A desire realized is sweet to the soul…

Proverbs 13:19a

University of Alberta

Processability of Weathered Oil Sand Ores

by

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Abstract

Processability of weathered oil sands ore remains a challenge in oil sands industry because it is characterized by low bitumen recovery and poor froth quality. Investigating the reasons for poor processability of weathered ores and improving their processability is the goal of this thesis.

Each ore used in this study was characterized. The effects of process parameters (such as temperature and pH) on the processability of weathered ores were investigated. Effects of increased flotation temperature and kerosene addition were studied. Both of these approaches significantly enhanced the processability of the weathered ore.

Oil sand weathering was studied by artificially treating ores in oven under air or nitrogen environment. Effects of solids wettability, formation water loss and oxidation on the processability of laboratory weathered ores were investigated. Processability deteriorated with water loss and increase in hydrophobicity of solids.

Morphological differences between bitumen froths of good processing and weathered ores were identified.

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Finally, to my parents and siblings, I say thank you for all the love, support, encouragement and being there, always.

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The symbols listed below are intended for general references.

Greek

μ	micro
Ω	ohms

Chapter 1

Introduction

1.1 General introduction

A critical look makes it clear that scientific advancement and technological innovations have tremendously transformed the deposit of oil sand ores found in Alberta from a marginal resource to an attractive business investment over the past thirty five years. Oil sands also known as bituminous sands or tar sands are a mixture of solids (coarse and fines), water and bitumen. The bitumen fills up the interstices between the sand grains, either as a continuous phase in high-grade ores or as a discontinuous phase in low-grade ores (Kasperski, 2001). Oil sands can also be viewed as unconsolidated sand deposits impregnated with high molar mass viscous bitumen. At room temperature, bitumen appears like cold molasses with very high viscosity (Masliyah et al., 2004). Oil sands contain bitumen, which is chemically similar to conventional crude oil, but has a lower API gravity (greater density) and a much greater viscosity (Schramm et al., 2002).

Alberta, Canada has the largest known deposit of economically recoverable oil sand with bitumen reserves estimated to be between 1.7 to 2.5 trillion barrels (Morgan, 2001; Alberta Energy, 2002; Munoz et al., 2003). The Athabasca oil sand deposit has been estimated to contain about 830 billion barrels (Morgan, 2001; Masliyah et al., 2004). Using the currently available open pit mining and in-situ extraction techniques, about 300 billion barrels are estimated to be recoverable (Mathieson and Stenason, 2001).

The water-based extraction technology developed in the 1920's by Dr. Karl Clark of the Alberta Research Council remains the fundamental process that is currently employed in oil sand extraction in Canada. Here is a quote from Dr. Karl Clark (1944) "It is time that a theory for hot water separation should commence to emerge". In the hot water-based processes, hot water is added to oil sands to form slurry from which bitumen is liberated by applying some degree of shear to the slurry. The liberated bitumen is aerated and further recovered by flotation (Long et al., 2006; Helper, 1989; Clark, 1944).

Collaborative research work among the industries, universities, and government laboratories have led to more economical and environmentally sustainable operations. Both the surface mining and in-situ techniques of bitumen production are the two main commercial oil sand recovery techniques used in Alberta (Lipsett, 2004). With open pit mining operations, about 95% of bitumen may be recovered under optimal conditions for a good processing ore. However, problem ores and in particular, weathered and "oxidized" ores are characterized with extremely low bitumen recovery (about 20%). The menace caused by this low processability in terms of low bitumen recovery and poor bitumen froth quality is becoming rampant hence; there is a need to address and combat this problem in order to enhance the processability of this type of ores.

1.2 Overview of bitumen production

To obtain some bitumen out of mined oil sand ore is very easy but to economically maximize and have optimum recovery of bitumen with a minimal negative impact on the environment is not. Adequate understanding of the factors that affect the extraction process is needed in order to maximize bitumen recovery from oil sand ores. Athabasca oil sand ores are commercially extracted using both open pit and in-situ techniques. Open pit mining is employed when the deposit of oil sands are found near the surface with thin overburden/shallow deposit of ore, typically from 0 to about 75 meters from the surface. The oil sands are extracted from the earth by using world's largest trucks and shovels. Deeper deposit of oil sand ores with overburden depth greater than 200 meters are extracted using in-situ techniques. In the in-situ technique, a pair of two horizontal wells is drilled parallel to each other and one above the other at a separating distance of about 5m. Steam or/and solvent is injected through the upper injection well to reduce the viscosity and raise the pressure for flow of oil through the lower production well to the surface for further separation. Debate is still on as to how to efficiently and commercially extract oil sand deposits lying between a depth of 75 meters and 200 meters.

Material that has little or no economic value but has to be excavated during the process of mining is known as overburden or waste. One important economic criterion for open pit mining is the amount of overlying waste which must be removed to access the oil sand ores. The ratio of the weight of overburden to the weight of ore deposit is referred to as the stripping ratio as expressed mathematically in equation (1.1). Generally, the lower the stripping ratio, the more likely an ore body is to be mined by open pit extraction techniques.

$$\text{Stripping Ratio} = \frac{\text{Weight of overburden (tonnes)}}{\text{Weight of ore deposit (tonnes)}} \qquad (1.1)$$

Bitumen production using the open pit technique encompasses three fundamental operations: mining, oil sand ore extraction and upgrading (Masliyah et al., 2004). Innovation in mining technology has led to the use of shovel and truck operations as against the draglines used in 1970's. The mined oil sands are conditioned in either slurry pipelines (40-55°C) or tumblers (70-80°C) with the aim of achieving the liberation of bitumen from the sand grains. Liberation of bitumen from sand grains is the first step in oil sand extraction and it is a very important step to the recovery of bitumen from the oil sand ore. Bitumen droplets that have been liberated from sand grains attach to bubbles and float to the top of the slurry in the primary separation vessels by the buoyancy force to form a bitumen-rich froth product. In the froth treatment plant, the bitumen-rich froth is processed with the addition of diluents (naphtha or paraffin) to reduce the viscosity and density of the diluted bitumen. The removal of entrained solids and water is achieved using centrifuges, settlers and/or thickeners. The treated bitumen product from the froth treatment plant is further upgraded to light synthetic crude oil.

By using either cyclo-separators and hydrocyclones, or induced air flotation in mechanical flotation cells and tails oil recovery vessel; the bitumen droplets (mostly un-aerated bitumen) remaining in the slurry may be further recovered (Cymerman and Kwong, 1995). The tailings from the bottom of the flotation cells and the primary separation vessels, is treated by the addition of coagulants or flocculants to consolidate the fines and capture the coarse sand before discharging the water into the tailings pond (Zhou et al., 2004). Figure 1-1 shows a generic process diagram from oil sand extraction (mining) to bitumen upgrading operations.

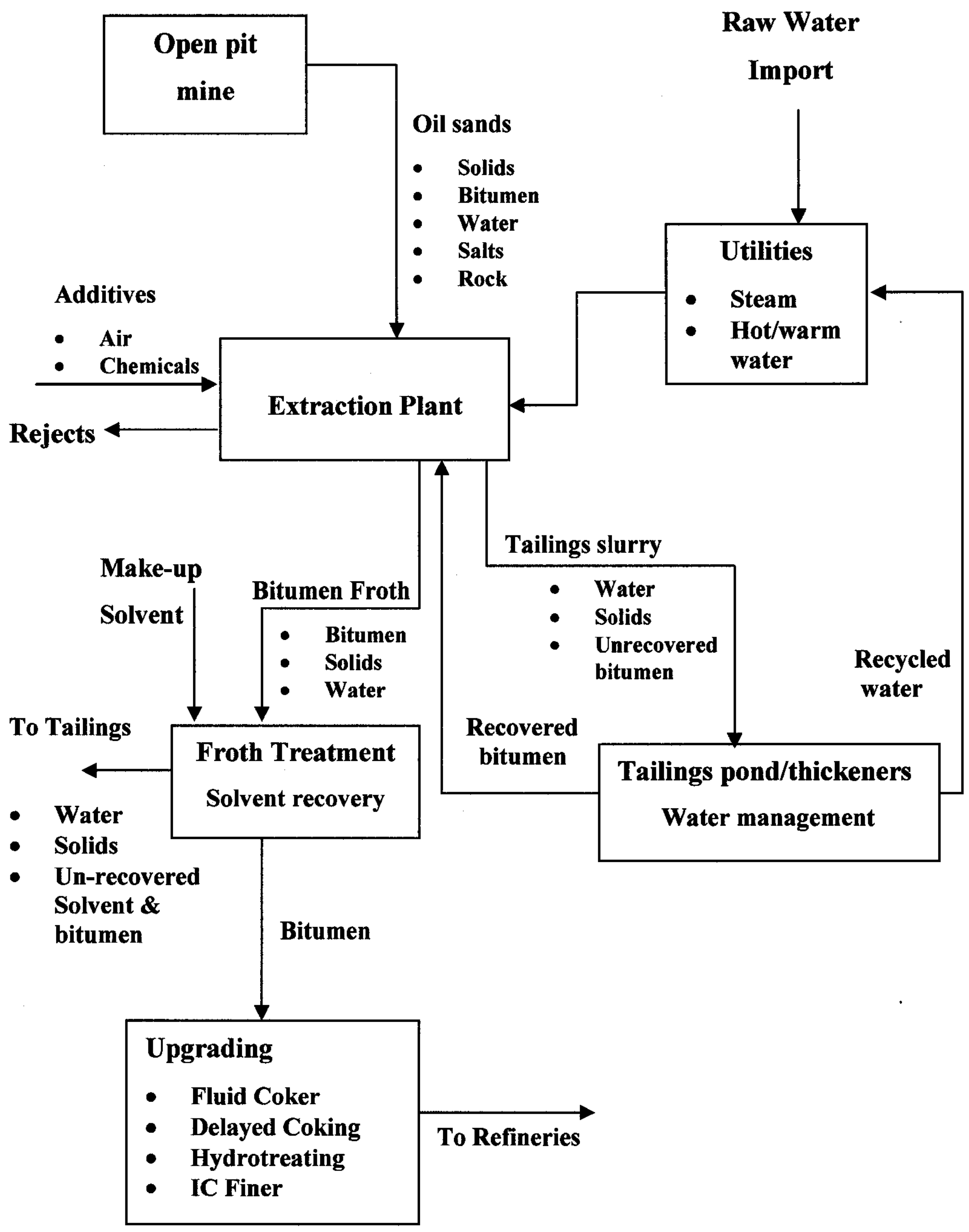

Figure 1-1: Generalized scheme for oil sands processing using water-based extraction processes (adaptation from Advances in Oil Sands Tailings Research by the Fines Tailings Consortium, 1995, Vol. 1).

Figures 1-2 and 1-3 show the oil sand extraction operations using the dragline and shovel/trucks, respectively. The type of operation shown in Figure 1-2 ceased by mid 1990's.

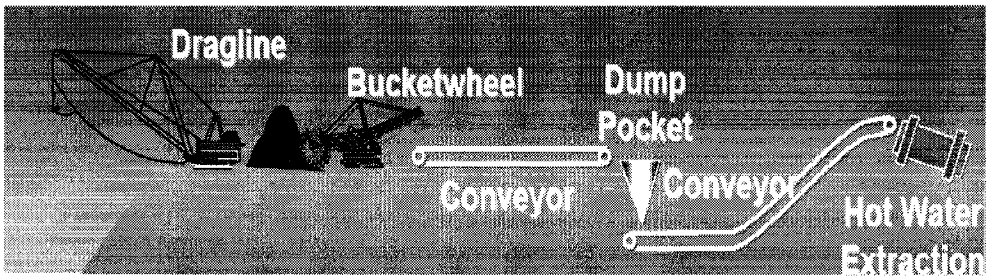

Figure 1-2: Oil sands extraction operations using dragline (Plessis, 2004).

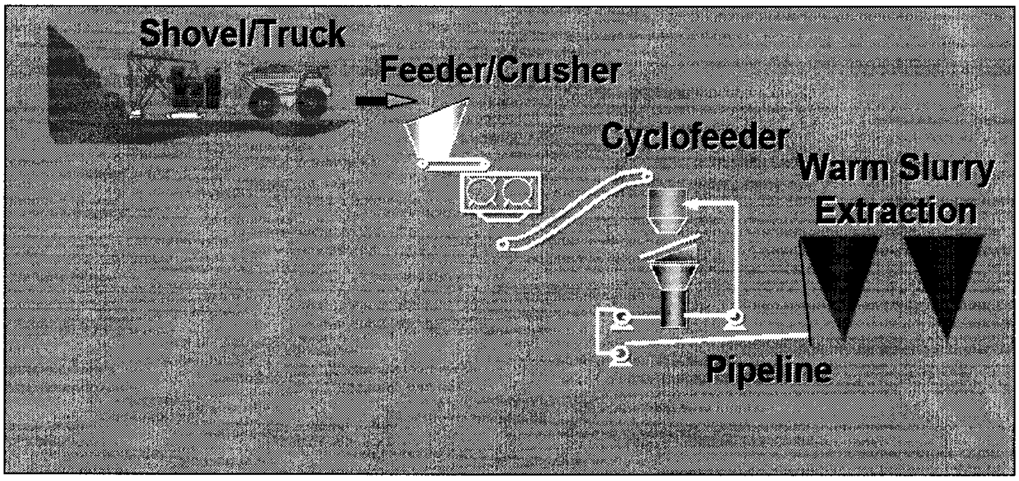

Figure 1-3: Oil sand extraction operations using shovel and truck (Plessis, 2004).

1.3 Thesis Objective

This thesis intends to gain a better understanding of processing weathered ores normally referred to as "oxidized" ore by Suncor Energy Inc and laboratory weathered ores by thermal weathering. Some specific objectives of this thesis are listed below:

- Characterization of Suncor weathered ore and good processing ore.
- Examination of the processability of the Suncor weathered ore and good processing ore by visualization using shaking jar tests.
- Investigation of the effect of process parameters such as temperature, pH, and chemical additives on the processability of weathered ores.
- Wettability study of solids extracted from oil sands with the aim of correlating solids hydrophobicity to the processing behavior of oil sand ores.
- Investigation of the relationship between oil sand ore formation water loss during oil sands weathering and ore processability for laboratory weathered oil sand ores.
- Identification of the differences in the bitumen froth morphology of good processing and weathered ores using optic microscope.
- Investigation of oxidation on ore processability (bitumen recovery and froth quality) by artificially weathering a good processing oil sand in laboratory oven under nitrogen or air environment.

- Investigation of the effect of oil sand ore formation water loss on ore processability.
- Improvement of the processability of weathered ores.

Chapter 2

Literature Review

2.1 Historical perspective of oil sands in Canada

A fur trader named Peter Pond was the first white man to see the Athabasca oil sands in 1778. An early commercial plant, Abasand Oils Ltd was founded in 1936 by Max Ball in the west of Fort McMurray where diesel oil was produced from oil sands. He employed solvents instead of the water-based extraction processes to extract bitumen from the oil sand because solvent extraction is more environmentally friendly (Morgan, 2001). In the 1967, Great Canadian Oil Sands, GCOS (now Suncor Energy Inc) initiated and developed the first large scale surface mining operation of oil sand ores by applying Clark Hot Water Extraction process (CHWE). In the late 1970s, Syncrude Canada Ltd commenced its open-pit mine operation at Mildred Lake, Fort McMurray area. In 2003, Albian Sands Energy Inc. started their open pit mining operation to recover bitumen from oil sands. Other potential operators such as CNRL and Exxon Mobil are expected to commence mining and extraction of oil sands (Masliyah et al., 2004).

2.2 Characterization of oil sands

Oil sand ores mined at different locations in Alberta possess different characteristics and properties that influence their extractability using water-based extraction technology (Liu et al., 2005). Deposits of oil sands found in Alberta typically contain 80–85 wt% mineral solids (mainly quartz and clays such as kaolinite and illite), 6–14 wt% bitumen and the formation water making up the balance. Fines (defined as the mass fraction of total mineral solids smaller than 44 μm) may range from 5-34 wt% in mineral solids (Bichard, 1987). Some ores have been confirmed to contain fine solid content as low as 3.0 wt%. Based on the bitumen content in the oil sands, oil sand ores can be classified as low grade, average grade or high grade. Another classification for oil sands is based on the amount of fines reported as percent fines in mineral solids. Table 2-1 shows the classification based on fines and bitumen content in the oil sands. Also, oil sands classification can be based on a combination of bitumen content and fines content. The most commonly identified classifications in this category are low bitumen/high fines, average bitumen/average fines and high bitumen/low fines. Other classifications include low bitumen/average fines, average bitumen/low fines, high bitumen/average fines, and high bitumen/high fines (Kasperski, 2001).

Table 2-1: Oil sand classification based on content of fines and bitumen (Kasperski, 2001).

	Low	*Average*	*High*
Fines[1] (<44 μm) (wt%)	<6	12-14	>18
Bitumen[2] (wt%)	<9	9-12	>12

[1] expressed as weight percent of total mineral solids

[2] expressed as weight percent of the entire oil sand ores

Content of the soluble ions in the aqueous phase of oil sand slurry is an important property of the ores, as it often determines the bitumen recovery and bitumen froth quality.

2.3 Properties of bitumen

The properties of bitumen are of immense importance in determining the potential of bitumen recovery from oil sand ores. Bitumen itself does not seem to vary from ore to ore in the same geographical location.

2.3.1 Bitumen viscosity

The viscosity of bitumen plays an important role in the extraction of oil sand ores. Viscosity can be defined as a measure of the resistance to fluid flow. It describes the internal friction of a moving fluid. Bitumen viscosity affects the various steps in ore processability such as the breaking and reduction of oil sand lumps, liberation of bitumen from sand grains and aeration of the liberated bitumen. According to Sepulveda et al. (1978), the viscosity of bitumen from Utah oil sands was found to be about two orders of magnitude greater than Athabasca bitumen. Studies have shown that blending of bitumen with light distillates (especially naphtha, high pressure light gases or pure organic solvent) can significantly reduce the bitumen viscosity (Seyer and Gyte, 1989).

2.3.2 Bitumen density

The bitumen density for Athabasca oil sands ranges from approximately 990 kg/m^3 to 1060 kg/m^3, depending on the temperature (Robinson, 1984). The densities of water and bitumen are very close to each other. There exists an inverse relationship between bitumen density and temperature. The density of bitumen decreases with an increase in temperature as shown in Figure 2-2. Figure 2-2 provides a comparison between the density of water and density of bitumen as a function of temperature.

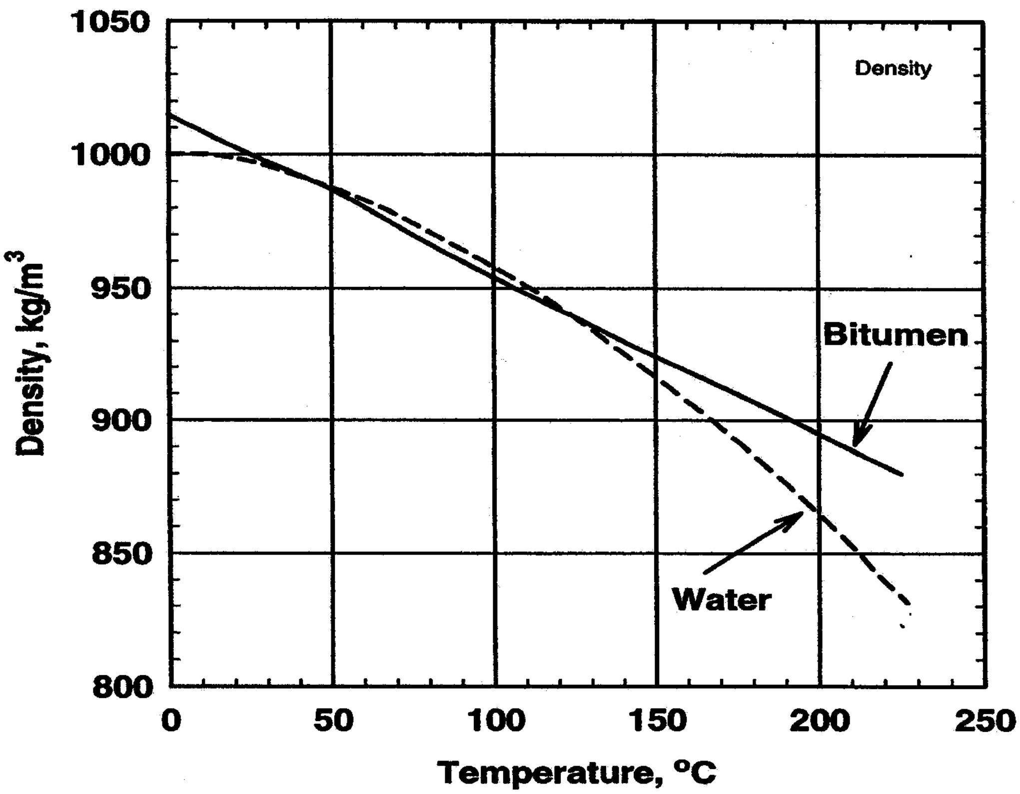

Figure 2-1: Comparison of densities between bitumen and water as a function of temperature (Masliyah et al., 2004).

In the range of oil sands extraction temperature, one can assume that both the bitumen and water have the same density.

2.4 Oil sand extraction techniques using water-based technology

Water-based extraction technology is the commercially employed process for bitumen recovery from Athabasca oil sands. Commercial bitumen recovery from Athabasca oil sands using the water-based technology was successful because of the fact that the sand grains in the Athabasca oil sands are hydrophilic or "water wet" (Masliyah et al., 2004). According to Mossop (1980), there exists a thin layer of water surrounding individual sand grains. The presence of water film around the sand grains in the Athabasca oil sands deposit has been inferred from the initial stage of oil sands development (Clark and Pasternack, 1932; Ball, 1935; Clark, 1944). The thickness of the thin water film between sand grains and bitumen was estimated to be about 10 ηm (Takamura, 1982; Hall et al., 1983) but there is no experimental verification of the existence of such a water film.

Bitumen recovery in a water-based process can be sub-divided into two important steps: conditioning and flotation. According to Clark (1944), the conditioning step is the key step to the bitumen extraction process. During the conditioning step which occurs in a hydrotransport pipeline, mechanical shear is applied to the ore slurry. The applied shear results in the break down of oil sand ore lumps, followed by the liberation of bitumen from sand grains. The liberated bitumen droplets then attach to air bubbles (Clark, 1945). Any coagulation of fines during the conditioning stage can trap bitumen, and hence results in poor bitumen froth quality (Wallace et al., 1989). In the primary flotation step which occurs in primary separation vessels (PSV), aerated bitumen floats to the top of the PSV to form bitumen froth (Kasperski, 2001). Any factor that leads to poor bitumen liberation from sand grains or/and poor aeration of the liberated bitumen would result in poor processability of oil sand ores The extraction of ore using a water-based technology requires some combination of mechanical energy and thermal energy to maximize the processability of the ore (Sury et al., 1993).

Some steps involved in the extraction of bitumen from oil sand using the water based technology are detailed in Sections 2.5.1 to 2.2.5.

2.4.1 Oil sand ore slurry preparation

Crushed lumps of oil sand ores are conveyed through a conveyor belt and then mixed with a combination of fresh and recycled warm/hot water in mixing boxes, cyclo-feeders or rotary breakers to reduce the bitumen viscosity. Chemical additives may be added at this stage.

2.4.2 Ablation of oil sand lumps

By mixing oil sand lumps with hot water and subsequent application of mechanical shear to the slurry in a tumbler, rotary breakers or hydrotransport pipelines, the outer layer of the lumps becomes heated which results in a reduction in the bitumen viscosity. As a result of the applied thermal energy, the outer layer of the lump exposed to the warm water can be sheared and ablated away. The newly formed oil sands outer layer is then heated and eventually ablated. The process of a fresh lump getting exposed to the warm slurry and getting sheared is repeated until the whole oil sand lumps is ablated away (Masliyah et al., 2004).

During this process of ablation, thermal energy and mechanical energy are employed to break down the oil sand lumps in order to achieve relatively well dispersed slurry (Schramm et al., 2003). This step does not necessarily depend on water chemistry.

2.4.3 Liberation of bitumen from sand grains

This is a very critical step in oil sands extraction. There is a need to achieve good liberation of bitumen from the sand grains in order to have a high bitumen recovery from oil sands and good bitumen froth quality. Bitumen disengagement from mineral solids is favoured if the mineral solid surface can be made more hydrophilic since a lowering of surface-free energy will accompany the separation. In the liberation step, bitumen thinning leads to bitumen recession to form distinct bitumen droplets which are subsequently liberated from the sand grains by the application of thermal energy and mechanical shear (Masliyah et al., 2004). The phase separation is enhanced by the effects of mechanical shear and disjoining pressure (Schramm et al., 2003). Reduction in the interfacial tension leads to reduction in the energy of interaction between the mineral solid and bitumen (Kasperski, 2001). This step is highly dependent on the temperature of the slurry and water chemistry. Figure 2-3 shows a model of bitumen recession from a sand grain surface.

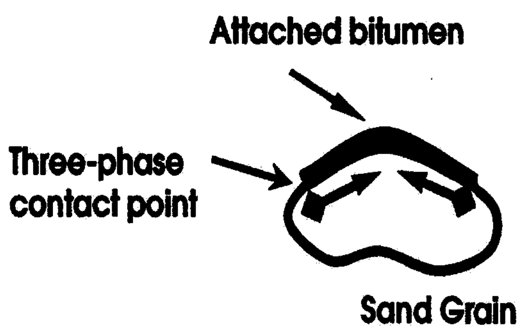

Figure 2-2: Bitumen liberation from a sand grain surface (Masliyah et al., 2004).

2.4.4 Aeration of liberated bitumen

The liberated bitumen attaches to available air bubbles. In the low temperature process (temperature less than 35°C), the bitumen attaches to an air bubble while at higher temperatures (temperature ranging from 45°C to 80°C), the bitumen engulfs an air bubble (Masliyah et al., 2004). As the densities of bitumen and water are nearly the same over the temperature range used in the water-based extraction processes, to achieve good separation of these two phases, there is a need for bitumen aeration in order to increase the density difference between bitumen and water.

The role of gas bubbles in bitumen recovery has been studied by Drelich et al. (1995). They found that when air was eliminated during the digestion stage in oil sands processing, poor bitumen recovery was observed. On the contrary, when the oil sands were saturated with dispersed air during the digestion step, bitumen recovery was enhanced.

2.4.5 Flotation of the aerated bitumen

In this step of the oil sand extraction process, bitumen is separated from most of the mineral solids and water in the primary separation vessel (Kasperski, 2001). The aerated bitumen froth is floated to the top surface of the slurry as froth for collection. The collected bitumen froth can be treated in the froth treatment plant.

