EFFECT OF PULP RHEOLOGY ON FLOTATION: THE NICKEL SULFIDE ORE WITH ASBESTOS GANGUE SYSTEM

by

ISIL KILICKAPLAN

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ABSTRACT

Pulp rheology is a sensitive indicator of the state of aggregation/dispersion of mineral particles. In this study, possible correlation between the rheological properties of flotation pulp and the flotation response was investigated through a series of rheological and batch flotation tests.

The nickel-sulfide ore from the Mt. Keith plant (Western Australia) was used for the tests. It is a low-grade nickel sulfide ore which is characterized by complex mineralogical composition with a large content of serpentine minerals.

The experimental program included agglomerate flotation tests and rheological tests with the use of a slurry produced by grinding the nickel-sulfide ore. In order to see the effect of solids content on flotation, a series of tests were performed at various pulp densities in the absence and presence of oil in both conditioning and flotation stages. The agglomerate flotation and rheological tests were repeated with dispersants in order to investigate the correlation between degree of agglomeration/dispersion and flotation response.

The agglomerate flotation tests conducted at various solids content in the flotation stage revealed that the fastest flotation rate was observed at the lowest solids content. The entrainment was found to increase with increasing solids content due to elevated pulp viscosity. The “boiling” was observed in the flotation cell in the tests at a high pulp density, the effect likely associated with a high pulp viscosity. High pulp viscosity is clearly increasing the size of bubbles in the flotation system. In line with this the high recoveries by entrainment are observed in the tests at high pulp density. Under such conditions the concentrate grade was low since a large proportion of the nickel recovery resulted from entrainment. In distilled water the use of a dispersant improved considerably flotation performance.

The presence of chrysotile in the system strongly affects the pulp viscosity. In the tests with pure chrysotile the pulp with a solids content of around 0.8% by volume is close to the critical packing fraction. However, in the tests with the nickel ore containing more than 80% of chrysotile the viscosity is still far from the critical packing even at 7.6 % solids content by volume. This shows a very beneficial dilution effect; the presence of other non fibrous minerals dramatically decreases the viscosity of the chrysotile suspensions. Without such minerals processing of this ore would have been entirely impossible.
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1. INTRODUCTION

This thesis is based on the assumption that the low recoveries of pentlandite in the slime flotation circuit of Mt. Keith (Western Australia) plant result from the fine sizes of the particles and the interactions between this mineral and serpentine particles (which includes fibrous minerals like chrysotile and lizardite). To better understand such interactions rheological tests were carried out in parallel with batch flotation tests, and the objective of the thesis was to study whether rheological methods could be used to improve our understanding of such flotation systems.

The experimental program of this thesis was split into three parts. In the first part the flotation and rheological tests were carried out with the use of the nickel sulphide ore from Western Australia. Since it was found that agglomerate flotation provided better results more detailed rheological tests were needed. The second part of the experimental program involved examination of the correlation between the degree of agglomeration/dispersion and flotation response through oil agglomeration and slimes dispersion with polymers. Carboxymethyl cellulose (CMC) was used as a dispersant to eliminate the possibility of heterocoagulation and formation of slime coating in the flotation tests. Tests with CMC were conducted with both process water and distilled water. By monitoring both the concentrate yield and water recoveries it was possible to calculate the true flotation and the flotation by entrainment. In the third part, rheological measurements were conducted to characterize the effect of shearing on the rheology of aggregated pulp. Tests were performed in either the absence or presence of the dispersant in both distilled and process water.
1.1 OBJECTIVE

The primary objective of this project is the investigation of a possible correlation between rheological properties of flotation pulp and flotation response. This study was deemed worthwhile because the studied nickel sulfide ore contains fibrous minerals (e.g. chrysotile) as a main component of the gangue which makes the pulp very viscous even at low pulp densities.
2. LITERATURE REVIEW

2.1 Rheology

2.1.1 Rheology Fundamentals

Rheology is the science of the deformation and flow of matter. In practical terms, it is the study of the relation between the applied stress and the resulting deformation generally represented in the form of flow curves.

Rheology allows a correlation between the observed mechanical behaviour and the structure of the studied material to be tested. Since rheological behaviour of the disperse systems reflects not only the characteristics of the liquid medium in which solid particles are dispersed and the suspended solid particles but also particle-particle interactions, the rheological measurements are often applied to study such interactions.

Slurry rheology is strongly dependent on different slurry properties like particle size, particle size distribution, particle shape, solids content, pulp chemistry and surface properties of the particles. The rheological behaviour of mineral slurries has a significant influence on the processing performance and hence a slight change in any of these slurry properties could have a significant impact on the process such as flotation.

Viscosity is the measure of the internal friction of a fluid. Figure 2. 1 shows a liquid sandwiched between two parallel plates of area A with lower plate held stationary and the upper plate pulled with a force F. The force acting in the x direction per unit area applied to the material is called shear stress, \( \tau \) (units Pascal, Pa=1 N/m\(^2\))

![Figure 2. 1 Illustration of the relationship between applied force per unit area and fluid velocity.](image)

Figure 2. 1 Illustration of the relationship between applied force per unit area and fluid velocity.
According to Newton’s model (Figure 2.1) the relationship between the shear stress and the velocity gradient is given by:

\[ \frac{F}{A} = \eta \frac{dv}{dx} \]  

where \( \eta \) is dynamic viscosity.

The velocity gradient, \( \frac{dv}{dx} \), is a measure of the change in speed at which the intermediate layers move with respect to each other. It describes the shearing the liquid experiences and is thus called shear rate, \( \gamma \) (units reciprocal second, \( \text{sec}^{-1} \)). Newton assumed that all materials have, at a given temperature, a viscosity that is independent of the shear rate. Using these simplified terms, viscosity may be defined by:

\[ \eta = \frac{\tau}{\gamma} = \frac{\text{shear stress}}{\text{shear rate}} \]  

where \( \tau \) is shear stress (Pa) and \( \gamma \) is shear rate (s\(^{-1}\)). The fundamental unit of viscosity measurement is “Poise.” In the SI system, it is expressed in “Pascal-seconds” (Pa.s) or “milli-Pascal-seconds” (mPa.s) (1 cP= 0.001 Pa·s = 1 mPa·s, 1P=100 cP).

For Newtonian fluids, the shear stress plotted versus shear rate, gives a straight line of zero intercept and slope of the line gives \( \eta \). Non-Newtonian fluids display non-linear plots which are given in Figure 2.2.

![Rheological curves for different rheological systems.](image)
2.1.2 Newtonian Fluids

Ideal liquid-like behaviour is described as Newtonian behaviour and in that case the applied shearing stress is directly proportion to the rate of shear (Figure 2.3 A). Figure 2.3 B shows that the fluid’s viscosity remains constant as the shear rate is varied; viscosity is independent of how fast the liquid is being sheared (provided that the shear rate is still within a laminar flow range).

![Figure 2.3 Newtonian fluid behaviour](image)

2.1.3 Non-Newtonian Fluids

In the case of a Newtonian fluid such as water, gases, hydrocarbons, the viscosity is constant throughout the entire shear rate range (laminar flow range). For non-Newtonian systems, shear stress is not linearly proportional to shear rate anymore, the viscosity for this system is called “apparent viscosity (η_a)”. In most cases, the viscosity decreases with increasing shear rate and this is called “shear thinning” behaviour. Figure 2.4 shows the shear thinning behaviour and apparent viscosity which is defined as the slope of a line passing through the origin that intersects the flow curve at a specified shear rate. For pseudoplastic systems which exhibit shear thinning behaviour, viscosity has the highest value at the lowest shear rate and decreases with increasing shear rate values.
2.1.4 Thixotropy and Rheopexy

For sheared systems, the apparent viscosity of a suspension sometimes depends also on the shear rate and the time of shearing. The time-dependent behaviour of the viscosity is related to changes occurring in the inner structure of the fluid (changes in the sizes of the dispersed particles caused by either aggregation or aggregate breaking, bubble coalescence etc) due to particle interaction forces like the van de Waals forces, which are responsible for aggregation (Bekkour. et.al, 2005).

The most common phenomena of this type of behaviour is **thixotropy** which is the property of some non-Newtonian pseudoplastic fluids that show a time-dependent change in viscosity. Thixotropy is defined as the time-dependent decrease in viscosity due to shearing.
Shearing the material at a given shear rate may breakdown the network of aggregating particles gradually with time. After shearing over a long enough period, equilibrium or steady-state curve which is usually pseudoplastic can be obtained.

There is another behaviour which is essentially the opposite of thixotropic which is referred to as **rheopexy**. In this case, the fluid’s viscosity increases with time at a constant shear rate. In such systems the structure can be rebuilt at lower shear rates and thus the viscosity will increase. In other words, thixotropy is time-dependent thinning and rheopexy is time-dependent thickening which is shown in the Figure 2. 6. Both phenomena can be irreversible, reversible or partially reversible.

![Time-Dependent Behavior](image)

**Figure 2. 6** Time dependent behaviour of fluid.

The apparent viscosity is not the only rheological quantity which is related to the strength of the links between particles. The yield stress also implies that the suspension has a structure that must be overcome to initiate flow.

**Yield stress** is a rheological property that is defined as the minimum shear stress corresponding to the first evidence of flow. The presence of the yield stress indicates a network of interacting particles; this structure must be overcome to initiate flow. It is a common property of highly concentrated suspensions containing fine aggregated particles. Such systems are often characterized by a plastic viscosity. In such systems only when the applied stress is higher than yield stress value, the network collapses and the suspension starts flowing. The maximum stress which is just before network collapsing is the yield stress (Scales *et al.*, 1998).
The yield stress is an important rheological parameter in mineral industry. A too high yield stress may result in unnecessarily high power consumption and hence high operating costs (Condoliosand and Chapus, 1963; Thomas, 1977). On the other hand, yield stress measurements can be used to study aggregation/dispersion that occurs due to the inter-particle interactions in the suspension (Schofield et.al, 1954; Nyugen and Boger, 1983, 1985; Johnson et.al, 1999). Flow curves are commonly used to determine the yield stress value.

The estimation of yield stress involves the extrapolation of shear stress-shear rate data corresponding to a zero shear rate using a given analytical rheological models. Bingham and Casson models can often be used to describe the flow behaviour of the suspensions.

2.2 Aggregation Processes

2.2.1 Colloidal Forces

As the particle size decreases, the surface area of the suspended particles increases. There exists a point at which the bulk properties of the dispersed solid become dominated by its surface properties; this is the colloidal state of matter. Colloidal behaviour usually becomes noticeable at particle size range below 1 µm (1000 nm).

Colloidal systems are always thermodynamically unstable and tend to aggregate with time due to their large surface energies. The rate of aggregation can be slowed down when the particles are electrically charged and the resulting repulsive electrical forces are larger than the van der Waals attractive forces.

