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Development Towards a New Test Procedure for Flotation at Brønnøy Kalk

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Problem Description

Brønnøy Kalk is currently using a standardised flotation test procedure to check the quality of their products. The aim of the project is to revise this test so it can be used with a new collector. The effect of various test variables will be investigated (including the particle size distribution of the feed) in order to determine their relative importance. This should result in recommendations for a new test procedure.

Keywords:

1. Froth flotation
2. Amine collector
3. Particle size distribution

(sign.)

Abstract

Brønnøy Kalk AS is a mining company established in 1997 and is localized in Brønnøy, Norway. Approximately 2 million tonnes of ground calcium carbonate (GCC) raw material is mined annually. No processing of the marble is performed by Brønnøy Kalk; the raw material is sent to an off-site processing plant where the impurities are removed using reverse froth flotation. However, the quality of the blocks in the open pit is analysed in a laboratory procedure at Brønnøy Kalk. In short, the blocks are analysed for expected brightness, to enable the correct mixing of different grades. As part of the quality inspection, the raw material is subjected to bench scale reverse froth flotation. The processing plant successfully switched the collector used in the flotation process at the plant, prompting the same change of the collector used in the bench scale flotation procedure at Brønnøy Kalk. However, initial testing with the new collector proved difficult, as the results achieved with the standard flotation procedure used with the new collector resulted in a high deviation from the results achieved when using the original collector, as well as low reproducibility in replicates when using the new collector.

The first part of the study addresses the use of an off-site laboratory for performing flotation experiments, as part of reducing the need to travel to Brønnøy Kalk to perform experiments. The second part of the study addresses the assumed reasons for the difficulties in using the new collector in the bench scale flotation procedure at Brønnøy Kalk. The main experiments were on the particle size distribution (PSD) of the flotation feed, as it was assumed that the lifting capacity of the new collector was less than for the original collector. The reduction of the PSD was assumed to increase the reproducibility of the flotation procedure.

The main findings of the study includes the difficulties faced when using off-site laboratories, and that the reduced PSD of the flotation feed improved the flotation process when using the new collector. Using the laboratories at NTNU proved problematic, as many aspects of the flotation procedure were different from the procedure used at Brønnøy Kalk. The flotation cell and the flotation operators are emphasized. The study on the PSD revealed increased brightness in the final concentrate when reducing the PSD of the flotation feed. The experiments also showed reduced variance in the replicate trials when reducing the PSD of the flotation feed. The thesis includes a suggestion for a new flotation procedure for flotation using amine B.

Sammendrag

Brønnøy Kalk AS ble etablert i 1997, og er lokalisert i Brønnøy, Norge. Dagbruddet produserer omtrent 2 millioner tonn kalsium karbonat (kalsitt/kalkspat) som råmateriale til papirindustrien. Kalken oppredes ikke av Brønnøy Kalk, men transporteres med skip til en oppredningsfabrikk hvor forurensningsmineralene fjernes med revers flotasjon. Kvaliteten av blokkene i dagbruddet analyseres hos Brønnøy Kalk for å kunne blande de forskjellige "gehaltene", og som del av kvalitetssjekken fjernes forurensningsmineralene i råmaterialet med benkeskala flotasjon. "Gehalten" ble hovedsakelig analysert ved å måle hvitheten til konsentratet av flotasjonsprosessen.

Oppredningsfabrikken har byttet flotasjonssamleren og det samme byttet er ønsket i benkeskala flotasjonen i kvalitetssjekken hos Brønnøy Kalk. Forberedende testing med den nye samleren viste at dette var vanskelig. Resultatene oppnådd av flotasjon med den nye samleren viste store avvik fra resultatene oppnådd av flotasjon med den originale samleren. Det viste seg også at reproduserbarheten av resultatene ved bruk av den nye samleren var dårlig. Replikatene viste høy varians.

Den første delen av prosjektet omhandler bruken av NTNU sitt flotasjonslaboratorium for å utføre de planlagte flotasjoneksperimentene. Dette var for å potensielt redusere behovet for å reise til Brønnøy Kalk for å utføre eksperimentene. Den andre delen av prosjektet tar for seg de antatte årsakene til hvorfor den nye samleren ikke fungerer like bra som den originale. Hovedforsøkene var på partikkelstørrelsesfordelingen (PSF) av flotasjonspågangen, ettersom løftekapasiteten til den nye samleren var antatt til å være lavere

enn den originale. Reduksjonen av partikkelstørrelsesfordelingen ble antatt å øke reproduserbarheten av flotasjonsprosessen.

De viktigste funnene i prosjektet omfatter problemene ved bruk av andre laboratorier, og at redusert partikkelstørrelsesfordeling av flotasjonspågangen forbedret flotasjonsprosessen ved bruk av den nye samleren. Bruk av laboratoriet ved NTNU viste seg å være problematisk. Mange aspekter ved flotasjons prosedyren var forskjellig fra prosedyren ved Brønnøy Kalk. Spesielt flotasjonscellen og flotasjonsoperatørene er trukket fram som hovedårsaker. Studiet på partikkelstørrelsesfordelingen viste økt lysstyrke i flotasjonskonsentratet ved redusert partikkelstørrelsesfordeling av flotasjonspågangen. Forsøkene viste også redusert varians i replikatene ved redusert partikkelstørrelsesfordeling av flotasjonspågangen. Til slutt er et forslag til en ny prosedyre for flotasjon ved bruk av amin B inkludert.

Abbreviations, particle size and minerals

Abbreviations

NTNU - Norges Teknisk-Naturvitenskapelige Universitet (Norwegian University of Science and Technology).

BK - Brønnøy Kalk.

PP - Processing Plant.

g/t - gram/tonne. All units used in this thesis are SI-units.

Particle size

1 µm 1 micrometer = $\frac{1}{1.000.000}$ m. 100 µm is the average diameter of a human hair.

-250 µm the same as <250 µm.

d_{num} Example: d_{90}=250 µm. This implies that 90% of the particles have a diameter less than 250 µm.

Minerals involved in the thesis

Calcite $CaCO_3$

Dolomite $CaMg(CO_3)_2$

Graphite C

Silicates mainly Quartz (SiO_2), Plagioclase ($(Na,Ca)(Al,Si)_4O_8$) and Muscovite ($KAl_2(AlSi_3O_{10})(F,OH)_2$)

Sulphides Pyrite (FeS_2), Pyrrhotite ($Fe_{1-x}S$)

Acknowledgement

Many people have contributed to this thesis and deserve recognition for their help.

First of all, I would like to thank Brønnøy Kalk for the opportunity to work on this project, as well as the financial support of the analyses.

Vivian Molid and Anne Berit Hansen at the laboratory at Brønnøy Kalk have been a huge help with the laboratory work, AIR analyses and brightness measurements related to the project. I would like to thank them both for their help.

Torill Sørløkk, at the chemical and mineralogical laboratory at the department of Geology and Mineral Resources Engineering at NTNU, has been very helpful with the arrangement of acid measurements at NTNU.

The particle size distribution measurements of the slurries with a sedigraph could not have been performed without the help from the laboratory at the processing plant, and I would like to thank them for contribution.

I would also like to thank Erik Larsen at the Mineral processing lab at the department of Geology and Mineral Resources Engineering at NTNU for assistance with the flotation equipment and setup at NTNU.

Last but not least, I would like to thank my supervisors, professor Rolf Arne Kleiv and Ingjerd Bunkholt. Rolf Arne has been a great help guiding both the project and the report, with ideas and valuable input. Ingjerd was the organizer of the project, and helped with both experiments and the report. I would like to extend my sincere gratitude for all the help I have received throughout the project from them both. I have learned a lot.

Håkon Havskjold, Trondheim, June 2016

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1 Introduction

1.1 Brønnøy Kalk and ground calcium carbonate (GCC) slurry

Brønnøy Kalk AS is a mining company established in 1997, localized in Brønnøy, Norway. The company employs 40-50 skilled professionals, and produces approximately 2 million tonnes annually of ground calcium carbonate (GCC) raw material for use as paper filler or coating (Brønnøy Kalk AS, 2016). This is derived from a high quality marble deposit, the Akselberg deposit, situated in Brønnøy, about 250 km north of Trondheim, Norway.

No processing of the marble is performed at Brønnøy Kalk; the raw material is crushed and shipped to a processing plant where the impurities are removed using froth flotation. However, the quality of each block in the open pit is analysed for expected brightness, enabling the mine to mix the different grades of marble and continuously deliver the required grade.

The quality inspection is based on a laboratory procedure meant to represent the full scale process at the processing plant. In short, the material is crushed and milled to the necessary particle size, then subjected to reverse froth flotation. The flotation concentrates are micronized, and the micronized slurries dried and pressed into a tablet of which the brightnesses are measured. The brightness is the most significant parameter for determining the expected grade of the block.

Prior to this thesis, the flotation collector used at the processing plant was successfully switched to a new one, from amine A to amine B. The switch was of an environmentally friendly nature; the collector usage of amine B was significantly less than for amine A. This prompted the same change of collector used in the laboratory flotation procedure at Brønnøy Kalk. However, an internal study on the new collector in the laboratory flotation procedure at Brønnøy Kalk showed ambiguous results; the results did not follow the same trends as the original collector (amine A) and were difficult to reproduce. The reasons for the discrepancy of the brightness results were not determined but some possibilities were suggested, such as the flotation procedure not being tailored to amine B and the particle size distribution of the feed being too coarse.

It is essential for the production to use a procedure which produces the same results on the same feed material every time. Obtaining the same results when using amine B as when using amine A would be beneficial, however deemed not important; the cut-offs[1] can be redefined based on the new results when using amine B as long as the results are reproducible.

1.2 Aim

The company's goal is to implement the use of amine B in the batch flotation procedure of the quality inspection at Brønnøy Kalk. However, for this study the overall aim was limited to examining how different procedures and inherent settings affected the reproducibility of the quality parameters.

As part of the overall aim of increasing reproducibility of the quality inspection when using amine B, three subordinate aims are identified:

i) Validating the use of the flotation laboratory at NTNU. For practical reasons, a comparison of the standard quality inspection performed at NTNU and Brønnøy Kalk was conducted with the aim of enabling the use of the flotation laboratory at NTNU. Validating the use of external laboratories is beneficial for the mobility of studies on the subject, reducing the need to travel to Brønnøy Kalk's laboratory to perform experiments.

[1] A cut-off value constitutes a limit of a quality parameter, determining whether the analysed subject is within the required domain, such as the brightness determining whether a block is of required grade or not.

ii) Gaining information on how altering the flotation procedure when using amine B affects the brightness results.

iii) Finding the correct particle size distribution curve for the flotation feed. The correct particle size distribution in this regard would be a distribution where every particle size in the feed has a high probability of being affected by the collector.

1.3 Scope

Froth flotation is the main feature in this thesis, which is a multidisciplinary field of study. In this thesis, mainly the mechanical aspects of froth flotation are examined, such as the yield, AIR, tailing kinetics and the particle size distributions. The chemical aspects, such as collector chemistry, pH of flotation pulp, zeta-potential etc., are not considered.

The most appreciable limitation of this study was the available time. The duration of the thesis was 20 weeks, making prioritization inevitable. Several aspects of the experiments performed in this study were shortened, such as:

i) The flotation cell combination tested at NTNU was limited to just one out of many possible combinations. The study was ultimately discontinued to prioritize the other parts of the thesis.

ii) Only GSM (high quality) raw material was used as the feed material. The collector might behave differently using the other marble qualities, such as sulphide bearing marble or banded marble.

iii) Not every curve of every grade were examined in the particle size distribution experiments.

The process at the processing plant is not emphasized in this thesis. However, the use of amine B at the processing plant is successful. The process at the processing plant combines several milling steps with corresponding continuous flotation (Vik, 2011), and can not be directly compared to the batch flotation process used in the quality inspection at Brønnøy Kalk. No study on the differences between these processes was performed.

1.4 Structure of the thesis

The thesis is separated into two parts, part A and part B, with a joint background. This structure was chosen because the background applies for both parts while the methodology of each part is different. Part A addresses the first two subsidiary aims: the comparison of the flotation procedure at NTNU and Brønnøy Kalk, and the experiments using amine B in the flotation procedure at NTNU. Part B addresses the last aim: examining the effect of the particle size distribution curve of the feed material when subjected to flotation using amine B. An overall discussion and conclusion summarizes the studies.

1.5 Confidentiality

Some parts of the quality inspection process addressed in this thesis are confidential. The confidential parts will be lacking in description and detail, and the results transformed. This includes the brightness results, the micronization mills and the flotation collectors.

The flotation collectors used in this thesis are dubbed *amine A* and *amine B*, amine B being the new collector. A graphite collector was used in some of the flotations supplementing amine B, and is simply abbreviated as *GC*. The composition of the collectors will not be discussed or described further. The processing plant is addressed as the processing plant (PP), and the process at the processing plant will not be described in detail.

2 Background

2.1 Minerals in paper

The paper industry utilizes minerals as fiber filling and coating to achieve greater brightness, gloss and printing abilities (Bunkholt, 2015). Four minerals are commonly used as filler: kaolinite ($Al_2Si_2O_5(OH)_4$), rutile (TiO_2), talc ($Mg_3Si_4O_{10}(OH)_2$), and calcite ($CaCO_3$). The use of minerals also reduces the need for cellulose fibres and decreases costs. A typical sheet of copy paper contains approximately 15% mineral filler. High gloss and coated paper contain more, even above 40% (Bunkholt, 2015).

2.2 Colourimetry

Colour is an important quality of a mineral product in the paper industry, and the brightness is a major quality parameter. The human perception of colour is subjective, and the colour depends on many factors, such as illumination (the light source), reflection and observation. Several systems where these factors are standardised is in use today. The CIE L*a*b* system is the preferred colour space system in the paper industry, and the Elrepho Datacolor is the paper industry's own spectrophotometer (Bunkholt, 2015). Four parameters are measured, the Rx, Ry, Rz and brightness R457. The Rx, Ry and Rz are the reflectance expressed as weighted averages over three broad wavelength bands, of which the CIE L*a*b* values are calculated from. The brightness R457 is the brightness at a wavelength of 457 nm. The brightness R457 is shown as tappi in this thesis, and is the main colour parameter used in the studies.

A more detailed explanation of the colour measurements used at Brønnøy Kalk and the paper industry can be found in Bunkholt (2015).

2.3 Fragmentation and liberation

Fragmentation is one of the most basic mineral processing processes, and is performed primarily with two goals: comminution (particle size reduction) and mineral liberation (Kleiv, 2013). To be able to handle a large deposit, the masses need to be of a manageable size. In short, this is typically done by blasting, grinding and milling. By blasting, the rock is reduced to sizes possible to transport to a grinder. Grinding and milling is usually performed in stages, depending on the wanted product. In processes such as quarrying, the size and shape of the rock is most important. For mineral processing where the separation of minerals is necessary, the particle reduction becomes more complicated. To achieve a high separation of the minerals, some trade-offs are encountered.

First, some terminology must be defined. From Aasly (2015), the distinction between grain, particle and particle texture is defined as such: a *grain* is a particle consisting of one mineral (mono-mineral). A *particle* is an aggregate of one or more minerals (poly-mineral). *Particle texture* is how the minerals behave in relation to each other within a particle.

When fragmenting, the breakage of a particle either occurs along the grain boundaries or within the grains, depending on the strength of the adhesion between the minerals (Wills & Napier-Munn, 2006). Sedimentary minerals typically have low adhesion between the minerals, making liberation easier; a greater degree of liberation is achieved at larger particle sizes. The degree of liberation refers to the percentage of a mineral occurring as grains in the ore in relation to the total content (Wills & Napier-Munn, 2006).

To achieve a good separation of minerals, the mineral liberation is important. Typical particle liberation is shown in figure 2.1. Composite particles in either concentrate or waste is unwanted regardless of where it ends up. A greater liberation theoretically improves the separation, however the separation methods often handles smaller particles poorer, and fragmenting is energy-intensive. *Over grinding* happens when already

liberated grains is further fragmented. This increases the energy consumption and makes the separation methods less efficient without increasing the possible separation of the minerals. This leads to a trade-off between greater liberation with smaller particle sizes and greater energy consumption.

All the particles are never fully liberated, resulting in trade-off between the amounts of composite particles in the concentrate and tailing. The greater amounts of composite particles allowed in the concentrate increases the yield, and decreases the grade (more gangue minerals is included)[2]. Less amounts of composite particles decreases the yield and increases the grade.

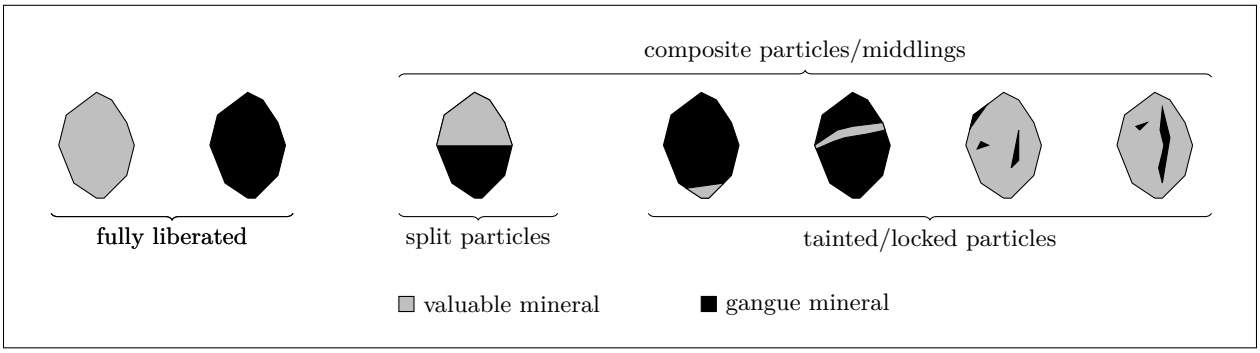


Figure 2.1: Particle liberation. *Based on illustrations from Kleiv (2013)*

When separating minerals, the necessary size reduction depends on the particle texture and the wanted separation method(s). The separation method often determines the possible range of the particle size. For flotation, the efficient particle size range is typically 3-300 µm (Sandvik, Digre, & Malvik, 1999).

2.4 Principles of flotation

Froth flotation is one of the most important and versatile mineral separation techniques, and is used to separate valuable minerals from gangue (Wills & Napier-Munn, 2006). Froth flotation utilizes the surface properties of the different minerals in the feed for selective separation. In short, it separates hydrophilic (water-loving) and hydrophobic (water-fearing) particles. The process involves solids, water and air, with complex and not fully understood interactions Wills and Napier-Munn (2006). However, the principles necessary to understand the project is included here.

To better explain the principle mechanisms of flotation, a schematic of a batch flotation procedure is included in figure 2.3. A cell is filled with mineral particles and water, creating a *pulp* which is agitated by a spinning rotor. An air flow is introduced in the bottom of the cell, and the bubbles are reduced in size and dispersed by the rotor and stator combination. The stator acts as a distributor of both the turbulence caused by the rotor, and the bubbles caused by the air flow. When hydrophobic particles collide with an air bubble, it attaches to the bubble and is carried to the top. The hydrophobic particles are trapped in a froth, which is skimmed off mechanically, or in the case of typical batch flotation, manually. The hydrophilic particles remain at the bottom of the cell.

For easier understanding of the process, the particles are thought of as fully liberated grains and are just called minerals. Most minerals are hydrophilic in nature, with a few exceptions such as graphite and talc. Therefore, creating a hydrophobic surface on the minerals for separation is necessary. A surfactant (*surface active agent*) is added to reduce the surface tension between two phases. Simplified, a surfactant

[2]The *yield* is the quantity of the total amount of a mineral present in the concentrate, while the *grade* is the concentration of the mineral in the concentrate.

is made up of a polar and a non-polar group, where the polar group is hydrophilic and the non-polar group is hydrophobic. The polar group can be positively or negatively charged (cationic or anionic), or both (amphoteric). Some surfactants are used to selectively attach on the mineral phases, and are called collectors. In this project, two amine collectors were used. The polar group (the amine) is cationic, and the collector attaches to the mineral surfaces through electrostatic adsorption (Bunkholt, 2015). Surfactants are also used to increase the lifetime of the froth, called frothers. In the case of froth flotation, transient foams with a lifetime of approximately 20 sec is obtained with (typically) short chain alcohols and carboxylic acids (Leja, 1982). The effect of collectors and frothers are shown graphically in figure 2.2. Frothers are not explained further, as no extra frother was added in the flotation experiments in this project due to the collector achieving a sufficient froth.

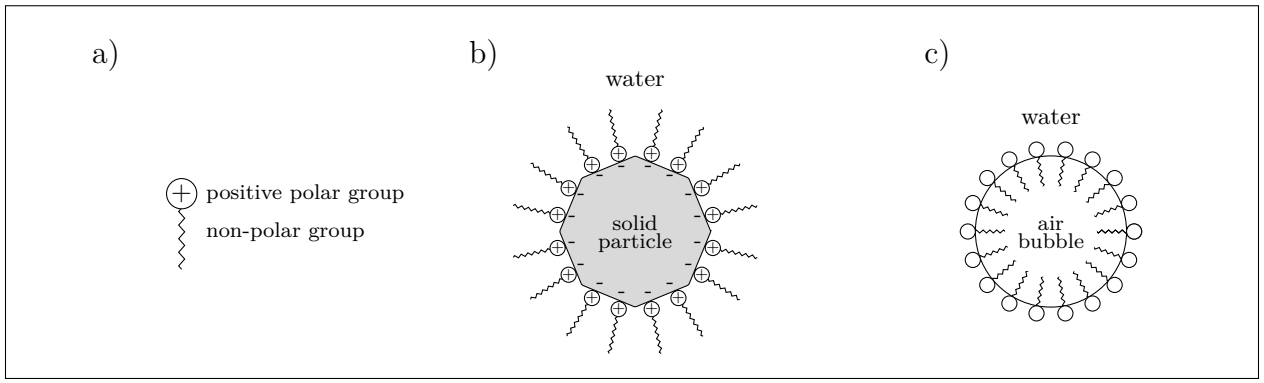


Figure 2.2: Effect of collector and frother. *Based on illustrations from Wills and Napier-Munn (2006)*
a) Cationic surfactant b) Cationic collector adsorption on particle surface c) Effect of frother.

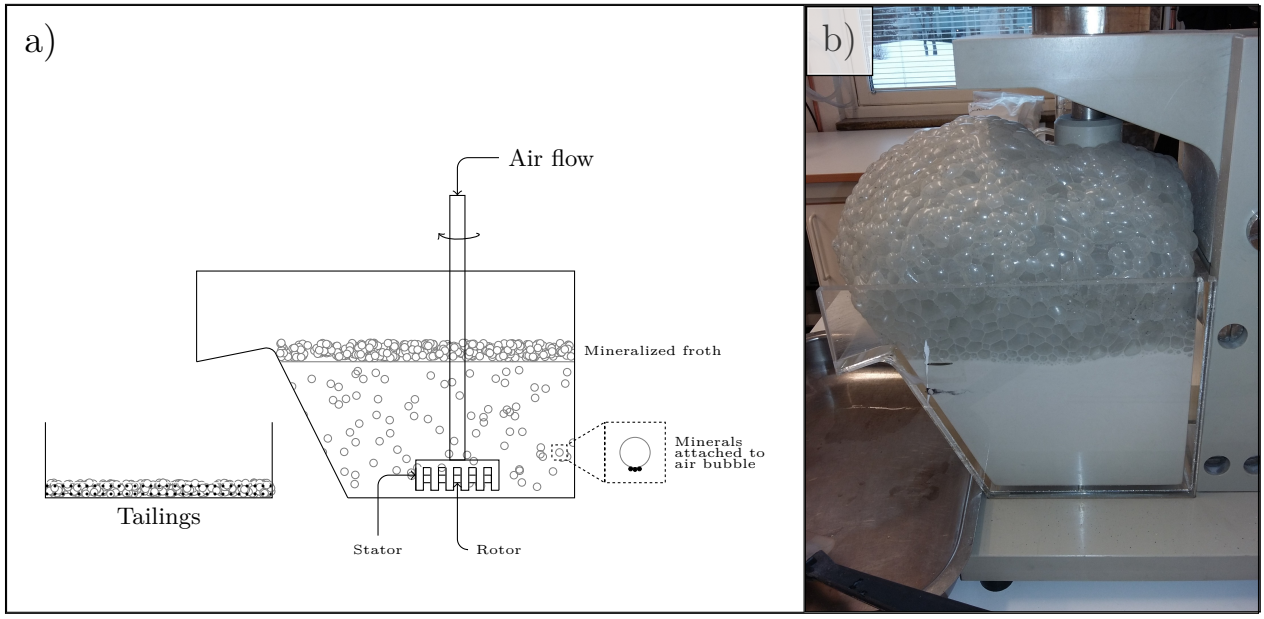


Figure 2.3: Flotation cell. *Based on illustrations from Bunkholt (2015)*
a) Schematic of the Maelgwyn flotation cell.
b) Maelgwyn flotation cell depicted showing a sample with extraordinarily stable froth.