2.5 Oil sands weathering

Weathering is a process that naturally occurs. It is mostly common in an oil sand deposit having a shallow overburden. Freshly mined ores have been observed to process better than ores that have been mined and left sitting for some time (Clark, 1923). It should be noted that some freshly mined ores may have undergone in-situ weathering that make them to process as if they have been mined after being exposed for a long time showing poor processability. These types of ores have extractability affected by changes in composition (formation water, bitumen and/or mineral) and some time-dependent reaction either in-situ or after they have been mined. Weathered ores (sometimes also referred to as aged ores or oxidized ores) are characterized with low bitumen recovery and poor bitumen froth quality in low temperature extraction processes. Processability of a mined ore typically deteriorates with storage time. In a laboratory setting, storage of ores in plastic bags and keeping the ore in freezers can only slow down the aging process but may never stop weathering (Kasperski, 2001).

Several reasons have been proposed for oil sand ore processability deterioration by ore weathering. Some proposed explanations for the poor processability include desiccation of formation water, degradation of bitumen, bacterial activity, oxidation of pyrite, changes in water chemistry, to mention a few (Schramm et al., 1984; Schramm and Smith, 1985; Munoz et al., 2003).

2.5.1 Oxidation of oil sands

A marker for weathered ore is an increase in sulphate content due to sulphide oxidation which was observed with storage time (Wallace, 1984; Wallace et al., 1989). The oxidation of pyrite to ferric sulphate as summarized in equation (2.1) below is accompanied with acidification which results in dissolution of both calcium and iron carbonate to yield soluble cations such as calcium, magnesium and iron as well as sulphate anions. Natural carboxylate surfactants, which are known to enhance bitumen flotation, are precipitated by these produced cations leading to an adverse effect on bitumen recovery from the oil sand (Schramm and Smith, 1985).

$$4FeS_2 + 15O_2 + 2H_2O \rightarrow 2Fe_2(SO_4)_3 + 4H^+ + 2SO_4^{2-} \qquad (2.1)$$

Hindrance in flotation of bitumen can be due to increased coagulation of clays and fines as a result of the acidification that accompanied the oxidation process (Wallace et al., 1989). Wallace found that ores stored at 3°C or 20°C showed increasingly poor recovery, which seemed to correlate with increasing sulphate and divalent cation concentrations. Water chemistry can be used as an indicator to determine the oxidation of oil sand ores. Determination of the degradation or oxidation of oil sands can be readily assessed by quantifying the soluble anions and cations (Munoz et al., 2003). By artificially weathering a good processing ore in an oven under air or nitrogen environment to investigate the effect of oxidation on ore processability, Gu (2006) found that the processability of treated ore in nitrogen environment gave a lower bitumen recovery than un-weathered ores, but higher than that weathered in air. This finding suggests that among other factors, oxidation plays a role in oil sands weathering.

2.5.2 Desiccation of oil sand ores

Poor processability of weathered ore has been attributed to many effects. Among them includes Clark's assertion that desiccation of oil sand ores (that is, loss of formation water in the oil sand) is the reason for the low bitumen recovery and poor froth quality. In his study, Clark stated that formation water plays a critical role in the liberation of bitumen from sand grains because water is an internal phase in the bitumen. Also, he reported that bitumen containing water does not adhere strongly to the mineral solids while dry bitumen does adhere strongly to minerals (Clark, 1923; Clark, 1929). It can be said therefore that weathering may have altered the bitumen-solid association due to water leaving the solids or a change in the oil-wet to water-wet nature of the solids.

Loss of the formation water in oil sand ores will result in the direct contact between the bitumen and solid phases thereby making the liberation of the bitumen from sand grains difficult, resulting in poor processability of the weathered ore.

By artificially treating oil sands samples in an oven at temperature of 50°C under controlled conditions, it was shown experimentally that weight loss during the treatment was mainly due to the evaporation of oil sand formation water. Over 90% of the weight loss upon thermal weathering was ascribed to the formation water loss (Liu et al., 2005). According to Gu (2006), by artificially weathering a good processing ore in oven under air or nitrogen environment, formation water loss from the ore plays an important role in oil sands weathering. He reported that when the formation water content of weathered oil sands decreases, the recovery decreases and the froth quality deteriorates. Also, the poor processability of the weathered ore was attributed to an increased hydrophobicity of the solids obtained from the artificially weathered ores where there was significant loss of oil sand ore formation water. The solids obtained from both the original good processing ore and laboratory treated ore in oven without water loss were found to be highly hydrophilic, whereas the solids extracted from treated ores with significant amount of formation water loss became highly hydrophobic (Liu et al., 2005; Gu, 2006).

2.6 Role of temperature in bitumen extraction

The role of temperature on oil sand ore processability has been extensively studied (Bichard, 1987; Schramm et al., 2002; Ding et al., 2004). Laboratory experiments have shown that at a temperature lower than 35°C, there is a significant reduction in bitumen recovery. According to Bichard (1987), bitumen recovery was reduced from about 90% at temperature of 37.8°C to 40% at temperature of 26.7°C for a poor processing oil sand ore. For a particular good processing ore with the addition of illite clay, magnesium ions and calcium ions, the bitumen recovery dropped from between 85% and 90% at temperature of 35°C to about 30% at temperature of 25°C (Ding et al., 2004). It was reported that for an average ore, there was a large reduction in the primary bitumen recovery from 88% at temperature of 50°C to 8% at temperature of 25°C (Schramm et al., 2002). According to the studies conducted by Stasiuk et al. (2004), the observed drastic reduction in bitumen recovery with temperature reduction is due to the bitumen viscosity and that there is a bitumen viscosity threshold at about 3 Pa.s.

There is a significant reduction in bitumen viscosity with increasing temperature (Helper and Smith, 1994). The force of adhesion between bitumen and clay in process water decreases with increasing temperature until a critical temperature value of between 32°C and 35°C. According to Long et al. (2005), bitumen recovery should be operated at a temperature higher than this critical temperature range. Sepulveda and Miller (1978) found that for Utah oil sands, high bitumen recovery was obtained at a temperature of 95°C but the recovery gradually decreased until it abruptly dropped at a temperature below 60°C. Here, the bitumen froth quality was not affected significantly. They credited the low bitumen recovery to increase in viscosity of bitumen due to the decrease in temperature. Bitumen froth obtained from extraction at lower flotation temperature (between 10°C and 30°C) contained more solids and water than bitumen froth obtained at higher temperature of about 80°C (Lam et al., 1995). Also, studies from Masliyah's model show that the increased solids content in froth at low temperature might be a result of the increase in the viscosity of the slurry. By reducing temperature, the extent of air bubble engulfment by bitumen reduces and adhesion of bitumen to silica particles increases (Kasperski, 2001).

2.7 Role of pH in bitumen extraction

In the initial water-based extraction processes (both hot and warm), processability of the ore was enhanced at an alkaline pH by adding chemical reagents such as sodium hydroxide (NaOH), sodium carbonate (Na_2CO_3), or sodium silicate (Na_2SiO_3) (Clark, 1922). Sodium hydroxide is widely used because it is effective, economical and readily available. With the addition of NaOH in the extraction process, which subsequently results in an increased pH, more oil was recovered in the primary step (Clark, 1944). In general, the amount of NaOH added and the maximum recovery obtained vary but bitumen recovery from an ore usually increases to a maximum and then decreases with increasing pH. According to Sanford (1983), there is an optimum for NaOH addition for most ores, beyond which bitumen recovery decreases again.

2.8 Role of kerosene in bitumen extraction

One role of kerosene among many as a collector is to make the bitumen more hydrophobic. This helps in the attachment of air bubble to bitumen (Sury et al., 1993; Hrouda et al., 1995). For the Utah oil sand, a pretreatment step which involves kerosene addition to the oil sand ore is necessary to reduce the bitumen viscosity (Drelich et al., 1995). According to the studies from the oil sands extraction group at University of Alberta, bitumen recovery from oil sand ores that process poorly could be increased significantly by the addition of kerosene to an air stream feeding to oil sands slurry. By using kerosene-coated air bubbles instead of air bubbles, there was an increase in bitumen recovery from about 10% to 98% for the weathered ore from Suncor Energy Inc and from about 20% to 65% for the poor processing ores from Syncrude Canada Ltd, (Wallwork et al., 2003).

2.9 Microscopic study of bitumen froth

Processability of oil sand ores has been related to the type and presence of morphology found in the extracted bitumen froth obtained from such oil sand ores. According to Munoz et al. (2003), the morphological features present in bitumen froths from laboratory samples are similar to those found in commercial operations. Also, in their study, they stated that the bitumen in an oil sand ore is the phase most susceptible to oxidation process. By treating fresh oil sand ores in a low-temperature oven under air revealed that the morphology of bitumen froth obtained using a light microscope and confocal laser scanning microscopy (CLSM) changed drastically as compared to that of fresh ore of the same bulk composition and extraction water chemistry. Morphology of bitumen froth obtained from poorly processing ore shows the presence of low-fluorescence bitumen bands which can be correlated to elevated inorganic solids content in the froth while the morphology of good processing ore shows normal fluorescence bitumen bands. Morphology of froth samples from batch extractions of weathered oil sands ores show areas or bands of low- fluorescence bitumen alternating with bands of bitumen of normal fluorescence.

Figure 2-3 shows the morphology of bitumen froth obtained from a poor processing ore using batch extraction process. The morphology shows both low fluorescence bitumen band (bottom left) and normal fluorescence bitumen band (top right). According to Munoz et al. (2003), froth morphology with bands of low-fluorescence bitumen is an indication of poorly processing ores. Such froth may be said to have elevated inorganic solids content. The low-fluorescence area may also be attributed to differences in chemical composition as compared to the normal bitumen. The dark circular spots correspond to water droplets.

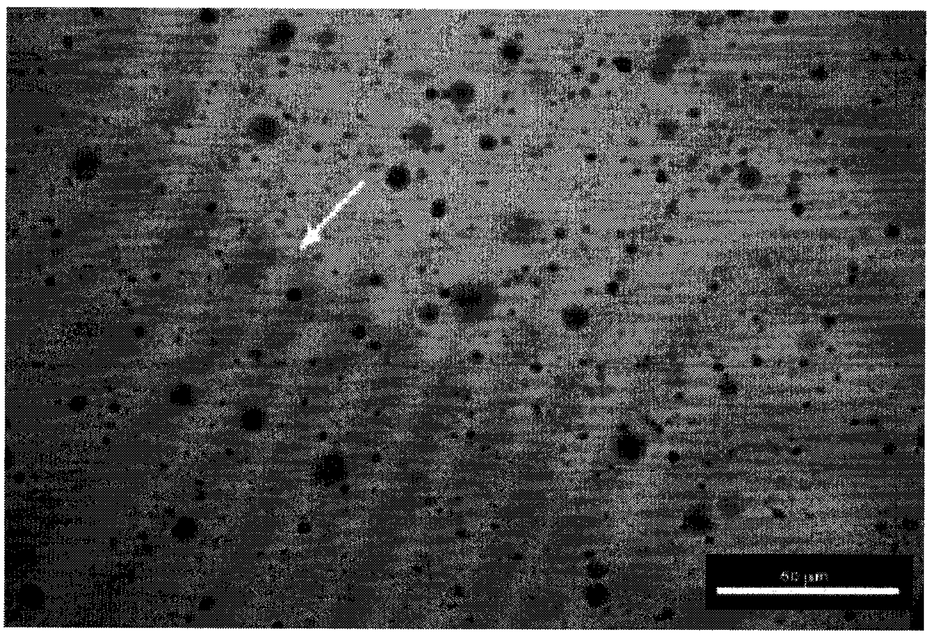

Figure 2-3: Confocal micrograph of bitumen froth obtained from poor processing ores using batch extraction showing an area of lower fluorescence intensity (the direction of the arrow) (Munoz et al., 2003).

Figure 2-4 shows the morphology of bitumen froth obtained from a poor processing ore using batch extraction process with alternating bands of low-fluorescence bitumen band and normal fluorescence bitumen band.

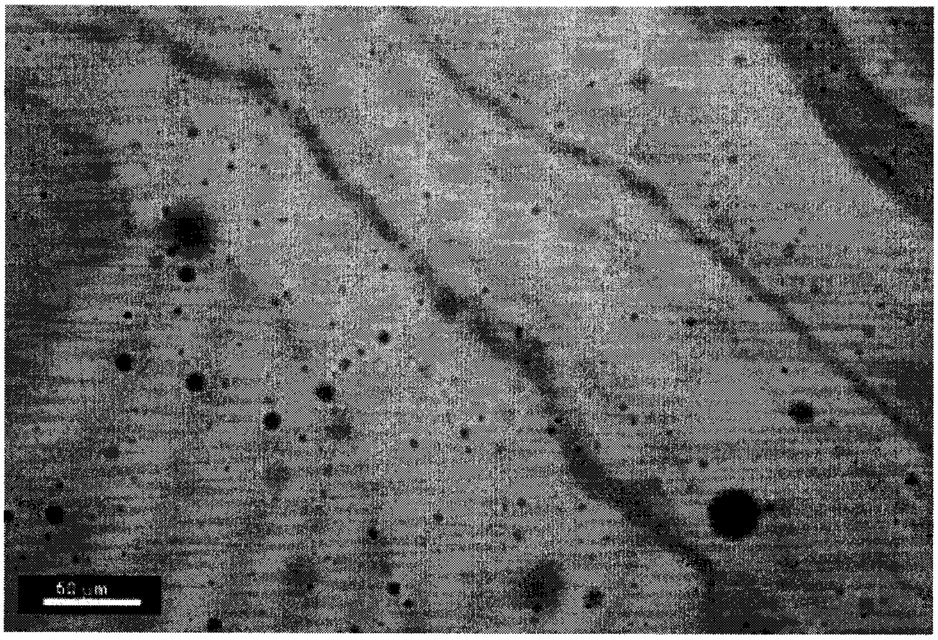

Figure 2-4: Morphology of bitumen froth obtained from poor processing ores using batch extraction indicating alternating bands of high and low fluorescence (Munoz et al., 2003).

Morphology of bitumen froth from a good processing ore is almost featureless, unlike the morphology for bitumen froth from weathered and oxidized ores. The bitumen froth morphology of weathered or oxidized ores has several types of irregular structures and features. The presence of degraded bitumen in froth is always indicated distinctly by various characteristic structures and features. In their study, they identified and named structures such as string, skin or flat, dendrite, sheet and globule structures which were associated with bitumen froth obtained from weathered and oxidized ores (Munoz, 1994; Munoz et al., 2003).

String-type structures are fibre-like with length ranging from about 5 μm to over 100 μm. This type of structure is normally found in bitumen froths having minimal concentrations of degraded bitumen, often found in the very initial stage of bitumen degradation. String-type structures may be associated with inorganic particles. The skin or flat structures appear as skin on normal bitumen. They have irregular shapes. Similar to the string structures, they may be associated with inorganic particles. They are also commonly found in bitumen froths having low concentrations of degraded bitumen. Dendrite structures can be described as string-type structure that bifurcates. They are mostly three-dimensional, and may symbolize trapped inorganic particles. Sheet and globule structures are the most complicated. They are basically three-dimensional and they are the largest type of structures commonly found in highly degraded bitumen froths. These two types of structure can be found in froth sample independently or together. Unlike the other structures earlier mentioned, they are found in froth samples having high concentrations of degraded bitumen with dimensions in x–y size ranges from about 150 μm to over 300μm and depth (in z-axis) of about 200 μm (Munoz et al., 2003). Figure 2-5 shows most of the described structures found in degraded bitumen froth. In this figure, St symbolizes string-like structure, Sk symbolizes skin structure, D symbolizes dendritic structure and Dn symbolizes dendritic network structure. The observed dark circles correspond to water droplets. The arrow in white color points to the bifurcation of a string to form a dendrite structure.

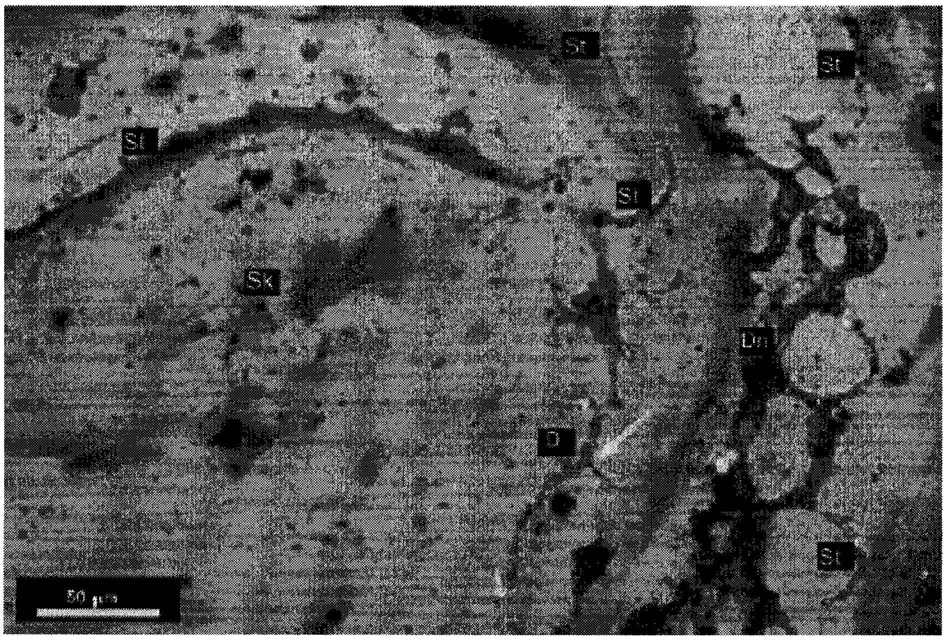

Figure 2.5: Morphology of bitumen froth obtained from oxidized ores using batch extraction showing several types of degraded bitumen structures (Munoz et al., 2003).

The Figure 2-6 shows envelope that traps both solid particles and air bubbles thereby resulting in poor froth quality and formation of very stable foams of the froth obtained from the oxidized ore using batch extraction and formation of very stable foams.

Figure 2-6: Morphology of bitumen froth obtained from oxidized ores using batch extraction depicting large structures referred to as sheets (Munoz et al., 2003).

Figure 2.7 shows morphology of bitumen froth from the ore subjected to air oxidation for 42 hours at oven temperature of 50°C. It showed a very high increase in degraded bitumen structures and can be said to be similar to micrographs of bitumen froth obtained using oven-oxidized ores.

Figure 2.7: Confocal micrograph of bitumen froth from an oven-oxidized ore (42 hours, oven temperature of 50°C) (Munoz et al., 2003).

Chapter 3

Experimental

3.1 Materials

Weathered oil sand and good processing oil sand ores obtained from Suncor Energy, Inc and Syncrude Canada, Ltd, respectively were used for the flotation test and laboratory weathering tests in this study. Deionized water, prepared with Elix 5 followed by a Millipore-UVPlus water purification system (Millipore Inc., Canada), was used where applicable throughout this experimental study. The resistivity of the de-ionized water is about 18.2 MΩcm. Reagent grade toluene (Fisher Scientific) was used as solvent to extract bitumen from oil sands. Mineral oil (reagent grade, Fisher) was used as the oil phase in evaluating surface wettability of solids obtained from the oil sands samples. Reagent grade sodium silicate, Na_2SiO_3 (Fisher Scientific) was added to de-ionized water to prepare 25 wt% of Na_2SiO_3 solution. The 25 wt% Na_2SiO_3 solution was added to Aurora recycle process water at different dosages to make up the water used for the flotation tests with the aim to investigate the effect of Na_2SiO_3 on the processability of Suncor weathered ore. Reagent grade kerosene (Fischer Scientific) was used to study the effect of kerosene on the processability of Suncor weathered ore.

Plant water used during the flotation and shaking jar experiments was Aurora recycle process water from Syncrude Canada, Ltd. The pH of the process water was about 8.5. Concentrations of the soluble cations for the Syncrude good processing ore, Suncor weathered ore and Aurora recycle process water were measured using Atomic Absorption Spectroscopy.

3.2 Experimental procedures

This section provides a detailed description of the procedures involved in carrying out the various experiments in the course of this thesis research.

3.2.1 Oil sands ore storage

Mined oil sand ores from Suncor Energy, Inc (weathered ore) and Syncrude Canada, Ltd (good processing ore) were each homogenized. Samples of 600g each were packed in plastic bags, and stored in a deep freezer at a temperature of about -29°C in order to minimize aging or weathering during the storage.

3.2.2 Oil sands ore assay/composition determination

The bitumen-water-solids analysis was performed by a Dean-Stark soxhlet extraction, followed by direct gravimetric determination of bitumen in the toluene extract. The method requires a fairly large sample to ensure the accuracy of analysis. In this case, 100g of oil sands sample was placed in Whatman filtration thimble (Fisher Scientific) which was then placed in the extractor. The extractor was connected to a round bottom flask containing about 200 mL of toluene (extraction solvent). A condenser was connected above the extractor containing the sample ore. Heat was applied to the bottom of the round bottom flask which resulted in the boiling of the toluene and oil sand ore formation water. By the application of heat to the round bottom flask, water and toluene vapor rose and passed through the bypass arm of the extractor to the condenser. The condenser condensed the vapor and the liquid dripped into the side arm of the extractor.

At the extraction temperature, the density of toluene is lower than the density of water. The water phase therefore settled on the bottom of the side arm while the toluene was on the top due to the density difference between the toluene and water. The condensed liquid kept rising in the side arm of the apparatus until it got to a stage where the condensed toluene reached the top of the side arm. At this point, the reflux of toluene went back into the lower flask and extracted the bitumen into the toluene reservoir in the flask. The solvent is re-boiled, and the cycle was repeated until the reflux of toluene solvent through the Whatman filtration thimble became colorless. When the toluene becomes colorless, it implied that the bitumen in the oil sand has been completely extracted by the toluene. Periodic check was necessary to ensure that the solvent at the bottom of the flask does not get too low.

The water trapped inside the side arm of the extractor was separated from the condensed toluene and weighed. This gave the amount of formation water in the oil sand ore. The mixture of bitumen and toluene collected in the flask was emptied into a 250ml volumetric flask and the volume was adjusted to 250ml by the addition of fresh toluene. The mixture of the bitumen and toluene was then placed in Teflon centrifuge tubes and centrifuged at 25000g force for 30 minutes to remove the solids from the solution. Exactly 5ml bitumen solution was then taken from the centrifuge tube using a pipette and carefully sprayed on a weighed filter paper. The filter paper was hung in a fume hood for about 18 hours so as to achieve complete evaporation of the toluene. The dried filter paper was weighed to obtain the bitumen content in the ore. The solids (mainly fines) in the centrifuged bottle after centrifugation were washed with fresh toluene to obtain clean solids. The clean solids from the centrifuge tube were added to the solids in the thimble. The thimble containing the solids was dried in a vacuum oven at a temperature of about 110°C to remove the residual toluene. After cooling, the solids were weighed. This gave the amount of solids in the ore sample. The final results were confirmed by simple mass balance.

3.2.3 Bitumen extraction using shaking jar test

A precisely weighed (around 20g) homogenized weathered oil sand ore was placed in a glass jar and 30 g of pre-heated Aurora recycle process water (pH of 8.5) was added to the jar to achieve a desired slurry temperature (usually a slurry temperature of 50°C). To investigate the effect of pH on the processability of the weathered ore, higher pH of the process water was achieved by adding sodium hydroxide solution to the process water.

To investigate the effect of kerosene on the processability of the oil sand sample, 0.5g of kerosene per 20g of ore was added to the weathered oil sand ore. After 5 minutes of kerosene addition, Aurora recycle process water was added. The water bath shaker was filled with de-ionized water and pre-heated to the desired temperature of 50°C for the oil sand extraction. Agitation of the jar containing the slurry was conducted in water bath shaker controlled at the desired temperature by a circulating water bath for 20 minutes. After the 20 minutes of shaking, the jar was removed from the water bath shaker. The slurry in the jar was placed on a table to allow the phase separation. After 2 hours of settling, photographs were taken.

3.2.4 Bitumen flotation

For each run of a batch bitumen extraction test, the defrosted ore sample was chopped and homogenized. A desired volume (900 mL) of Aurora recycle process water (pH of 8.5) from Syncrude Canada, Ltd was preheated to the desired flotation temperature (in this experiment, the flotation temperature were 35°C, 45°C and 75°C, respectively) in a 1 litre Denver cell via a water jacket attached to the cell for temperature control. The agitator was lowered and made to come almost to rest on the bottom of the Denver cell. A precisely weighed 200g sample of the homogenized ore was randomly picked up and introduced to the Denver cell containing 900 mL of the preheated Aurora recycle process water.

The oil sands slurry was conditioned for 5 minutes under mechanical agitation at 1500 rpm to achieve liberation of the bitumen from sand grains. Air was then introduced through a sparger at an adjusted aeration rate of 25 mL/minute. Upon air addition, the liberated bitumen droplets were aerated and floated to the top of the slurry to form a bitumen-rich froth in the Denver cell.

By using spatula, three froth samples were collected separately in Whatman filtration thimbles (Fisher Scientific) placed in a jar over incremental flotation periods of 2, 5 and 10 minutes to determine bitumen recovery kinetics. While collecting the bitumen froth, caution was taken to avoid dragging too much water and to ensure that all aerated bitumen were collected within the given periods. To obtain bitumen recovery kinetics information, the initial weight of each empty Whatman filtration thimble placed in jar was recorded and the final weights when filled with bitumen froth were recorded. Also, the initial pH of the water used for the slurry and the final pH of the tailings water were recorded. The three collected froth containers were taken aside for assay using the Dean and Stark method described in Section 3.2.2.

3.2.5 Tailings settling

Tailings slurry obtained from the bitumen extraction experiments were transferred into a one liter graduated cylinder. The slurry was homogenized by inverting the graduated cylinder six times and then allowed the solids to settle. The level of the solids-liquid interface (mud line) was monitored and recorded as a function of time using stop watch.

3.2.6 Extraction of solids from ores

Solids from an oil sands ore sample were collected by water extraction and centrifugation method. The oil sand sample was placed in a centrifuge bottle and de-ionized water at a temperature of 70°C was added. The centrifuge tube containing the slurry was then subjected to agitation using a mechanical shaker for several minutes. The tube was then centrifuged at a speed of 25000g at 22°C for 30 minutes to allow the solids to settle. The mixture of water and extracted bitumen was carefully decanted while minimizing the removal of fines. This process of water washing, centrifugation and decantation was repeated until the solids become clean, judged by colorless washing water (indicating that the bitumen has been completely extracted by the water). The clear water was decanted and the clean solids were left to dry in a fume hood for several days.

