POWER DRAW MEASUREMENTS AS A METHOD OF CHARACTERIZATION OF
PARTICLE AGGREGATION

by

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ABSTRACT

Power consumption is one of the critical parameters in optimization of industrial operations. In the present work, the power draw measurements were used as a method of characterizing and optimizing particle aggregation. A series of agglomerate flotation and oil agglomeration tests were carried out to find a correlation between power draw, particle aggregation and flotation performance.

The experimental program included the agglomerate flotation of a complex nickel sulfide ore with fibrous gangue minerals, oil agglomeration of a fine suspension of bituminous coal used as model systems and oil agglomeration tests on a mixed mineral system with various types of gangue minerals. The effect of the gangue properties on oil agglomeration were studied by changing the coal-to-gangue ratio and the type of the gangue. Power draw measurements were conducted with either a turn-table or an electrical method to characterize and optimize agglomeration.

The results indicate that the system behavior was strongly dependent on the mineralogical composition of the ore. The presence of fibrous gangue minerals in the flotation feed produced by grinding the nickel sulfide ore had a dominating effect on the performance of the flotation process.

Power draw measurements were found to be much more useful in system optimization when compared to the characterization of particle agglomeration. The small portion of nickel sulfide particles in the ore and the complex entangled structure resulting from the presence of large amounts of fibrous gangue minerals probably made the agglomeration process difficult to be followed by power draw measurements. In the case of a simple coal-oil agglomeration system, the power draw measurements were sensitive enough to follow the oil agglomeration process. Concentration (valuable-to-gangue particle ratio), size and shape of these gangue minerals were the factors which affected the system significantly.
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1. INTRODUCTION

Particle size is one of the most important parameters in flotation and it has been well established that the effectiveness of flotation is limited to a particle size range specific to each flotation system.

Fine grinding is commonly required to achieve sufficient liberation. However, fine particle flotation has always been a challenge in mineral processing. Very fine particles, even if hydrophobic, float poorly owing to their small mass, large surface area and high surface energy (Sivamohan, 1990). It is generally accepted that low collision probability limits the flotation of fine particles and the only effective method to increase the flotation rate is to increase the particle size by selective aggregation of fine particles.

Oil agglomeration is a common technique for increasing the size of hydrophobic particles and thereby improving flotation. Oil droplets when present in a mineral suspension tend to attach to hydrophobic particles and may cause agglomeration depending on hydrodynamic conditions (Laskowski, 1992).

In agglomerate flotation, size of the particles is increased by hydrophobic aggregation of desired mineral fines and then the agglomerates are recovered by flotation. The agglomeration process is selective since only very hydrophobic particles can be agglomerated with oil.

The efficiency of agglomerate flotation strongly depends on the properties of the ore. Sulfides can be agglomerated with oil when rendered hydrophobic (House and Veal, 1989; Kocabab et al., 1990; Laskowski and Lopez-Valdivieso, 2004). Pentlandite is one of the main sources of nickel. It occurs with other sulfides, such as pyrrhotite and chalcopyrite, and with various silicate gangue minerals. Low grade nickel ores containing large amounts of magnesium-silicate minerals have historically been difficult to process. The gangue portion including serpentine minerals, with their mineralogical diversity and metallurgical complexity often interfere with the concentration of nickel sulfides. Suspensions of serpentine minerals are characterized by high viscosity; the problem is further aggravated when fine grinding is required. Estimation of the system behavior is very difficult when the particles have an unusual shape and are anisotropic as chrysotile.
The kinetic energy of interacting particles is one of the most important parameters in determining the probability of particle aggregation. This energy can be altered by adjusting the intensity of conditioning. It is therefore obvious that the power input in conditioning affects the aggregation process. In the tests described in this thesis, power draw was monitored to find the optimum agglomerate flotation conditions. The objective was better understanding of the correlation between power draw and agglomerate flotation performance. Power consumption is one of the critical parameters in industrial operations and power draw measurements are needed in any optimization attempt.

The power consumption of impeller is the amount of power required to rotate an impeller at a given speed in a tank with a specific configuration and containing a fluid of certain physical properties (Mhaisalkar et al., 1985). Since the power draw during conditioning depends on the pulp viscosity, which in turn changes with particle aggregation, the measurements could also be used to characterize particle aggregation. The power draw measurements have been shown to be a promising technique for characterization of agglomeration (Bensley et al., 1977; Swanson et al., 1977; Lapidot and Mellgren, 1978; Szymocha et al., 1989) and it can be measured mechanically using a turn-table setup, or using electrical methods. Both methods were used in this study.

The aggregation of particles, even if very hydrophobic, was found to be dependent on the valuable-to-gangue particle ratio. The role of gangue minerals and their effect on the oil agglomeration process has never been studied extensively. To better understand the possible effect of gangue on agglomeration of nickel sulfides, the test program was later modified to include a coal-oil model agglomeration system. By changing the coal-to-gangue ratio and the type of gangue, it was then possible to study the effect of the gangue properties (particle shape) on oil agglomeration.
1.1 OBJECTIVES

The objectives of this study include:

− testing the use of power draw measurements as a way of characterizing and optimizing fine particle agglomeration,
− finding the possible correlation between power draw and agglomerate flotation performance,
− investigating the effect of power input on degree of aggregation and its relation with the flotation performance.

1.2 HYPOTHESES

The thesis is based on the following hypotheses:

1) **Power draw in the conditioning stage affects particle aggregation.** In oil agglomeration, aggregation of particles is affected by the collision of particles. Particles should be able to react with reagents and collide with each other. Collision probability is determined by the kinetic energy of particles which can be altered by the intensity of conditioning. The intensity of conditioning can be monitored and controlled through the power draw measurements. Therefore, it can be assumed that the power draw in the conditioning stage affects the aggregation process.

2) **Particle aggregation results in the formation of a structure between aggregating particles, that would give higher power draw and in turn better flotation performance.** Power consumption should increase due to an increase in aggregation and flotation performance should improve due to an optimal particle size.

3) **There exists an optimum energy input that would result in maximum particle aggregation without breaking up the aggregates.**
2. LITERATURE REVIEW

2.1 EFFECT OF PARTICLE SIZE ON FLOTATION

Liberation in mineral processing is essential and it is achieved by crushing and grinding of the ore. Size reduction and flotation processes are closely interrelated. Grinding liberates valuable components from the host rock whereas flotation separates them into valuable minerals and gangue. Optimum performance of mineral processing plants requires optimum size reduction; both unnecessary overgrinding (high cost, slow flotation) and insufficient grinding (poor liberation) must be avoided.

Particle size is an important parameter in flotation and has been the focus of the flotation research for decades. Perhaps, the first detailed study on the effect of particle size on flotation was carried out by Gaudin et al. (1931). This study analyzed the performance of a number of concentrators and the data indicates the existence of an optimal size range in the flotation of lead, zinc and copper sulfides.

It is now well established that the effectiveness of flotation is limited to a particle size range between 10-100 µm (Trahar and Warren, 1976; Smith and Warren, 1989) and there is an optimum particle size range for each flotation system.

The effect of particle size on flotation is commonly presented as the recovery-size curves in which the recovery from a particular size fraction is plotted against the average size of particles in that fraction. These recovery versus particle size curves have a characteristic shape, as illustrated in Figure 1, which can be divided into three regions. The region below 5-10 µm comprises the fines which are difficult to float; the region from 10 µm to around 70 µm comprises the intermediate particles which are usually the most floatable and in the region above 70 µm flotation again becomes difficult (Trahar, 1981).
The particle size range with maximum floatability depends on the density of the particles. For instance, in the case of coal, high recoveries can be achieved at particle sizes up to 500 µm. For lead flotation, the optimum particle size lies between 5 and 70 µm.

Even though the optimum particle size range can differ for different minerals, the shape of recovery-size curves is usually fairly stable for a given set of flotation conditions. Outside the optimum size range, flotation recoveries of both fine and coarse particles decrease for various reasons. The difficulty in floating fine particles is attributed to the low probability of bubble–particle collision, while the problem with coarse particle flotation is due to detachment.

It is generally accepted that for a particle to be collected by a bubble, there must be:

a. a collision between particle and bubble \( (E_C) \),
b. an adhesion (attachment) step where particle and bubble form an aggregate \( (E_A) \),
c. a formation of bubble-particle aggregate which is stable enough to resist the action of detachment forces. \( (E_S) \) (Whelan and Brown, 1956; Derjaguin and Dukhin, 1961; Jameson et al., 1977; Schulze, 1984; Spedden and Hannan, 1984; Dobby and Finch, 1987)
Collision of a particle with a bubble is determined by hydrodynamics and inter-particle interactions. Once the particle approaches the bubble, the surface forces become significant at a shorter separation distance and the attachment process begins. The success of the flotation process depends on the stability of the particle-bubble aggregates that are formed as a result of this collision and attachment process. In the case of coarse particles, the bubble–particle aggregates may not be strong enough to prevent the particle detachment from the bubble surface. The inability of the bubbles to lift the attached particles causes losses in the recovery of coarse particles.

Recovery rates are also affected by the disruption of bubble-particle aggregates in turbulent zones (Morris, 1950; Schulze, 1977). In mechanical flotation cells, high shear rates may break the formed particle-bubble aggregates. As illustrated in Figure 2, a particle-bubble couplet caught in a turbulent eddy will rotate with a frequency appropriate to the eddy size, and if the kinetic energy of the particle exceeds the work of rupture, the particle will detach (Schulze, 1984).

![Figure 2](image_url)

**Figure 2** – Mechanism of detachment of a particle from a bubble in a turbulent eddy; a. The bubble rotating around its centre. b. The forces acting on the particle (Ahmed and Jameson, 1989). (Courtesy of Taylor&Francis)

The performance of mechanical cells deteriorates with increasing particle size. As the particles in the flotation feed become coarser, higher impeller speeds are required to keep the particles in suspension and off the bottom of the cells. However, it is not possible to adjust
impeller speed without affecting other functions such as, bubble creation and bubble–particle collision/attachment/detachment.

The probability of a stable particle-bubble aggregate formation depends not only on the particle size and external detaching forces, but also on particle hydrophobicity. Another possible way of improving the recovery of coarse particles is to increase the hydrophobicity with collector addition. The degree of hydrophobicity required to promote a high level of floatability increases with the particle size. Trahar (1981) showed that as the amount of collector added to the system increases, the region of maximum floatability becomes progressively wider. Figure 3 demonstrates the influence of xanthate dosage on the recovery of chalcocite. The effect of increased collector dosage on finest material was less when compared to the coarse size ranges.

![Figure 3](image.png)

**Figure 3**—Response of a chalcocite ore to collector addition (Trahar, 1981). (Courtesy of Elsevier)

Chen *et al.* (1999) studied the effect of high intensity conditioning on flotation of a nickel ore with a size-by-size analysis. Significant decrease in the recovery was observed for pentlandite particles larger than 106 µm, under all experimental conditions. The particles being too large for sufficient bubble-particle stability and the incomplete liberation of pentlandite particles were the reasons for this behavior.
Incomplete liberation is another problem that is common in coarse particle flotation. There must be sufficient exposure of the target mineral on the particle surface for selective flotation to take place. Thus, another way of improving the flotation recoveries of coarse particles is by further grinding the ore to improve the liberation degree of particles.

2.2 FLOTATION OF FINE PARTICLES

2.2.1 PROBLEMS IN FINE PARTICLE FLOTATION

Flotation plants have been historically designed to recover medium-sized particles characterized by good liberation and high flotation rate constant. In almost all mineral processing operations, conditioning and flotation stages have not been properly optimized for the fine fractions (Trahar, 1981; Rubio et al., 2003). Fine particles mostly show slow recovery rates owing to their small mass, high surface area and high surface energy.

Small mass leads to: (Sivamohan, 1990)
- Low particle momentum,
- Low probability of collision with a bubble,
- Particle entrainment in concentrates,
- Heterocoagulation.

High surface area leads to:
- High reagent consumption,
- High dissolution rate in water,
- Rigidity of froth (Froth stabilization),
- High pulp viscosity.

High surface energy leads to:
- Higher dissolution (Increased dissolution from the surface of fine particles may introduce undesirable ions into solution, affecting collector-mineral interactions),
- Rapid oxidation (Unintentional activation of undesirable minerals or depression of desired minerals),
- Non-specific collector adsorption,
- Froth stabilization.
A schematic diagram to illustrate the relationship between the physical and chemical properties of fine particles and their behavior in flotation is given in Figure 4.

Figure 4 - The relationship between the physical and chemical properties of fine particles and their behavior in flotation. (G) and (R) refer to whether the phenomena affect grade and/or recovery respectively (Fuerstenau, 1980). (Courtesy of Society of Mining Engineers Publishing)

The response of fine particles to collision, attachment and detachment sub-processes is different from the response of coarse particles. Some factors which have a vital importance in coarse particle flotation can be negligible in fine particle flotation. For instance, the particle-bubble detachment is one of the most challenging problems in coarse particle flotation. On the other hand, it is usually negligible in fine particle flotation, since the attached particles are small enough to prevent the particle detaching from the bubble surface under gravitational and dynamic forces existing in flotation cells.

The recovery of fine particles requires an effective particle-bubble collision. The collision step has been the most extensively investigated among the sub-processes of particle-bubble interactions. Sutherland (1948) proposed a model for collision efficiency as a function of the ratio of particle diameter over bubble diameter for potential flow around the bubbles and for solid particles. After this pioneering work of Sutherland (1948), several researchers have studied the consecutive stages of particle-bubble collision and numerous models have been proposed for the evaluation of the collision efficiency (Gaudin, 1957; Derjaguin and Dukhin, 1961; Flint and
Howarth, 1971; Yoon and Luttrell, 1989B). Any comparison between the collision efficiency models is difficult since they were derived with different assumptions (Dai et al., 2000). Yet, all of these collision models predict that the collision efficiency increases with particle size.

