

Development of measurement of froth characteristics

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Abstract

Flotation is a very useful process. It is very popular due to its ability to collect small particles from mineral slurry, for example reducing the grade of silica in iron ore; to separate two kinds of mineral from each other. As more complex ores have to be mined and higher-grade iron ore concentrates with lower impurities are requested by the steel industry, achieving lower grades of silica in iron ore is a constant struggle.

The aim of this work was to investigate the influence of different parameters on the froth stability of silica flotation where silica is separated from magnetite. Magnetite is a ferromagnetic mineral; its chemical formula is Fe₃O₄. The high-grade concentrates of magnetite are used for iron ore pellet production in steel industry and for advanced metallurgical applications.

Many parameters can affect the froth stability in mineral flotation. For example, type of collector, concentration of collector, particle size, electrolytes and concentration of electrolytes, airflow and stirring rate. In this work, the focus has been the effect of stirring rate, airflow and concentration of collector.

There are a few factors used in froth stability evaluating, for example, froth maximum height in equilibrium, growth velocity, decay velocity, froth retention time, air recovery, froth half life time, etc. In this work, except froth half life time, the rest of other five factors are compared to have a whole picture of the froth stability with the change of different parameters.

The airflow plays an important role for froth stability when stirring rate is constant. However, the stirring rate has major effect on the froth stability only when the airflow is high. The alternation of the collector concentration can significantly impact the froth height when the stirring rate and airflow are constant, nevertheless the growth velocity is not affected by the collector concentration change.

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1. Introduction

Froth flotation, which is over 100 years old as a patented process is widely used in mineral processing plants. Nowadays, froth structure and stability attract more and more attention because its significant role in determining the mineral grade and recovery achieved from a flotation operation.

The aim of this master thesis was to investigate the influence of different parameters on the froth stability of silica flotation where silica is separated from magnetite. The aim of this project is to further develop the froth measuring method and to build up understanding of how different parameters such as airflow, agitation and amount of surfactant affect the characteristics of the froth.

A few factors were used to evaluate froth stability in this work, froth maximum height in equilibrium, growth velocity, decay velocity, froth retention time, air recovery. These factors can help to get integrate understanding of the froth.

This work has been divided in five parts:

- ♦ Study the influence of different stirring rate on the froth behavior at constant airflow.
- ♦ Study the effect of different airflow on the froth behavior at constant stirring rate.
- ♦ Study the impact of different collector concentration on the froth behavior at constant stirring rate and airflow.
- ♦ Reproducibility of the test
- ♦ MODDE-design of the two parameters: stirring rate and airflow

2. Theoretical background and literature review

2.1 Froth Flotation

Froth flotation, which was commercially introduced in mineral industry early in the 20th century about 100 years ago, is applied in many instances in the primary mineral industries for separations of solids. Besides mineral industries, flotation can also be used in wineries, food factories, smelters and refineries. After mining the material from any deposit on the earth, crushing and grinding operations are applied to liberate the mineral species. Processing water is added to produce the sample insuspension, which is then added in the cell. Bubbles are induced into the slurry. Surfactant acts in the interface of gas and liquid, and change the interfacial properties. Modifying agent can be added in to the slurry depend on the kind of the ore sample floated. The hydrophobic particles attach to the bubbles and are carried to the surface of the slurry where the froth is formed. The surfactant is used to make certain mineral particles hydrophobic, and leaving others in a hydrophilic form. [1]

2.1.1 Role of surfactant

Various particles with different surface properties can be separated by flotation process. In this process, hydrophobic particles or hydrophilic particles which are changed into hydrophobic by surfactants, attach to the gas bubbles in the pulp and are floated to the froth, with most other particles remaining in the bulk. By separating the froth from the bulk pulp, the hydrophobic particles thus can be separated from the slurry.

Flotation reagents can control or modify the surface properties of minerals selectively. By developing the flotation reagents, significant advances in flotation can be made. Flotation depends on the probability of attachment of the bubbles to the particles. The ability of the gas bubbles to attach to the particles is a function of the hydrophobicity of the particles. When a solid particle is attached to a gas bubble in a liquid, the resulting contact angle is given by Young's equation in terms of the interfacial energies

$\gamma_{sg} - \gamma_{sl} = \gamma_{lg} \cos \theta$

where γ_{sg} , γ_{sl} , and γ_{lg} are solid-gas, solid-liquid, and liquid-gas interfacial tensions, respectively, and θ is the contact angle. Adhesion of the gas bubbles to the particles in the suspension requires a contact angle that is significantly larger than zero. The larger the contact angle, the greater is the hydrophobicity of the particle, and the easier it is for the bubble to attach. [3]



Figure 1: Contact angle between bubble and particle in an aqueous medium [4]

2.1.1.1 Cationic collectors

Because of the uniqueness of every ore, the reagents require careful test work to be determined in each case. However, based on the prior similar test work, it might be possible to predict the suitable reagent for a new composition of ore.

There are a few minerals, such as natural sulfur and graphite, which are naturally hydrophobic. In most cases, the mineral particles are hydrophilic and surface-active agents are added to impart the hydrophobicity to the particles. [3]

Both anionic and cationic collectors can be used for this purpose depending on the charge of particles. The cationic collector will be emphasized below.

In cationic collectors, the nitrogen atom carries the cationic charge. Common products are amine and quaternary ammonium-based products [2]. These types of collectors have been in industrial use since the mid 1930's and are especially useful in the flotation of silicate minerals.

The general structure is:

$R-NH_2$

NH2 is the polar hydrophilic group and R is the hydrophobic tail, hydrophobic carbon chain comprises 8-24 carbon atoms. The longer the carbon chain is, the harder the amine is to disperse.

These molecules will ionize in solution. The ionized molecule has a positively charged head that will attach on the silicates' negatively charged surfaces, and the hydrophobic tail will attach to the air bubbles [14].

Since pH will change the polarity, cationic collectors must be used in carefully controlled pH. The amines are weak bases and occur as cationic ammonium ion at pH below 10.

The change in the hydrophobicity of the adsorbed layer, and not the occurrence of

the adsorption bond itself influences the floatability of mineral particles. It is known that an increase in the alkyl chain of a given collector improves floatability and lowers the threshold concentration needed for flotation.[1]

the formation of ion-molecular complex at a certain pH range is an important property of amine collectors.

2.1.1.2 Critical micelle concentration, CMC

When measuring the different physicochemical properties of an aqueous solution of a surfactant, many peculiarities will be encounter, for instance cationic surfactants, as exemplified in figure 2.



Figure 2: Schematic representation of the concentration dependence of some physical properties for solutions of a micelle-forming surfactant[2]

From the above figure, at low concentrations, most properties are similar to those of a simple electrolyte. But surface tension shows distinct exception. Giving a notable physical property to be measured to get CMC of the surfactant, since it decreases rapidly with surfactant concentration,.

Along with the increasing concentration of the surfactants, they tend to assembly. The first-formed aggregates are generally approximately spherical in shape. Such aggregates are called micelles, and the concentration where they start to form is known as the critical micelle concentration, abbreviated to CMC.

The CMC will change with type and concentration of electrolytes and of course with changes in pH.[2]

2.1.2 Role of electrolyte

There are many factors that can influence plant flotation. The presence of interfering ions in the process water is one of the most common one. These ions may come from many different places in the process, for example it could be organic chemicals such as tannic acid and lignins or it can come from the water added to the process. The process water is recirculated and will be almost saturated with dissolved ions from the ore.