3.2.7 Hydrophobicity study of extracted solids using solid partitioning method

Partitioning of the solids in water and mineral oil phases was employed to characterize the hydrophobicity of the solids. About 3g of clean solids obtained using the procedure outlined in section 3.2.6 were placed in a glass bottle. Added to the bottle were 10 ml of de-ionized water and 10 ml of mineral oil (Fischer Scientific). Due to density difference, the mineral oil floats on the top of the aqueous phase. The bottle was shaken using an agitator for about 3 minutes. The mixture in the glass bottle was allowed to settle for phase separation. The hydrophilic solids stay in the aqueous phase while the hydrophobic solids report in the oil phase. Photographs were taken for visual investigation.

After taking the photographs, the mineral oil together with the solids that stayed in the oil phase (referred to as hydrophobic solids) was carefully decanted. The bottle containing water and the solids in the aqueous phase (referred to as hydrophilic solids) was dried in an oven. The hydrophilic solids were quantified by weighing. The percent of solids in the mineral oil phase over the total added solids serves as a measure of the hydrophobicity of the solids.

3.2.8 Sample preparation for bitumen froth morphology study

For morphological evaluation of the froth, a small sample of the bitumen froth collected from the Denver flotation cell was transferred into a hollow flat-bottom metallic cell of dimension 20 x 20 x 2 mm. A hydrophobic cover of silicon was used to cover the froth placed in the metallic cell. This was gently done to avoid destroying the surface of the froth. A siliconized glass cover was chosen because it minimizes wetting of the cover by the water in the froth, which is extremely important in the examination of the bitumen froth under the microscope. The siliconized glass cover was well fitted to the metallic cell by adhesive tape to avoid the direct contact between the microscope lens and the bitumen froth and to avoid evaporation of the water to cause morphological change of the froth due to bitumen coalescence

3.2.9 Weathering of good processing ores under air or nitrogen environment

Laboratory weathering of good processing ore was conducted in a well-controlled oven under either air or nitrogen environment at different well controlled operating conditions. Figure 3-1 shows the schematic diagram for the laboratory treatment of oil sands.

Weathering using air (complete[3] formation water loss)

About 600g of the good processing ore was placed in an aluminum tray. The ore was well spread in the tray to a thickness of 5 mm. The temperature of the input air was 21°C. The tray containing the ore was heated in the oven at temperature of 60°C for 7 days. The temperature of 60°C was chosen for the oven because it was found experimentally that at this temperature, oil sands could be effectively weathered inside the oven for a period of 7 days. The oil sands sample inside the oven was dehydrated because the inlet air was not water saturated.

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[3] virtually all the formation water content was removed from the ore

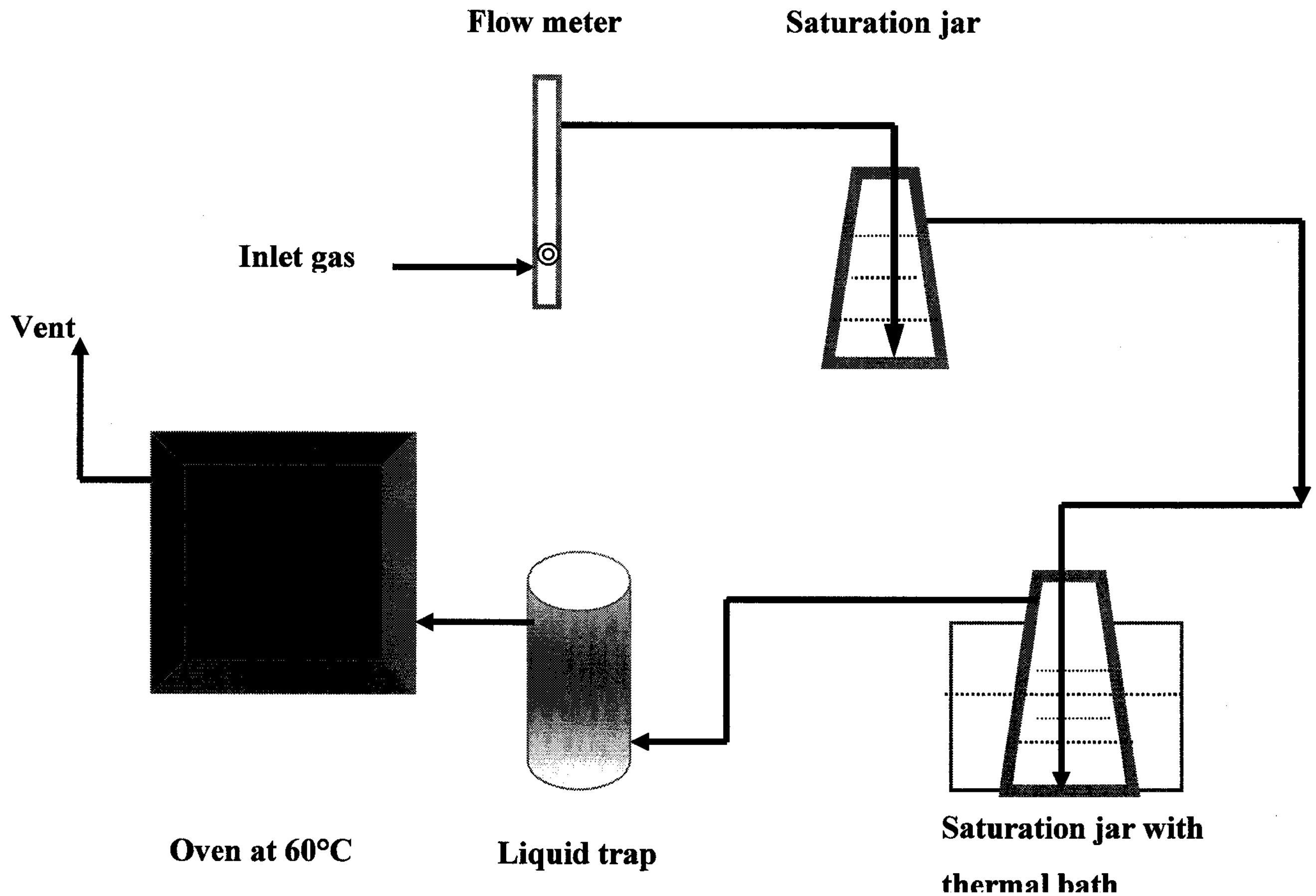


Figure 3-1: Schematic diagram of oil sand ore treatment

Weathering using air (negligible formation water loss)

About 350g of the good processing ore was placed in an aluminum tray. The ore was well spread in the tray to a thickness of 5 mm. The temperature of the oven was set at 60°C. Two bottles filled with de-ionized water were used for water saturation of the input air. One bottle was held at room temperature while the other bottle was placed inside a thermal bath at a given bath temperature. The temperature of the input air was controlled at about 68°C in order to minimize formation water loss. Water content in the input air at 68°C is higher than that of the air inside the oven at 60°C thereby ensuring that the air was saturated in the oven. A sparger was used to generate small bubbles which enhanced water saturation efficiency. Two trays filled with de-ionized water were placed inside the oven to ensure negligible water loss from the ore that was being treated. When the oven temperature reached 60°C and the bath temperature reached the desired temperature, the system was kept running for about 1 hour to attain steady state. Once steady state temperatures were reached in the oven and water bath, the aluminum tray containing oil sand ores to be treated was placed and kept in the oven for 7 days.

Weathering using nitrogen (complete formation water loss)

For nitrogen treatment, it is very important to ensure air removal from the oven and maintain an inert atmosphere inside the oven throughout the entire period of the treatment. Approximately 350g of the good processing ore was placed in an aluminum tray with a layer of 5 mm thick. The tray containing the ore was inserted into the oven. The oven was evacuated over a short period of time. The oven was then continuously flushed with nitrogen at a large flow rate so that the oven pressure quickly reached atmospheric pressure. After this stage, the oven was flushed with dried nitrogen gas at a flow rate of about 20 mL/min for 4 hours at room temperature. The procedure (that is, evacuation and flashing) was done for a few hours. The sample was then heated to 60°C for 7 days under continuous nitrogen gas flow. The ore sample was then allowed to cool to room temperature under nitrogen environment. The cooled ore was immediately taken out for flotation experiment.

Weathering using nitrogen (negligible formation water loss)

About 350g of the good processing oil sand ore was placed into a flask. The flask was flushed with nitrogen and then covered. The oil sands inside the flask were then placed into the oven at room temperature. In order to avoid breakage of the flask, the flask was tied onto the oven shelf. The oven was evacuated over a short period of time and then continuously flushed with nitrogen at a large flow rate so that the oven pressure quickly reached atmospheric pressure. After this stage, the oven was flushed with dried nitrogen gas at a flow rate of about 20 mL/min for 4 hours at room temperature. The pressure inside the oven was kept slightly higher than atmospheric pressure. The nitrogen gas was connected to the saturation bottle and the stopper was removed from the flask containing the oil sands sample. The sample was then heated to 60°C for 7 days under continuous nitrogen gas flow.

3.2.10 Soluble cation concentrations for oil sand ores

To determine soluble cation concentrations for oil sands sample, about 45g of de-ionized water was heated to a temperature of 100°C. The heated de-ionized water was added to about 50g of oil sands sample in a glass bottle. The slurry in the glass bottle was mixed using a stirrer and then allowed to settle. A small quantity of the water on the top of the ore was collected in a centrifuge tube. The water collected in the centrifuge tube was subjected to centrifugation at 25000g force for 30 minutes with the aim to remove fine solids. The slurrying water was then analyzed using the Atomic Absorption Spectroscopy.

3.2.11 Size distribution of solids

In order to evaluate the particle size distribution of the solids, solids have to be extracted. In this study, solid particles are extracted from oil sands using the procedures described in Section 3.2.6. A Mastersizer 2000 is used to measure the particle size distribution of the solids. In Mastersizer 2000, an optical device captures the actual scattering patterns from a field of particles. Using the Mie theory, the size of the particles that create the scattering patterns can be calculated. About 15g of the solids sample was taken and placed in a beaker. Added to the beaker was 100 ml of de-ionized water. A few drops of 10 wt% sodium silicate were added to the beaker to achieve a well dispersed solution. Sonication method was used to break the aggregates of solids. The solids suspension inside the beaker was carefully stirred using a stirrer. After the sample had been well dispersed, the particle size distribution of the sample was determined by introducing a given volume of the suspension into the Mastersizer sample holder.

3.3 Characterization of ore samples and plant water

3.3.1 Characterization of Syncrude good processing and Suncor weathered ores

Table 3-1 shows the composition of the Syncrude good processing ore and Suncor weathered ore. The detailed procedure for determining the composition of oil sand ores was described in Section 3.2.2. Table 3-2 shows the soluble cation concentrations of the Syncrude good processing ore and Suncor weathered ore. From the results shown in Table 3-1, it can be noted that the Suncor weathered ore has lower fines and formation water content as compared to the Syncrude good processing ore. Table 3-2 shows that the Syncrude good processing ore has lower divalent ion concentrations than the Suncor weathered ore.

Table 3-1: Composition of the Syncrude good processing ore and Suncor weathered ore.

Constituent	Composition[4] (wt%)	
	Syncrude good processing ore	*Suncor weathered ore*[5]
Bitumen	14.5	14.9
Formation water	4.0	0.3
Mineral solids	81.1	84.5
Fines (<44 μm)	10.0	5.6

Note: Fines are reported as weight percent of mineral solids while bitumen, water and mineral solids contents are reported as weight percent of the entire oil sands.

Table 3-2: Soluble cation concentrations of the Syncrude good processing ore and Suncor weathered ore.

Ion	Concentration (mg/kg)	
	Syncrude good processing ore	*Suncor weathered ore*
Ca	2.3	6.2
Mg	0.2	1.8
Na	63.0	2.5
K	14	1.7

[4] average value of 5 repeated experimental runs.

[5] Suncor weathered ore refers to the weathered ore obtained from Suncor Energy, Inc

3.3.2 Characterization of Aurora recycle process water

The pH of the Aurora recycle process water is approximately 8.5. Table 3-3 shows the soluble ion concentrations for the Aurora recycle process water used.

Table 3.3: Soluble ion concentrations of Aurora recycle process water

Ion	*Concentration (mg/L)*
Ca	41.1
Mg	19.2
Na	527.3
K	16.2
SO_4	76.6
HCO_3	649.0

Chapter 4

Processability of weathered ores[6]

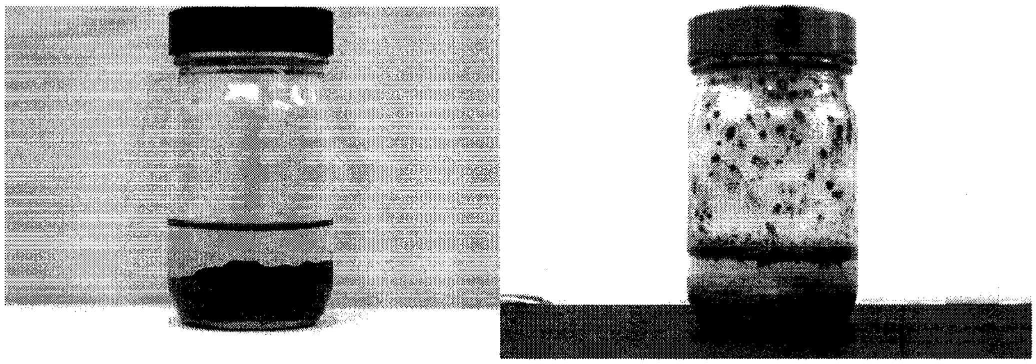
[6] data of the figures in chapter 4 are given in Appendix A

4.1 Visualization tests on the processability of Suncor weathered ores using shaking jar test

Bitumen extraction tests using a water bath shaker were a preliminary test conducted on the Suncor weathered oil sand ore to investigate its processability by visualization. Process parameters (such as extraction temperature and pH of the water used to make ore slurry) and addition of kerosene to the oil sands were investigated. The purpose of the tests was to obtain a preliminary understanding of the processability of the Suncor weathered oil sand ore.

4.1.1 Photographs showing the effect of temperature on processability of ores

From Figure 4-1(a), it can be seen by visual inspection that at room temperature, there was no liberation of bitumen from the sand grains hence, no bitumen could be recovered from the ore at this low extraction temperature. In Figure 4-1(b), an appreciable liberation of bitumen from sand grains was observed. It can be seen that because of the good liberation of bitumen from sand grains and bitumen aeration, an increase in bitumen recovery is anticipated. The liberated and aerated bitumen floated to the top of the water and also sticked to the side of the bottle. The same Aurora recycle process water with pH of 8.5 was used for both scenarios and the pH of the slurry was 8.3. The only conclusion is that extraction at temperature of 50°C is better than extraction at room temperature. It is evident that by increasing the extraction temperature from room temperature to 50°C, bitumen recovery can be improved.

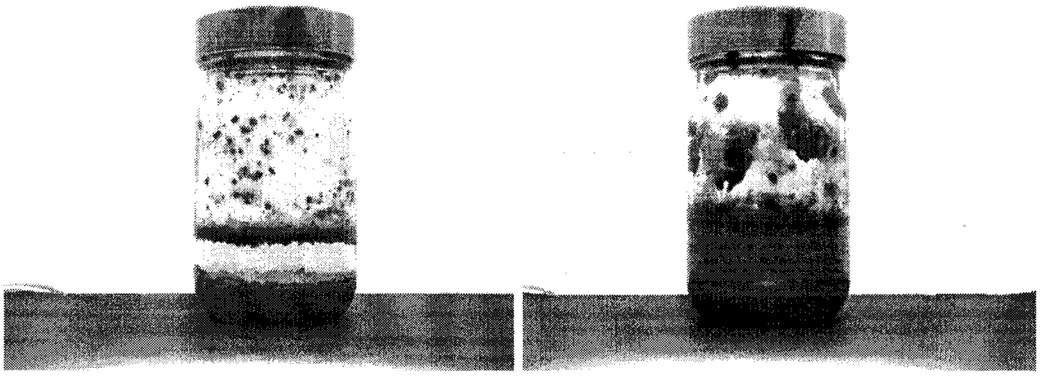


(a) Room temperature, pH of 8.3 **(b) Temperature of 50°C, pH of 8.3**

Figure 4-1: Photographs showing the effect of temperature on processability of Suncor weathered ore using Aurora recyle process water at extraction pH of 8.3.

4.1.2 Photographs showing the effect of pH on processability of ores

The same extraction temperature of 50°C was employed in the recovery experiments but the initial pH of the water was modified by the addition of sodium hydroxide solution. Figure 4-2 (a) shows that the solids appeared very clean at pH 9.2 indicating significant liberation of bitumen from sand grains. Figure 4-2 (b) shows that at pH 10.5, there was also good liberation of bitumen from sand grains but there was evidence of the formation of emulsification which would make the recovery of the bitumen difficult (Malmberg et al., 1968). It is well established that higher pH leads to very low interfacial tension between bitumen and water, resulting in bitumen emulsification.

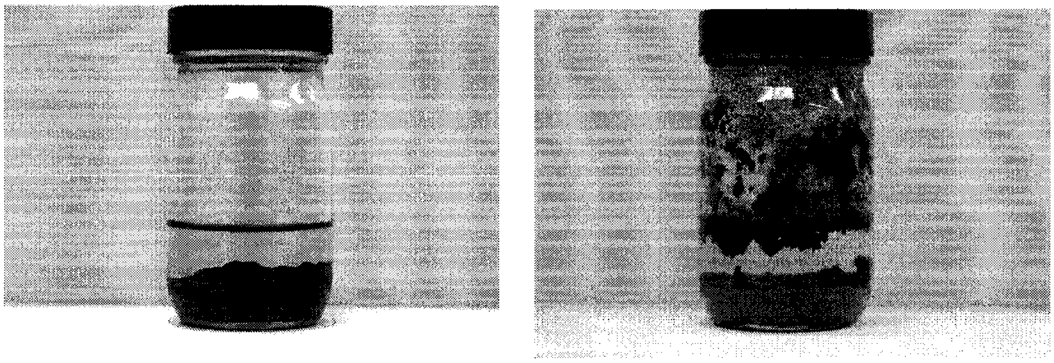


(a) pH of 9.2, Temperature of 50°C (b) pH of 10.5, Temperature of 50°C

Figure 4-2: Photographs showing the effect of pH on processability of Suncor weathered ore using Aurora recycle process water at temperature of 50°C for two different initial water pH.

4.1.3 Photographs showing the effect of kerosene on processability of ore

With no addition of kerosene to the oil sands before extraction at room temperature, there appeared to be no bitumen liberation from sand grains and little or no recovery of bitumen from the weathered oil sand ore as shown in Figure 4-3 (a). With the addition of kerosene (0.5g of kerosene/20g of weathered ore) prior to the mechanical agitation of the ore slurry in a temperature controlled shaker, there was a significant degree of bitumen liberation from the sand grains, anticipating highly effective bitumen recovery. It can be said that addition of kerosene helped to reduce the bitumen viscosity which is another good reason for the enhanced bitumen recovery from oil sand ores (Drelich et al., 1995).



(a) No kerosene addition, pH of 8.3 **(b) 0.5g kerosene/20g ore, pH of 8.3**

Figure 4-3: Photographs showing the effect of kerosene addition on processability of Suncor weathered ore using Aurora recycle process water at pH of 8.3.

4.2 Effect of flotation temperature on processability of Suncor weathered ore using Denver flotation cell

In an attempt to ascertain the problems encountered with the processing of weathered ores, the effect of temperature on processability of Suncor weathered ore was investigated. In this set of experiments, flotation temperatures of 35°C, 45°C and 75°C were employed to extract bitumen from the ore. The test was performed in a Denver flotation cell using 200g of Suncor weathered ore and 900 mL of Aurora recycle process water. The pH of the slurrying water was 8.5 and the tailings pH ranged from 8.3 to 8.4. Each data point on the plots is an average value of 5 repeated experimental runs. Figure 4-4 shows bitumen recovery as a function of flotation time at different flotation temperatures for Suncor weathered ore.

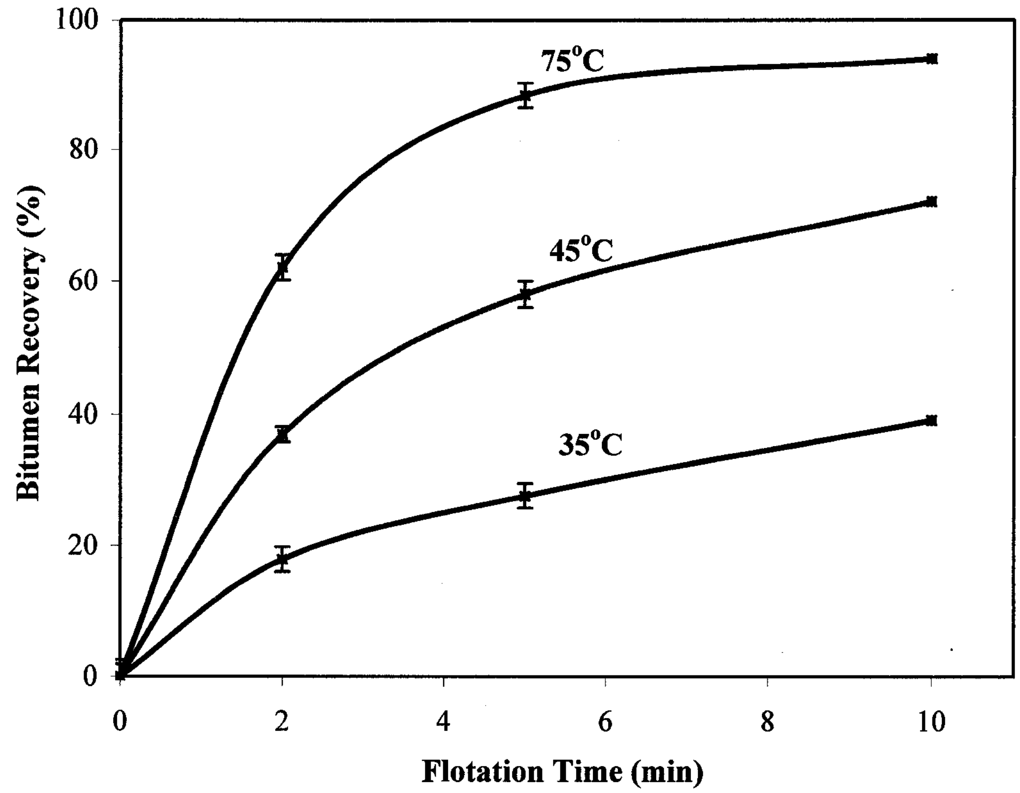


Figure 4-4: Bitumen recovery as a function of flotation time at different flotation temperatures for Suncor weathered ore using Aurora recycle process water. Extraction pH range of 8.3 to 8.4.

From the flotation kinetic results shown in Figure 4-4, the cumulative bitumen recovery after 10 minutes of flotation at 35°C is 39%. This recovery is very low and may be partly due to the low formation water content of the Suncor weathered ore. The cumulative bitumen recovery increased at higher flotation temperatures. The cumulative recovery was increased from 39% at flotation temperature of 35°C to 72% at flotation temperature of 45°C. A further increase in the cumulative recovery of bitumen from 72% at flotation temperature of 45°C to

94% at flotation temperature of 75°C was obtained. The result showed that processing temperature played a large role in the recovery of bitumen from the Suncor weathered oil sand. At the lower temperature of 35°C, the Suncor weathered ore exhibited very low bitumen recovery with little froth layer on the surface of the slurry in the flotation cell. At increased flotation temperatures of 45°C and 75°C, the bitumen recovery improved significantly with a very thick froth layer formed on the surface of the slurry in the flotation cell.

The results implied that significantly high bitumen recovery can be achieved from Suncor weathered oil sand ores by increasing the flotation temperature in the oil sand extraction process. The increase in the bitumen recovery at higher processing temperature may be attributed to the reduced bitumen viscosity and any other factors that may be influenced by temperature.

Figure 4-5 gave the results of the effect of temperature on bitumen to solids ratio of the froth obtained with Suncor weathered ore. From the results shown in Figure 4-5, it can be noted that the bitumen froth obtained at lower flotation temperature contained more solids than the froth obtained at higher flotation temperature. At the lower temperature of 35°C, the Suncor weathered ore exhibited very poor froth quality in terms of the bitumen to solids ratio. At this low temperature, the bitumen to solids ratio after 10 minutes of froth collection was only 0.16. The froth collected at this low temperature was very loosely packed and foamy. The froth looks like "a solid matrix". At increased flotation temperatures of 45°C and 75°C, the bitumen to solids ratio improved and the froth collected during the flotation experiment was less loosely packed. The bitumen to solids ratio after 10 minutes of froth collection at temperatures of 45°C and 75°C were 0.24 and 0.38, respectively.

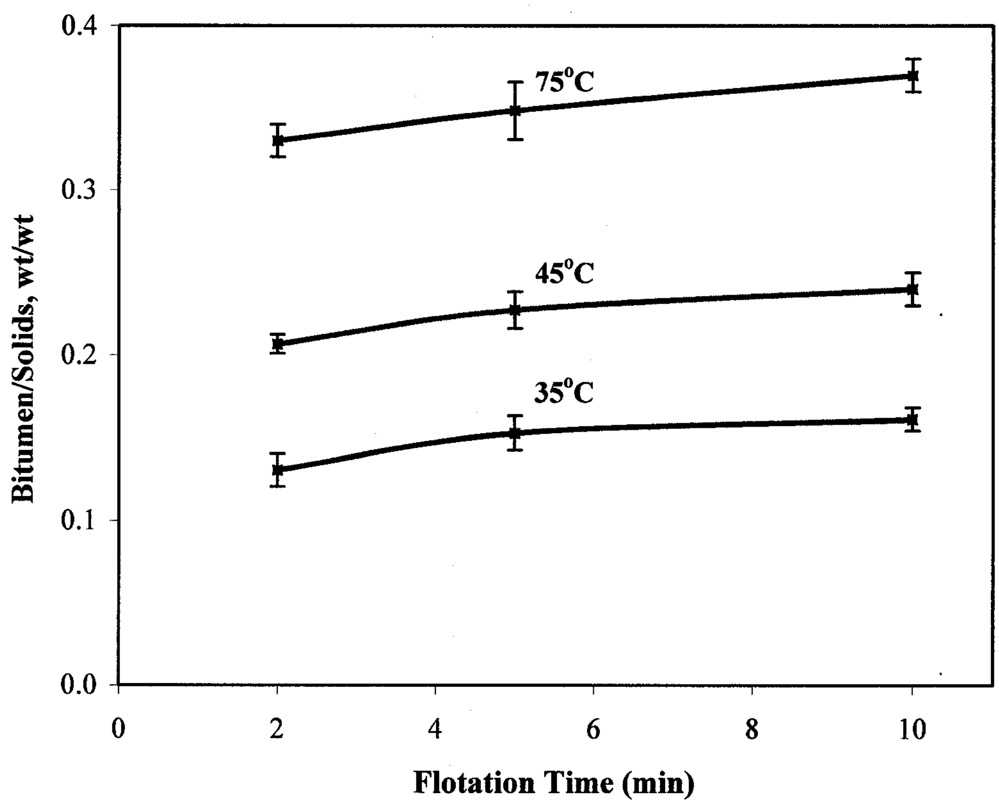


Figure 4-5: Bitumen/solids ratio as a function of flotation time at different flotation temperatures for Suncor weathered ore using Aurora recycle process water. Extraction pH ranges from 8.3 to 8.4.