Unlike the bubble-particle collision sub-process, an attachment strongly depends on the surface chemistry of a solid particle and a bubble (Nguyen et al., 1998). The difficulty in overcoming the energy barrier between a particle and bubble causes low adhesion probability and results in low recoveries.

Mineral particles can be recovered by two mechanisms; true flotation or entrainment. The recovery of fines is often due to water recovery or entrainment rather than genuine flotation via bubble attachment (Trahar, 1981). Figure 5 shows the entrainment mechanism in which fine hydrophilic particles (white-colored) are dragged from the pulp into the froth zone in the interstitial liquid. While true flotation is a selective process, entrainment is a non-selective process which can account for the low selectivity.

![Diagram of flotation recovery](image-url)

**Figure 5**— Flotation recovery in the froth by true flotation($R_F$) and entrainment($R_E$). Black-colored particles represent the hydrophobic particles and white particles represent the fine hydrophilic particles: (a) flotation froth, (b) froth lamella(Schubert, 2008). (Courtesy of Elsevier)

The impact of entrainment on fine particle flotation is evident from the study of Engelbrecht, Woodburn (1975) and Subrahmanyam, Forssberg (1988b). Engelbrecht and Woodburn observed a linear relationship between entrainment and water recovery in cases of hydrophobic fine pyrite ($<7$ µm) and silica ($< 12$ µm). The recovery of coarse pyrite was found to be dependent on hydrophobicity. A similar observation was made by Subrahmanyam and
Forssberg (1988b) in an investigation on the performance of different flotation frothers with a copper ore containing mostly graphite and silica.

Since the entrainment mechanism was found to be related to water recovery, any variable that can change the recovery of water will also change the recovery of fine particles. The variables that affect the water recovery can be manipulated to obtain better grades (Trahar, 1981).

Residence time is one of those variables. In the study of Subrahmanyam and Forssberg (1988a), it was observed that higher recoveries and low grades were obtained with short froth residence times whereas long froth residence times resulted in higher grade concentrates with less entrained minerals.

The other problem of fine particles is the high surface area. Fine particles consume excessive amount of collector due to their high surface area and show a tendency to form slime coatings on coarse material. When a mixture of fine and coarse particles come in contact with a collector at a given dosage, most of the collector will be consumed by the fine particles and will leave no sufficient amount available for coarse particles (Bazin and Proulx, 2001).

The large surface area of fine particles can also cause high dissolution rate in water, high pulp viscosity and can increase rigidity of froth (Sivamohan, 1989). These factors can have a considerable impact on grades and recoveries, depending on the dominant effects in operation.

### 2.2.2 METHODS TO IMPROVE FLOTATION OF FINE PARTICLES

In order to reduce the losses of valuable fine particles, fine particle generation must either be prevented or the recovery of the fines must be improved.

The fine particle generation can be reduced by controlling and optimizing grinding stage. However, in the case of some complex ores, fine grinding is necessary in order to obtain proper liberation.

There are several ways of improving the flotation response of fine particles (Figure 6). These include reduction of the size of air bubbles that increase the probability of collision and generation of gas bubbles that will directly nucleate on hydrophobic surface as in vacuum flotation.
Particle–bubble interaction is a fundamental sub-process in flotation. The recovery of fine particles can be increased by optimizing the capture between a bubble and a hydrophobic fine particle.

For an efficient size-by-size flotation, there must be a wide bubble size distribution (Yoon, 2000; Franzidis and Manlapig, 1999; Phan et al., 2003). Since the ore feed is composed of a wide particle size distribution, the flotation cell should also have a wide bubble size distribution, including fine bubbles which are suitable to catch the fine particles (Zhou et al., 1997, Yoon, 2000; Rubio et al., 2003; Rubio et al., 2007). However, conventional flotation cells do not provide the required bubble size distribution. Therefore, other options to improve selective recovery must be taken into account such as increasing the probability of collision.

There are several ways to increase the collision probability: (Schubert, 2008)

a. Using an efficient impeller-stator system,

b. Increasing the power input by means of a high intensity conditioning,

c. Dispersing the flotation slurry,

d. Reducing the turbulence damping.

It is important to use an efficient impeller-stator system having a large power number at an adequately high rotational speed. Moreover, a satisfactory dispersion state in the pulp is

---

**Figure 6** – Methods of improving selective recovery of fines.
needed to improve rheological properties (low apparent viscosity) and also to reduce turbulence damping (Schubert, 2008).

Column flotation with its counter-current principle increases the probability of particle-bubble collisions. It is also possible to increase the chance of collision by increasing the residence time. The recovery of fine sized particles requires long residence times in flotation cells. With longer residence times, the chance of particles getting attached to the bubbles increases and therefore, the recovery may increase. However, this solution is not economically feasible when considering long process times and high energy consumption.

In the flotation of fines, the problem is mainly due to a slow flotation rate and therefore, the real improvement requires an increased flotation rate. The only effective method to achieve better results in fine particle flotation is to increase the particle size by selective aggregation.

2.3. AGGLOMERATION

In an agglomeration process, fine particles in a dispersed system are aggregated into large aggregates. These large aggregates, when hydrophobic, float well due to their increased particle momentum and have a higher probability of collision with bubbles. The agglomerates interact with the air bubbles instead of individual fine particles, avoiding the problem of low particle-bubble collision and also reducing the entrainment.

The dependence of the flotation on particle size can be explained by the equation;

\[ P = P_c P_a (1 - P_d) \]  

in which \( P_c \) is the probability of bubble-particle collision, \( P_a \) the probability of adhesion, and \( P_d \) the probability of detachment (Schuhman, 1942; Sutherland, 1948). Providing that the particles are very fine and sufficiently hydrophobic, detachment forces can be neglected and it can be assumed that the probability of formation of a stable particle-bubble aggregate can be equal to 1. Under such conditions \( P \) in Equation 1 is given by

\[ P = P_c P_a \]

Various hydrodynamic models suggested that the probability of particle-bubble collision can be summarized in the form

\[ P_c = A \left( \frac{d_p}{d_b} \right)^n \]

in which \( A \) is a function of the Reynolds number of the bubble and \( n = 2 \) for the bubbles used in flotation (Yoon, 1989B). This equation shows that the particle-bubble collision depends on
particle size \((d_p)\), bubble size \((d_b)\) and also on the stream function of the fluid flowing past the bubble.

Accordingly, it is expected that by selective aggregating the fine particles and increasing the aggregate size, the probability of a particle being collected would increase and thus flotation kinetics should improve (Equation 4).

\[
P_c \propto \left( \frac{d_p}{d_b} \right)^2
\]  \[\text{[4]}\]

Aggregation of fine particles in mineral suspension systems can be achieved by various mechanisms such as coagulation, polymeric flocculation, shear flocculation or oil agglomeration.

a. Coagulation / Polymeric Flocculation

The behavior of colloidal particles in water is strongly influenced by their electrical charge. Each particle immersed in water carries an electrical charge; if the particles carry the same type of surface charge, they repel each other and this prevents their aggregation. As a result, charged fine particles tend to remain dispersed in suspension forming a stable system. Coagulation is the destabilization of colloidal system by neutralizing the electrical charge of the interacting particles. Dispersed systems are destabilized when electrostatic forces that are responsible for repulsion are minimized. The coagulation can be achieved either by reduction of the inter-particle repulsion through neutralization of particle charge or by compression of the electrical double layer surrounding the interacting particles (Somasundaran, 1980; Laskowski and Pugh, 1991; Laskowski and Palmes, 1993; Klimpel, 1997; Hogg, 2000).

Flocculation is the bridging of particles with the use of long polyelectrolytes (floculants) (Kitchener, 1978; Attia, 1991; Laskowski, 1992). Polymeric flocculants are adsorbed at mineral/water interfaces and the aggregation is believed to be the result of this bridging action caused by the binding of the adjoining particles within the flocs (Slater and Kitchener, 1966). Selective flocculation takes place due to the differences in the physical-chemical properties of various mineral components in mixed suspensions (Kitchener, 1978).
b. **Hydrophobic Aggregation**

Hydrophobic particles tend to aggregate in aqueous systems. In flotation, it is important for aggregate formation to be selective and to result in hydrophobic aggregates. Gaudin *et al.* (1942) had suggested a method of aggregation which is potentially more selective than electrolytic coagulation or flocculation by soluble polymers. The study of Gaudin *et al.* (1942) showed that in the presence of ethyl xanthate, all galena particles finer than 4 µm floated equally well and this behavior was explained by the formation of aggregates (Trahar and Warren, 1976). Such aggregates were hydrophobic and therefore, much more suitable to be recovered by flotation than the aggregates resulting from the coagulation or flocculation phenomena.

A confirmation of these results had been provided by Rehbinder (1950). It was shown that with the use of ethyl xanthate, the galena particle surfaces were rendered hydrophobic and their floatability was increased at the same time. The contact angle was changed from 60 to 95 degrees and such a change immediately led to the aggregation of particles (under proper hydrodynamic conditions) and improved the flotation.

There are two important mechanisms which may account for better flotation of fine particles; shear flocculation and oil agglomeration. Both of them are based on hydrophobic aggregation but their mechanisms are different.

c. **Shear Flocculation**

Shear flocculation is a method of destabilizing a suspension and selectively aggregating very fine particles in a convenient stirring regime after rendering them hydrophobic by the adsorbing collectors.

As the adsorption of anionic collectors (e.g. xanthates, fatty acids, etc) increases the negative charge of mineral particles, such particles can form a stable suspension in spite of the fact that they are hydrophobic. An energy barrier resulting in repulsion of similarly charged hydrophobic particles prevents the particle-bubble adhesion.

In shear flocculation, the energy barrier between fine particles is overcome by intense stirring (Warren, 1975; Subrahmanyam and Forssberg, 1990).
There are two conditions for shear flocculation to take place:

- Particles must be rendered hydrophobic prior to aggregation,
- A high shear rate is needed to initiate the aggregation process (Warren, 1991).

For a typical shear flocculation system, the effect of agitation speed or shear rate on particle aggregation has been well established. In 1975, Warren observed the aggregation of ultrafine particles of scheelite, in the presence of sodium oleate by applying a shear field of sufficient magnitude. Scheelite particles have a negative zeta potential in dilute alkaline medium, which is large enough to stabilize the suspension. Addition of sodium oleate to the suspension rendered the scheelite particles hydrophobic and made the zeta potential of scheelite particles more negative at the same time. However, there were no aggregation at low stirrer speeds; only stirring at higher speeds produced an extensive aggregation.

Warren coined the term “shear-flocculation” to distinguish this process from coagulation by electrolytes or flocculation by soluble polymers. The aggregates were found to be much stronger than those produced by coagulation or flocculation, which tend to break up at higher shear rates (Warren, 1991).

Shear flocculation/flotation was first tested on the beneficiation of finely disseminated scheelite ores by Koh and Warren (1977). Scheelite fine particles were made hydrophobic by adsorbing oleic acid, and then were aggregated when the shear rate was sufficient.

Even though collector usage is generally required for most of the minerals to render them hydrophobic prior to agglomeration, some minerals like sulfides, depending on oxidation, may exhibit some degree of hydrophobicity even without using collectors. Figure 7 confirms that galena even without xanthate is slightly hydrophobic (Song et al., 2000).
Figure 7— Contact angle of galena as a function of pH in the absence and in the presence of KAX (Song et al., 2000). (Courtesy of Elsevier)

Figure 8— Effect of agglomeration on flotation of galena at $10^{-4}$ mol/l concentration of KAX (Song et al., 2000). (Courtesy of Elsevier)

As Figure 8 demonstrates, flotation of galena can be much more improved when particles are aggregated. Galena does not float well in the case of conventional flotation tests in which the suspensions were only mildly conditioned with PAX using a magnetic stirrer. On the other hand, aggregate floatability was better when conditioning was carried out in a mixing tank at 700 rpm for 20 minutes (Song et al., 2000).
d. Oil Agglomeration

Oil agglomeration is another example of the processes which can be utilized to improve flotation of fines. It is a beneficiation process that is based on selective aggregation of particles in which water-insoluble oil is utilized. The oil droplets when present in a mineral suspension tend to attach to hydrophobic particles and, depending on hydrodynamic conditions, may cause their agglomeration (Laskowski, 1992) (Figure 9).

![Figure 9](image)

**Figure 9**—Oil agglomeration process in which particles are tethered by bridging oil that forms the aggregates (After Szymocha et al., 1989). (Courtesy of IChemE Publishing)

Only very hydrophobic particles can be agglomerated with oil. Except for naturally hydrophobic minerals, selective agglomeration process requires adsorption of a collector onto a desired mineral. For this reason, the first stage in the oil agglomeration may involve the conditioning with appropriate collector to render the particles hydrophobic. The second stage is the addition of oil, followed by an intense conditioning to bridge the particles together. Intensive mixing of sufficient duration is needed to disperse the oil and to increase the probability of collision.