2.2.1.1 Surface Electrical Charge

An interface can acquire an electrical charge by several possible mechanisms. The most important were set out by Kitchener (1969) in the following way:

a) Ionic Solids: In the case of ionic solids such as AgI, BaSO₄, CaF₂ etc. the surface charge arises from the transport across the interface of ions constituting the lattice. Those particular ions that are free to pass between both phases and therefore establish the electrical charge are called potential-determining ions (PDI). For AgI, the potential-determining ions are Ag⁺ and I⁻, for a solid like calcite, CaCO₃, the potential-determining ions are Ca²⁺ and CO₃²⁻ but since their concentration also depends on pH, the potential-determining ions for this mineral are Ca²⁺, CO₃²⁻ and also H⁺, OH⁻ and HCO₃⁻.
b). Oxides (e.g. SiO$_2$, SnO$_2$, TiO$_2$, Fe$_2$O$_3$, Al$_2$O$_3$, etc.). It is customary to ascribe the origin of the electrical charge at the oxide surface/aqueous interface to protonation/deprotonation of the surface hydroxyls:

\[
\begin{align*}
-MOH + H^+ &= -MOH_2^+ \\
-MOH + OH^- &= -MO^- + H_2O
\end{align*}
\]

As these reactions reveal, H$^+$ and OH$^-$ ions are potential-determining ions for oxides and therefore the electrical charge of oxides depends on pH. Since oxides are amphoteric, their surfaces are variable; they are positively charged at low pH and negatively charged at higher pH values. Zeta potential versus pH values of various oxides minerals under different pH values is shown in Figure 2.7 (Laskowski, 2007). The concentration of potential-determining ions (pH) at which the zeta potential value is equal zero is referred to as the iso-electric point (i.e.p).

![Figure 2.7 Zeta potential versus pH values of oxide minerals at constant ionic strength](image)

**Figure 2.7** Zeta potential versus pH values of oxide minerals at constant ionic strength (after Laskowski, 2007, with the permission of Janusz Laskowski).

c). Alumino-silicates: The best known example in this group is clay minerals (kaolinite, montmorillonite, etc). These minerals have laminar crystal structure.
Montmorillonite is a 2:1 layer mineral composed of an octahedral alumina sheet sandwiched between two silica sheets. As a result of isomorphous substitution of some silicon atoms for aluminum, the face surfaces carry negative electrical charge while $=\text{Al(OH)}$ groups at the edges are either positively or negatively charged depending on pH (see Figure 2.24).

![Figure 2.8 Idealized sketch of the atomic structure of montmorillonite.](image)

**Figure 2.8** Idealized sketch of the atomic structure of montmorillonite.

Kaolinite is a 1:1 sheet silicate composed of a silicate tetrahedral layer and an alumina octahedral layer. Below pH 9 this would thus result in plate-like particles with “top” and “bottom” surfaces carrying different electrical charges.

Clays are typical anisotropic minerals. Also talc belongs to this group (Burdukova, 2007). Chrysotile and lizardite, the main components of the gangue in the sulfide ore from Western Australia studied in this thesis belong to this group (see chapter 2.6.) as well.

**Figure 2.9** Origin of electrical charge at the montmorillonite edge.
(D). Sulphide minerals: Strictly speaking the electrical charge at a sulfide/aqueous solution interface is generated in the same way as in the case of ionic solids (group A). So, for galena, Pb$^{2+}$ and S$^{2-}$ ions should be potential determining. However, as a result of corrosion reactions that take place at the sulfide/water interface in the presence of oxygen, the surface of sulfide may be coated by various oxidation products and this further complicates the possible mechanism of charge generation. It is known that in the case of sulfides, the surface charge depends not only on pH but also on the pulp redox-potential ($E_h$).

2.2.1.2 Electrical Double Layer Interaction

The electrical surface charge influences the distribution of ions in the layers adjacent to the solid surface: while ions of opposite charge (counter-ions) are attracted towards the surface, co-ions are repelled away from the surface. The combined system of surface charge and the excess charge in solution is known as the electrical double layer (Gregory, 2006). Distribution of ions in the electrical double layer is schematically depicted in Figure 2.10.

![Diagram](https://example.com/diagram.png)

**Figure 2.10** Schematic representation of the structure of the electric double layer according to Stern’s theory.

The potential changes from $\Psi_o$ (surface potential) to $\Psi_d$ (Stern potential), and decays from $\Psi_d$ to zero in the diffuse double layer. If it is assumed that the shear plane is located near
the Stern layer the Stern potential can be estimated from electrokinetic measurements and it is customary to assume that $\Psi_d \approx \zeta$.

### 2.2.1.3 Zeta Potential

For most real systems, the surface potential cannot be measured directly and can only be estimated from the measured zeta potential value. Zeta potential is defined as the potential on the shear plane. It is assumed that the electrokinetic plane of shear, where the potential is $\zeta$, coincides with the Stern plane, which represents the closest approach of counter-ions to the surface (Figure 2.10).

If all the particles in suspension have large negative or positive zeta potentials, then they will tend to repel each other and there will be no tendency for the particles to aggregate. However, if the particles have low zeta potential values, the attractive van der Waals forces may be a dominating factor which will result in coagulation.

In commercial zeta-meters, zeta potential is obtained from electrophoretic experiments: it is calculated from the electrophoretic mobility of the tested fine particles in electric field via Smoluchowski equation. As already discussed, there is no a model that can describe the movement of for plate like anisotropic particles in electric field and in such cases the use of Smoluchowski equation must be questioned.

### 2.2.1.4 DLVO Theory

A comprehensive theory of the interaction between colloidal particles was introduced by Derjaguin and Landau (1941) and Verwey and Overbeek (1948). According to DLVO theory of colloid stability, the degree of particle aggregation and dispersion is controlled by repulsive and attractive forces. The total potential energy of the interaction between two particles is given by:

$$V_T = V_E + V_A$$  \[2.4\]

where $V_E$ is the energy of electrostatic double layer repulsion and $V_A$ is the van der Waals attraction. According to this theory an energy barrier resulting from the repulsive forces prevents the two particles from aggregation. However, if the particles collide with sufficient kinetic energy they can overcome that barrier, and the attractive forces will pull them into contact. Therefore, if the particles show a sufficiently high repulsion, the dispersion will be stable and the particles will not aggregate. In the absence of the repulsive forces the particles coagulate.
Different definitions were introduced for coagulation and flocculation in the mineral processing field (these terms are treated as synonyms by colloid chemistry). In the case of the coagulation the particles aggregate due to the neutralization of electrical charge of the interacting particles. When aggregation is induced by a polymer bridging action, the process is referred to as flocculation.

The interaction between two approaching colloidal particles is commonly described by the use of a potential energy diagram. It combines an attractive interaction due to van der Waals forces with repulsions due to electrostatic and/or steric effects. The curve of total potential energy versus inter-particle distance can also be used to describe the stability of the system. The broken curves show the potential energy of repulsion \( V_E \) that is due to the electrical force whilst potential energy of attraction \( V_A \) is the van der Waals attraction force. The full curves are obtained by summing the broken \( V_E \) curve with the \( V_A \) curve. If the zeta potential of the interacting particles is small then the electrical repulsion \( V_E \) (2), is also small, and the total interaction energy \( V_T \) (2) does not show any energy barrier. Coagulation occurs between these particles whenever they collide. The potential energy of \( V_E \) (1) is higher than that of \( V_E \) (2) and the total interaction energy curve for this case shows existence of the energy barrier which will oppose particle-to-particle attachment; the system will be stable if these barriers sufficiently high.

Figure 2. 11 Total interaction energy curves, \( V_T(1) \) and \( V_T(2) \) is the summation of an attractive curve, \( V_A \), with different repulsion curves, \( V_E(1) \) and \( V_E(2) \) (after Laskowski, 2007, with the permission of Janusz Laskowski).
Increased electrolyte concentration causes compression of the double layer and reduction of the zeta potential value. At low ionic strength, the diffuse layers around the particles are extended and since the thickness of the double layer determines the range of action of electrical forces the particles with such thick double layers will feel electrical repulsion even if they are far away from each other. As the electrolyte concentration is increased, the diffuse layer becomes thinner and the solid particles will feel electrical repulsion only when they are very close to each other. At such small distances the van der Waals forces are very large and can be sufficient to prevail over the double-layer repulsion.

### 2.3 Isotropic and Anisotropic Particles

All surfaces of isotropic particles are created by breaking the same bonds and the resulting mineral surfaces are homogenous and do not vary as a function of position on the surface of a particle. In classical use of the DLVO theory the isotropic minerals are used to calculate interactions between such particles suspended in water. The interaction energy is calculated assuming either a constant potential case, or a constant charge case (which means that the potential, or charge, are determined by concentration of potential determining ions only and do not depend on a distance between interacting particles). According to the DLVO theory, maximum aggregation (fast coagulation) takes place at the iso-electric point (i.e.p.) that is the point at which there is no electrical repulsion and each particle-particle collision results in the attachment and formation of an aggregate.

Important information on the nature of the particle surface charge can be provided by comparison of the experimentally determined yield stress versus solution pH curves with the zeta potential-pH curves (since for most systems, potential-determining ions are H\(^+\) and OH\(^-\) ions). The shape of the yield stress-pH curves roughly follows a parabolic form as in Figure 2.12. As the pH moves away from the i.e.p, the yield stress values decrease both in higher and lower pH ranges; in these ranges particles are stabilized against aggregation by electrical double layers.
Figure 2.12 Electrophoretic measurements show that the maximum coagulation occurs at the iso-electric point of zirconia suspensions (after Boger, 1999, with the permission of Taylor & Francis).

A maximum yield stress (determined from rheological measurements) occurs at pH of the i.e.p. At this point, the van der Waals attraction is not opposed by any electrical repulsion (Hunter, 1982 and Scales et al., 1998). A well dispersed suspension does not exhibit a yield stress, while coagulated/flocculated suspensions are characterized by high yield stress values. Aggregating suspensions show shear thinning behaviour and reach their maximum yield stress value at the iso-electric point (Boger, 1999).

In the case of anisotropic particles the surface charges on different sides of the crystal are different (as in the case of clays). The Figure 2.13 shows that the surface charge on kaolinite edges becomes negative with increasing pH values, while the charge on the particle faces remains negative throughout the pH range (see Figure 2.9) which shows the case of montmorillonite). At the point where the charge on the particle edges becomes positive, the electrostatic force between faces and edges of particles changes from repulsive to attractive.
Figure 2.13 The likely zeta potential values for faces and edges of kaolinite (after Johnson et al., 1998, with the permission of Elsevier).

Therefore, the maximum coagulation in the suspension of anisotropic mineral will occur when the attractive electrostatic force between particle edges and planes is at its maximum. This kind of coagulation process is usually referred to as “heterocoagulation”. The maximum heterocoagulation is observed over the pH range where the difference between negatively charged upper faces and the edges is large.