Multivalent ions such as calcium and magnesium can be particularly devastating for amine flotation [15]. Together with the increase of concentration of magnesium and calcium, an increase in collector consumption and loss of selectivity will take place, because of the increased competition for negative sites on the surface of the mineral particles [16].

pH will dramatically influence the effect of these ions on the flotation. The recoveries are affected very little by calcium or magnesium at pH 7. The recoveries gradually decrease over pH 10.5. An explanation to this could be the absorption of the ions on the mineral. Adsorption measurements have shown that the calcium ion is adsorbed as hydrated Ca²⁺ at pH 7, whereas it is adsorbed as CaOH⁺at pH 11. [17].

These include the action of the electrolytes in disruption of hydration layers surrounding the particles and enhancing bubble-particle capture, reduction of the electrostatic interactions, and an increasing charge on the surface of the bubbles to prevent primary bubble coalescence.

The flotation efficiency will increase with additional salt concentration.

The inhibition of bubbles to coalescence in electrolytes can be realized by the application of a combining rule based on the nature of the cationic/anionic pair. Viscosity and electrostatic repulsion were ruled out as possible explanations. In fact, following conventional electrostatic double-layer theory, and increase in salt concentration would reduce the double-layer repulsion and should induce inhibition [1].

2.1.3 Role of pH

In flotation, the pH of the slurry is crucial, as in solution, the extent of ionization, hydrolysis of surfactants and the charge on the mineral surface is determined by pH. This in turn influences the possibility of the collector to attach on the surface. Therefore, at the various ionized solid/liquid interfaces, pH can either helps or hinders the adsorption of the surfactant, contributing to greater or lesser selectivity of flotation [1].

Researchers have established that at an approximate pH of 2, silica has zero charge, amphiboles at 5 and magnetite at 7. This phenomenon explains why at a pH of 8-9, silica floats more easily, amphibole floating next and magnetite only when excess of reagents is added to the pulp.

Flotation usually takes place in the pH range of 8 to 11[18]. It is not favorable to run the process with an acid pH since this will damage the equipment due to the high

tendency of corrosion and dissolve iron oxide.

2.1.4 The major forces in surfactant adsorption

Electrostatic adsorption of surfactants

Electrostatic interactions play a governing role in the mineral-surfactant adsorption process in the system where the surfactants and the particles are oppositely charged. Charge on the particle surface, in the case of oxides, is a result of the hydrolysis of surface species followed by pH-dependent dissociation of the surface hydroxyl groups:

 $MOH = MO^- + H^+$

 $H^+ + MOH = MOH_2^+$

where M represents the interfacial metal atom. The pH at which the surface charge is zero is called the point of zero charge (PZC). In the solutions which are more acidic than the PZC, the oxides carry a positive charge and a negative charge in those is more alkaline [3]. The adsorption of amine-silica follows this mechanism. When the solution is more alkaline than PZC, in this case pH=8, the surface charge of silica is negative, attracts the positive charged etherdiamine collector.

Chemical adsorption of surfactants

Between the surface site of the particles and the polar head group of the surfactant, chemical adsorption or "chemisorption" takes place through covalent or coordinate bonds. The result is the formation of a new surface compound. Chemisorption takes place in the systems where metal ions don't leave their lattice sites. This kind of chemisorptions is termed "surface reaction", when metal ions composing the mineral move out of their lattice sites and interact with the collector,.

If chemisorption took place in the system, it would be the dominant froth compared with electrostatic adsorption of surfactants.

Hydrogen Bonding

For adsorption of amount of reagents, particularly those containing hydroxyl, phenolic, carboxylic and amine groups, hydrogen bonding between the surfactants and the particle surface is accountable.

2.1.5 Hydrodynamics of mineral flotation

The collision between particles and bubbles in the liquid suspension, attachment of the particles to the bubbles, and levitation of the particle-bubble aggregates to the surface of the suspension assume vital roles in flotation in addition the forces between the surfactant and the particle surfaces.

Film rupture between minerals and bubbles

In determining the stability of bubbles and droplets in froth, rupture of thin liquid films between liquids, gases, and solids plays a governing role.

Particle-Bubble interactions in Solutions

Many varieties of forces can result in attachment of particles to bubbles, such as electrical double-layer forces between the particle and the bubble (with adsorbed surfactant on both), Van Der Waals forces, the energy change due to the transfer of the hydrocarbon chains adsorbed on the particles to the gaseous phase.

Electrical Double-Layer Interactions

In the mechanism of the electrostatic stabilization of colloids, electric Double Layer is the phenomenon playing a fundamental role. When negatively charged ions of the dispersion medium are adsorbed on particles surface, colloidal particles get negative electric charge. A negatively charged particle attracts the positive counterions surrounding the particle. Electric Double Layer is the layer surrounding a particle of the dispersed phase and including the ions adsorbed on the particle surface and a film of the countercharged dispersion medium. As shown in figure 3.

Zeta potential is electric potential in the interfacial double layer (DL) at the location of the slipping plane versus a point in the bulk fluid away from the interface. In other words, zeta potential is the potential difference between the dispersion medium and the stationary layer of fluid attached to the dispersed particle as in figure 3. [3]



Figure 3: Electric double layer and Zeta potential [20]

Zeta potential varies with the distance from the particle as it is shown in figure 3; The zeta potential decreases when the distance to the surface increase. When two

particles move closer to each other, the overlap of double layers will lead to the interaction energy change.

Van der Waals interactions

When the bubble and particle move together, an energy change will be associated with van der Waals forces, because the present system is comprised of three different media (solid, air, and intervening solution).

Interaction due to Steric Repulsion

When the interacting spheres (bubble and particle) contain adsorbed layers, their adhesion will be sterically hindered, mainly because of the physical size of the molecules.

2.2 Two phase foam and three phase froth

The froth phase in column flotation plays a very important role in determining the mineral recovery and the grade of produced concentrate. The role of the froth has only relatively recently been recognized as important, and as being more than a simple mass transfer mechanism for carrying particles attached to bubbles from the pulp- froth interface to the concentrate launder.

2.2.1 Two phase foam

Two phase foam is a dispersion of a gas in a liquid or solid. Here only liquid foam will be elaborated. [2] In the foam structure, the bubbles are separated by a thin water film, called lamellae, and the intersection of three lamellae is called Plateau.



Figure 4: Bubbles consist of thin films (lamellae) meeting in lines (Plateau borders)[6]



Figure 5: Real structure of foams showing lamellae, Plateau borders and nodes: aqueous foam (above) and solid foam (below)[6]

Surfactant plays a critical role in foam stability. They adsorb at the interface of gas and liquid, decrease the surface tension of the interface, and therefore increase the formability of the liquid.

Table 1 below lists out the forces acting in the foam system. Among them all, gravitational force is the most common one, it causes drainage of the liquid between the air bubbles. By either increasing the viscosity of the bulk liquid or by adding particles either solid particles or emulsion droplets, the drainage can be slowed down. Particles can stabilize the foam by two mechanisms: 1) These particles are trapped in the plateau borders, so by a local increase in the viscosity, the drainage is hindered.



Figure 6: Emulsion droplets, or suspended particles, can hinder further drainage of the foam liquid, if they are trapped in the plateau borders [2]

2) the particles themselves are surface active. By adsorbing, or chemically reacting, hydrophobic moieties on the surface of the particles, hydrophilic particles can be changed into hydrophobic. Too high hydrophobicity of the particles will precipitate the particles and destroy the foam. If the particles are made only partly hydrophobic, however, they will show surface activity, i.e. they will have an affinity for the liquid and air interface. Such systems give very stable foams. When the contact angle of water is around 90°, the best foam stability is achieved, i.e. when one half of the particle is in the liquid and the other half is in the air, as shown in Figure 7.



