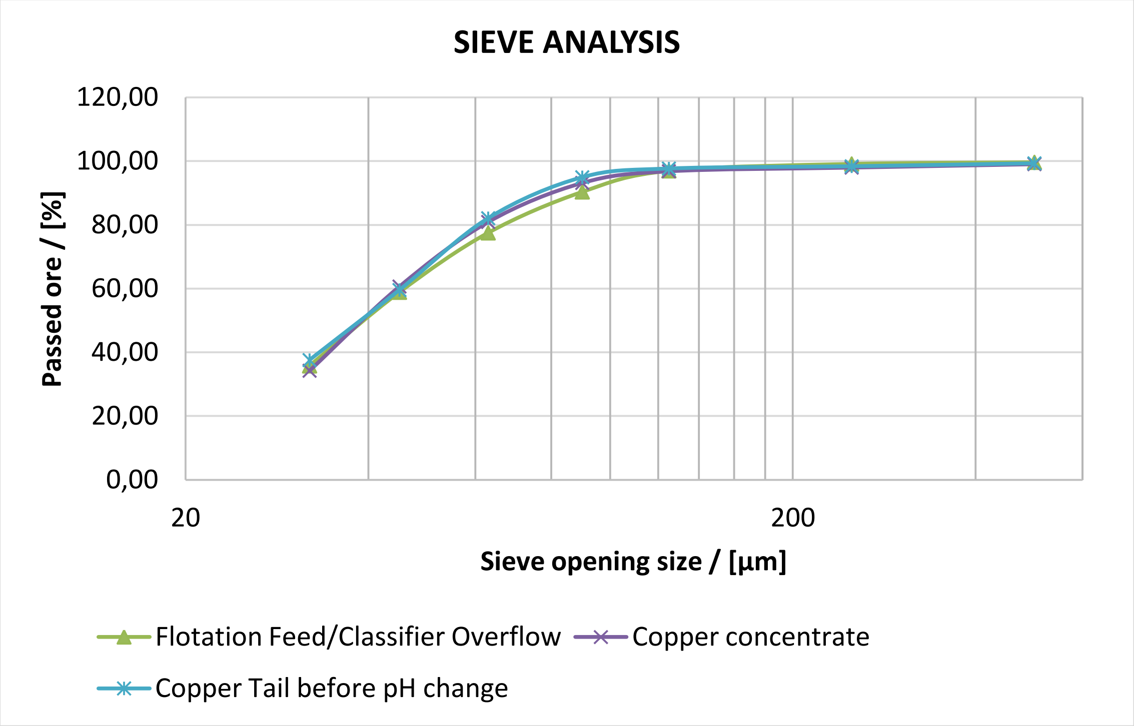

Figure 31. Particle size distribution for the flotation.

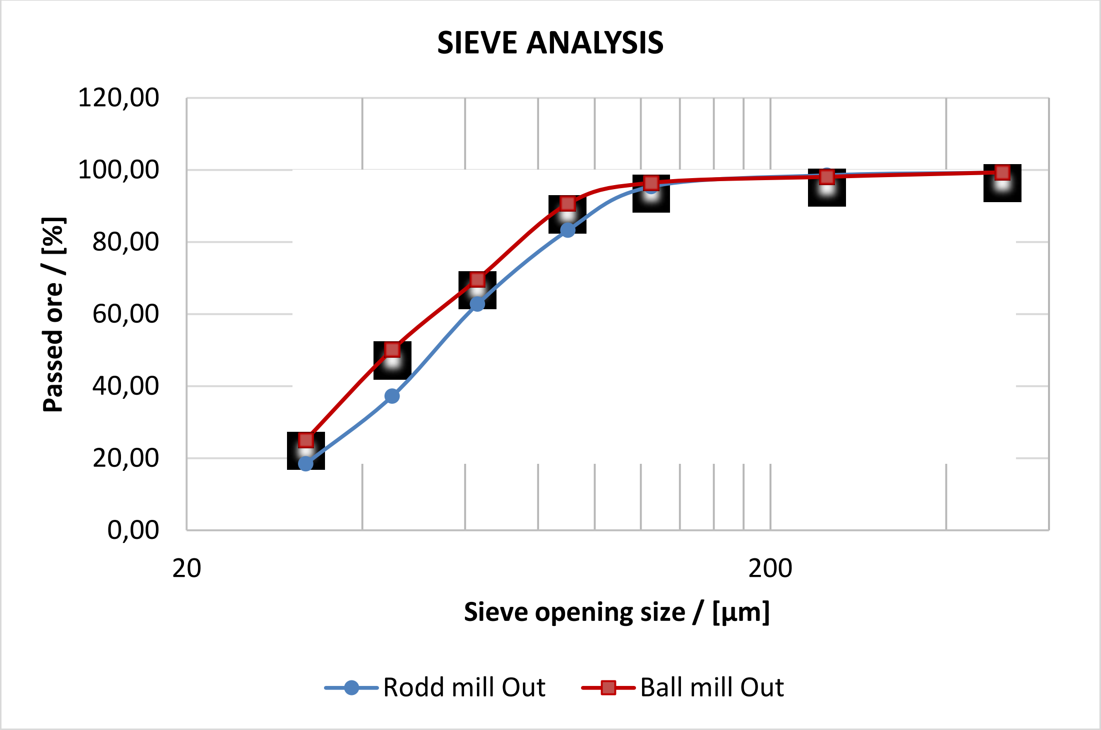

Figure 32. Particle size distribution for the rod and the ball mill.

4.2.4 Observations

The whole process was working as planned and the step change made to the milk of lime feed was accomplished as hoped. The step change was seen from the XRF and pH analyses. The milk of lime feed rate of 300 ml/min was noticed to be too low to achieve the desired pH level 12 for each of the flotation cells. For the final run, the milk of lime feed rate was increased to 400 ml/min. This feed rate should also be applied for future runs.

4.3 Third experimental run

The findings of the measurements and laboratory analyses from the third minipilot trial run are introduced here. First, all the result from the XRF analyses are presented and explained. Second, the result from the pH measurements are reported and studied. Third, the particle size analyses from different streams of the process are presented. Finally, the noteworthy observations made during the run are collected and presented.

4.3.1 XRF analyses

In Figures 33 and 34, the contents of copper, zinc and iron in the samples from the sampling location 00 (feed to the rod mill) and 01 (feed to the flotation) are presented as a function of time. In both figures on the left vertical axis the copper and zinc contents of the ore are shown. On the right vertical axis, the iron content of the sample is presented.

As seen from Figure 33 the changes of the copper and zinc contents are relatively small in samples 00. The copper content changes between ca. 1% to 1.2%. The average copper content is 1.07% and the standard deviation is 0.04% as presented in Appendix 9. Average content of zinc in samples from the location 00 is 3.7% and the standard deviation is 0.1% as shown in Appendix 9. In Appendix 9, also the element contents, the average contents and the standard deviations of iron, sulphur and lead are presented.

Figure 34 shows that the copper content between different samples is relatively stable. As presented in Appendix 10 the average content of copper in samples taken from the location 01 is 1% and the standard deviation is 0.17%. In Appendix 10, also the element contents, the average contents and the standard deviations of zinc, iron, sulphur and lead are presented.