The correlation between zeta potential measurements and the yield stress values of kaolinite is shown in Figure 2.14. The iso-electric point of kaolinite which is measured by zeta potential is around pH 3.5, while the point of maximum coagulation of kaolinite lies around pH 5.5 (Johnson et al. 1998). The iso-electric point and maximum yield stress does not appear in the same pH range.
Figure 2.14 Zeta potential measurements show that the maximum coagulation does not occur at the iso-electric point of kaolin suspensions at different solid concentrations (after Johnson et al., 1998, with the permission of Elsevier). (●, Φ=0.200; ○, Φ=0.060; ▲, Φ=0.090; Δ, Φ=0.130; ♦, Φ=0.180).

Various particle associations can form due to the differently charged edge and face surfaces (i.e. edge-to-face, edge-to-edge association). These associations can lead to different Bingham yield stresses of kaolinite suspensions (Rand and Melton 1977; Ma and Pierre 1999).
Figure 2. 15 demonstrates the variation of yield stress with pH due to the type of association.

Rand and Melton (1977) indicated that when pH is higher than the iso-electric point of edge (i.e. $p_{	ext{edge}}$ is 7.3) the edge and face are both negatively charged, so double-layer repulsion prevails, which results in a dispersed structure and a lower yield stress. However, at pH which is lower than the i.e.$p_{	ext{edge}}$, the edge-to-face coagulation prevails because of electrostatic attraction between oppositely charged face (negative) and edge (positive). This leads to stronger inter-particle forces and a higher yield stress. At the iso-electric point of the edge of kaolinite, there should always be a finite yield stress due to edge-to-edge coagulation.

The other property that affects the yield stress is ionic strength of suspension (Rand and Melton, 1977). As Figure 2. 16 shows the yield stress decreases with increasing electrolyte concentration where pH values are lower than the i.e.$p_{	ext{edge}}$ (pH 7.3), resulting in a decrease in the value of the corresponding Bingham yield stress. Since the addition of electrolyte compresses double layer at pH which is lower than the i.e.$p_{	ext{edge}}$ this translates into weaker particle-particle interaction. But, the face-to-face aggregation is promoted due to a reduction in the repulsion between faces (above the i.e.p). At the pH which is higher than the i.e.$p_{	ext{edge}}$, the increase in the electrolyte concentrations gradually increases the yield stress.
Figure 2.16 Effect of pH and NaCl concentration on the Bingham yield stress of homoionic Na kaolinite suspensions (9 wt%) (after Rand and Melton, 1977, with the permission of Elsevier). (∆, pH=5; ○, pH=6; ●; pH=7, ▽, pH=7.3; □, pH=8.5; ▲, pH= 9; ■, pH= 8)

2.4 Particle Aggregation

2.4.1 Agglomerate Flotation

Flotation depends on particle size. It is widely known that the recovery of minerals by flotation is successful in the 10-100 μm range. The recoveries of both very fine and coarse particles outside the optimal size range are low for different reasons. The low flotation rate of the fine particles results from insufficient collision rates, whereas the flotation of the coarse particles is impeded by the stress acting on the particle-bubble aggregates, leading to the particle-bubble detachment. Typical size effects in flotation on size-by-size recovery of valuable minerals and pulp components are illustrated in Figure 2.17.
Fine particles have been the focus of a lot of research for many years and fine particle recovery is one of the most difficult problems in the mineral processing industry (Trahar, 1981, Sivamohan, 1990, Collins and Read, 1971, Shibata and Fuerstenau, 2003, Rubio et al., 2003, 2006). Valuable minerals are quite often in finely disseminated form that requires very fine grinding in order to achieve liberation. Although the recovery of these fine mineral particles is generally exceedingly difficult, diminishing raw material resources necessitates the recovery of such mineral values.

Fine particles may float poorly even if they are hydrophobic. The reasons of poor flotation result from small mass, high surface area and high surface energy. Small mass leads to low particle momentum, particle entrainment in concentrates (e.g. froth) and low probability of collision with a bubble; the latter leads to low flotation rate. High surface area leads to high reagent consumption. High surface energy leads to a high dissolution rate in water, adsorption of a large quantity of chemicals, rigidity of froth and high pulp viscosity and undesirable coating of the valuable particles by ultrafine gangue particles (Klassen and Mokrousov, 1963; Fuerstenau, 1980). This situation can be improved only by increasing the size of the floated particles by aggregation.
There are several ways of increasing the particle size of fine particles. The best known examples are oil agglomeration and agglomerate flotation.

### 2.4.2 Oil Agglomeration

Oil agglomeration is based on the preferential wetting of solid particles by immiscible liquid. In this process the mineral suspension containing hydrophobic and hydrophilic particles is conditioned with emulsified oil. When particles collide with oil droplets, the oil droplets attached to hydrophobic particles merge and bridge the particles into larger agglomerates. Thus, the process is selective, oil droplets attach only to hydrophobic particles and only such particles agglomerate. Since only very hydrophobic particles can be agglomerated by oil agglomeration, this technique is used for solids which are hydrophobic by nature. But, hydrophilic minerals can also be beneficiated by this method; the only difference is that such particles have first to be rendered hydrophobic with the use of properly selected collectors (Straton-Crawley, 1979). Sulfhydryl collectors such as xanthates are used to float sulfides. The oil agglomeration of sulfide particles rendered hydrophobic with xanthate is therefore possible. In this process, a collector is added in order to make the particles hydrophobic and then extender oil to initiate agglomeration.

![Figure 2.18](image.png)

**Figure 2.18** Schematic illustration of agglomerate flotation.

The agglomerates are hydrophobic and thus this technique can be used to increase the size of the particles which are then recovered by flotation. The only difference between agglomerate flotation and oil agglomeration is the extent of agglomeration. In oil agglomeration the formed agglomerates must be large enough to be recovered by screening whereas in the agglomerate flotation the size of the formed agglomerates must be sufficient for flotation (Laskowski and Castro, 1999; Laskowski and Valdivieso, 2004).
2.4.3 Particle Dispersion

2.4.3.1 Dispersing Agents

Dispersing agents are used to prevent fine particle aggregation. The aggregation of very fine particles (particle finer than 10µm are commonly referred to as slimes) onto coarse particles is referred to as slime coating; this is responsible for poor floatability of valuable minerals in many operations.

Slimes can interfere with the flotation of valuable minerals in different ways. First of all, they decrease hydrophobicity of the valuable particles by coating their surface. In the presence of slime coatings flotation rates of the hydrophobic particles decrease and so does the flotation efficiency.

Removal of slimes from the surfaces of valuable coarser particles can be achieved by using of dispersing agents. Adsorption of dispersants onto the slime particles (and also onto the valuable minerals) commonly makes these particles more negatively charged and stabilize the slimes against aggregation (Sun, 1943; Parsonage, 1985). Thus, it is obvious that these agents improve selectivity of flotation.

The commonly used dispersants in mineral processing are water glass, sodium hexametaphosphate, dextrin, sodium fluorosilicate, CMC (carboxymethyl cellulose), tannic acid and lignosulfonates, etc. (Laskowski and Pugh, 1992).

While the most common dispersants produce strong electrostatic repulsive forces between the particles by increasing their electric charge (electrostatic stabilization), it is also possible to stabilize disperse system by steric stabilization. This results from the adsorption of polymer on the dispersed particles; if the thickness of the adsorption layer is sufficient this layer can prevent the interacting particles from approaching each other (the van der Waals attractive forces are large only at small distances). In many cases, stabilization can be due to both electrostatic and steric stabilization (Laskowski and Pugh, 1992). These three different cases are shown in the Figure 2.19.
2.5 Control of Rheological Properties

Rheological properties of slurries affect a host of different mineral processing operations (Boger, 1999). Controlling the slurry transportation, flotation, dewatering and milling efficiency strongly depends on the slurry rheology.

2.5.1 Effects of Solid Contents on Rheology of Mineral Suspensions

Increasing the solid content in the slurry has a drastic influence on its rheological properties. If the solids content is low, the average distance between neighbor particles will be much larger than their size. In such a case, inter-particle forces are small. As the solids concentration is raised, the importance of interaction between particles increases.

The influence of particle concentration on the viscosity of the concentrated suspensions is best described by the maximum packing fraction ($\Phi_{\text{max}}$). The utility of this approach is in the fact that it takes into account the effect of particle size distribution and particle shape, since they both control the maximum packing fraction.

The Krieger-Dougherty equation is:

$$\frac{\eta}{\eta_w} = \left(1 - \frac{\phi}{\phi_{\text{max}}}\right)^{-\phi_{\text{max}}[\eta]}$$  \hspace{1cm} [2.5]

where $\eta$ is the apparent viscosity of the suspensions, $\eta_w$ is viscosity of water, $\Phi_{\text{max}}$ is the maximum volume fraction and $[\eta]$ is the intrinsic viscosity, which is expected to be 2.5 for
spheres. As the volume fraction of solids in the system goes up: the particles become more closely packed together; it becomes more difficult for them to move freely; particle-particle interactions increase; and viscosity rises. As the volume fraction nears maximum for the sample, viscosity rises very steeply. The important conclusion of this equation is that the viscosity strongly depends on $\Phi_{\text{max}}$.

At lower solid contents, dispersions typically exhibit Newtonian behaviour (Krieger, 1972) whilst non-Newtonian rheological behaviour is characteristic at higher solids content. Non-Newtonian behaviour is strongly related to particle size and it is observed in cases of highly concentrated fine particle suspensions. If the suspension consists of coarse particles, the apparent viscosity does not increase significantly with increasing solids content and Newtonian behaviour may be seen up to a relatively high solids concentrations.

The existence of a yield stress can be considered as an indicator of an internal structure. Figure 2. 20 shows that the yield stress increases with volume fraction and depends on aggregation (which is maximum at i.e.p.).

![Figure 2. 20](image-url)

**Figure 2. 20** The yield stress values of concentrated $\alpha$-alumina suspensions as a function of pH and the solids volume fraction (after Johnson et al., 2000, with the permission of Elsevier).
The effects of solids concentration on suspension viscosity have been studied for spherical particles. Particle shape has a strong effect on maximum packing fraction and so on the concentration/viscosity relationship. When particles have irregular shapes, particle packing density is also affected.

The effect of irregularly shaped particles on viscosity was investigated by Barnes et al. (1989) who indicated that rod-shaped particles have a greater effect on suspension viscosity than disc-shape particles. The effects of concentration of the fibrous particles on viscosity are even more pronounced (Horie and Pinder, 1979). It can then be expected that the minerals like chrysotile and lizardite must also strongly affect the pulp rheology. A high ratio of the surface area to volume for such particles results in elevated apparent viscosities and yield stresses of such suspensions (Leong et al., 1996; Megias-Alguacil et al., 2000).