Regulating agents such as pH regulators and electrolytes are used to further manipulate the surface properties of the minerals, controlling and improving the flotation process (Leja, 1982). However, no regulating agents were used in this project and is therefore not explained further. The flotation process occurred in the natural alkaline pH of calcite in water (pH of roughly 8-8.5).

In direct froth flotation, an environment is created where the valuable minerals attaches to air bubbles, and are trapped in a froth. In the case of marble flotation, the opposite is true; an environment where the valueless gangue minerals attaches to the air bubbles is created. This is called a *reverse froth flotation*.

Batch flotation is a single and static environment, where the tailings are removed continuously, without addition of feed. In a real, large scale continuous processes, the tailings and concentrate are removed continuously, and new feed introduced continuously. Such a process is often many large flotation cells in a row. A continuous process behaves somewhat differently from a batch flotation, however the same principles apply.

As particle size and particle size distribution is a big part of the project, the effect of the particle size in the flotation procedure is elaborated on here (although simplified). For separation to happen, a collision between a particle and an air bubble must occur, and the particle must attach to the bubble. The probability of a particle being carried to the froth can be divided into the probability of collision, P_k, the probability of attachment, P_v, and the probability of transportation in the pulp, P_{TP}. From Sandvik et al. (1999), the probability of collision increases with increasing mass of the particle and increasing size of the bubble. The probability of attachment depends on the hydrophobic properties of the particle. The probability of transportation in the pulp, depends on the dynamics of the pulp (agitation). Simplified, these three probabilities determine the total probability of a certain particle size to be floated. The smallest particles are less likely to attach to a bubble, the largest particles have a decreased probability of attachment and transportation, due to decreased surface area and increased detachment due to larger affinity to agitation in the pulp. The total probability of these three probabilities is regarded as the lifting capacity in this thesis.

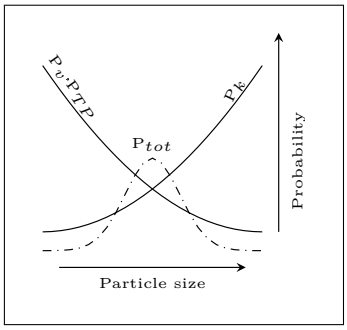


Figure 2.4: Flotation probability. *Based on illustrations from Sandvik et al. (1999)*

As the collector attaches to the surface of a particle, it follows that the degree of liberation of the grains is important. In the case of reverse flotation with such small amounts of impurities as is the case in this project, the entrainment of valuable minerals is less important. This is especially true for small scale batch flotation where the yield of the flotation process is of lesser interest.

Many other factors, such as stability of froth and drainage also plays a role in the final result of a flotation process. However, these are assumed to have lesser effect on the processes used in this thesis, and not elaborated on. In the rest of the thesis, the reverse flotation is just called the flotation.

2.5 The Akselberg deposit

Figure 2.5: The Akselberg deposit, from the northern wall looking south. *source: H.H. (August, 2015)*

Four main marble types are characterised and described by Watne (2001). In descending order of quality:

1. Graphite Speckled Marble (GSM)
2. Banded Marble (Band)
3. Sulphide Bearing Marble (SBM)
4. Impure Marble

The marble types are described in Watne (2001), and a short description is included here.

The graphite speckled marble is white to light gray, coarse grained (5-10 mm) with characteristic graphite specks of agglomorated platy graphite crystals in the 10-250 µm range. Impurities are graphite, quartz and plagioclase, with some muscovite and pyrite.

The banded marble is white to dark gray, medium grained (2-7 mm) with alternating layers of lighter and darker bands on a cm to dm scale. The impurities are concentrated in the darker bands, and are mainly graphite, pyrite, pyrrhotite, muscovite, quartz and feldspar.

The sulphide bearing marble is light to dark gray, typically very coarse grained (1-3 cm) with characteristic sulphide specks (mainly pyrrhotite).

The graphite speckled marble is regarded as the highest quality, and was used as raw material for the experiments in this thesis.

2.6 Initial study on amine B at Brønnøy Kalk

An initial study on the flotation procedure using amine B was performed at Brønnøy Kalk laboratory, 2015-2016. In this study (from here on called *the initial study*) the standard flotation procedure was used with amine B as the collector, on daily production samples (drill cuttings). The same samples were subjected to flotation using amine A as well, which were used as a reference. Different concentrations of amine B and graphite collector (GC) were experimented with.

The brightness results from the study are simplified and summarized in figure 2.6 and 2.7 below, with permission from Brønnøy Kalk. For amine B, the study showed increased brightness with increased collector concentration, as well as increased brightness when graphite collector was added. This is shown for the AB_1849_A8 sample in figure 2.6, where the collector use is displayed. However, comparing samples subjected to flotation using amine A and amine B, the samples subjected to flotation using amine B achieved lower brightness, with increasing deviation with decreasing brightness. This can be seen in figure 2.6, as the deviation from the dashed line increases with decreasing brightness.

From figure 2.7, a trend of increasing deviation in brightness results (ΔT) with increasing concentration of impurities in the sample feed is apparent. The amount of impurities is represented as acid insoluble residue (AIR%), which is the concentration of the feed not dissolved in a weak acid. The acid insoluble residue is explained in section 3.2.6. The variations occur both when comparing samples subjected to flotation using amine A and amine B, and amongst samples subjected to flotation using amine B. The study suggests that the deviations might be due to the flotation procedure not being tailored to amine B, or that the particle size distribution of the feed might be too coarse. The lifting capacity of amine B is assumed to be lower than of amine A.

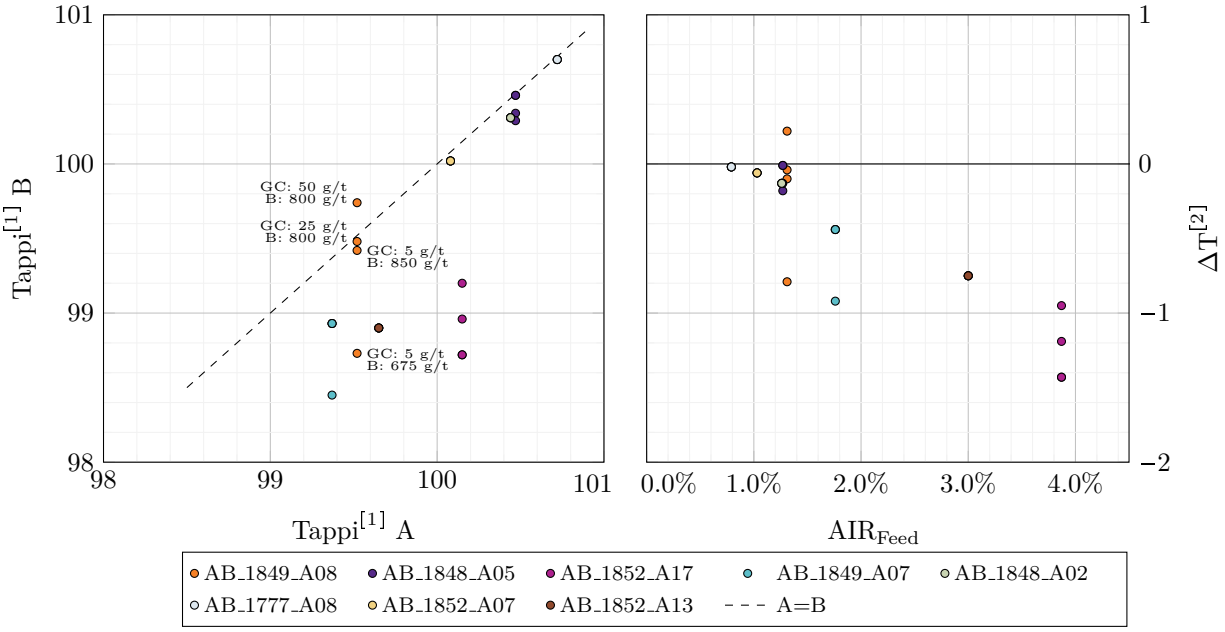


Figure 2.6: Brightness results from the initial study at Brønnøy Kalk.

Figure 2.7: Brightness results from the initial study at Brønnøy Kalk compared to the AIR content in the feed.

[1]Tappi values are transformed with a constant value due to confidentiality.

[2]ΔT = Tappi B - Tappi A, representing the deviation from the dashed line.

3 Part A - Validation of laboratory and testing of amine B at NTNU

3.1 Introduction

In this part of the project, all the flotation experiments were performed at NTNU. This involved using different equipment from what is being used at Brønnøy Kalk. A range of equipment was available at the flotation laboratory at NTNU, and a flotation cell similar to the ones used at Brønnøy Kalk was chosen.

The experiments performed at NTNU are divided into two parts:

a) Confirming the reliability and comparability of the equipment setup at NTNU.

NTNU, the Norwegian University of Science and Technology, is located in Trondheim, Norway. The Department of Geology and Mineral Resources Engineering at NTNU has a flotation laboratory available to students. To allow the usage of this laboratory in favour of travelling to Brønnøy Kalk, a comparison study was performed to assure that the results achieved at NTNU were representative of the results achieved at Brønnøy Kalk; the results achieved when a sample was subjected to flotation at NTNU had to be similar to the results achieved when the same sample was subjected to flotation at Brønnøy Kalk. The standard quality inspection procedure (using amine A) was followed at NTNU on several samples which were already processed at Brønnøy Kalk, and the brightness results achieved at NTNU were compared to the brightness results achieved at Brønnøy Kalk.

b) Experimenting with amine B.

With the presumption that the comparison study was successful, experiments on flotation with amine B were performed in parallel with the comparison study in part a. The new collector (amine B) was tested to gain experience on how it behaved, what worked and what did not, with the intent of achieving the most reproducible results. This also enabled the comparison of amine B with amine A on the same material subjected to flotation at NTNU. Several parameters of the flotation procedure were experimented with, such as conditioning time, addition of graphite collector, and ageing of amine B.

3.2 Methodology

3.2.1 Overview of the studies

In part a, the representability of the flotation setup at NTNU was examined by comparing the achieved brightness of the same samples subjected to flotation at both NTNU and Brønnøy Kalk, using amine A. The workflow is shown in the flowchart in figure 3.1.

In part b, samples were subjected to flotation using amine B at NTNU. The achieved brightness results were compared to the achieved brightness of the same samples subjected to flotation using amine A at NTNU.

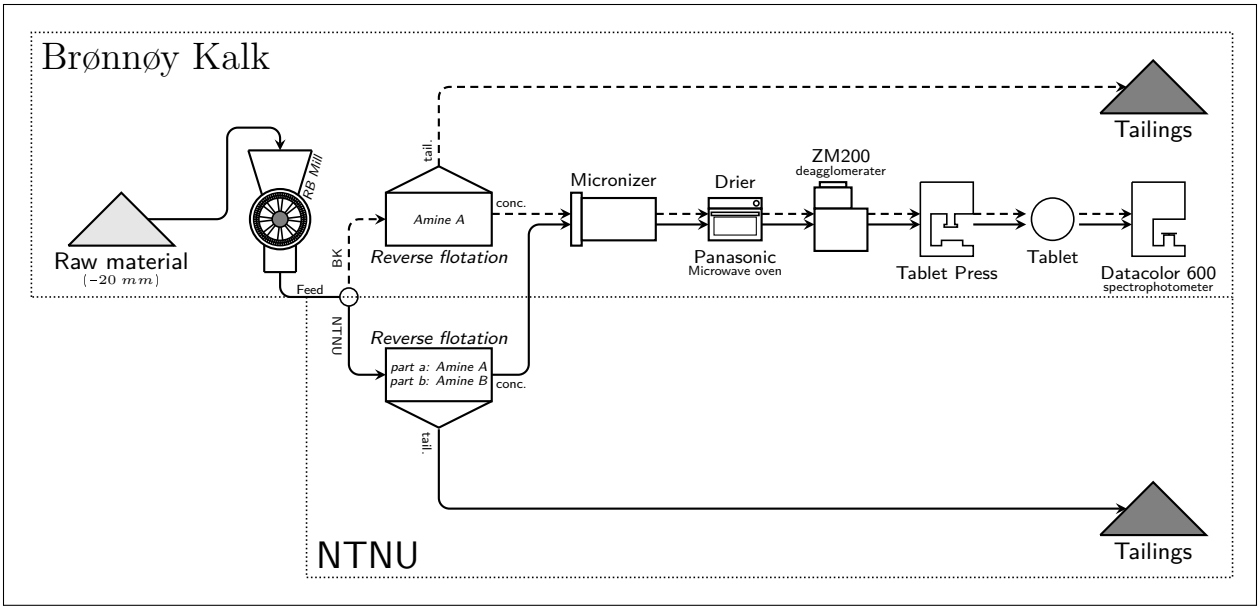


Figure 3.1: Flowchart of the comparison study (part a) and the experimentation with amine B (part b). Note that both the concentrate and tailings were dried subsequent to flotation (not shown in the flowchart), see section 3.2.4.

3.2.2 Sample description and notation

The study involved laboratory work, and several different sample types were used. This can be difficult to perceive in a report, and to increase the comprehensibility for the reader, a simplified flowchart of the quality inspection is shown in figure 3.2, with corresponding sample description below. The different samples are depicted in figure 3.3.

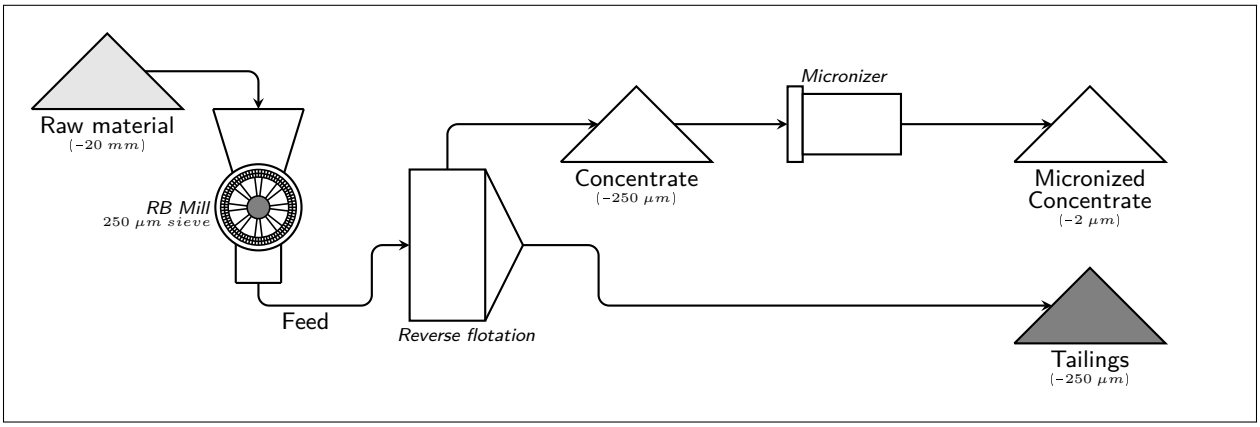


Figure 3.2: Simplified flowchart of the standard quality inspection at Brønnøy Kalk. Note that both the concentrate and tailings were dried subsequent to flotation (not shown in the flowchart), see section 3.2.4.

The *raw material* used consisted of drill cuttings and represented a block in the mine. The raw material particle size was fine grained, typically -1 mm, with some coarse particles, typically -20 mm. The raw material was dried and milled dry to the standard particle size of -250 µm, controlled by a sieve in the mill. This milled material is called the *feed* material in this thesis, depicted second from the left in figure 3.3. The feed was subjected to reverse froth flotation, where the tailings float and the concentrate sink. The concentrate was micronized together with water, creating a *slurry*. This slurry was dried and deagglomerated, producing a *dry micronized concentrate* sample, and pressed to a tablet to measure brightness.

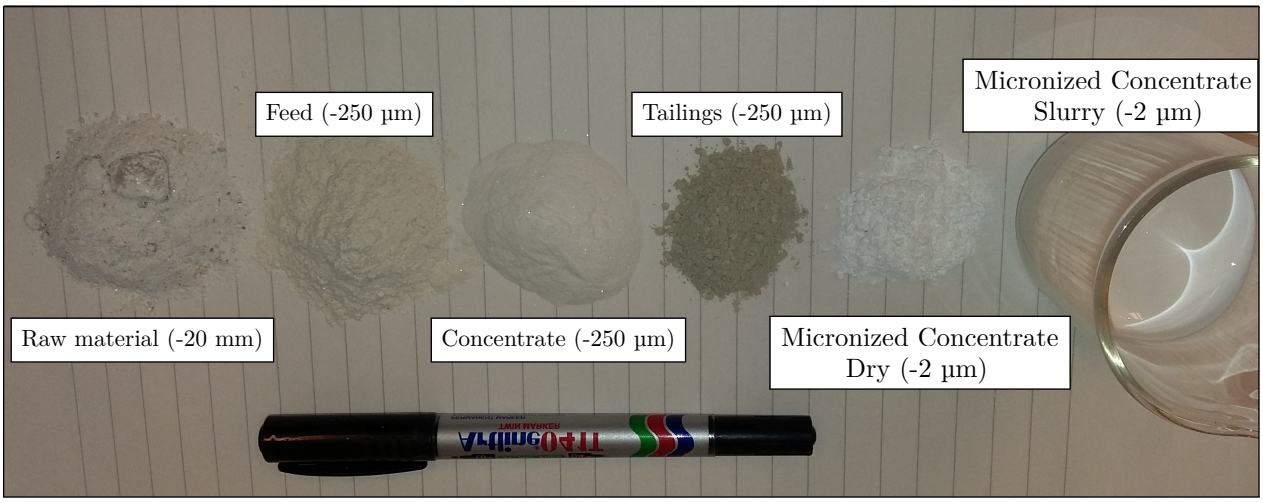


Figure 3.3: Overview of the different samples (with rough particle size) used in the study.

Collectors

Three different collectors were used in this thesis: amine A, amine B, and a graphite collector (GC), all shown in figure 3.4. Amine A is the original collector used in the flotation procedure at Brønnøy Kalk. Amine B is the collector used by the processing plant, and the one experimented with in this thesis. The graphite collector was used in some experiments as a supplement to amine B, as amine B did not contain a graphite collector. No more information on these chemicals is included due to confidentiality.

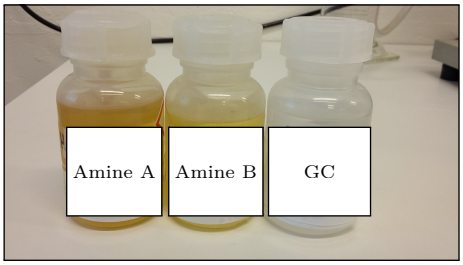


Figure 3.4: Overview of collectors used in the study.

Sample notation

Two different sets of sample notations were used in this study. The two sets differentiates the different studies as well as the different stages of a sample, from raw material to the tailings samples extracted in the kinetics analyses. In this part of the study, the samples were noted as the one below. The raw material notation used at Brønnøy Kalk was maintained and the different subnotations corresponding to the different stages of the procedure were appended.

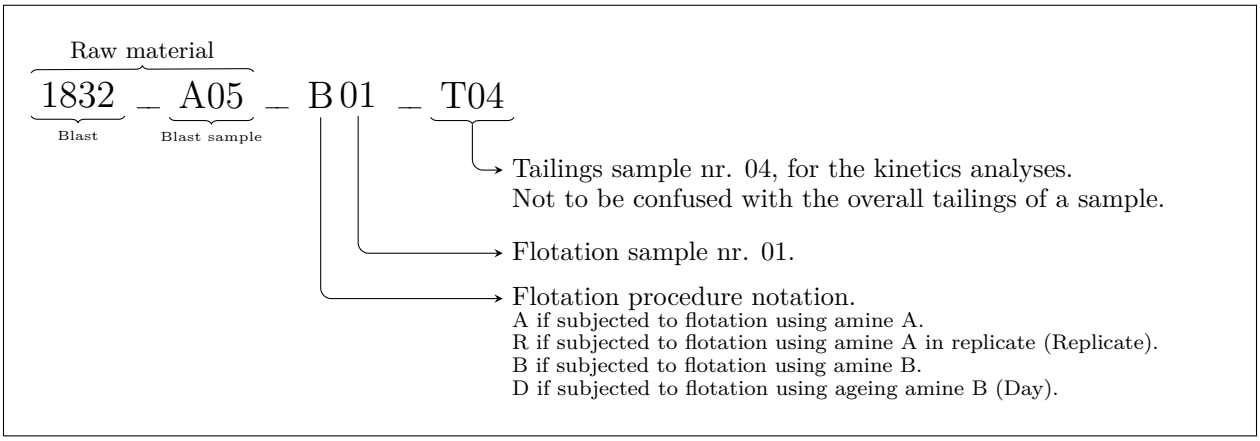


Figure 3.5: Standard sample notation for part A explained.

This notation can make it ambiguous whether the sample is of feed, concentrate or tailings. This is addressed in the text and figures by appending *feed*, *conc.* or *tail*.

To increase the distinguishability of the samples, some of them are color coded in the figures and tables where necessary. In this part of the thesis, the samples are color coded as follows:

1852_A18_A02 - Samples subjected to flotation using amine A

1852_A09_R01 - Replicate samples subjected to flotation using amine A

1852_A18_B01 - Samples subjected to flotation using amine B

1852_A18_B02 - Samples subjected to flotation using amine B with graphite collector (GC)

1852_A18_B04 - Samples subjected to flotation using amine B with conditioning

1852_A03_D01 - Samples subjected to flotation using aged amine B

3.2.3 Feed samples

The feed samples for flotation at NTNU were sent from Brønnøy Kalk. All the samples were drill cuttings collected from the mine and milled down in one step using a Retsch SR300 Rotor Beater Mill with a 250 µm screen, using the standard feed rate. Each drill cuttings sample weighed roughly 5 kg (dry), and represented a block in the mine. Some of each sample was used in the regular laboratory work at Brønnøy Kalk, and the rest sent to NTNU, making it possible to compare the results. Typically 3 kg of each sample was available for this study, restricting the comparable flotation experiments to 6 trials (0.5 kg necessary for each flotation).

3.2.4 Flotation

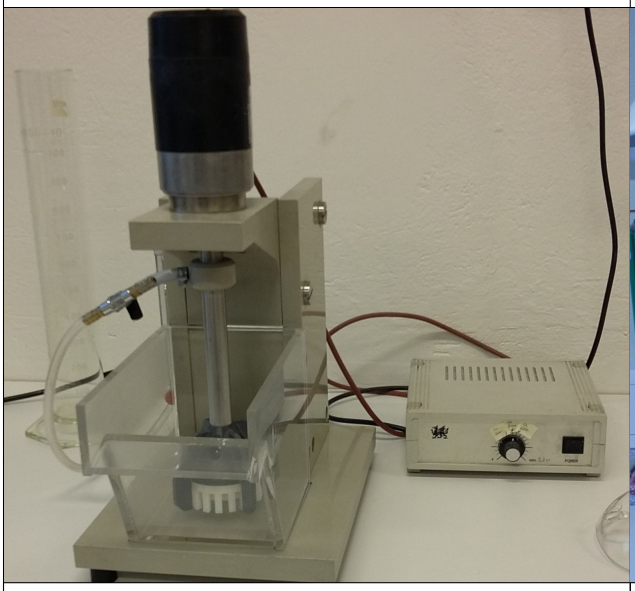
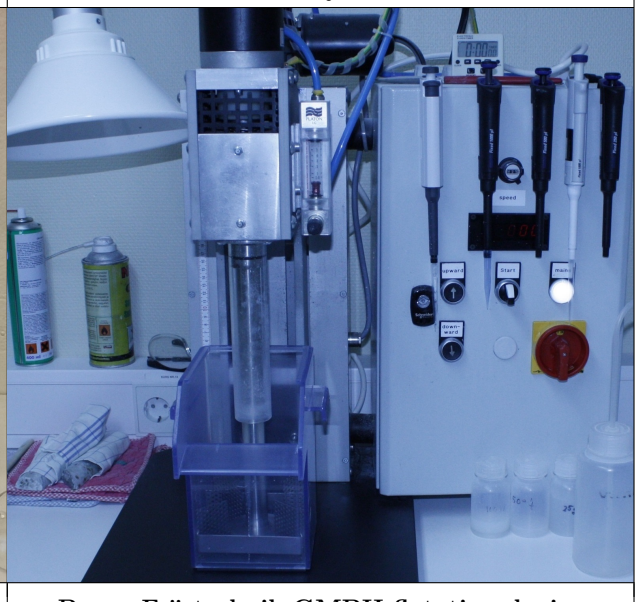
To compare the results achieved by flotation at NTNU with the results achieved by flotation at Brønnøy Kalk, the standard procedure for bench scale flotation used at Brønnøy Kalk was imitated. The standard procedure for batch flotation at Brønnøy Kalk was 500 g feed material with 1.4 L filtered tap water at roughly 28°C, in a Bauer Frästechnik GMBH flotation device. 3 L/min air flow and 1600 RPM rotor speed was used. The dimensions of the flotation cell were roughly 12.5x12x12 cm^3 (HxWxD), about 1.8 L, and the cell contained a large cubical stator. The flotation lasted for 20 min, with no additions of collector the last 2 min. A start concentration[3] of 200 g/t of amine A was used, with no additions during the first 2 min of the flotation. After the first 2 min, amine A was continually added in decreasing increments: firstly 100 g/t, then 50 g/t, and lastly 25 g/t, added when the stability of the froth was insufficient for mineral separation. The froth was skimmed off using a spatula. A flocculation agent was added when the flotation process was completed to increase the sedimentation speed and decrease the filtration time. The cell was decanted and the tailings and concentrate were de-watered by vacuum filtration. The concentrates were dried in a Panasonic microwave oven and the tailings in a Termaks drying cabinet.