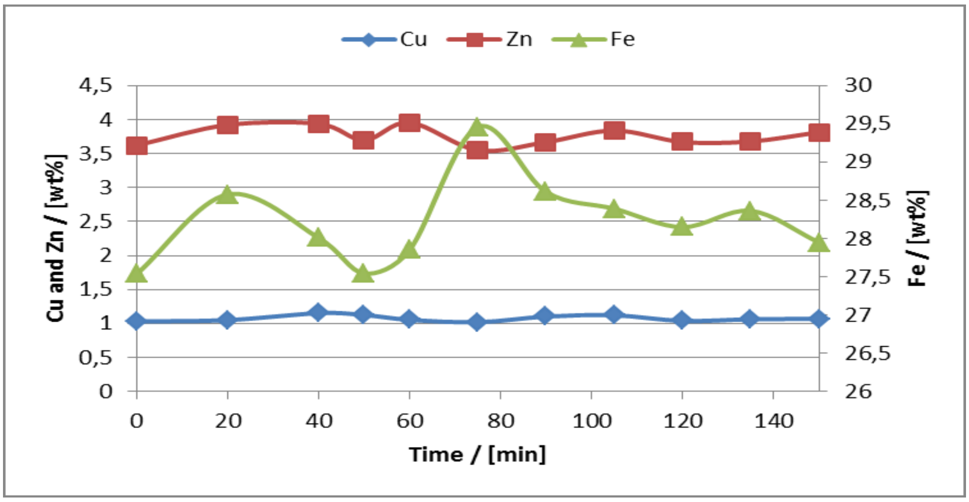

Figure 33. Cu, Zn and Fe contents on XRF samples from the sampling location 00.

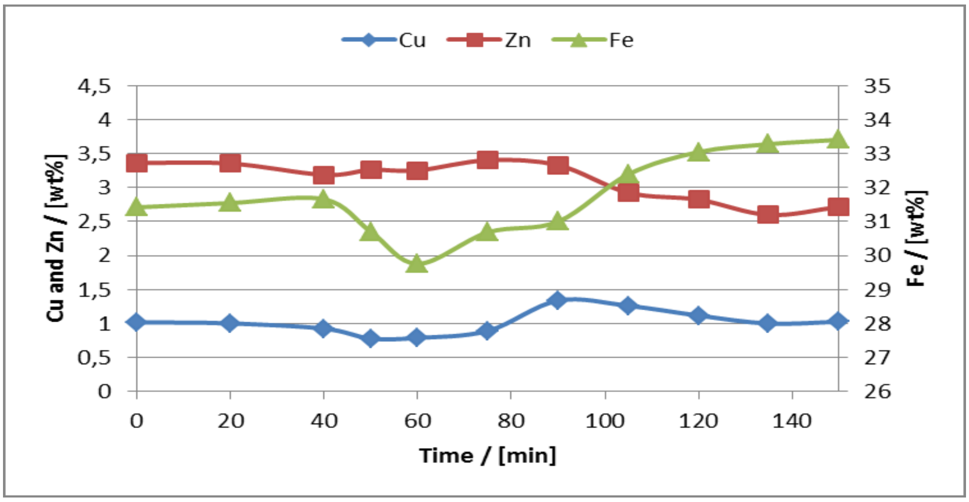

Figure 34. Cu Zn and Fe contents on XRF samples from the sampling location 01.

In Figures 35 and 36 the copper, zinc and iron contents in samples taken from sampling locations 02 (cleaner concentrate) and 03 (copper tailing) are presented. As explained in Chapter 3.5.3, the final concentrate samples were already taken after the first cleaner flotation, due the problems occurred during the run. As shown in Figure 35 the copper content is radically dropped after 90 minutes, due to the change made in the milk of lime feed. The copper content reaches its highest value of 12.4% after 90 min of running. From there the copper content is descends to ca. 4%. Overall, the pH level in the flotation is not directly affecting to the flotation of copper particles but it is depressing iron particles. As presented in Figure 35, the iron and the copper contents seem to be somehow related to each other. When there is a drop in the copper content the iron content increases and when the copper content increases the iron content drops. In Appendix 11, the element contents, the content averages and the standard deviation of each of the elements for the sampling location 02 are presented.

As shown in Figure 36 the copper and zinc contents in samples taken from sampling location 03 are relatively stable. The iron content rises from 30% to 32%, during the run. In Appendix 12, the element contents, the content averages and the standard deviation of each element for the sampling location 03 are presented.

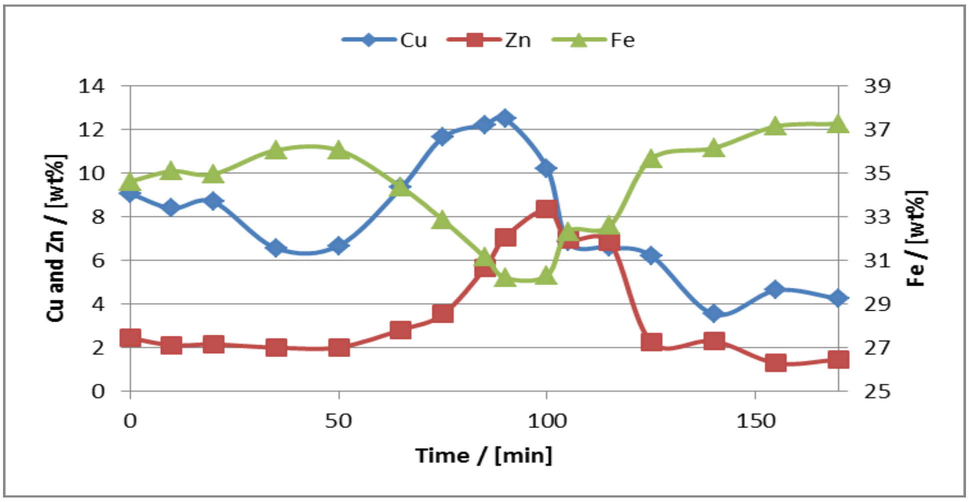

Figure 35. Cu, Zn and Fe contents on XRF samples from the sampling location 02.

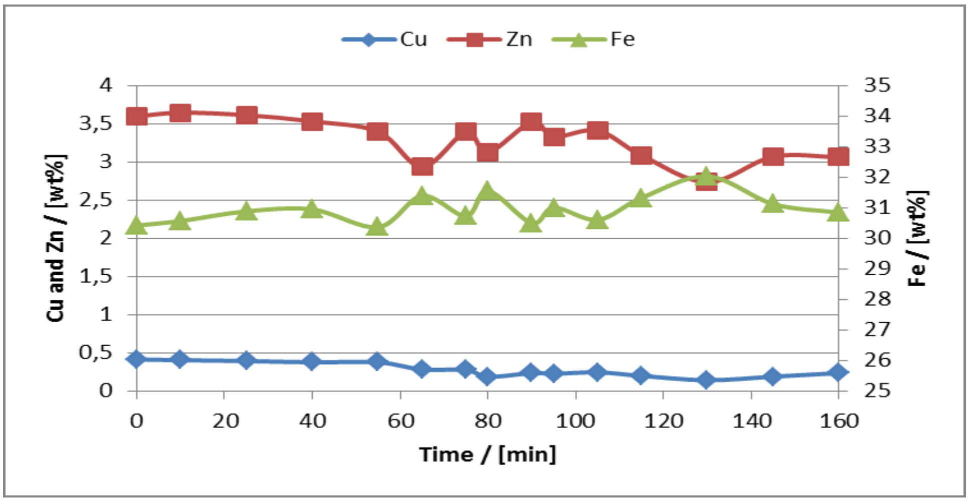

Figure 36. Cu, Zn and Fe contents on XRF samples from the sampling location 03.