![Figure 2. 21 The effects of differently shaped particles on apparent viscosity at various volume concentrations (at a shear rate of 300 s⁻¹)” from Clarke (1967) (after Barnes, 1989, with the permission of Elsevier).](image)

Horie and Pinder (1979) found that the larger the length-to-diameter (L/D) ratio of the particles, the higher is the yield stress at any concentration as shown in Figure 2. 22. It can also be seen that the higher the particle concentration, the higher is the yield stress.
2.5.2 Effects of Particle Aggregation on Rheology of Mineral Suspensions

Rheological behaviour of mineral slurries can be used as an indicator of the level of interaction/aggregation in a pulp. Therefore, rheology is a useful processing control variable (Muster and Prestidge, 1995).

Both plastic viscosity and yield stress were shown to increase significantly with decreasing particle size (Valentik and Patton 1976; Klein et al. 1988).

Any aggregation between particles results in a loss of stability and fast sedimentation in a very dilute disperse system whereas in a heavily loaded suspension the aggregation may prevent particles from settling so it stabilizes the system towards settling. The aggregation creates a network of interacting particles, which sharply increases the viscosity (Laskowski, 2001).

In the system which also contains irregular non-spherical particles their effect on viscosity may be prevalent. The system which was studied in this thesis contains fibrous serpentine mineral (chrysotile). The problematic rheological behaviour of these minerals consequently necessitated the slurry streams to be diluted to 10% solids (by weight) in commercial flotation, thus reducing production capacity.
2.5.3 Effects of Dispersing Agents on Rheology of Mineral Suspensions

Various chemical additives, both organic and inorganic, have been used to reduce viscosity and to increase stability of the aqueous suspensions. The general effect of these modifiers (e.g. carboxymethyl cellulose, guar gum, dextrin) is to reduce the yield stress rather than the plastic viscosity as claimed by Schrantz (1954) and Meerman (1959). In Klassen’s tests (1963) the increasing polymer addition decreased both the viscosity and yield stress initially, until a plateau was reached. Aplan and Spedden (1964), who used apparent viscosity to characterize the magnetite dense medium rheology, showed that the addition of a dispersant agent (sodium hexametaphosphate) reduces the apparent viscosity and increases the suspension stability (He and Laskowski, 1999). Coal-water slurries, the concentrated suspensions of fine coal in water, are pumpable only because of the use of dispersing agents (Laskowski, 2001).

2.5.4 Effects of Rheological Properties of Suspension on Flotation

Particles in flotation pulp are carried into the froth by two mechanisms; true flotation and entrainment. True flotation results from the attachment of hydrophobic particles to air bubbles (Smith and Warren, 1989). True flotation is a selective separation process. Entrainment is another way of transporting fine particles to the froth. It can be defined as a mechanical carry-over of all fine particles (hydrophobic and hydrophilic) by the upward movement of water between the bubbles in the froth phase. These hydrophilic particles are not actually attached to the bubbles; they are transported up and out of flotation cell between bubbles. The fine fraction of these entrained particles does not have sufficient time to drain out of the froth zone and so is recovered. Some of the larger particles and water, trapped between bubbles, drain back into the pulp but the remaining water and fine particles are carried upwards and recovered in the concentrate.

The entrainment is a problem in flotation because it is non-selective process. There is no discrimination between hydrophobic and hydrophilic particles, both of which may be present in the inter-bubble water.

Many authors have indicated that entrainment is strongly associated with the amount of water carried in the froth (Engelbrecht and Woodburn, 1975; Bisshop and White, 1976; Lynch et al., 1981; Smith and Warren, 1989; Govindarajan et al., 1991, Maachar and Dobby, 1992; Savassi et al., 1998; Tao et al., 2000, Rahal et al., 2001). It has been found that the
higher the recovery of water, that is wetter the froth, the higher the proportion of gangue appears in the concentrate and lower its grade. (Jowett, 1966; Johnson et al., 1974).

In general, it has been observed that the finer the particle, the more likely it is to be recovered by entrainment. Johnson et al. (1974) and Trahar (1981) showed how the entrainment factor for gangue increases with decreasing particle size. The entrainment mechanism becomes more important as the particle size decreases whilst it is insignificant for particles which are coarser than 50 µm (Smith and Warren, 1989). The probability that particles will be recovered by entrainment is higher for finer particles. The gangue minerals in general appear in the pulp as very fine particles, and the flotation of fine gangue particles by entrainment decreases the grade.

Jowett (1966) showed that the gangue recovery was linearly related the water recovery.

The solids content in the pulp has a significant effect on the recovery by entrainment. The probability of collisions between particles and bubbles increases with a larger amount of particles, thus enhancing the recovery of the valuable mineral. However, the increase in solids content also increases the pulp viscosity making pulp mixing more difficult. Lynch et al. (1981) showed that at the same water recovery, the recovery of non-floatable gangue increases with increasing solids content in the feed.

The other factor that affects entrainment is froth structure. Booth and Freyberg (1962) claimed that froth structure has a strong influence on recovery and grade. Thus, in general closely knit froth composed of small bubbles allows higher recovery of both entrained and attached particles due to lower drainage and less coalescence (Subrahmanyam and Forssberg, 1988). On the other hand, a loosely textured froth composed of larger bubbles tends to release gangue particles and tend to produce concentrates of higher grade.

In the case of asbestos ore (such as the one from Mt. Keith), the density of the flotation pulp is reduced to about 15% in the industry due to low recoveries. Apparently in the case of the fibrous gangue, the probability of collision between valuable particles and bubbles in the suspension of the fibrous gangue is very much affected by the concentration of such gangue particles, which also affects bubble size. The increasing solids content dampens the turbulence intensity in the cell, which cause bubble coalescence (Rubinstein, 1995).

The topic of the effect of pulp viscosity on flotation is an “uncharted territory” in the flotation research.
2.6 Characterization of Nickel-Sulfide Ore

The nickel ore from Western Australia used is a low-grade ore (0.52% Ni), which is classified as a serpentinised ultramafic nickel (Ni) deposit. The main nickel bearing minerals are pentlandite (NiFe)₉S₈, millerite (NiS) and violarite (Ni₂FeS₄) (Kerr, 2002). Pentlandite is the most common mineral and contributes 60-80%; millerite 2-20%, while violarite 0-10% to the nickel-bearing mineralogy in this specific ore. The detailed mineralogical analyses of the ore for various particle size ranges are given in APPENDIX I. The chemical composition of the ore is presented in Table 2.1.

Table 2.1 Chemical composition of nickel sulfide ore from Western Australia (Senior and Thomas, 2005)

<table>
<thead>
<tr>
<th>Process Stream</th>
<th>Assays %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
<td>Ni</td>
</tr>
<tr>
<td></td>
<td>0.52</td>
</tr>
</tbody>
</table>

The gangue minerals of the ore are mainly members of serpentine group, such as chrysotile and lizardite; talc in minor amount, as well as iron sulfides with pyrite and pyrrhotite as the main contributors. Pyrrhotite is generally much more abundant in nickel sulfide ore deposits than pentlandite. One of the difficult challenges for the nickel sulfide ores processing is the separation of pentlandite from pyrrhotite (Kerr, 2002). One of the difficult challenges for the nickel sulfide ore is the presence of violarite and magnetite.

The most problematic gangue in Western Australia nickel-sulfide ore are serpentine minerals. These minerals compose over 90% of gangue in the ore, while hydroxyl-carbonates, including magnesite, the main mineral of the carbonate group, compose the remaining part of the gangue minerals.

The difficulty in treating ores that contain a high amount of serpentine group minerals was encountered in processing of the low grade nickel deposits (Senior and Thomas, 2005; Fornasiero and Ralston, 2006). Serpentine minerals depress pentlandite flotation and pentlandite recovery gradually decreases with serpentine content (Mani et.al, 1997) which can be seen in Figure 2.23.
Very poor separation of pentlandite from the serpentine gangue was explained as the slime coatings of lizardite and chrysotile onto the surface of pentlandite (Edwards et al., 1980 and McQuie, 1999). Slime coating of serpentine minerals reduces hydrophobicity of the sulfide particles and may also reduce collector adsorption (Learmont and Iwasaki, 1984). In each case, the flotation rate and the recovery of the valuable sulfide minerals decrease (Trahar, 1981; Wellham et al., 1992). Edwards et al. (1980) indicated that the formation of slime coatings is directly related to the surface potentials of the sulfide particles and slimes.

The importance of particle charge and zeta potentials for the formation of slime coatings has been highlighted in a number of studies. In one of these studies, Parsonage (1985) suggested that slime coatings occur when there is no potential energy barrier for heterocoagulation, which certainly influenced the zeta potentials of the mineral and slime particles.

As serpentine minerals carry positive charges over a broad pH range, it is likely that the serpentine particles will attach through electrostatic attraction to the negatively charged pentlandite surface and result in severe depression (see Figure 2. 24). When slimes are negatively charged, the slime coating on pentlandite does not occur and flotation is not impeded (Edwards et al., 1980).

Slime coating can be prevented by ensuring that the slime and the valuable mineral particles have high and either negative or positive but the same surface charge. These coatings
are removed by the addition of dispersants which adsorbs preferentially onto slimes, their surface charges are reversed and electrostatic attraction is reversed. The effect of pH on electrokinetic potential of chrysotile (Ney, 1973) and lizardite and pentlandite particles (Bremmel, 2005) are shown in Figure 2.24. (Carboxymethyl cellulose (CMC), which is an anionic polymer, can be used to reduce slime coating in the process (Bremmel et al, 2004).

Figure 2.24 Effect of pH on electrokinetic potential of chrysotile, lizardite and pentlandite particles in 0.001M KNO₃ as a function of pH (after Bremmel, 2005, with the permission of Elsevier).

Chrysotile is the major gangue component in the nickel-sulfide ore; it constitutes about 80% of the ore by mass. The presence of serpentine minerals in nickel sulfide mineral is often associated with processing problem. The fibrous structure of chrysotile is generally believed to be the cause of its problematic rheological behavior which affects the process negatively. The shape of the particle can dramatically affect solution viscosity. The fibrous chrysotile results very high surface area to volume ratio of the particles. This high L/D ratio results in a dramatic increase in interactions between particles which increases yield stresses and viscosities (Leong et al., 1996; Megias-Alguacil et al., 2000). Besides, there are other visible problems in the flotation processes which are the tendency of air bubbles to coalesce, slow flotation rate and high mechanical recovery of gangue (Kirjavainen and Heiskanen, 2007).
reason of all these effects, nickel recovery and also nickel grade reduces (Mani et al., 1997; Senior et al.1995).