The chosen flotation cell at NTNU was the one most similar to the Bauer Frästechnik GMBH flotation device used at Brønnøy Kalk, a Maelgwyn flotation device. The flotation cell was cubical with an angular front, and the dimensions were roughly 11x13x(13-18) cm^3 (HxWxD), about 2.2 L, and the cell contained a small circular stator. 500.0 g feed material was used with *1.8 L* tap water at roughly 28°C. An air flow of 3 L/min was used, the same as at Brønnøy Kalk. With the size and shape of the flotation cell used at NTNU, a rotor speed of 1600 RPM was not enough to achieve sufficient suspension of the solids. A rotor speed of approximately 2300 RPM[4] was found to be sufficient. The froth was skimmed off using a spatula. A flocculation agent was not added when the flotation was completed. The cell was decanted and the tailings and concentrate were de-watered by vacuum filtration, and both dried in a Termaks drying cabinet.

[3] Every collector concentration is the amount of collector compared to the solids.
[4] Rotor speed measured using a Elma DT-2236 tachometer

The stator used at NTNU was a small circular stator fixed outside the rotor causing one corner of the cell (the top right) to be far more turbulent than the rest. The cell was larger, resulting in a lower solids content than what was used in the standard flotation procedure at Brønnøy Kalk. The difference in the amount of water was not discovered until late in the study and is possibly a source of error. The most obvious differences in the flotation cells are summarized in table 3.1 below.

Table 3.1: A summary of the two flotation devices with emphasis on the differences between them.

NTNU	Brønnøy Kalk
	
Maelgwyn flotation device	**Bauer Frästechnik GMBH flotation device**
Dimensions (HxWxD): 11x13x(13-18) cm^3 (~2.2 L)	12.5x12x12 cm^3 (~1.8 L)
Cell shape: Cubical with angled front	Cubical with perpendicular front
Rotor speed: ~2300 RPM	1600 RPM
Air flow: 3.0 L/min	3.0 L/min
Solids in pulp: 21.7%	26.3%
Stator: Small circular	Large cubical
Flocculation agent: No	Yes

With the intention of having comparable results, every parameter were kept as equal as possible throughout the study. One of the largest uncertainties of the experiments was believed to be the manual handling during the batch flotation; the operator adds collectors and skims off the froth based on a subjective evaluation of the froth. As multiple experiments on samples were conducted, a standardisation of a dosage profile was created for each sample. The very first dosage profile of each unique raw material (and chemical) set the standard for the following experiments on the same raw material. This was logged and monitored using an application in Microsoft Excel, developed during the study. The program is explained in more detail in appendix A. The dosage profiles of the flotations performed at Brønnøy Kalk were unknown, however the total amount of collector was maintained for the samples where it was known. The log enabled adding the collector at $\pm$10 sec, creating a better basis for comparison than the addition based on subjective evaluation of the froth.

a) Confirming the reliability and comparability of the setup at NTNU

The implementation of the standard flotation procedure at NTNU explained above was used for all the flotations with amine A. A typical dosage profile for amine A is shown in table 3.2, however the dosage profile varies greatly from sample to sample, depending on the grade and marble type of the raw material.

Table 3.2: Typical dosage profile for amine A on typical grade raw material

Typical grade	Time [min:sec]:	00:00	02:00	03:30	05:30	08:30	11:30	14:30	17:00	20:00	Total
	Amine A [g/t]:	200	100	50	50	25	25	25	25	-	500

b) Experimenting with amine B

When testing amine B, the standard flotation procedure was used only as a guide as no standard procedure was developed for amine B. Several parameters were modified to see what affected the results. Start concentration, conditioning time, addition of graphite collector, and ageing of the collector were tested, amongst others. The start concentration when using amine B was increased to 300 g/t early in the study, which was a satisfying start concentration for all the samples tested. No typical dosage profile for amine B is included here, as they were all experimental.

Conditioning is well known to increase the effect of collectors. Giving the surfactant more time to adsorb on the mineral surfaces allows better exposure and increases the probability of selective separation. Conditioning of the samples was done by adding amine B and turning on the rotor while withholding the air flow. After a given duration, the air flow was turned on and the regular flotation procedure continued. Several increments of conditioning durations were planned, but due to the discontinuation of the amine B testing at NTNU, only the 5 min mark was tested for two samples.

Amine B includes no graphite collector. By the initial study at Brønnøy Kalk (section 2.6), the graphite collector was shown to improve the flotation results when added to the batch flotation. However, it was also known to destabilize the froth at too high concentrations. A balance was sought for, and the effect was examined by adding increasing amounts of graphite collector on the same samples: firstly none, then 5 g/t and 50 g/t. Higher concentrations were planned, but cut short due to the discontinuation of the testing of amine B at NTNU.

The collectors are diluted in ion exchanged water before use to decrease the concentration and make it more manageable for bench scale flotation. The deterioration of flotation properties was assumed to be significant with prolonged storage time when diluted, and freshly mixed batches of amine A and amine B were prepared for each day. The deterioration of amine B's flotation properties was believed to be worse than amine A's, and was examined by comparing the flotation results obtained when using a freshly mixed batch of amine B with an ageing batch. The same sample was not used for all the days, but the comparison of fresh and old was the same sample each day.

3.2.5 Micronization and brightness measurements

Samples of the concentrates produced by flotation at NTNU were sent to Brønnøy Kalk for micronization, as the micronization mills are specialized for Brønnøy Kalk. 100 g of the concentrate was micronized with 400 mL filtered tap water, creating a milky white slurry (depicted to the far right in figure 3.3). The micronized slurry was then dried in a microwave oven and deagglomerated using a Retsch ZM200 Centrifugal Mill. A sample of the dried fine powder was compressed to a tablet and the reflectiveness measured using a Elrepho Datacolor 600 spectrophotometer.

3.2.6 Acid Insoluble Residue (AIR)

As calcite is readily dissolved in hydrochloric acid, the amount of impurities (non-carbonate minerals) can be measured by dissolving a sample in acid and weighing the remains. The remains are called the acid insoluble residue, abbreviated *AIR*. If the acid or water is warm, the dolomite will also be dissolved (Geology.com, 2016). For feed and concentrate samples, 10 g of material were dissolved. For tailings samples, only 5 g were dissolved due to greater concentration of insoluble minerals. The samples were dissolved in 100 mL 15% HCl and, after the effervescence was completed, diluted with 400 mL warm to boiling water (about 65°C was achieved from the tap at NTNU). For samples with insufficient material, the amounts were scaled down to keep the material-acid-water ratio constant. The solution was vacuum pumped through a cellulose nitrate filter with 0.45 µm pore size, and the filter residue weighed.

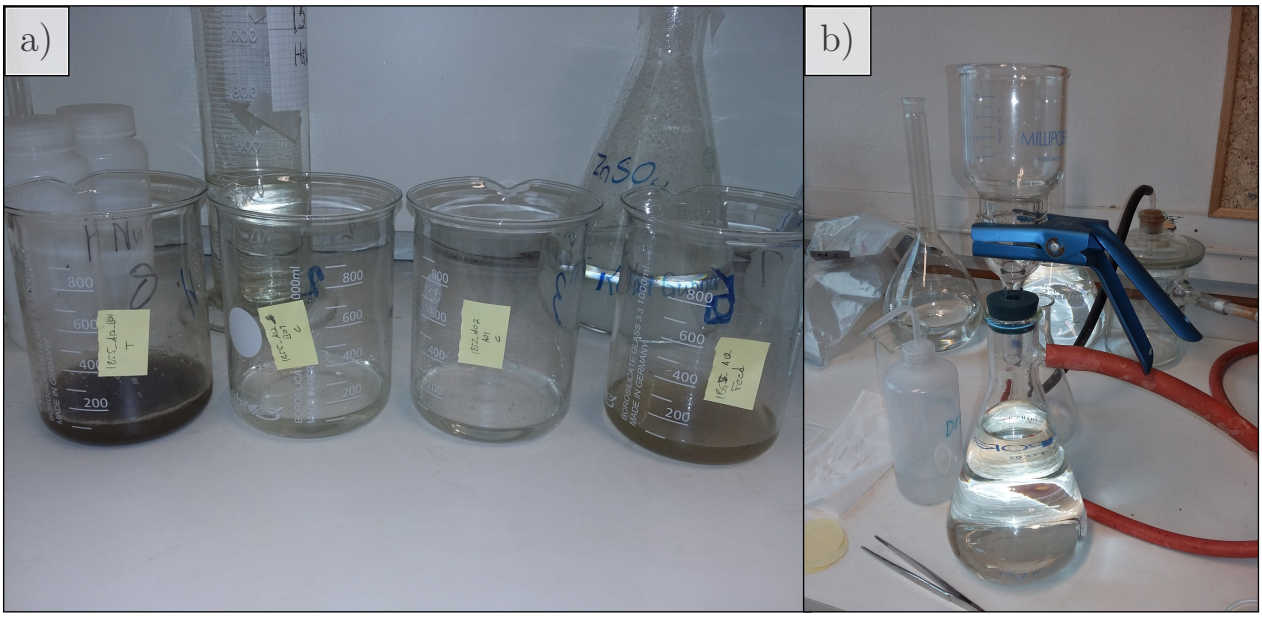


Figure 3.6: AIR setup at NTNU.
a) Samples dissolving in acid, prior to adding warm water. From left: tailings sample, two concentrate samples and lastly a feed sample. Darker color indicate higher concentrations of insoluble minerals.
b) Filter setup, vacuum pump not shown.

3.2.7 Particle Size Distribution (PSD)

The particle size distribution in a sample was measured using a Malvern Mastersizer 3000 laser diffraction particle size analyser. The samples were dispersed in deionized water, and the refractive index set to 1.33. The particle type was set to non-spherical, and measured for calcite (refractive index 1.57235, absorption index 0.1, and density set to 1 g/cm^3, with equal blue-light properties). Ultrasound was used for 60-90 sec prior to measurements, to separate agglomerated particles.

Sample preparations were believed not to be necessary. However, graphite readily floats in water, making it difficult to achieve a representative curve for tailing samples (much greater amount of graphite). Pre-wetting the samples in water was tried by vigorously mixing it with a magnetic stirrer, however it did not affect the graphite much. No surfactants for making the graphite hydrophilic were tested during the study. However, this is recommended for studies where the particle size distribution of the tailings are more important. Measuring the micronized samples proved difficult as well, as the very fine particles agglomerated easily when dispersed in water. This is shown for the micronized slurries in the appendix, table F.1. No dispersing agents were tested on the micronized samples during this study. However, this is recommended for studies where the particle size distribution for slurries are important. From experience, the slurry samples were easier to measure than the dry powder samples, as the dry powder samples were highly agglomerated prior

to measurements. When using the Mastersizer 3000, 100 µL of slurry was the perfect amount of sample to obtain the necessary obscuration in a 500 mL beaker filled with water.

Figure 3.7: a) Malvern Mastersizer 3000 laser diffraction particle size analyser with Hydro EV wet dispersion. b) Close-up on the Hydro EV stirrer (head raised), showing problems with floating graphite (tailings sample).

3.3 Results

3.3.1 Confirming the reliability and comparability of the setup at NTNU

The results from the comparison study are displayed by comparing the achieved brightness from samples subjected to flotation at Brønnøy Kalk and NTNU in figure 3.8. A point represents a sample subjected to flotation using amine A at both NTNU and Brønnøy Kalk (BK), and plotted according to the measured brightnesses. If the brightness was the same for both, the point occurs on the dashed line. Deviation from the line represents dissimilar results. A trend of increasing deviation with decreasing brightness is apparent, where the samples subjected to flotation at NTNU achieved lower brightness. The three blue diamonds, highlighted with a red square, are results from a triplicate flotation experiment, showing decent reproducibility. The complete list is included in appendix B, table B.2.

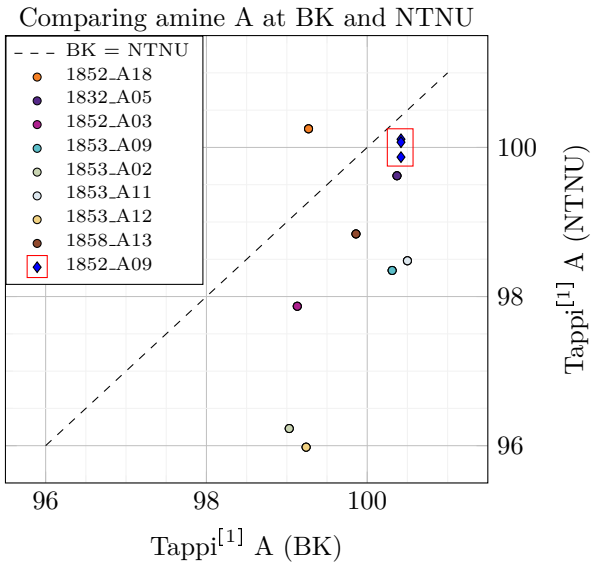


Figure 3.8: Comparison of brightness achieved from samples subjected to flotation with amine A at both NTNU and Brønnøy Kalk.

[1] Tappi values are transformed with a constant value due to confidentiality.

An acceptable deviation of 0.3 percentage points were allowed between the results at each laboratory, determined by Brønnøy Kalk. Based on the results shown in figure 3.8, the comparison study was deemed unsuccessful, as a deviation 1-3 percentage points was not even close to the accepted deviation. Other equipment setups and settings could have been tested, however as the thesis had limited time, it was determined that the rest of the experiments were to be performed at Brønnøy Kalk. The results are deliberated on in the discussion (section 3.4).

3.3.2 Experimenting with amine B

The results from the study on amine B are firstly displayed by comparing the achieved brightness of samples subjected to flotation using amine A and amine B at NTNU in figure 3.9 below. If the achieved brightness of a sample subjected to flotation using amine A and amine B was the same for both, the point occurs on the dashed line. Deviation from the line represents dissimilar results. The deviation is displayed compared to the impurities present in the feed in figure 3.10. An increased brightness deviation as the impurities in the feed increases is apparent.

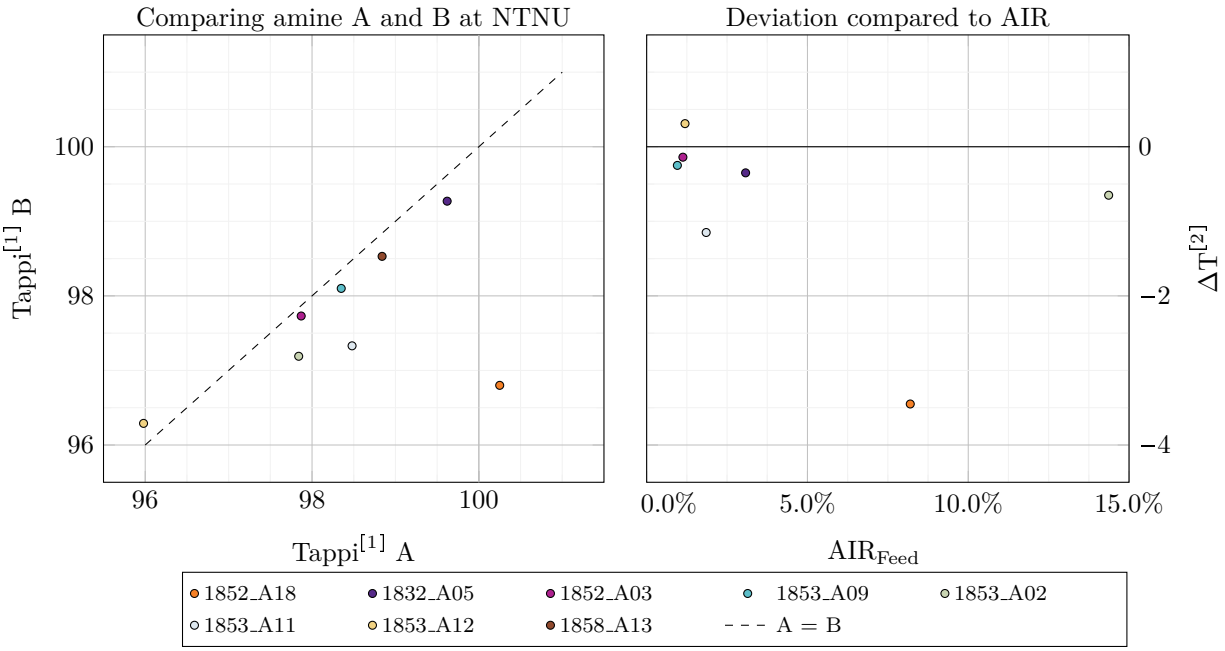


Figure 3.9: Brightness results of samples subjected to flotation using amine A and amine B at NTNU.

Figure 3.10: Brightness results of samples subjected to flotation using amine A and amine B at NTNU compared to the AIR content in the feed.

[1]Tappi values are transformed with a constant value due to confidentiality.

[2]ΔT = Tappi B - Tappi A, representing the deviation from the dashed line.

Several parameters of the batch flotation procedure when using amine B were experimented with at NTNU. The ageing of amine B, conditioning time and addition of graphite collector are emphasized here.

Only a few samples were subjected to flotation with conditioning of amine B. The conditioning was achieved by adding the start concentration and letting the rotor run, withholding the air flow. For the experiments performed, the conditioning elapsed 5 min, which is significant compared to the 20 min flotation time. Results are summarized in figure 3.11 below. An increase in the brightness due to conditioning was found for both samples.

The effect of graphite collector (GC) was examined by adding it together with the start concentration of collector in the flotation experiments. The results are summarized in figure 3.12 below. An increase of brightness with increasing concentration of graphite collector is apparent.

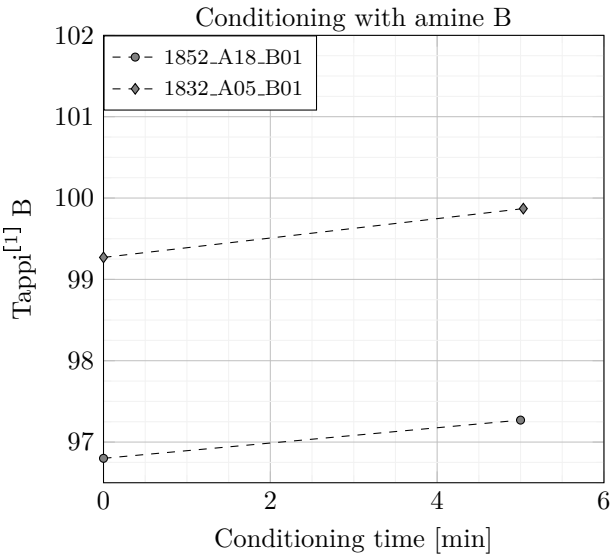


Figure 3.11: Effects of conditioning with amine B at NTNU. A higher brightness result is obtained by conditioning the pulp with amine B.

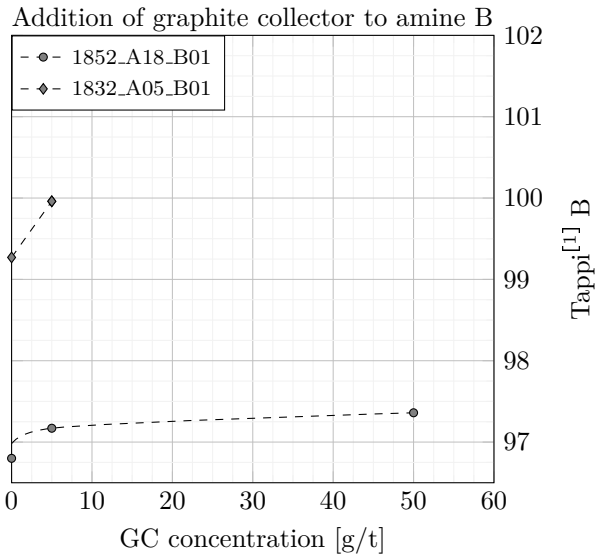


Figure 3.12: Effects of adding graphite collector on amine B. Increasing graphite collector in small dosages improves the flotation results.

[1]Tappi values are transformed with a constant value due to confidentiality.

For amine B, the flotation properties were believed to rapidly deteriorate over storage time. The deterioration was assumed to start when the concentrated collector was diluted in water (the collectors are diluted in deionized water to make it more manageable when adding to a batch scale flotation process). The effect on flotation properties from the ageing of diluted amine B was tested by comparing the brightness results of subjecting a sample to flotation using a fresh amine B batch and an ageing batch of amine B. The summarized results are shown in figure 3.13 below. The y-axis is shown as relative brightness, as the same material was not used over the week, and the results show how the old amine B batch compares to a fresh one. No distinct deterioration of the flotation properties of amine B was observed during the 4 days; the ageing batch gave a lower brightness than the fresh only on one of the days (day 2).

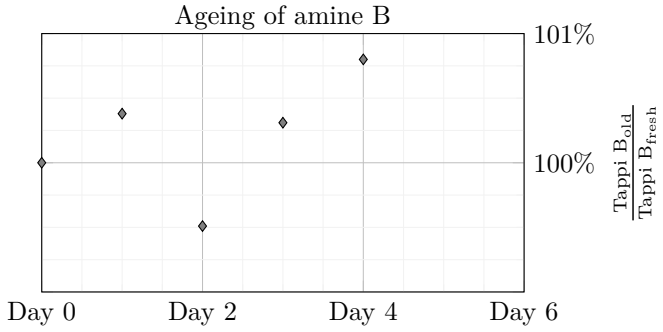


Figure 3.13: Effects on brightness results with increased ageing of amine B. 100% represents the same brightness for the old amine B compared to the freshly mixed.

3.3.3 Particle Size Distribution (PSD)

The particle size distribution (PSD) was assumed to affect the flotation when using amine B more than when using amine A, as the lifting capacity was assumed to be less for amine B than amine A. A lower lifting capacity would increase the variations and decrease the brightness results when using amine B, as more of the coarse gangue particles present in the feed would be less likely to be lifted and separated from the concentrate. The particle size distribution of several samples were examined, comparing the sample feeds with the concentrates produced when subjected to flotation using amine A and amine B. This is summarized in figure 3.14 below. The figure shows the PSD for a selection of samples, ordered by decreasing deviation in brightness (ΔT).

No mineral liberation study was performed on any of the materials, and no PSD analysis was performed on the impurities (and measuring the PSD of the tailings proved difficult). The distribution of impurities in the feed is unknown. However, considering the gangue minerals are harder than the calcite, the impurities are assumed to be coarser. In any case, as the feed sample was assumed homogeneous, both collectors should in theory produce the same PSD for each concentrate.

As figure 3.14 shows, a larger difference in PSD between the concentrates produced from a sample subjected to flotation using amine A and amine B corresponds with a larger difference in the measured brightness. The most equal sample, ΔT = -0.14, shows very equal PSDs for feed and both concentrates. However, caution should be taken when reading into the PSD results. The amount of fines included in the concentrates were variable, as no flocculation agent was used and the sedimentation time before decantation of the flotation cell was variable.