4.3.2 pH

Figure 37 presents the pH changes in the rougher, scavenger and third cleaner flotation units during the third trial run. At the beginning of the run, the pH level in the rougher flotation changes from 10 to 11. In the scavenger and the third cleaner flotation, the change is from 9 to 10 as shown in Figure 37. 40 min after the run was started, the pH in each cell went up to 12.5. Figure 16 shows, that the time delay is the shortest between the conditioner and rougher flotation, and the longest between the conditioner and scavenger flotation.

From Figure 37, a time constant, a time delay and a slope for the rougher, the first cleaner, the final concentrate and the scavenger flotation were determined. The time constant represents the time it takes the pH to reach 63.2% of its final value. The time constant for the rougher flotation was 16 min and for the first cleaner flotation it was 18 min. The pH of the scavenger flotation was not stabilised before the sampling was ended. Thus, the time constant for the scavenger flotation was not counted. The milk of lime feed to the conditioner was stopped 83 min after the first sample was taken. The time delay represents the time it takes to notice changes on the pH after the milk of lime feed to the conditioner was stopped. The time delay for the rougher flotation was 13 min, for the first cleaner flotation 21 min and for the scavenger flotation 31 min. From the slopes of the pH curves the rate of change can be defined. The slopes of the pH curves of the flotation cells during the change are: The rougher flotation 65 1/min, the first cleaner flotation 63 1/min, the first cleaner flotation 60 1/min and the scavenger flotation 36 1/min. Figure 38 presents the online measurements of the pH from the conditioner. As presented in Figure 38, the conditioner pH curve has similar form as the pH curve of the rougher flotation.

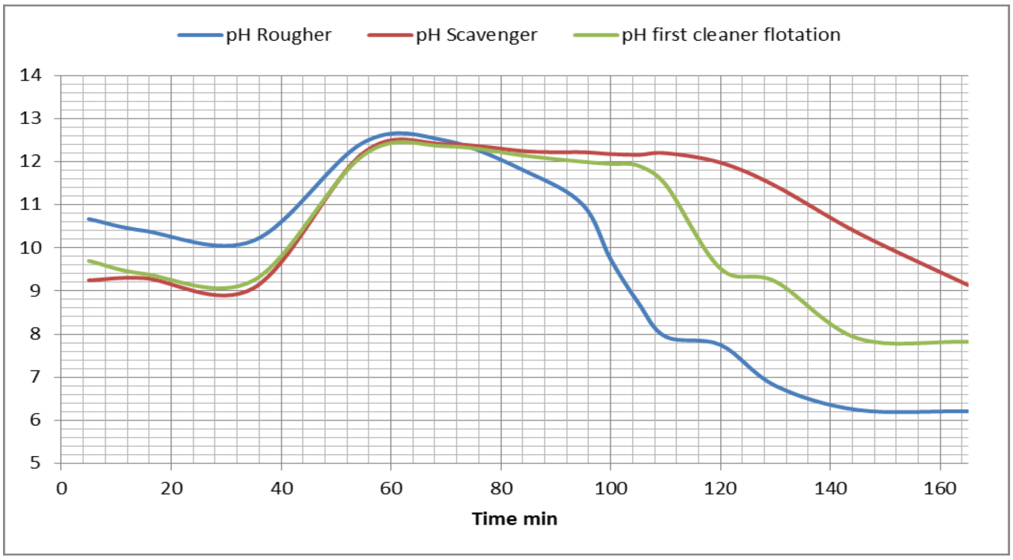

Figure 37. The pH from different cells.

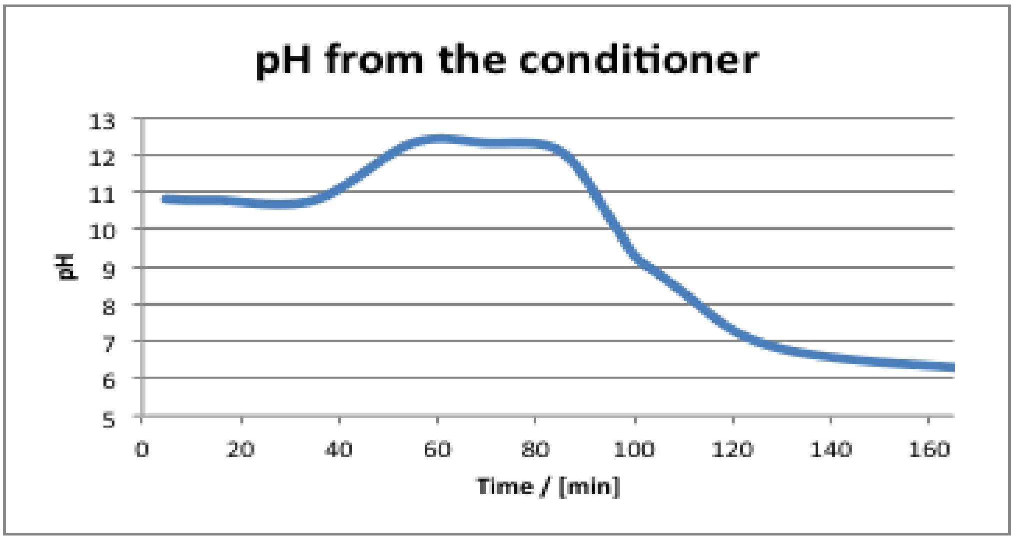

Figure 38. The pH from the conditioner

4.3.3 Particle size distribution

The particle size analyses were made for the outputs of the rod and ball mills, the classifier overflow, the copper concentrate and the copper tailing before and after the pH change as shown in Figure 39 and 40. Sieve opening size is presented as a logarithmic scale and the vertical axis presents the percentage of ore passed the sieves.

As shown in Figure 39, small particles in the flotation are in the copper concentrate and bigger particles in the copper tailing. 80 percent of the copper concentrate particles are smaller than 55 µm, when in the tailing the d80 value is 70 µm. Figure 40 presents that the rod mill outflow includes bigger particle sizes than the ball mill outflow. The 80% ore passed value for the rod mill is 75 µm, and 65 µm for the ball mill.