Although the words ‘flocculation’ and ‘agglomeration’ are often used interchangeably, they refer to two distinct processes. If aggregation is induced by polymer bridging action, the process is known as flocculation and the aggregates are referred to as flocs. When aggregation results from the action of an immiscible liquid, such as oil, the process is referred to as agglomeration and the aggregates are refereed to as agglomerates (Attia, 1991). The main difference between oil agglomeration and agglomerate flotation is the extent of agglomeration. In the oil agglomeration, the formed agglomerates must be large enough to be recovered using a
procedure such as screening, whereas in the agglomerate flotation, the agglomerates must be hydrophobic enough to be recovered by flotation (Laskowski and Castro, 1999; Laskowski and Valdivieso, 2004).

Several stages of oil agglomeration have been described in the literature (Lapidot and Mellgren, 1968; Swanson et al., 1977; Szymocha et al., 1989).

Szymocha et al. (1989) investigated the growth mechanism of the agglomerates in the spherical agglomeration of coal. The results of the study showed that the growth of agglomerates occurs in a step-wise manner. Seven stages of spherical agglomeration of coal have been identified:

- **a.** Wetting period,
- **b.** Quick growth period,
- **c.** Consolidation period,
- **d.** Second growth period,
- **e.** Compaction period,
- **f.** Third growth period,
- **g.** Equilibrium period.

The first stage, wetting period, begins with the addition of the bonding liquid. The oil is dispersed by intensive mixing that disperses the oil into fine droplets which are then deposited on hydrophobic particles. No growth of agglomerates is observed in this period (Szymocha et al., 1989).

The wetting period is followed by a quick period of growth of the agglomerates in which the build-up of liquid bridges between particles begins and the primary agglomerates are formed. These aggregates are further densified and consolidated during the consolidation period. Due to the multiple collisions, the consolidation of the aggregates takes place through rearrangement of the particles and forms a more stable system.

After consolidation period, a second stage of agglomerate growth takes place. Dense primary agglomerates coalesce and associate into larger secondary agglomerates. This period is followed by a compaction period in which the secondary agglomerates undergo plastic deformation and porosity reduction as the spaces are filled with the dispersing liquid. Depending on the concentration of the bonding liquid, the agglomerates can maintain the size they have achieved or they can grow further if there is enough binder to cause agglomeration.
again. After reaching an equilibrium size, the agglomerates are very strong and rigid (Szymocha et al., 1989). In the presence of excess oil, agglomerates are wet in surface state, soft, weak and very susceptible to coalescence. Under prolonged mixing, agglomerates undergo plastic deformation as shown in Figure 10.

![Diagram of agglomerate formation and deformation](image)

**Figure 10**—Schematic representations of the agglomerate formation and deformation (Szymocha et al., 1989). (Courtesy of IChemE Publishing)

Lapidot and Mellgren (1968) studied the growth mechanism of agglomerates in the oil agglomeration process. They measured the power consumption to monitor the phenomena occurred during the conditioning of the ilmenite ore. Their results revealed that power consumption during the conditioning showed a peak and then a sharp decrease. The peak was explained by the considerable agglomeration of the pulp observed during the conditioning and the decrease was a result of a subsequent de-agglomeration of the pulp. On the basis of these observations, five distinct periods were identified on the power consumption curves:

a. The induction period,

b. The agglomeration period,

c. The agglomeration peak,

d. The de-agglomeration period,

e. The dispersion period.

Their results showed that if agglomerate flotation is preceded by a short conditioning (induction period), a very low concentrate yield is obtained. In the agglomeration period, a gradual non-selective increase in both ilmenite and gangue recoveries took place and reached a
maximum at the agglomeration peak with low flotation selectivity. Conditioning during de-agglomeration period resulted in a rapid depression of the gangue while the ilmenite recovery increased further. Maximum selectivity was obtained at the very end of this period (Figure 11). In the dispersion period, both ilmenite and gangue recoveries decreased gradually (Lapidot and Mellgren, 1968).

2.4 EFFECT OF CONDITIONING ON AGGLOMERATE FLOTATION

In the froth flotation process, the suspension of finely ground minerals in water is subjected to conditioning. The conditioning is the period of agitation, before air is turned on, which allows the surface of the mineral particles to react with the reagents. It is needed to disperse mineral particles and keep them in the ‘liberated’ state. But most importantly, it involves the adsorption of collector onto valuable particles and distribution of the reagents throughout the cell.

In 1958, Kihlstedt studied the use of the tall oil-fuel oil mixture in the flotation of Swedish hematite ores. The tests revealed that while using the tall oil-fuel oil mixture, long conditioning times at high solids content were essential in the conditioning stage.

Runolinna *et al.* (1960) advanced the concept of agglomerate flotation with the same tall oil and fuel oil combination on an ilmenite ore from Finland. A similar experimental technique to the one in the Kihlstedt’s study (1958) was used, which involved long conditioning time with fatty acid-fuel oil mixture at high pulp density. They observed that adsorption of the tall oil-fuel oil mixture on wet mineral surfaces was slow and the agglomeration initially was not selective. The oil was shown not to spread spontaneously; and the spreading needed a conditioning process. Only after some conditioning time, the fuel oil droplets attached to the hydrophobic tall oil coated surfaces and the selective agglomeration of the ilmenite particles was achieved. The need to spread the oil on the ilmenite surface was claimed to be the main reason for the vigorous conditioning.

Lapidot and Mellgren (1968) used the same technique for the flotation of ilmenite ore. They conditioned the ilmenite ore with reagents at 70% solids content. The variations in the net power consumption indicated that the pulp became viscous several minutes after the addition of the reagents and that the viscosity altered with conditioning time.
On the basis of these observations, some characteristic points were identified on the power consumption curves. The ore was conditioned to these different characteristic points and the flotation results were then presented as a function of them. The plot of ilmenite and gangue recovery curves obtained in the tests against the characteristics points is shown in Figure 11.

![Figure 11— Ilmenite recovery and gangue recovery as a function of the characteristic points on the power consumption curve (Lapidot and Mellgren, 1968). (Courtesy of Maney Publishing)](image)

As seen in Figure 11, satisfactory selectivity in agglomerate flotation could only be obtained after sufficient conditioning. After a period of unselective agglomeration of all the material, further conditioning resulted in a rapid depression of the gangue and better recovery of ilmenite. The oil was transferred from hydrophilic gangue to hydrophobic ilmenite with the increasing conditioning time. This correlation indicated that when conditioning is conducted at a high pulp density, the ilmenite ore flotation results were a direct function of the conditioning.

High pulp density is an important parameter in the conditioning stage that permits bulk agglomeration. It improves the spreading of oil over solid particles and enhances transfer of oil to the hydrophobic surfaces (Jordan and Davis, 1995; Damodaran et al., 1996).

Jordan and Davis (1995) and Damodaran et al. (1996) showed that there is a definite relationship between the solids content at the conditioning stage and the flotation response of phosphate ore. While conditioning of the pulp at 66% solids with a fatty acid-fuel oil mixture
had to be long (over 9 min) to achieve a high P₂O₅ recovery, higher recoveries could be obtained after just 2 minutes of conditioning at 74% solids. The flotation performance after conditioning at the solids content below 70% varied quite dramatically whereas the flotation response was good and predictable when conditioning was carried out at the solids content exceeding 70%.

Dell et al. (1964) showed that long conditioning at high pulp density was advantageous in agglomerate flotation of coal as well. Increasing the conditioning time produced a rapid initial improvement in flotation, followed by a less rapid decline and then, a slower recovery to the former peak value. It was suggested that the improvement in flotation was due to the bonding of the oil droplets on the surface of particles to form bridges causing the aggregation (Dell et al., 1964).

Mechanical action during conditioning is another factor that plays a major role in particle aggregation. In the conditioning stage, mixing is applied to maintain the mineral particles in a state of suspension, to increase the probability of collision, to distribute the reagents evenly within the pulp and to provide sufficient time for particles to react with these reagents.

In 2000, Song et al. concluded that sufficient mixing intensity and duration is required to achieve a maximum degree of hydrophobic aggregation. They showed that aggregate floatability was better when conditioning was carried out in a mixing tank at 700 rpm for 20 minutes (Figure 8).

**Figure 12**—Floatability of galena aggregates induced by potassium amyl xanthate as a function of the stirring speed at pH 8 (Song et al., 2000). (Courtesy of Elsevier)
However, aggregate floatability can only be increased up to a maximum point by increasing the mixing intensity (Figure 12). Song et al. (2000) showed in their study that the intensity of agitation is closely associated with hydrophobic aggregation.

Perhaps the best example of the fact that particle aggregation depends on intensity of conditioning is the concept of shear flocculation. Shear flocculation has been extended to the conditioning stage ahead of flotation by many researchers (Rubio, 1978; Bulatovic and Salter, 1989; Stassen, 1990, 1991; Rubio et al., 1994). These authors claimed that the energy transferred in the conditioning stage, often expressed as conditioning time at constant impeller speed, has a pronounced effect on the concentrate recovery, grade and flotation rate.

Bulatovic and Salter (1989) investigated the improvements in recovery and selectivity with increasing energy input to the conditioning stage. They carried out a series of tests on a finely ground copper sulfide ore with varying energy input to the conditioning stage. They described a technique which involved long conditioning times at high pulp density prior to flotation. This phenomenon was termed "High Intensity Conditioning (HIC)" (Valderrama and Rubio, 1998). High intensity conditioning with the addition of a collector and suitable modifiers significantly improved both the recovery and the selectivity of ultrafine sulfide slimes. Bulatovic and Salter (1989) claimed that the improvement was due to the selective collector adsorption and to the formation of fine particle aggregates which are readily floatable. In other words, they regarded "shear flocculation" (Warren, 1975) as the mechanism taking place during a high intensity conditioning (Chen et al., 1999).

In 1998, Valderrama and Rubio studied the effect of high intensity conditioning (HIC) on carrier flotation of gold by varying the energy transferred to the pulp. They concluded that high intensity conditioning, as a pulp pre-treatment step, enhanced flotation recovery of gold fines by about 24%, flotation rates were at least 2-3 times faster and the gold concentrate grade was increased by 50%. These results were explained by the increase in the concentration of floatable particles as a result of the aggregation of gold fines.

Valderrama and Rubio (1998) also emphasized the importance of long conditioning times and the effect of particle size in case of high intensity conditioning. At short periods of HIC, very fine particles adhered to the surface of coarse particles better, causing carrier flotation. However, at longer times of HIC, fine particles attached to the coarse particles began to detach due to the high shear forces. They formed aggregates within themselves that yielded

Engel et al. (1997) studied high intensity conditioning as a means of improving flotation of a Western Australian nickel sulfide ore. In terms of energy input, major improvements to the nickel grade-recovery results occurred after using HIC, with the best values found after the highest HIC work input. The highest values of shear rate and power per unit volume gave the fastest flotation rates and the best grade-recovery relationship. These were also the conditions for the highest selectivity for nickel recovery over MgO. In other words, the highest intensity mixing has the highest number of inter-particle interactions and better surface cleaning (Engel et al., 1997).

Similar conclusions have been reported by Chen et al. (1999) who investigated the effect of HIC on the same ore sample as used in the study of Engel et al. (1997). They concluded that surface cleaning plays a dominant role during high intensity conditioning. The experimental results showed that HIC improved flotation behavior on both the whole and deslimed feeds of the nickel ore, due to surface cleaning of pentlandite particles. This was reflected by significantly higher flotation rate constant values of pentlandite for intermediate and coarse particle size fractions (Chen et al., 1999).

### 2.5 EXPERIMENTAL METHODS OF STUDYING AGGREGATION

A wide range of experimental methods for monitoring the aggregation of fine particles has been described in technical literature. These include techniques for measuring aggregation rates in fundamental studies and those which are useful in monitoring and control of the aggregation in commercial processes. The two experimental methods of studying aggregation that are described in this chapter include:

- Rheology of aggregating mineral suspensions,
- Power draw measurements.
2.5.1 RHEOLOGY OF AGGREGATING MINERAL SUSPENSIONS

A considerable number of materials of technological importance in the mineral industry are handled in the form of highly concentrated suspensions. Such concentrated solid-liquid suspension systems having strong inter-particle interactions often exhibit a unique plastic flow behavior in the presence of a yield stress. Under the application of small stress, these systems deform elastically with finite rigidity, but when the applied stress exceeds the yield value continuous deformation occurs with the material flowing like a viscous fluid (Boger and Nguyen, 1983).

Rheological measurements can detect conformational changes in solutions of polymers that depend on polymer-solvent interaction; such interactions determine flocculation/stabilization phenomena in mineral suspensions (Laskowski, 2001). These measurements have been widely used to characterize the dispersibility and stability of mineral systems although the exact interpretation of the observed phenomenon for heterodispersed non-spherical particles in concentrated systems is difficult (Laskowski and Pugh, 1991).

A mineral suspension can be dispersed by promoting net repulsive inter-particle forces, or can be aggregated by promoting attractive inter-particle forces. Such systems exhibit quite different rheological behavior (Johnson et al., 1999). While the former most likely will behave as a Newtonian fluid, the latter will exhibit a non-Newtonian behavior with a yield stress. Stable suspensions at low and moderate solids contents exhibit Newtonian behavior, coagulated suspensions show reversible plastic behavior (decreasing viscosity up to a constant value as the shear rate increases) and the flocculated suspension shows irreversible pseudo-plastic behavior with initial viscoelasticity (Laskowski and Pugh, 1991). While good dispersion of suspended particles and Newtonian behavior of the system improves wet grinding, particle aggregation improves thickening and filtration in solid/liquid separation (Laskowski, 2005).