Figure 7: Solid particles act as foaming agents if they are made partly hydrophobic. The best foam stability is achieved when the contact angle is around 90°[2]

Cause	Effect
Gravitation	
Pressure difference in lamellae and plateau borders	Drainage to foam base
Pressure difference of the gas in bubbles of different size	Drainage to plateau borders Diffusion of gas from small to large bubbles
Overlap between the electrical double layers	Increase in foam stability

Table 1: a summary of the four forces acting on foam[2]

2.2.2 Three phase Froth

In the flotation process it is clearly recognized that there are two distinct particulate systems: a pulp phase and a froth phase. [9]

Froth is a complex three phases system consisting of air bubbles, particles and water, its ability to transport particles into the concentrate launder is crucially determined by the structure and stability of the froth. Flotation froths reach a stable height in the column after a period of time, however, two-phase foams which do not reach an equilibrium height but continue to grow indefinitely. [9] Froth structure (including the bubble size distribution, solids loading and liquid content) are known to play a

significant role in determining the grade and recovery achieved from a flotation process.[10]

2.3 Froth test

A variety of methods has been developed to assess the stability of froth. In general, two types of tests can be identified: dynamic tests and static tests.

A dynamic test is one in which the foam has reached a state of dynamic equilibrium between rates of formation and decay.

A static test is one where the rate of foam formation is zero: the foam, once formed, is allowed to collapse without regeneration by further agitation or input of gas.

Dynamic test comes closest to the dynamics of a real flotation system. The air is continuously introduced into the solution, the foam starts forming at the bottom and then moves upwards. The liquid drains downwards so that quality of the foam varies with height. The top of the foam then reaches the breaking condition and the top layer collapses. Those features are similar to the real flotation and make the dynamic method more likely to give a true indication of the froth stability during a flotation test. [5]

2.4 Influencing parameter of froth stability

The surfactants play a major role in foam stability. They adsorb at the gas-liquid interface and change the interfacial properties. The influence of surfactants on foam behavior is not clear. But it is worth noted that froth stability is mainly controlled by particles, rather than surfactant molecules.[5]

2.4.1 Motion of the particles

For both hydrophobic or hydrophilic particles in the froth, two possible types are considered: those particles attached to the bubble lamellae or those particles that are unattached and free to move through the Plateau borders. The attached particles will mainly only be hydrophobic, whereas the unattached particles may be hydrophobic or hydrophilic.

In most cases, the bubbles are significantly larger, often as much as two orders of magnitude, than the particles which are attached to the bubble lamellae.

The Attached Particles

Because of bubbles bursting at the top surface or through coalescence in the froth, attached hydrophobic particles detach from bubble lamellae and become unattached. Some of the bubble surface area is lost, and the attached particles on that lamella transfers to the Plateau borders, when bubbles coalesce or rupture. The role of the unattached particles is often much more significant than that of the attached, because there is a tremendous number of coalescence occurring in the froth, and

also a significant fraction of the air bursting on the surface.

The Unattached Particles

The unattached particles move through the Plateau border network and will tend to follow the liquid.

2.4.2 Nature of the particles

The nature and concentration of the particles present in the system can also influence the froth stability. Three important parameters are 1. the capability to stabilize the liquid films separating the bubbles; 2. the facility to form a three-dimensional network in the bulk aqueous phase; 3. the ability of the particles to form coherent shell around the bubbles.

Particle size

The dynamic froth stability depends on the particle size. Previous work shows that equilibrium height increase when particles become finer, and hereinafter, the dynamic froth stability increase. Particles can arrange at the liquid/gas interface and stabilize the films by capillary mechanism, if the particle size is small compared to the film thickness. However, particles can bridge and rupture the foam films if they are large. It has been reported that as the particle size decreases, when using <0.7um silica particles, froth become more stable.

Particle shape

The shape of the particles is also critical. The thinning and rupture of liquid film can be accomplished in roughly 0.1s by rounded or spherical particles, however sharp-edged particles rupture the liquid film in about 0.02s. [5]

Hydrophobicity of the particles

When hydrophilic particles are present, the stability of particle-stabilized foams is the lowest. Dippenaar and Harris discovered that very hydrophilic particles of all sizes can destroy froth, and the destabilizing effect is independent of the frother type, unless the frother adsorbs onto the particles. Similar effect would appear, when it comes to the particles of high hydrophobicity. These particles may break films and suppress the froth stability, whereas those with lower hydrophobicity have little effect on the froth stability because they remain dispersed in lamellae.[3]

Gourram-Badri (1997) found that the presence of particles in the foam determine the total foam height, the total foam height is lowered with the increased amount of particles, moreover they found that more hydrophobic particles do not produce a stable steady-state height.[7]

2.4.3 Bubble size

Bubbles, having hydrophobic particle load associated with them, enter the froth from

the pulp/froth interface. The bubble size, density, and coverage of the particles determine the particle load that a bubble can carry. The particles are found on the lower hemisphere of the bubbles, and are free to move on the surface of the bubble, rising through the pulp.

Both by the length and volume of Plateau borders containing the unattached particles and by the lamellae surface area containing, the attached particles The bubble size can affects the flotation process dramatically. The lamellae size distribution is not as the same as the bubble size distribution, and will depend not only on the bubble size, but also the shape.

2.4.4 Bubble coalescence

The rupture of thin liquid films that separates two adjacent bubbles is one of the main reasons for bubble coalescence in froth. [6] When the liquid content in the froth falls below a critical value, this occurs. The presence of particles and surfactants stabilize the bubbles, therefore they are believed to have a detrimental effect on the film rupture.

Due to the presence of hydrophobic particles, bubble coalescence in flotation froths will also occur. A spherical particle will attempt to move toward a central position to meet the contact angle requirement, when it enters a foam film and bridges the two interfaces. A stable orientation will be attained and the life of the film will be prolonged, if the contact angle is less than a critical degree of wetting. The particle will easily dewet through both sides of the lamella, resulting in the film rupture as shown in the fig below, in the case where the bridging particle has a contact angle more than the critical degree of wetting.



A) Contact angle of surfactant solution B) Contact angle of surfactant solution with particle< 90°. with particle > 90°.

Figure 8: Bridging particle behavior in a foam. A) Moderately hydrophobic, B) highly hydrophobic particle [6]

2.4.5 Detachment of hydrophobic particles

Hydrophobic particles detach from the surface of bubbles within flotation froths. The detached particles may be recaptured and transported into the froth when they return to the liquid zone. There are several reasons of the detachment of floatable particles from the froth: Coalescence of bubble is one of the primary reasons. In flotation froths especially at the base and surface of the froth, bubble coalescence occurs constantly. A high rate of coalescence may eventually lead to froth overloading (maximum carrying capacity) to a point where further transportation of floatable particles to the concentrate may not be possible.

In a relatively heavily loaded froth or at the top of the froth where the surfaces of the bubbles are fully covered, the detachment due to insufficient surface area is expected to be effective. At the pulp-froth interface where bubbles rising from the liquid beneath start to crowd and pack together, it is also largely occurs.

During the merging process as a result of the oscillations caused by the coalescence, the coalescence of two bubbles could be vigorous and that the particles residing on the surface of the bubbles could be detached. With adsorbed surfactant used to increase contact angle and thus reduce particle detachment, particle hydrophobicity played an important role. [6]

2.4.6 Frother concentration

The bubble coalescence can be decreased by increasing frother concentration, and the coalescence of the bubbles is prevented at a particular concentration, known as critical coalescence concentration (CCC). As it is shown in the schematic figure 9, coalescence does not occur at concentrations higher than CCC.



Figure 9: Effect of frother concentration on bubble size (schematic)

The effect of flotation frothers on bubble size and its implications for foam stability were studied by Cho and Laskowski (2002). The CCC values and the dynamic foamability index were correlated, and that is determined by bubble coalescence. Foam stability under dynamic conditions is determined by bubble coalescence. [7]

2.4.7 Operating parameters

Conditioning time

Conditioning of flotation feed particles with various chemical reagents is an important parameter. The maximum froth height declines as the conditioning time increases, it might be because the particles hydrophobicity increase and exceed the critical value for stabilizing the froth.