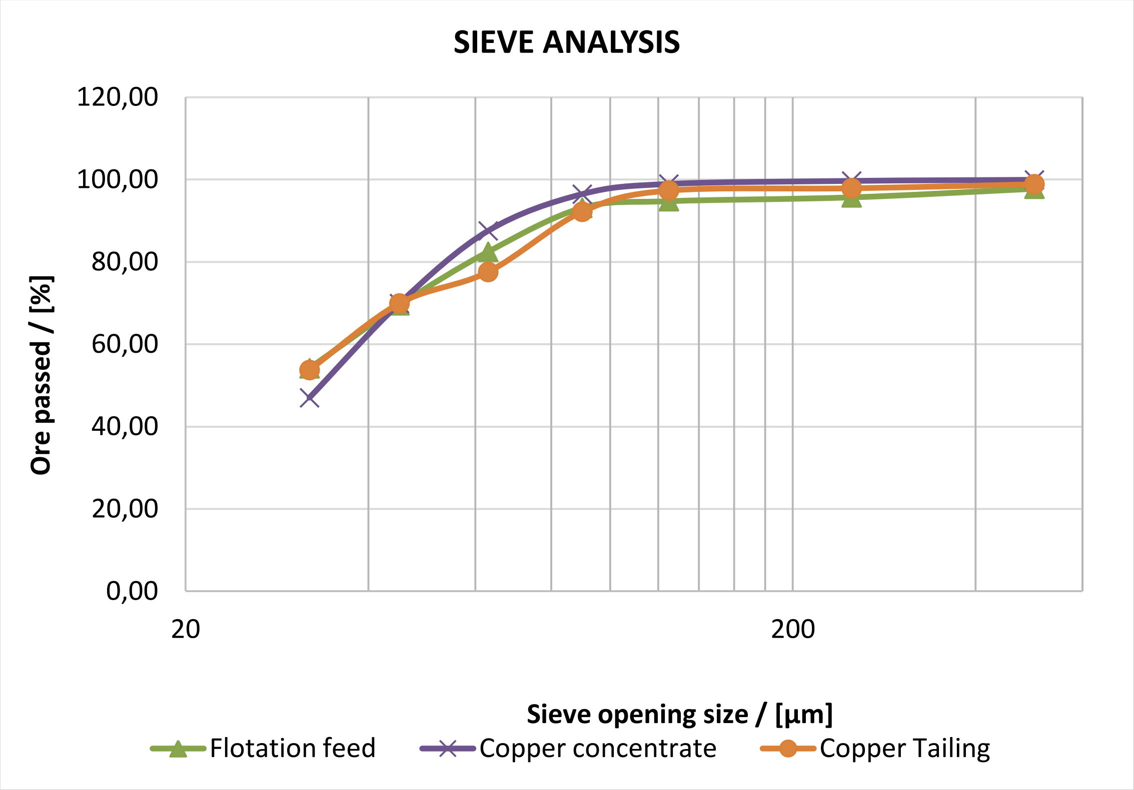

Figure 39. Particle size distribution for the flotation.

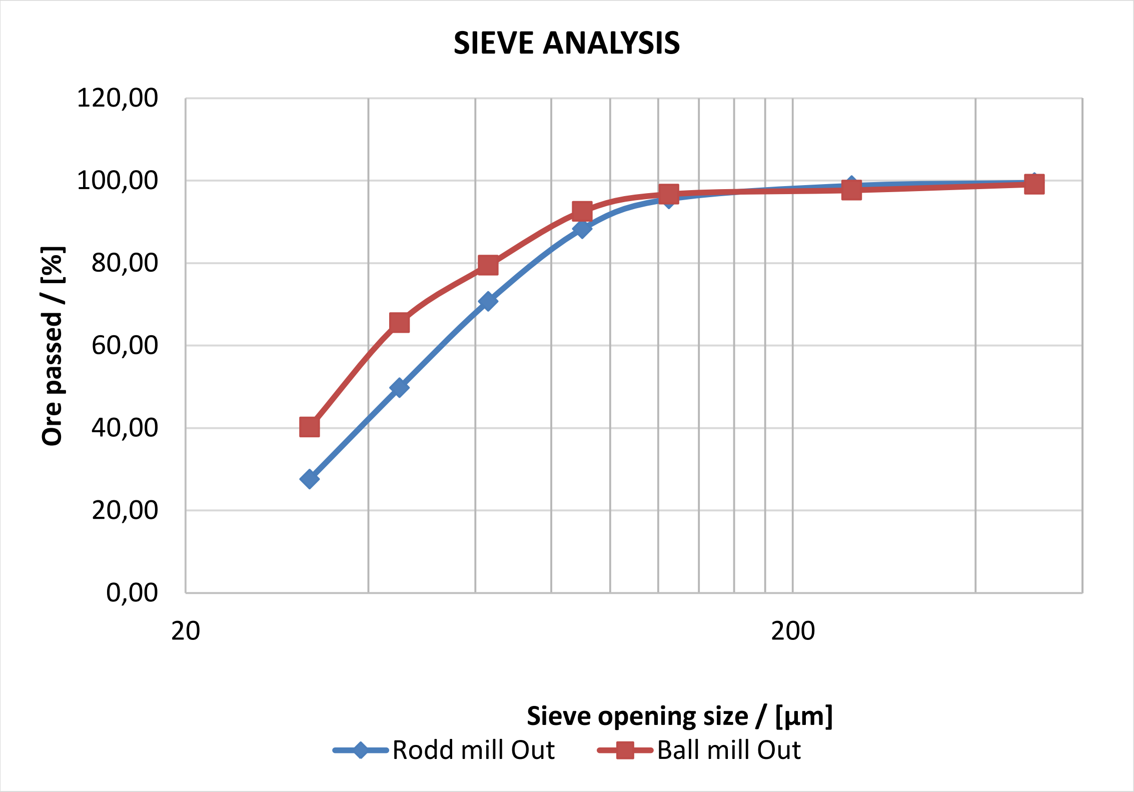

Figure 40. Particle size distribution for the rod and the ball mill.

4.3.4 Observations

The mixing impeller in flotation cells 1 and 2, one in each, were adrift. It caused problems due to the blocked air feeding and lack of mixing. Bypassing the broken section of the cell and feeding the slurry to the next section solved the problem. However it decreased the bubbling area of flotation cells 1 and 2, which decreased the concentrate flow rates from the cells. Thus it was decided to take the final concentrate already after the first cleaning flotation.

With the milk of lime feed rate of 400 ml/min the slurry pH level 12 was reached. For some reason the slurry pH level was first under 12 and suddenly, without any changes made to the process, rose to 12. The step change was seen from the XRF and pH analyses.

5 Discussion and future aspects

The ore beneficiation process is a highly complex process with multiple variables affecting the results. To achieve good concentrate grades and recoveries all the unit processes including grinding, conditioning and flotation must be working flawlessly. As expected, the results obtained throughout the three trial runs of the minipilot experiments performed in this thesis work highlighted the fact that even in pilot scale ore beneficiation process the individual runs are extremely hard to be replicated. For the future runs, first only the rougher flotation should be used when the influences of different variables to process are investigated. Also the recycling flows from the flotation back to the conditioner can be taken off, to obtain an open loop system. The circulating flows cause disturbances to the process, which have an effect on the results. The open loop system also makes the response faster between the conditioner and the flotation.

The analysed samples of the sampling location 00 shows that the ore handling, presented in Chapter 3.2, was executed correctly and the ore had relatively uniform quality in all runs. The average values and standard deviations from the trial runs are presented in Table 9. Overall the results from the sampling location 00 were quite stable and only relatively small changes were observed during the runs. As shown in Table 9, ore samples from the trial runs 2 and 3 were really close to each other.