As the particle size of the feed becomes finer, ores tend to exhibit a much more complex behavior which can result in adverse effects on plant performance (Nel et al., 2007). Solid-liquid suspensions become more non-Newtonian as the particle size decreases, the volume fraction of particles increases and/or the net force of interaction between particles become attractive. For the particles showing net attractive forces, a network of particles that form aggregated structures develops, and rheological properties of such a system are characterized by a yield stress (Laskowski, 2005). The yield stress is the minimum shear stress corresponding to
the first evidence of flow, that is, the value of the shear stress at zero velocity gradient. The yield stress can thus be regarded as a material property denoting a transition between solid-like and liquid-like behavior (Boger and Nguyen, 1983).

Rheology of suspensions is strongly affected by a large number of factors. This includes particle size as well as the distribution of particle sizes (Farris, 1968). Increasing the particle size by aggregation affects the viscosity and the yield stress, which in turn would affect the rheology. There is a limit for the yield stress in optimum and economical operating conditions. A precise quantitative knowledge of the yield stress is very important in handling, storage, processing, and transport of concentrated suspensions in industry (Charles et al., 1971). An extremely high yield stress may result in unnecessarily high power consumption and hence high operating costs (Boger and Nguyen, 1983). Therefore, the changes in the yield stress and system stability should be monitored to characterize aggregation-dispersion of fine particles. At this point, rheological measurements can be utilized as a useful method to identify the behavior of a dispersed system.

2.5.2 POWER DRAW MEASUREMENTS

Another method of characterizing the aggregation of fine particles suspended in liquid involves the measurements of power draw required to drive an impeller. Power consumption of the impeller is the amount of power required to rotate an impeller at a given speed in a tank with a specific configuration (tank shape, baffle size etc.) and containing a fluid of certain physical properties (Mhaisalkar et al., 1985).

Power required to rotate an impeller depends on several factors such as the properties of the liquid (viscosity), the tank or vessel size, liquid depth and geometry of the rotor, stator and container. Since there are many factors that affect power consumption, no single equation is capable of defining the power requirement of different conditioners operating under various conditions. A convenient method involves the use of power curves.

Power curves provide a means of correlating power consumption as a function of impeller speed, impeller diameter and fluid properties. An individual power curve is valid only for a specific geometric configuration and is independent of size of the container (Uhl and Gray, 1966).
Several researchers have used the power draw measurements to study the stages of coal-oil agglomeration (Swanson et al., 1977; Bensley et al., 1977; Szymocha et al., 1989). During the first stage of coal-oil agglomeration, the wetting period, there were no agglomerates formed and the power consumption in the conditioner was observed to be steady. At the end of this period, power consumption rapidly increased due to particle agglomeration.

The peak of the power draw curves represents the phase inversion time. In the case of coal-oil agglomeration, the phase inversion marks the beginning of separation of coal and mineral particles and can be characterized by a change of color of suspension (Swanson et al., 1977; Bensley et al., 1977). The coal particles, which were previously wetted by water, become wetted by the oil droplets after the phase inversion.

Factors influencing phase inversion time was investigated by Swanson et al. (1977). It was shown that the phase inversion time, taken as the time at which the stirrer power consumption reaches maximum, dramatically decreases with increasing solid concentration (Figure 13) and can further be reduced by emulsifying the utilized oil. The results also revealed that as the amount of gangue mineral (kaolinite) added to the system increases, the agglomeration of hydrophobic coal particles becomes more difficult and phase inversion time increases (Figure 14) (Bensley et al., 1977).

![Figure 13](image-url)  
**Figure 13**– Influence of pulp density on inversion time; coal suspensions with various solids content were mixed at 300 rpm with 15% wt. kerosene (Swanson et al., 1977). (Courtesy of Society of Mining Engineers)
Figure 14—Effect of kaolinite addition on oil agglomeration kinetics; different kaolinite quantities were added to coal suspensions (40% pulp density) and mixed with 15% wt. kerosene at 300 rpm (Swanson et al., 1977). (Courtesy of Society of Mining Engineers)

At the phase inversion, the viscosity of the slurry rapidly changes as the system achieves a state of maximum aggregation (Szymocha et al., 1989; Swanson et al., 1977; Bensley et al., 1977). Consequently, there is a rapid measurable increase in power consumption of the stirrer motor agitating the slurry.

Perhaps the first attempt to correlate agglomerate flotation with power consumption was that of Runolinna et al. (1960). They studied the agglomerate flotation of Otanmaki ilmenite ore. It was observed that the energy required for conditioning was reduced when a few percent of emulsifying agent was added to the system and the selective agglomeration was found to be accelerated with the addition.

The fundamentals of this process were further studied by Lapidot and Mellgren (1968). They examined the relationship between pattern of power consumption during conditioning and flotation results of an ilmenite ore. When the ore was conditioned at 70% solids content with the tall oil-fuel oil mixture, a bulk agglomeration was followed by de-agglomeration. Agglomeration and breaking up of the agglomerates led to the variations in the viscosity of the
pulp and, as a consequence, to corresponding variations in the power consumption. The measured power consumption during the conditioning gave the curves shown in Figure 15.

![Figure 15](image)

**Figure 15**—Typical curves of power consumption versus conditioning time at constant impeller speeds (Lapidot and Mellgren, 1968). (Courtesy of Maney Publishing)

All three curves which correspond to various impeller speeds are characterized by a peak and then a sharp decrease. The peak represented the considerable agglomeration of the pulp and it was therefore assumed that the increased pulp viscosity was the result of this bulk agglomeration. In the same manner, it was again assumed that the subsequent sharp de-agglomeration of the pulp was the result of the decreasing viscosity.

These correlations showed that the phenomena occurring during conditioning were related to the changes in the viscosity as revealed by the power consumption curves. A preliminary study of the rheological properties of agglomerating fine coal suspensions was carried out by Burdukova (2004). In this work, the rheological properties of coal and water
mixture were measured after it was agglomerated with the oil. All tests were carried out at 10 wt% oil addition and the vane method was used to determine directly the yield stress of the system.

![Graph showing effect of solids content on oil agglomeration of fine bituminous coal as characterized by rheological measurements.](image)

**Figure 16**– Effect of solids content on oil agglomeration of fine bituminous coal as characterized by rheological measurements (Burdukova, private communication).

It was observed that the yield stress curve obtained (Figure 16) has a similar bell-shape when compared with the power draw curve obtained in the previous studies (Figure 15). The yield stress of the agglomerating system was first increased with the conditioning time and then, decreased with the subsequent de-agglomeration. These results prove that the increase in power consumption during conditioning was, indeed, caused by the increased viscosity of the system. Hence, it may be concluded that the power draw curves obtained from the power draw measurements can be used as a helpful tool to study particle aggregation.

Stability-aggregation of fine particles is determined by a balance between the energy barrier opposing aggregation (DLVO theory) and the kinetic energy of the interacting particles. The energy transferred to the pulp in the conditioning stage (commonly expressed as conditioning time at constant impeller speed, or as impeller speed at contact time) increases the kinetic energy of the interacting particles and may induce aggregation. Increased conditioning
intensity increases a chance of aggregation, but on the other hand, too intense conditioning may break the aggregates. It is thus obvious that the HIC requires measurement of the amount of energy transferred to the pulp and characterization of the intensity of conditioning via power draw measurements.

Valderrama and Rubio (1998) determined an optimum energy input for carrier flotation of gold particles. Depending on the intensity of conditioning fine gold particles either adhered to the surface of coarser particles and floated along with them (carrier flotation) or aggregated among themselves, yielding more floatable species.

According to their results, carrier flotation was promoted at rather mild shear energy input (0.5-2 kWh/m³) of pulp, at higher energy inputs (2-3 kWh/m³) detachment of the fine particles from larger carrier particles was observed, but at even higher energy inputs (3-4 kWh/m³) the aggregation of fine particles was again promoted. Between 3-4 kWh/m³, the fine gold particles aggregated themselves and the highest recovery values were obtained. At the energy input levels exceeding these values, the aggregation was severely affected, no aggregates were formed and much lower recoveries were obtained (Figure 17).

*Figure 17*— Particle aggregation phenomena during the conditioning stage illustrated by Valderama and Rubio (1998). (Courtesy of Elsevier)
The power consumption of the mixing impeller can be measured using various experimental techniques. It has been studied by several researchers and some of these methods are briefly discussed in the following section.

a. **Electrical Method**

Mhaisalkar *et al.* (1985) developed a simple electrical method to measure the power input by an impeller to the system. The value of net power draw was obtained from the difference in power input to the motor for a given speed of the impeller at load (impeller in pulp) and no load conditions (impeller in air).

Palmes and Laskowski (1993) used the same electrical method to determine the power input of the stirrer motor and thereby to measure the shear rate in flocculation tests. The net power input (in Watts) was again calculated from the difference in power input to the motor; i.e., power input without load and with load. In each case, power input was calculated from the voltage and current measured across the motor armature for a given rotational speed.

This method has not been commonly used because of its shortcomings. In electrical power draw measurements, for geared motors or any coupled systems other than the direct one, losses in gearing or in the coupling system were difficult to assess under load and no load conditions. In addition, deducting the value measured at no load from the observed value for measuring the power input was not convenient when a large number of data was being processed. Moreover, the current and voltage values must be precisely measured to obtain reproducible results. For this reason, a second method that involves a turn-table setup for measuring torque has been chosen as the experimental method of this study.

b. **Torque Measurement on Turn-table**

In the torque turn-table method, the conditioning tank is placed on a low friction turn-table which rotates when the impeller is turned on. A thin wire attached to the base of the cell, passes over a pulley and is attached to a weight placed on a balance (Figure 18). By knowing the mass (m) required to counter rotation, the distance of the pulley from the cell (l), and the mass of fluid in the cell (ρV), the mean energy dissipation in the cell can be calculated (Newell and Grano, 2007).
Figure 18 – Turn-table setup with electronic balance which has been used by previous researchers (as designed by Dr. Negeri)

There are several methods to measure mean energy dissipation. While studying the effect of operation parameters on three geometrically similar Rushton turbine flotation cells, Newell and Grano (2007) compared two methods of measuring mean energy dissipation, one of which is torque turn-table method. The design of the equipment that they used was provided by Dr. T. Negeri (personal communication, 2002). They observed that the turn-table method showed a good agreement with the second method, Laser Doppler Velocimetry and this confirmed the validity of the turn-table method. The mean energy dissipation was determined from an average of point values obtained within the conditioning tank by these two methods.

Patil et al. (2001) obtained the mean shear rate in a stirred tank by using the torque table method for modeling purposes. The power dissipation (P) of the impeller is calculated from the measured torque.
The mean shear rate is calculated from,
\[
\bar{G} = \left( \frac{P}{V \mu} \right)^{1/2} = \left( \frac{\bar{\varepsilon}}{\nu} \right)^{1/2}
\]  
[5]

where \( V \) is the volume of the fluid in the vessel, \( \mu \) and \( \nu \) are the dynamic and the kinematic viscosity of the fluid, respectively, and \( \varepsilon \) is the mean energy dissipation rate per unit mass of the fluid (Patil et al., 2001).

Engel et al. (1997) also used a torque measuring device to investigate the high intensity conditioning mechanism. A circular HIC tank was attached to a steady base support, (beneath a single-phase suspended motor) suspended from its mounting via a counterweight. Low friction pulleys reduced experimental inaccuracies. A lever arm was attached horizontally to the motor and thus tangentially to the rotation of the shaft. This was connected via a stiff piece of wire to a load cell. The load cell fed an electric signal to the meter readout. Since the force imparted onto the fluid by the impeller causes an equal and opposite force on the lever arm, the meter readout was directly related to the force applied to the pulp. Meter readings could then be plotted versus the weight of the masses.

Overall, these results from the literature survey suggest that turn-table method was a more reliable and convenient method when compared with the electrical method.

3. EXPERIMENTAL PROCEDURE and MATERIALS

3.1 INTRODUCTION

This study had started as a part of a research project whose major objective was to improve the flotation of a fine (slime) fraction of nickel-sulfide ore from Western Australia.

The experiments were carried out using three different materials;

a. Nickel sulfide ore samples
b. Bituminous coal
c. Mixed mineral systems
   – Coal with dolomite
   – Coal with nickel sulfide ore
Finely ground nickel sulfide ore was used in the first batch of experiments. Agglomerate flotation had been chosen as the method to improve flotation results. Since agglomeration depends on conditioning, the power draw measurements were used to study the effect of conditioning on agglomeration. A turn-table setup had been constructed and the changes in the system were recorded as a function of the power draw dissipation.

In the second part of the experiments, a bituminous coal was used as a hydrophobic model system to verify the turn-table method in studying particle aggregation. Oil agglomeration of fine coal, which had been studied by several researchers, was chosen as a model system. The following set of conditioning experiments have been carried out to:

- determine the sensitivity of the power draw measurements to the physicochemical conditions,
- see whether this equipment can give the same results that were obtained by previous researchers,
- define the limitations of the setup.

The experiments were conducted on the same turn-table setup which was used in the agglomerate flotation of the nickel ore; a specially designed tank was used in the conditioning tests instead of the flotation cell.

In the third section of the test work, mixed mineral systems with different types of gangue minerals were tested. A dolomite mineral sample was added to the same coal system that was used in the second part of the test work. Dilution effect on oil agglomeration was investigated by increasing the amount of dolomite added to the system thereby changing the mineralogical composition. It was expected that oil agglomeration would not only be affected by the mineralogical composition of the gangue but also by the size and shape of the particles. To substantiate this hypothesis further, a finer dolomite sample and the nickel sulfide ore containing asbestos type of gangue were added to the coal model system.