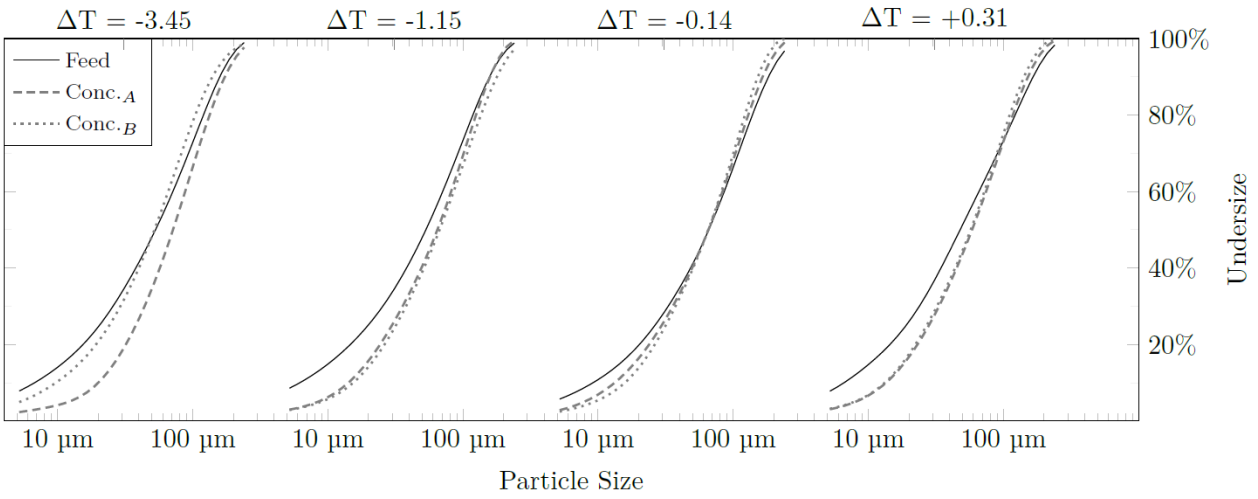


Figure 3.14: Particle size distribution of samples with increasing deviation in Tappi.
ΔT = Tappi B - Tappi A. The samples shown are (from left to right): 1852_A18, 1853_A11, 1853_A03, 1853_A12.

Table 3.3: PSD data for the selected samples, based on ΔT. (d_{num} defined in section [ref])

Sample	AIR_{Feed}	$AIR_{Conc.}$	Conc. wt%	Tappi	ΔT[1]	d_{90}	d_{80}	d_{70}	d_{60}	d_{50}	d_{40}	d_{30}	d_{20}	d_{10}
1852_A18_feed	**8.20 %**	-	-	**-**	-	**158.9**	**120.2**	**93.1**	**71.4**	**53.1**	**37.9**	**25.7**	**15.4**	**6.8**
1852_A18_A02	”	NM[2]	82.7 %	100.25	**-3.45**	173.4	133.6	107.7	87.9	71.5	57.1	44.0	32.0	20.0
1852_A18_B01	”	NM[2]	78.9 %	96.80		140.0	104.6	82.5	65.9	52.0	40.0	29.2	19.3	9.7
1853_A11_feed	**1.85 %**	**-**	**-**	**-**	-	**157.6**	**118.6**	**92.0**	**70.9**	**53.2**	**38.3**	**25.5**	**14.7**	**6.2**
1853_A11_A01	”	0.03 %	89.4 %	98.48	**-1.15**	158.7	124.9	101.3	81.9	64.7	49.3	35.8	24.4	14.2
1853_A11_B01	”	0.05 %	88.7 %	97.33		178.7	136.1	107.5	85.4	67.2	51.5	38.0	26.3	15.4
1852_A03_feed	**1.13 %**	**-**	**-**	**-**	-	**182.8**	**138.9**	**109.3**	**85.9**	**66.0**	**48.4**	**33.1**	**20.2**	**9.2**
1852_A03_A01	”	0.04 %	91.7 %	97.87	**-0.14**	165.3	129.4	104.1	83.5	65.6	49.7	35.8	23.9	13.3
1852_A03_B01	”	0.06 %	94.2 %	97.73		152.8	122.3	100.4	82.1	65.5	50.6	37.5	26.3	15.8
1853_A12_feed	**1.19 %**	**-**	**-**	**-**	-	**166.0**	**122.3**	**90.5**	**66.0**	**47.9**	**34.5**	**23.9**	**14.5**	**6.6**
1853_A12_A01	”	0.07 %	88.8 %	95.98	**+0.31**	152.5	117.5	93.5	74.5	58.7	45.1	33.4	23.1	13.5
1853_A12_B01	”	0.05 %	89.0 %	96.29		142.2	111.2	89.8	72.4	57.3	44.0	32.3	22.3	13.3

[1]ΔT = Tappi B - Tappi A.
[2]NM = Not Measured.

3.4 Discussion

3.4.1 Confirming the reliability and comparability of the setup at NTNU (part a)

The purpose of comparing the brightness results achieved at NTNU with the brightness results achieved at Brønnøy Kalk was to examine the accuracy of the flotation procedure at NTNU; a validation that the flotation procedure used at NTNU were representative of the flotation procedure at Brønnøy Kalk. The results achieved at Brønnøy Kalk were treated as the true results and were attempted to be replicated at NTNU. Validating the accuracy and precision of performing flotation trials at NTNU would enable the future use of this laboratory, reducing the need to travel to Brønnøy Kalk to perform experiments. The distinction between accuracy and precision is shown in figure 3.15. However, as the results from this study shows (figure 3.8), the accuracy of the flotation procedure at NTNU was low, and precision was high. The high deviation from the dashed line represents the low accuracy and the low variance in the triplicate trial represents the high precision.

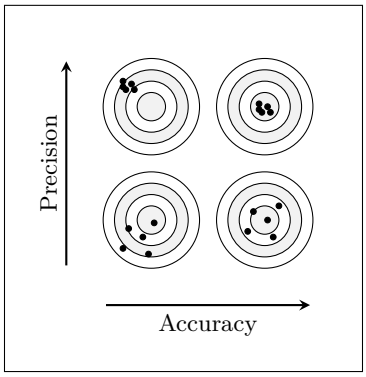


Figure 3.15: Accuracy and precision.

It is worth noting that the precision was only examined as one triplicate study, and was incidentally on one of the samples with lower deviation from the brightness results at Brønnøy Kalk. A lower precision may be found in a sample showing higher deviation. More triplicate trials were desired, but was deprioritized in favour of the other experiments.

Comparing the results achieved from flotation at NTNU and Brønnøy Kalk, the deviation was large. The four samples achieving a brightness of 99.0-99.3 by flotation at Brønnøy span the range of 96.0-100.6 by flotation at NTNU. This was clearly an unacceptable deviation when considering the 0.3 percentage point limit set by Brønnøy Kalk. The results achieved at NTNU could not be trusted, and the flotation setup was deemed not representative of the flotation process at Brønnøy Kalk. Further experimenting with the flotation setup at NTNU was abandoned due to limited time. Flotation is a complex process with many aspects interacting with each other. Some of the aspects of the flotation process assumed to have the largest effect on the results is discussed here.

Every step of the quality inspection procedure, except the flotation procedure at NTNU, was performed by the laboratory at Brønnøy Kalk. This is shown by the flowchart in figure 3.1. The steps performed at Brønnøy Kalk was not considered a large contributor of the variations in the brightness results, as they were performed by the same operators with the same equipment as the samples subjected to flotation at Brønnøy Kalk. The high precision shown by low variance in the triplicate trials, suggests that the low accuracy was probably not caused by variations inherent in the procedure used at NTNU or the external processes at Brønnøy Kalk. The difference in procedure and different flotation operators are more likely to be the cause of the low accuracy. The different equipment is discussed first, followed by the operators.

In this study, the samples were subjected to flotation at NTNU using a different flotation cell and a slightly altered flotation procedure compared to the same samples subjected to flotation at Brønnøy Kalk. Most notable difference in the setup was the flotation cell and the lack of flocculation agent. The flotation cell used at NTNU was larger, causing a lower pulp density, increased froth area, and altered the air flow to cell volume ratio; the air flow was kept equal to the standard flotation procedure. The pulp density could easily have been changed to match the one used at Brønnøy Kalk, however the different cell volume was not discovered until late in the study. The rotor speed used at NTNU was increased compared to the standard procedure at Brønnøy Kalk due to insufficient particle suspension in the larger cell. Only a small stator was present in the flotation cell at NTNU, and no flocculation agent was used to increase the sedimentation speed. The small stator probably caused less bubble dispersion, and the lack of flocculation agent caused less of the fine particles to be included in the concentrates compared to the concentrates at Brønnøy Kalk; the finest particles were still in suspension when the cell was decanted. The lack of the finest particles possibly increased the

overall PSD which may have altered the measured brightness purely due to different particle size distribution. However, as the study in part B showed (figure 4.12), the final PSD was not affected much by finer feed PSD, suggesting that less finer particles in the concentrates probably have little effect on the final PSD as well.

The flotation operator on the samples subjected to flotation at NTNU was the author. The flotation operators on the samples subjected to flotation at Brønnøy Kalk were the experienced staff at Brønnøy Kalk. As the flotation procedure involved manual handling, such as addition of collector and skimming of the froth, the different operators probably evaluated the froth differently. The subjective evaluation of the froth determined the dosage profile, which the operators at Brønnøy Kalk are more experienced at. A large difference in the amount of collector used, as well as the addition time probably contributed to the high deviation.

One key difference independent of any setup was the storage duration of the milled raw material. The samples used in this study were subjected to flotation and analysed at Brønnøy Kalk shortly after milling, and was stored for a longer duration (days to weeks) in plastic bags before being subjected to flotation at NTNU. Some days after being subjected to flotation at NTNU, the concentrates were sent to Brønnøy Kalk for analyses. The effect of this storage duration is unknown, but as samples were milled, the mineral surface area is large, and possibly interacts with air. This possibly alters the flotation properties. Especially the sulphides are prone to alterations due to exposure (Bunkholt, 2015). However, these alterations happen quickly, and the effect might not be any difference over the long storage time, given that the samples were not subjected to flotation immediately after milling at Brønnøy Kalk.

Lastly, the water quality and chemistry used is also something to consider. The water used in the flotation procedure at Brønnøy Kalk was filtered tap water, and the water used at NTNU was unfiltered tap water. From Havskjold (2015), it is known that the flotation using amine A is poor when using deionized water, and that different water qualities affect the flotation process. An analysis of the water chemistry used at NTNU was not performed during the study, however, a difference in the content of the water is expected, and it most likely had an impact on the flotation process.

3.4.2 Experimenting with amine B (Part b)

Under the presumption that the validation of the flotation setup at NTNU would be successful, experiments with amine B were performed at NTNU in parallel with the validation study. The validation study proved unsuccessful, and as a result, the experimenting with amine B at NTNU was discontinued. The decision to discontinue this study in favour of other studies was taken with the supervisors on the project. Regardless of the shortened study, some experiences with amine B were gained. As the validation study proved unsuccessful, the results from the experiments with amine B could not be compared to the results from Brønnøy Kalk. However, the single triplicate trial in the validation study showed decent reproducibility, so the trends are considered credible. The results are most likely not correct, but the trends suggests what to expect from further experimenting.

The study showed an improved flotation process when the samples were subjected to flotation using amine B with conditioning. This was based on only two samples but is still considered very probable. The study also showed improved flotation when adding graphite collector (GC) at the start of the flotation procedure when using amine B. The improvement is possibly increased further by increasing the added amount of GC, however this assumption is based on just one trial. No clear deterioration was found using an ageing batch of amine B in the flotation procedure, possibly allowing for longer use of each mixed batch. The deterioration was not examined for longer than one work week, however this interval is the most interesting one when considering the diluted batch from a practical standpoint.

The initial study at Brønnøy Kalk showed a trend of increasing deviation of the brightness measurements with increased amounts of impurities in the feed. The samples with high AIR content achieved significantly

higher brightness when subjected to flotation using amine A compared to when subjected to flotation using amine B. The same trend was apparent from the flotation trials at NTNU. This suggests problems with the probability of lifting the largest particles when using amine B. The lower probability leads to high variations in the flotation trials of the same samples due to some of the large gangue particles being floated in one trial, and not in another. This variation is expected to decrease with finer feed particle size distributions, as the grains are more liberated and smaller in size.

3.4.3 Particle size distribution (PSD)

The measuring of the particle size distribution of some of the samples in the project proved difficult. Due to floating graphite in the water dispersion of the samples when using the Mastersizer 3000, the particle size distribution analyses performed did not account for the entire sample. The amount of impurities in the concentrates was small, typically less than 0.1%, and the measurements were assumed representative of the sample. The feed contained a larger amount of impurities, typically 1-2%, and the results may have been skewed due to lack of impurities in the measured samples. The tailings had too much impurities in the samples, typically more than 20%, and the measurements were deemed not representative of the sample.

No flocculation agent was added after the flotation experiments at NTNU. This most likely prevented some of the fines from ending up in the concentrate, as they were suspended in the water when the cell was decanted. This probably resulted in coarser PSDs for the concentrates than it should. The sedimentation time was variable during the flotation experiments, resulting in different amount of fines included in the concentrates. This makes it difficult to compare the PSDs of the concentrates to the feeds.

No mineral liberation study or particle size distribution of the impurities was done. The particle association and distribution of the impurities were unknown. From the PSD measurements, it was apparent that both the finest and coarsest particles were under-represented in the concentrates compared to the feeds. From the (inaccurate) PSD measurements of the tailings over time, the coarse particles are removed first, followed by the fines. This was the case for both collectors, and is only mentioned here, as the tailings measurements were deemed not representative of the true PSD of the tailings.

Without knowing the mineral liberation and the particle size distribution of the impurities in the feed, discussing the concentrate PSD compared to feed will only be speculations. However, the concentrate PSD produced when subjected to flotation using amine A and amine B can be compared, as the mineral liberation and particle size distribution of the feed was the same. Figure 3.14 shows a trend of increasing deviation in brightness results with increasing differences in the particle size distribution of the two concentrates produced by flotation using amine A and amine B. As different amounts of fines is present in every concentrate due to different sedimentation time and not due to the flotation process, this comparison should not be viewed as correct. However, the most equal brightness result was achieved when the PSD of both concentrates as well as the feed were approximately equal. This suggests that the feed size might not be the optimal for amine B, and opens the possibility of a study on the optimal feed PSD when using amine B.

3.5 Conclusion

The validation study performed on the flotation setup at NTNU proved unsuccessful. The study showed low accuracy when comparing brightness results achieved from samples subjected to flotation at NTNU with the same samples subjected to flotation at Brønnøy Kalk. However, given the high precision when comparing the samples subjected to flotation at NTNU, it is the authors belief that a representative equipment setup can be obtained at NTNU and other laboratories. To allow for the usage of the flotation laboratory at NTNU, more experiments must be performed to fine-tune the setup in such a way that the same results are achieved at NTNU and Brønnøy Kalk.

Four sources of the variation are emphasized in this study: the storage of raw material, the flotation equipment, the flotation operators and the water chemistry. To achieve a representative flotation setup at NTNU, these variations have to be addressed. A study on the effect of prolonged storage of the milled feed material would establish whether it is possible to use another laboratory in the first place, or if the material would need to be milled on site. On addressing the flotation equipment at NTNU, it is suggested to try different flotation cells, both machine and the cell shape. A setup more similar to the setup at Brønnøy Kalk is beneficial, especially towards the pulp density and bubble dispersion, altered by the feed-water ratio and different stators, respectively. A cell shape with the same surface area as Brønnøy Kalk's cells, ~144 cm^2, is also preferred. A digitalization of the dosage profile used on each sample would be beneficial in reducing the variations due to different operators, reducing the variance by different evaluations of the froth. The water chemistry should be compared, and the effect of the different water qualities can be tested by testing them with the same flotation equipment and procedure.

Even though the validation study proved that the flotation setup at NTNU was non-representative of the flotation setup at Brønnøy Kalk, the precision of the triplicate trial when using amine A suggests that the trends found by the experiments with amine B were somewhat credible. From the flotation experiments using amine B it was shown that conditioning of the pulp as well as addition of graphite collector both improved the flotation of impurities in the flotation process. The study also suggest that the storage of diluted amine B is less detrimental to the flotation of impurities than initially believed. It is the authors belief that conditioning and addition of graphite collector should be considered when a new flotation procedure is developed for amine B.

Using the Mastersizer 3000 to analyse the particle size distributions of the samples in this study was both successful and unsuccessful. The material needed was very small (typically less than 1 g), the analysis was quick, and the results were easily available. However, due to the floating graphite, the samples containing high amounts of impurities were problematic. The micronized samples were also problematic due to agglomeration, however, as the Mastersizer is far more efficient than methods such as sieving, it is suggested to test surfactants to make the graphite hydrophilic, and dispersing agents to decrease the agglomeration of micronized samples.

3.6 Suggestions for future work

Further testing of the flotation equipment at NTNU is suggested, with the aim of obtaining a setup representative of the setup at Brønnøy Kalk. The differences emphasized in this study should be considered. The flotation cell shape should be as equal to the Brønnøy Kalk cell as possible, and the other aspects of the flotation such as solids content, the stator and use of flocculation agent, should also be considered. The rotor speed and air flow should be adjusted to the cell volume. A tracking of the dosage profile used at Brønnøy Kalk is also suggested, as this would create a better basis for comparison of the achieved brightness results. As part of such a study, the water quality used should also be compared. A triplicate trial using water from Brønnøy Kalk should be compared to a triplicate trial using water from NTNU, on the same flotation device.

The experimentation with amine B in the flotation procedure was shortened, and further experimentation is suggested for further work. Both adding graphite collector and conditioning showed promising results. Experiments on the concentration of GC and on the duration of the conditioning should be performed, with emphasis on the variations of the brightness measurements. To address this, a replicate study is suggested, examining whether the variations reduces with increased GC concentration and/or conditioning duration.

A mineral liberation study could be a step in diagnosing the cause of the alterations in the flotation experiments when using amine A and amine B. A liberation analysis would reveal whether the graphite and other gangue minerals are liberated or not. A well liberated feed is necessary for a good separation result. The diagnosis could by improved further by comparing mineral liberation of concentrates produced

by a feed subjected to flotation using amine A and amine B. This could possibly reveal problems with the non-liberated minerals when using amine B. A mineral liberation study performed using a scanning electron microscope (SEM) with particle texture analysis (PTA) at NTNU could produce the particle size distribution of the impurities as well as the calcite.

A more extensive PSD study than performed here is suggested. Given comparable concentrates and known particle size distribution of the impurities in the feed, the problematic sizes and/or particle types can be revealed. Comparable concentrates can be achieved with the use of flocculation agent at the end of the flotation procedure, assuring the inclusion of the fines. The particle size distribution of the impurities can be achieved by SEM with PTA.

As the flotation using amine B at the Processing Plant is known to work, the particle size distribution used in the process at the Processing Plant could be examined. An emphasis on the final particle size distribution used at the Processing Plant is suggested.

4 Part B - Variance in micronization and flotation using amine B at Brønnøy Kalk

4.1 Introduction

The initial study performed by Brønnøy Kalk and the results from part b in Part A both suggests the particle size distribution of the feed to be problematic for flotation using amine B. This was examined, with the aim of finding a more correct particle size distribution. As the validation study in part A proved flotation experiments at NTNU unsuccessful, the rest of the experiments were determined to be performed at Brønnøy Kalk. This enabled the use of the same equipment as the laboratory uses in the daily production, effectively removing any variance caused by different equipment.

The experiments with amine B at NTNU in part A showed the same divergent behaviour as was found in the initial study at Brønnøy Kalk; samples subjected to flotation using amine B showed lower brightness than the same samples subjected to flotation using amine A, with increasing deviation as the amount of impurities increase in the feed. The cause was assumed to be the lifting capacity of amine B, that amine B might not be capable of reliably lifting the coarsest particles in the feed. A reduced probability of particles with gangue minerals being lifted increases variation between each flotation. In other words, a variable amount of gangue minerals ends up in the concentrates, independent of feed and procedure. It was shown by Bunkholt (2015) that even very low concentrations (<0.01 wt%) of impurities display a significant decreasing effect on brightness. It is therefore likely that even small differences in how the coarser particles behave in replicate experiments will influence the brightness results. Therefore, the company wanted to know more about how the particle size distribution (PSD) of the flotation feed affected the process and if the variance decreases with finer PSD curves.

The subsequent experiments were divided in two, part I and part II, based on where the variances were assumed to be the largest: I) the micronization process and II) the flotation with different PSD curves of the feed.

The micronization process used at Brønnøy Kalk was examined because four separate batch micronizers were in use, in two different models (model α and β). It is known that the particle size has an effect on the brightness measurement, and if some of the mills creates a different micronized PSD, it could produce variance in the brightness measurements independent of the flotation process. Establishing the inherent variance in the micronization process was important for the reliability of the results in part II.

Part II addresses the effect of finer particle size distributions of samples subjected to flotation using amine B.

4.2 Methodology

4.2.1 Overview of the studies

The brightness was measured on the same material micronized in each mill, comparing both the inherent variance of each mill, as well as comparing the mills. A flowchart of the micronization experiments is shown in figure 4.1 below.

The effect of finer particle size distributions was examined because this was assumed to be the cause of variations in the brightness results, and was examined by floating samples milled finer than the standard particle size distribution. This was examined for three grade groups of the marble, dubbed standard (STD), medium (MID), and sub-standard (SUB), of which one to four different PSD curves were examined. A flowchart of the PSD experiments is shown in figure 4.2 below.

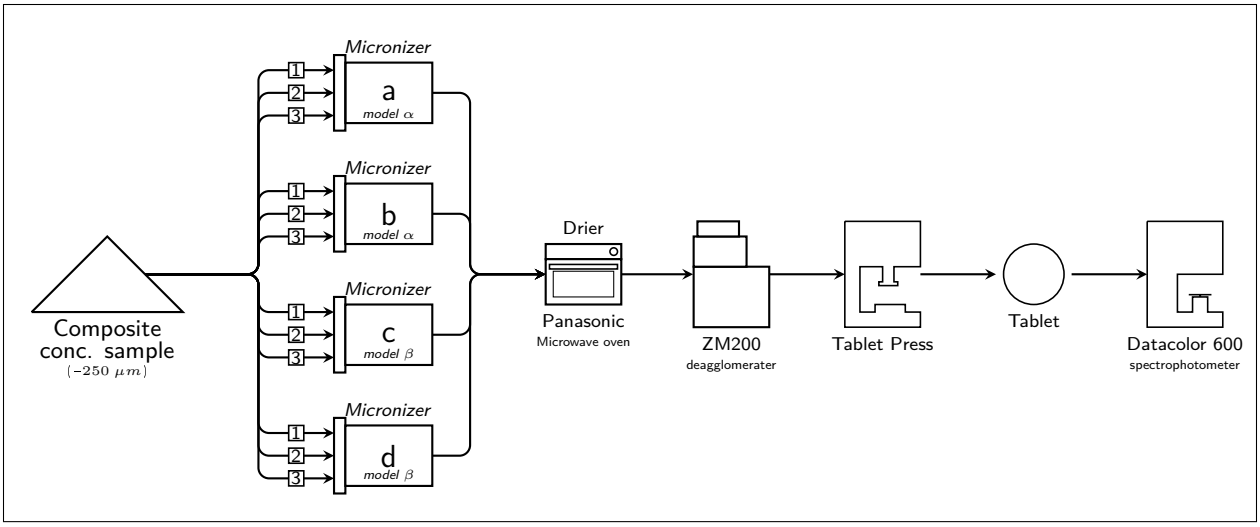


Figure 4.1: Flowchart of micronization setup (part I).