Figure 4-6 shows the effect of flotation temperature on the bitumen to water ratio in the collected froth from the Suncor weathered ore.

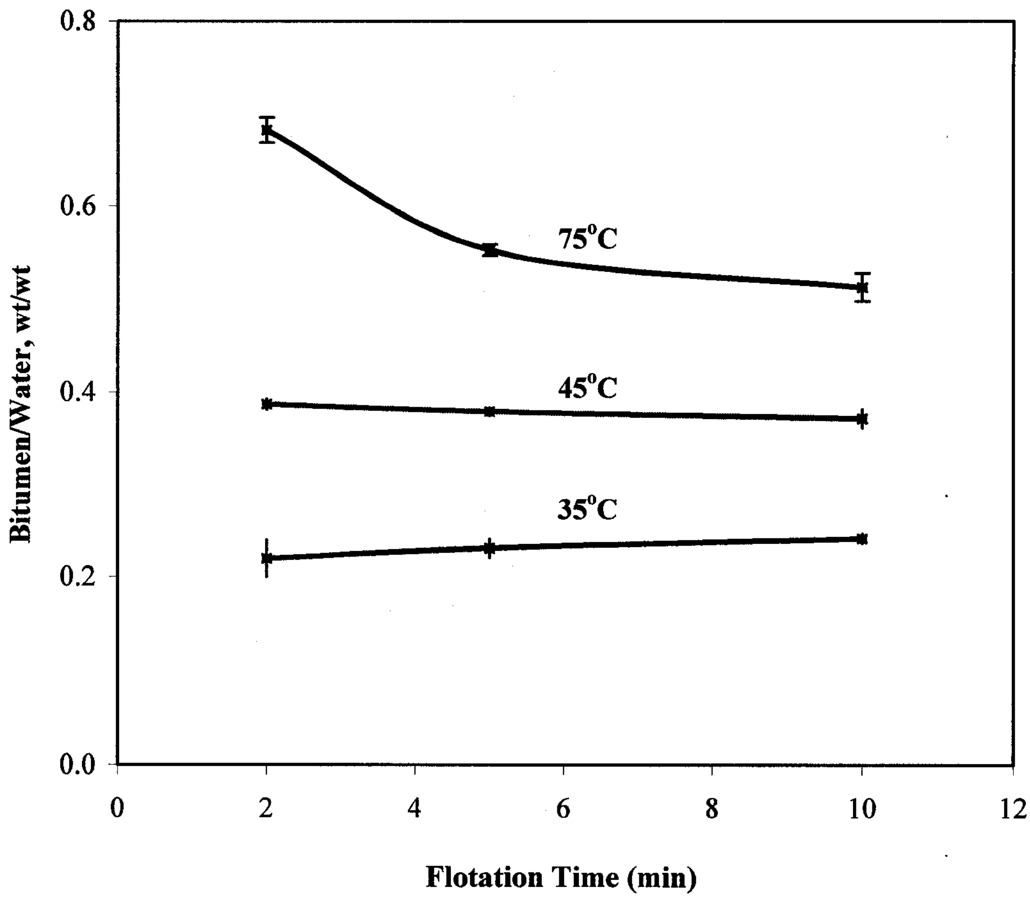


Figure 4-6: Bitumen/water ratio as a function of flotation time at different flotation temperatures for Suncor weathered ore using Aurora recycle process water. Extraction pH ranges from 8.3 to 8.4.

From this figure, one can see that at a lower temperature, more water was trapped in the froth. At the lower temperature of 35°C, the Suncor weathered ore exhibited extremely poor froth quality in terms of bitumen to water ratio. The bitumen to water ratio after 10 minutes of froth collection gradually increased from 0.24 at flotation temperature of 35°C to 0.37 at 45°C and finally to 0.51 at flotation temperature of 75°C. It can be seen from Figure 4-6 that at the flotation temperature of 75°C, the bitumen to water ratio decreases from 0.68 after 2 minutes of froth collection to 0.51 after 10 minutes of froth collection but for the other two flotation temperatures, the bitumen to water ratio remained fairly constant throughout the 10 minutes of froth collection.

The results shown in Figure 4-7 gave the amount (in grams per 100 grams of froth) of the various components contained in the froth obtained from the three different extraction temperatures of 35°C, 45°C and 75°C after a collection time of 10 minutes. The results show more bitumen content in the froth as the temperature increases, while the reverse was the case for solids and water contents in the froth. This observation confirmed improved processability at higher temperature.

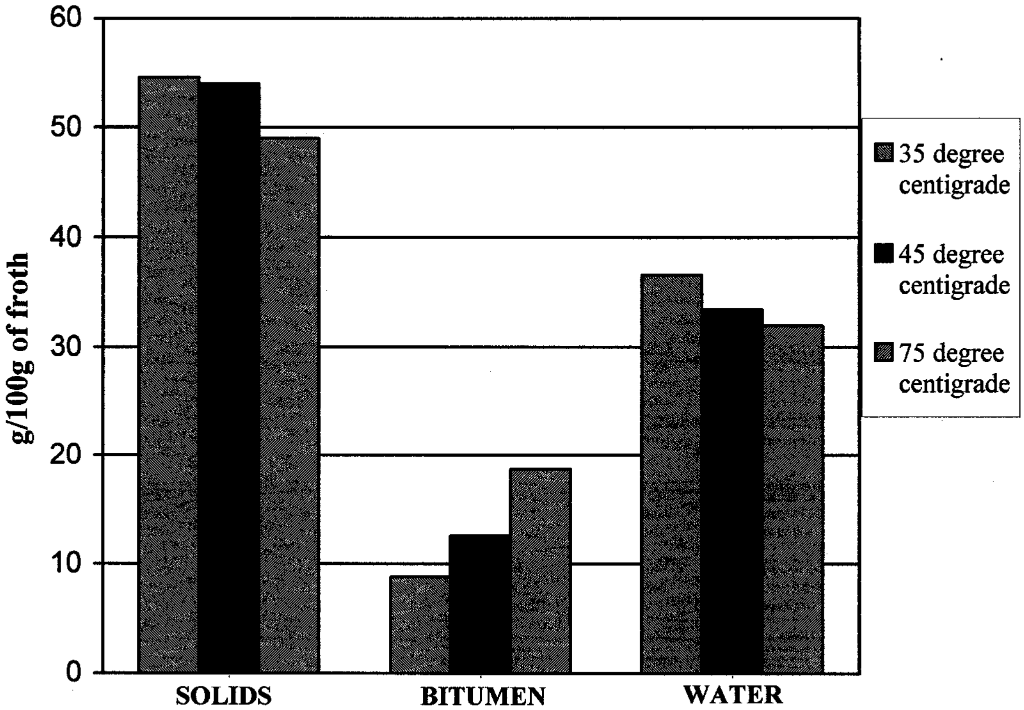
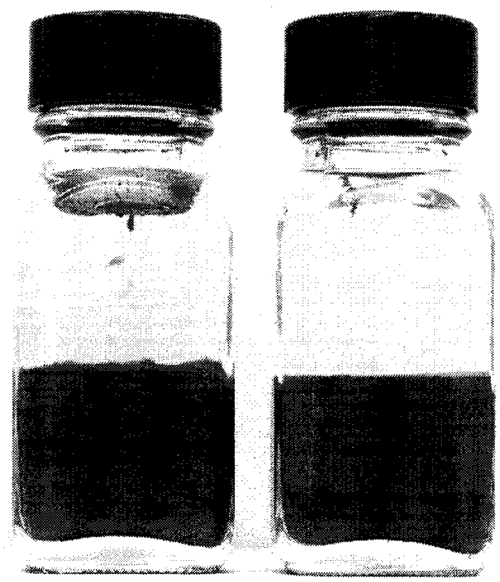


Figure 4-7: Froth analysis of Suncor weathered ore at flotation temperatures of 35°C, 45°C and 75°C using Aurora recycle process water. Extraction pH ranges from 8.3 to 8.4.

4.2.1 Photograph of the tailings at flotation temperatures of 35°C and 75°C

Figure 4-8 shows the photograph of tailings obtained from flotation experiments using Suncor weathered ores and Aurora recycle process water at flotation temperatures of 35°C and 75°C. Visual observation of photograph shown in Figure 4-8 would suggest that at a flotation temperature of 35°C, the tailings contains much bitumen and appears more like the original oil sand ore. This means that there was little liberation of bitumen from sand grains. For the higher temperature of 75°C, there was a very good liberation of bitumen from sand grains and hence, very good bitumen recovery of about 94%. At 75°C, there was no noticeable bitumen in the tailings stream as indicated by the “clean” solids.



Flotation at temperature of 35°C **Flotation at temperature of 75°C**

Figure 4-8: Photograph of tailings obtained from flotation experiment for Suncor weathered ore at flotation temperatures of 35°C and 75°C using Aurora recycle process water.

4.3 Effect of additives on Suncor weathered ore using Denver flotation cell.

4.3.1 Effect of sodium silicate on bitumen recovery and froth quality

In this section, the role of sodium silicate, Na_2SiO_3 on the extractability of Suncor weathered ores was investigated by the addition of different dosage of Na_2SiO_3 in the flotation test using a Denver flotation cell. The effect of sodium silicate as a dispersing agent and pH modifier on the processability of Suncor weathered ores using low temperature extraction processes (flotation temperature of 35°C) was studied. Sodium silicate (Na_2SiO_3) has strong dispersing ability of clay fines (Li et al., 2005). Table 4-1 and Figure 4-9 summarize the data from the flotation tests conducted on Suncor weathered ore using different dosage of sodium silicate.

As shown in Table 4-1 and Figure 4-9, without sodium silicate addition, the cumulative recovery of bitumen was 39%. The cumulative recovery of bitumen with 500 ppm sodium silicate addition increased to 40.3%. A further increase in sodium silicate addition to 2000 ppm increased bitumen recovery to 45.2%. However, the addition of 3500 ppm sodium silicate resulted in a bitumen recovery of 42%. The results show that addition of sodium silicate during the flotation slightly enhanced bitumen recovery from the Suncor weathered ore. An optimum dosage of 2000 ppm gave the highest bitumen recovery. At the higher dosage of 3500 ppm, the bitumen recovery dropped from 45.2% to 42%. This decline in bitumen recovery may be attributed to the high slurrying pH of 10 at 3500 ppm sodium silicate addition. It has been clearly shown that the hydrophobicity of bitumen decreases rapidly when the pH is about 10 and higher (Masliyah et al., 2004). With bitumen becoming less hydrophobic, there will be reduced attachment of air bubble to bitumen droplets and subsequently results in a lower bitumen recovery. The lower bitumen recovery may also be due to the emulsification of bitumen to produce bitumen droplets that are too fine to be effectively recovered.

Table 4-1: Summarized data for the flotation tests at 35°C using different dosage of sodium silicate in Aurora recycle process water.

Concentration of Na_2SiO_3 (ppm[7])	*pH of slurrying water containing Na_2SiO_3*	*pH of tailings water*	*Cumulative Recovery after 10 minutes (%)*
0	8.5	8.3	39.1
500 (0.1g/200g of ore)	9.3	8.8	40.2
2000 (0.4g/200g of ore)	9.8	9.5	45.2
3500 (0.7g/200g of ore)	10.0	9.8	42.0

[7] ppm value is on the basis of the weight of oil sands.

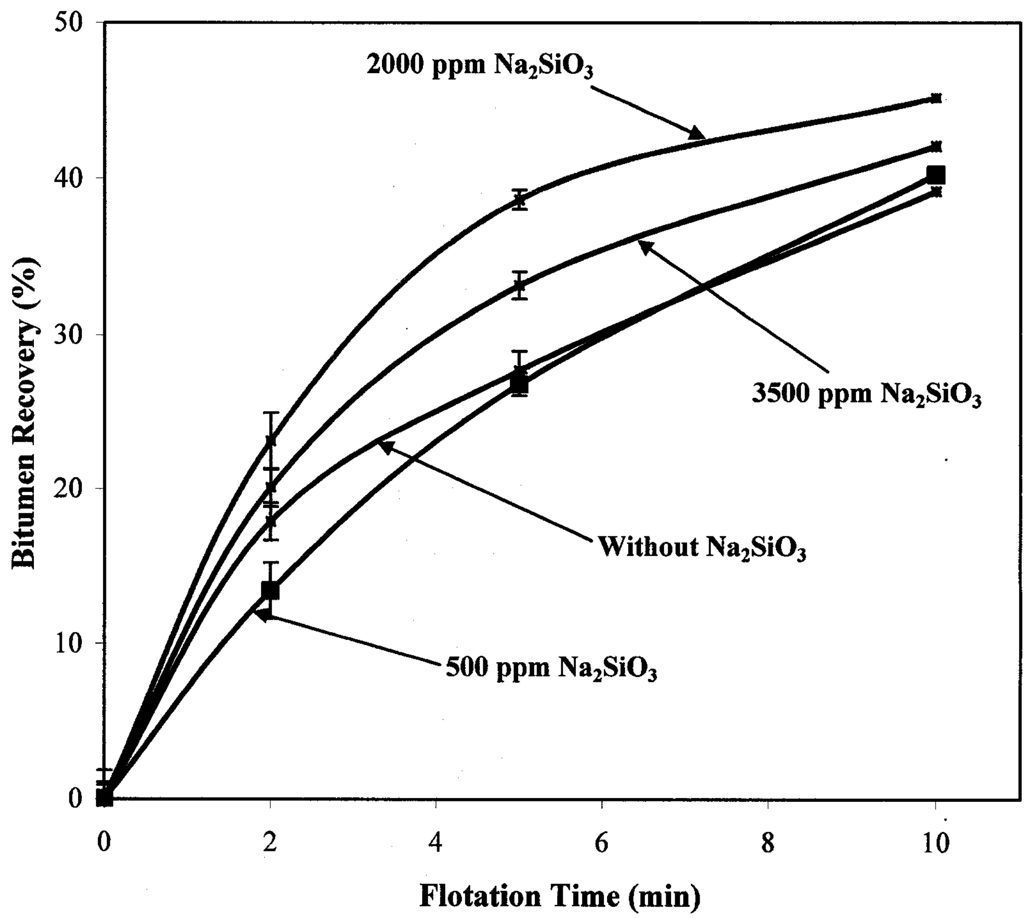


Figure 4-9: Effect of sodium silicate addition on bitumen recovery for Suncor weathered ore using Aurora recycle process water at flotation temperature of 35°C and pH 8.3 to 9.8.

Figure 4-10 shows the bitumen to solids ratio as a function of flotation time for Suncor weathered ores using different dosage of sodium silicate. As shown in this figure, there was a small increase in the bitumen to solids ratio as the dosage of sodium silicate increased. However, the difference after 10 minutes of froth collection for the different dosages of sodium silicate was not significant. The reason could be due to the low fines content of the Suncor weathered ore (which is about 5.6 wt% in mineral solids) since an important role of sodium silicate as dispersing agent is to minimize fines/bitumen heterocoagulation.

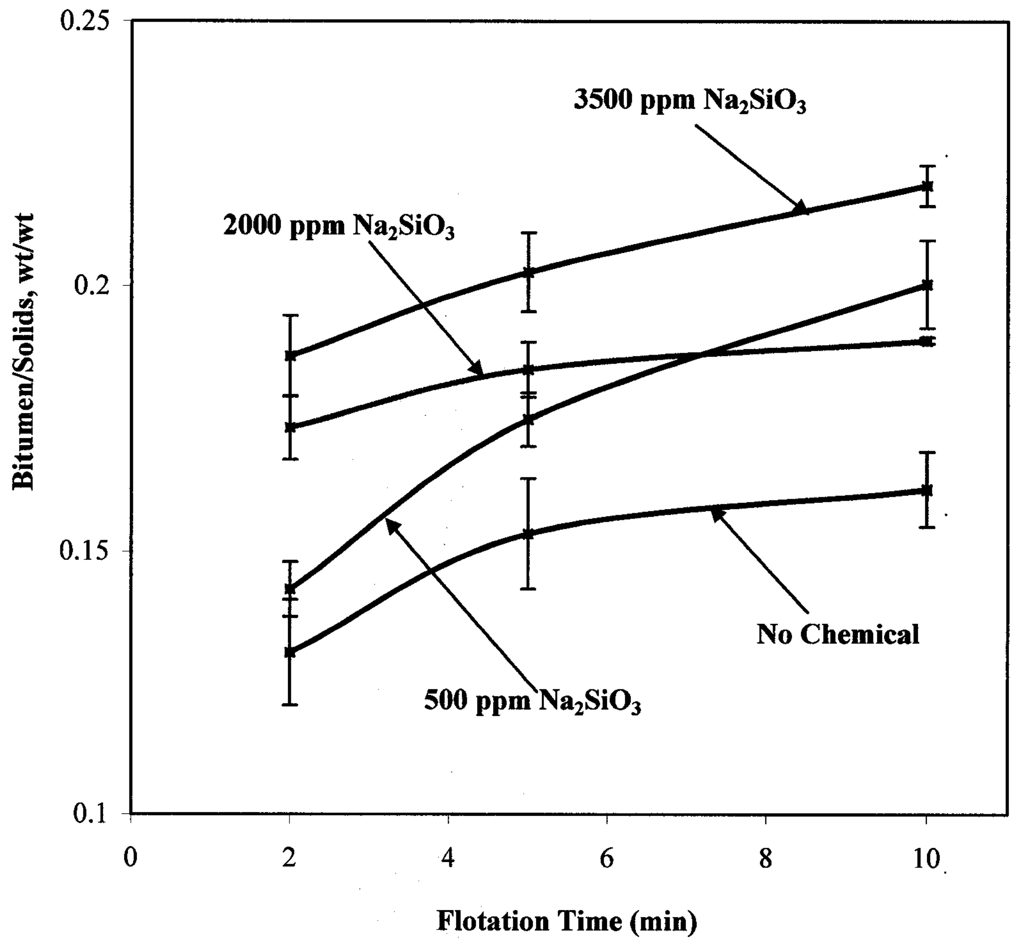


Figure 4-10: Effect of sodium silicate on bitumen/solids ratio for Suncor weathered ore using Aurora recycle process water at flotation temperature of 35°C and pH 8.3 to 9.8.

4.3.2 Effect of chemical dosage on process and tailings water pH

As shown in Figure 4-11, there is a direct relationship between sodium silicate dosage and pH of both the slurrying water and tailings water. It shows that the pH of the slurrying water was consistently higher than that of the tailings water at the same sodium silicate addition.

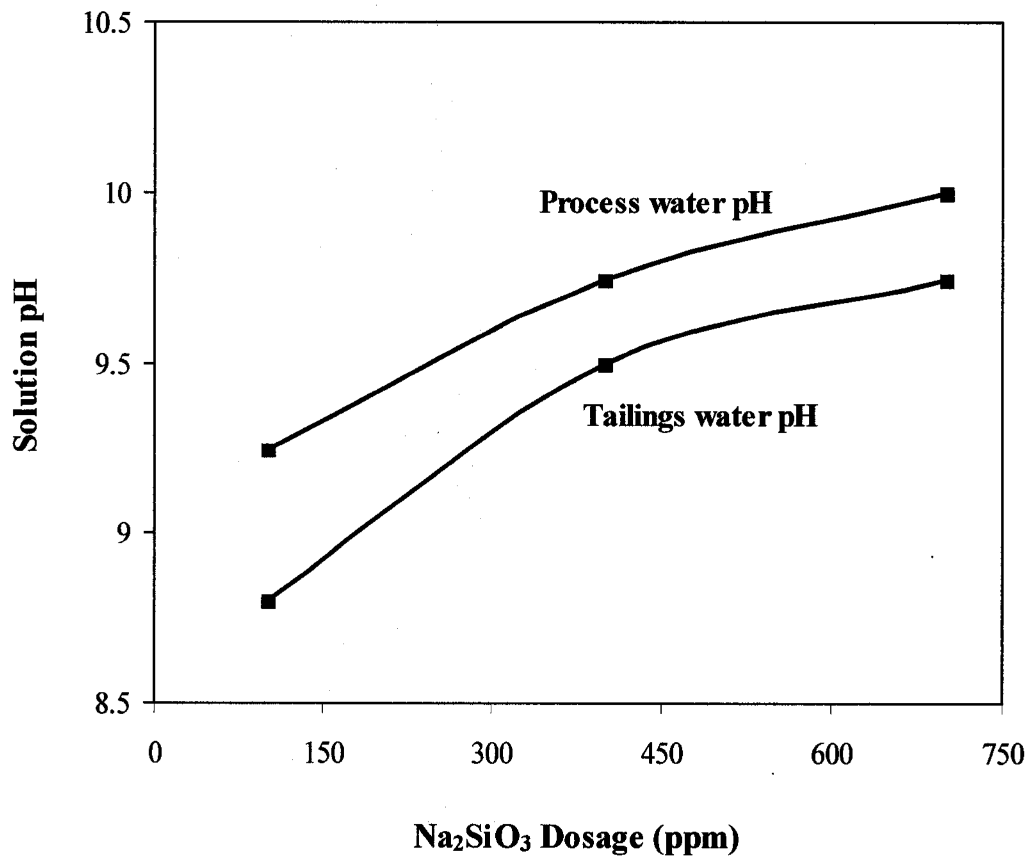


Figure 4-11: Effect of chemical (Na_2SiO_3) dosage on process and tailings water pH for Suncor weathered ore at flotation temperature of 35°C.

4.3.3 Effect of kerosene addition on bitumen recovery and froth quality

The flotation experiments were performed in a Denver cell with kerosene addition at a flotation temperature of 35°C using Aurora recycle process water. The concentration of kerosene is 2500ppm that is based on the amount of oil sand ores and it is 500 ppm based on the total slurry. Figure 4-12 shows the effect of kerosene addition on bitumen recovery from Suncor weathered ores as a function of flotation time.

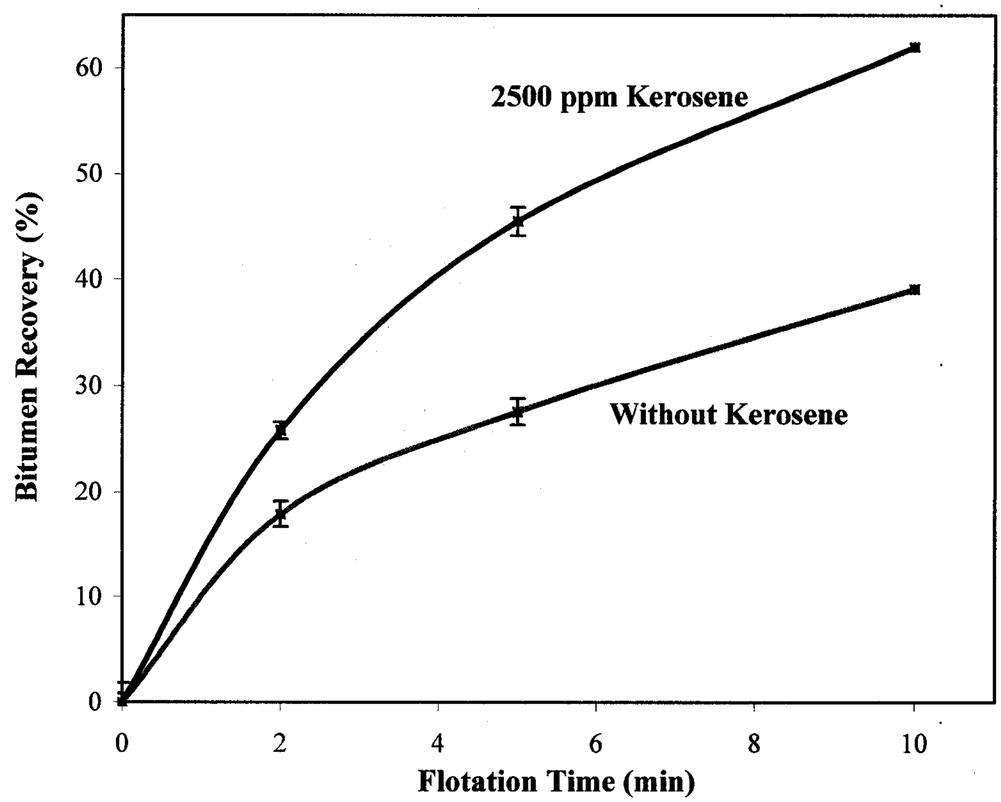


Figure 4-12: Effect of kerosene addition on bitumen recovery from Suncor weathered ores using Aurora recycle process water at a flotation temperature of 35°C and pH 8.3.

The positive effect of kerosene on bitumen extraction has again been confirmed in our experimental results shown in Figure 4-12. With the addition of 2500 ppm kerosene during the conditioning of the oil sands slurry, the bitumen recovery from the Suncor weathered ore significantly increased from 39% to 62%. The results suggest that at low temperature extraction of oil sand ores, kerosene addition is beneficial in enhancing bitumen recovery. The increase in bitumen recovery may be attributed to the reduction in bitumen viscosity achieved by kerosene addition (Drelich et al., 1995). The results in Figure 4-13 show significant increase in the bitumen to solids ratio with the addition of 2500 ppm of kerosene.