The power draw measurements in this project were performed to identify the optimum conditions and to characterize the system behavior. It was measured at regular time intervals during conditioning stage in all tests.
In addition to the power draw measurements conducted with the use of the turn-table, the electrical measurements were performed to verify the results by an independent experimental technique.

In this chapter, the procedures and the materials used in the test work are described.

3.2 NICKEL SULFIDE ORE

The ore used in this study was from a low grade (0.52 % Ni) disseminated nickel sulfide deposit hosted in a dunite, serpentinised ultramafic rock. Mineralization consists of around 1% sulfides. The chemical composition of the nickel ore sample is given in Table 1.

**Table 1**— Chemical composition of the nickel ore from Western Australia (Senior and Thomas, 2005)

<table>
<thead>
<tr>
<th>Assays</th>
<th>Ni</th>
<th>MgO</th>
<th>Fe</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
<td>0.52%</td>
<td>39.6%</td>
<td>4.78%</td>
<td>0.8%</td>
</tr>
</tbody>
</table>

The nickel bearing minerals in the ore are predominantly pentlandite (60-80%) with smaller amounts of millerite and violarite. The mineral composition of the nickel bearing minerals is given in Table 2.

**Table 2**— Mineral composition of the nickel bearing minerals (Senior and Thomas, 2005)

<table>
<thead>
<tr>
<th>Ni Bearing Minerals</th>
<th>Pentlandite (NiFe)₉S₈</th>
<th>Millerite (NiS)</th>
<th>Violarite (Ni₂FeS₄)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content (%)</td>
<td>60-80%</td>
<td>2-20%</td>
<td>0-10%</td>
</tr>
</tbody>
</table>

The detailed mineralogy of the feed is given in Appendix A. The gangue minerals are mainly from the serpentine group; the predominant mineral being chrysotile, together with significant quantities of lizardite and minor talc. Iron sulfides (pyrite, pyrrhotite), chlorite, brucite, magnetite and hydroxy-carbonates (magnesite) compose the remaining rest of the gangue minerals.

Serpentine minerals making up about 90% of the ore, were the main challenge in the flotation. Serpentine is a group of rock-forming hydrous magnesium iron phyllosilicate ((Mg, Fe)₃Si₂O₅(OH)₄) minerals. The three most common members of the serpentine group are:
a. Antigorite-(Mg,Fe)$_3$Si$_2$O$_5$(OH)$_4$,

b. Lizardite- Mg$_3$Si$_2$O$_5$(OH)$_4$,

c. Chrysotile-Mg$_3$Si$_2$O$_5$(OH)$_4$.

Chrysotile and lizardite are the main contributors of the gangue in this particular ore. Chrysotile is the major gangue component, making up about 80% by mass. It is a complex phyllosilicate mineral containing approximately 40% of silica and 40% magnesium oxide. The stoichiometric chemical composition may be expressed as Mg$_3$Si$_2$O$_5$(OH)$_4$. The flexible fibrous structure of chrysotile is generally believed to be the cause of the ore problematic behavior and it is this aspect that poses a major challenge in processing of the ore and in the analysis of the degree of agglomeration.

Chrysotile is a member of the asbestos mineral group and is commonly referred to as ‘white asbestos’. Although all asbestos minerals are fibrous, it does not follow that a material is asbestos if it is fibrous (Myer, 1989). The term asbestos is applied only to those varieties that have been commercially exploited (Campbell et al., 1977; Ross et al., 1984). There are only six asbestos minerals (Figure 19) and chrysotile is the single asbestiform variety that belongs to serpentine group.

![ASBESTOS Diagram](attachment:asbestos_diagram.png)

**Figure 19** – The two major fibrous asbestos silicate groups.
Asbestos minerals are composed of fibrous particles which are extremely flexible, thin and have a very high length to diameter ratio (Zussman, 1979). For ground chrysotile, a length to diameter ratio of greater than 50:1 can be expected (Zussman, 1979). Figure 20 shows the fibers of one of the samples used in the experiments, thickness as low as 0.1 µm, but up to about 100 times in length.

![Figure 20](image)

**Figure 20** – An SEM micrograph showing the flexibility of individual chrysotile fibers.

The curled tubular structure of chrysotile is much more complex than the simple edge/plate structure of clays. Most serpentines and other layered silicate minerals, such as micas and clays, are composed of tetrahedral and octahedral sheets that lie virtually flat. However, in chrysotile samples, the dimensions of the silicate tetrahedral layer are about 9% smaller than the corresponding ones in the octahedral brucite layer (Myer, 1989). This is illustrated in Figure 21A. The imperfect fit of octahedral layers and the tetrahedral layers causes the crystal structure to bend, curl and form concentric hollow cylinders. The bending of the sheets is continuous and results in tubes that give the mineral its fibrous nature.
As the chrysotile have a spiral shape, the tetrahedral-octahedral edge is likely to occur at the end of each tube as well as run along the length of it (Figure 21B). The tubes curl in a way to expose the magnesium rich octahedral layer on the outside. Therefore, the surface charge of this site is likely to be similar to the brucite which is positively charged over a broad pH range. However, T-O edges are likely to undergo a change from positive to negative. Due to this separation of charge between edges and faces, as well as the flexibility of the chrysotile fibers, they are able to align themselves in a number of configurations with forces existing both within (intra) and between (inter) individual fibers when heterocoagulating. This results in a very complex tangled structure with adverse effects on chrysotile slurry rheology. The entangled fibers tend to form low density mineral aggregates (Senior and Thomas, 2005). Formation of such low density aggregates decreases the effective voidage of the mineral slurry by immobilizing the water within the aggregates. Both the decrease in the effective particle density and the effective voidage has a strong influence on the rheological properties of particulate suspensions (Laskowski and Pugh, 1992).
Chrysotile minerals, especially in fine fractions, increase the viscosity of the flotation slurry and reduce the efficiency of the separation. The problematic behavior consequently necessitated the flotation feed to be highly diluted (to below 10 wt% solids), thus reducing production capacity (Senior and Thomas, 2005). The presence of chrysotile particles in the feed after conditioning was confirmed by the SEM Micrograph shown in Figure 22. The particle is large, approximately 150 microns long and 20 microns wide and shows signs of disintegrating into ultrafine follicles.

![SEM micrograph of flotation feed after conditioning](image)

**Figure 22**— SEM micrograph of flotation feed after conditioning (taken by E. Burdukova).

The other difficulties in recovering pentlandite from such ores are often attributed to the interactions between pentlandite and gangue minerals. Pentlandite surface oxidation, slimes coating and unintentional activation are caused by these interactions and can detrimentally affect pentlandite flotation.

The ore assays approximately 40% MgO. Magnesium silicate (MgO) gangue minerals often report to the concentrate causing downstream processing problems and increasing the smelter costs in nickel sulfide processing,. Flotation of the MgO particles may be via composite particles, through an attachment to the valuable minerals as “slime coatings” or by entrainment.

The formation of slime coatings is directly related to the surface potentials of pentlandite and MgO gangue particles. Edwards et al. (1980) investigated the effect of slime coating of
chrysotile and lizardite on pentlandite flotation at pH 9 and found that the positively charged serpentine (chrysotile) was coagulating on negatively charged pentlandite.

In addition, Bremmel et al. (2005) showed that the zeta potential of lizardite is positive and independent of pH while pentlandite is negative at pH values larger than 4.5, and increases in magnitude as the pH increases. At pH values larger than 4.5, it is likely that the positively charged fine lizardite particles attach to the negatively charged pentlandite particle surface through electrostatic attraction (Figure 23). As a result of lizardite and chrysotile slime coatings, the surface of pentlandite becomes less hydrophobic and its flotation recovery decreases. Carboxymethyl cellulose (CMC), anionic common dispersant, was recommended to be used to eliminate the slime coating phenomena in the flotation of nickel sulfide ores (Edwards et al., 1980).

![Figure 23](image)

**Figure 23** – Effect of pH on zeta potential of chrysotile (after Ney, 1973) and zeta potential of lizardite and pentlandite particles in 0.001MKNO₃ (after Bremmel et al., 2005). (Courtesy of Elsevier)

A further dramatic illustration of the negative influence of serpentine minerals on pentlandite recovery was reported by Mani et al. (1997). The dosage of as little as 30% by weight of serpentine to a pentlandite ore in a laboratory mill reduced the pentlandite recovery from above 80% to below 30% as shown in Figure 24.
Figure 24 – The effect of serpentine content in the feed on pentlandite recovery by flotation (Mani et al., 1997). (Courtesy of Canadian Institute of Mining Publishing)

3.2.1 EXPERIMENTAL PROCEDURE

The nickel sulfide ore used in this study was already crushed below 3 mm and packed in 1 kg bags. The experimental procedure included grinding, classifying, splitting followed by conditioning and flotation (Figure 25).

Figure 25 – Flowsheet of the experimental procedure followed in the experiments.
Wet grinding of the ore was carried out in a mild steel rod mill at 40% solids content. Each 1 kg ore was ground in synthetic process water with 40% of the 180 g/t collector addition. The composition of the process water is given in Table 3. About 80% of this ground ore was below 125 µm. The ground product was washed out using 1.5 L of the process water which resulted in a 25% solids content pulp. The feed after grinding was classified by a C700 Mozley cyclone test rig. The cut-size of the ore was 25 µm. The yields of the underflow and overflow were about 45% and 55%, respectively. The overflow was then left for settling for a few hours and the clear water on top was collected. The pulp was separated into 8 portions by using a splitter and the clear water was used as wash water in splitting and flotation. The grinding and flotation were carried out in the same day to prevent oxidation of the ore. Particle size distribution of the flotation feed was determined by Malvern Mastersizer (Appendix B).

Table 3 – Composition of process water used in the experiments.

<table>
<thead>
<tr>
<th>Mineral</th>
<th>NaCl</th>
<th>CaCl₂</th>
<th>KCl</th>
<th>NaHCO₃</th>
<th>MgSO₄.7H₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content (g/L)</td>
<td>48.2 g/L</td>
<td>1.25 g/L</td>
<td>2.10 g/L</td>
<td>0.29 g/L</td>
<td>24.4 g/L</td>
</tr>
</tbody>
</table>

After grinding, classifying and splitting, a given amount of nickel sulfide ore sample was transferred to a 2 L Denver flotation cell and calculated amount of process water was added. The solids concentration at the conditioning stage was varied over the range of 5–30%. The experiments were performed with 180 g/t of Flex-41 collector (equivalent of iso-butyl xanthate); 40% of the collector was added to the mill, the remaining 60% added to the flotation pulp and conditioned for 10 minutes prior to the addition of emulsified diesel oil. At the end of the 10 minute conditioning period with collector, 1% emulsified oil was added (whenever used) with a dose of 200 g/t or 500 g/t. Conditioning time with emulsified oil was another controlled variable that varied from 0.5 minutes to 30 minutes. In all experiments, 30 g/t of Terric 407 frother was added to the cell and conditioned for one minute just prior to flotation.

The flotation tests were conducted at 10% solids content for 30 minutes. Impeller speed was maintained constant at 1500 rpm during flotation and the air flow rate at 4 L/min. The variables that were kept constant and varied during the experiments are summarized in Table 4. Multiple tests were performed at each condition to test the reproducibility. Power draw measurements were recorded throughout the conditioning stage at regular time intervals.
Table 4 – The variables which were kept constant and varied during the experiments.

<table>
<thead>
<tr>
<th>Constants</th>
<th>Varying parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>─ Collector dosage (180 g/t) (Flex-41)</td>
<td>─ Solids content of the pulp at the conditioning stage</td>
</tr>
<tr>
<td>─ Frother dosage (30 g/t) (Terric 407)</td>
<td>─ Oil dosage added to the tank after conditioning with collector</td>
</tr>
<tr>
<td>─ Diesel oil emulsification ratio (1%)</td>
<td></td>
</tr>
<tr>
<td>─ Impeller speed (1500 rpm)</td>
<td></td>
</tr>
<tr>
<td>─ Solids content of the pulp during the flotation stage (10%)</td>
<td></td>
</tr>
<tr>
<td>─ Air flow rate at flotation (4 L/min)</td>
<td></td>
</tr>
<tr>
<td>─ Geometry and volume of the flotation cell</td>
<td></td>
</tr>
</tbody>
</table>

3.3 BITUMINOUS COAL

A medium-volatile bituminous metallurgical coal (G-120) provided by the Fording River mine, British Columbia, Canada was used in the tests. The particle size distribution of a representative coal sample was determined by a laser diffraction based instrument (Malvern Mastersizer 2000) (Appendix C). The proximate analysis of this coal is given in Table 5.

Table 5 – Proximate analysis of the tested coal.

<table>
<thead>
<tr>
<th>Coal</th>
<th>Moisture (%)</th>
<th>Ash (%)</th>
<th>Volatile Matter (%)</th>
<th>Volatile Matter d.a.f. (%)</th>
<th>Fixed Carbon d.a.f., %</th>
</tr>
</thead>
<tbody>
<tr>
<td>G-120</td>
<td>0.46</td>
<td>13.31</td>
<td>23.02</td>
<td>26.7</td>
<td>73.3</td>
</tr>
</tbody>
</table>

Scanning Electron Microscope images were taken to get the information about the external morphology (texture). The SEM micrographs of the sample surface revealed that the coal particles were breaking into cube-like fragments due to its even-textured structure and were forming two sets of joints at right angles. A SEM micrograph of coal particles is shown in Figure 26.
3.3.1 EXPERIMENTAL PROCEDURE

The coal samples were firstly crushed, ground and screened below 0.7 mm. In order to properly disperse the coal particles in water, 250 grams of fine coal was mixed with water in a blender at high impeller speed for a standard time of 2 minutes. After that, the suspension was transferred to a conditioning tank. The amount of water in the tank was calculated to make the solids content 45%, 50% or 55% in a series of tests. The coal-water suspension was mixed in the conditioning tank at 900 rpm for 10 minutes. High intensity mixing of the pulp was carried out to ensure complete wetting, to homogenize the system and to create a non-settling suspension. At the end of 10 minutes, impeller speed was adjusted to 650 rpm and the calculated amount of oil emulsion was added to the system. The reason for mixing at high intensity, followed by low intensity mixing was to enable the coal-oil agglomerates to enlarge and strengthen. The oil dose was set to 10% by weight (versus dry coal) in all coal-oil agglomeration tests. The emulsion was prepared by mixing 25 grams of fuel oil in 75 g of water at high speed. This was then injected directly into the slurry with a syringe to create an even distribution of the dispersed oil in the cell. The slurry was then further conditioned for an additional 20 minutes. All experiments were repeated three times to get more reliable results.