2.6.1 Surface Properties of Chrysotile

Chrysotile, referred to as white asbestos, is a naturally occurring fibrous hydrated magnesium silicate belonging to the serpentine group of minerals. Chrysotile fibres are composed of two layers of chemicals a brucite layer and a silica tetrahedral layer. Due to the differing chemical and physical characteristics of the two materials, these fibres tend to curl into hollow tube which is illustrated in Figure 2. 25. The bending of the layer is continuous which give the mineral its fibrous habit. This fibrous structure also can be seen in Figure 2. 25 as an electron micrograph which is produced by Yada (1971).

The building blocks of chrysotile are shown in (A). The notations shown are: T = tetrahedral ‘silica’ component; O = octahedral ‘brucite’ component. The hydroxyl group is shown by the heavy ‘dot’ One of the stacking orientations of the tetrahedral and octahedral layers is shown in (B). The apical oxygens of the silica sheet substitute within the brucite layer. This figure is modified from Skinner et al. (1988) by Langer and Nolan (1994).

Figure 2. 25 Schematic of tubular structure and TEM micrograph of cross section of chrysotile (after Yada, 1971, with the permission of IUCr).

The curled tubular structure of chrysotile makes everything more complex than the more familiar edge/plate structure. The spiral structure indicates that there are octahedral and tetrahedral edges occurring at the end of each tube. The curvature occurs with the brucite layer on the outer surface, the electrokinetic behaviour of brucite is similar to chrysotile and both of them are positive over a broad range of pH. However, the surface charge of the chrysotile tends to become negative as pH increases (pH>10), which causes heterocoagulation.
of these particles (see Figure 2.24). This results in a very intricate, tangled structure which makes chrysotile rheology very complex. Senior and Thomas (2005) indicated that elevated serpentine content has a dramatic increasing effect on the Bingham yield stress of mineral slurries. Bingham yield stress and Bingham plastic viscosity of the suspension increases as volume fraction of chrysotile increases. It is worth pointing out that the increase in yield stress was observed at particularly low volume fractions which prove the detrimental effect of the chrysotile on the rheology of suspension.
3 EXPERIMENTAL

3.1 Introduction

The experimental program of this thesis was divided into three parts. The flotation and rheological tests were conducted with the use of the nickel-sulfide ore from Western Australia. In order to see the effect of solids content on flotation, the tests were done with variable solids content in the absence and presence of oil. Besides, the tests were performed at variable solids content in both conditioning and flotation stage.

The second part of the experimental program is the investigation of the correlation between degree of agglomeration/ dispersion and flotation response through agglomerate flotation and slimes dispersion with CMC. Tests with CMC were conducted with both the process water and distilled water. By monitoring both the concentrate yield and water recoveries it was possible to calculate the true flotation and the flotation by entrainment.

In the third part, rheology measurements were conducted to investigate the time dependency of the rheological properties of the tested slurry (nickel sulfide ore) under different conditions. The tests were performed in both distilled water and process water in the presence and absence of CMC and, each test was performed at different shear rate values.

3.2 Materials

3.2.1 Reagents

Flotation tests were conducted using Flex-41 xanthate as a collector. Flex-41 xanthate, a proprietary brand of Prospec Chemicals, is an equivalent of iso-butyl xanthate. Collector dosage was 180 g/t in all the flotation tests. Emulsified diesel oil (EDO) was used (500 g/t) as agglomerating additive.

Carboxymethyl cellulose (CMC), obtained from Polysciences Inc., was used as a dispersant at concentration of 400 g/t to prevent slime aggregation. Its molecular weight is 80,000 g/mol.

Agglomerating and dispersing agents were used in emulsified form in the experimental study: 1 gram of these agents was added into distilled water and stirred vigorously to obtain 100 ml solutions, 1% of which were those agents. And the stock solution of Flex-41 was prepared by dissolving 1 g of Flex-41 in 100 g distilled water.
Teric 407, a registered trademark of Hunstman Corporation Australia Pty Ltd., was applied as a frothers. It was added at concentrations of 30 g/t. The stock solution of Teric 407 was prepared by dissolving 0.4 g of Teric 407 in 200 g distilled water.

### 3.2.2 Ore Characterization

Nickel-sulfide ore from Western Australia was used in this study. The particle size distribution of the flotation feed (-25 µm) was performed by Malvern size analyses using laser light, which is shown in Figure 3.1.

![Particle Size Distribution](image)

**Figure 3.1** The particle size distribution of nickel-sulfide ore.

The technique of laser diffraction relies on the fact that particles passing through a laser beam scatter light at an angle that is inversely proportional to their size (small particles scatter light at high angles whereas large particles scatter light at low angles). It is therefore possible to calculate particles size distribution if the intensity of light scattered from a sample are measured as a function of angle. However, these analyzes are designed for regularly shaped particles, the size estimation of fibrous particle (such as chrysotile) is doubtful. For this reason, scanning electron microscope (SEM) analysis was used for particles size approximation of the chrysotile fibres.
Figure 3. 2 a) and b) confirmed the presence of chrysotile in flotation. The long fibrous chrysotile particles are seen in the figure. Figure 3. 3 shows chrysotile’s fibrous structure. The SEM micrograph shows fibres with thickness of about 0.1 µm, and length of up to about 100 times the thickness.

Figure 3. 2 a) and b) show SEM micrographs of flotation feed of nickel-sulfide ore.
3.3 Methods

3.3.1 Grinding and Classification

Grinding: A mild steel rod mill 30 cm long and 20 cm in diameter was used for grinding. The mill was loaded with 15.5 kg of mild steel rods. The crushed and packed ore which is below 3 mm was received in 1kg plastic bags. For each grinding test, one bag of ore was first placed into the mill, then 1.5 litre of synthetic process water or distilled water was added. Composition of the process water is given in Table 3. 1. Following addition of water 40% of the collector was added to the mill.

Table 3. 1 Composition of the process water

<table>
<thead>
<tr>
<th>Compound</th>
<th>Content, g/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaCl</td>
<td>48.2</td>
</tr>
<tr>
<td>CaCl₂</td>
<td>1.25</td>
</tr>
<tr>
<td>KCl</td>
<td>2.10</td>
</tr>
<tr>
<td>NaHCO₃</td>
<td>0.29</td>
</tr>
<tr>
<td>MgSO₄·7H₂O</td>
<td>24.4</td>
</tr>
</tbody>
</table>
The solids content in grinding was 40% and the grinding time was 18 minutes so that 80% of the ground ore was below 125 µm. The sample contained 0.5 % Ni. After grinding, the ground product was washed out using 1.5 L of the synthetic process water (or distilled water) with the resulting 25% solids content in the ground product. The products of every four grinding tests were collected together in one bucket for next stage which was classification.

Classification: The product of grinding was classified using C700 Mozley cyclone test rig. The pulp containing 4 kg of ore (25% solids content) was poured into the rig with 4L additional process water (or distilled water), resulting in a feed solids content of 20% in the classification feed. The produced cut-size of ore was 25 µm. This product contained 0.48 % Ni. The overflow and underflow were collected separately. The yields of the underflow and overflow were about 45% and 55%, respectively.

Splitting: Since the obtained overflow contained a large amount of water, the clear water drew off from the sample after 4 hours of settling. The sediment after decantation (around 30% solids content) was then separated into 8 portions using a splitter. Each portion (containing 229 g of solids) was used separately in batch flotation tests. The clear water from decantation was used as wash water in splitting and as dilution water in flotation.

3.3.2 Flotation

All flotation tests were carried out in a 2-litre Denver laboratory flotation machine with the nickel-sulfide ore. Ore was ground just before flotation in order to prevent oxidation of the ore; grain size of the ore was -25 µm. The impeller speed was set at 1500 r.p.m. and the airflow rate was maintained at 4 L/min.

The experiments performed with 180 g/t of Flex 41 collector; 40% of the collector was added to the mill, with the remaining 60% added to the flotation pulp and conditioned for 10 minutes prior to diesel oil (500g/t) addition, thereafter the pulp was conditioned for a further 5 minutes with oil. Dispersing agent was added into the pulp if it was necessary and conditioned for 5 minutes. Finally, 30g/t of Teric 407 frother was added and conditioned for 1 minute in all the tests. The froth product was scraped off from the lip of the cell every 5 seconds. Concentrates were collected after 2, 5, 10, 20 and 30 minutes. Multiple tests were performed for different solids contents in the flotation stage.

The pulp level was maintained constant at all times by adding process water as required. Seven bottles filled with water were also weighed out before conducting the tests.
The five of these bottles were used to maintain the pulp level constant during the tests. The other two bottles were used to wash off the froth attached to the scrapper when collecting the concentrates. After each test, the water remaining in each bottle was weighed in order to determine the amount of wash water added to each tray. Thus in addition to the evaluation of the grade and recovery of the flotation tests, water recovery was also monitored in order to estimate the degree of entrainment within the flotation system.

Flotation tests were done under different conditions; in the first part, agglomerate flotation tests were carried out at different solids content in the flotation stage. In the second part, agglomeration tests were done both at different solids content and using dispersing agent (CMC). Tests with CMC were done both in distilled water and in process water. The procedure for the tests in distilled water and process water was exactly the same. The sequence of adding reagents was also same, but dispersant was added and conditioned just before frother addition. All flotation tests were carried out at natural pH of 8-8.5.

The flotation concentrates were dried in an oven at 100-150 °C over night. A sample of each concentrate and tailing was collected for each test to do mass balance.

3.3.3 Rheology

Rheological tests were conducted in a controlled rate co-axial rheometer Haake Viscotester 550. The samples of the slurry were taken from the flotation pulp just before aerating the pulp to initiate flotation.

All measurements were conducted using a MV-1P bob and cup fixture where P indicates a ribbed surface to reduce errors associated with wall slip. The fixture is illustrated in Figure 3. 4 and the dimensions of the fixture are given in Figure 3. 4.

![Figure 3. 4](image)

Figure 3. 4 Schematic representation of the double gap rheological measuring system.
Table 3.2 The dimensions of bob and cup fixture

<table>
<thead>
<tr>
<th>Sensor</th>
<th>MVP</th>
<th>MV 1P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inner Cylinder (Rotor)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Radius $R_I$ (mm)</td>
<td>20.04</td>
<td></td>
</tr>
<tr>
<td>Height $L$ (mm)</td>
<td>60.00</td>
<td></td>
</tr>
<tr>
<td>Outer Cylinder (Cup)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Radius $R_a$ (mm)</td>
<td>21.00</td>
<td></td>
</tr>
</tbody>
</table>

The shear stress versus shear rate data were recorded. Rheogram was generated in the shearing rate range between 0 and 200 s$^{-1}$ for 1 minute to obtain complete flow curves. The flow curve data were modeled by using both the Bingham and the Casson equation. Casson model was selected to represent the flow curve since it fits better the experimental flow curves. An example of the rheogram obtained for the tested nickel sulfide ores is given in Figure 3.5.