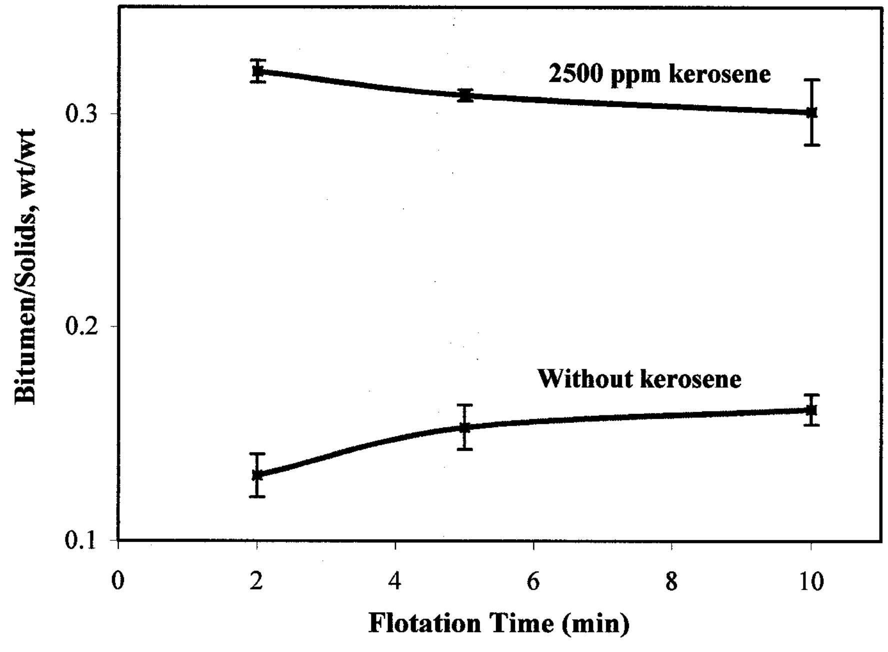


Figure 4-13: Effect of kerosene addition on froth quality obtained from Suncor weathered ore using Aurora recycle process water at a flotation temperature of 35°C and pH 8.3.

4.4 Processability of laboratory weathered oil sand ores

To understand the effect of weathering on the processability of oil sand ores, the good processing ore from Syncrude Canada Ltd was weathered in a laboratory oven under well controlled conditions prior to flotation experiments in a Denver flotation cell. The experimental procedure for the weathering of ore has been described in detail in Section 3.2.9.

4.4.1 Effect of formation water loss on processability of laboratory weathered oil sand ores

The purpose of the tests was to investigate the relationship between the processability (bitumen recovery and froth quality) and formation water loss in oven treated oil sands under air environment. The flotation experiment was carried out at 35°C. Clark in his early studies believed that formation water plays a critical role in the liberation of bitumen from sand grains. He derived such conclusion by noting that bitumen containing water does not adhere strongly to the mineral solids but dry bitumen does adhere strongly to mineral solids (Clark, 1923 and Clark, 1929). Weight loss during the artificial treatment of oil sands samples in an oven at 50°C under controlled environment has been reported to be mainly due to the evaporation of formation water that is inherent in the ore (Liu et al., 2005). Weathering study by Gu (2006) confirmed that after weathering of a good processing ore in an oven using air or nitrogen, bitumen recovery decreased as the remaining formation water content of the weathered oil sands decreased.

He ascribed the poor bitumen recovery to the formation water loss during the weathering process.

The Figure 4-14 shows a correlation between bitumen recovery and formation water loss from the laboratory weathered ore. The original ore has about 4g of formation water per 100 g of oil sand ores, which was reported in Table 3-1.

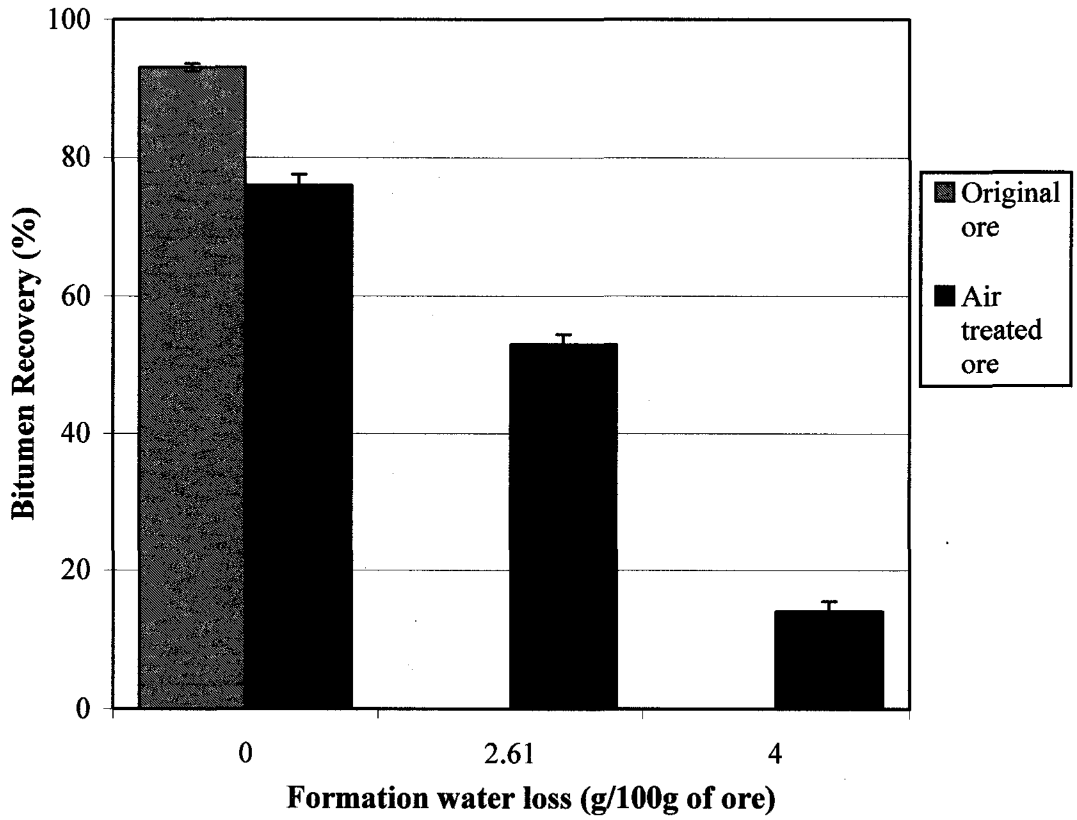


Figure 4-14: Bitumen recovery as a function of the formation water loss for air treated ores (7 days and oven temperature of 60°C) using Aurora recycle process water at flotation temperature of 35°C. The original untreated ore has 4g of formation water per 100g of ore.

The results of Figure 4-14 show that with an increase in the formation water loss due to laboratory oven treatment, the bitumen recovery of the treated ore was decreased. With about 2.61g of formation water loss, the bitumen recovery decreased from 93.0% to about 53.0%. For the ores of complete formation water loss, the bitumen recovery was the lowest at 14.0%. In comparison, the bitumen recovery from the ores treated similarly but with negligible or no formation water loss decreased from original 93.0% to about 76.0%. In this case, a thick froth layer in the flotation cell was observed.

For the case of complete formation water loss, it was observed that the tailings slurry after the flotation experiment looked like the fresh treated ore. This implied that there is really insignificant liberation of bitumen from sand grains. It is for this reason that very poor bitumen recovery was obtained. By the evaporation of formation water from ores using air treatment in the oven, the water film which is generally believed to be between the sand grains and bitumen was assumed to be eliminated. This water film plays an important role in the separation of bitumen from sand grains. The loss of the inherent water in oil sand ores would result in the direct contact between the bitumen and sand grains thereby making the liberation of the bitumen from sand grains difficult and subsequently resulting in poor processability of the laboratory weathered ore.

As for the case of negligible or no formation water loss, the bitumen recovery was only slightly lower than the bitumen recovery of the original good processing ore. This is most likely due to the preservation of the water film which helped in the liberation of bitumen from the sand grains. The bitumen froth formed was thicker as compared with the case of complete formation water loss. Some layer of un-liberated bitumen was observed in the tailings after the extraction, which implies that some bitumen did not float but rather sank with the solids into the tailings stream. This observation suggests that in addition to water loss there may be other reason(s) for the lower bitumen recovery associated with laboratory weathered oil sands. These results further confirmed previous findings reported in literature (Liu et al., 2005; Gu, 2006). Figure 4-15 shows the relationship between the bitumen to solids ratio and formation water loss for laboratory air weathered good processing ore.

Figure 4-15 clearly shows that bitumen to solids ratio of the collected froth decreased with increasing formation water loss. With about 2.61g of formation water loss, the bitumen to solids ratio decreased from 17.0 to about 3.7. For the case of complete formation water loss, the bitumen to solids ratio was reduced to 0.5. This meant that the bitumen froth collected during the flotation test was mainly solids. The recovered froth was very small in quantity and loosely packed together. With partial formation water-loss, the froth quality was not as poor as the case of complete formation water loss.

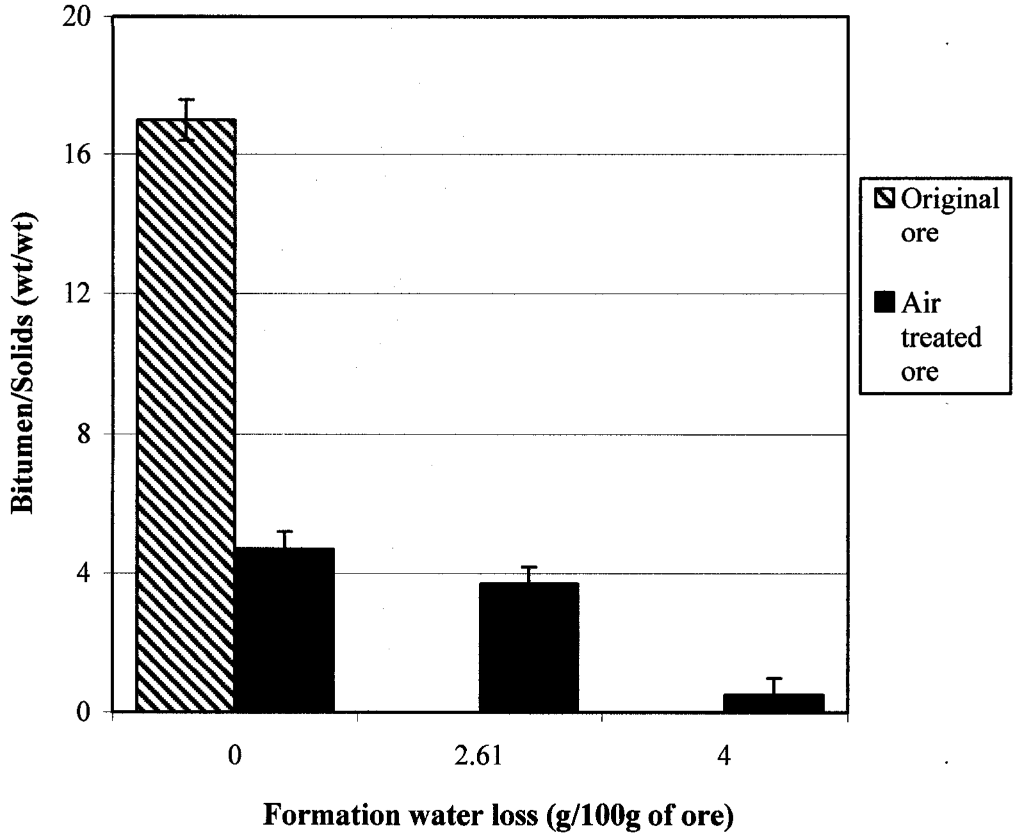


Figure 4-15: Bitumen-solids ratio as a function of the formation water loss for air treated ore (7 days and oven temperature of 60°C) using Aurora recycle water at flotation temperature of 35°C. The original untreated ore has 4g of formation water per 100g of ore.

It should be noted that with negligible or no formation water loss during the laboratory weathering process, froth quality in terms of bitumen to solids ratio of 4.73 was obtained. Better bitumen liberation from sand grains could be attributed to the enhanced bitumen to solids ratio. In this case, thicker bitumen froth was observed. The results shown in Figure 4-14 and Figure 4-15 both emphasized the adverse effect of weathering on ore processability (bitumen recovery and froth quality) as a result of formation water loss when a good processing oil sand was treated in an oven. It should be noted that bitumen to solids ratio and bitumen recovery for the case of negligible or no formation water loss are lower than the case of the original untreated ore. This is also the case of bitumen recovery.

4.4.2 Effect of weathering using nitrogen on processability of laboratory weathered ores

As shown in both Figures 4-14 and 4-15, the bitumen recovery and froth quality of laboratory weathered ore in air with negligible or no formation water loss are lower than that of the original untreated ore. This poor processability made it clear that there is at least one other factor that is responsible for the lower processability of laboratory weathered ores apart from formation water loss. This led us to investigate whether ore oxidation is a contributing effect on the processability of weathered ores. To study the effect of oxidation on laboratory treated ore, the good processing ore was treated in the oven under nitrogen environment. The flotation test was performed in the Denver flotation cell at temperature of 35°C. The results for the bitumen recovery and froth quality are shown in Figure 4-16 and Figure 4-17, respectively. From the results, it is clear that nitrogen-treated good processing ores showed the same trend of ore processability as air-treated good processing ore, but to a less extent.

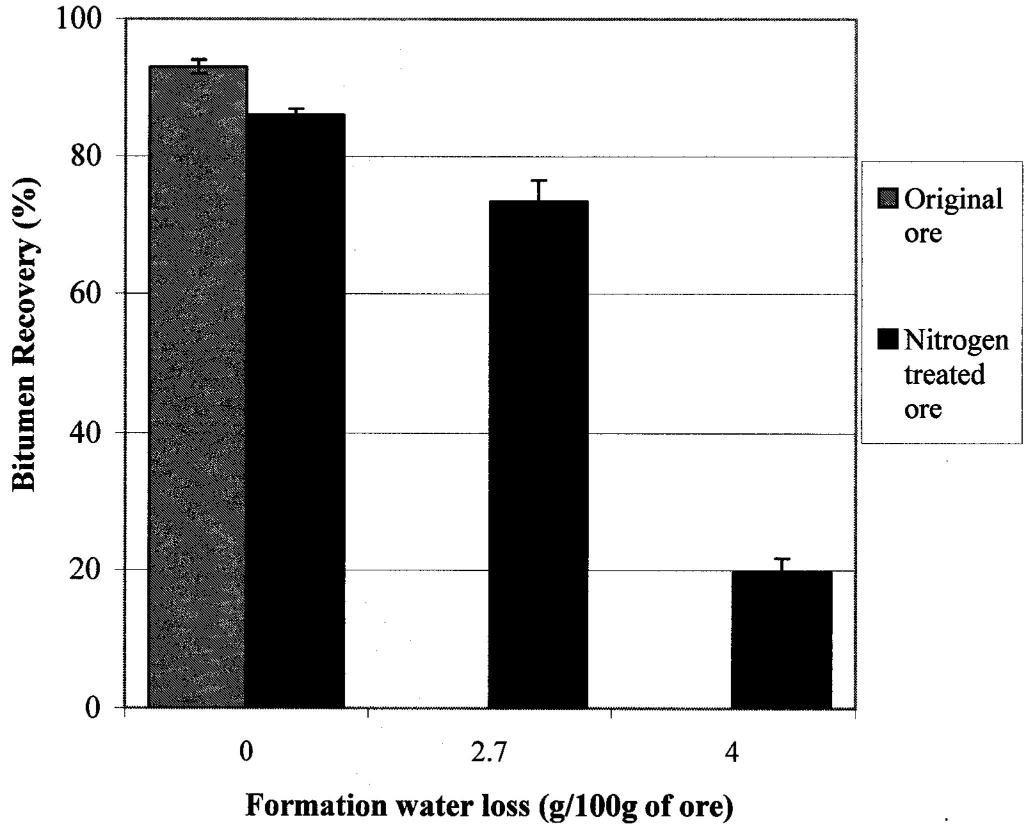


Figure 4-16: Bitumen recovery as a function of the formation water loss for nitrogen treated ore (7 days in oven temperature of 60°C) using Aurora recycle process water at a flotation temperature of 35°C. The untreated original ore has 4g of formation water per 100g of ore.

As shown in Figure 4-16, the bitumen recovery for the nitrogen treated ore with complete formation water loss was 19.7%, indicating a very poor processability. The tailings looked dark, just like an ore without any visible bitumen liberation from the sand grains. The bitumen recovery was found to be less affected with lower formation water loss during laboratory treatment. A maximum bitumen recovery of 86.0% was obtained with negligible or no formation water loss during the nitrogen treatment in an oven. In this case, a significant liberation of bitumen from sand grains was observed which is one reason for a high bitumen recovery.

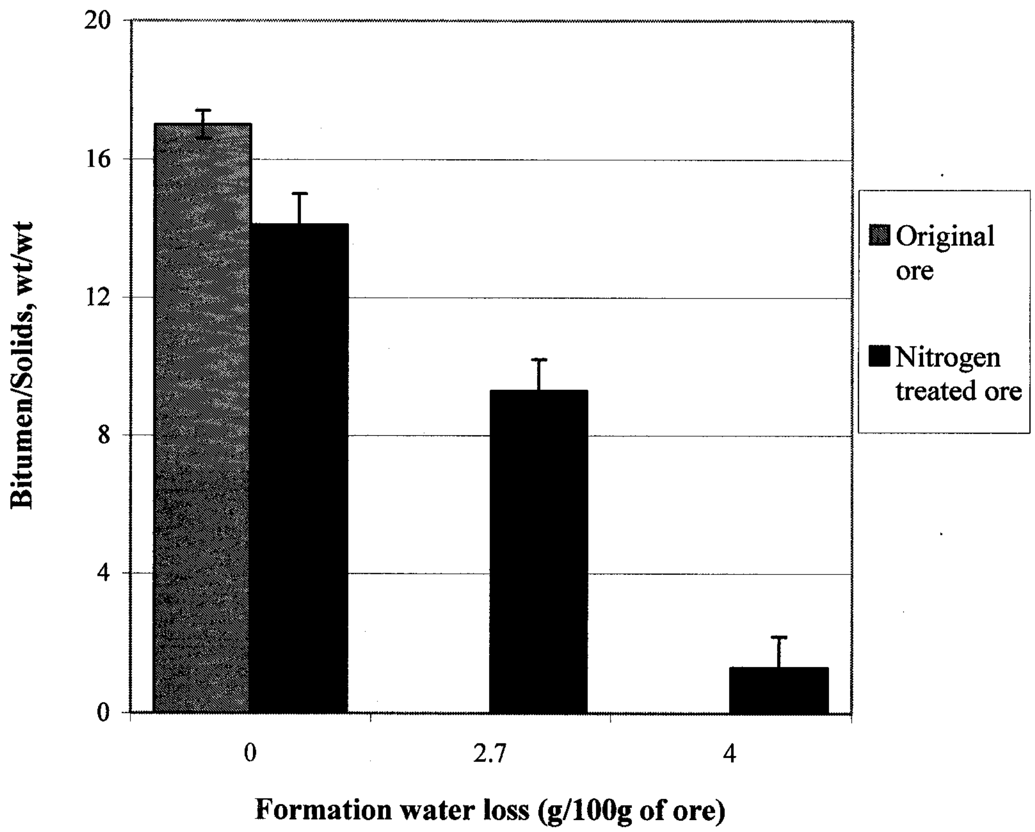


Figure 4-17: Bitumen-solids ratio as a function of the formation water loss for nitrogen treated ore (7 days in oven temperature of 60°C) using Aurora recycle process water at flotation temperature of 35°C. The untreated original ore has 4g of formation water per 100g of ore.

The results in Figure 4-17 show that bitumen to solids ratio for the nitrogen treated ore with complete formation water loss (that is, loss of the total 4g of formation water) was 1.31 in contrast to 9.3 for the case of partial formation water loss (about 2.7g). The bitumen to solids ratio was about 14.1 when the ore was nitrogen treated with negligible or no formation water. It is clear from the above results that there is a strong and consistent correlation between formation water loss and ore processability. As the formation water loss increases, the processability (both bitumen recovery and froth quality) deteriorates.

In order to appreciate the effect of oxidation on processability of laboratory weathered ores, a comparison between bitumen recovery obtained with air treatment and nitrogen treatment is shown in Figure 4-18. A comparison between the bitumen to solids ratio obtained with air treatment and nitrogen treatment is shown in Figure 4-19.

Figure 4-18 shows a bitumen recovery as a function of formation water loss for air and nitrogen treated ores. For a given formation water loss, air treatment results in a more severe detrimental effect on bitumen recovery than nitrogen treatment. As mentioned earlier, in the case of negligible or no formation water loss, there was a more significant decrease in bitumen recovery with air treatment than with nitrogen treatment. The observed difference in bitumen recovery is attributed to ore oxidation. With air treatment, oxidation can occur while with nitrogen, oxidation could be considered minimal. Therefore, oxidation appears to be an important factor that affects bitumen recovery from weathered ores. This finding is in agreement and consistent with previous study of Schramm et al. (1984), Schramm and Smith (1985), Wallace et al. (1989), Gu (2006) and Liu et al. (2005).

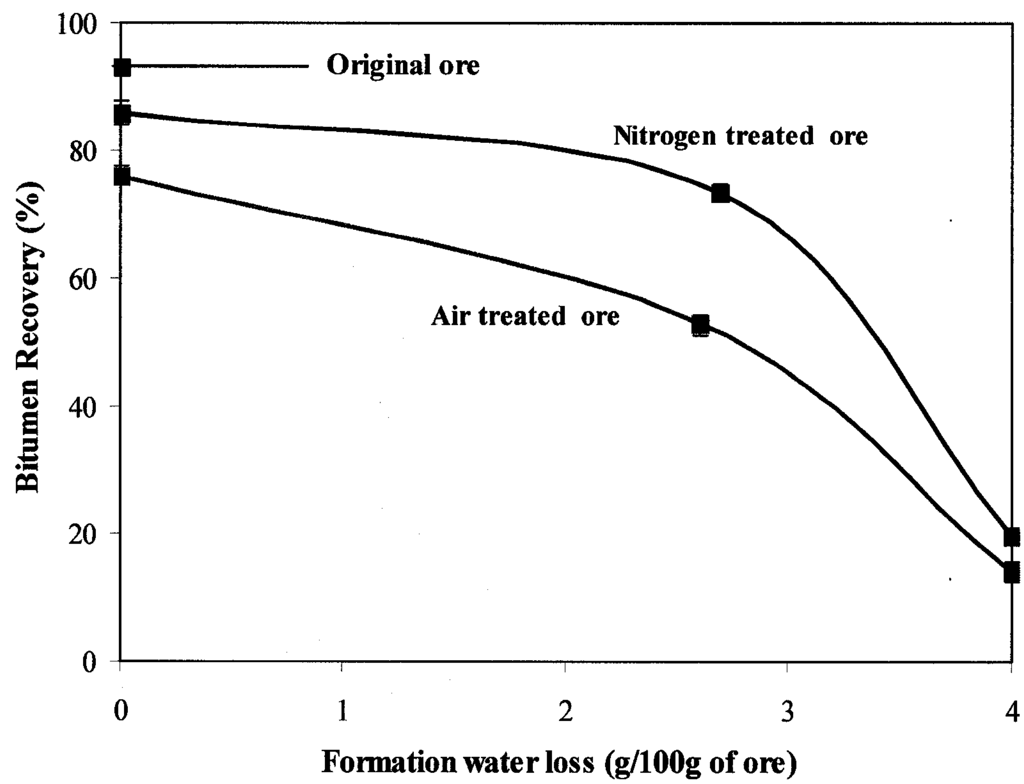


Figure 4-18: Comparison between bitumen recoveries of air and nitrogen treated ores (7 days in oven temperature of 60°C) as a function of formation water loss using Aurora recycle process water at flotation temperature of 35°C. The untreated original ore has 4g of formation water per 100g of ore.

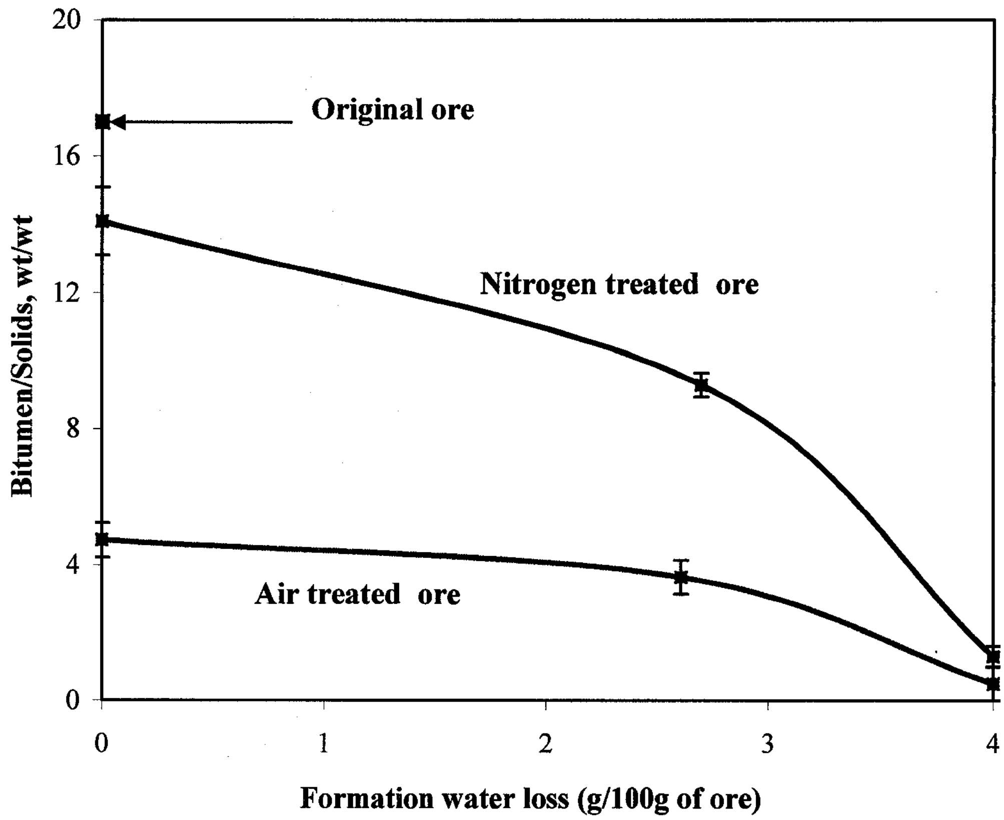


Figure 4-19: Comparison of bitumen to solids ratios for air and nitrogen treated ores (7 days in oven temp. of 60°C) as a function of formation water loss using Aurora recycle process water at a flotation temperature of 35°C. The untreated original ore has 4g of formation water per 100g of ore.

The results in Figure 4-19 shows that with no formation water loss, there was a large difference in the froth quality obtained with air and nitrogen treatment. For the nitrogen treated ore, the bitumen to solid ratio was 14.1 in contrast to 4.7 for the air treatment. This difference confirms the adverse effect of ore oxidation on processability of weathered ores. For the complete formation water loss, the difference in bitumen to solids ratio between the air and nitrogen treatments was not significant but the two treatments (that is, air and nitrogen) under this condition showed very poor froth quality. This finding emphasizes important role of formation water on bitumen recovery from oil sands using water -based extraction processes.