Figure 26 – A SEM micrograph of a coal sample.
3.4 COAL-DOLOMITE SYSTEM

In this set of experiments, dolomite samples in varied amounts were added to the coal samples to study the effect of dilution on oil agglomeration of coal. The same coal samples that are described in Section 5.3 were used and highly pure natural dolomite was purchased from Wards Natural Science. The mineral composition of the dolomite sample determined by quantitative X-ray diffraction is given in Table 6.

Table 6 – Composition of dolomite mineral samples used in experiments.

<table>
<thead>
<tr>
<th>Component</th>
<th>Dolomite</th>
<th>Calcite</th>
<th>Quartz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage, %</td>
<td>97.4</td>
<td>2</td>
<td>0.6</td>
</tr>
</tbody>
</table>

After determining the effect of dolomite on the coal-oil agglomeration system, the effect of particle size of dolomite was investigated by using the same dolomite sample but with different particle size range. The particle size distributions of the coarse and fine dolomite samples were determined using a Malvern Mastersizer (Appendix D and E). The difference between size distributions of the two mineral samples can also be seen from their SEM micrographs in Figures 27 and 28. These images were also helpful in characterizing the shape of the particles. As it is seen from the Figures 27 and 28, the dolomite tested is regularly shaped and form rhombohedrons which are typical for this mineral.

Figure 27 – SEM image of a fine dolomite sample.  
Figure 28– SEM image of a coarse dolomite sample.
**Ash Content**

Ash content was determined by burning a known amount of coal in a crucible at 750°C for two hours following the standardized procedure. The remaining residue (ash) is then carefully weighed out to calculate the ash content according to the following equation:

\[ \text{%Ash} = \frac{\text{Residue remaining after burning (g)} \times 100}{\text{Initial weight of coal (g)}} \]

Average ash contents of the coal samples with different dilution degrees were determined for:

- **a.** Coal sample with 10% dolomite: 17.69±0.34% on a 95% confidence interval.
- **b.** Coal sample with 20% dolomite: 22.57±0.95% on a 95% confidence interval.
- **c.** Coal sample with 30% dolomite: 26.13±1.15% on a 95% confidence interval.

### 3.4.1 Coal–Dolomite Oil Agglomeration Procedure

The same procedure used in the coal-oil agglomeration tests was followed. Dolomite was added on the mass basis. Each coal-dolomite mixture was vigorously mixed with water in blender for 2 minutes to ensure wetting before being transferred to the conditioning tank. The slurry was then agitated for 10 minutes in the conditioning tank at 900 rpm. At the end of 10 minutes, impeller speed is lowered down to 650 rpm and the oil emulsion is added to the system. The slurry was then further conditioned for an additional 20 minutes. The oil dose was again 10% by weight which means that for a coal-dolomite mixture of 250 grams, 25 grams of oil was emulsified and added to the slurry.

Solids content in the conditioning tank was maintained constant at 50% in each test. The total amount of the material was also kept constant but the dolomite (gangue) concentration was changed from 10% to 30% by weight.

An identical procedure was followed for the tests with fine dolomite.

### 3.5 Coal–Nickel Sulfide Mineral System

The same nickel sulfide ore samples described in Section 5.2 and the same coal samples described in Section 5.3 were used in the following set of experiments.
3.5.1 EXPERIMENTAL PROCEDURE

The procedure of Section 5.4.1 was followed for the coal - nickel sulfide mineral systems. To test the effect of the shape of gangue particles on coal oil agglomeration the nickel sulfide ore samples containing mostly asbestos gangue were added to the coal.

3.6 EXPERIMENTAL METHODS

3.6.1 POWER DRAW MEASUREMENTS WITH TURN-TABLE SETUP

The technique developed to measure power draw in the present study was based on the torque method with a turn-table setup. The design of the turn-table setup was kindly borrowed from Dr. Tesfaye Negeri (personal communication, 2002). The tank was placed on a low friction turn-table that rotates when the impeller is turned on. A lever arm was attached horizontally to the base of the cell and thus tangentially to rotation of the shaft. This was then connected via thin wire to a gauge (Figure 29). Since the force imparted onto the fluid by the impeller causes an equal and opposite force on the lever arm, the gauge reading was directly related to the force applied to the pulp. The force required to resist to the rotation of the impeller in the pulp was measured by the gauge. Gauge readouts were recorded in a computer.

A linear relationship between gauge readout and force was obtained. The force reading $F$ is converted to give power in watts ($P$) by using the equation,

$$P[\text{Watt}] = F[\text{g}] \times r[\text{m}] \times 0.009807 \times 2\pi \times \text{RPM} \times 0.016667$$  \[6\]

$P$ : Power input in Watt,
$r$ : The distance of the pulley from the cell in m,
RPM: The shaft speed in rotations per minute,
$F$ : Gram-force (balance reading).

As power in watts was calculated using the above formula, power dissipation in kilowatts can also be calculated by dividing the power by the pulp volume [$\text{m}^3$].

$$\text{Power Dissipation [kW/ m}^3\text{]} = P[\text{Watt}]/1000 \times \text{Pulp Volume [m}^3\text{]}$$  \[7\]
Figure 29 – Turn-table setup which has been used during nickel sulfide flotation tests.

The main difference of this setup from the one designed by Dr. Negeri was the use of the force gauge instead of a balancing weight (Figure 29). In our tests a large number of measurements were needed for reliable results and the use of the force gauge hooked to a computer allows for continuous acquisition of the data.

As the power draw measurements were used to characterize aggregation in the conditioning tests, every factor that affects aggregation would also affect the power draw measurements. Particle aggregation depends on the intensity of conditioning and the mixing intensity is influenced by the relative speed and configuration of the impeller as well as the geometry of the mixing container (Mhaisalkar et al., 1985). Two types of mixing containers were used in this experimental work. In the first part of the study, the conditioning and flotation tests of nickel sulfide ore were carried out in a 2.5 L Denver flotation cell. However, as these tests gave some controversial results and in order to test validity of the turn-table setup and to create a more stable system, two modifications have been introduced. A specially designed
conditioner tank was used instead of the flotation cell and a LIGHTNIN LabMaster™ mixer was used instead of the Denver Flotation machine. The setup that has been used is shown in Figure 30. The technical drawing of the conditioning tank is given in Appendix F and G.

![Diagram of the conditioning tank setup](image)

**Figure 30**– The turn-table setup which was used at conditioning tests in the 2nd and 3rd part of the study.

The two major differences between flotation cell and conditioning tank were the presence of four baffles and an annular lid located at the pulp level. The role of baffles in a mechanically agitated vessel was to promote the stability of power drawn by the impeller, to prevent swirling and formation of the vortex and thus, to improve the mixing of liquid (Lu
W.M. et al., 1997). Without the baffles, tangential velocities coming from the impeller causes the entire fluid mass to spin with a little shear. The suspension behaves under the influence of centrifugal forces and a vortex forms all the way down to the impeller.

Subrahmanyam et al. (1990) compared a baffled and a non-baffled cell. Particle collisions and particle aggregation were very effective in baffled cells whereas a little particle aggregation was observed in the unbaffled cells. Figure 31(A, B) shows unbaffled cells where swirl and vortex formation are common in low viscosity fluids. Baffled cells with radial or axial flow impellers produce top to bottom turnover with maximum shear rate (Subrahmanyam and Forssberg, 1990) (Figure 31 C, D).

Figure 31– Unbaffled tanks with minimum shear rate (A, B) and baffled tanks with maximum shear rate (C, D) (Subrahmanyam et al., 1990). (Courtesy of Elsevier)

Another feature of the conditioning tank that was critical to the outcome of the experiments was the presence of an annular lid. This annular lid in the shape of a disc was designed to eliminate the suction of air from the conditioning tank (Engel et al., 1997). In the study of Engel et al. (1997), the use of an annular disc fitted just below to the level of the pulp was proven to be the simplest modification that can ensure maximum shear.
Figure 32 illustrates the comparison of the power inputs of the cells with and without the annulus lid. It was shown that in small scaled laboratory cells, an error up to 50% could occur at high speeds due to the induction of air.

This effect was explained by the formation of an air vortex around the impellers which reduces the effective work input and shear at any rotation speed. The annular disc prevented the vortex inducement of air into the cell when operated at high speeds (Engel et al., 1997). For this reason, a similar annular disk was designed and used in the experiments. The position of the lid was adjustable. The height of the disk was adjusted by sliding and fitting it to a position where it was just below the pulp level. By keeping the position of lid at constant level the liquid volume hence the liquid depths for all repetition tests could be kept the same.

The impeller speed was kept constant throughout the conditioning and flotation stages. Since the impeller speed stayed stable in all tests, a tachometer was not needed.
3.6.2 ELECTRICAL METHOD

The power input was firstly measured by using the torque turntable method, and then it was verified by an independent measurement using an electrical method. The electrical measurements were recorded using a computer. The circuit diagram for measurement of power input to the impeller is presented in Figure 33. An ampere-meter and a voltmeter were attached to the system to measure the current and voltage values.

![Figure 33— Electrical measurement circuit used in the conditioning tests of coal-oil agglomeration.](image)

Voltage and current were measured across the motor of impeller and the power draw was calculated from the following formula,

\[ P = I \times V \]  

Where \( P \) = Power draw,

\( I \) = Current across the motor of the impeller at load condition,

\( V \) = Voltage across the motor of the impeller at load.
4. RESULTS AND DISCUSSION

4.1 FLOTATION OF NICKEL SULFIDE ORE

In this thesis, the agglomerate flotation was tested as a way of improving the flotation performance of fine nickel sulfide ore. Agglomerate flotation tests were performed under a variety of conditions to investigate the following aspects:

- Efficiency of agglomerate flotation
- Characterization of agglomeration by power draw measurements
- Optimization of agglomerate flotation by power draw measurements

Firstly, flotation tests were initially carried out to enable a conclusive demonstration of the efficiency of agglomerate flotation. It was assumed that the degree of particle agglomeration would affect the effective particle size, which in turn would affect the flotation performance.

After ensuring that the agglomerate flotation is an efficient method, power dissipation in the flotation cell was monitored to:

- characterize the aggregation of particles,
- study the effect of power consumption during conditioning and,
- find a relationship between energy input in conditioning and flotation efficiency.
4.1.1 EFFICIENCY OF AGGLOMERATE FLOTATION

a. Effect of Oil Addition on Nickel Recovery

Figure 34 – Effect of emulsified oil addition (500 g/t) on Ni recovery. (EDO = Emulsified diesel oil).

Figure 34 shows the effect of oil addition on Ni recovery at two different solids concentrations in the conditioning stage. The experiments were 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 cell) and conditioned for 10 minutes prior to diesel oil addition. Thereafter, the pulp was conditioned for another 5 minutes. The conditioning was carried out either at 10% or at 30% solids content whereas the flotation tests were carried out at 10% solids for 30 minutes. 30 g/t of Terric 407 frother was added at last in all the tests and conditioned for one minute. Five concentrates were collected after 2, 5, 10, 20 and 30 minutes of flotation and multiple tests were performed for each condition.

The flotation tests performed after conditioning at 10% solids in the absence of oil gave lower recoveries than the ones in the presence of oil. However, 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. These results imply that flotation recovery was not significantly affected by oil addition.

The reason why Ni recovery values were not improved could probably be attributed to a high degree of entrainment in the system. In addition to the evaluation of the grade and recovery in the flotation tests, water recovery was also determined.

The results showing the effect of oil addition to flotation pulp during the conditioning stage on flotation are summarized in Table 7. The table shows the mean values of flotation performance indicators for the tests carried out either with 500 g/ton of emulsified diesel oil or without it. The table also includes the confidence in the difference between the means of each indicator obtained from statistical t-tests for experiments with and without oil.

### Table 7– Effect of oil addition on the flotation performance

<table>
<thead>
<tr>
<th>Flotation Performance Indicators</th>
<th>No Oil Addition</th>
<th>With 500 g/t Oil</th>
<th>Confidence in the Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
<td>Average</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Grade</td>
<td>2.6± 0.4</td>
<td>4.0± 0.6</td>
<td>99%</td>
</tr>
<tr>
<td>Ni Recovery</td>
<td>55.4±1.1</td>
<td>53.4±2.6</td>
<td>87%</td>
</tr>
<tr>
<td>Water Recovery</td>
<td>10.5±1.5</td>
<td>7.7±1.2</td>
<td>99%</td>
</tr>
</tbody>
</table>

The results summarized in Table 7 confirm that none of the differences between overall Ni recoveries for the two sets of the conditions were significant on a 95% confidence interval. On the other hand, the grade was found to be increasing from 2.6± 0.4% to 4.0± 0.6% with oil addition and this difference was significant on a 99% confidence interval.