Casson model equation can be given as:

$$\tau^{1/2} = \tau_C^{1/2} + (\eta_C \dot{\gamma})^{1/2}$$  \[3.1\]

where, $\tau$ is shear stress
$\tau_C$ is yield stress
$\eta_C$ is Casson viscosity coefficient
$\dot{\gamma}$ is shear rate
Figure 3.5 Example of a rheogram of the slurry with the ground nickel-sulfide ore, fitted with Casson model.

3.3.3.1 Stress Decay Tests

Stress-decay tests were conducted to characterize the time dependent properties of the suspension by using Haake Viscotester 550. For the determination of the time dependent flow behaviour, a constant shear rate was set and the independent variables were recorded as a function of time. Measurements were carried out in duplicate. For each run, a fresh sample was used. These tests involved measuring the shear stress over a 100 second period at pre-defined shear rates. Equilibrium flow curves were generated from stress decay tests by plotting the equilibrium stress versus the shear rate.

During the stress-decay tests, at increasing shear rates (10, 50 and 100 s\(^{-1}\)) the shear stress values were recorded after 100 seconds of shearing at each rate.

Table 3.3 summarizes the conditions for the stress decay tests for different solid contents and conditions.
Table 3. 3 The conditions of the stress decay tests

<table>
<thead>
<tr>
<th>Solids Content</th>
<th>Water</th>
<th>Dispersing Agents</th>
<th>Shear Rate (1/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>Process Water</td>
<td>CMC</td>
<td>10, 50, 100</td>
</tr>
<tr>
<td></td>
<td></td>
<td>No CMC</td>
<td>10, 50, 100</td>
</tr>
<tr>
<td></td>
<td>Distilled Water</td>
<td>CMC</td>
<td>10, 50, 100</td>
</tr>
<tr>
<td></td>
<td></td>
<td>No CMC</td>
<td>10, 50, 100</td>
</tr>
<tr>
<td>20</td>
<td>Process Water</td>
<td>CMC</td>
<td>10, 50, 100</td>
</tr>
<tr>
<td></td>
<td></td>
<td>No CMC</td>
<td>10, 50, 100</td>
</tr>
<tr>
<td></td>
<td>Distilled Water</td>
<td>CMC</td>
<td>10, 50, 100</td>
</tr>
<tr>
<td></td>
<td></td>
<td>No CMC</td>
<td>10, 50, 100</td>
</tr>
</tbody>
</table>
4 RESULTS

4.1 Effect of Solids Content in Conditioning Stage on Flotation Performance

The tests were conducted at two different solids content in order to investigate the effects of varying solids content in conditioning stage. It should be noted that in flotation experiments the pulp density was 10 % by weight regardless of the solids content during conditioning.

The results which represent the effect of solids content in the conditioning stage on flotation are summarized in Table 4.1. The table shows the mean values of flotation performance indicators (such as nickel grade and nickel recovery) for the tests in which the slurry was conditioned either at 10% or at 30% solids without oil respectively.

Table 4.1 Effects of solids content in the conditioning on flotation performance

<table>
<thead>
<tr>
<th>Flotation Performance Indicators</th>
<th>500 g/ton Oil</th>
<th>No Oil Addition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grade</td>
<td>4.0±0.6</td>
<td>2.6±0.4</td>
</tr>
<tr>
<td>Recovery</td>
<td>53.4±2.6</td>
<td>55.0±1.1</td>
</tr>
<tr>
<td>Water Recovery</td>
<td>7.7±1.2</td>
<td>10.5±1.5</td>
</tr>
</tbody>
</table>

The results in Table 4.1 conclusively demonstrate that the amount of solids content in the conditioning stage has no effect on flotation performance under the conditions tested in this study (without oil addition). None of the differences in any of the flotation performance indicators were significant within a 95% confidence interval.

To complete these results an additional set of tests were carried out with conditioning either at 10% or at 30% solids but with 500 g/t of emulsified diesel oil.

4.2 Effects of Oil Addition on Flotation Performance

Figure 4.1 demonstrates that the flotation tests performed after conditioning at 10% solids in the absence of oil exhibited lower recoveries compared to conditioning at 30% solids. No difference was found between the tests performed with 0 and 500 g/ton of diesel oil when the pulp was conditioned at 30% solids. When the pulp was conditioned at 10% solids,
the nickel recovery was higher with 500 g/ton diesel oil addition compared to the test with no diesel oil.

Figure 4. 1 Effect of diesel oil addition on flotation of the nickel sulfide ore. The solids content in the flotation was 10 % by weight, the conditioning was carried out either at 10 or at 30% solids.

Figure 4. 2 The effect of diesel oil addition (EDO) on flotation of nickel-sulfide ore. The solids content in the flotation was 10 % by weight, the conditioning was carried out either at 10 or at 30% solids.
Figure 4.2 shows the obtained grade versus recovery relationships for the tests carried out either with 500 g/ton of emulsified diesel oil or without oil at 10% and 30% solids content. Conditioning at 10% solids in the absence of diesel oil provides significantly lower concentrate grade compared to 30% solids content. The summary of the results pertaining to the effect of oil addition at 10% solids content during the conditioning stage is given in Table 4.2. A statistical t-test confirmed that the decrease in nickel grade is significant on a 95% confidence interval. However, the differences in the concentrate grades for the remaining three conditions were found to be insignificant.

**Table 4.2 Effects of oil on flotation performance at 10% solids content.**

<table>
<thead>
<tr>
<th>Flotation Performance Indicators</th>
<th>500 g/ton Oil Average</th>
<th>No Oil Addition Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grade</td>
<td>4.0±0.6</td>
<td>2.6±0.4</td>
</tr>
<tr>
<td>Recovery</td>
<td>53.4±2.6</td>
<td>55.0±1.1</td>
</tr>
</tbody>
</table>

In order to clarify the improvement in the selectivity in the presence of oil, nickel grade versus nickel recovery graph was plotted. These results imply that the selectivity of nickel-sulfide ore is low in the absence of oil, which is probably resulted from higher entrainment. With addition of oil, nickel recovery improved and this leads to a higher grade of the concentrate. The relationship between nickel grade and nickel recovery for all the tests is illustrated in Figure 4.3 as a result of various tests of flotation. The figures shows 5 minute flotation results.
Figure 4.3 Effect of oil addition on selectivity of the flotation of the tested nickel ore.

The figure demonstrates that in spite of a high scatter of the results, both higher nickel grades and higher true nickel recoveries were achieved in the tests when 500 g/t of oil was used. Figure 4.3 also demonstrated that agglomerate flotation improves the floatability of fine nickel bearing particles.

4.3 Effects of Pulp Density on Flotation

As the obtained results revealed changing the solids content during conditioning had no apparent effect on flotation performance. In order to further investigate this aspect, the flotation and rheological tests were conducted when varying solids content in the flotation stage between 5% to 20% in the presence of diesel oil. Agglomerate flotation tests were conducted for 30 minutes with 180 g/t of Flex-41 xanthate and 500 g/t of emulsified diesel oil.

The conditioning and flotation experiments that are investigated in this part were both performed at the same solids content. The recovery of nickel as a function of the solids content in the flotation pulp is shown in Figure 4.4.
Figure 4.4 Nickel recovery as a function of flotation time at varying solids content in agglomerate flotation (with 500g/t diesel oil).

As Figure 4.6 demonstrates the concentrate yield increases with the pulp density with flotation feed. Comparison with Figure 4.6 indicates that this concentrate mass increase is caused by increasing entrainment; the concentrate grade dramatically declines with increasing pulp density.

Figure 4.5 Effect of pulp density of the flotation feed on the concentrate yield-nickel recovery.
Figure 4.6 The grade – recovery curves for the agglomerate flotation tests carried out at varying solids content in the pulp. The flotation concentrates were collected after 2 minutes of flotation (point A), 5 minutes of flotation (point B), 10 minutes of flotation (point C), 20 minutes of flotation (point D) and 30 minutes of flotation (point E).

Figure 4.6 shows the effect of solids content on the grade-recovery curves. The agglomerate flotation tests were carried out over varying period of time. The marked points explain experimental procedure. The results obtained after collecting the concentrate over 2 minutes is shown as Point A. Point B gives the flotation results for 5 minutes of flotation, and so on. 10% solids content only has two points; one of them is for 2 minute and the other is for 30 minutes flotation results.

The “cleanest” flotation conditions were apparently created when the solids content in the pulp was very low (5% solids) and over the first 2 minutes when the content of the valuable sulfides was relatively high. While recovery increases when the concentrate is collected over 5 minutes, the grade clearly deteriorates (point B), and the grade deteriorates further for longer flotation times. This indicates that while true flotation is the best at the beginning of the flotation tests, the entrainment increases with time. Apparently entrainment increases a lot when the solids content in the pulp is raised and the ratio of valuables-to-gangue is low. Even a 2 minute flotation results at 20% solids content show low grades.
It was observed during the flotation tests at 20% solids content that air dispersion within the cell was significantly affected in these tests. A smaller number of much larger bubbles was seen creating a “boiling” impression. Similar observations were reported by Grau and Heiskanen (2005) who indicated that with increasing solids content, the bubble size increased. They also reported that the influence of the solid concentration on bubble size was more evident at solid concentrations exceeding 20% by weight (Rubinstein, 1995).

As Figure 4.7 shows, the water recovery at 5% solids and at higher pulp densities is very different. At the beginning of the flotation (2 and 5 minutes) the water recoveries are practically identical for all the tests (5%, 10%, 15% and 20% solids content). The situation in Figure 4.6 is very similar, the three grade-recovery curves for 10%, 15% and 20% are very close to each other. Only the 5% curve is different from the other three; the water recovery curves show the same trend as the grade-recovery curves. If we analyze the case of flotation at 5% solids content then it is quite obvious that when the water recovery increases the concentrate grade is reduced, which indicates entrainment. As Figure 4.6 reveals, the entrainment at a high pulp density (20%) is much higher. All this seems to indicate that the cases of flotation at the 10%, 15% and 20% solids content are so dominated by rheology of the system that all other effects seem to be secondary. Only the 5% solids content curve is “normal” and displays the trend that could be expected.

![Figure 4.7](image-url) Effect of flotation time and solids content on water recovery.
4.4 Effects of Solids Content in Flotation Stage on Pulp Rheology

The rheological curve of the flotation pulp is shown in Figure 4. 8.

![Rheological curve for the fine fraction of Mt Keith ore](image)

**Figure 4. 8** Rheological curves for the fine fraction of Mt Keith ore as a function of solids content.

The figure shows the effect of solids content on the rheological properties of the slurry of nickel ore (flotation feed). The curves indicate that the yield stress values increase markedly with increasing solids content especially when 15% solids content is exceeded. The figure also shows that with increasing solids content from 5 to 20%, almost-Newtonian flow behaviour changes to clearly non-Newtonian when solids content exceeds 10%. The curves reveal some thixotropy. The yield stress values imply that even at this low solids content some structure develops in the suspension.