Stirring rate

In froth test, mechanic stir is needed in the cell in order to break down the bubbles which are produced from the nozzle. This in turn will lead to larger surface area in total and make the particle attach to the bubbles and float up more easily.

Airflow rate

Airflow rate is of great importance in determining the flotation rate constant. It can influence the superficial gas velocity in the froth, which is defined as the gas blew in the cell and that grows up into the column as bubbles regardless of the bubble collapse and coalescence.

Froth stability to flotation performance with a series of coal column flotation experiments was directly related by Tao (2000). Airflow, froth height, and feed solids concentration rate was manipulated, it is found that an increase in the airflow rate stabilizes the froth, however operating at superficial gas velocities higher than 2 cm/s results in a significant increase in the ash recovery due to non-selective entrainment. [11]

2. 5 Analyzing factor for froth stability

Used for separating mineral ores, the froth flotation process depends on the establishment of a carefully controlled and stable froth. Measuring the froth height for a given frother dosage and a given airflow rate in a measuring cylinder is a simple, semi-quantitative method of estimating the frothing power of a frother. The relationship between flotation variables, performance and froth structure was determined by previous work. It has become clear that froth stability plays a major role in flotation performance, from these studies. Nevertheless, there is no unique measure, or precise definition, of froth stability as yet. Many different methods to measure froth stability have been employed over the years.

Froth maximum height at equilibrium

From the pictures which are taken from the froth test, the froth maximum height at equilibrium is measured directly. The froth height H is measured as a function of time, t, using the pulp-froth interface as the reference when the froth rises up inside the flotation cell and grows upwards in the measuring column. The froth rise stops and it reaches a constant height, H_{max} , after some time, depending on the froth stability

and operating conditions. [12]

The rate of froth growth and the maximum equilibrium height change for different airflow rates and frother concentrations was studied by Barbian(2003). In this study, when increasing airflow rate and frother concentration, the maximum equilibrium froth height was found to increase. However, the maximum equilibrium height decreases, at high aeration rates and frother concentration. Under these conditions the froth is no longer stable and collapse. [7]

Froth growth velocity

Froth growth velocity is the rate or velocity of movement of froth up the column from a pre-determined starting height to a maximum height of the froth in the column. A patent "Measuring froth stability" measures the froth height in a cell of a flotation circuit for slurry and plots the data over time. They used a fitted model $H = Ho \left(1 - e^{-\frac{t}{r}}\right)$ to fit the curve and their data is described perfectly by this model, as shown in the fig below.



Figure 10: Column forth height as a function of time

They also introduced plenty of other parameters to make the most use out of the model. For instance,

The superficial gas velocity Jg

which is defined as $Jg = \frac{volume \text{ flow of gas}}{cross \text{ sectional area of the cell}}$

It is the gas flow rate per unit area of the column, this parameter is calculated based on the assumption that all the air entering the froth from the pulp was retained in the froth; bubbles at the surface of the froth did not burst and release their contained air.

The air recovery α , represents the fraction of air retained in the froth. Given the consideration of the preceding paragraph, one equation can be presented relating the Jg, α and froth rise velocity u: U=Jg(1- α). Since the instantaneous rise velocity can be calculated from the froth behavior curve and the Jg can be measured by the airflow rate, then the instantaneous bursting fraction (1- α) can be calculated for a given froth height. Consequently, the line plotted by the change of α with H is accessible, a relationship between froth height and bursting fraction can be studied, as it is shown below. [8]



figure 11: Froth height as a function of time; Air retained in froth as a function of froth height [8]

The dynamic froth stability factor and Froth retention time (FRT)

The dynamic froth stability factor Σ , was originally proposed by bikerman (1973). It

represents (the average time that gas remains entrained in the foam) lifetime of a bubble in the froth, and is defined as the ratio of the total volume of froth at equilibrium to the volumetric gas rate introduced into the system. It is expressed as

$$\Sigma = \frac{V_f}{Q} = \frac{H_{max}A}{Q}$$

Where V_f is the foam volume, Q is the gas volumetric flow rate and H_{max} and A are the total foam height and the cross-sectional area of the vessel, respectively.

The experiments at laboratory scale to measure the dynamic stability factor and equilibrium volume of the froth under different operating conditions—different airflow and frother concentration was carried out by N.Barbian (2003). It was found that the dynamic stability factor decreases as the airflow rate increases. At low frother concentration, Σ is lower than at higher frother concentration, however as the airflow rate is increased the value of Σ rapidly decreases. This implied that the operating variables can significantly influence froth stability; Σ is affected by airflow rate and frother concentration. Together with the result which was achieved from the froth growth and the maximum equilibrium height measurement, it was also found that dynamic stability factor were used to predict the expected fraction of air overflowing below the maximum froth height.[7]

It is necessary to note that in some articles, the Froth retention time (FRT) is used to describe the average lifetime of an air bubble in the froth system, from mathematic point of view FRT is the same as Σ .

Froth retention time defined as $FRT = \frac{Hmax}{Jg}$ by Laplante (1983), where H_{max} is the froth depth and Jg is the superficial gas value it.

froth depth and Jg is the superficial gas velocity.

The froth retention time is physically a measure of the average lifetime of an air bubble in the froth system. For a given froth the longer the froth retention time FRT is, the greater the probability for an attached particle to be dropped back into the slurry is. The increase of water drainage and reduce of entrainment, which is often desirable, take place when the froth height is increased. On the other hand, the froth recovery of the valuable minerals may also be reduced.

A more accurate definition of the mean froth retention time was given by Zheng (2006), who consider that: a) not all of the volume of the froth is occupied by air, and b) due to bubble bursting on the surface, only a fraction of the total airflow introduced to the cell reports to the concentrate.[9]

Air recovery

Air recovery, which is the fraction of air, flew into a flotation cell that overflows the cell lip as unburst bubbles. The air recovery is, in general, surprisingly low, and values greater than 50% are rarely observed.

Air recovery empirically determines the balance between the valuable particle recovered by attachment to the lamellae and those recovered from the Plateau borders. The air recovery determines, to a large extent, water recovery, which is related to gangue recovery.

The air recovery, α , is the fraction of air overflowing the weir that entered the cell: $\alpha = Q_{air, \text{ concentrate}}/Q_{air, \text{ pulp}}$ $Q_{air, \text{ pulp}}$ relates to the airflow rate which is set for the experiment, $Q_{air, \text{ concentrate}} = u_{froth} * H_{froth}$

Half life time $t_{1/2}$

Half life time is a characteristic time is used to describe froth stability. It is the time taken for the foam to drop to one half of its initial height(Hmax) when the gas flow is discontinued. In addition to the factor to determine the froth stability, it is also a parameter for a model for decay, $t_{1/2}$ together with α is crucial parameters to determine froth decay. It is described in the study of Zanin(2008) as : $\frac{H}{H_{max}} = \frac{1}{2} - \alpha *$

 $ln \underbrace{t}_{t_{1/2}}^{t}$, this model fitted the experimental data they gathered from a plant. [9]

2.6 Iron ore

2.6.1 Magnetite

 Fe_3O_4 , is exactly the same name says a magnetic ore. Magnetite is a member of the spinel group. This group has the general formula AB_2O_4 . A and B usually represent different metal ions but in this case A is Fe^{2+} and B is Fe^{3+} . This arrangement is called an inverse spinel and causes a transfer of electrons between the different irons in a structured way. It is this "electricity" that causes the magnetic field that is characteristic for magnetite.

Magnetite is a common mineral found in most types of igneous rocks. The largest magnetite deposits in the world are found in northern Sweden.

Magnetite is black with a metallic luster. The crystal system is isometric. The crystal usually forms as octahedral shape.