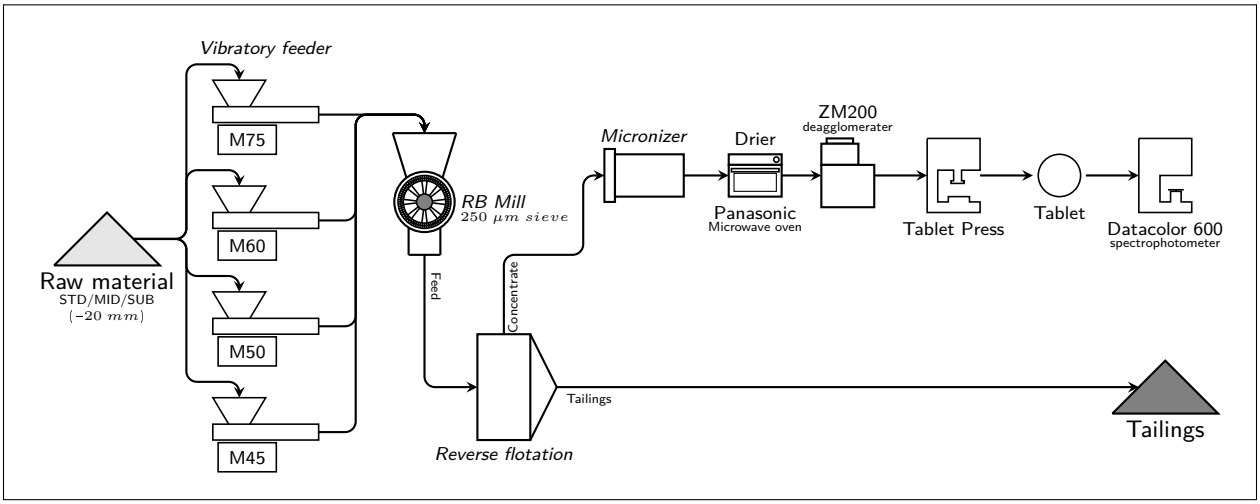


Figure 4.2: Flowchart of particle size distribution setup (part II).
The mill feed using different vibratory feeder intensities (feed rates) were fed separately into the mill, creating different flotation feed PSD. Note that both the concentrate and tailings were dried subsequent to flotation (not shown in the flowchart), see section 4.2.4.

4.2.2 Sample description and notation

The thesis deals with laboratory work, and several different sample types are used. Descriptions of the sample types and the collectors used are included in section 3.2.2, as they are the same for this part of the study. However, the notations of the samples are different. In part I, the notation is simple, and corresponds to the mill and trial number. In part II, the raw materials are composite samples of different grade groups, dubbed STD, MID and SUB, with a variable feed rate. The notation is shown in figure 4.3.

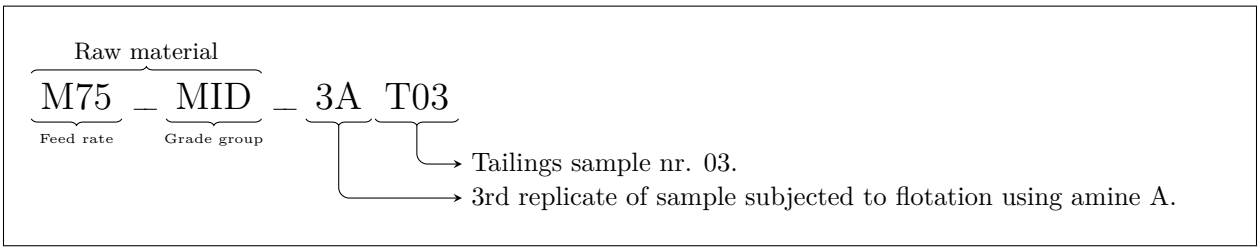


Figure 4.3: Sample notation for part II in part B explained.

This notation can make it ambiguous whether the sample is of feed, concentrate or tailings. This is addressed in the text and figures by appending *feed*, *conc.* or *tail*.

To increase the distinguishability of the samples, some of them are color coded in the figures and tables where necessary. In this part of the thesis, the samples are color coded as follows: **M75A**, **M75B**, **M60B**, **M50B** and **M45B**, where M75 corresponds to the feed rate, and A and B corresponds to the collector. Note that **M75A** and **M75B** are alternative notations for standard feed rate samples subjected to flotation using amine A and amine B, respectively, hence the same color coding as for the initial notation are used.

4.2.3 Sampling and milling

In part I, a composite sample of concentrates, roughly 2 kg, was created. Seven concentrate samples following regular trends[5] for the standard grade were gathered and mixed thoroughly in a barrel. No milling or flotation was necessary, as the samples were already processed concentrates.

In part II, the raw materials were composite samples created from samples of drill cuttings collected from the daily process at Brønnøy Kalk. Three grade groups were selected as follows:

1) **High grade**, dubbed standard grade (STD). Samples showing tappi[6] values in the vicinity of 100 were selected.
2) **Medium grade** (MID). Samples showing tappi[6] values in the vicinity of 99 were selected.
3) **Sub-standard grade** (SUB). Samples showing tappi[6] values in the vicinity of 98 were selected.

Roughly 13 kg of each grade group were collected and mixed thoroughly using a sample riffler.

Figure 4.4: Feeder and mill.

To create different particle size distributions, the throughput of the mill was varied. The throughput was determined by a vibratory feeder, and was altered by varying the intensity of the vibrations. The intensity, called *amplitude* on the feeder, operated on a continuous spectrum going from 0 to 100, and was changed by turning a knob. The different intensities used in this thesis are dubbed *feed rates* and corresponds to the used intensity.

The mill was unaltered throughout the study; the particle size distributions were altered purely by altering the speed of the feed. The feed speed was reduced compared to the standard feed speed to increase the energy used on each particle, reducing the overall particle size. The feed rate was controlled by a Retsch DR 100 vibratory feeder, feeding a Retsch SR300 Rotor Beater Mill. The Rotor Beater mill controlled the top size of the particles with a circular 250 µm sieve. The vibratory

[5] Reagent consumption, brightness, tailings weight and AIR were determining factors
[6] Tappi values are transformed with a constant value due to confidentiality.

feeder and the mill is depicted in figure 4.4. Four rates were chosen: M75 (standard rate), M60, M50 and M45, where the numbers corresponds to the intensity ("amplitude") used on the vibratory feeder. The composite sample of each grade group was divided using a sample riffler, and milled using the different feed rates. M45 was the slowest practical speed, and M75 the fastest speed not choking the mill. Each feed rate was assumed to create a different PSD, where M45 was the finest.

From experience, the mill is known to overheat when run for too long or when milling too much material in one run. When the mill was overheated, the particles were discoloured and it smelt burnt. In this study, only 3 kg were milled at a time, which is generally an unproblematic amount. However, for the slowest feed rates (M50 and M45), the mill had to be stopped during the milling due to very long milling durations. To not impair the samples in this study, the continuous run time was conservatively experimented with, and a maximum run time of 5 min was assumed to be the longest for the slowest feed rates. The cooling period for the mill was at least 5 min between the runs, often more than 20 min if the rotor was hot to the touch. The downtime is referred to as "stops", and represents the inefficiency of milling at this rate.

4.2.4 Flotation

In part I, no flotation was necessary as the composite sample was created from concentrate samples available at the laboratory.

In part II, a Bauer Frästechnik GMBH flotation device was used, with all the standard settings as the standard flotation procedure at Brønnøy Kalk. 500 g feed material with 1.4 L filtered tap water at roughly 28°C, 3 L/min air flow and 1600 RPM rotor speed was used. The cell also included a large cubical stator. The flotation procedure lasted for 20 min, with no addition of collector the last 2 min. A start concentration of 200 g/t were used in the flotation procedure when using amine A and 300 g/t when using amine B, with no additional collector added during the first 2 min of the procedure. For the samples subjected to flotation using amine B, graphite collector was added shortly after the start concentration of amine B. A concentration of 100 g/t GC was used based on the observations gained in part A, of improved flotation results with increased GC concentration. 100 g/t was considered a high concentration, but not so high as to destabilize the froth. However, a runny froth was experienced at this concentration. After the first 2 min, collector was continually added in decreasing increments: firstly 100 g/t, then 50 g/t, and lastly 25 g/t, added when the stability of the froth was insufficient for mineral separation. The froth was skimmed off using a spatula when necessary. After the flotation procedure was completed, 20 mL of flocculation agent was added before the cell was decanted. The concentrate and tailings were de-watered by vacuum filtering. The concentrate was then dried in a Panasonic microwave oven, and the tailings in a Temrak drying cabinet.

A reference was created for each grade group by subjecting the standard feed PSD, M75, to flotation using amine A. This also worked as a confirmation of the homogeneity of the feed. Each feed rate of each grade group was then subjected to flotation in quadruplicate using amine B. Not all the feed rates were tested for all the grade groups due to insufficient time.

In the process of the flotation experiments with different PSD curves, the Flotation Log was important. The study involved both quadruplicate flotation experiments of each PSD curve as well as different PSD curves from the same grade group. To achieve comparable results, it was imperative to assure the equality of each flotation within each grade group. The Flotation Log[7] enabled the usage of a standard dosage profile for each grade group. A standard dosage profile was constructed based on the first flotation procedure of each grade group, of both amine A and amine B, dosed based on the subjective evaluation of the froth. The standard dosage profile was followed for every sample subjected to flotation within the same collector and grade group, enabling a more accurate basis for comparison. The dosage profile for each grade group is listed in tables 4.1 and 4.2 below. Using the Flotation Log to keep track enabled an accuracy of $\pm$10 sec for each addition.

[7] The Flotation Log is explained in appendix A.

As table 4.2 shows, the total amount of amine B used for all three grade groups were similar. This was coincidental and only based on the subjective evaluation of the froth in each initial flotation procedure for each grade group.

Table 4.1: Dosage profile for amine A on the three grade groups

STD	Time [min:sec]:	00:00	02:09	03:38	05:35	06:52	11:15	15:44	20:00			Total
	Amine A [g/t]:	200	100	100	50	50	50	25	-			575
MID	Time [min:sec]:	00:00	02:10	03:52	05:22	10:47	13:21	16:07	20:03			Total
	Amine A [g/t]:	200	100	100	50	25	25	25	-			525
SUB	Time [min:sec]:	00:00	02:37	04:23	05:48	07:57	10:43	13:20	15:03	17:47	20:00	Total
	Amine A [g/t]:	200	100	50	50	50	25	25	25	25	-	550

Table 4.2: Dosage profile for amine B on the three grade groups

STD	Time [min:sec]:	00:00	00:32	02:22	04:52	06:44	08:49	11:16	13:02	14:06	15:57	17:28	20:01	Total
	Amine B [g/t]:	300		100	50	50	50	25	25	25	25	25	-	675
	GC [g/t]:		100											100
MID	Time [min:sec]:	00:00	00:30	02:40	05:14	07:18	09:24	11:23	14:06	15:19	17:20	20:00		Total
	Amine B [g/t]:	300		100	50	50	50	50	25	25	25	-		675
	GC [g/t]:		100											100
SUB	Time [min:sec]:	00:00	00:29	02:32	04:50	07:31	09:32	12:34	14:10	15:16	16:53	17:56	17:18	20:00	Total
	Amine B [g/t]:	300		100	50	50	50	25	25	25	25	25	25	-	700
	GC [g/t]:		100												100

4.2.5 Flotation kinetics

The flotation kinetics were analysed on the last of the quadruplicate trials of each PSD curve of each grade group, by weighing the tailings obtained at sequential stages of the flotation. This was done by separating the tailings in four time intervals, roughly 00:00-02:30, 02:30-05:00, 05:00-10:00 and 10:00-20:00. The practical separation was done just before the addition of more collector, when the froth was at a minimum. This also allowed the tailings to be more accurately portrayed by the collector use. The interval timing was logged precisely in the Flotation Log.

4.2.6 Micronization and brightness measurements

Four micronization mills were used at Brønnøy Kalk, in two different models. Mills a and b was of model α, c and d of model β. The speed of each mill was measured by the operators at Brønnøy Kalk prior to the experiments in this part of the thesis, and was deemed to be the correct speed for all four mills.

In part I, the composite sample was divided into 12 samples, three samples for each micronization mill. The standard procedure for micronization at Brønnøy Kalk was followed: 100 g of concentrate was micronized with 400 mL filtered tap water. The milling procedure is not discussed further, due to confidentiality.

The temperatures of the slurries were measured, as previous micronizations had shown noticeable differences in slurry temperature, but never quantitatively measured. It was assumed that differences in temperature could indicate differences in PSD when micronized, due to mechanical differences in the mills. The temperature of each slurry sample was measured on the surface (after stirring) of the slurry shortly after the micronization finished, using a IP54 infrared thermometer. The temperature of the filtered tap water

held roughly 13°C at the time of the study, however some of the water samples used in the micronization process were most likely warmer than this, as some samples were subjected to room temperature (roughly 22°C) for longer periods than others. This is a possible source of error. The concentrate samples used in the micronization process all held room temperature.

In part II, each concentrate from the flotation experiments were micronized following the standard procedure for micronization at Brønnøy Kalk, the same as in part I. After micronization, the slurries were dried using a Panasonic microwave oven, then deagglomerated using a Retsch ZM200 Centrifugal Mill. The micronized dry concentrates were pressed to a tablet and the brightness measured using a Elrepho Datacolor 600 spectrophotometer.

4.2.7 AIR in the coarser fractions

Given the assumption of reduced lifting capacity of amine B for coarser particles, a high variance in the brightness measurements and an increase of impurities in the coarser fractions of the concentrates was expected. To examine this, three concentrate samples were collected from one of the PSD curves from one of the grade groups showing a high variance in brightness measurements of the samples subjected to flotation using amine B. The three concentrate samples were

1) a **reference**. The concentrate showing the median brightness result from the sample subjected to triplicate flotation using amine A was chosen.
2) a **best case**. The concentrate showing the highest brightness result from the sample subjected to quadruplicate flotation using amine B was chosen.
3) a **worst case**. The concentrate showing the lowest brightness result from the sample subjected to quadruplicate flotation using amine B was chosen.

As the samples in part II were analysed at this point, a sample matching the requirements was found: **M75_MID**. Five fractions of each of the three concentrate samples were separated using a sieve tower for 15 min. The fractions were determined by the available sieve-sizes (Tyler mesh), and were: 0-53 µm, 53-104 µm, 104-147 µm, 147-208 µm, and 208+ µm. The amount of impurities in each fraction was determined by the amount of AIR, measured following the standard AIR procedure explained in section 3.2.6.

In addition to the AIR measurements of the M75_MID sample, AIR measurements of the fractions of every PSD curve (M45, M50, M60 and M75) in the MID grade group were performed. The concentrate showing the lowest brightness of the samples subjected to flotation using amine B for each PSD curve was chosen. The lowest brightness samples were chosen to represent the worst case for every PSD curve. This was done to examine whether the concentration of impurities in the coarse fractions declined with finer PSD curves. As the samples with finer curves contained less coarse material and the finest fractions were less interesting, only three fractions were selected: 0-104 µm, 104-147 µm, 147+ µm.

4.2.8 Particle size distribution (PSD) measurements

The particle size distribution of several samples were measured using a Malvern Mastersizer 3000 laser diffraction particle size analyser. The setup was the same as for the measurements in part A, section 3.2.7.

The particle size distribution of the feed and the concentrate produced when subjecting the feed to flotation using amine B were measured for the four feed rates. This was performed on the MID grade group, as this was the only completed series. Of the quadruplicate flotation experiments, the last trial (4B) was chosen, as the flotation yield over time were also measured on these samples. The intent of the PSD measurements was to examine the difference of the flotation concentrate compared to the feed, for the decreasing PSD of the feed.

One of the concerns when reducing the particle size distribution of the flotation feed was how it affected the particle size distribution of the micronized concentrate. Given the same concentrate sample, a coarser particle size distribution is known to give higher brightness measurements, simply due to the particle size. To examine if the finer PSD curves of the feed had an effect on the final PSD of the micronized concentrate, the final PSD of the micronized concentrate was measured on each of the feed curves used in this study (M45, M50, M60, and M75). The samples were measured using the Malvern Mastersizer 3000 at NTNU, as well as a sedigraph at the processing plant. The sedigraph samples were run with a dispersant agent, reducing the agglomeration. No dispersing agent was used in the samples analysed with the Mastersizer.

4.2.9 Deviation of flotation, micronization and measurements

The quality inspection used in this study involved many procedures. Every part of the inspection was assumed to contribute to the final variance in the brightness measurements of the concentrates. The variances in the procedure was broken down to the procedures where it was possible to measure the variance. Assuming the feed is homogeneous, the measured variance can be written as

$$\sigma_T^2 = \sigma_{flotation}^2 + \sigma_{micronization}^2 + \sigma_{tablet}^2 + \sigma_{measurement}^2 \tag{4.1}$$

where σ_T is the standard deviation of the brightness measurements of samples, $\sigma_{flotation}$ is the standard deviation in the flotation process, $\sigma_{micronization}$ the standard deviation in the micronization process, σ_{tablet} the standard deviation in the tablet making process, and $\sigma_{measurement}$ the standard deviation in the brightness measurement of the tablet.

In part I, a 2.3 kg composite sample of concentrates was created. The composite sample allowed for several micronization samples of the same concentrate, and assuming the composite sample was homogeneous, $\sigma_{flotation} = 0$. Equation 4.1 reduces to

$$\sigma_T^2 = \sigma_{micronization}^2 + \sigma_{tablet}^2 + \sigma_{measurement}^2 \tag{4.2}$$

This can be reduced further by measuring several tablets made from the same micronized dry concentrate, leaving $\sigma_{micronization} = 0$. Equation 4.2 reduces to

$$\sigma_T^2 = \sigma_{tablet}^2 + \sigma_{measurement}^2 \tag{4.3}$$

By measuring the same tablet several times, leaving $\sigma_{tablet} = 0$, reduces equation 4.3 to

$$\sigma_T^2 = \sigma_{measurement}^2 \tag{4.4}$$

Note that σ_T is unique for every equation, it is the calculated standard deviation of the sample size in each unique case.

4.3 Results

4.3.1 Variance in micronization process

A total of 12 concentrate samples were micronized using four micronization mills, three samples in each. The four mills are denoted as a, b, c and d. The values of the composite sample were calculated based on a linear relationship between the colour data and weight proportion of the seven chosen concentrates, and represents the expected value of every sample. The results are listed in table 4.3 below, with a graphical representation to the right. Mill b and c shows results close to the expected results, and were chosen for the subsequent experiments in part II.

Table 4.3: Tappi- and temperature values for each mill

Sample	**Temperature [°C]**	**Tappi**[1]	**avg. Tappi**[1] $\pm\sigma$
a_1	52.5	**99.83**	
a_2	50.3	**99.77**	99.83 ±0.06
a_3	51.8	**99.88**	
b_1	46.3	**99.74**	
b_2	53.4	**99.76**	99.74 ±0.02
b_3	52.5	**99.73**	
c_1	49.4	**99.74**	
c_2	51.2	**99.76**	99.70 ±0.08
c_3	51.1	**99.61**	
d_1	50.4	**99.93**	
d_2	50.8	**100.00**	99.95 ±0.04
d_3	48.6	**99.92**	
Comp. sample	**-**	**99.72**	99.72

[1]Tappi values are transformed with a constant value due to confidentiality.

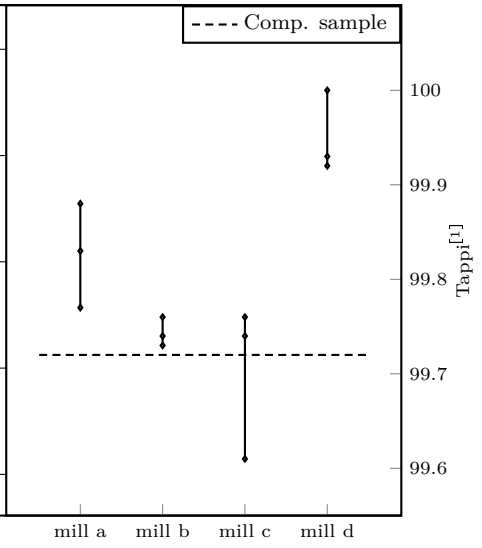


Table 4.4: Mill speeds for the four mills

Mill	Model α		Model β	
	a	b	c	d
Mill speed[2]	99.5%	100.0%	100.0%	99.8%

[2]Mill speeds are normalized such that the speeds of mills b and c represents 100% of their respective model, as the results from these mills were closer to the expected result. The exact measurements are not included, due to confidentiality.

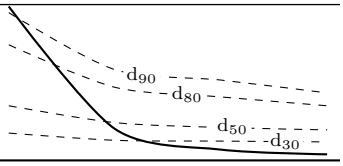
Based on the results from the micronization testing in part I, only mills b and c were used for subsequent micronizations.

4.3.2 Mill throughput and particle size distribution

In order to create finer particle size distributions of the flotation feed, the throughput of the mill was reduced by reducing the intensity of the vibratory feeder. The achieved throughput for each feed rate is listed and shown graphically in table 4.5. The data can be found in appendix C, tables C.1 and C.2. The particle size distribution obtained at these feed rates are shown in figure 4.5.

Table 4.5: Rough throughput of the mill (SR300 with a 250 µm screen) on the different feed rates[3]

Feed rates	Throughput [kg/min]	# Stops[1] per 3 kg	Cool down per 3 kg	PSD d_{90}	d_{80}	d_{50}	d_{30}	M75 M60 M50 M45
M75	~8.0[2]	0	0 min	180	140	64	30	
M60	~1.5	0	0 min	109	87	43	21	
M50	~0.4	1	~10 min	96	75	37	20	
M45	~0.15	4	~60 min	80	64	34	19	



[1] Due to overheating, the mill was stopped and cooled during the milling. The cooling period was roughly 10 min, longer for consecutive milling of samples.
[2] The throughput for M75 was not measured directly, but calculated from M80 and M70 measurements (appendix C, table C.1).
[3] Measurements listed in appendix C, tables C.1 and C.2.

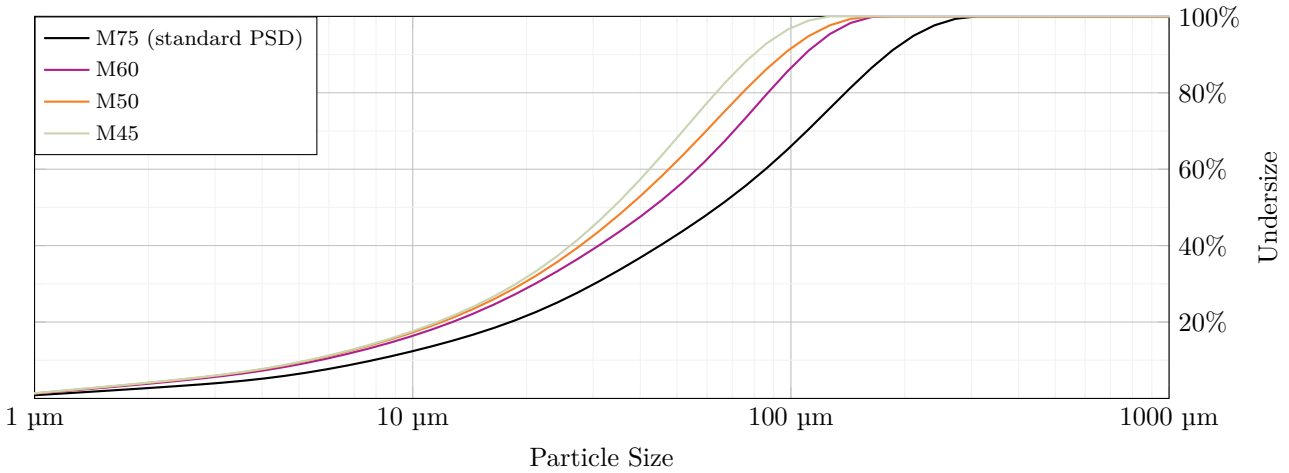


Figure 4.5: Obtained particle size distribution of the chosen feed speeds (Retsch SR300 Rotor Beater Mill with a 250 µm screen with a Retsch DR 100 vibratory feeder).

4.3.3 AIR in the coarser fractions

Three samples were chosen from the same raw material (M75_MID), and floated using amine A and amine B. M75_MID_1B was the concentrate showing the highest brightness of the quadruplicate flotation of M75_MID using amine B, and M75_MID_3B was the concentrate showing the lowest brightness. The difference between the two measured samples was almost 1 percentage point, a very large difference considering they were from the same feed and subjected to the same flotation procedure. Note that from the M75_MID samples subjected to flotation using amine B, the concentrate achieving the highest brightness (M75_MID_1B) shows as high brightness as the concentrates produced from flotation using amine A (here represented by M75_MID_1A). The results from the AIR measurements on the fractions in each sample is shown in figure 4.6. A higher concentration of impurities were found in the coarser fractions of the sample showing worst the lowest brightness, and the sample showing the highest brightness, as high as the reference, shows the same low concentration in all fractions. Note the decreasing weight distribution of the increasing fractions. The concentration is higher for the coarser fractions, but most of the impurities are in the finer fractions. The results from the AIR measurements on the lowest brightness samples from each PSD curve in the MID grade group is shown in figure 4.7. This also shows increased concentration of impurities in the coarser fractions, which is not decreasing for the finer PSD samples. Note the low weight amount in the coarsest fractions of the finest curvers (M45 and M50).