Table 9. Element content averages and standard deviations for the sampling location 00.

	Cu (%)	Zn (%)	S (%)	Fe (%)	Pb (%)
Run 1					
Average	0,943636	3,214455	37,14491	30,64327	0,069636
Standard deviation	0,177498	0,418368	1,123983	1,942628	0,004081
Run 2					
Average	1,070455	3,837455	35,59182	28,00373	0,073
Standard deviation	0,047905	0,108783	0,309015	0,362905	0,004025
Run 3					
Average	1,074727	3,760091	36,00182	28,22182	0,125727
Standard deviation	0,043587	0,140026	0,491655	0,55252	0,176566

The results from the sampling location 01 presents that the material fed to the flotation was relatively of uniform quality as shown in Table 10. Overall the results from the sampling location 01 indicate that the grinding section of the process is reproducible when the ore handling is made correctly. The average contents of elements are close to values presented in Table 9. The standard deviation values in Table 10 are mostly increased compared to Table 9. The reason for this is that the sampling location 01 is further, and influenced for more disturbances, in the process than sampling location 00.

Table 10. Element content averages and standard deviations for the sampling location 01.

	Cu (%)	Zn (%)	S (%)	Fe (%)	Pb (%)
Run 1					
Average	0,923941	3,522412	37,58412	30,77429	0,073294
Standard deviation	0,061476	0,360484	1,290755	1,153199	0,001896
Run 2					
Average	1,021	3,055364	38,21809	32,07882	0,076182
Standard deviation	0,192986	0,435997	0,96727	1,246735	0,002401
Run 3					
Average	1,012818	3,11	37,83573	31,71645	0,076
Standard deviation	0,174806	0,290163	1,283588	1,18752	0,002757

The influence of pH to the copper content of the final concentrate is presented in Figures 41 and 42. Figure 41 shows the results from the second run and Figure 42 from the third run. Results from the first run are not shown, due to the low pH values in the third cleaning flotation. The copper content is clearly correlating with the changes in pH as shown in both of the Figures. Unfortunately the desired pH level 12 was not reached in second run as presented in Figure 41. Thus, the results in Figures 41 and 42 are not fully comparable. In both figures, there are some unexpected changes in the copper content, which is not explicable by changes in pH. As shown in Figure 41, there is a drop in the copper content, at the beginning of the run, even though the pH value is stable. In the third run the copper content dropped from 12% to 7%, when 90 min of time was passed. There are no clear evidences that explain why these changes happened. Thus, further trial runs and closer investigation are needed in the future.

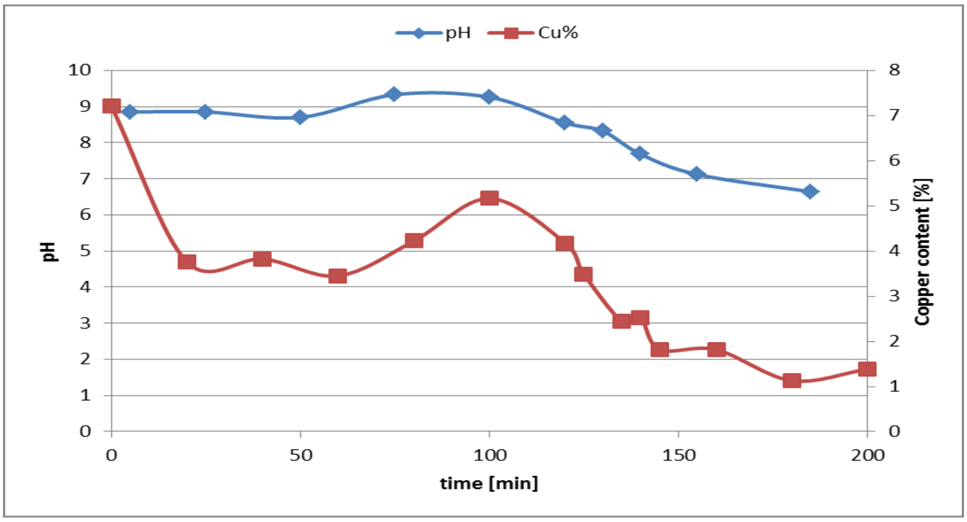

Figure 41. Concentrate copper content of the second run when the pH is changing.

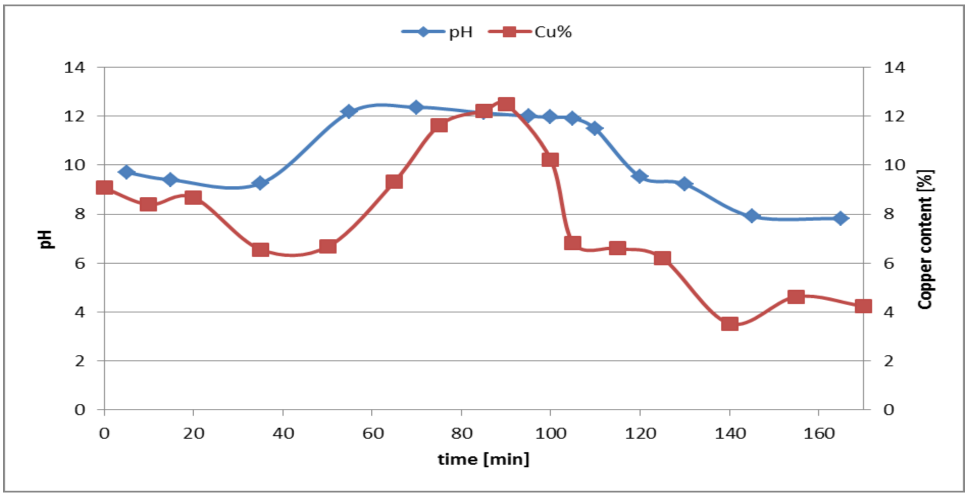

Figure 42. Concentrate copper content of the third run when the pH is changing.

Only one set of particle size analyses for the outputs of the rod and ball mills, the classifier overflow, the copper concentrate and the copper tailing were made for each trial run. Results from the analyses were relatively similar between the different trial runs. As expected the particle size of the ball mill outflow is smaller than the rod mill outflow. As introduced in the results, the copper concentrate samples have smaller particle sizes compared with the copper tailing samples. Thus, the effective and reproducible grinding is highly important to accomplish good results from the flotation.

Each of the trial runs was different and multiple problems were occurred during them. In the first trial run, there was a problem with the feed of the milk of lime into the mills. The problem was solved, by shifting the milk of lime feeding tubes from the mills to the conditioner. The next problem was surprisingly long time delays between the conditioner and flotation cells. After the milk of lime feed was started during the first run, it was noticed that the pH level in the cleaner and scavenger was not increasing as fast and as high as expected. For the next trial runs, the change was executed in the opposite direction.

In the second trial run, everything was working as planned. The only problem was the pH level in the flotation, which was too low to accomplish a high copper grade for the concentrate samples. For the third run, the milk of lime feed rate was increased from 300 ml/min to 400 ml/min and the desirable pH level was accomplished. In the third trial run, there were some mechanical problems with the mixing impellers in the rougher and scavenger flotation’s. This caused great decreases in the flow rates between the cells. Thus the final concentrate samples were taken already after the first cleaning flotation.