The best way of recognizing this ore is by the magnetism, thereafter by its streak and by its crystal habit.

2.6.2 Silica

Silica, also known as silicon dioxide, is an oxide of silicon with the chemical formula SiO_2 . Since antiquity, it has been known for its hardness. In nature, silica is most

commonly found as sand or quartz. There are three stable forms of silica structure, i.e., quartz (stable below 870 °C), tridymite (870-1470 °C), and cristobalite (above 1470 °C). The Si-O bonds possess a considerable degree of covalent character, with oxygen atoms arranged tetrahedrally around Si in such a manner that every oxygen atom is shared by two SiO₄ tetrahedra. [1]

2.7 Experimental design

The method will give an organized approach of a set of experiments and it helps to see interactions between the factors are one of the biggest benefits with experiment design. This is a very effective method to reach a better result, if there is more than two variables, probably with fewer trials than there would have been otherwise. Therefore experimental design is often used in optimization work where the experimental cost otherwise would be very high.

General

There are three objectives in optimization work where experimental design could be used: screening, optimization and robustness testing. Three main problems that often occur in those kinds of works are coped with by the method:

- $\diamond\,$ How to monitor a systems where many factors simultaneously influence the system
- ♦ Separation of real effects and noise
- ♦ Producing reliable maps

A good experimental design will give a set of experiments where all relevant factors are varied simultaneously and this set of experiments should of course be representative for the given question.

Create a standard representative experiment, a centre point, and then perform other representative experiments that lie symmetrically around this standard is one way to look at the experimental design. This gives a square pattern. If there are more than three factors to change, it will be a more complex pattern. One of the most important things with experimental design is that these trials should be performed randomly. [19]

3. Experiments

This work was focused on the different parameters which influence the froth stability. The experiment is launched by changing the stirring rate, airflow and the collector concentration respectively with the other two parameters constant. It is important to note that electrolyte is also one of the significant parameters for froth stability. In this work the electrolyte remained the same in all tests.

3.1 Parameters

Process water:

For all mineral flotation, the electrolytes in the process water are different. In this work electrolytes were added in the water to simulate the process water in a plant. The table shows the concentrations under which experiments were carried out. All salts are held high quality.

	pН	Ca	Mg	SO_4	C1	HCO3
		mg/1	mg/1	mg/1	mg/1	mg/1
process water	8	600	50	1900	530	50

Table 2: Salt type and concentration of the process water

Collector:

An Etherdiamine is used in this work. The chemical structure of this Alkyl etherdiamine is R-O-(CH2)3-NH-(CH2)3-NH2 with isotridecyl as alkyl chain.

Ore sample:

The iron ore used in this work is the magnetite, 56% Fe, 16 % acid insoluble (the amount of silicates is measured as acid insoluble) Particle size: K80 = 34,5 μ m (K80 means that 80% are smaller than 34,5 μ m).

3.2 Procedure:

General:

Every single test is run by the same procedures below with different parameters setting. This froth test instrument is similar to many froth studies in laboratory scale. The flotation cell with the mechanical stir at the bottom in the froth test is a rebuilt open-top flotation machine.

- ♦ Collector solution preparation: make 1.0 wt% solution
- $\diamond~$ The solution is stirred with magnetic stirrer for 30 minutes in order to fully mix the collector with water
- ♦ Airflow regulation: set the airflow to the experimental request every time before each test
- Preparation of the flotation cell: add 644g ore and the process water up to 1.3l, set the stir rate and stir the dispersion for 2 minutes before adding the certain

amount of collector. Stir the dispersion for 2 minutes after the collector is added.

♦ Build up the froth column and place the measuring ruler tape on the top of it as the figure below.



Figure 12: Picture of the whole experimental set

- ♦ The airflow and the camera, the camera is set to take a picture of the froth column every 20 second.
- → Turn off the air when the froth has reached its first maximum height, wait a couple of minutes to allow the froth collapse without any external disturbance. The
- Spray tap water on the top of the froth in order to get faster decay of the froth.
 The water is sprayed approximately every 2 minutes, twice or three times depends on the froth height of self decay.
- ♦ Stop the test when the froth approximately decayed to the interface level between pulp and bubbles
- ♦ Data reading from the pictures taken by the camera

Specific:

- 1. Froth behavior under the change of stirring rate with 1l/min airflow, and collector concentration of 144g/ton.
 - ♦ Stir set: 650rpm, 900rpm, 1050rpm and 1200rpm
- 2. Froth behavior under the change of stirring rate with 3l/min airflow, and collector concentration of 144g/ton.
 - ♦ Stir set: 650rpm, 900rpm, 1050rpm and 1200rpm
- 3. Froth behavior under the change of airflow with 900rpm stirring rate, and collector concentration of 144g/ton.

- Airflow set: 0.5I/min, 1l/min, 1.5I/min, 2I/min, 2.5I/min, 3I/min and 5I/min
- 4. Froth behavior under the change of collector concentration with 900rpm stirring rate, and1l/min airflow.
 - ♦ Concentration set: 36g/ton, 72g/ton, 144g/ton and 288g/ton
- 5. Reducibility set: collector concentration 144g/ton
 - ♦ 650rpm, 1l/min; 900rpm, 1l/min, 900rpm, 3l/min, 1200rpm, 1l/min and 1200rpm, 3l/min

It should be mentioned,

- ☆ The dry weight of every ore sample, which is used in froth test, is 644g, and the content of solid-in-pulp is 35.5wt%.
- ♦ When running the experiment with constant collector concentration, 144g/ton of collector is used as standard dosage in this work. This is because in the flotation test, the target 4% acid insoluble in the magnetite concentrate is reached at 144g/ton of collector. Acid insoluble is an indirect factor to show gangue content in magnetite concentrate.
- ♦ Because 900rpm has longer froth equilibrium stage, this stirring rate is chosen as the standard stirring rate for the change of airflow rate and concentration.

3.3 Analysis:

Froth height

This is the most direct data we can get from the pictures taken by the camera, and is also the y axis factor in the froth behavior diagram.

Froth growth velocity u (cm/s)

This is measured from the maximum slope in the froth growing part in the diagram.

Froth retention time (s)

Besides froth height and growth velocity, the froth retention time (FRT) is a factor used in this work

$$FRT = \frac{Hmax}{Jg}$$

FRT is calculated by knowing the froth height at equilibrium from the diagram.

Froth decay rate v (cm/s)

This factor is achieved from the fitting line fitted out from the point when the airflow is turned off to the end of the test, during the time water is sprayed on the top of the froth with the amount 11-12 g three times.

Air recovery α (%)

Air recovery has been recognized empirically as determining the balance between the valuable particle recovered by attachment to the lamellae and those recovered from the Plateau borders. $\alpha = Q_{air, concentrate}/Q_{air, pulp} = Jg/u$

3.4 Experimental design

In this work a computer program called MODDE which is invented by Umetrics is used to do the experimental design.

Design set up

To begin the design some input conditions have to be defined. There has to be a number of different factors, for example stirring rate and airflow, and in which range they should be varied or if they are uncontrolled. Also the number of responses and their units has to be specified. The experimental data was putted into MODDE. Each experiment will give a result (a response) that will be analyzed by regression and a model will be given where the changes of factors will be related to the changes in responses. This model will indicate which factors that is most important for the optimization and how they should be combined to get the desired results.

In this work, the external influencing parameters for froth stability, which in this case are four different stirring rates and seven different airflows, maximum height and growth velocity, are studied by the help of MODDE.