4.4.3 Effect of flotation temperature on bitumen recovery from laboratory weathered ores

Earlier in this thesis, it has been confirmed in Figure 4-4 that increased flotation temperature during the extraction of Suncor weathered oil sands led to a significant increase in the bitumen recovery from 39% at flotation temperature of 35°C to about 94% at flotation temperature of 75°C. In order to improve bitumen recovery of laboratory weathered ores and to further ascertain the significant positive effect of higher flotation temperature on the processability of weathered or aged ores, the flotation temperature of the laboratory air and nitrogen weathered ores was increased from 35°C to 75°C. Figures 4-20a and 4-20b show the bitumen recovery from air treated ores at flotation temperatures of 35°C and 75°C, respectively while Figures 4-21a and 4-21b show the bitumen recovery from nitrogen treated ores at flotation temperatures of 35°C and 75°C, respectively. Both nitrogen and air treatments were performed in the oven operated at temperature of 60°C for 7 days.

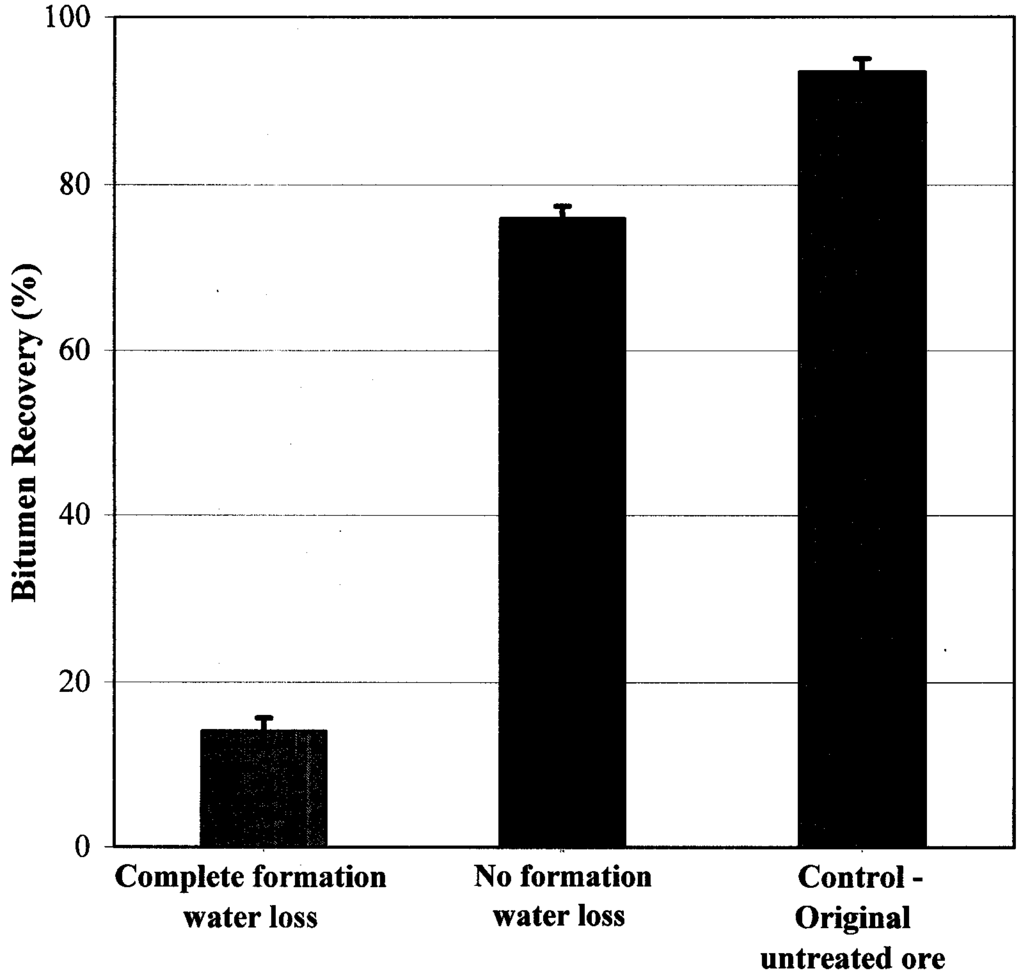


Figure 4-20a: Bitumen recovery for air treated ores (7 days in oven temperature of 60°C) using Aurora recycle process water at flotation temperature of 35°C and pH 8.1 to 8.2.

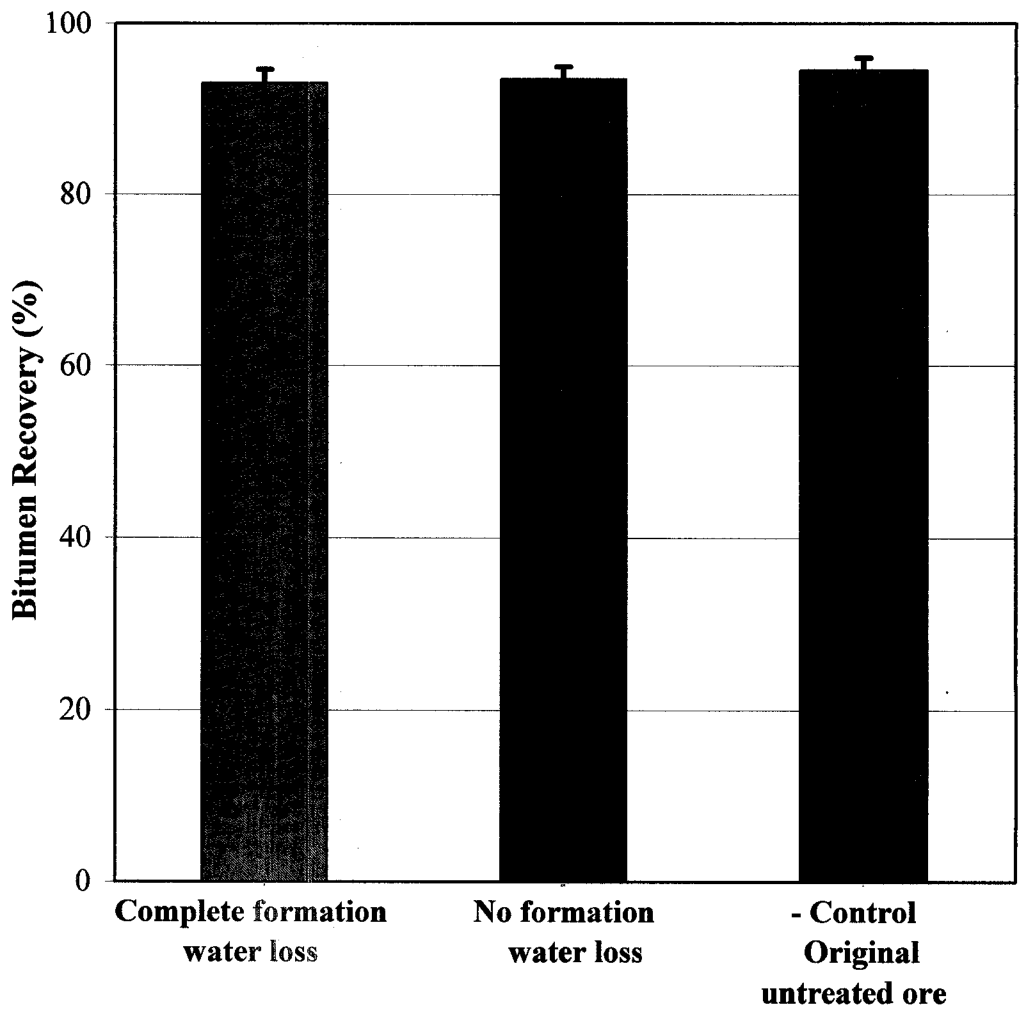


Figure 4-20b: Bitumen recovery for air treated ores (7 days in oven temperature of 60°C) using Aurora recycle process water at flotation temperature of 75°C and pH 8.1 to 8.2.

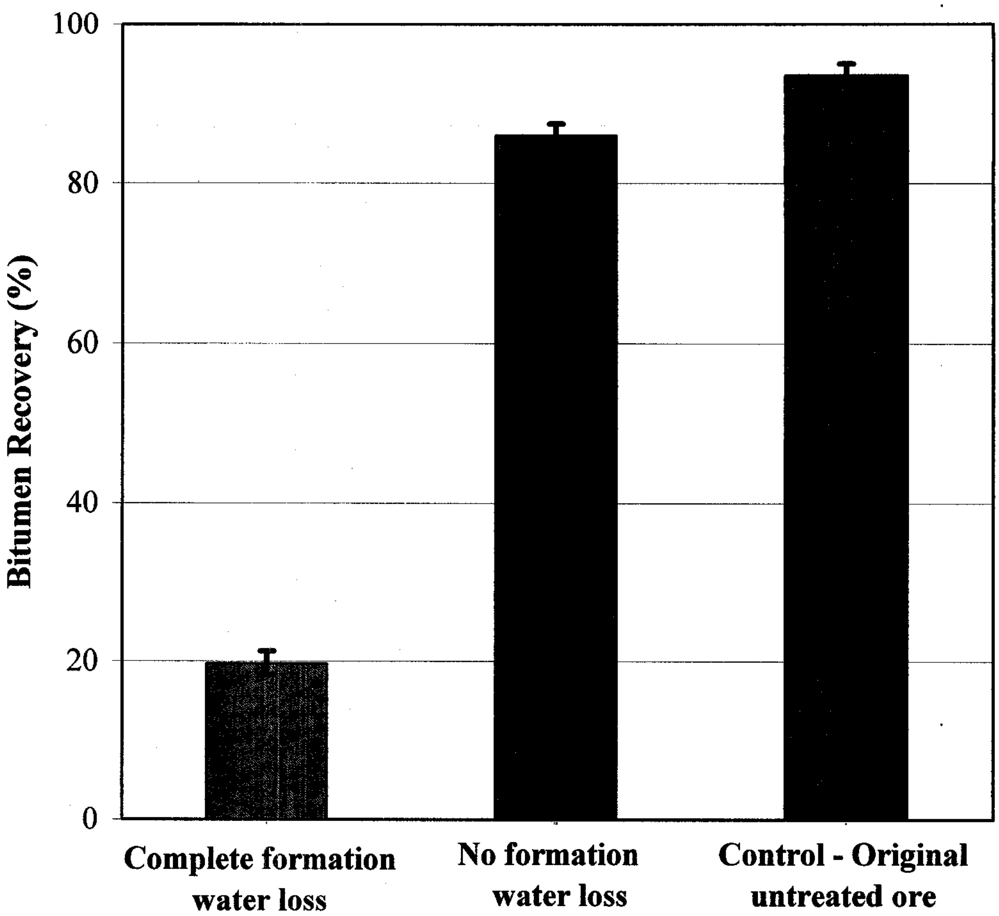


Figure 4-21a: Bitumen recovery for nitrogen treated ores (7 days in oven temperature of 60°C) using Aurora recycle process water at flotation temperature of 35°C and pH 8.1 to 8.2.

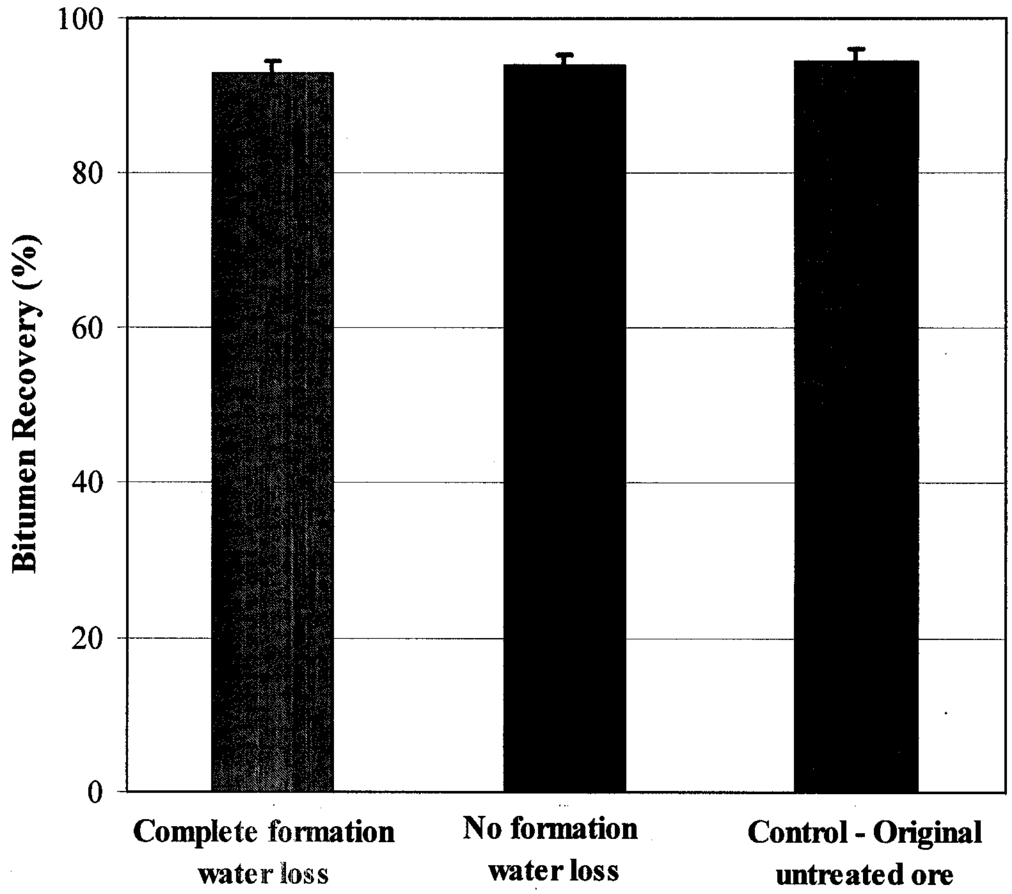


Figure 4-21b: Bitumen recovery for nitrogen treated ores (7 days in oven temperature of 60°C) using Aurora recycle process water at flotation temperature of 75°C and pH 8.1 to 8.2.

It was clear from the Figures 4-20a, 4-20b, 4-21a and 4-21b that by increasing the temperature of oil sands extraction from 35°C to 75°C, the processability of both air and nitrogen weathered ores improved drastically. Comparison of Figures 4-20a and 4-20b show an increase in bitumen recovery from 14% to 93% when flotation of air treated ore with complete water loss increased from 35°C to 75°C. The bitumen recovery of air treated ore with negligible or no water loss increased from 76% to 93% when flotation temperature increased from 35°C to 75°C. Similar trend for nitrogen treated ores is seen in Figures 4-21a and 4-21b. The bitumen recovery of nitrogen treated ores with complete water loss increased from 19.7% to 94% when flotation temperature was increased from 35°C to 75°C. Likewise, for the case of nitrogen treatment with negligible or no water loss, the bitumen recovery increased from 86% to 94% when flotation temperature was increased from 35°C to 75°C. These results are in agreement with the previous results shown in Figure 4-4 for the Suncor weathered ore. It can therefore be generally concluded that increased flotation temperature plays a major role in enhancing processability of weathered ores.

Chapter 5

Effect of weathering on wettability of solids[8]

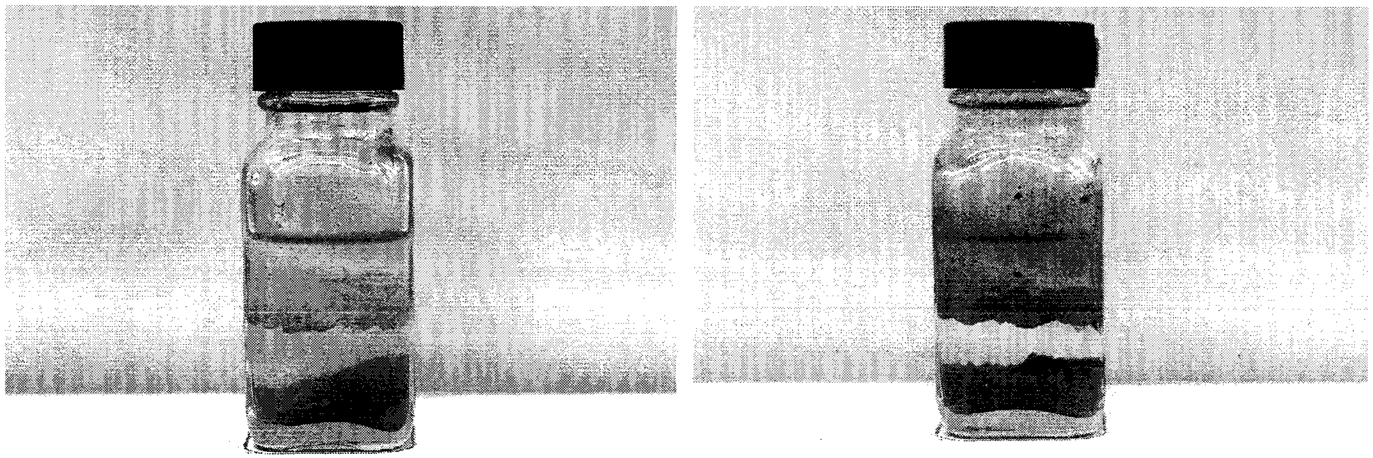
[8] data of the figures in chapter 5 are given in Appendix B

5.1 Wettability/hydrophobicity of solids from oil sand ores

Previous studies on Athabasca oil sand ores have correlated the good processing ability of the Athabasca oil sands to the hydrophilic nature of the solids. Hydrophobicity of solids in oil sands plays a critical role in controlling interactions between bitumen and solids. The hydrophobic feature of the solids can induce strong attractive forces between bitumen and solids, thereby leading to poor liberation or/and slime coating. A good marker for the hydrophobicity of solids is the partitioning of the solids in mineral oil phase and aqueous phase. The solids that are distributed in the mineral oil phase can be considered to be hydrophobic while the solids that stay in the aqueous phase are referred to as being hydrophilic. With this knowledge at hand, a hydrophobicity study of the solids was carried out by the extraction of solids from the oil sand ore followed by solids partitioning tests as described in Sections 3.2.6 and 3.2.7.

5.1.1 Wettability/hydrophobicity of solids from the Syncrude good processing ore and Suncor weathered ore

Photographs of solid partitioning test conducted for the extracted solids from both good processing ores (as a control) and Suncor weathered ores are shown in the Figure 5-1. The photograph in Figure 5-1(a) shows that for the good processing ore, almost all the extracted solids remained in the aqueous phase. An insignificant amount of solids can be seen at the mineral oil-water interface. In essence, almost all the extracted solids from the good processing ore are hydrophilic (water loving). This could be one of the reasons for the good processability of oil sand ores with bitumen recovery being 93% even at a low flotation temperature of 35°C.



(a) Solids extracted from good processing ores **(b)** Solids extracted from Suncor weathered ores

Figure 5-1: Photographs of the partitioning test for solids extracted from good processing ores and Suncor weathered ores

A visual examination of the photograph in Figure 5-1(b) indicates that more solids from the Suncor weathered ore, resides in the oil phase. It can be seen that large dark chunks of coarse hydrophobic solids that were originally in the oil phase fell down to the aqueous phase because they were too heavy to be supported by interfacial forces to keep at the interface. It may therefore be said that a significant portion of the solids from the Suncor weathered ore are more hydrophobic than solids from the Syncrude good processing ore. This could be one of the reasons for the poor processability of the Suncor weathered ore at a bitumen recovery of 39% when floated at 35°C using Aurora recycle process water.

A quantitative analysis of the tailings solid distribution in the aqueous phase (that is, hydrophilic solids) is shown in Figure 5-2. The quantitative results in Figure 5-2 show that 97% and 64% of solids from good processing ores and Suncor weathered ores are partitioned in the aqueous phase. It is clear that the good processing ore contains more hydrophilic solids than the Suncor weathered ore. This shows the importance of solids wettability in oil sands extraction and processing. An increase in hydrophilicity of solids would in general lead to a better processability.

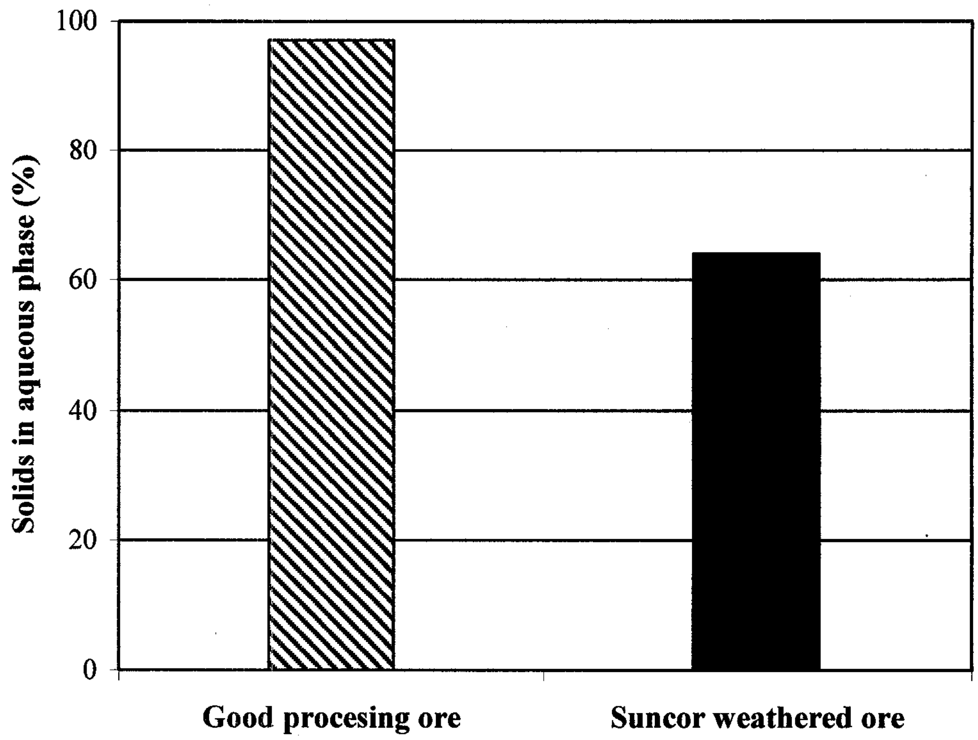
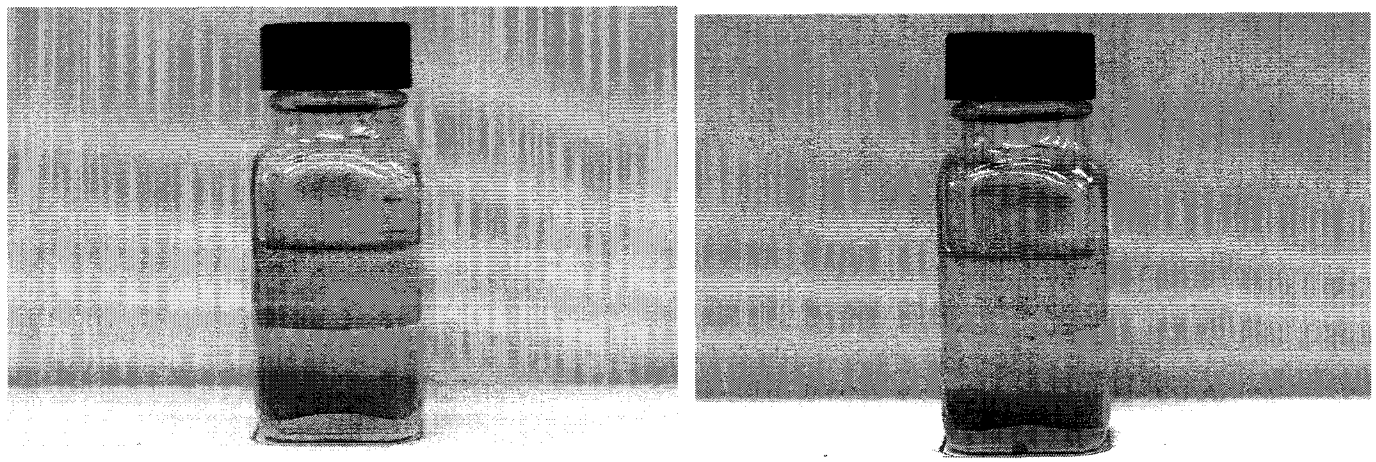


Figure 5-2: Wettability quantification for solids extracted from Syncrude good processing ores and Suncor weathered ores.

5.1.2 Effect of flotation temperature on wettability of tailings solids from Suncor weathered ores

This section is focused on wettability of solids obtained from tailings after flotation experiments were carried out at 35°C and 75°C using Suncor weathered ores. Visual observations of solids distributions in aqueous and oil phases from the partitioning test is shown in Figure 5-3, and the quantitative analysis of tailings solids distribution in the aqueous phase (that is, hydrophilic solids) is shown in Figure 5-4.



(a) Solids obtained from flotation test at flotation temperature of 35°C

(b) Solids obtained from flotation test at flotation temperature of 75°C

Figure 5-3: Photographs of the partitioning test for solids obtained from process tailings of Suncor weathered ores

A visual interpretation of the photographs shown in Figure 5-3 indicates that there is no significant difference in the tailings solids that remained in the mineral oil phase and aqueous phase for both flotation temperatures of 35°C and 75°C, as anticipated. The results in Figure 5-4 confirm the visual interpretation of no measurable difference between the wettability of the tailing solids obtained at flotation temperatures of 35°C and 75°C. The percent solids in the aqueous phase for both cases are around 98%. This implies that almost all the tailings solids are in the aqueous phase.

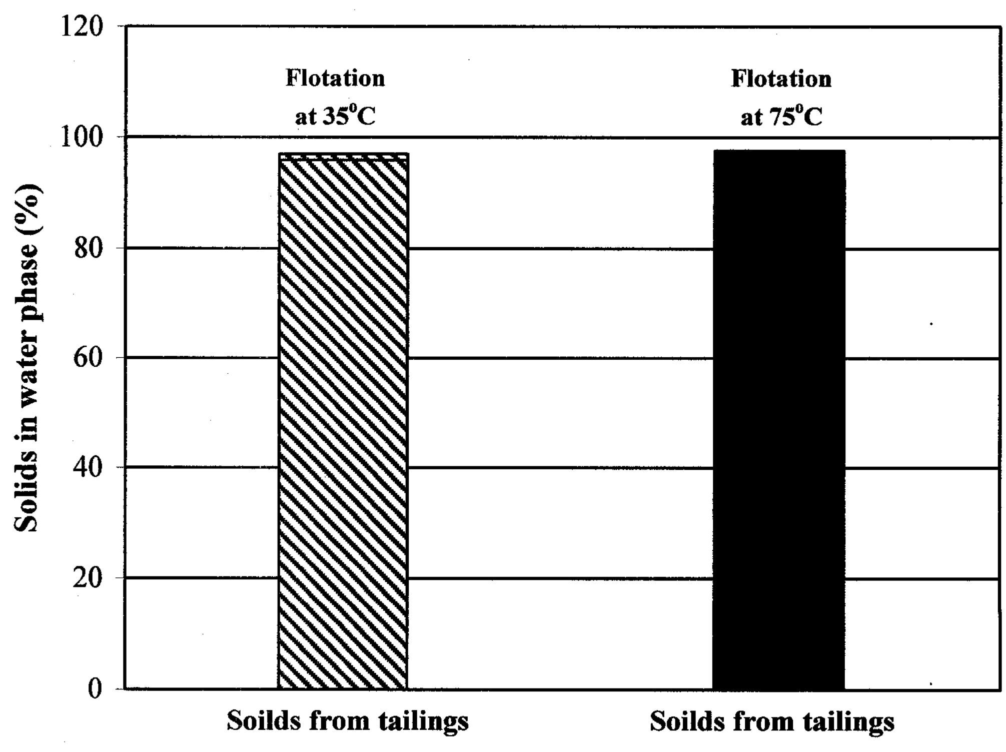


Figure 5-4: Wettability quantification for solids obtained from tailings at flotation temperatures of 35°C and 75°C for Suncor weathered ores using Aurora recycle process water.