The data was analyzed using the Casson model, and the Casson yield stress values are given in Table 4. 3 and Figure 4. 9. Since error bars were very small in these experiments, no vertical error bars are shown. As the solids concentration increases, so does the yield stress of the flotation pulp and also the viscosity. This proves the presence of aggregation at higher solids contents.
Table 4.3 Casson yield stress values versus solids content.

<table>
<thead>
<tr>
<th>Solids Content (%)</th>
<th>Casson Yield Stress (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.37±0.001</td>
</tr>
<tr>
<td>10</td>
<td>1.20±0.004</td>
</tr>
<tr>
<td>15</td>
<td>1.35±0.004</td>
</tr>
<tr>
<td>20</td>
<td>1.35±0.011</td>
</tr>
</tbody>
</table>

Figure 4.9 Casson yield stress of the flotation feed as a function of solids content.

The increased yield stress causes the damping of the turbulence in the flotation system described by Schubert, 2008. This has two effects: the decrease in the amount of air dispersion and the decrease in the kinetic energy of bubbles and particles. Therefore, the increase in the solids concentration in the flotation pulp gives rise to two opposing effects. The increase in the solids content improves flotation by increasing the probability of bubble/particle collision which appears from the data presented in Figure 4.4. However, over 15% solids content, the opposing effect related to viscosity of the system affects adversely the flotation.
Figure 4.10 Nickel recovery and grade as a function of solids content in agglomerate flotation. Solids contents are shown with different graphical symbols; 5% solid (♦), 10% solid (■), 15% solid (▲) and 20% solid (●).

The experimental points for 30 minutes flotation from Figure 4.6 are shown again in figure 4.10 as either cumulative nickel recovery or nickel grade plotted versus the slurry yield stress. With 20% solids content, the cumulative nickel recovery is lower compared to the tests conducted at other pulp densities. This implies that the effect of “boiling” on flotation is significant. In comparing these results with those in Figure 4.6, it is important to remember that these are 30 minute flotation results.

4.5 Effects of Dispersant Addition on Flotation Performance

Figure 4.11 shows the nickel recovery as a function of solids content under different conditions, indicating higher recoveries for the tests conducted without dispersant in process water (these are 30 minutes flotation results).
Figure 4.11 The effect of dispersant addition on nickel recovery in the agglomerate flotation as a function of solids content (400 g/t CMC).

The highest recoveries without dispersant in the process water likely result from higher recovery of non-selectively entrained particles in the system, which increases the total recovery but decreases the nickel grade. As Figure 4.12 shows the lowest concentrate grades were obtained when the flotation experiments were conducted without dispersant.

The highest grades (Figure 4.12) were found in the tests conducted with CMC in distilled water, which are characterized by the lowest recoveries. Wellham et al. (1992) tested flotation of the nickel sulfide ore from Western Australia and concluded that the flotation performance was much better when the tests were conducted in saline pulp (10% NaCl) without dispersant. These results were better than the results of the tests with CMC (1kg/t). However, the findings of these thesis (Figure 4.11 and Figure 4.12) show that the tests conducted with CMC in distilled water seem to be successful. Since the use of CMC in the flotation of this type of ore in distilled water was shown to improve flotation of pentlandite (Edwards et al., 1980) the lack of beneficial effects in these tests can possibly be explained by a high ionic strength of the process water.
Figure 4.12 The effect of dispersant addition on nickel grade as a function of solids content (400 g/t CMC).

Figure 4.13 The effect of dispersant on the Casson yield stress under different conditions as a function of solids content (400 g/t CMC).

Figure 4.13 shows the relationship between Casson yield stress and solids content in the pulp under different conditions. The detailed results of the tests are given in APPENDIX.
IV. Figure 4. 13 points out that the Casson yield stress increases as the solids content increases.

The highest yield stresses were observed in distilled water without any dispersant whereas the lowest values were found in the process water (without dispersant). In distilled water, positively charged lizardite and chrysotile particles are likely to aggregate on the surface of negatively charged pentlandite (Figure 2. 24). With increasing ionic strength, the double layer is compressed and this decreases the range of the attractive electrical forces. In accordance with Edwards et al (1980) the addition of CMC to the flotation pulp increases selectivity due to dispersion of slimes; under such conditions the measured yield stress values decrease.

Figure 4. 14 shows the relationship between nickel recovery and Casson yield stress. These nickel recovery curves show the same trends as those observed in Figure 4. 11. The effect of CMC on the concentrate grade is shown in Figure 4. 14.

![Figure 4. 14](image)

**Figure 4. 14** Nickel recovery versus Casson yield stress under different conditions as a function of solids content. 5% solid (♦), 10% solid (■), 15% solid (▲) and 20% solid (●).

Figure 4. 15 shows the effect of pulp rheology on grade. The figure indicates that the nickel grade decreases with the yield stress value for all conditions. This would be explained by the recovery of higher amount of impurities with increasing solids content.
Figure 4.15 The effect of dispersant addition on nickel grade and pulp rheology under different test conditions as a function of solids content. 5% solid (♦), 10% solid (■), 15% solid (▲) and 20% solid (●).

4.6 Effect of Shearing on Rheological Properties of the Tested Flotation Pulp

Figure 4.16-Figure 4.21 show the results of the stress decay tests at shear rates of 10, 50 and 100 s⁻¹ in the absence of dispersant. The tests were carried out with the finely ground flotation feed either in distilled or in process water. These figures demonstrate that apparent viscosity of the suspension decreases with time of shearing for each condition and assumes constant values after a long equilibration time. The tested slurry thus exhibits thixotropic time dependent properties. The time dependency tests were also conducted with addition of CMC in both distilled and process water in order to investigate the effects of dispersants on rheology at different shear rates. Results show that the difference in the apparent viscosity was found more obvious at lower shear rates, but the difference was disappeared with increasing shearing rate.
Without dispersant:

**Figure 4.16** Apparent viscosity versus time of shearing at $10s^{-1}$ shear rate at 20% solids content either in the presence and absence of the dispersant in distilled water.

**Figure 4.17** Apparent viscosity versus time of shearing at $10s^{-1}$ shear rate at 20% solids content either in the presence or absence of the dispersant in process water.
Figure 4. 18 Apparent viscosity versus time of shearing at 50 s$^{-1}$ shear rate at 20% solids content either in the presence or absence of the dispersant in distilled water.

Figure 4. 19 Apparent viscosity versus time of shearing at 50s$^{-1}$ shear rate at 20% solids content either in the presence and absence of the dispersant in process water.
Figure 4. 20 Apparent viscosity versus time of shearing at 100 s$^{-1}$ shear rate at 20% solids content either in the presence or absence of the dispersant in distilled water.

Figure 4. 21 Apparent viscosity versus time of shearing at 100s$^{-1}$ shear rate at 20% solids content either in the presence or absence of the dispersant in process water.
Aggregated systems usually display very high viscosities at low shear rate which can be seen in the figures (Figure 4.16-Figure 4.21), and are very shear thinning. At low shear rates, the aggregates form weak networks which give the suspension its structural properties. The apparent viscosity starts to decrease with increasing shear rate, reflecting the tendency of anisotropic particles to be aligned by the shear and thereby offer an average volume lower than that provided by the same particles when randomly oriented. When aggregated slurries are sheared, the aggregates rotate, possibly deform and, if the applied stress is high enough, begin to break down. All aggregated structures take time to break down, and thixotropic behaviour is usually associated with the aggregated suspensions. The decreasing viscosity with time can be explained by assuming that the loose aggregates of gangue are broken up by increasing shear rate. Michaels and Bolger (1962) pointed out that the broken aggregates by shearing forces release trapped water reducing the effective volume concentration. This phenomenon is illustrated in Figure 4. 22 which shows the stages of breaking of aggregates of long fibrous chrysotile particles with increasing rate of shear. The small points symbolize the nickel-sulfide particles. The tangled chrysotile and nickel-sulfide particle aggregates start to break up with increasing shear rate.

![Image](image-url)

**Figure 4. 22** The illustration of break-up process of agglomerates

The results for the tests that were conducted at 15 and 20% solids content without dispersant either in distilled or in process water are replotted in the 3-D form and shown in Figure 4. 23-Figure 4. 26. The results for tests which were conducted with dispersant either in distilled or in process water are shown in Figure 4. 27-Figure 4. 30.
Without dispersant:

Figure 4.23 Effect of time of shearing at 15% solids on the rheological curves for the tested flotation pulp. The tests were carried out in distilled water without dispersant.

Figure 4.24 Effect of time of shearing at 15% solids on the rheological curves for the tested flotation pulp. The tests were carried out in processed water without dispersant.
Figure 4.25 Effect of time of shearing at 20% solids on the rheological curves for the tested flotation pulp. The tests were carried out in distilled water without dispersant.

Figure 4.26 Effect of time of shearing at 20% solids on the rheological curves for the tested flotation pulp. The tests were carried out in processed water without dispersant.
With dispersant addition:

**Figure 4.27** Effect of time of shearing at 15% solids on the rheological curves for the tested flotation pulp. The tests were carried out in distilled water with dispersant (400 g/t CMC).

**Figure 4.28** Effect of time of shearing at 15% solids on the rheological curves for the tested flotation pulp. The tests were carried out in processed water with dispersant (400 g/t CMC).
Figure 4. 29 Effect of time of shearing at 20% solids on the rheological curves for the tested flotation pulp. The tests were carried out in distilled water with dispersant (400 g/t CMC).

Figure 4. 30 Effect of time of shearing at 20% solids on the rheological curves for the tested flotation pulp. The tests were carried out in processed water with dispersant (400 g/t CMC).
The measured shear stresses in the tests conducted in process water without dispersant were higher than those in the distilled water. The figures demonstrate that under the intense shearing conditions of the flotation tests in the cell mechanical properties of the tested slurry, mostly resulting from the size and shape of the particles, are a dominant factor in this case. For this suspension containing mostly fibrous gangue particles the differences between the rheological properties as caused by different physico-chemical conditions are negligible.

All these results imply that mechanical properties of the tested slurry, mostly resulting from the size and shape of the particles, are a dominant factor in this case. The time dependent properties can be explained in terms of the time required for particle orientation to occur. These anisotropic fibrous particles tend to align in the direction of flow and the viscosity decreases with time. In time dependency tests, it was observed that the viscosity decreases with time for variable solids concentrations, then become constant (after 20-40 seconds), which means that particles align with the flow and reach the plateau.

Plastic viscosity of suspensions is a function of solids content

\[ \eta_p = f(\phi) \]  \[4.1\]

where \( \phi \) is the solid volume fraction.