Factors: Stirring rate (experimental set, controlled, 650, 900, 1050, 1200) [rpm] Airflow (experimental set, controlled, 0.5, 1, 1.5, 2, 2.5, 3, 5) [I/min]

Response: Maximum height (cm) Growth velocity (cm/sec) Air recovery (%) Decay velocity (cm/sec)

4. Result and discussion

4.1 Reproducibility

The figures below show the repeatability of the froth test. As it is shown, the growth velocity has high reproducibility, froth height has fare reproducibility, however, the decay velocity is not controllable, that might be because it is hard to control the water amount sprayed on the surface of the froth every time.



Figure 13: Repeatability of the froth with 650rpm stirring rate and 11/min airflow



Figure 14: Repeatability of the froth with 900rpm stirring rate and 31/min airflow



Figure 15: Repeatability of the froth with 1200rpm stirring rate and 11/min airflow



Figure 16: Repeatability of the froth with 1200rpm stirring rate and 3l/min airflow

4.2 Influencing parameters on froth stability



4.2.1 Influence of the stirring rate

Figure 17: Froth behavior with different stirring rate at 11/min airflow

The figure 17 shows the result from the influence of stirring rate 650 rpm, 900 rpm, 1050 rpm and 1200 rpm on the froth stability with the airflow of 1 l/min and collector concentration 144g/ton. For all the stirring rates, they had almost linear growth after 60 seconds up to the equilibrium height. 650 rpm and 900 rpm respectively 1050 rpm and 1200 rpm have similar growth rate. However, the difference between two rates is not so obvious. The equilibrium heights (maximum height) are among the same level for all stirring rates. The froth of 1200 rpm didn't behave like others. It had an abrupt drop right after achieving the maximum height. However for the stirring rates which are lower than 1200rpm, when the froth height slight froth height fluctuations. The decay stage is quite difficult to interpret from this diagram, because of the different time of the water spray, and the different bubble coalescence for different stirring rate.



Figure 18: Froth behavior with different stirring rate at 3 l/min airflow

The figure 18 shows the result from the influence of stirring rate 650 rpm, 900 rpm, 1050 rpm and 1200 rpm on the froth stability with the airflow of 3 l/min and collector concentration 77 mg/l. As it seems in the figure for 3 l/min, the froth had a linear growth before it came to the equilibrium height. The differences of growth velocity and maximum height for the different stirring rates were quite obvious. The growth velocity and the maximum height decrease as the stirring rate decreases.

Besides maximum froth height and growth velocity, there are other froth stability factors which are also valuable for froth stability evaluation. Froth retention time

which represents the life time of a bubble in the froth, is defined as $FRT = \frac{Hmax}{Jg}$, Jg is the superficial gas velocity. Air recovery α (%), $\alpha = Q_{air, concentrate}/Q_{air, pulp} = u/Jg$

The tables 3 and 4 are a quantitative comparison of the results of different stirring rate in 1 l/min and 3 l/min

	Hmax(cm)	Jg(cm/s)	FRT(s)	u(cm/s)	v(cm/s)	α (%)
stirring rate	maximum froth height	superficial gas velocity	froth retension time	froth growth velocity	decay velocity	air recovery
650	29.3	0.11	266.3636364	0.064	0.021	19.09%
900	30.6	0.11	278.1818182	0.066	0.031	28.18%
1050	29.5	0.11	268.1818182	0.075	0.032	29.09%
1200	30.5	0.11	277.2727273	0.074	0.047	42.73%

Table 3: Froth stability factors for different stir rate at 11/min, collector concentration of 144 g/ton

	Hmax(cm)	Jg(cm/s)	FRT(s)	u(cm/s)	v(cm/s)	α (%)
stirring rate	maximum froth height	superficial gas velocity	froth retension time	froth growth velocity	decay velocity	air recovery
650	36.7	0.33	111.2121212	0.096	0.053	16.06%
900	42.9	0.33	130	0.147	0.04	12.12%
1200	46.5	0.33	140.9090909	0.194	0.127	38.48%

Table 4: Froth stability factors for different stir rate at 31/min, collector concentration of 144 g/ton



Figure 19: Variation in maximum height for different stirring rate and airflow



Figure 20: Variation in growth velocity for different stirring rate and airflow

Figure 19 and 20 are plotted out from the table 3 and table 4. It shows directly the distribution and the variation trends of the stability factors between 1 l/min and 3 I/min. Figure 19 and 20 shows the height and growth velocity distribution for different stirring rate at different airflow respectively. The reason that these two factors at 3 I/min are higher than the ones at 1 I/min is: the higher the airflow is, the larger the amount of bubbles which build up the froth are in the system. For 3 l/min, these two factors are ascending as the increase of the stirring rate. This is because the higher the stirring rate, the smaller the bubbles becomes. Consequently, there are more surface area for the particles to load on, in turn the froth can be stabilized more easily. From the distribution for the factors at 1 l/min, it is reasonable to conclude the stirring rate is not the dominant parameter for the froth stability when the airflow is low. This might be because the bubble amount is the crucial parameter for particle loading when the air content is low. Even though increasing the stirring rate can increase the total surface area of the bubbles, the amount of particles that can load on to the bubble surface depends on the bubble amount instead of the bubble surface area. It is acceptable to presume there is a critical airflow rate, which is the watershed of these two influencing mechanism.



Figure 21: Variation in decay velocity for different stirring rate and airflow

Figure 21 shows the decay velocity (v) distribution for different stirring rates at different airflow. As be seen from the figure, the froth at 3 l/min has higher decay rate than the froth at 1 l/min. That is because of that the bubbles are bigger at 3 l/min in comparison with the ones at 1 l/min. That consequently influences the bubble coalescence and collapse with the disturbance of water on the top of the froth in the column, another reason is that for lower airflow, the particle content in the froth is low, that gives a less stable froth, and the bubbles on the surface are easier to coalescence and collapse.

It is necessary to note that for the reason that 900rpm has longer froth equilibrium stage; this stirring rate is chosen for the later airflow rate and concentration change.

4.2.2 Influence of the airflow



Figure 22: Froth behavior of different airflowat 900rpm stirring rate

Figure 22 shows the froth behavior of the different airflow under the stirring rate of the 900 rpm and collector concentration of 144 g/ton. Distinct growth velocity variations are revealed up to 3 l/min. From 0.5 l/min to 3 l/min, the growth velocity accesses as the airflow increases, until it comes to 3 l/min. The maximum height also shows the similar behavior, as the airflow rise up to 2 l/min, the maximum height increase. However, when the airflow is higher than 2 l/min, it remains on the similar level for all airflows.

Table 5 shows a quantitative comparison of the results of different airflow rate in 900rpm, and also these are the initial data to draw the diagrams below.

air flow	Hmax(cm) maximum froth height	Jg(cm/s) superficial gas velocity	FRT(s) froth retension time	u(cm/s) froth growth velocity	v(cm/s) decay velocity	α (%) air recovery
0.5	22.4	0.055	407.2727273	0.039	0.012	21.82%
1	30.6	0.11	278.1818182	0.066	0.031	28.18%
1.5	34	0.17	200	0.086	0.031	18.24%
2	40.9	0.22	185.9090909	0.098	0.041	18.64%
2.5	44.1	0.27	163.3333333	0.126	0.031	11.48%
3	42.9	0.33	130	0.147	0.04	12.12%
5	44.8	0.55	81.45454545	0.164	0.058	10.55%

Table 5: Froth stability factors for different airflows at 900rpm stirring rate



Figure 23: Variation in maximum height with different airflow at 900 rpm



Figure 24: Variation in growth velocity with different airflow for 900 rpm



Figure 25: Variation in decay velocity with different airflow for 900 rpm

Figures 23 and 24 verify the results from figure 22. This phenomenon is explained by the air content increase in the froth when the airflow rises up. More bubbles exit in the system which leads to more hydrophobic particles attached to the lamellae, also gangue particles reside in the plateau. This can make the froth more stable. However, when the amount of bubbles grows up to a certain number, in this case at 3 l/min, the growth velocity is not increasing any more. This is predicted by knowing that the particles content is constant, even though higher airflow rate gives a higher amount of bubbles. The froth height comes to a certain level when the airflow rate rises up to 2l/min. This is also due to the same reason.