5.1.3 Solids wettability study of air treated oil sand ores

In this section, we attempt to correlate solids wettability and formation water loss for the air-treated good processing ore in a laboratory oven. The results of the wettability study using the partitioning tests as a function of the formation water loss during the air treatment of the good processing ore are shown in Figure 5-5. In this figure, it is evident that the percent solids in the aqueous phase increases with decreasing formation water loss during the air treatment. As the formation water loss increases, the hydrophilicity of the extracted solids decreases significantly. When the treatment in the oven was performed to ensure that there is no formation water loss, the percentage of solids in the aqueous phase remained high at 93%. With partial formation water loss (about 2.61g of formation water loss), the hydrophilic solids reduced to about 26.1%. With complete formation water loss from the oil sand ore after the treatment the percent solids in the aqueous phase was reduced to only 5.5%.

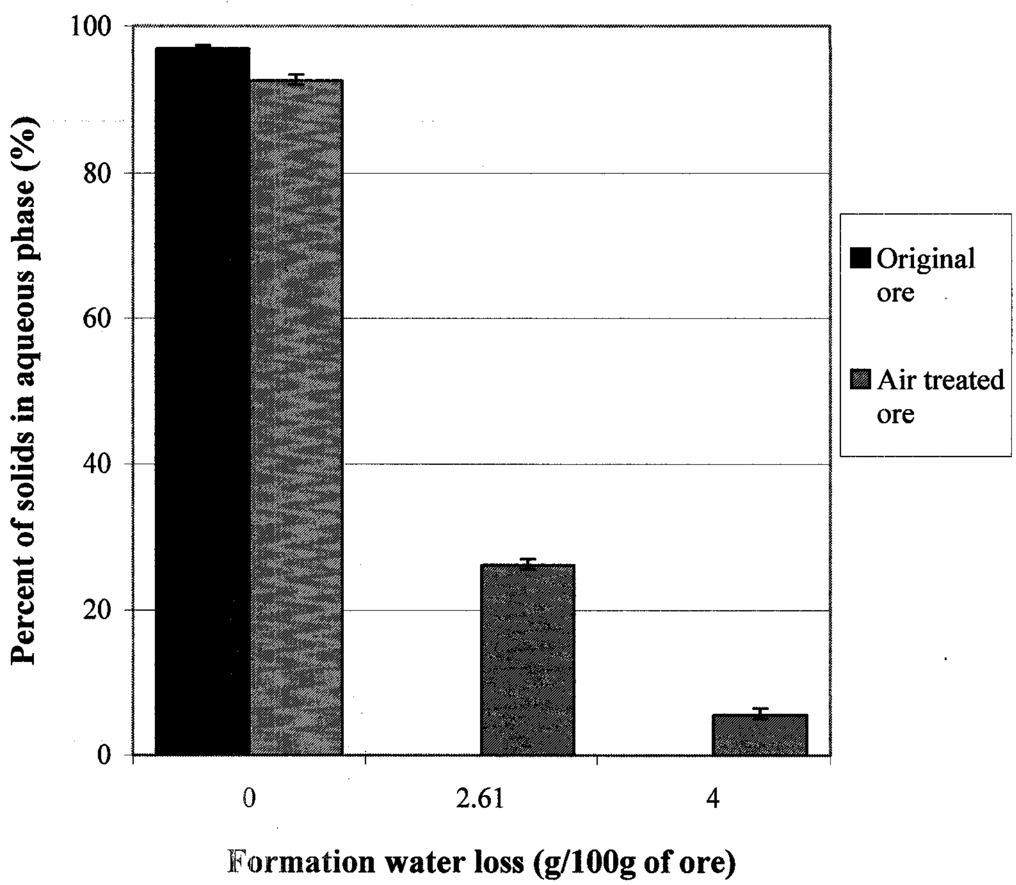


Figure 5-5: Quantitative analysis of solids wettability as a function of formation water loss for air treated ores (7 days and oven temperature of 60°C).

5.1.4 Solids wettability study of nitrogen treated oil sand ores

In this section, we attempt to make a correlation between solids wettability and formation water loss for nitrogen-treated good processing ores in a laboratory oven. The result obtained using the partitioning test as a function of the formation water loss is shown in Figure 5-6. From the results shown in Figure 5-6, it is clear that the percent solids in the aqueous phase decreases with increasing formation water loss during the nitrogen treatment. With no formation water loss, the percent solids in the aqueous phase of 97.0% are nearly the same as that of the original good processing ore prior to treatments. With partial water loss (about 2.7g of formation water loss), the hydrophilic solids decreased to about 78.0%. With total formation water loss from the ore during the treatment process, the percent solids in the aqueous phase decreased to 8.0%. A comparison of Figure 5-5 with Figure 5-6 reveals that air-treatment and nitrogen-treatment have similar effect on the change of solids wettability.

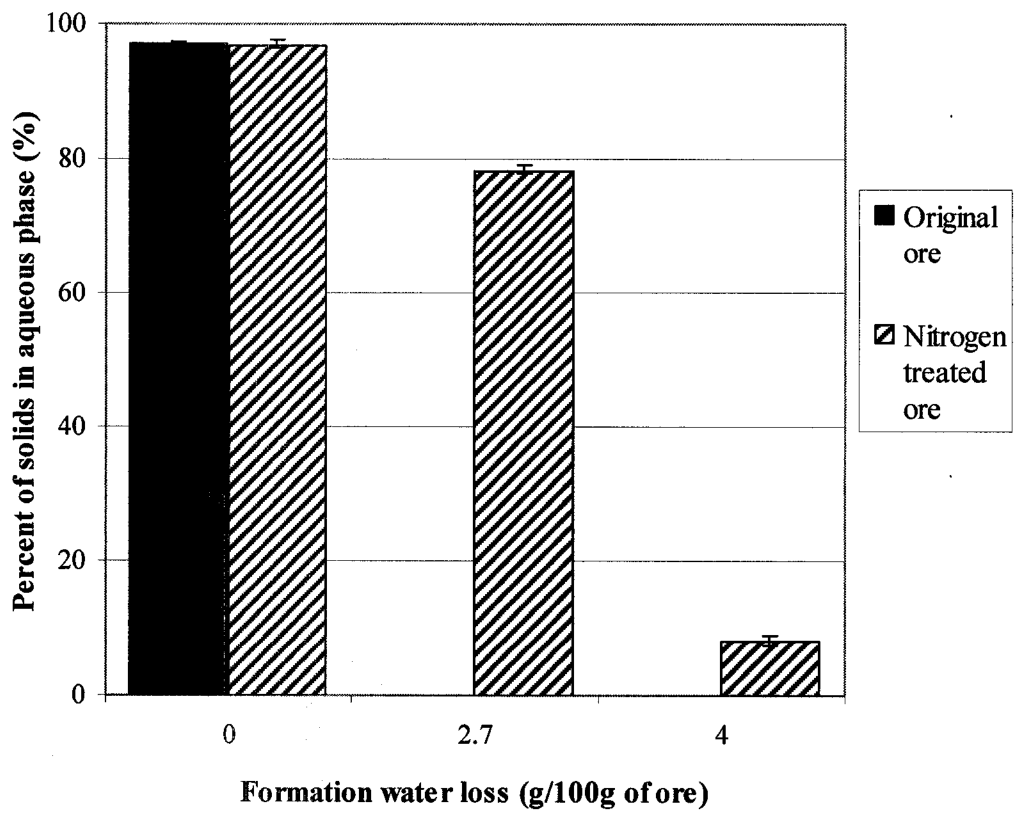


Figure 5-6: Quantitative analysis of solids wettability as a function of formation water loss for nitrogen treated ores (7 days and oven temp. of 60°C).

A close comparison of the results shown in Figures 5-5 and 5-6 reveals a lower percent of hydrophilic solids of air treated ores than nitrogen treated ores at the same formation water loss of 2.7g/100g of ore. It can therefore be concluded that both oxidation of ore and formation water loss are key factors that adversely affect the wettability of solids extracted from the laboratory treated ores.

5.1.5 Relationship between bitumen recovery and solids wettability for treated ores

An attempt was made to investigate the relationship between the bitumen recovery for treated ores and the wettability of the extracted solids. The results of this investigation are shown in Figure 5-7. From this figure it can be concluded that there is a strong and direct correlation between the bitumen recovery of weathered ores treated in laboratory oven and the percent of hydrophilic solids. Higher bitumen recovery was obtained with increasing percent of hydrophilic solids in the treated ores. One other important fact from the results shown in Figure 5-7 is that bitumen recovery of nitrogen treated ores was higher than that of air treated ores. This would imply that oxidation is an important contributing factor that adversely affects bitumen recovery from weathered oil sand ores.

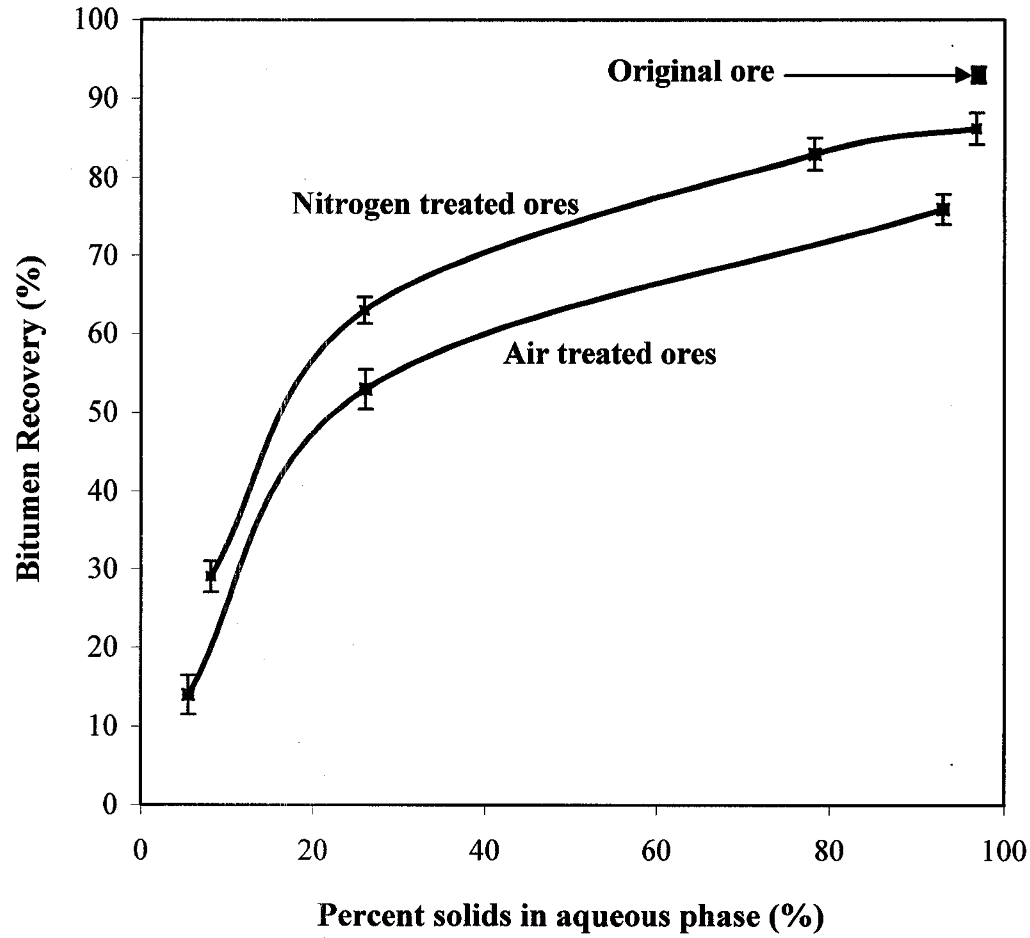


Figure 5-7: Bitumen recovery as a function of percent solids in aqueous phase for laboratory treated ores using Aurora recycle process water at a flotation temperature of 35°C.

Chapter 6

Bitumen froth morphology

6.1 Microscopic study of bitumen froth

The presence of structural features and the type of the structures observed in the morphology of bitumen froth can be directly linked with the processability of the oil sand ore from which the froth was extracted by flotation tests. Studies have shown that there is a distinct difference in the morphology of bitumen froth from good processing ores and weathered ores of poor processability (Munoz et al., 2003). The morphology of the froth obtained from a good processing ore has normal fluorescence bitumen band while the froth from a weathered ore depicts a low-fluorescence bitumen band. Also, the morphology of the froth obtained from a good processing ore is relatively featureless as compared to the morphology for the froth from the oxidized or weathered poor processing ore. The latter shows several types of structures and features. Most of these structures symbolize trapped inorganic particles in the froth. The majority of these large structures are mostly three-dimensional, and they are more visible when a confocal laser scanning microscopy (CLSM) is used (Munoz et al., 2003).

6.2 Optic microscope and instrumentation

Morphology of the bitumen froth obtained from the flotation tests were obtained by using Carl Zeiss Axioskop 40 Pol optic microscope. The microscope is equipped with a video camera that can be used to take digital photographs of the froth morphology in the field. Fluorescence mode was used for the examination of the bitumen froth sample. Fluorescence micrograph was obtained using a high-pressure mercury lamp with an average brightness of 170000 cd/cm^2.

The selection of the wavelength of the incident beam was accomplished with a combination of filters which provided a range from 440 to 490 nm (blue light). The separation of the emitted fluorescence from the incident light was achieved with a 515-nm barrier filter. The microscope has both reflected and transmitted light systems.

6.3 Morphology of bitumen froth from different types of ores at flotation temperature of 35°C using optic microscope

The following bitumen froth micrographs from Figure 6-1 to Figure 6-8 were obtained using the optic microscope.

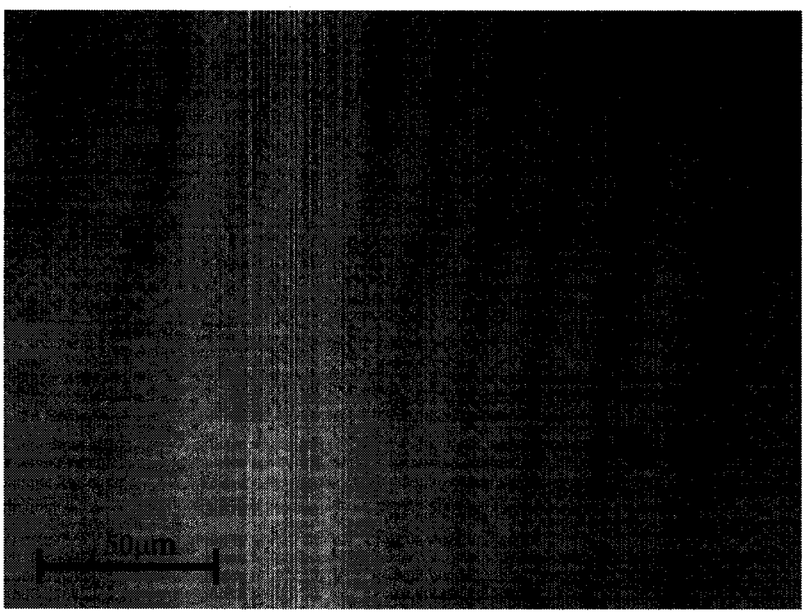


Figure 6-1: Morphology of bitumen froth obtained using the Syncrude good processing ore and Aurora recycle process water in flotation at 35°C and pH 8.4.

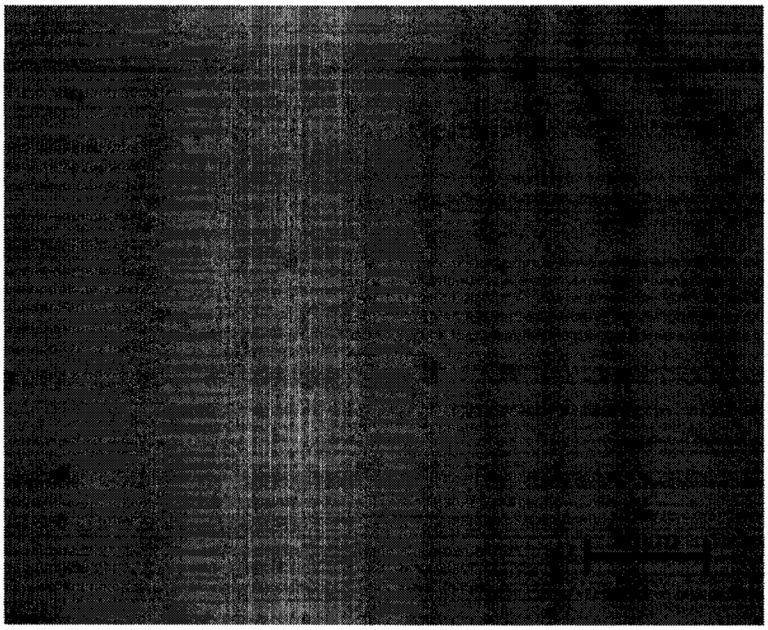


Figure 6-2: Another field of view of morphology of bitumen froth obtained using the Syncrude good processing ore and Aurora recycle process water in flotation at 35°C and pH 8.4.

Micrographs of bitumen froth obtained from a good processing ore are shown in Figure 6-1 and Figure 6-2. Figure 6-1 shows froth morphology of almost no distinct structures or features. This is typical of good processing ores. The spherical objects seen in the morphology corresponds to water droplet that must have been collected with the bitumen froth during the flotation. The same description can be attributed to the froth shown in Figure 6-2. Figures 6-3 to 6-5 are micrographs of bitumen froth obtained at a flotation temperature of 35°C using Suncor weathered ores, air-treated and nitrogen-treated good processing ores, respectively. Each shows large structures or features that are absent in the micrographs of the froth from their good processing ore counterparts. These structures could have trapped water droplets and air bubbles which may result in the formation of stable foam. The presence of trapped air bubble could be one of the reasons as to why froth collected from weathered or oxidized ores are foamy. They appeared grey in color because they contain large content of inorganic solids. The term degraded bitumen is employed to characterize these structures observed in Figures 6-3 to 6-5 (Munoz et al., 2003). The presence of large content of inorganic solids in the froth may give rise to the development of structures observed in the micrographs.

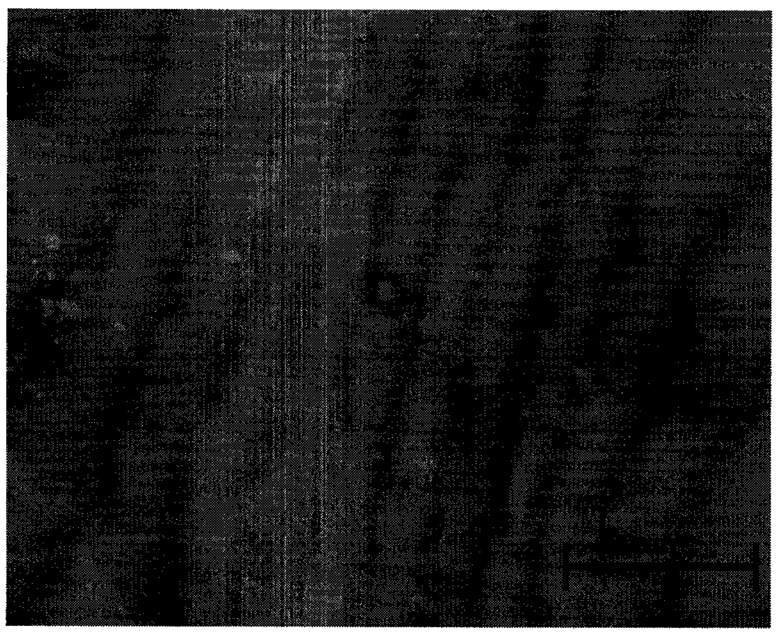


Figure 6-3: Morphology of bitumen froth obtained using the Suncor weathered ore and Aurora recycle process water in flotation at 35°C and pH 8.2.

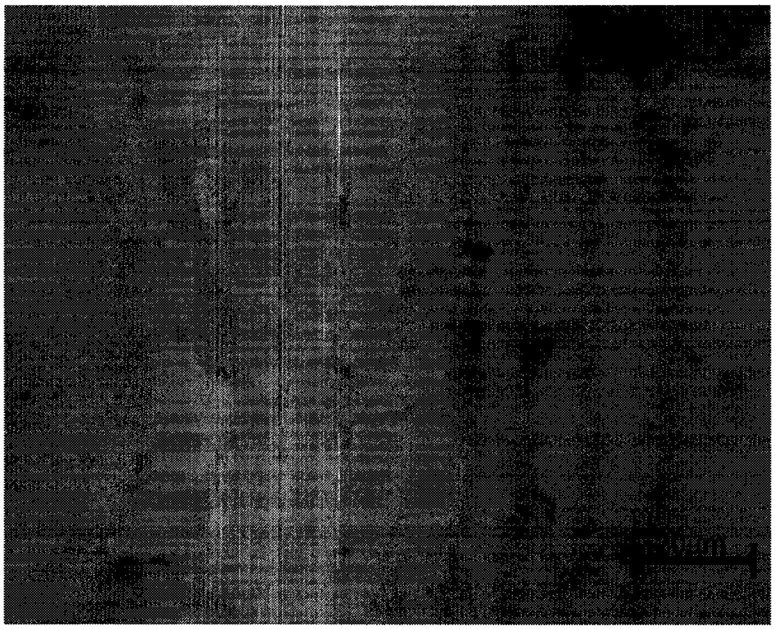


Figure 6-4: Morphology of bitumen froth obtained using the air-treated good processing ore (7 days, oven temperature of 60°C) and Aurora recycle process water in flotation at 35°C and pH 8.1.

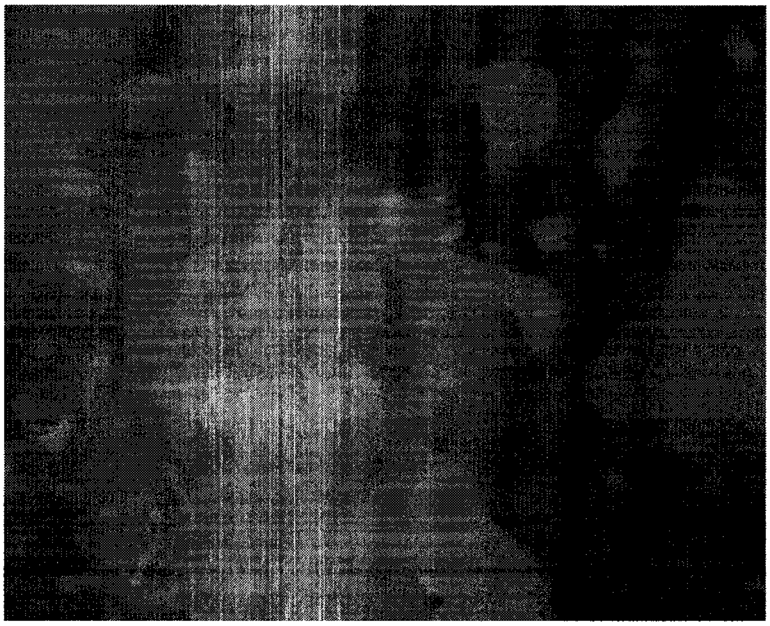


Figure 6-5: Morphology of bitumen froth obtained using the nitrogen-treated good processing ore (7 days, oven temperature of 60°C) and Aurora recycle process water in flotation at 35°C and pH 8.1.

6.4 Morphology of bitumen froth from different types of ores at flotation temperature of 75°C using optic microscope

The bitumen froth morphology of air-treated and nitrogen-treated good processing ores at a flotation temperature of 75°C are shown in Figure 6-6 and Figure 6-7, respectively. It appears that the structures and the low fluorescence bitumen band that were present while processing the ore at 35°C are no longer present. Instead, normal fluorescence bitumen band is observed. The results may imply that increased flotation temperature revert the bitumen froth morphology to the case of the original untreated ore prior to weathering under air or nitrogen environment.

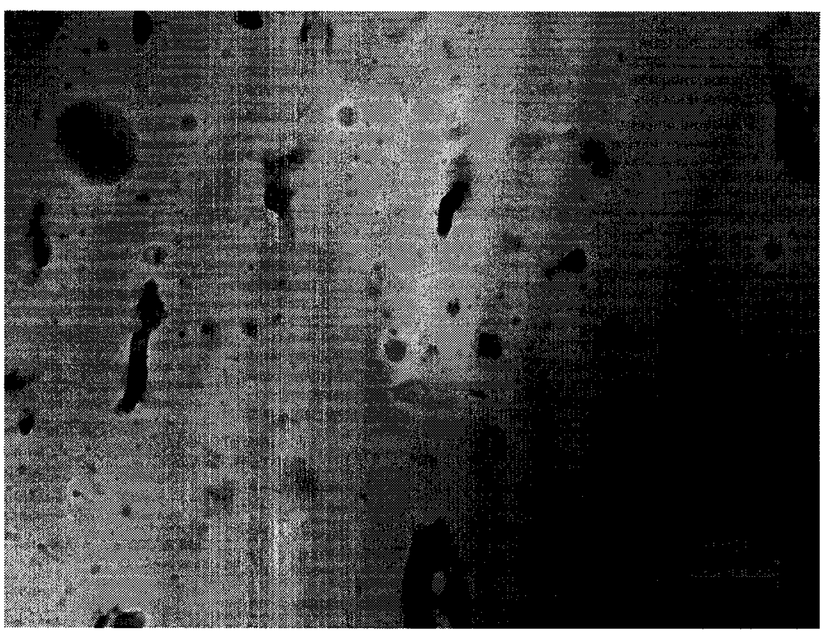


Figure 6-6: Morphology of bitumen froth obtained using the air-treated good processing ore (7 days, oven temperature of 60°C) and Aurora recycle process water in flotation at 75°C and pH 8.1.