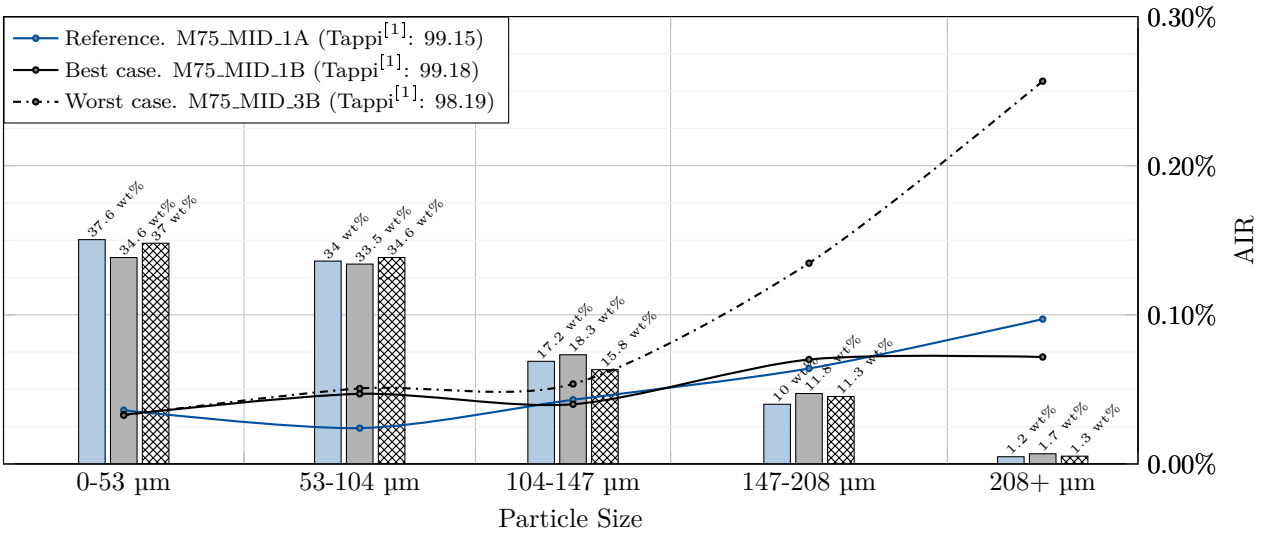


Figure 4.6: Acid insoluble residue (AIR) concentration in the fractions of the concentrates of M75_MID subjected to flotation using amine B. The columns represent the weight distribution of the fractions in the samples.

[1]Tappi values are transformed with a constant value due to confidentiality.

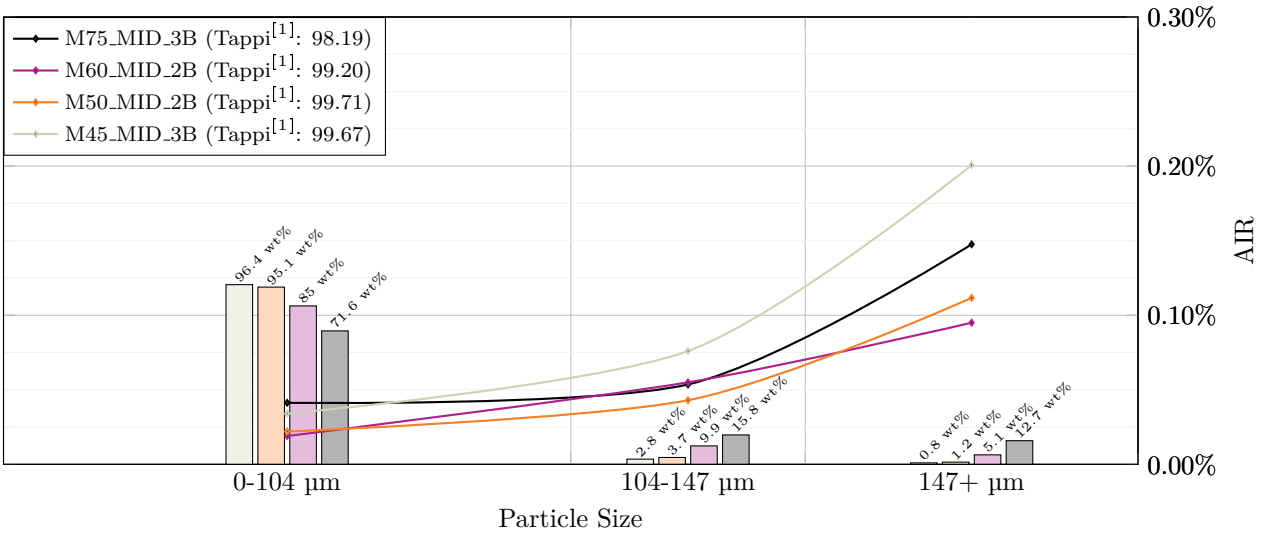


Figure 4.7: Acid insoluble residue (AIR) concentration in the fractions of the lowest brightness concentrates of M75_MID subjected to flotation using amine B. The columns represent the weight distribution of the fractions in the samples.

[1]Tappi values are transformed with a constant value due to confidentiality.

4.3.4 Particle size distribution of concentrate compared to feed

The differences in the particle size distribution between the feed and the produced concentrates from the feed subjected to flotation using amine B were examined for all the four PSD curves tested in this thesis. The last of the quadruplicate samples were analysed, as the kinetics analysis were also performed on these samples. The results are shown as undersize in figure 4.8 and as frequency in figure 4.9 below. The frequency is included as this differentiates the lines more and shows differences not easily seen when presented as undersize.

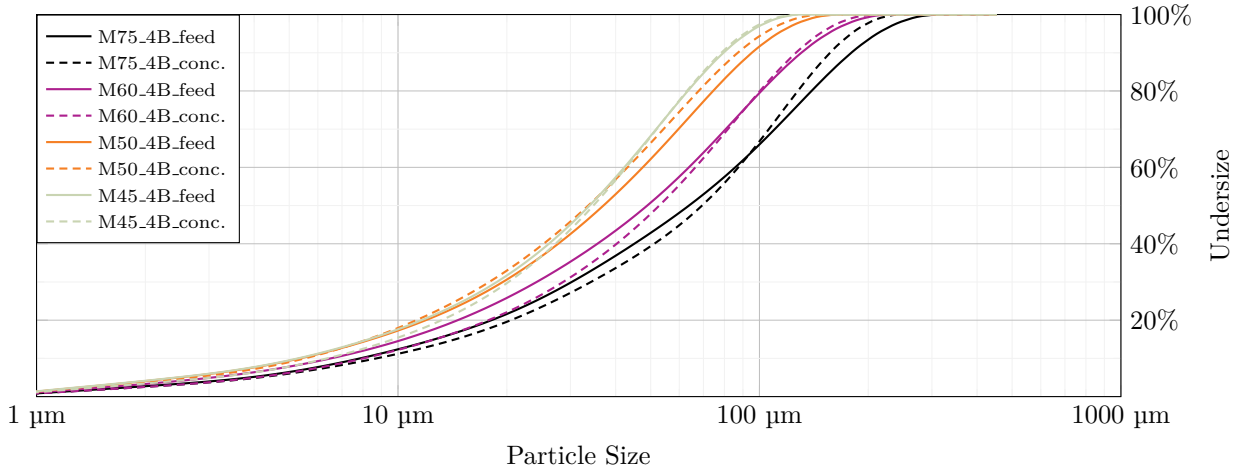


Figure 4.8: Comparison of the PSD of concentrate and feed for each feed rate as undersize. Concentrate getting closer to feed as particle size decreases.

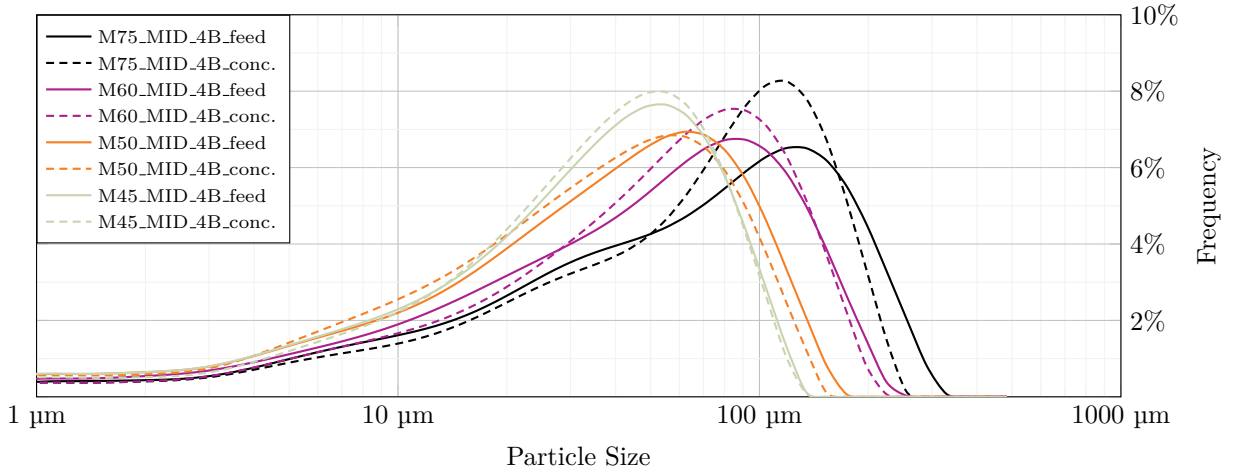


Figure 4.9: Comparison of the PSD of concentrate and feed for each feed rate as frequency. The PSD of the concentrates more similar to the PSD of the feed as is decreases.

4.3.5 Flotation kinetics

Flotation kinetics were measured by weighing the tailings over time. The separation times of the tailings and the addition of collectors were logged using the Flotation Log. This enabled a graphical representation of the tailings over time, and a comparison of the kinetics for the two collectors as well as for different grade groups and different curves within each grade group.

The first observation was that amine B floats the impurities more quickly than amine A, however the start concentration is also larger. After 5 min, the amount of tailings from the samples subjected to flotation using amine B quickly decreased, despite the continuous addition of collector. The results for each grade group are shown in figures 4.10, 4.11 and 4.12.

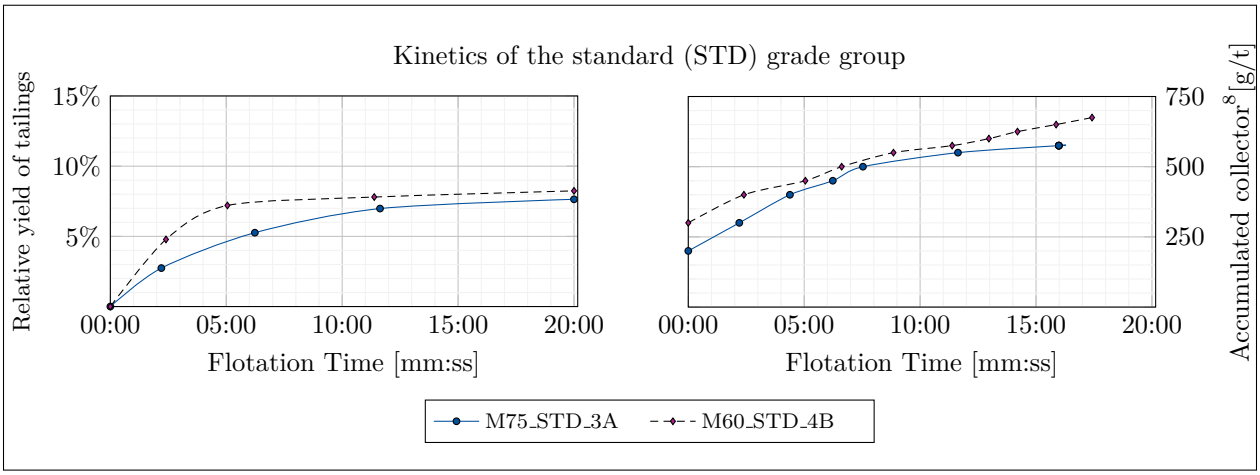


Figure 4.10: Kinetics of STD grade group over elapsed flotation time.

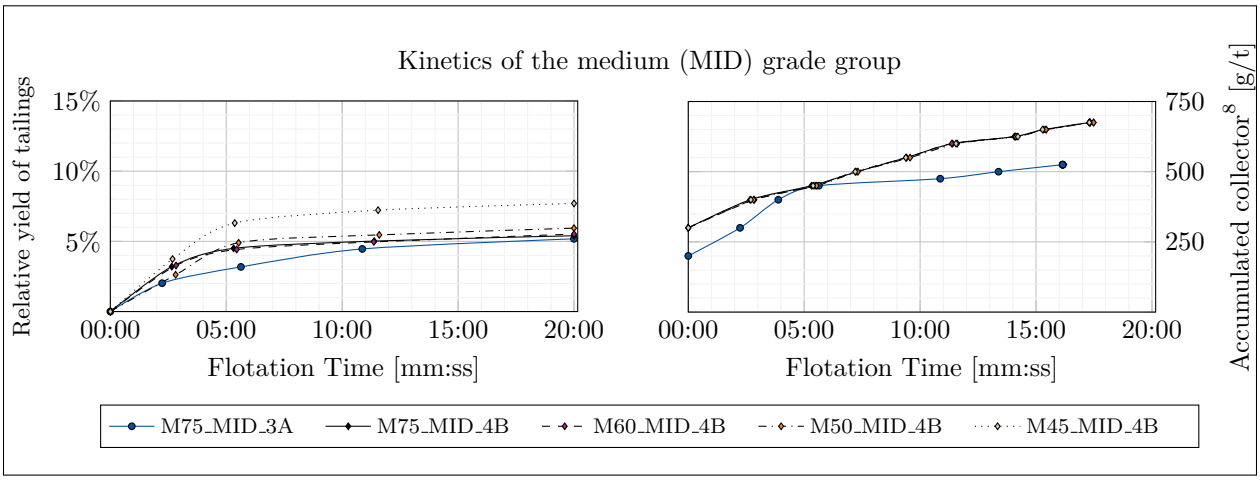


Figure 4.11: Kinetics of MID grade group over elapsed flotation time.

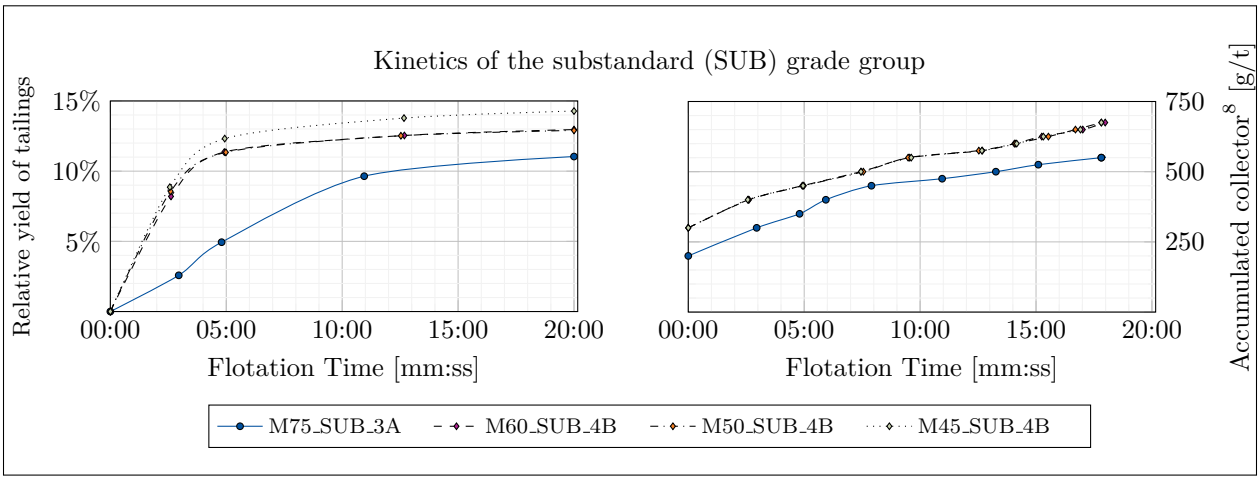


Figure 4.12: Kinetics of STD grade group over elapsed flotation time.

[8]Graphite Collector not included, 100 g/t GC added at the start for every sample subjected to flotation with amine B.

4.3.6 Particle size reduction when micronizing finer curves

It is known that the particle size distribution (PSD) of the slurry affects the brightness measurements; a finer distribution decreases the brightness. It was therefore important to determine how the PSD of the feed affected the final PSD after micronization. To examine this, the slurries produced using the standard micronization process on the different finer curves of the MID grade group were analysed and compared. The slurries (not the dry micronized concentrates) were analysed both using the Micronizer 3000 at NTNU, and with a sedigraph, performed by the processing plant. The results are shown in figure 4.12 below. The sedigraph gives the most accurate representation of the PSD, as the slurries quickly agglomerated when using the micronizer without a dispersing agent. With the micronizer 3000, the very first measurement was considered the most accurate, as the sequential measurements were significantly coarser. An example of the agglomeration of the slurries is included in the appendix, figure F.1. No significant change of the final PSD was found.

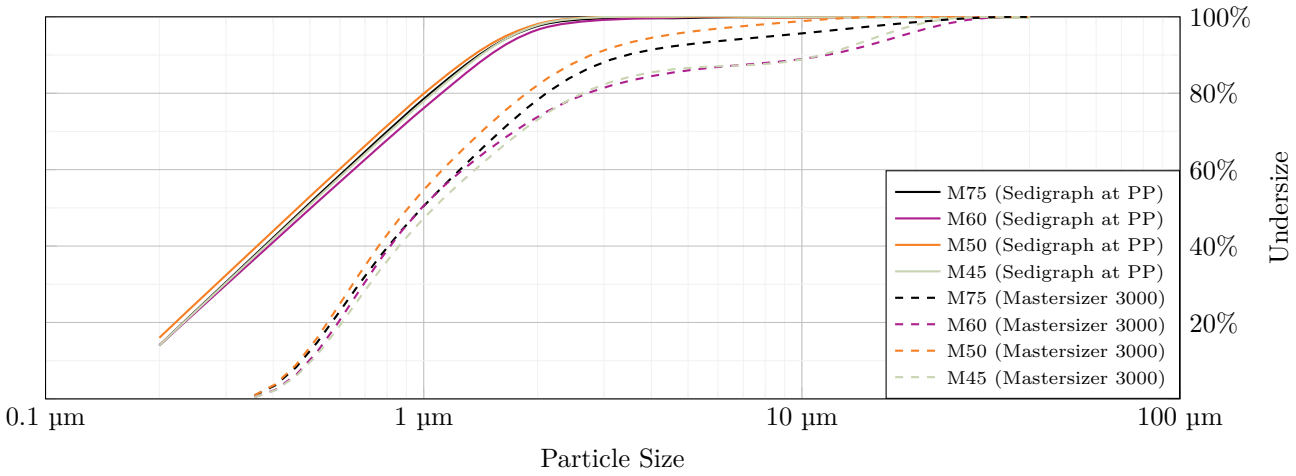


Figure 4.12: Obtained particle size distribution after micronizing the different feed speeds materials. Results from the processing plant's (PP) sedigraph and the laser diffraction at NTNU are shown.

4.3.7 Deviation of flotation, micronization and measurements

The deviations inherent in the quality inspection at Brønnøy Kalk were calculated from the experiments performed during this study. The measurements used for the calculations are included in appendix D, table D.1. 's' is used here, as the calculations were from a sample and not the true deviation.

From the calibration tests at Brønnøy Kalk, the deviation of the measurements on the same tablet was calculated from 13 measurements performed over two weeks. $s_T \approx 0.010$ was calculated from the measurements, and following equation 4.4

$$s^2_{measurement} = s^2_T$$
$$s^2_{measurement} \approx (0.010)^2$$
$$s_{tablet} \approx 0.010$$

Six tablets were made from two different concentrates, three in each. $s_T \approx 0.013$ was calculated from the measurements, and following equation 4.3

$$s^2_{tablet} = s^2_T - s^2_{measurement}$$
$$s^2_{tablet} \approx (0.013)^2 - (0.010)^2$$
$$s_{tablet} \approx 0.009$$

The deviation in the micronization process was calculated from the measurements in part I. Here, the deviation is distinguished between all four mills, a b c and d, and the two used in part II of this study, b and c. For mill b and c, $s_{T(bc)} \approx 0.057$ was measured, and following equation 4.2

$$
\begin{aligned}
s^2_{micronization} &= s^2_T - s^2_{tablet} - s^2_{measurement} \\
s^2_{micronization(ab)} &\approx (0.057)^2 - (0.009)^2 - (0.010)^2 \\
s_{micronization(ab)} &\approx 0.055
\end{aligned}
$$

For all the mills, $s_{T(abcd)} \approx 0.109$ was calculated from the measurements, giving $s_{micronization(abcd)} \approx 0.108$.

The variance in the flotation results are dependent on many aspects, such as feed material and collector used. In this subsection, it is limited to the MID grade group subjected to flotation using amine B in part II of the study, as this series was completed. The $s_{T(M75\_MID\_B)} \approx 0.476$ was calculated from the measurements in part II, and following equation 4.1

$$
\begin{aligned}
s^2_{flotation} &= s^2_T - s^2_{micronization} - s^2_{tablet} - s^2_{measurement} \\
s^2_{flotation(M75\_MID\_B)} &\approx (0.476)^2 - (0.055)^2 - (0.009)^2 - (0.010)^2 \\
s_{flotation(M75\_MID\_B)} &\approx 0.473
\end{aligned}
$$

Following the same equation, using the deviation for mill b and c, the rest of the deviations were as follows:

$$
\begin{aligned}
s_{T(M75\_MID\_A)} \approx 0.092 &\implies s_{micronization(M75\_MID\_A)} \approx 0.072 \\
s_{T(M75\_MID\_B)} \approx 0.476 &\implies s_{micronization(M75\_MID\_B)} \approx 0.473 \\
s_{T(M60\_MID\_B)} \approx 0.198 &\implies s_{micronization(M60\_MID\_B)} \approx 0.189 \\
s_{T(M50\_MID\_B)} \approx 0.232 &\implies s_{micronization(M50\_MID\_B)} \approx 0.225 \\
s_{T(M45\_MID\_B)} \approx 0.147 &\implies s_{micronization(M45\_MID\_B)} \approx 0.136
\end{aligned}
$$

Note that all these calculations are based on small sample sizes and is only meant as a rough overview of which processes contributes most to the overall variation in the brightness measurements. In this case, it shows that the flotation process contributes most of the variations in the brightness measurements for samples subjected to flotation using amine B, and that the standard deviation generally decreases for the sample with lower PSD curves of the feed.

4.3.8 Flotation with different PSD curves of the feed

The main part of this thesis was the examination of how finer particle size distribution (PSD) curves affected the flotation process when subjected to flotation using amine B, especially how it affected the variation of the measured brightness. Decreased variation in the brightness measurements of the quadruplicate flotation trials was expected with decreasing PSD.

Four different PSD curves were tested for three grade groups. Due to insufficient time, only one of the grade groups was completed with all the four curves including the reference (standard curve subjected to flotation using amine A). However, a trend of improved flotation with decreasing PSD curves, as well as decreased variation in the brightness measurements was apparent. The brightness measurements for all the measured PSD curves for all the grade groups are summarized in figure 4.13 below.

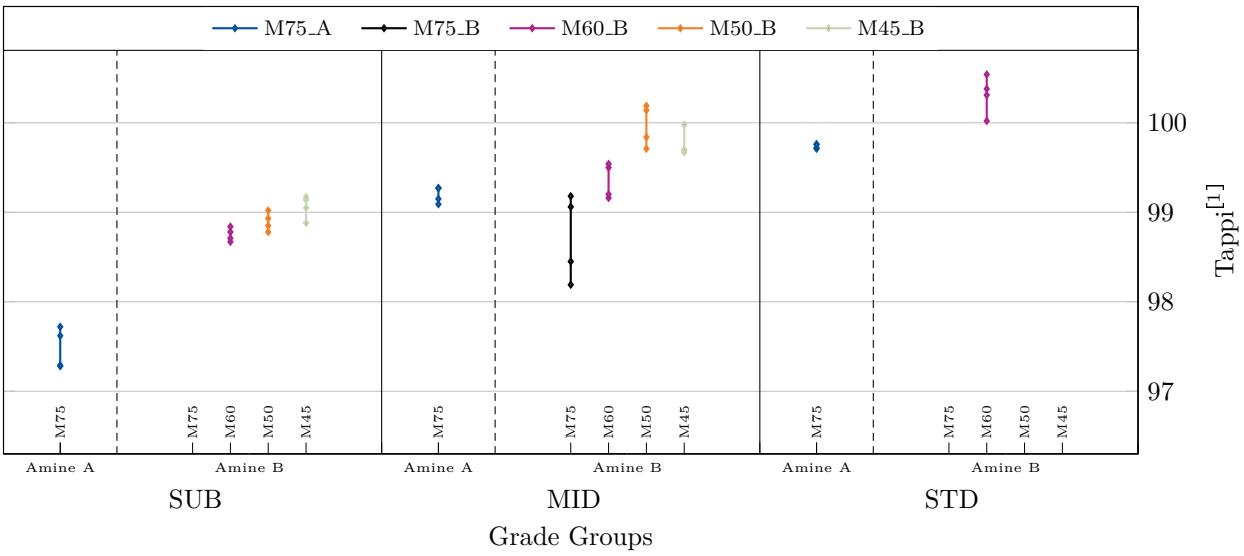


Figure 4.13: Brightness of each grade group, grouped by collector and feed speed

[1]Tappi values are transformed with a constant value due to confidentiality.