Figure 25 shows the decay velocity with the change of airflow. As it is shown in the figure, decay velocity increase with the increase of airflow rate.

4.2.3 Influence of collector concentration



CMC of the collector:

Figure 26: CMC-measurement, for etherdiamine in process water

As shown in figure 26, the CMC of etherdiamine in process water is 0,065 g/l of water, which is approximately 130g/ton of ore.



Figure 27: Froth behavior of different collector concentration at 1 l/min airflow and 900 rpm stirring rate

The figure 27 shows the froth behavior under the change of collector concentration at 900 rpm stirring rate, and 1 l/min airflow. With the increase of the collector concentration, the maximum height increases. However, the growth velocity is not influenced too much. This is explained by the fact that the airflow is constant, the bubble amount is constant consequently. Nevertheless, the higher the collector concentration is, the more particles surface can be changed into hydrophobic, and in turn the more particles will attach to the air bubbles and can build up the froth to a higher level. Also table 5 shows that higher the concentration of collector in the system, longer the single bubble life time will be, as shown in the table below, the FRT is longer, with the increase of collector concentration that gives higher maximum froth height.

Table 6 shows a quantitative comparison of the results of different collector concentration in 900 rpm and 1 l/min airflow.

1l/min	Hmax(cm)	Jg(cm/s)	FRT(s)	u(cm/s)	v(cm/s)	α(%)
36 g/ton	17	0.11	154.5455	0.082	0.043	0.745455
72 g/ton	27.2	0.11	247.2727	0.083	0.066	0.754545
144 g/ton	30.6	0.11	278.1818	0.066	0.031	0.6
288 g/ton	46.8	0.11	425.4545	0.081	0.024	0.736364

Table 6: Froth stability factors for different collector concentration at 11/min airflow and 900rpm stirring rate



Figure 28: Froth behavior of different collector concentration at 3 l/min airflow and 900 rpm stirring rate

Figure 28 shows similar result as for 1 l/min, the growth velocity remains unchanged when the collector concentration is increased. However, the maximum height is increased.

Table 7 is a quantitative comparison of the results of different collector concentration at 900 rpm and 3 I/min airflow. Together with the table above, the below diagram is plotted.

3l/min	Hmax(cm)	Jg(cm/s)	FRT(s)	u(cm/s)	v(cm/s)	α(%)
36 g/ton	26.5	0.33	80.30303	0.227	0.09	0.272727
72 g/ton	37.7	0.33	114.2424	0.193	0.057	0.172727
144 g/ton	42.9	0.33	130	0.147	0.04	0.121212
288 g/ton	69	0.33	209.0909	0.279	0.061	0.184848

Table 7: Froth stability factors for different collector concentration at 3 l/min airflow and 900 rpm stirring rate

max height (cm) 1l/min 31/min collector concentration (g/ton)

Figure 29 and 30 verify the result from figure 28.







Figure 30: Variation in growth velocity for different collector concentration and airflow

Figure 31: Variation in decay velocity for different collector concentration and airflow

4.3 Experimental design by MODDE software

As previously stated, these experiments were done at a dosage of 144 g/ton. The main responses that were looked at were:

- i. Maximum height (cm)
- ii. Growth velocity (cm/s)
- iii. Air recovery (%)
- iv. Decay velocity (cm/s)

The results from these experiments are presented in table below

Fac	Factors: 3 (uncontrolled: 1) Responses: 4 Runs: 18 Objective: RSM CCF design, quadratic model Fitted with MLR 🖕											
	1	2	3	4		5	6	7	8	9	10	11
1	Exp No	Exp Name	Run Order	Incl/Ex	cl	Rotor speed	Air flow	Time	Max height	Growth	Growth/Nominal Growth	Decay
2	1	N1	15	Incl	•	650	1		30,5	0,075	69,237	0,02
3	2	N2	16	Incl	•	1200	1		28,5	0,1	92,316	0,101
4	3	N3	24	Incl	Ŧ	650	3		36,7	0,107	32,926	0,053
5	4	N4	1	Incl	•	1200	3		47	0,217	66,7752	0,133
6	5	N5	13	Excl	•	650	2					
7	6	N6	17	Excl	•	1200	2					
8	7	N7	14	Incl	•	900	1		30,6	0,077	71,0833	0,031
9	8	N8	12	Incl	•	900	3		40,2	0,158	48,6198	0,049
10	9	N9	9	Incl	•	900	2		40,9	0,111	51,2354	0,041
11	10	N10	5	Incl	•	650	1		30,9	0,075	69,237	0,021
12	11	N11	21	Incl	•	1200	1		30	0,11	101,548	0,091
13	12	N12	18	Excl	•	650	3					
14	13	N13	6	Incl	•	1200	3		44,5	0,2	61,544	0,023
15	14	N14	22	Excl	•	650	2					
16	15	N15	3	Excl	•	1200	2					
17	16	N16	26	Incl	•	900	1		30,1	0,079	72,9296	0,034
18	17	N17	23	Incl	•	900	3		44,3	0,15	46,158	0,049
19	18	N18	2	Incl	•	900	2		38,6	0,147	67,85	0,02
20	19	N19	10	Incl	•	650	1		32,3	0,075	69,237	0,022
21	20	N20	8	Incl	•	1200	1		30,5	0,105	96,9318	0,047
22	21	N21	20	Excl	•	650	3					
23	22	N22	11	Incl	•	1200	3		46,5	0,213	65,5444	0,127
24	23	N23	19	Excl	-	650	2					
25	24	N24	27	Excl	•	1200	2					
26	25	N25	25	Incl	•	900	1		30,5	0,078	72,0065	0,006
27	26	N26	7	Incl	-	900	3		44,3	0,134	41,2345	0,024
28	27	N27	4	Excl	Ŧ	900	2					

Figure 32: Trial details of experimental design by MODDE

The trial number which was designed by the MODDE is 27. In this table, 18 results are presented due to the lack of time of the repetitive tests for some parameters, and also some tests with unstandarded water spray results are excluded. However, this would not modify the accuracy of the model. The purpose to use MODDE in this work is to perceive the trend of froth stability changing with the two parameters.



Figure 33: A summary of the basic model statistics in four parameters; 1 is perfect = 100%. Figure 33 shows the sumary of the basic model statistics in four parameters as shown below:

R2 shows the model fit; 0.5 is a model with rather low significance.

Q2 shows an estimate of the future prediction precision. Q2 should be greater than 0.1 for a significant model and greater than 0.5 for a good model.

Model validity is a test of diverse model problems. A value less than 0.25 indicates statistically significant model problems, such as the presence of outliers, an incorrect model, or a transformation problem.

Reproducibility is the variation of the replicates compared to overall variability. A value greater than 0.5 is wanted.

Correct model tuning like removing non significant model parameters or selecting the appropriate transformation results in higher summary statistics. The best and most sensitive indicator is Q2.

Note: Model validity might be low in very good models (Q2 > 0.9) due to high sensitivity in the test or extremely good replicates.



Figure 34: Maximum height of froth (MODDE)

Figure 34 shows the maximum froth height with the influence of the airflow and the stirring rate. The froth height is not changed with the increment of the stirring rate when the airflow is below 2 l/min. When the airflow is above this number, the stirring rate starts to affect the maximum froth height. But it only plays a minor role as it is shown in curvature in the figure, only for the lowest and highest stirring rate, the maximum height is varied with airflow. The maximum froth height is achieved when both the airflow and the stirring rate is the highest, as it is in the red region.