The relationship between Ni grade and Ni recovery for all the tests is summarized in Figure 35. The figure demonstrates that in spite of a high scatter of the results, higher Ni grades could be detected in the tests with 500 g/t of oil.
These results confirmed that agglomerate flotation of this particular nickel sulfide ore is beneficial. It was found that the selectivity of the process can be significantly improved with the use of agglomerate flotation.

4.1.2 CHARACTERIZATION OF AGGLOMERATION BY POWER DRAW MEASUREMENTS

a. Correlation of Power Draw with Agglomerate Flotation

As the flotation results revealed that oil agglomeration is possible for this particular ore, power draw measurements were performed on the same system to characterize particle agglomeration in the flotation cell. Figure 36 demonstrates the effect of oil dosage on power dissipation during conditioning at 30% solids content. Experiments with various diesel oil dosages were repeated three times and the mean power dissipation values were presented in the figure.
Figure 36— Effect of oil dosage on power dissipation during conditioning at 30% solids content.

The time range between 0 and 600 seconds represents the 10 minute conditioning with xanthate prior to the addition of oil. The oil was added at the end of this conditioning period. As the figure reveals, in all cases the power dissipation per cubic meter started to increase at 600 seconds. This increase could be attributed to the addition of diesel oil. However, the power dissipation progressively increased throughout the tests irrespective of whether or not the oil was added to the system. This indicates that the measurements of changes in power dissipation could not be used to identify the effect of parameters such as oil dosage and conditioning time on the agglomerate flotation of the tested ore.

Power draw values obtained from the tests were compared with the results of flotation tests performed at the same set of conditions. The experiments were 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 addition, thereafter the pulp was conditioned for another 5 minutes. 30 g/t of Terric 407 frother was added at last. A single concentrate was collected after 5 minutes of flotation and multiple tests were performed for each condition.
In order to assess the correlation between power draw and a number of flotation performance indicators, the coefficient of determination (R² value) and the correlation probability were calculated. Coefficient of determination is the square of correlation coefficient (R) which indicates the strength and direction of a linear relationship between test variables and it determines how well the regression line approximates the real data points. The results are summarized in Table 8.

Table 8 – Correlation of power dissipation with various flotation parameters as a function of varying agglomerate flotation chemistry.

<table>
<thead>
<tr>
<th>Flotation Parameter</th>
<th>Coefficient of Determination (R²)</th>
<th>Probability of Correlation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentrate Yield (%)</td>
<td>0.36</td>
<td>98.64%</td>
</tr>
<tr>
<td>Water Recovery (%)</td>
<td>0.26</td>
<td>95.60%</td>
</tr>
<tr>
<td>Ni Grade (%)</td>
<td>0.39</td>
<td>99.04%</td>
</tr>
<tr>
<td>Total Ni Recovery (%)</td>
<td>0.10</td>
<td>76.83%</td>
</tr>
<tr>
<td>Ni Recovery by Entrainment (%)</td>
<td>0.29</td>
<td>96.90%</td>
</tr>
<tr>
<td>Recovery by True Flotation (%)</td>
<td>2.95 × 10⁻⁴</td>
<td>5.03%</td>
</tr>
</tbody>
</table>

None of the obtained R² values presented in Table 8 were higher than 0.4, which indicates that in all cases only 40% of the data could be explained by a linear relationship. No strong correlation was found between flotation parameters and the system power dissipation as a function of varying oil addition. However, in the majority of cases, the probability of correlation was above 95%. This indicates that while a correlation may theoretically exist, the employed measurement techniques were not reflecting it. This is mainly attributed to the lack of sensitivity in power draw measurements to small changes in the degree of aggregation of the mineral suspension, arising from a small proportion of Ni sulfide particles in the ore.

b. **Effect of Solids Content on Power Draw**

Since increased particle concentration during conditioning stage should affect the probability of particle-particle collision leading to an increased degree of particle agglomeration, additional tests were designed to test this hypothesis.
A series of power draw measurements were carried out using different percentages of solids concentrations ranging from 5% to 20% both in conditioning and flotation stages. The pulp was conditioned with 180 g/t of Flex-41 collector for 10 minutes, then 500 g/t emulsified oil was added to the system and further conditioned for 5 minutes. Finally, 30 g/t of Terric 407 frother was added to the cell and conditioned for another minute just prior to flotation. The raw power dissipation measurements over a 10 minute conditioning period as a function of %solids concentration are shown in Figure 37.

![Figure 37](image)

**Figure 37**– Power draw measurements at various % solids concentrations in the conditioning stage.

The figure indicates that increasing the solids content resulted in higher power dissipation per cubic meter in the cell. If this graph is plotted as power dissipation versus % solids content as in Figure 38, it becomes clear that there exists a strong linear correlation between slurry solids content and power draw. The mean power dissipation values were calculated using the raw power dissipation data at the conditioning stage.
c. Effect of particle agglomeration on rheology/power draw measurements

Rheology is strongly dependent on the interactions between suspended particles in a concentrated suspension. Viscosity of highly concentrated suspensions is high due to the greater resistance of flow as the particles move out of each other’s way. Even more resistance arises when the particles form aggregated structures. The aggregates, by enclosing and immobilizing some of the continuous phase, increase the apparent phase volume and the viscosity (Barnes et al., 1989).

It was assumed that since the rheology of agglomerating slurry was known to change quite dramatically when it is conditioned with oil (as in the coal-oil agglomeration tests), the power draw should also vary considerably.
Starting from this point of view, it was expected to find a relationship between the power draw in the conditioning stage and rheological properties of the slurry. Using developed power dissipation - yield stress curves, the changes in the rheological properties of the system during conditioning were compared. Casson yield stress was calculated from the formula:

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

[11]

where, \( \tau \) is shear stress
\( \tau_C \) is Casson yield stress
\( \eta_C \) is Casson viscosity coefficient
\( \gamma \) is shear rate

The Casson yield stress values corresponding to the %solids content in the flotation stage were plotted from the results of Kilickaplan (2009) (Figure 39). Error bars on this plot and elsewhere represent 95% confidence interval of the mean. However, it is difficult to map these values in this figure since they are very small.

![Error Bars - 95% Confidence Interval](image)

**Figure 39**- Relationship between solids content and Casson yield stress (Kilickaplan, 2009).

This figure implies that Casson yield stress increases with increasing solids concentration. It was confirmed that the increase in concentration of the finely ground tested ore
has a strong influence on the apparent viscosity and on the yield stress of the suspension. In the case of this particular ore, it is likely that the rheological properties of this system mainly result from the presence of fibrous gangue particles. The increased concentration of such fibrous particles may mask the possible effect of agglomerating nickel sulfide particles on the rheology of this system.

In order to determine the correlation between power draw and rheology, power dissipation values were plotted against Casson yield stress for corresponding %solids concentration (Figure 40).

Figure 40—Effect of the Casson yield stress of the flotation pulp on the measured power dissipation during conditioning.

Figure 40 demonstrates that, as expected, power draw changes due to the variations in the yield stress and a direct relationship exists between the power draw and rheological properties of the slurry.
Overall, the evaluation of the power draw measurements indicates that the system was more complex than originally expected and it needed further investigation. Strong and significant correlations can be picked up when measuring power dissipation of a system with strong variables such as the solids content in the slurry. However, sensitive measurements with weaker variables such as oil dosage have been unsuccessful.

4.1.3 OPTIMIZATION OF THE AGGLOMERATE FLOTATION BY POWER DRAW MEASUREMENTS

a. Correlation of power input with flotation

Agglomeration process depends on particle-particle collisions which are controlled by the hydrodynamics in flotation cells. As the agglomerate flotation was found to be beneficial for this particular ore, it was then decided to test the effect of energy input on degree of agglomeration and its relation with the flotation performance.

The effect of large step changes in the hydrodynamics of the mineral slurry were investigated by changing the mechanical power input into the flotation cell, that is, the impeller speed. It was assumed that there is an optimum energy input that would result in maximum particle aggregation without breaking up the nickel sulfide aggregates.

In order to ascertain the effect of power input on flotation performance, a number of flotation tests were performed as a function of increasing impeller speed (and consequently power input) during the conditioning stage.

The experiments were performed at 10% solids content with 180 g/t of Flex-41 collector and conditioned for 10 minutes. 500 g/t oil was then added and conditioned for another 5 minutes. 30 g/t of Terric 407 frother was added at last and conditioned for one minute. Impeller speed was varied from 750 to 1800 rpm but in the flotation stage it was kept constant at 1500 rpm. A single concentrate was collected after 5 minutes of flotation and multiple tests were performed for each condition.
The relationship between power input to the flotation cell in the conditioning stage and Ni recovery is presented in Figure 41. As these results reveal, Ni recovery is strongly affected by the power input in the conditioning stage of the flotation process. This was specifically significant in the region below 8 kW/m$^3$, where the power input is similar to that of an industrial size cell.

It seems that at low power inputs (around 2 kW/m$^3$), the nickel sulfides do not form aggregates. Increased power input provides the particles with sufficient kinetic energy to aggregate with the aggregates being characterized by higher floatability. Once the power input exceeds 8 kW/m$^3$, the aggregates no longer grow but instead start to break apart, resulting in poor flotation performance.

It is important to note that all the conditioning tests in the previous section (characterization of agglomeration through power draw measurements) were performed at an impeller speed of 1500 rpm. This impeller speed is typical for bench scale flotation tests. It provides power input in the range of 12-16 kW/m$^3$ and obviously exceeds by far the optimum conditioning intensity for agglomerate flotation of this ore.
Variations in the Ni recovery with the power input may also be explained by the rheological properties of the gangue particles. Rheological properties of this particular system were found to be time dependent (Kilickaplan, 2009). The effect of conditioning on rheology of chrysotile suspension indicated severe changes with time; the conditioning of the pulp reduced the yield stress and the apparent viscosity. The decrease in viscosity may suggest that gangue minerals were either aligned with the flow or large fibrous aggregates were being broken with time resulting in a less viscous system.

Figure 42 illustrates an initial random orientation of elongated particles in the ore at low shear rates (high apparent viscosity), and possible increasing alignment with the flow as the shear rate is increased.

![Figure 42](image.png)

**Figure 42**– Possible alignment mechanism of gangue particles with the increasing conditioning time. The black dots represent the sulfide minerals and the long elongated particles represent the fibrous chrysotile gangue.

Overall, the power draw measurements that were performed to characterize the process of particle agglomeration have not been conclusive. The measurements may not be sensitive enough to record changes in the system, or the content of the agglomerating nickel sulfides is so small (when compared to the content of fibrous gangue) that recording the effect resulting from the agglomeration of such sulfides is impossible. The power draw measurements were found to be much more beneficial in case of system optimization when compared to the characterization of the oil agglomeration.

The reason for poor characterization of oil agglomeration by power draw measurements could result from either the mineralogical composition of the ore or from insensitivity of the used turn-table setup.

The nickel sulfide ore used in the tests is a low grade ore (0.52% Ni) that contains approximately 1% of nickel sulfides. It is likely that the presence of large amount of fibrous
gangue minerals strongly affect the agglomeration process. Chrysotile, being the predominant
gangue mineral, has an unusual fibrous shape with unique combination of physical and chemical
properties. Its complex structure may pose a major challenge in the analysis of the degree of
agglomeration.

At this point of the study, there was a possibility that the turn-table setup was not
sufficiently sensitive. In order to test this possibility, a model system with bituminous coal was
used in the following tests.

4.2 MODEL TESTS WITH THE USE OF BITUMINOUS COAL

Since power draw measurements with nickel sulfide ore gave some controversial results,
the sensitivity of the power draw measurements to the physicochemical conditions was tested
using a well-studied model system in the second part of the thesis.

Coal has been studied by many researchers and oil agglomeration of fine coal is a well
understood concept. Bituminous fine coal constitutes an ideal product to be beneficiated by oil
agglomeration. The process relies on the fact that bituminous coals, if not oxidized, are
generally hydrophobic so that when agitated with a mixture of oil and water, they can be
agglomerated by oil droplets. Since oil droplets in water can only attach to hydrophobic
particles, the hydrophilic mineral matter would remain in aqueous suspension and the process
would be selective.

The following aspects were investigated in the coal-agglomeration tests:

a. Effect of oil addition on power draw,
b. Effect of solids content on phase inversion time,
c. Electrical measurements.
a. **Effect of Oil Addition on Power Draw**

![Graph showing power draw measurements for three coal-oil agglomeration tests.](image)

**Figure 43**– Power draw measurements for three coal-oil agglomeration tests.

Figure 43 shows three power draw curves obtained during the conditioning of the coal slurry at 50% solids content. The coal slurry was mixed at 900 rpm for 10 minutes, thereafter the impeller speed was lowered to 650 rpm and 10 wt% of emulsified oil was added to the pulp. The initial decline in the power draw was caused by the change in the impeller speed. The oil was conditioned in the conditioning tank for another 20 minutes at 650 rpm.

The figure verified the reproducibility of the tests since repeated experiments showed a similar phase inversion phenomenon. The power dissipation was found to increase in all tests as oil was added to the system (at 600 s). The increase was followed by a decrease and power dissipation became stable around 17 kW/m$^3$.