4.4 Discussion

4.4.1 Variance in the micronization process

The study on the variance due to use of different micronization mills revealed a systematic difference in the brightness measurements purely from the use of different mills. The calculations of the variances of measuring the micronized concentrate (the tablet making and measuring) showed that the variance found in the micronization mill study was mostly due to the micronization process. As the material was considered homogeneous, the different measurements were most likely due to different final particle size distribution. Based on the results and on the experience of coarser PSDs producing higher brightness measurements, mills a and d were possibly producing a coarser micronized concentrate than mills b and c. The intensity of the four mills were measured just prior to the study, and were considered to be equal. However, both a and d were measured to run at slightly lower speeds than mills b and c, suggesting that this slight difference in speed might be the cause (slower speeds creates a coarser final PSD, leading to increased measured brightness). If the difference in the brightness measurements was due to different particle reduction in the micronization mills, a PSD measurement of the final micronized slurries could reveal it. A coarser PSD for the mills with lower speeds is expected. This was not performed in this study.

From previous experience with the mills, a temperature difference between the mills was assumed to be indicative of differences in the milling process. However, no clear trend was found by measuring the freshly micronized slurries. The temperature differences previously experienced when using the mills may be due to viscosity differences in the slurries; it is known that especially the sulphide bearing marble (SBM) attains a lower viscosity than the standard graphite speckled marble (GSM) when micronized.

To minimize the variance in the brightness measurements, mills b and c were chosen for the rest of the micronizations in this project. These mills produced micronized concentrates which showed brightness results closer to the expected value with lower relative variance, thus theoretically reducing the standard deviation in the rest of the experiments. Using only one mill would further decrease the variance, however, the use of two mills was decided for practical reasons, as the micronization process was time consuming.

4.4.2 Creating finer particle size distribution of the feed

The particle size distribution of the flotation feed was successfully altered by decreasing the intensity of the vibratory feeder, increasing the energy used on size reduction of the particles. However, the finer curves were very time consuming to create. For the two lowest feed rates (obtaining the two finest PSD curves), cooling of the mill was necessary. This was both time consuming and impractical, and would be unacceptable in a fast-paced laboratory procedure where several samples must be possible to mill consecutively.

4.4.3 Variance in the brightness measurements when subjected to flotation using amine B

From the acid insoluble residue (AIR) measurements performed on the fractions of the feed sample (M75_MID) showing a high variance in the brightness measurements of the concentrates (figure 4.6), the low brightness concentrate showed a significantly higher amount of AIR in the coarsest particle size fractions. In addition to this, the high brightness concentrate showed both the same high brightness and the low amount of AIR in all the particle size fractions as the reference concentrate created by flotation using amine A. This supports Brønnøy Kalk's hypothesis that the high variance is caused by a low probability of floating the coarsest impurities when feeds with standard PSD is subjected to flotation using amine B. From these results, it follows that the brightness measurements of flotations with finer curves should be less variable.

The AIR measurements of the size fractions in the concentrates produced by flotation of decreased PSD feed (figure 4.7) revealed some unexpected results. It was expected that when subjecting the feeds with finer PSD to flotation using amine B, the concentration of AIR in the coarsest fractions was reduced. However, for the finest feed sample (M45), the concentration of AIR was largest of all. This was possibly an effect of the amount of material in the fractions, as the finest curves had almost no particles larger than 147 µm. The increase of AIR was probably due to poor flotation of the coarsest particles containing gangue minerals, causing a relative increase of AIR in the small amounts of the coarsest fractions, even though the overall flotation was improved. The measurements may also be less correct for the finest curves, as the coarse fractions were small (only a few grams).

By decreasing the particle size distribution of the feed, two effects were assumed to affect the flotation process: increased mineral liberation and decreased particle size. Firstly, the mineral liberation was assumed to be greater. A greater mineral liberation improves the selective flotation, as the liberated grains have a greater probability of flotation due to increased surface area, where more collector can attach to the mineral surface. Composite grains have a low gangue mineral surface area compared to the particle weight, and has a lower probability of flotation. Secondly, the particle size decreases, which increases the total surface area for the collector to attach on, increasing the probability of attachment to a bubble. However, this means increased collector use, and for the finest particles the probability of colliding and attaching to a bubble decreases (explained in section 2.4).

Comparing the particle size distribution of the flotation feed and the resulting flotation concentrate when subjected to flotation using amine B, the frequency of the particle sizes suggests that the finest (-30 µm) and the coarsest particles (150+ µm) are readily separated from the feed compared to the middle particle sizes, especially around 100 µm. This difference decreases significantly for the finer curves. For the finest curve, M45, the PSD of the feed and the produced concentrate when subjected to flotation using amine B is almost identical. This suggests a good flotation, given that the impurities have the same PSD as the calcite in the feed. However, this is just speculation, as the PSD of the impurities is unknown.

The improvement of the flotation with decreasing particle size distribution can be seen by the increased achieved brightness, and for the MID grade group, the steady decline of AIR in the concentrate. The same decline of AIR in the concentrates is not seen in the SUB grade group, but a steady incline of the brightness measurements with decreasing particle size is found. This suggests increased mineral liberation with finer particle size distribution. The increase of surface area for the finer curves did not seem to strongly impact the flotation process, as the same total amount of collector was used for all the curves within the grade

groups. However, the lower brightness result for the M45 curve in the MID grade group may possibly be due to the increased surface area, as the small particles consume more of the collector.

Unexpectedly, flotation with the different collectors showed different trends in the variance. Feed samples subjected to flotation using amine A showed lower variance with increasing grade, while the opposite was true for flotation using B, where the SUB grade group showed the lowest variance. This is best seen by comparing the amine A blocks with the amine B blocks in figure 4.13.

The asymptotic behaviour of the relative yield of tailings of the flotations using amine B after only 5 min suggests that every particle should have had a long time and many possibilities of attaching to a bubble for all the replicate trials. In the authors opinion, this strengthens the hypothesis of a particle size, composite grains or minerals being the problem. If it was purely probabilistic, the pulp had a very long time to interact in the flotation process. When comparing the kinetics of the flotation procedure with the two different collectors, amine B shows a quicker flotation than amine A. This may have to do with the increased start concentration, however this concentration was needed to obtain a froth.

The deviation calculations showed that most of the variance is due to the flotation procedure. The micronization mills showed a high standard deviation when all four mills were used, which was halved when using only b and c. However, all the calculations were based on small sample sizes, and should be considered approximate at best.

Figure 4.13 summarizes all the brightness measurements in part II of the study. It shows both the relative brightness in each grade group and the variance within each replicate trial of each PSD curve in each grade group. A trend of increasing brightness with decreasing PSD curves is apparent. This is probably a combination of increased mineral liberation as well as decreased size and increased surface area of the particles, increasing the probability of flotation of gangue minerals. This improves the flotation process and reduces the variance in the replicates. On the other hand, the finest curves have a very large total surface area compared to the coarsest curves. The same amount of collector was used in all the flotation trials in each grade group, and this is possibly the reason for the lower brightness measurements for the M45 series compared to the M50 series in the MID grade group.

Due to limited time, not all the planned trials were performed. However, even with incomplete series, the trends of improved flotation process with decreased variance when decreasing the particle size distribution is apparent.

4.4.4 Particle size reduction when micronizing finer curves

The particle size distribution of the concentrates was shown to have little to no effect on the final micronized PSD, ensuring the final results to be comparable. The known effect of decreasing brightness for finer particle sizes was not a problem with the finer curves, as the final PSD of the micronized concentrates were not affected by the PSD of the concentrates. This suggests that the micronization procedure does not need to be tailored to a new particle size distribution of the feed.

4.5 Conclusion

The study on the micronization mills used in the project showed a systematic and significant difference in the brightness measurements from the use of the different mills. The cause was assumed to be different particle size reduction in the four mills. A coarser particle size distribution in the two mills showing the highest brightness measurements was expected, due to slightly lower milling speeds than the other two mills. The two mill showing results closest to the expected result and with low variance was chosen for the rest of the project, as to effectively minimize the overall variance in the measurements. The calculations based on

the variance along the brightness measurements also showed that the flotation process contributed most of the variance, followed by the micronization process.

Four different feed rates of the vibratory feeder was chosen, and resulted in four different PSD curves for the flotation feed (d_{80} of 64 µm for the finest curve and d_{80} of 140 µm for the coarsest curve). The effect of finer particle size distribution of the feed subjected to flotation using amine B was improved flotation results with reduced variance, indicated by increased brightness measurements with lower variance. The improved flotation results is believed to be because of greater mineral liberation, as well as reduced particle size which increases the surface area and increases the probability of flotation of the gangue particles. However, for the MID grade group, the finest PSD curve (M45) showed decreased brightness results compared to the M50 curve. This was believed to be because of increased collector consumption due to increased surface area, and a decreased probability of colliding with bubbles for the finest particles, reducing the amount of floated gangue particles.

The examination of the flotation kinetics showed that amine B floats the particles more quickly than amine A, and is practically finished after 5 min flotation. The finest curve (M45) also showed slightly increased tailings yield.

Based on these results, even though not all the grade group series were completed, the PSD of the feed for flotation must be reduced in size when subjected to flotation using amine B. The standard feed curve is simply too coarse for the lifting capacity of amine B, leading to increased variance in the brightness measurements and reducing the reproducibility. Lowering the PSD to something more resembling the M50 or M45 curve (a d_{80} of -75 µm) proved more reproducible. By any practical measure, the mill currently used at Brønnøy Kalk, a Rotor Beater mill with a 250 µm sieve, is not well suited for a finer PSD than a d_{80} of roughly 90 µm. To be able to produce the finer PSD as suggested here, a new mill capable of efficiently producing these finer curves must be acquired. In addition to this, a calibration of the micronization mills is recommended.

As the micronization mills showed systematic differences in the brightness measurements of the same sample, adjusting the micronization mills to achieve more equal results is recommended. The micronization of the finer concentrates was shown to have little to no effect on the final micronized PSD, and no adjustment of the micronization procedure is necessary if the feed PSD is decreased prior to flotation, as is recommended when using amine B in the flotation procedure.

4.6 Suggestions for future work

For future work, a calibration of the micronization mills is suggested. This could be performed by micronizing a homogeneous composite concentrate sample such as in this project, and measure the PSD of the slurries. This could be done with variable speeds of the mills, comparing each mill within the two models.

As not all the trials in the project was completed, the completion of these is suggested for future work, as all the materials are still available. The variance is expected to be the highest in the M75 samples and decrease with the finer curves. The same variance trials should also be performed on the different marble qualities, such as the sulphide bearing marble.

As samples subjected to flotation using amine B is still more variable than the same samples subjected to flotation using amine A, experimenting with the flotation process when using amine B is suggested. This should be performed with finer feed PSD curves, such as M45 or M50. As using 100 g/t GC in the flotation process gave a runny froth, a study into the correct concentration is suggested. Conditioning might also reduce the variance.

5 Overall discussion and conclusion

The overall aim of increasing reproducibility in quality inspection when using amine B in the flotation procedure was subdivided into three studies: i) validation of the laboratory at NTNU, ii) experimenting with amine B, and iii) finding the correct particle size distribution of the flotation feed.

The validation study compared the brightness results achieved from flotation using amine A at NTNU with the brightness results achieved from flotation using amine A at Brønnøy Kalk. The study showed low accuracy, but a triplicate trial at NTNU showed high reproducibility which opens up the possibility of obtaining a representative flotation procedure outside of the laboratory at Brønnøy Kalk. However, many aspects of the process affects the results, and with the high requirements of brightness as well as the high requirements for reproducibility, the procedure is difficult to replicate outside of the laboratory at Brønnøy Kalk. Even if the flotation devices used are exactly the same, human aspects such as manual handling of the flotation process determines the dosage profile and intensity of skimming the froth. Given the exact same dosage profile, the material is most likely milled then subjected to flotation at different times, altering the surface chemistry of the raw material. If not that, the water chemistry can affect the flotation results. In total, given the high requirements of the flotation process, a representative procedure is difficult to achieve. However, it is the authors belief that it is possible, given enough studying on the area.

The experimenting with amine B at NTNU showed expected improvement when adding graphite collector and with conditioning of the pulp. The deterioration of the diluted amine B collector over storage time was less than first assumed as well. These results were considered positive in the pursuit of reproducible results when using amine B in the flotation procedure as part of the quality inspection. Conditioning and addition of graphite collector should be considered when developing the new procedure for flotation using amine B. As the kinetics in part B showed, most of the impurities are quickly removed from the pulp. From this, it is suggested to insert a conditioning time, and shortening the flotation duration accordingly.

Even though not all the grade group series were completed, the results showed an improvement of the flotation with amine B with finer PSD curves. The finer curves also showed significant decrease of the variance in the brightness measurements in the replicate trials. M50 is suggested as the coarsest PSD curve for the flotation feed when using amine B. This finer particle size distribution did not seem to affect the final PSD of the micronized concentrate, and can be safely used without altering the rest of the quality inspection. The recommendation for a new flotation procedure for flotation when using amine B is included in section 6.2.

6 Recommendations for the company

6.1 Dosage profile application

As pointed out in the validation study, one of the sources of variance was the different flotation operators. Different operators evaluate the froth differently, which is assumed to affect the final brightness results. This is especially true when comparing and replicating results. To address this, a digitalization of the dosage profile of each flotation is suggested. As of now, the addition of collector is written on paper, and the total amount of collector is logged digitally. The digitalization of the entire dosage profile could be done by developing a simple application, where all the data is stored. The development of such an application to be run on a generic tablet is not particularly difficult or expensive, and can possible be done by a programming student. The program developed by the author for the logging of the dosage profile in this study was crude and not very user friendly, as the author is no programmer.

6.2 Suggested flotation procedure using amine B

Based on the results from the studies performed in this thesis, the following procedure for flotation using amine B is suggested. The standard flotation procedure for flotation using amine A is the basis, with finer PSD of the feed, and with increased start concentration of amine B including graphite collector. Only 50 g/t of graphite collector is suggested, as the initial froth when using 100 g/t GC was runny.

Flotation device:	Bauer Frästechnik GMBH flotation device. Cell size of 12.5x12x12 cm^3 (HxWxD).
Feed material:	a PSD similar to the M50 is recommended - d_{90}<100 µm, d_{80}<75 µm, d_{50}<40 µm.
Pulp:	500 g feed material with 1.4 L filtered tap water at roughly 28°C.
Settings:	3 L/min air flow and 1600 RPM rotor speed.
Start conc. coll.:	300 g/t amine B and 50 g/t graphite collector.
Dosage profile:	same as the standard flotation when using amine A. No addition of collector in the first and last 2 min of the flotation. Additional amine B added after the first 2 min, in decreasing amounts (100 g/t, 50 g/t, 25 g/t).
Flotation duration:	20 min. Maybe shorter if conditioning of the pulp is included.
Flocc. agent:	added when the flotation is finished, to increase sedimentation time and include the fines otherwise suspended in the cell when decanted.

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Appendices

A Flotation Log

To conveniantly log and monitor the dosage profile and other data in each flotation experiment, an Excel application was created. This was creatively called the ”Flotation Log”.

The application was initiated some months prior to this thesis, during another flotation project on the same material. In the flotation procedure at Brønnøy Kalk, the addition of reagent during flotation is written down on paper. This was the first feature of the application: digitalizing the dosage profile as well as getting the addition-time of the reagent(s). The application was further developed during the thesis, making it easier to both log and look up the dosage profile of each sample, as well as logging a lot more data during each flotation. The logging was made easier (and safer) by creating a remote control with predefined buttons, saving the laptop from exposure to water and flotation spray. The remote control was a Exibel Wireless Numeric Keyboard, of which the output was manipulated using HID macros (software). With Excel 2016, this was simply done by passing a predefined keyboard sequence for each unique button, corresponding to the shortcut key sequence triggering the wanted macro/action in the application. For earlier versions of Excel, this was more difficult due to input events not being bound to a keyboard in this (and probably earlier) versions. This was initially circumvented by a script deactivating excel each time a button was pressed on the remote, which made it much less reliable. This was not a problem with Excel 2016, in which HID macros can be used without a scripted action.

When a flotation procedure is completed, the information is stored in full as a seperate sheet, as well as compressed to make it easier to export to a workbook holding overall information. In short, parameters such as the date and time of flotation, the chemical(s) used, feed, water and water temperature, calculated pulp density, air flow, rotor speed, the total dosage of each chemical (as well as profile), the flotation time and conditioning time are logged. A picture of the application in use is shown in figure A.1 below.

Figure A.1: Flotation Log in use. Wireless remote located below the laptop.

B Flotation results for part A (Validation study)

Table B.1: Flotation procedure for samples in part A

SAMPLE	Date and time	Flotation procedure												
		Chem	Dry feed [g]	Water [L]	Water temp. [°C]	Solids content	Air flow [L/min]	Rotor speed [RPM]	GC [g/t]	Chem cons. [g/t]	Start cons. [g/t]	Cond. Time [min:ss]	Flotation duration w/o cond. [min:ss]	Total Flotation Duration [min:ss]	Amine conc. for stable froth [g/t]
1852_A06_1A	05.02.2016 14:10	A	500	1,8		27,8 %	3,0	-	-	600	200	00:00	20:00	20:00	450
1852_A08_1A	05.02.2016 13:39	A	500	1,8		27,8 %	3,0	-	-	625	200	00:00	20:00	20:00	450
1852_A09_1A	05.02.2016 11:42	A	500	1,8		27,8 %	3,0	-	-	550	200	00:00	20:00	20:00	400
1852_A09_1B	11.02.2016 10:55	B	500	1,8		27,8 %	4,0	-	0	750		02:00	19:31	21:31	600
1852_A13_2A	08.02.2016 10:51	A	500	1,8		27,8 %	3,0	-	-	625	200	00:00	20:00	20:00	550
1852_A18_2A	08.02.2016 11:21	A	500	1,8		27,8 %	3,0	-	-	525	200	00:00	20:00	20:00	400
1832_A05_A01	19.02.2016 11:47	A	500	1,8		27,8 %	3,0	-	0	575	200	00:00	20:02	20:02	500
1832_A05_B01	19.02.2016 12:16	B	500	1,8		27,8 %	3,0	-	0	825	300	00:00	25:07	25:07	450
1832_A05_B02	19.02.2016 14:09	B	500	1,8		27,8 %	3,0	-	0	825	300	05:02	24:58	30:00	450
1832_A05_B03	19.02.2016 14:47	B	500	1,8		27,8 %	3,0	-	5	825	300	00:00	25:03	25:03	450
1832_A05_B04	22.02.2016 11:23	B	500	1,8		27,8 %	3,0	-	50	850	300	00:00	25:12	25:12	400
1852_A03_A01	22.02.2016 13:30	A	500	1,8		27,8 %	3,0	-	0	475	200	00:00	20:00	20:00	400
1852_A03_B01	22.02.2016 12:22	B	500	1,8		27,8 %	3,0	-	0	875	300	00:00	25:00	25:00	400
1852_A03_D01	23.02.2016 12:23	D	500	1,8		27,8 %	3,0	-	0	875	300	00:00	25:00	25:00	400
1852_A09_R01	09.03.2016 13:49	A	500	1,8	29,1	27,8 %	3,0	2400	0	550	200	00:00	20:02	20:02	425
1852_A09_R02	09.03.2016 14:22	A	500	1,8	29,3	27,8 %	3,0	2400	0	550	200	00:00	20:01	20:01	425
1852_A09_R03	09.03.2016 14:58	A	500	1,8	29,5	27,8 %	3,0	2400	0	550	200	00:00	20:06	20:06	425
1852_A18_A02	08.02.2016 11:21	A	500	1,8		27,8 %	3,0	-	0	525	200	00:00	20:00	20:00	400
1852_A18_B01	18.02.2016 10:55	B	500	1,8		27,8 %	3,0	-	0	775	300	00:00	22:21	22:21	500
1852_A18_B02	18.02.2016 13:04	B	500	1,8		27,8 %	3,0	-	5	775	300	00:00	22:30	22:30	450
1852_A18_B03	18.02.2016 13:46	B	500	1,8		27,8 %	3,0	-	50	775	300	00:00	22:20	22:20	-
1852_A18_B04	18.02.2016 14:31	B	500	1,8		27,8 %	3,0	-	0	775	300	05:00	22:21	27:21	550
1853_A02_A01	24.02.2016 13:49	A	500	1,8		27,8 %	3,0	-	0	900	200	00:00	20:06	20:06	800
1853_A02_A02	24.02.2016 15:03	A	500	1,8		27,8 %	3,0	-	0	900	200	00:00	20:00	20:00	750
1853_A02_B01	24.02.2016 14:29	B	500	1,8		27,8 %	3,0	-	0	900	300	00:00	20:02	20:02	500
1853_A02_D04	26.02.2016 12:29	D	500	1,8	29,4	27,8 %	3,0	-	0	900	300	00:00	20:02	20:02	500
1853_A09_A01	24.02.2016 11:51	A	500	1,8		27,8 %	3,0	-	0	500	200	00:00	20:02	20:02	400
1853_A09_B01	24.02.2016 12:28	B	500	1,8		27,8 %	3,0	-	0	750	300	00:00	20:01	20:01	400
1853_A09_D02	24.02.2016 13:05	D	500	1,8		27,8 %	3,0	-	0	750	300	00:00	20:00	20:00	400
1853_A09_D07	29.02.2016 09:35	B	500	1,8	28,8	27,8 %	3,0	2300	0	750	300	00:00	20:01	20:01	400
1853_A11_A01	25.02.2016 11:45	A (1 d old)	500	1,8		27,8 %	3,0	-	0	600	200	00:00	20:00	20:00	550
1853_A11_B01	25.02.2016 12:22	B	500	1,8		27,8 %	3,0	-	0	950	300	00:00	20:00	20:00	800
1853_A11_D03	25.02.2016 13:01	D	500	1,8		27,8 %	3,0	-	0	950	300	00:00	20:01	20:01	750
1853_A12_A01	25.02.2016 13:55	A (1 d old)	500	1,8		27,8 %	3,0	-	0	600	200	00:00	20:04	20:04	200
1853_A12_B01	25.02.2016 14:36	B	500	1,8		27,8 %	3,0	-	0	1300	300	00:00	20:01	20:01	1100
1858_A13_A01	09.03.2016 10:46	A	500	1,8	30,0	27,8 %	3,0	2400	0	500	200	00:00	20:01	20:01	400
1858_A13_B01	09.03.2016 11:29	B	500	1,8	29,4	27,8 %	3,0	2400	0	750	300	00:00	20:10	20:10	500
1855_A02_A01	09.03.2016 12:21	A	500	1,8	27,6	27,8 %	3,0	2400	0	650	200	00:00	20:20	20:20	500
1855_A02_A02	09.03.2016 13:00	A	500	1,8	28,8	27,8 %	3,0	2400	0	650	200	00:00	20:04	20:04	550