Figure 35: Growth velocity of the forth (MODDE)

Figure 35 shows the growth velocity under the change of airflow and stirring rate. Unlike the maximum froth height, the growth velocity is influenced by both the

airflow and the stirring rate. The froth grows faster when both the airflow and the stirring rate are high.



Figure 36: Air recovery of froth (MOODE)

Figure 36 shows the air recovery, which is the fraction of air that flow into the cell and stay as unburst bubbles. The lower airflow and higher stirring rate can give a larger air recovery. Increasing the airflow the air recovery decrease, this must because the particle and collector content is constant in the cell, the excessive bubble couldn't be stabilized and collapse. The air recovery is ascending with the increase of stirring rate is due to the fact increasing the stirring rate, the bubble can break down into smaller size and more surface area for the particles to reside, in turn more air bubbles can be stabilized in the froth with little collapse. Consequently the surface area is bigger, and more particles can stay on and stabilize the froth.



Figure 37: Decay velocity of froth (MODDE)

Figure 37 is the last one from MODDE and it shows how the decay velocity is influenced by the airflow and the stirring rate. The froth can decay faster when both the airflow and the stirring rate is higher, so the froth system is less stable and easier to disturb by spraying water onto the froth surface in the froth column.

6. Conclusion

Flotation process is a huge and complex area of research. The chemistry behind this process is extremely complicated. Froth as discussed earlier play a tremendous role in flotation. A lot of research has been conducted to study its effect on the flotation results, especially the froth stability attracts an abundant amount of attention from the researchers.

This work shows that stirring rate, airflow and collector concentration all can influence the froth stability.

The repeatability tests of some of the parameters show good repeatability of the growth velocity, moderate repeatability of the maximum height, but repeatability of the decay velocity is below the average.

For stirring rate, the froth stability, both the maximum height and growth velocity, depends on the airflow. At low airflow, the stirring rate is not the dominant parameter. The froth stability doesn't change along with the stirring rate. That is because in this case, the amount of air bubbles is of importance instead of surface area of the bubbles and also the amount of the hydrophobic particles is constant, the amount of particle stabilized bubbles are the same in different stirring rates. Also the air recovery is constant, means the amount of unburst bubble in the column is constant, which is another explanation of this fact. When the airflow exceeds the critical value, which is 2l/min predicted by MODDE, the stirring rate starts to affect the froth stability. The higher the stirring rate is, the higher the maximum height and the faster the growth velocity is. This is because in this case the surface area of the bubbles are, and consequently larger the surface area of the bubbles in the cell. That can attract more hydrophobic particles, therefore the froth is more stable.

Airflow can affect both the maximum height and the growth velocity. They both increase, when the airflow is increased. That is because the amount of the bubbles increase as the airflow is increased, and more hydrophobic particles can load on to the surface of the bubbles. However, when the airflow comes to a certain number, the maximum height and the growth velocity will not change with further increase of the airflow and achieve peak values, respectively. That is because the constant concentration of the collector in different airflow changing lead to the constant amount of hydrophobic particles in the cell. There would not be enough particles to stabilize the excessive bubbles, which would coalesce and collapse in the system.

The collector concentration can significantly influence the froth height, but not so much on the growth velocity. The froth height can increase along with the increment of the collector concentration. Nevertheless, the grow rate remains at the same level with the change of the collector concentration. This is explained by the fact that the

airflow is constant, the bubble amount is constant consequently. Nevertheless, the higher the collector concentration is, the more particles surface can be changed into hydrophobic, and in turn the more particles will attach to the air bubbles and can build up the froth to a higher level.

The experimental design by MODDE software shows that the experimental results fit the model very well and the prediction out of MODDE is trust worthy. Some of the prediction of the MODDE can verify the results of the experiments.

The prediction of the MODDE shows both the maximum height and growth velocity come to the peak value when the airflow and the stirring rate are the highest out of the experimental parameter set up. But for air recovery the peak value is in the region where the highest stirring rate and the lowest airflow present. This is because the hydrophobic particles content wouldn't change with the increasing of the amount of the bubble when the airflow is increased. The excessive bubbles would not be stabilized by the particles and collapse, consequently there would be smaller amount of unburst bubbles in the system. The higher the stirring rate is, the larger the surface area of the bubbles would be, and more hydrophobic particles would attach to the surface, and preventing the bubbles to collapse. MODDE also give a well prediction of the decay velocity.

6.Reference

- [1] Rao, S. (2004) *Surface Chemistry of Froth Flotation*. Second Edition. New York: Kluwer Academic/ Plenum Publishers.
- [2] Holmberg, K. (2003) Surfactants and polymers in aqueous solution. Second Edition. England: John Wiley & Sons Ltd.
- [3] Fuerstenau, M., Jameson G. and Yoon R. (2007) *Froth Flotation A Century of Innovation*. USA: Society for Mining, Metallurgy, and Exploration, Inc.
- [4] Gence, N. (2005). Wetting behavior of magnetite and dolomite surfaces. Applied Surface Science, vol 252, pp. 3744-3750.
- [5] Farrokhpay, S. (2011). The significance of froth stability in mineral flotation-A review. *Advances in Colloid and Interface Science*, vol 166, pp. 1-7.
- [6] Ata, S. (2012). Phenomena in the froth phase of flotation-A review. *International Journal of Mineral Processing*, vol 102-103, pp. 1-12.
- [7] Barbian, N., Ventura- Medina E., Cilliers, J.J. (2003). Dynamic froth stability in froth flotation. *Minerals Engineering*, vol 16, pp. 1111-1116.
- [8] Technological Resources PTY Limited, University of Manchester Institute of Science and Technology (2004) *Measuring Froth Stability*. WO 2004/080600 A1.
- [9] Zanin, M., Wightman, E., Grano, S.R., Franzidis, J.-P. (2009) Quantifying contributions to froth stability in porphyry copper plants. *Int. J. Miner. Process.*, vol 91, pp. 19-27.
- [10] Barbian, N., Hadler, K., Ventura-Medina, E., Cilliers, J.J. (2004) The froth stability column: linking froth stability and flotation performance. *Minerals Engineering*, vol 18, pp. 317-324.
- [11] Tao, D., Luttrell, G.H., Yoon, R.-H. (2000) A parametric study of froth stability and its effect on column flotation of fine particles. *Int. J. Miner. Process.*, vol 59, pp. 25-43.
- [12] Barbian, N., Hadler, K., Cilliers, J.J. (2006) The froth stability column: measuring froth stability at an industrial scale. *Minerals Engineering*, vol 19, pp. 713-718.
- [13] Ventura-Medina, E., Cilliers, J.J., (2002) Amodel to describe flotation performance based on physics of foams and froth image analysis. Int. J. *Miner. Process.*, vol 67, pp. 79-99.
- [14] Manser, R.M. (1975) Handbook of Silicate Flotation, Warren Spring Laboratory.
- [15] Dingeman, D. (1991) *How Froth Flotation Works-Practical Side*, Oriox Technologies, Inc.
- [16] Zachwieja, J.B., (1993) An Overview of Cationic Reagents in Mineral Processing, Akzo Chemicals, Inc. New York: Dobbs Ferry.
- [17] Iwasaki, I. (1983) Iron Ore Flotation, *Mining Engineering*, pp. 622-631.
- [18] Kremidas, J.P. (1977) *Fundamental Concepts of the Mineral Flotation Processes*, Armak Company, Minerals Processing Laboratory.
- [19] Eriksson, E., Johansson, E., Kettaneh-Wold, N., Wikström and Wold, S. (2000), Unimetrics Academy, ISBN: 91-973740-0-1.
- [20]<u>http://www.substech.com/dokuwiki/doku.php?id=stabilization_of_colloids&Dok_uWiki=1a7660dced264b04eed3e7f</u>