Even though all the runs had some problems, the time constants and delays for the trial runs 2 and 3 were analysed based on the measured step responses. The time constant between the conditioner and the rougher flotation for the second run was 26 min and for the third run 16 min, but for the first cleaner flotation the time constant was the same for the both runs, 18 min. It is not clear why the time constant value between the conditioner and the rougher flotation changed so much between the runs, but there might been some changes in the flotation rates of the slurry. For further conclusions more trial runs and data from the process is needed. The time constant of the process can also be evaluated from the slopes of the curves during the descent of the pH. From the slopes of the pH curves the rate of change can be defined. The calculated slope of the rougher flotation in the second trial run was 66 1/min and in the third run 65 1/min. The slope of the scavenger flotation in the second run was 33 1/min and in the third run 36 1/min, and for the first cleaner flotation 63 1/min and 60 1/min. The slope values are relatively similar between the runs, thus the rates of change between the runs are close to each other even though the time constants do not match. The time delay between the conditioner and rougher flotation on the second run was 7 min. To define the time delay of the third run's rougher flotation was challenging due to the shape of the curve. The time delay between the conditioner and the rougher flotation was carefully evaluated to be 13 min. The time delays of the first cleaner flotation and the scavenger flotation were relatively close in both runs. In the second run the first cleaner flotation had the time delay of 25 min and in the third run 21 min. The time delay of the scavenger in the second run was 35 min and in the third run 31 min. More trial runs are needed in future to confirm the results.

Multiple mechanical problems other than those mentioned in Chapter 3.5 were observed during the runs. The list of the problems is presented below:

- Water feed to the feeding funnel before the rod mill is poorly designed. Ore gets jammed and has to be poked inside to the rod mill.
- The hydraulic circuit of the screw classifier was not working properly. Slurry was lodged in the bottom and on the walls of the classifier, which caused flooding.
- The hose pumps on the grinding circuit were sized for larger flow rates. The feed from the pumps was not continuous, which caused problems for the flow rate measurements.
- The hose pumps on the flotation circuit were sized for smaller flow rates. If the flow rates or recycled streams were increased even a little, the conditioner was flooding.
- The chemical reagent hose pumps were sized for larger flow rates. The chemical reagent feed was inconstant, which caused trouble in the conditioner where slurry and chemical reagents were mixed. When the

chemical reagent flow is stopped, the ore gets inside the tubes, causing problems and jamming the tubes.

- The mixing impellers for the flotation cells were too powerless to produce a complete mixing of the slurry. The heaviest particles were sunk to the bottom.

From the each trial run around 60 XRF samples were taken. The amount of samples is problematic because each of the samples has to be dried, grind and pressed to pellets before it can be analysed by the XRF. To analyse all the samples from the trial run it took approximately one week. There are multiple different online and semi-online analysers in the markets, which give information about the conditions of the flotation. In example online froth cameras can be used to analyse the bubble size and colour to get directional knowledge of flotation conditions. This would decrease the number of samples taken, thus the sampling can be started when the condition in the flotation is as wanted. The sampling time of the process can also be adjusted, thus the cameras detect the changes in the froth. For the faster sampling, different semi-online XRF analysers had been developed. The samples can be analysed after dried, without further treatments. When analysing the outputs of the dynamic process as the flotation process, the above-mentioned analysing devices can ease the task.

Gathering information of the slurry flow rates of the input and output flows of the flotation, and also between the flotation's units, would make the calculation of mass balances and recoveries possible. A mass balance is used to calculate mass flow rates of different streams entering or leaving the process. In the flotation process investigated in the thesis, there were one input flow (from the conditioner to the rougher flotation) and two output flows (The copper concentrate and the copper tailing). To create a mass balance for the flotation, the flow rate measurements from two of them are required. The mass balance can be used for developing the process, planning the needed controls and to get knowledge of the bottlenecks of the process. To calculate the recoveries of each element measured at the XRF, the input and output flows of the flotation would be needed.

For the first trial runs, the chosen process area, from the grinding to the third cleaning flotation, was too wide. For the future runs, the rougher flotation should be first studied individually in the open loop system by closing the circulating flows from the flotation cells to the conditioner. From there the effects of the changes of different variables could be measured and controlled better. When the rougher unit is analysed, flotation units can be added to the analysis one at a time, until the whole flotation train is investigated and the problematic sections are found.

In the future different simulation software can be used for developing and analysing the process, and for the control design. The results and data collected during the trial runs introduced in this work can be used as a basic data for developing the simulator. Further reliable trial runs are needed to achieve and identify a reliable simulator of the minipilot.

6 Conclusions

The continuous minipilot beneficiation plant is situated in the Department of Process and Environment Engineering of the University of Oulu. The planning basic for the minipilot plant was the ore beneficiation process of the Pyhäsalmi mine in scale of 1:5000. Outotec Oyj and Schneider Electric together with the experts from the Geological Survey of Finland and Pyhäsalmi mine have been the main planners and equipment suppliers.

The main target of this work was to produce a guideline manual for the use of the minipilot ore beneficiation plant and collect data from the process during various trial runs. The collected data was later used to investigate the dynamics and time delays of the process. All the occurred problems and observations done during the trial runs were note, and are introduced in this thesis. Practical measurement device which would improve the repeatability and continuity of the process were also discussed. All the trial runs and experiments were executed on the minipilot ore beneficiation plant and the milk of lime feed was the manipulated variable in all experiments.

The main object of the experimental part of this work was to collect data from the minipilot process under different operating conditions and subjected to step changes of certain variables. Three trial runs were executed during the experimental part of the thesis. For each trial run, XRF, pH and particle size samples were taken. The collected data was later used to investigate the process dynamics and time delays. Performed investigations give important knowledge about how to improve the repeatability and continuity of the process. Also the measuring devices and improvements needed for the process are discussed based on the data and knowledge received from the trial runs. As expected, the results obtained throughout the three trial runs of the minipilot experiments performed in this thesis work highlighted the fact that even in pilot scale ore beneficiation process the individual runs are extremely hard to be replicated.

The analysed samples of the sampling location 00 shows that the ore handling was executed correctly and the ore had relatively uniform quality in all runs. The results from the sampling location 01 presents that the material fed to the flotation was relatively of uniform quality. Overall the results from the sampling location 01 indicate that the grinding section of the process is reproducible when the ore handling is made correctly. The standard deviation values are mostly increased compared values from sampling location 00 and 01. The reason for this is that the sampling location 01 is further, and influenced for more disturbances, in the process than sampling location 00.

The results from the experimental runs 2 and 3 are not fully comparable, but from both runs the step change made to the milk of lime feed was clearly seen from the XRF results. Even though all the runs had some problems, the time constants and delays for the trial runs 2 and 3 were analysed based on the measured step responses. The time constant between the conditioner and the rougher flotation for the second run was 26 min and for the third run 16 min, but for the first cleaner flotation the time constant was the same for the both runs, 18 min. From the slopes of the pH curves the rate of change can be defined. The calculated slope of the rougher flotation in the second trial run was 66 1/min and in the third run 65 1/min. The slope of the scavenger flotation in the second run was 33 1/min and in the third run 36 1/min, and for the first cleaner flotation 63 1/min and 60 1/min. The slope values are relatively similar between the runs, thus the rates of change between the runs are close to each other even though the time constants do not match.