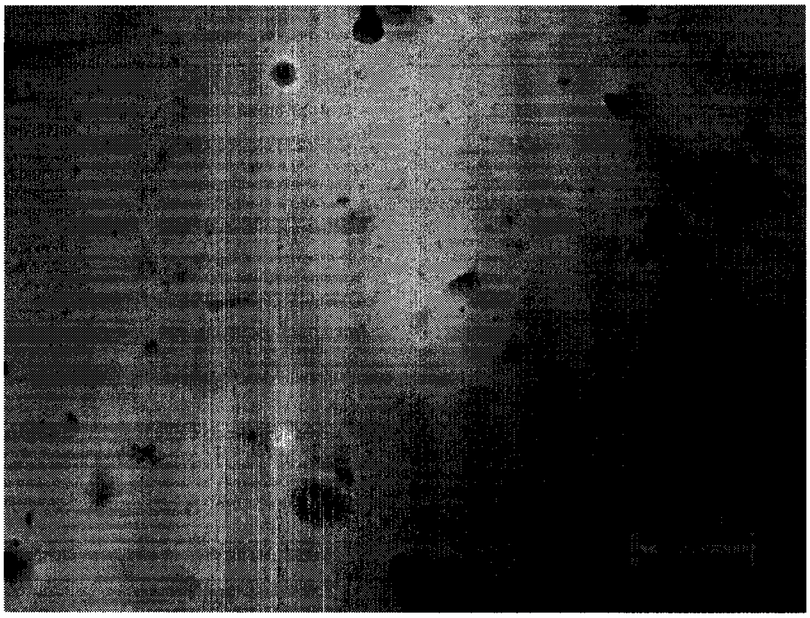


Figure 6-7: Morphology of bitumen froth obtained using the nitrogen-treated good processing ore (7 days, oven temperature of 60°C) and Aurora recycle process water in flotation at 75°C and pH 8.1.

Figure 6-8 and Figure 6-9 are micrographs of bitumen froth obtained at a flotation temperature of 75°C using Suncor weathered ores and Syncrude good processing ores, respectively.

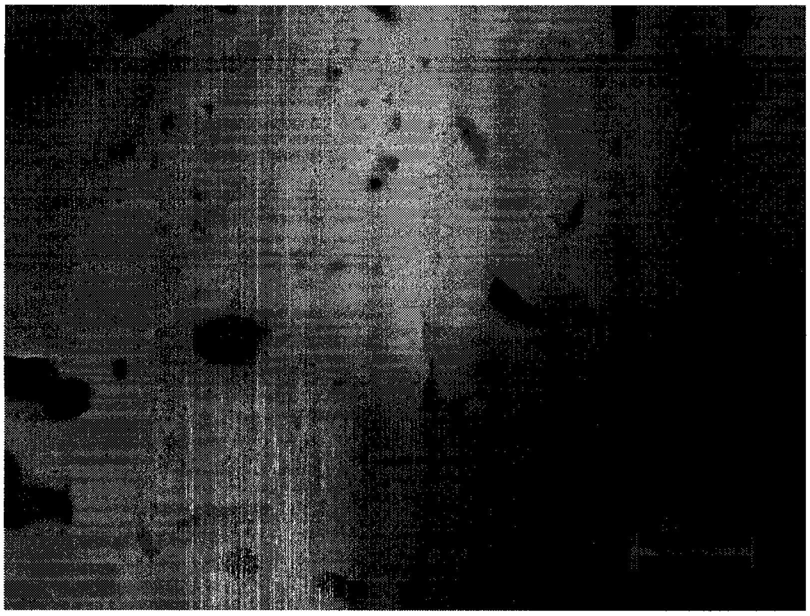


Figure 6-8: Morphology of bitumen froth obtained using Suncor weathered ores and Aurora recycle process water in flotation at 75°C and pH 8.2.

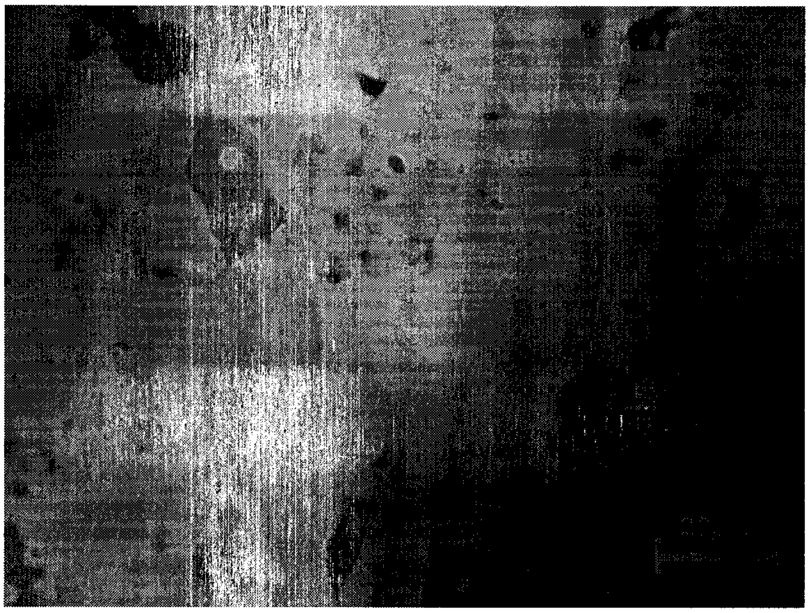


Figure 6-9: Morphology of bitumen froth obtained using Syncrude good processing ores and Aurora recycle process water in flotation at 75°C and pH 8.4.

Chapter 7

Conclusions

Based on the various tests conducted on the Suncor weathered ore, a poor processability of the ore is clear despite its high bitumen content. The visualization of shaking jar tests for the Suncor weathered ore revealed that a higher extraction temperature, addition of kerosene in the extraction process and a higher extraction pH (not higher than 10.5) enhance the processability of the ore.

The Denver cell flotation tests on the same Suncor weathered ore confirmed the observation of the shaking jar tests. At the flotation temperature of 35°C, there appeared to be no liberation of bitumen from sand grains. Higher flotation temperatures of 45°C and 75°C improved the processability of the ore. The addition of kerosene as a collector significantly enhanced the processability of the ore but only a slight improvement was recorded by the addition of sodium silicate which acted as a dispersant in the flotation tests. Our study shows that higher bitumen recovery can be achieved by tuning bitumen-sand interaction.

Further study on processability of weathered oil sand ores was implemented by artificially weathering a good processing ore in an oven under air or nitrogen environment. Such an approach was necessary to control formation water loss from the ore. It was found that bitumen recovery from the laboratory weathered ore decreased drastically and the quality of the froth became poorer as a result of weathering. Formation water inherent in the oil sand is of major importance in the processability of oil sand ores. When the ore was treated under either nitrogen or air environment to reduce the formation water content in the ore, the laboratory weathered ore became less processable with low bitumen recovery and poor froth quality. As the formation water loss during treatment increases, the laboratory weathered ore became progressively more difficult to process. Accordingly, formation water loss has been identified as a contributor to the poor processability of weathered oil sand ores.

Nitrogen-treated oil sands in the oven exhibited better bitumen recovery and froth quality as compared to air-treated oil sands for the same formation water loss. It can therefore be implied that oxidation of the ore and especially the minerals in the ore also play a significant role in determining the processability of the weathered oil sand ores.

The processability of the laboratory weathered ore was improved by increasing the flotation temperature from 35°C to 75°C, a situation similar to Suncor weathered ores. Generally, the processability of weathered or oxidized ores can be enhanced by increasing the flotation temperature.

A strong and direct correlation between the hydrophobicity of the extracted solids from oil sand ores and formation water loss exists. The more hydrophilic the solids are, the better is the processability of the ore. Solids wettability of the weathered oil sands was found to change as a result of formation water loss during oil sands weathering. Extracted solids from both the original untreated ore and treated ores with negligible or no formation water loss were virtually all hydrophilic while extracted solids from the treated ores with complete formation water loss were almost all hydrophobic.

Finally, examination of the bitumen froth obtained from flotation tests using optic microscope shows distinct differences between bitumen froth morphology of a good processing ore and weathered ores. The presence of irregular structures is attributed to trapped inorganic solids which are visible in the morphology of weathered or oxidized ores. On the other hand, the bitumen froth morphology of good processing ore has normal fluorescence of bitumen band and featureless with no visible structure.

References:

1. Ball, M.W., "Athabasca Oil Sands: Apparent Example of Local Origin of Oil", *Bull. Amer. Assoc. Petroleum Geol.* 19, 153–171 (1935).

2. Bichard, J.A., "Oil sands Composition and Behaviour Research" *AOSTRA Technical Publication Series #4* (1987).

3. Chow, R. S. and K. Takamura, "A mechanism for initiation of bitumen displacement from oil sand", *Ann. Tech. Mtng. Can. Inst. Min. Metall.*, Calgary, AB, (1982).

4. Clark, K.A. "The Bituminous Sands of Northern Alberta. Their Separation and their Utilization in Road Construction", 8, 42-58 Alberta Research Council, Edmonton Research Council of Alberta Annual Report 1922, (1923).

5. Clark, K.A., "The Separation of the Bitumen from Alberta Bituminous Sands", *Can. Inst. Min. Metall. Bull.* 20, 1385-1395 (1929)

6. Clark, K.A and D.S. Pasternack, "Hot Water Separation of Bitumen from Alberta Bituminous Sand", *Ind. Eng. Chem.* 24, 1410–1416 (1932).

7. Clark, K.A., "Hot-Water Separation of Alberta Bituminous Sand", *Trans. Can. Inst. Min. Metall.* 47, 257-274 (1944).

8. Clark, K. A., "Bituminous sands of Alberta", The Oil Weekly, (1945).

9. Cymerman, G. J. and T. Kwong, "Improvements in the Oil Recovery Flotation Process at Syncrude Canada Ltd.", Processing of Hydrophobic Minerals and Fine Coals: Proceedings of the 1st UBS McGill Bi-Annual International Symposium on Fundamentals of Mineral Processing, J.S.Laskowski and G.W.Poling, Eds., CIM, Vancouver, BC 319–328 (1995).

10. Czarnecki, J., J. K. Liu, R. G. Smith, and W. J. R. Tyerman, "Extraction of Bitumen from Athabasca Oil sands: Hot Water Process Theory, addressing temperature effects, in "*Can. Chem. Eng. Conf.*", Vancouver, BC, 1 (1991).

11. Ding, X., Z. Xu, and J.H. Masliyah, "Effects of Divalent Ions, Illite Clays and Temperature on Bitumen Recovery", *54th Canadian Chemical Engineering Conference*, Calgary, Canada, October 3-6, (2004).

12. Drelich, J., D. Lelinski, J. Hupka, and J. D. Miller, "The Role of Gas Bubbles in Bitumen Release during Oil Sand Digestion", *Fuel,* 74 (8), 1150-1155 (1995).

13. FTFC (Fine Tailings Fundamentals Consortium), "Advances in Oil Sands Tailings Research", AOSTRA, Edmonton, AB (1995).

14. Gu, Y., "Oil Sands Weathering", M.Sc thesis, University of Alberta, Fall, 2006, Department of Chemical and Materials Engineering.

15. Hall, A.C., S.H. Collins, and J.C. Melrose, "Stability of Aqueous Wetting Films in Athabasca Tar Sands", *Soc. Petrol. Eng.,* 23, 249–258 (1983).

16. Hepler, L.G. and C. His, *AOSTRA Technical handbook on oil sands, bitumen, and heavy oils, AOSTRA technical publication series #6*, Alberta Oil Sands Technology and Research Authority: Edmonton, Alberta, Canada (1989).

17. Hepler, L. G. and R.G. Smith, *The Alberta oil sands: industrial procedures for extraction and some recent fundamental research, AOSTRA technical publication series #14*, Alberta Oil Sands Technology and Research Authority: Edmonton, Alberta, Canada, (1994).

18. Hupka, J., J.D. Miller, and A. Cortes, "Importance of Bitumen Viscosity in the Hot Water Processing of Domestic Tar Sands", *Mining Engineering* 35, 1635-1641 (1983).

19. Hrouda, J., G. B. Lim, R. Paul, L.C. Stevenson, and K.N. Sury, "Hot Water Extraction Process and Apparatus, Esso Resources Canada Ltd., Canada Patent Number 2,004,352 (1995)

20. Kasperski, K.L., "Review of Research on Aqueous Extraction of Bitumen from Mined Oil Sands", Natural Resources Canada, CANMET-WRC, CONRAD Extraction TPG (2001).

21. Lam, W.W., W.J.R. Tyerman, C. Payette, R.J. Mikula, and E.C. Sanford, "Physical Structure of the Primary Froth from Athabasca Oil Sand Extractions: Cryogenic Sampling and Microscopic Observations of Pilot Plant Froths", *Fuel Sci. & Tech. Int.* 13, 483-508 (1995).

22. Li, H., Z.A. Zhou, Z. Xu, and J.H. Masliyah, "Role of Acidified Sodium Silicate in Low Temperature Bitumen Extraction from Poor-Processing Oil Sand Ores", *Ind. Eng. Chem. Res.* 44, 4753 - 4761 (2005).

23. Lipsett, M.G., "Oil Sands Extraction Research Needs and Opportunities", *Can. J. Chem. Eng.* 82, 626-627 (2004).

24. Liu J., Z. Xu, and J.H. Masliyah, "Processability of Oil Sand Ores in Alberta", *Energy & Fuels,* 19, 2056-2063 (2005)

25. Long, J., Z. Gu, and J. H. Masliyah, "On the Role of Temperature in Oil Sands Processing", *Energy & Fuels,* 19, 1440-1446 (2005).

26. Long, J., Z. Gu, and J. H. Masliyah, "Role of Illite–Illite Interactions in Oil Sands Processing", *Colloids and Surfaces A: Physicochem. Eng. Aspects* 281, 202–214 (2006).

27. Malmberg, E. W. and B. Roger, "Hot Water Processing of Athabasca oil sands: II. Microscope Studies of the Conditioning Process", American Chemical Society, San Francisco, F25-F37 (1968).

28. Masliyah, J.H, Z. Zhiang, Z. Xu, J. Czarnecki, and H. Hamza, "Understanding Water-Based Bitumen Extraction from Athabasca Oil Sands", *Can. J. Chem. Eng.,* 82, 628-654 (2004).

29. Masliyah J., Fundamentals of Oil sands Extraction – ChE 534 Courseware, Chemical and Materials Engineering Department, University of Alberta, Winter, (2005).

30. Mathieson, D. and D. Stenason, "Investing in Oil sands", Scotia Capital, *The Drill Bit,* (2001).

31. Miller, J.D. and M. Misra, "Hot Water Process Development for Utah Tar Sands", *Fuel Process. Technol.* 6, 27-59 (1982).

32. Moran, K., A. Yeung, and J.H. Masliyah, "Factors Affecting the Aeration of Small Bitumen Droplets", *Can. J. Chem. Eng.,* 78, 625-634 (2000).

33. Morgan, G., "An Energy Renaissance in Oil sands Development", *World Energy,* 4, 46–53 (2001).

34. Mossop, G. D., "Geology of the Athabasca Oil Sands", *Science* 207, 145–152 (1980).

35. Munoz, V. A., "Some Applications of Confocal Laser Scanning Microscopy in the Oil Sands and Heavy Oil Industries", Division Report CWRC 94-41 (TR); CANMET, Energy, Mines, and Resources Canada: Devon, Alberta (1994).

36. Munoz, V.A, K.L. Kasperski, O.E. Omotoso, and R.J. Mikula, "The Use of Microscopic Bitumen Froth Morphology for the Identification of Problem Oil Sand Ores", *Petroleum Science and Technology,* 21, No. 9 & 10, 1509–1529 (2003).

37. Plessis, D., "Alberta's Oil Sands Resources, Production Growth, Products and Markets", Alberta Economic Development and Alberta Energy Research Institute Calgary, Alberta, Canada (2004).

38. Robinson, D.B., "The Thermodynamic and Transport Properties of Bitumen and Heavy Oils", AOSTRA Technical Report No 2, Edmonton Alberta Oil Sands Technology and Research Authority (1984).

39. Sanford, E.C. and F.A. Seyer, "Processability of Athabasca Tar Sand Using a Batch Extraction Unit: The role of NaOH", *Can. Inst. Min. Metall. Bull.* 72, 164-16 (1979)

40. Sanford E.C., "Processability of Athabasca Oil Sands: Interrelationship Between Oil Sand Fine Solids, Process Aids, Mechanical Energy and Oil Sand Age after Mining", *Can. J. Chem. Eng.* 61, 554-567 (1983).

41. Schramm, L. L., R. G. Smith, and J. A. Stone, "The Influence of Natural Surfactant Concentration on the Hot Water Process for Recovering Bitumen from the Athabasca Oil Sands", *AOSTRA J. Res.* 1, 5–13 (1984).

42. Schramm, L. L. and R. G. Smith, "The Influence of Natural Surfactants on Interfacial Charges in the Hot Water Process for Recovering Bitumen from the Athabasca Oil Sands", *Colloids Surfaces* 14, 67–85 (1985).

43. Schramm, L.L., E. N. Stasiuk, and D. Turner, "The Influence of Interfacial Tension in the Recovery of Bitumen by Water-Based Conditioning and Flotation of Athabasca Oil Sands", *Fuel Processing Tech.* 80, 101– 118 (2003).

44. Schramm, L.L., E.N. Stasiuk, H. Yarranton, B.B. Maini, and B. Shelfantook, "Temperature Effects in the Conditioning and Flotation of Bitumen from Oil Sands in Terms of Oil Recovery and Physical Properties", *J. Can. Pet. Tech.,* 42, 55-61, (2003).

45. Sepulveda, J.E. and J.D. Miller, "Separation of Bitumen from Utah Tar Sands by a Hot Water Digestion-Flotation Technique", *Soc. Min. Eng.* 30, 1311-1320 (1978).

46. Seyer, F. A. and C. W. Gyte, "Viscosity" in "AOSTRA Technical Handbook on Oil Sands, Bitumens and Heavy Oils", *AOSTRA Technical Publication Series # 6* (1989).

47. Stasiuk, E. N., L.L. Schramm, H. Yarranton, and B. Shelfantook, "Shear and Interfacial Phenomena Involved In Reducing Process Temperature for the Recovery of Bitumen from Athabasca Oil Sand", 227th ACS National Meeting, Anaheim, CA, March 28-April 1, (2004).

48. Sury, K. N., R. Paul, T. M. Dereniwski, and D. G. Schulz, "Next Generation Oil Sands Technology: The New OSLO Processes", Oil Sands Conference, Alberta Chamber of Resources, AOSTRA, EMR Canada, Edmonton, 1-36 (1993)

49. Takamura, K., "Microscopic Structure of Athabasca Oil Sand", *Can. J. Chem. Eng.* 60, 538–545 (1982).

50. Takamura, K. and R.S. Chow, "A Mechanism for Initiation of Bitumen Displacement from Oil Sand", *J. Can. Pet. Tech.* 22, 22-30 (1983).

51. Takamura, K. and E. E. Isaacs, Interfacial Properties in "AOSTRA Technical Handbook on Oil Sands, Bitumens and Heavy Oils", Hepler, L.G. and Hsi, C. (eds.), 100-128 (1989).

52. Wallace, D. and D. Henry, "Aging of Oil Sand During Storage", Engineering Foundation Conf. on the Processing of Energy Minerals: Shale, Tar Sands and Coal, Henniker, New Hampshire, 1-14 (1984).

53. Wallace, D., D. Henry, and K.A. Takamura, "A Physical Chemical Explanation for the Deterioration in the Hot Water Processability of Athabasca Oil Sand Due to Aging", *Fuel Sci. Technol. Int.* 7(5–6), 699–725 (1989).

54. Wallwork, V., Z. Xu, and J. H. Masliyah, "Bitumen Recovery with Oily Air Bubbles". *Can. J. Chem. Eng.* 81, 993–997 (2003).

55. Zhou, Z., T. Kasongo, Z. Xu, and J. Masliyah, "Assessment of Bitumen Recovery from the Athabasca Oil Sands Using a Laboratory Denver Flotation Cell", *Can. J. Chem. Eng.* 82, 696-703 (2004).

Appendix A

Data of Figure 4-4

X Flotation Time (min)	*Y* Bitumen Recovery (%)		
	Flotation at 35°C	*Flotation at 45°C*	*Flotation at 75°C*
0	0.00	0.00	0.00
2	17.87	37.03	62.13
5	27.74	58.05	88.39
10	39.20	72.13	93.99

Data of Figure 4-5

X Flotation Time (min)	*Y* Bitumen/Solids (wt/wt)		
	Flotation at 35°C	*Flotation at 45°C*	*Flotation at 75°C*
2	0.13	0.21	0.33
5	0.15	0.23	0.35
10	0.16	0.24	0.37

Data of Figure 4-6

X *Flotation Time* *(min)*	*Y* *Bitumen/Water (wt/wt)*		
	Flotation at 35°C	*Flotation at 45°C*	*Flotation at 75°C*
2	0.22	0.39	0.68
5	0.23	0.38	0.55
10	0.24	0.37	0.51

Data of Figure 4-7

X	*Y* *g/100g of froth*		
	Flotation at 35°C	*Flotation at 45°C*	*Flotation at 75°C*
Solids in froth	54.60	54.00	49.00
Bitumen in froth	8.80	12.54	18.70
Water in froth	36.60	33.43	32.00

Data of Figure 4-9

X Flotation Time (min)	*Y* Bitumen Recovery (%)			
	Without Na_2SiO_3	*500 ppm Na_2SiO_3*	*2000 ppm Na_2SiO_3*	*3500 ppm Na_2SiO_3*
0	0.00	0.00	0.00	0.00
2	17.87	13.42	23.13	20.09
5	27.74	26.85	38.68	33.20
10	39.20	40.27	45.17	42.10

Data of Figure 4-10

X Flotation Time (min)	*Y* Bitumen/Solids (wt/wt)			
	Without Na_2SiO_3	*500 ppm Na_2SiO_3*	*2000 ppm Na_2SiO_3*	*3500 ppm Na_2SiO_3*
2	0.13	0.14	0.17	0.19
5	0.15	0.17	0.18	0.20
10	0.16	0.20	0.19	0.22

Data of Figure 4-11

X Na_2SiO_3 *dosage (ppm)*	*Y* *Solution pH*	
	Process water	*Tailings water*
100	9.25	8.80
400	9.75	9.50
700	10.00	9.75

Data of Figure 4-12

X *Flotation Time (min)*	*Y* *Bitumen Recovery (%)*	
	Without Kerosene	*2500 ppm Kerosene*
0	0.00	0.00
2	17.92	25.86
5	27.62	45.48
10	39.11	62.04

Data of Figure 4-13

X *Flotation Time (min)*	*Y* *Bitumen/Solids (wt/wt)*	
	Without Kerosene	*2500 ppm Kerosene*
2	0.13	0.32
5	0.15	0.31
10	0.16	0.30

Data of Figure 4-14

X *Formation water loss* *(g/100g of ore)*	*Y* *Bitumen Recovery (%)*	
	Air treated ores	*Original ores*
0.00	76.00	93.00
2.61	53.00	-
4.00	14.00	-

Data of Figure 4-15

X Formation water loss (g/100g of ore)	*Y* Bitumen/Solids (wt/wt)	
	Air treated ores	*Original ores*
0.00	4.70	17.00
2.61	3.70	-
4.00	0.50	-

Data of Figure 4-16

X Formation water loss (g/100g of ore)	*Y* Bitumen Recovery (%)	
	Nitrogen treated ores	*Original ores*
0.00	86.00	93.00
2.70	73.40	-
4.00	19.70	-

Data of Figure 4-17

X *Formation water loss (g/100g of ore)*	*Y* *Bitumen/Solids (wt/wt)*	
	Nitrogen treated ores	*Original ores*
0.00	14.10	17.00
2.70	9.30	-
4.00	1.31	-

Data of Figure 4-18

X *Formation water loss (g/100g of ore)*	*Y* *Bitumen Recovery (%)*		
	Air treated ores	*Nitrogen treated ores*	*Original ores*
0.00	76.00	86.00	93.00
2.61	53.00	-	-
2.70	-	73.40	-
4.00	14.00	19.70	-

Data of Figure 4-19

X *Formation water loss* *(g/100g of ore)*	*Y* *Bitumen/Solids (wt/wt)*		
	Air treated ores	*Nitrogen treated ores*	*Original ores*
0.00	4.70	14.10	17.00
2.61	3.70	-	-
2.70	-	9.30	-
4.00	0.50	1.31	-

Data of Figure 4-20a

X	*Y* *Bitumen Recovery (%)*
Complete formation water loss	14.00
No formation water loss	76.00
Control - Original untreated ores	93.00

Data of Figure 4-20b

X	*Y* *Bitumen Recovery (%)*
Complete formation water loss	93.00
No formation water loss	93.50
Control - Original untreated ores	94.50

Data of Figure 4-21a

X	*Y* *Bitumen Recovery (%)*
Complete formation water loss	19.70
No formation water loss	86.00
Control - Original untreated ores	93.00

Data of Figure 4-21b

X	*Y* *Bitumen Recovery (%)*
Complete formation water loss	93.00
No formation water loss	94.00
Control - Original untreated ores	94.50

Appendix B

Data of Figure 5-2

X *Name of ore*	*Y* *Percent of solids in aqueous phase (%)*
Good procesing ores	97.00
Suncor weathered ores	64.00

Data of Figure 5-4

X	*Y* *Percent of solids in aqueous phase (%)*
Tailing solids (Flotation at 35°C)	97.10
Tailing solids (Flotation at 75°C)	97.70

Data of Figure 5-5

X *Formation water loss* *(g/100g of ore)*	*Y* *Percent of solids in aqueous phase (%)*	
	Air treated ores	*Original ores*
0.00	92.60	97.00
2.61	26.10	
2.70	-	-
4.00	5.50	-

Data of Figure 5-6

X *Formation water loss (g/100g of ore)*	*Y* *Percent of solids in aqueous phase (%)*	
	Nitrogen treated ores	*Original ores*
0.00	96.80	97.00
2.61	-	
2.70	78.30	-
4.00	8.10	-

Data of Figure 5-7

X *Percent of solids in aqueous phase (%))*	*Y* *Bitumen Recovery (%)*		
	Air treated ores	*Nitrogen treated ores*	*Original ores*
5.50	14.00	-	-
8.10	-	29.00	-
2.60	-	63.00	-
2.61	53.00	-	-
78.30	-	83.00	-
93.00	76.00	-	-
96.80	-	86.20	-
97.00	-	-	93.00