It was observed that the power draw curves of Figure 43 had a similar shape when compared with the results published by Lapidot and Mellgren (1968) (Figure 16). This means that the used turn-table setup was sensitive enough to detect rheology changes caused by agglomeration of coal particles. Phase inversion characterized by the peak of power dissipation, could be easily identified from the power draw measurements.
The increase in power consumption during conditioning is likely due to the increased viscosity of the system. The preliminary study carried out by Burdukova (2004) showed that the yield stress of a coal slurry increases dramatically when the particles agglomerate (Figure 17). Thus, it may be concluded that the increase in power draw data measured with the turn-table setup is due to an increase in the yield stress caused by agglomeration.

Overall, these results confirmed that the power draw measurements were reproducible and sensitive enough to follow agglomeration taking place in the slurry of fine coal.

b. Effect of Solids Content on Phase Inversion Time

The effect of solids content on phase inversion time was studied by changing the solids content of the slurry used in the coal oil agglomeration tests. Power draw measurements were repeated three times to allow for statistical evaluation. The phase inversion time was taken as the time when the power dissipation reached its maximum. The mean phase inversion times for the tests with different solids content are plotted in Figure 44.

![Figure 44](image)

**Figure 44**– Effect of solids content in the oil agglomeration of fine coal on phase inversion time. All samples were first mixed at 900 rpm for 10 minutes, then the stirrer speed was lowered to 650 rpm and 10 wt% oil was introduced to the system.
As Figure 44 indicates, the phase inversion time is inversely proportional to the solids concentration. High solids content yields higher aggregation degree and therefore the time to reach the maximum power dissipation (maximum aggregation) decreases. These results demonstrate that conditioning with high solids content enhances spreading of the oil over hydrophobic surfaces which in turn improves the formation of aggregates. The agglomeration of hydrophobic coal particles becomes easier with increasing number of interactions and thus, the phase inversion time decreases. Similar findings on the effect of solids concentration have been reported by Swanson et al. (1977) (Figure 14). This confirms that the turn-table setup provided results that were in a good agreement with previously published results.

c. Electrical Measurements

The reliability of power draw measurements was also confirmed by electrical measurements. Figure 45 shows the comparison of power dissipation values obtained by the electrical and turn-table methods.

**Figure 45**– The electrical and power draw measurements of a coal slurry with 50% solids content. The impeller speed was 900 rpm prior to oil addition (600 s), it was then lowered to 650 rpm and oil (10%wt) was added.
The power dissipation was found to be higher for electrical measurements. Mhaisalkar et al. (1985) calculated the net power input from the difference in power input to the motor for a given speed of the impeller at load (impeller in pulp) and no load conditions (impeller in air) (Equation 12);

\[
P = (P_L - I_L^2R) - (P_0 - I_0^2R) \tag{12}
\]

Where \(P_L\) = Power input when impeller is rotating in pulp (with load),
\(I_L\) = Armature current at load,
\(P_0\) = Power input when impeller is rotating in air (with no load),
\(I_0\) = Armature current at no load,
\(R\) = Armature resistance in D.

Since in general \(P=IV\), the equation can be rewritten as:

\[
P = (V_L I_L - I_L^2R) - (V_0 I_0 - I_0^2R) \tag{13}
\]

Where \(V_L\) = Armature voltage across the motor of the impeller at load,
\(V_0\) = Armature voltage across the motor of the impeller at no load.

As shown by Musara and Yu (1993), if \(V_L=V_0\), this equation can be further simplified into:

\[
P = V (I_L - I_0) - R (I_L^2 - I_0^2) \tag{14}
\]

where \(V\) is the voltage across the motor of the impeller.

Voltage across the motor of impeller (V) and the resistance (R) were constant parameters and the results of Musara and Yu (1993) revealed that \(I_0\) was practically constant as well. Basically the current at load (\(I_L\)) was the only parameter that was changing with load.

This explains the reason why higher values of the power draw were obtained with the use of the electrical method. In these tests the measured values were not corrected since \(I_0\) was not measured. Since the curves in Figure 45 are only used to determine the phase inversion time, the actual power dissipation values were not that important. The consistency in the timing of the peaks indicates that the electrical and power draw measurements were well-matched.

The results of similar tests (Lapidot and Mellgren, 1968; Swanson et al., 1977; Burdukova, 2004) and the electrical measurements carried out in this thesis indicate that the turn-table setup with the conditioning tank was working properly. Thus, the next question that arises is to find out whether the poor correlation between power draw and agglomerate flotation was due to the presence of peculiar properties of the gangue minerals.
4.3 MIXED MINERAL SYSTEMS

As the reproducibility of the results obtained from the turn-table setup were verified, the phase inversion phenomenon was further studied.

In the previous experiments on coal-oil agglomeration, the tests were carried out using coal without any additional gangue. However, the ore tested in the section 6.1 contains only about 1 % of nickel sulfides and about 99% of gangue. For this reason the additional experiments were designed to test:

a. Effect of gangue addition (dolomite was used) on coal oil agglomeration (this is so-called dilution effect);

b. Effect of particle size of the added gangue (by adding fine dolomite) on coal oil agglomeration;

c. Effect of the shape of the gangue particles (by adding nickel sulfide ore that contain fibrous gangue particles) on coal oil agglomeration.
4.3.1 COAL – DOLOMITE SYSTEM

a. Effect of gangue concentration

Figure 46 – Effect of dolomite addition on power dissipation in the coal-oil agglomeration tests at 50% solids content and 10% dose of oil.

If it is assumed that the phase inversion characterizes agglomeration of the coal particles, the power dissipation curves can be used to study the effect of gangue addition (dilution effect) on coal-oil agglomeration. Figure 46 shows the power dissipation curves for various coarse dolomite additions to the coal system ranging from 0 to 30% by weight. Coal slurries with various dolomite concentrations were first mixed at 900 rpm for 10 minutes, then the stirrer speed was lowered to 650 rpm and the oil was introduced to the system. The peak value of the power dissipation curves was found to be decreasing when the amount of added dolomite increased, making the detection of the phase inversion more and more difficult.
Figure 47— Effect of dolomite concentration on phase inversion. Coal slurries with various dolomite concentrations at 50% solids content were first mixed at 900 rpm for 10 minutes, then the mixer speed was lowered to 650 rpm and oil (10 wt%) was introduced to the system.

The plot of mean phase inversion time versus dolomite concentration is shown in Figure 47. The figure reveals that the phase inversion time increases with addition of dolomite apparently making the agglomeration of coal particles difficult. This means that the time needed to achieve agglomeration increases when gangue content increases. The increase in phase inversion time can be explained by decreasing frequency of successful collisions between hydrophobic coal particles. Probability of interactions between oil-coated coal particles is obviously reduced due to the intervention of dolomite particles.

The effect of gangue addition on phase inversion time is further illustrated by a plot of phase inversion time versus ash content of the coal-dolomite mixture (Figure 48). Coal slurries with various dolomite concentrations at 50% solids content were first mixed at 900 rpm for 10 minutes, then the mixer speed was lowered to 650 rpm and oil was introduced to the system.
A similar conclusion was derived by Swanson et al. (1977) who showed that increasing amount of clay material (fine kaolinite) in the system increases the inversion time of agglomerating fine coal (Figure 15). This further confirmed that the used setup was able to give the similar results to the ones that were obtained by previous researchers.

b. **Effect of Particle Size of Gangue on Phase Inversion Time**

The oil agglomeration process does not only depend on the concentration of the gangue minerals but also on the size of such particles. In order to test the effect of the particle size of the gangue, a series of tests were performed with a finer dolomite sample. The relationship between gangue concentration and the phase inversion time for the same dolomite sample but with different particle size distribution is given in Figure 49. The error bars shown represent the 95% confidence interval of the mean.
Figure 49 – Effect of concentration and particle size of dolomite in the gangue on phase inversion time for agglomerating fine coal slurry. Coal slurries with various dolomite concentrations at 50% solids content were first mixed at 900 rpm for 10 minutes, then the mixer speed was lowered to 650 rpm and the oil (10% by wt) was introduced to the system.

The comparison of two sets of the data showed that the influence of the fine gangue particles in the pulp is more pronounced. As the concentration of fine dolomite particles added to the system increases, it becomes more difficult to aggregate the hydrophobic coal particles. This effect can be attributed to the high surface area of the added dolomite. Large number of interacting fine gangue particles with high surface area can interfere with coal-oil and coal-coal particle interactions making oil agglomeration more difficult.

4.3.2 COAL – NICKEL SULFIDE ORE

a. Effect of Particle Shape of Gangue -Nickel Sulfide Ore Addition

In order to provide a link between the agglomerate flotation tests performed on nickel sulfide ore and the oil agglomeration of fine coal, a third series of tests were carried out by adding the nickel sulfide ore as a gangue to the agglomerating fine bituminous coal.
Figure 50– Effect of finely ground nickel sulfide ore addition to the fine bituminous coal slurry on phase inversion of the agglomerating coal. Coal slurries with Ni sulfide ore at total solids content of 50% wt was first mixed at 900 rpm for 10 minutes, then the stirrer speed was lowered to 650 rpm and oil (10% wt) was introduced to the system.

It was observed that the phase inversion phenomenon disappeared entirely from the experimental curves (Figure 50) even when the nickel sulfide ore added to the agglomerating coal was only in the range of 5%. These findings imply that the shape of the dominating fibrous gangue particles in the studied nickel sulfide ore has much more pronounced effect on rheological properties of the tested pulps than any other parameter. This result makes also clear that the aggregation of nickel sulfide particles taking place in the presence of fibrous gangue (e.g. chrysotile) cannot be detected by the experiments relying on the power draw measurements.
5. CONCLUSIONS

1) Agglomerate flotation is a very promising process for treating fine fraction of the nickel sulfide ore with “asbestos” gangue. Even though the flotation recovery was not significantly improved by oil addition, there was a significant increase in the concentrate grade. This suggests that introducing oil to the system resulted in agglomerate flotation.

2) The sensitivity of the power draw measurements to detect the changes in the degree of aggregation in the mineral suspension depends on the mineralogical composition of the system. In the case of a complex system like the nickel sulfide ore tested in this thesis, the changes in power dissipation could not be successfully measured as a function of weak variables such as oil dosage and conditioning time. Strong and significant correlations can be picked up when measuring power dissipation of a system with strong variables such as the solids content of the slurry. On the other hand, in the case of a simpler model system like coal-oil agglomeration, power draw measurements were found to be sensitive enough to follow agglomeration taking place in the slurry of fine coal.

3) No significant correlation was found between power draw and agglomerate flotation performance of the nickel sulfide ore as a function of oil addition. Even though the statistical calculations showed that a correlation may theoretically exist, the experimental results were not reflecting it. The poor characterization of oil agglomeration by power draw measurements is related to the mineralogical composition of the ore. The small portion of nickel sulfide particles in the ore and the complex entangled structure resulting from the presence of large amounts of fibrous gangue minerals probably makes the agglomeration process difficult to be followed by power draw measurements.
4) **Power draw measurements can be used as a method of characterizing the optimum conditioning conditions for agglomerate flotation.** The results of optimization tests revealed that Ni recovery is strongly affected by the power input to the conditioning stage of the flotation process. Power draw measurements were found to be much more beneficial in case of system optimization when compared to the characterization of oil agglomeration in the cell.

5) **Coal-oil agglomeration tests on coal showed that the agglomeration process strongly depends on the concentration, size and shape of the gangue minerals.** The presence of dolomite particles interfering with coal-oil and coal-coal particle interactions made the oil agglomeration process difficult. The time needed to achieve the maximum agglomeration was longer when the gangue concentration was increased which was probably due to the decreasing frequency of successful collisions between hydrophobic coal particles. When the particle size of the added gangue was reduced, it was even more difficult to agglomerate the hydrophobic coal particles due to the large number of interacting fine particles with the large surface area.

6) **The shape and quantity of the fibrous gangue particles in the studied nickel sulfide ore dominated the system behavior.** It was found that nickel sulfide gangue additions as low as 5% made the phase inversion phenomenon to disappear entirely. The studied nickel sulfide ore contains only about 1% of nickel sulfides and more than 80% of “asbestos” gangue. It is, therefore, quite likely that the agglomeration of the particles of nickel sulfides is entirely masked by this peculiar gangue.

7) To sum it up, the results presented in this thesis lead to the conclusion that the presence of fibrous gangue minerals in the flotation feed produced by grinding of the tested nickel sulfide ore has a dominating effect on the performance of the flotation process.
6. RECOMMENDATIONS FOR FUTURE WORK

As the experimental power draw – nickel recovery plot indicates the conditioning intensity probably exceeded the optimum range in most of the agglomerate flotation tests. It would therefore be very interesting to repeat many of the agglomerate flotation tests following the optimal conditioning conditions.
REFERENCES


Morris T.M., (1950). Measurement of equilibrium forces between an air bubble and an attached solid in water, Trans. AIME, 187, 91-95


Yoon, R.-H. (2000). The role of surfaces forces in flotation, Proc XXI IMPC pp B8a- 1-B8a7


APPENDICES

APPENDIX A: Mineralogy of Nickel Sulfide Ore Feed

Feed Mineralogy

Combined Size Fractions
APPENDIX B: Particle Size Distribution of Nickel Sulfide Ore
APPENDIX C: Particle Size Distribution of Coal

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APPENDIX D: Particle Size Distribution of Coarse Dolomite

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APPENDIX E: Particle Size Distribution of Fine Dolomite

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Graph showing particle size distribution with volume percentage on the y-axis and particle size (μm) on the x-axis.

- QA530011B - Average, Monday, June 01, 2009 1:32:34 PM
APPENDIX F: Design of Conditioner Tank (side view) (all units are in cm)
APPENDIX G: Design of Conditioner tank (upper view) (all units are in cm)