Each of the trial runs was different and multiple problems were occurred during them. To confirm the results obtained from the experiment runs multiple trial runs are needed in the future. For the future runs, first only the rougher flotation should be used when the influences of different variables to process are investigated. Also the recycling flows from the flotation back to the conditioner can be taken off, to obtain an open loop system. The circulating flows cause disturbances to the process, which have an effect on the results. The open loop system also makes the response faster between the conditioner and the flotation.

The results and data collected during the trial runs introduced in this work can be used as a basic data for developing the simulator. Further reliable trial runs are needed to achieve and identify a reliable simulator of the minipilot.

7 References

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LIST OF APPENDICES

Appendix 1. Element analysis of the first trial run (sampling location 00)

Run 1	29.8.2013						
			Sample 00		From the silo		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
	Time min		(%)	(%)	(%)	(%)	(%)
	0		1,136	3,596	37,201	30,456	0,069
	15		0,971	3,408	36,664	29,374	0,071
	30		0,775	3,103	37,387	31,511	0,065
	45		1,051	2,826	38,09	32,591	0,08
	60		0,744	2,36	38,756	33,778	0,069
	75		0,834	2,988	37,446	30,463	0,066
	90		0,841	3,131	37,329	30,65	0,067
	105		0,682	3,017	38,569	33,039	0,067
	120		1,084	3,762	35,128	27,653	0,071
	135		1,222	3,541	36,191	28,961	0,069
	150		1,04	3,627	35,833	28,6	0,072
Standard deviation			0,177498	0,418368	1,123983	1,942628	0,004081
Average			0,943636	3,214455	37,14491	30,64327	0,069636

Appendix 2. Element analysis of the first trial run (sampling location 01)

			Sample 01		From the conditioner		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
			(%)	(%)	(%)	(%)	(%)
	Time min						
	0		1,067	2,828	38,858	32,834	0,076
	15		0,934	3,057	38,174	31,824	0,075
	30		1,014	3,175	37,985	31,77	0,075
	45		0,971	3,334	37,809	31,166	0,075
	60		0,937	3,612	37,376	30,614	0,072
	75		0,89	3,694	37,129	30,153	0,072
	90		0,922	3,519	41,417	32,68	0,076
	95		0,858	3,926	36,45	29,444	0,072
	100		0,848	3,934	36,403	29,27	0,071
	105		0,85	3,942	36,303	29,406	0,07
	110		0,929	3,184	37,556	31,438	0,073
	115		0,849	4,157	35,443	28,896	0,072
	125		0,871	3,791	36,974	30,022	0,072
	135		0,983	3,298	37,962	31,18	0,076
	145		0,929	3,623	37,534	30,546	0,072
	160		0,946	3,393	37,906	31,061	0,073
	175		0,909	3,414	37,651	30,859	0,074
Standard deviation			0,061476	0,360484	1,290755	1,153199	0,001896
Average			0,923941	3,522412	37,58412	30,77429	0,073294

Appendix 3. Element analysis of the first trial run (sampling location 02)

			Sample 02		Copper concentrate		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
			(%)	(%)	(%)	(%)	(%)
	Time min						
	0		1,502	1,02	43,499	38,805	0,078
	15		1,369	1,007	43,146	38,658	0,079
	30		1,375	1,416	43,637	38,548	0,08
	45		1,197	1,301	43,479	38,759	0,08
	60		1,114	1,423	43,167	38,52	0,082
	75		1,161	1,937	43,221	38,146	0,083
	80		1,287	1,87	43,425	38,197	0,083
	85		1,62	1,682	43,224	38,11	0,083
	90		1,515	1,43	43,323	38,393	0,083
	95		1,376	1,233	42,952	38,335	0,081
	100		1,304	1,183	42,897	38,449	0,079
	105		1,575	0,99	43,273	38,594	0,079
	115		1,733	0,996	43,544	38,648	0,081
	125		1,953	0,808	43,514	38,533	0,081
	135		2,042	0,745	43,52	38,616	0,08
	160		1,692	0,886	43,534	38,558	0,085
	175		1,736	1,018	42,932	38,191	0,082
Standard deviation			0,268514	0,354926	0,234639	0,213749	0,0019
Average			1,503	1,232059	43,311	38,47412	0,081118

Appendix 4. Element analysis of the first trial run (sampling location 03)

			Sample 03		Copper tailing		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
			(%)	(%)	(%)	(%)	(%)
	Time min						
	0		0,29	4,531	32,509	26,017	0,081
	15		0,271	4,838	32,281	25,507	0,079
	30		0,264	4,889	31,965	25,566	0,08
	60		0,213	5,837	30,513	23,678	0,072
	65		0,179	6,667	28,255	22,527	0,064
	70		0,211	5,671	30,194	22,789	0,073
	75		0,211	5,768	29,538	22,397	0,072
	80		0,178	6,653	28,612	21,692	0,067
	85		0,176	6,71	27,256	20,458	0,065
	90		0,156	7,187	26,879	18,859	0,063
	100		0,17	6,874	27,089	19,193	0,066
	110		0,147	7,226	26,361	17,498	0,058
	120		0,162	7,177	27,12	18,411	0,061
	145		0,184	7,009	27,817	19,463	0,064
	160		0,13	9,188	26,755	19,409	0,052
Standard deviation			0,047185	1,194208	2,146089	2,786365	0,008308
Average			0,196133	6,415	28,87627	21,56427	0,0678

Appendix 5. Element analysis of the second trial run (sampling location 00)

Run 2	20.9.2013						
			Sample 00		From the silo		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
	Time min		(%)	(%)	(%)	(%)	(%)
	0		1,056	3,993	35,719	28,281	0,067
	20		1,045	3,723	35,746	28,313	0,073
	40		1,112	3,816	35,921	28,137	0,076
	60		1,042	3,909	35,394	28,014	0,082
	80		1,114	3,68	35,179	27,807	0,072
	100		0,983	3,794	35,799	28,165	0,069
	120		1,117	3,842	35,801	28,063	0,07
	140		1,068	3,98	35,451	27,902	0,075
	160		1,128	3,952	35,115	27,264	0,072
	180		1,01	3,718	36,041	28,546	0,072
	200		1,1	3,805	35,344	27,549	0,075
Standard deviation			0,047905	0,108783	0,309015	0,362905	0,004025
Average			1,070455	3,837455	35,59182	28,00373	0,073

Appendix 6. Element analysis of the second trial run (sampling location 01)