Table B.2: Flotation results for samples in part A

Sample	Flotation Yield				AIR			Colour Data[1]					
	Tail. [mm:ss]	Concentrate	Tailings	Loss	AIR feed	AIR prod	AIR tail	Rx	Ry	Rz	Tappi	Index	Verdi b
1852_A06_1A		-	-	-	-	0.02 %	-	98.15	98.04	97.87	97.90	4.92	4.81
1852_A08_1A		-	-	-	-	0.06 %	-	99.92	99.95	100.07	100.09	4.67	4.60
1852_A09_1A		-	-	-	-	0.07 %	-	99.20	99.24	99.42	99.43	4.45	4.56
1852_A09_1B		-	-	-	-	0.07 %	-	99.06	99.08	99.14	99.18	4.61	4.65
1852_A13_2A		-	-	-	-	-	-	98.02	97.95	97.64	97.67	5.09	4.91
1852_A18_2A		-	-	-	-	-	-	100.21	100.21	100.24	100.25	4.65	4.67
1832_A05_A01		81.4 %	3.4 %	15.2 %	3.08 %	0.01 %	79.7 %	99.25	99.33	99.59	99.62	4.33	4.51
1832_A05_B01		78.9 %	3.6 %	17.5 %	”	0.04 %	79.4 %	98.94	99.02	99.26	99.27	4.35	4.52
1832_A05_B02		75.5 %	4.2 %	20.2 %	”	0.03 %	71.8 %	99.58	99.63	99.86	99.87	4.40	4.54
1832_A05_B03		87.8 %	4.2 %	8.1 %	”	0.03 %	74.0 %	99.75	99.78	99.94	99.96	4.48	4.58
1832_A05_B04		90.9 %	4.5 %	4.6 %	”	0.04 %	69.7 %	99.02	99.08	99.29	99.31	4.40	4.54
1852_A03_A01		91.7 %	2.6 %	5.7 %	1.13 %	0.04 %	X	97.77	97.82	97.82	97.87	4.63	4.69
1852_A03_A01 T1	*06:07*		*1.46 %*										
1852_A03_A01 T2	*14:00*		*0.80 %*										
1852_A03_A01 T3	*20:00*		*0.39 %*										
1852_A03_B01		94.2 %	2.5 %	3.3 %	”	0.06 %	X	97.63	97.68	97.67	97.73	4.64	4.69
1852_A03_B01 T1	*03:55*		*1.74 %*										
1852_A03_B01 T2	*12:09*		*0.37 %*										
1852_A03_B01 T3	*19:58*		*0.26 %*										
1852_A03_B01 T4	*25:00*		*0.13 %*										
1852_A03_D01		89.4 %	2.5 %	8.1 %	”	0.02 %	45.7 %	98.10	98.11	98.02	98.08	4.77	4.75
1852_A09_R01		86.7 %	3.4 %	9.9 %				99.99	100.02	100.09	100.11	4.58	4.64
1852_A09_R02		88.0 %	3.4 %	8.7 %				99.73	99.77	99.85	99.87	4.56	4.63
1852_A09_R03		88.2 %	3.7 %	8.1 %				99.89	99.94	100.03	100.07	4.54	4.62
1852_A18_A02		-	-	-	8.20 %	-	-	100.21	100.21	100.24	100.25	4.65	4.67
1852_A18_B01		78.9 %	11.5 %	9.6 %	”	0.00 %	74.2 %	96.29	96.44	96.80	96.80	4.13	4.43
1852_A18_B02		76.8 %	11.5 %	11.7 %	”	0.03 %	72.4 %	96.61	96.75	97.13	97.17	4.13	4.42
1852_A18_B03		73.2 %	10.7 %	16.1 %	”	0.03 %	75.8 %	96.79	96.94	97.35	97.36	4.08	4.40
1852_A18_B04		76.2 %	10.8 %	13.0 %	”	0.05 %	78.3 %	96.83	96.96	97.26	97.27	4.22	4.48
1853_A02_A01		76.1 %	18.4 %	5.6 %	14.37 %	0.03 %	77.6 %	95.66	95.84	96.21	96.23	4.08	4.43
1853_A02_A02		76.1 %	18.2 %	5.7 %	”	0.03 %	78.7 %	97.45	97.58	97.80	97.84	4.31	4.53
1853_A02_B01		76.1 %	17.6 %	6.3 %	”	0.03 %	80.6 %	96.78	96.91	97.15	97.19	4.28	4.51
1853_A02_D04		75.5 %	18.8 %	5.7 %	”	0.03 %	75.8 %	97.80	97.86	97.84	97.93	4.64	4.70
1853_A09_A01		91.5 %	2.6 %	5.9 %	0.96 %	0.01 %	36.4 %	98.42	98.40	98.31	98.35	4.80	4.75
1853_A09_B01		90.5 %	2.1 %	7.4 %	”	0.01 %	44.5 %	98.57	98.46	98.07	98.10	5.21	4.96
1853_A09_D02		93.0 %	2.2 %	4.9 %	”	0.04 %	44.1 %	97.91	97.85	97.60	97.64	5.02	4.87
1853_A09_D07		91.6 %	2.2 %	6.2 %	”	0.02 %	42.6 %						
1853_A11_A01		89.4 %	3.7 %	6.9 %	1.85 %	0.03 %	46.8 %	99.58	99.29	98.39	98.48	5.94	5.32
1853_A11_B01		88.7 %	3.2 %	8.1 %	”	0.05 %	54.0 %	98.60	98.27	97.26	97.33	6.11	5.40
1853_A11_D03		88.7 %	3.4 %	7.9 %	”	0.04 %	54.9 %	98.88	98.56	97.55	97.62	6.10	5.39
1853_A12_A01		88.8 %	4.2 %	7.0 %	1.19 %	0.07 %	20.9 %	98.61	97.93	95.84	95.98	7.65	6.16
1853_A12_B01		89.0 %	4.2 %	6.8 %	”	0.05 %	25.8 %	98.50	97.94	96.17	96.29	7.17	5.93
1858_A13_A01		91.8 %	3.0 %	5.2 %				98.78	98.81	98.80	98.84	4.66	4.69
1858_A13_B01		90.4 %	2.5 %	7.1 %				98.59	98.59	98.48	98.53	4.80	4.77
1855_A02_A01		84.6 %	8.3 %	7.0 %				-	-	-	-	-	-
1855_A02_A02		86.5 %	7.9 %	5.5 %				98.35	98.44	98.59	98.60	4.43	4.58

[1] Colour data are transformed with a constant value due to confidentiality.

Table B.3: Flotation results for samples subjected to flotation at Brønnøy Kalk

Sample	Flotation procedure			AIR			Colour Data[1]					
	Analyzed	Chem	Chem cons. [g/t]	AIR feed	AIR prod	AIR tail	Rx	Ry	Rz	Tappi	Index	Verdi b
1852_A18 (BK)	25.01.2016	A	525	0.08	0.00	0.47	98.96	99.09	99.19	99.27	4.44	4.62
1832_A05 (BK)	14.01.2016	A	575	0.03	0.00	0.52	100.28	100.34	100.29	100.37	4.68	4.72
1852_A03 (BK)	20.01.2016	A	475	0.01	0.00	0.14	99.28	99.32	99.04	99.13	4.94	4.88
1853_A09 (BK)	01.02.2016	A	475	0.01	0.00	0.14	100.36	100.41	100.23	100.31	4.83	4.81
1853_A02 (BK)	27.01.2016	A	500	0.14	0.00	0.67	99.02	99.10	98.93	99.03	4.78	4.81
1853_A11 (BK)	01.02.2016	A	475	0.02	0.00	0.29	100.68	100.68	100.39	100.50	4.99	4.89
1853_A12 (BK)	02.02.2016	A	700	0.01	0.00	0.17	100.36	100.15	99.08	99.24	6.03	5.43
1852_A09 (BK)	20.01.2016	A	550	0.01	0.00	0.16	100.38	100.43	100.30	100.42	4.77	4.78
1858_A13 (BK)	25.02.2016	A	500	0.01	0.00	0.14	99.91	99.90	99.80	99.86	4.80	4.76
1855_A02 (BK)	03.02.2016	A	650	0.05	0.00	0.50	99.22	99.32	99.33	99.39	4.56	4.68

[1] Colour data are transformed with a constant value due to confidentiality.

C Mill Throughput

Table C.1: Rough throughput of the vibratory feeder without mill

Speed	Wt [kg]	time [s]	**Throughput [g/min]**
M80	3.715	26	**8526**
M70	3.715	32	**7054**
M60	1.835	60	**1835**
M50	0.118	60	**118**
M45	0.020	300	**4**
M40	0.005	360	**1**

Table C.2: Rough throughput of the vibratory feeder with mill

Speed	Sample	Wt [kg]	time [s]	**Throughput [g/min]**	Rounds [s]				
					#1	#2	#3	#4	#5
M60	STD	3.16	137	**1384**	137				
	MID	2.87	104	**1656**	104				
	SUB	2.86	110	**1560**	110				
M50	MID	2.90	475	**366**	165	180	130		
	SUB	2.89	401	**432**	240	161			
M45	SUB	2,92	1140	**154**	200	300	180	270	190

D Deviation measurements

Table D.1: Measurements for deviation calculations in part 4.3.7

	Tablet measurement		Tablet		Micronization		Flotation	
#	Sample	Tappi[1]	Sample	Tappi[1]	Sample	Tappi[1]	Sample	Tappi[1]
1	Calibration	102.81	M60_STD_1B	100.02	a_1	99.83	M75_MID_1A	99.15
2	Calibration	102.81	M60_STD_1B	100.00	a_2	99.77	M75_MID_2A	99.09
3	Calibration	102.81	M60_STD_1B	100.03	a_3	99.88	M75_MID_3A	99.27
4	Calibration	102.81	M60_STD_3B	100.54	b_1	99.74	M75_MID_1B	99.18
5	Calibration	102.82	M60_STD_3B	100.56	b_2	99.76	M75_MID_2B	98.45
6	Calibration	102.81	M60_STD_3B	100.54	b_3	99.73	M75_MID_3B	98.19
7	Calibration	102.82			c_1	99.74	M75_MID_4B	99.06
8	Calibration	102.80			c_2	99.76	M60_MID_1B	99.5
9	Calibration	102.79			c_3	99.61	M60_MID_2B	99.2
10	Calibration	102.80			d_1	99.93	M60_MID_3B	99.54
11	Calibration	102.82			d_2	100.00	M60_MID_4B	99.16
12	Calibration	102.80			d_3	99.92	M50_MID_1B	100.14
13	Calibration	102.82					M50_MID_2B	99.71
14							M50_MID_3B	99.84
15							M50_MID_4B	100.19
16							M45_MID_1B	99.69
17							M45_MID_2B	99.7
18							M45_MID_3B	99.67
19							M45_MID_4B	99.98
	s_T	0.010	$s_{T(M60\_STD\_1B)}$	*0.015*	$s_{T(abcd)}$	*0.109*	$s_{T(M75\_MID\_A)}$	*0.092*
			$s_{T(M60\_STD\_3B)}$	*0.012*	$s_{T(bc)}$	0.057	$s_{T(M75\_MID\_B)}$	*0.476*
			s_T	0.013			$s_{T(M60\_MID\_B)}$	*0.198*
							$s_{T(M50\_MID\_B)}$	*0.232*
							$s_{T(M45\_MID\_B)}$	*0.147*
	$\mathbf{s}_{measurement}$	**0.010**	$\mathbf{s}_{tablet}$	**0.009**	$\mathbf{s}_{mic(bc)}$	**0.055**	$s_{flot(M75\_MID\_A.bc)}$	*0.072*
					$s_{mic(abcd)}$	*0.108*	$\mathbf{s}_{flot(M75\_MID\_B.bc)}$	**0.473**
							$s_{flot(M60\_MID\_B.bc)}$	*0.189*
							$s_{flot(M50\_MID\_B.bc)}$	*0.225*
							$s_{flot(M45\_MID\_B.bc)}$	*0.136*

[1] Colour data are transformed with a constant value due to confidentiality.

E Flotation results for part B

Table E.1: Flotation results part B

SAMPLE	Flotation procedure[1]						Flotation Yield			AIR			Colour data[2]					
	Date and time	Chem	Water temp. [°C]	GC [g/t]	Start coll. cons. [g/t]	Coll. cons. [g/t]	Conc. wt.	Tail. wt.	Loss wt.	Feed	Conc.	Tail.	Rx	Ry	Rz	Tappi	Index	b
M75_SUB_1A	12.04.2016 11:55	A	28.0	0	200	550	87.7 %	11.3 %	1.0 %	7.52 %			98.00	97.96	97.64	97.72	5.06	4.91
M75_SUB_2A	12.04.2016 12:30	A	27.8	0	200	575	87.1 %	11.4 %	1.5 %	”			97.79	97.78	97.56	97.62	4.94	4.85
M75_SUB_3A	12.04.2016 16:42	A	29.2	0	200	550	87.9 %	11.0 %	1.1 %	”			97.47	97.45	97.20	97.29	4.97	4.86
M75_SUB_4A	12.04.2016 20:31	A	27.6	0	200	550	87.5 %	11.6 %	0.9 %	”			97.52	97.50	97.23	97.28	4.99	4.88
M60_SUB_1B	20.04.2016 17:20	B	28.1	100	300	675	81.3 %	13.2 %	5.5 %	7.83 %	0.06 %	58.5 %	99.24	99.17	98.70	98.78	5.25	5.01
M60_SUB_2B	20.04.2016 17:52	B	27.7	100	300	675	85.7 %	13.6 %	0.7 %	”	0.04 %	56.7 %	98.96	98.92	98.60	98.67	5.06	4.91
M60_SUB_3B	20.04.2016 18:29	B	27.9	100	300	675	81.9 %	16.2 %	1.9 %	”	0.06 %	49.4 %	99.16	99.11	98.77	98.84	5.10	4.93
M60_SUB_4B	20.04.2016 19:00	B	28.1	100	300	675	86.3 %	13.0 %	0.7 %	”	0.03 %		99.00	98.96	98.64	98.71	5.05	4.91
M50_SUB_1B	20.04.2016 00:32	B	27.9	100	300	675	85.2 %	12.5 %	2.3 %	7.82 %	0.09 %	62.9 %	99.21	99.16	98.76	98.85	5.15	4.96
M50_SUB_2B	20.04.2016 01:02	B	28.4	100	300	675	86.4 %	13.2 %	0.4 %	”	0.10 %	60.1 %	99.25	99.20	98.84	98.93	5.11	4.95
M50_SUB_3B	20.04.2016 01:33	B	28.4	100	300	675	86.1 %	13.2 %	0.6 %	”	0.06 %	60.4 %	99.42	99.36	98.94	99.02	5.19	4.98
M50_SUB_4B	20.04.2016 02:07	B	28.3	100	300	675	85.0 %	12.9 %	2.1 %	”	0.11 %		99.06	99.01	98.69	98.78	5.07	4.91
M45_SUB_1B	19.04.2016 19:20	B	27.9	100	300	675	83.3 %	15.0 %	1.6 %	7.84 %	0.08 %	47.2 %	99.43	99.39	99.06	99.14	5.08	4.92
M45_SUB_2B	19.04.2016 19:54	B	28.4	100	300	675	83.1 %	17.0 %	-0.2 %	”	0.09 %	47.7 %	99.51	99.46	99.11	99.17	5.10	4.93
M45_SUB_3B	19.04.2016 20:37	B	27.6	100	300	675	86.6 %	12.8 %	0.7 %	”	0.08 %	61.5 %	99.27	99.21	98.77	98.88	5.22	4.99
M45_SUB_4B	19.04.2016 21:11	B	27.4	100	300	675	84.5 %	14.3 %	1.2 %	”	0.03 %		99.39	99.35	99.00	99.05	5.10	4.93
M75_MID_1A	12.04.2016 13:18	A	28.3	0	200	525	93.6 %	5.5 %	0.9 %	2.46 %	0.04 %		99.08	99.10	99.12	99.15	4.63	4.67
M75_MID_2A	12.04.2016 13:52	A	28.1	0	200	525	93.6 %	4.6 %	1.8 %	”			99.07	99.07	99.05	99.09	4.70	4.70
M75_MID_3A	12.04.2016 17:11	A	27.8	0	200	525	93.2 %	5.2 %	1.6 %	”			99.19	99.21	99.24	99.27	4.63	4.67
M75_MID_1B	13.04.2016 08:16	B	28.5	100	300	675	93.2 %	5.9 %	0.9 %	2.46 %	0.04 %		99.20	99.19	99.11	99.18	4.77	4.74
M75_MID_2B	13.04.2016 08:50	B	28.5	100	300	675	93.8 %	4.8 %	1.4 %	”			98.43	98.44	98.42	98.45	4.69	4.70
M75_MID_3B	13.04.2016 09:25	B	27.4	100	300	675	94.1 %	4.7 %	1.2 %	”	0.04 %		98.07	98.10	98.14	98.19	4.61	4.66
M75_MID_4B	19.04.2016 02:16	B	28.0	100	300	675	93.2 %	5.4 %	1.4 %	”	0.03 %		99.10	99.09	99.02	99.06	4.77	4.80
M60_MID_1B	18.04.2016 13:54	B	28.1	100	300	675	89.0 %	6.6 %	4.4 %	2.38 %		35.8 %	99.54	99.52	99.44	99.50	4.79	4.75
M60_MID_2B	18.04.2016 14:27	B	27.9	100	300	675	93.9 %	5.4 %	0.7 %	”		48.4 %	99.32	99.29	99.18	99.20	4.83	4.76
M60_MID_3B	18.04.2016 19:37	B	27.7	125	300	675	93.4 %	5.6 %	1.0 %	”		42.3 %	99.61	99.58	99.48	99.54	4.82	4.76
M60_MID_4B	18.04.2016 20:21	B	28.0	100	300	675	93.2 %	5.5 %	1.3 %	”			99.17	99.17	99.13	99.16	4.73	4.72
M50_MID_1B	18.04.2016 18:34	B	28.3	100	300	675	93.3 %	5.9 %	0.7 %	2.38 %			100.31	100.27	100.09	100.14	4.91	4.81
M50_MID_2B	18.04.2016 19:05	B	27.6	100	300	675	93.5 %	6.0 %	0.6 %	”	0.02 %		99.75	99.74	99.67	99.71	4.77	4.74
M50_MID_3B	18.04.2016 15:03	B	28.3	100	300	675	93.4 %	5.7 %	0.8 %	”		44.8 %	99.88	99.88	99.79	99.84	4.78	4.75
M50_MID_4B	18.04.2016 15:36	B	28.4	100	300	675	92.9 %	5.9 %	1.2 %	”			100.37	100.33	100.13	100.19	4.93	4.82
M45_MID_1B	13.04.2016 14:18	B	28.1	100	300	675	94.6 %	4.5 %	0.9 %	2.42 %			99.87	99.84	99.66	99.69	4.90	4.81
M45_MID_2B	13.04.2016 14:49	B	27.5	100	300	675	94.7 %	4.4 %	0.9 %	”	0.01 %		99.94	99.89	99.69	99.70	4.96	4.83
M45_MID_3B	13.04.2016 15:27	B	28.4	100	300	675	94.1 %	5.0 %	0.9 %	”			99.84	99.81	99.64	99.67	4.90	4.81
M45_MID_4B	19.04.2016 02:52	B	27.8	100	300	675	91.2 %	7.7 %	1.1 %	”			100.09	100.06	99.93	99.98	4.85	4.78
M75_STD_1A	12.04.2016 14:26	A		0	200	575	92.8 %	6.3 %	0.9 %	3.63 %			99.42	99.48	99.69	99.72	4.40	4.54
M75_STD_2A	12.04.2016 15:50	A	31.0	0	200	575	92.0 %	6.8 %	1.2 %	”			99.52	99.57	99.73	99.76	4.47	4.58
M75_STD_3A	12.04.2016 17:44	A	28.1	0	200	575	91.0 %	7.6 %	1.3 %	”			99.40	99.47	99.68	99.71	4.38	4.54
M60_STD_1B	20.04.2016 22:12	B	27.9	100	300	675	92.5 %	7.0 %	0.6 %	3.49 %		53.8 %	99.86	99.90	99.99	100.02	4.54	4.63
M60_STD_2B	20.04.2016 22:43	B	28.1	100	300	675	91.2 %	8.2 %	0.5 %	”		43.7 %	100.19	100.22	100.27	100.31	4.60	4.65
M60_STD_3B	20.04.2016 23:36	B	28.2	100	300	675	90.6 %	8.9 %	0.5 %	”	0.02 %	39.0 %	100.47	100.48	100.50	100.54	4.64	4.67
M60_STD_4B	21.04.2016 00:27	B	27.9	100	300	675	86.5 %	8.2 %	5.2 %	”	0.07 %		100.29	100.30	100.34	100.38	4.62	4.66

[1] The standard flotation procedure was followed for every sample in this table: feed = 500 g and water = 1.4 L (solids content = 26.3%), air Flow = 3.0 L/min, rotor speed = 1600 RPM.

[2] Colour data are transformed with a constant value due to confidentiality.

F Agglomeration of micronized slurry

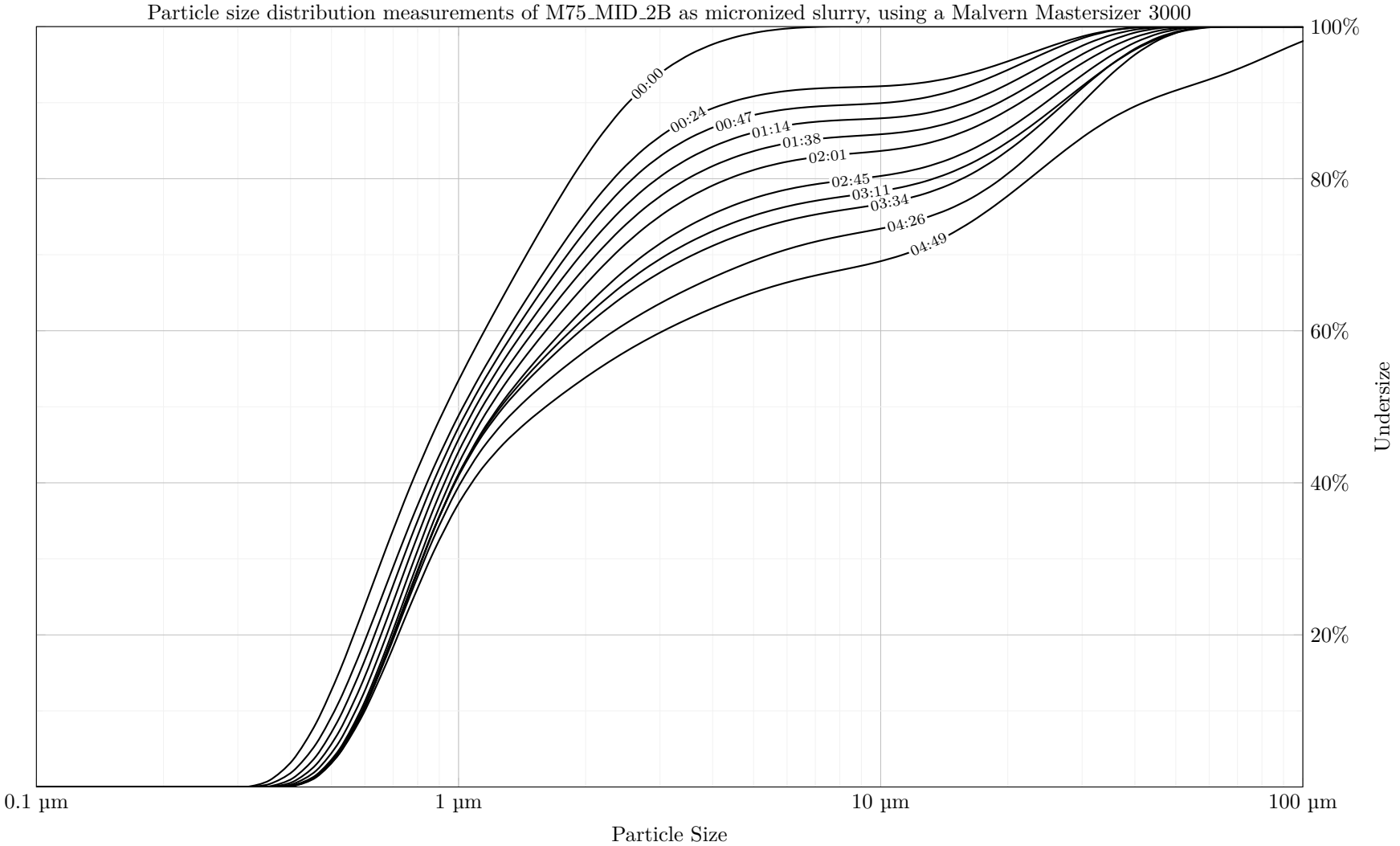

Figure F.1: Agglomeration of micronized slurry dispersed in water. The micronized slurry sample was measured on a Malvern Mastersizer 3000 with continuous measurements. The results are here shown as [min:sec] after the initial measurement (first measurement was shortly after the sample was subjected to the water). The particle size distributions are plotted here for the measurements [min:sec] after the initial measurement. This is all one sample, showing the agglomeration occurring during water dispersion without dispersing agent.