The viscosity at a high solid concentration is governed by the solid packing fraction \( p \); beyond this solid fraction the dispersed particles lock into a rigid structure and flow ceases. For each particle size distribution, and for each particle shape there is characteristic packing fraction (for instance, the maximum packing fraction for hexagonally packed monodisperse spheres \( p = 0.74 \)). Suspension viscosity starts dramatically increasing when the solids content approaches that of \( \Phi = p \) for a given suspension. Rand and Melton (1977) assumed that suspension plastic viscosity can be described by the Einstein expression:

\[ \eta_p / \eta_w = 1 + k\phi \]  \[4.2\]

where
- \( \eta_p \) is plastic viscosity
- \( \eta_w \) is viscosity of water
- \( \Phi \) is volume fraction
- \( k \) is the shape factor

This equation is used to calculate the \( k \) values. The authors found that the calculated values of \( k \) for kaolinite suspensions were the largest when the coagulating kaolinite particles formed the very voluminous edge-face aggregates.
Table 4.4 The effect of chrysotile on the apparent viscosity of the pure chrysotile aqueous suspension (Ndlovu, 2008).

<table>
<thead>
<tr>
<th>Solids Content (%)</th>
<th>Bingham Yield Stress (Pa)</th>
<th>Bingham Viscosity (Pas)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>0.17</td>
<td>0.07</td>
</tr>
<tr>
<td>0.2</td>
<td>0.40</td>
<td>0.78</td>
</tr>
<tr>
<td>0.3</td>
<td>1.21</td>
<td>0.89</td>
</tr>
<tr>
<td>0.4</td>
<td>2.02</td>
<td>1.80</td>
</tr>
<tr>
<td>0.5</td>
<td>2.97</td>
<td>3.16</td>
</tr>
<tr>
<td>0.6</td>
<td>4.03</td>
<td>3.85</td>
</tr>
<tr>
<td>0.7</td>
<td>5.61</td>
<td>1.90</td>
</tr>
<tr>
<td>0.8</td>
<td>15.49</td>
<td>8.32</td>
</tr>
</tbody>
</table>

The fibrous structure of chrysotile is generally believed to be the cause of its problematic rheological behaviour. Rheological properties of chrysotile suspensions were experimentally studied by Ndlovu (personal communication, 2008). The tests were performed as a function of chrysotile content at pH 9. Table 4.4 shows an increase in both Bingham yield stress and plastic viscosity with increasing solids content. It can be seen that maximum yield stress and viscosity values were obtained at 0.8% solids concentration (by volume). The $k$ values for the chrysotile suspensions were calculated using Ndlovu’s data (see Table 4.4) following the example provided by Rand and Melton (1977). For the highest volume fraction the value of $k$ of 83123 was obtained. This is much higher than the values provided for kaolinite by Rand and Melton and it seems to indicate the nature of the rheological properties of the suspensions formed by this mineral.

To realize how different the chrysotile suspensions are from other mineral systems it is enough to inspect Figure 4.31. The volume content was calculated from the weight content for nickel-sulfide ore assuming that the density of the ore is close to that of chrysotile ($d_{\text{chry}} = 2.63$). In the tests with pure chrysotile the pulp with solids content around 0.8% by volume is close to the critical packing fraction. At the solids content exceeding this value, the suspensions were found to be too viscous for performing rheological measurements. This is likely to be a result of the flexibility and subsequent high degree of aggregation between fibers which increases as the number of fibers in solution is increased. However, in the tests with the nickel ore containing more than 80% of chrysotile and the viscosity is still far from the critical packing even at 7.6 % solids content (by volume). This shows a very beneficial dilution effect; the presence of other non fibrous minerals dramatically decreases the viscosity of the chrysotile suspensions. In this regard, the ore displays less problematic rheological behavior than the pure chrysotile.
Figure 4.31 The effect of the chrysotile amount on the pulp rheology.
CONCLUSIONS

The kinetic tests carried out at varying pulp density, in flotation experiments revealed that at low pulp density (5%) flotation was fast during the first 2 minutes of flotation; the differences between the results of the tests conducted under various conditions were small after 30 minutes of flotation. While the ratio of the true flotation recovery to the recovery by entrainment was not changing much with time at low pulp density (5%) it was much lower for the tests carried out at 20% solids content. At this high pulp density the entrainment was very significant in spite of much lower water recovery under such conditions. This is in line with high viscosity values.

Newtonian behavior deviated to non-Newtonian when solids content exceed 10% and thixotropic behavior was observed at 20% solids content. The observed “boiling” in the flotation tests which were carried out at high pulp density (20%) likely results from a high pulp viscosity; this leads to the dampening of the turbulence in the flotation system described in literature.

Regarding the effect of dispersant on the flotation performance, the highest recovery was obtained using process water without CMC. This could be explained by increased recovery of non-selectively entrained particles in the system. While this improves recovery the concentrate grade is decreased. The highest entrainment was also observed in the tests conducted without CMC in the process water, which explains the low concentrate grades.

Use of CMC in distilled water provided the highest nickel grades of concentration. It was expected that the addition of CMC to the flotation pulp increased selectivity due to a better slime dispersion since the presence of CMC reduced the yield stress values. The highest yield stress values were obtained in the tests without CMC in distilled water and the yield stresses decreased with CMC addition.

The rheological tests show that increasing solids content leads to a higher yield stress and viscosity. The presence of chrysotile mineral strongly affects the pulp rheology. The increase in the viscosity becomes significant when the solids content approaches maximum packing fraction. When the solids content approaches 0.8% (by volume), the viscosity of chrysotile suspension becomes very high. This seems to indicate that the problematic rheology of the slurry is a result of the shape of chrysotile particles. The shape factor, calculated using Einstein equation, assumes as large values as 83123 for chrysotile suspension for further confirming unusual shape of the particles of this mineral. It was also found that an increase in the concentration of chrysotile has the most dramatic effect on the yield stress (its
maximum packing fraction is around 0.8% chrysotile by volume). The viscosity of pure chrysotile suspensions cannot be measured at higher than 0.8% chrysotile contents, in the case of the tested nickel ore that contains not only chrysotile but also other minerals, this effect is not so severe. Rheological tests could be conducted for nickel-sulfide ore even at 20% solids content by weight that is about 7.6% by volume. This shows a very beneficial “dilution effect” for processing this ore; the presence of other non-fibrous minerals dramatically decreases the viscosity of the chrysotile suspensions. Without such minerals processing of this ore would have been entirely impossible.
6 RECOMMENDATIONS FOR FUTURE WORK

Conditioning of this ore on one hand leads to aggregation of nickel sulfide particles but on the other it strongly reduces viscosity of this pulp. Both effects are beneficial for processing of the studied ore and new tests should be designed to study these two effects separately.
REFERENCES


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APPENDIX I : Mineral composition of nickel-sulfide ore

Figure I-1 Mineral composition of nickel-sulfide ore
Table II-1 The effect of dispersant addition on flotation performance either in process and distilled water.

<table>
<thead>
<tr>
<th>Solids Content (%)</th>
<th>Nickel Grade (%)</th>
<th>Nickel Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>3.14±0.03</td>
<td>62.07±2.16</td>
</tr>
<tr>
<td>10</td>
<td>2.51±0.58</td>
<td>63.63±2.71</td>
</tr>
<tr>
<td>15</td>
<td>1.76±0.20</td>
<td>66.63±2.57</td>
</tr>
<tr>
<td>20</td>
<td>1.65±0.02</td>
<td>69.54±1.33</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Solids Content (%)</th>
<th>Nickel Grade (%)</th>
<th>Nickel Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>3.16±0.26</td>
<td>62.86±3.03</td>
</tr>
<tr>
<td>10</td>
<td>3.44±0.84</td>
<td>58.58±2.79</td>
</tr>
<tr>
<td>15</td>
<td>2.45±0.29</td>
<td>50.59±1.96</td>
</tr>
<tr>
<td>20</td>
<td>2.24±0.02</td>
<td>53.96±6.73</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Solids Content (%)</th>
<th>Nickel Grade (%)</th>
<th>Nickel Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>3.14±0.03</td>
<td>62.07±2.16</td>
</tr>
<tr>
<td>10</td>
<td>2.51±0.58</td>
<td>63.63±2.71</td>
</tr>
<tr>
<td>15</td>
<td>1.76±0.20</td>
<td>66.63±2.57</td>
</tr>
<tr>
<td>20</td>
<td>1.65±0.02</td>
<td>69.54±1.33</td>
</tr>
</tbody>
</table>
APPENDIX III: Effect of dispersant addition on pulp rheology

Table III-2 The effect of dispersant addition on rheology either in process and distilled water.

With CMC in process water

<table>
<thead>
<tr>
<th>Solids Content (%)</th>
<th>Casson Yield Stress (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.37±0.02</td>
</tr>
<tr>
<td>10</td>
<td>1.02±0.13</td>
</tr>
<tr>
<td>15</td>
<td>2.27±0.41</td>
</tr>
<tr>
<td>20</td>
<td>3.32±0.15</td>
</tr>
</tbody>
</table>

With CMC in distilled water

<table>
<thead>
<tr>
<th>Solids Content (%)</th>
<th>Casson Yield Stress (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.38±0.01</td>
</tr>
<tr>
<td>10</td>
<td>1.24±0.05</td>
</tr>
<tr>
<td>15</td>
<td>2.58±0.42</td>
</tr>
<tr>
<td>20</td>
<td>3.14±0.25</td>
</tr>
</tbody>
</table>

Without dispersant in process water

<table>
<thead>
<tr>
<th>Solids Content (%)</th>
<th>Casson Yield Stress (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.37±0.001</td>
</tr>
<tr>
<td>10</td>
<td>1.20±0.004</td>
</tr>
<tr>
<td>15</td>
<td>1.34±0.004</td>
</tr>
<tr>
<td>20</td>
<td>5.01±0.010</td>
</tr>
</tbody>
</table>

Without dispersant in distilled water

<table>
<thead>
<tr>
<th>Solids Content (%)</th>
<th>Casson Yield Stress (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.40±0.07</td>
</tr>
<tr>
<td>10</td>
<td>1.29±0.12</td>
</tr>
<tr>
<td>15</td>
<td>3.56±0.05</td>
</tr>
<tr>
<td>20</td>
<td>5.78±0.37</td>
</tr>
</tbody>
</table>
Figure III- 41 through Figure III- 4 show the effect of dispersant on viscosity as a function of solids content under different conditions.

**Figure III- 1** The relationship between apparent viscosity and shearing time (between 0 and 200 s⁻¹) for 5% solids content.

**Figure III- 2** The relationship between apparent viscosity and shearing time (between 0 and 200 s⁻¹) for 10% solids content.
Figure III- 3 The relationship between apparent viscosity and shearing time (between 0 and 200 s$^{-1}$) for 15% solids content.

Figure III- 4 The relationship between apparent viscosity and shearing time (between 0 and 200 s$^{-1}$) for 20% solids content.