			Sample 01		From the conditioner		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
			(%)	(%)	(%)	(%)	(%)
	Time min						
	0		0,87	3,467	37,411	31,022	0,075
	20		0,813	3,41	37,433	30,913	0,073
	40		1,169	3,016	38,074	32,022	0,079
	60		0,831	3,55	37,3	30,67	0,072
	80		0,86	3,448	37,139	30,849	0,075
	100		0,976	3,341	37,441	31,221	0,076
	120		1,471	2,607	39,28	33,317	0,079
	140		1,146	2,298	39,931	34,081	0,079
	160		1,065	2,567	38,932	33,441	0,076
	180		1,003	3,164	38,303	32,055	0,076
	200		1,027	2,741	39,155	33,276	0,078
Standard deviation			0,192986	0,435997	0,96727	1,246735	0,002401
Average			1,021	3,055364	38,21809	32,07882	0,076182

Appendix 7. Element analysis of the second trial run (sampling location 02)

			Sample 02		Copper concentrate		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
			(%)	(%)	(%)	(%)	(%)
	Time min						
	0		7,211	1,644	41,978	36,101	0,09
	20		3,755	1,423	42,46	37,502	0,087
	40		3,825	1,316	42,499	37,518	0,092
	60		3,45	1,243	42,163	37,483	0,09
	80		4,221	1,352	41,941	37,245	0,096
	100		5,165	1,482	41,73	36,68	0,091
	120		4,178	1,363	42,256	37,284	0,094
	125		3,483	1,334	42,467	37,622	0,092
	135		2,441	1,173	42,633	38,062	0,093
	140		2,519	1,127	43,023	38,206	0,092
	145		1,817	1,09	42,831	38,506	0,084
	160		1,816	1,287	43,415	38,552	0,082
	180		1,128	1,321	43,624	38,626	0,079
	200		1,384	1,528	43,37	38,44	0,08
Standard deviation			1,642402	0,152923	0,586642	0,747839	0,005413
Average			3,313786	1,3345	42,59929	37,70193	0,088714

Appendix 8. Element analysis of the second trial run (sampling location 03)

			Sample 03		Copper waste		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
			(%)	(%)	(%)	(%)	(%)
	Time min						
	0		0,39	4,133	35,131	28,86	0,081
	20		0,319	4,349	35,091	28,802	0,077
	40		0,294	4,144	35,473	28,668	0,077
	60		0,247	4,154	35,451	28,484	0,073
	80		0,182	3,892	36,289	29,429	0,069
	100		0,188	4,052	36,653	29,573	0,07
	120		0,159	3,709	36,768	29,948	0,069
	125		0,15	3,593	36,763	30,019	0,073
	135		0,204	3,85	36,523	29,588	0,072
	140		0,118	3,543	37,023	30,182	0,068
	145		0,127	3,479	37,32	30,453	0,068
	160		0,131	3,76	36,736	29,98	0,068
	180		0,155	3,95	36,115	29,369	0,069
	200		0,137	3,873	36,243	29,344	0,07
Standard deviation			0,082639	0,257629	0,71274	0,604357	0,004065
Average			0,200071	3,8915	36,25564	29,4785	0,071714

Appendix 9. Element analysis of the third trial run (sampling location 00)

run 3	17.10.2013						
			Sample 00		From the silo		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
	Time min		(%)	(%)	(%)	(%)	(%)
	0		1,031	3,621	35,647	27,537	0,071
	20		1,05	3,922	36,573	28,577	0,078
	40		1,152	3,938	36,086	28,009	0,069
	60		1,125	3,7	35,125	27,543	0,658
	60		1,054	3,958	35,448	27,851	0,076
	75		1,019	3,55	36,829	29,461	0,068
	90		1,102	3,669	36,347	28,61	0,077
	105		1,121	3,839	36,11	28,39	0,071
	120		1,038	3,671	36,046	28,15	0,071
	135		1,062	3,679	36,057	28,363	0,07
	150		1,068	3,814	35,752	27,949	0,074
Standard deviation			0,043587	0,140026	0,491655	0,55252	0,176566
Average			1,074727	3,760091	36,00182	28,22182	0,125727

Appendix 10. Element analysis of the third trial run (sampling location 01)

			Sample 01		From the conditioner		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
			(%)	(%)	(%)	(%)	(%)
	Time min						
	0		1,018	3,363	37,732	31,418	0,078
	20		1,001	3,355	38,52	31,546	0,076
	40		0,925	3,187	37,705	31,656	0,075
	60		0,778	3,26	36,812	30,701	0,073
	60		0,788	3,252	35,249	29,755	0,07
	75		0,892	3,404	36,858	30,684	0,075
	90		1,337	3,329	37,143	30,999	0,078
	105		1,26	2,924	38,24	32,39	0,08
	120		1,117	2,822	39,168	33,046	0,076
	135		0,999	2,598	39,185	33,278	0,078
	150		1,026	2,716	39,581	33,408	0,077
Standard deviation			0,174806	0,290163	1,283588	1,18752	0,002757
Average			1,012818	3,11	37,83573	31,71645	0,076

Appendix 11. Element analysis of the third trial run (sampling location 02)

			Sample 02		Copper concentrate		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
			(%)	(%)	(%)	(%)	(%)
	Time min						
	0		9,066	2,446	41,01	34,614	0,097
	10		8,387	2,114	41,155	35,081	0,094
	20		8,675	2,154	40,999	34,949	0,09
	35		6,557	2,006	41,3	36,045	0,085
	50		6,669	2,016	41,458	36,042	0,086
	65		9,328	2,82	40,61	34,348	0,084
	75		11,625	3,528	39,69	32,847	0,087
	85		12,214	5,628	39,053	31,152	0,09
	90		12,476	7,073	38,872	30,193	0,096
	100		10,213	8,334	39,23	30,307	0,094
	105		6,828	6,953	40,156	32,32	0,095
	115		6,598	6,827	40,189	32,576	0,094
	125		6,201	2,256	41,282	35,654	0,087
	140		3,531	2,303	41,742	36,146	0,089
	155		4,633	1,289	42,175	37,136	0,094
	170		4,24	1,463	42,142	37,25	0,105
Standard deviation			2,770985	2,37725	1,058481	2,318578	0,005474
Average			7,952563	3,700625	40,69144	34,16625	0,091688

Appendix 12. Element analysis of the third trial run (sampling location 03)

			Sample 03		Copper tailing		
			Cu	Zn	S	Fe	Pb
			Cu4	Zn3	S3	Fe5	Pb2
			(%)	(%)	(%)	(%)	(%)
	Time min						
	0		0,418	3,599	37,398	30,426	0,081
	10		0,409	3,646	37,282	30,574	0,081
	25		0,397	3,614	37,58	30,89	0,08
	40		0,381	3,533	37,835	30,954	0,079
	55		0,381	3,398	36,651	30,374	0,078
	65		0,287	2,939	37,582	31,395	0,078
	75		0,282	3,395	36,872	30,735	0,072
	80		0,192	3,128	37,747	31,558	0,069
	90		0,24	3,524	35,926	30,505	0,069
	95		0,231	3,332	37,244	31,013	0,068
	105		0,25	3,412	36,599	30,614	0,069
	115		0,204	3,088	37,083	31,33	0,07
	130		0,147	2,737	37,875	32,035	0,069
	145		0,194	3,071	37,001	31,128	0,071
	160		0,24	3,065	36,944	30,848	0,071
Standard deviation			0,090533	0,276952	0,534433	0,466197	0,005094
Average			0,283533	3,298733	37,1746	30,9586	0,073667