University of Alberta

Effect of Oil Sands Slurry Conditioning on Bitumen Recovery from Oil Sands Ores

by

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Abstract

The effect of slurry conditioning on bitumen recovery and bitumen froth quality has been studied by using three oil sands ores tested with a laboratory hydrotransport extraction system (LHES) and a Denver flotation cell.

Tests with the LHES show that an increase in slurry conditioning time yielded a lowered bitumen recovery for a long flotation time (30 min). Longer slurry conditioning time led to a better bitumen froth quality regardless of flotation time. However the over conditioning could be compensated by higher conditioning temperatures and higher slurry flow velocities.

Tests with the Denver flotation cell show that the increase in slurry conditioning time resulted in a higher bitumen recovery and a better bitumen froth quality for both good and poor processing ores for a shorter flotation time of 5 min. For a longer flotation time of 20 min, increasing slurry conditioning time had little impact on bitumen recovery but led to a slightly better bitumen froth quality for the good processing ore whereas no effect on bitumen froth quality of the poor processing ore.

Results also show that higher slurry temperatures and stronger mechanical energy input were beneficial to both bitumen recovery and bitumen froth quality for all three oil sands ores tested on both devices.

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B%–bitumen recovery (%);

B/S–bitumen to solids mass ratio of bitumen froth;

B/W–bitumen to water mass ratio of bitumen froth;

B_{froth}–mass of bitumen reported to the bitumen froth (kg);

Bit%–bitumen content in bitumen-toluene solution (wt%);

B_{ore}–mass of bitumen in initial ore (kg);

k–the bitumen recovery rate constant (min^{-1});

L%–percentage of bitumen liberated over the total bitumen in the initial ore;

R_t –the bitumen recovery at time *t*;

R_∞ –the ultimate bitumen recovery;

ΔG_{bs} –Gibbs free energy of bitumen liberation (J/m^2);

ΔG_{ba} –Gibbs free energy of bitumen aeration (J/m^2);

ΔG_{sa} –Gibbs free energy of fines-air attachment (J/m^2);

ζ–volumetric fraction of drops with specific diameter d (μm^{-1});

ε–average energy dissipation rate or turbulence strength (W/kg);

γ_{ab} –interfacial energy of air-bitumen (J/m^2);

γ_{as} –interfacial energy of air-fines (J/m^2);

γ_{bs} –interfacial energy of bitumen-solid (J/m^2);

γ_{bw} –interfacial energy of bitumen-water (J/m^2);

γ_{sw} –interfacial energy of solid-water (J/m^2);

γ_{aw} –interfacial energy of air-water (J/m^2).

1. Introduction

1.1 Alberta Oil Sands

Oil sands, also known as tar sands, are referred to unconsolidated sand deposits containing high molar mass viscous bitumen, which is chemically similar to heavy oil. Oil sands deposits are one of the world's largest fossil fuel resources. Alberta of Canada has the largest bitumen source located in three areas of Northern Alberta: Athabasca (4.3 million hectares), Peace River (976 thousand hectares) and Cold Lake (729 thousand hectares), among which the one in Athabasca is the largest oil sands deposits (Alberta Energy and Utilities Board, 2004). It is estimated that the total bitumen (initial volume in-place) in Alberta oil sands is 272 10^9 m^3. The initial established reserves that can be recovered by current technologies are $28.39 \cdot 10^9$ m^3, of which $27.45 \cdot 10^9$ m^3 remains the established reserves. An estimation of about 18 per cent ($4.96 \cdot 10^9$ m^3) could be recovered by surface mining methods (ERCB ST98, 2008). The oil sands reserves amenable to surface mining methods are mainly located in Athabasca area.

Typically, mineable oil sands contain about 7~12% bitumen, 4~7% water, and 84% minerals by mass. The total content of bitumen and water is fairly constant at about 16% by mass (Masliyah, 2006). The minerals in the oil sands include both coarse solids and the fines (particle sizes less than 44 microns). The results of x-ray diffraction analysis on oil sands solids show a typical composition of 82 wt% quartz, 5 wt% feldspar, 7 wt% illite, 4 wt% kaolinite, 1 wt% chlorite, 1 wt% mixed layer clays, and trace amounts of calcite, smectite, etc. (Hepler and Smith, 1994). In the fines, it contains some clay minerals (particle sizes less than 2 microns), which have potential impacts on bitumen extraction. The particle size distribution (PSD) of the mineral solids is a function of bitumen content (Cameron Engineers, 1978).

Fortunately, oil sands ores in Athabasca region close to Athabasca River are near surface and the contained solids are hydrophilic. The near surface feature of oil sands deposits enables them to be recovered by surface mining techniques (FTFC, 1995). The hydrophilic characteristics of solid surfaces imply that Athabasca oil sands deposits are amenable for water-based extraction technologies to recover bitumen (Mossop, 1980). Several structural models were proposed to describe the occurrence of sand grain, water, bitumen and fines in the oil sands ore (Cottrell, 1963; Takamura, 1982). All these models describe that the bitumen phase is separated from the minerals by a thin film of water surrounding the sand grains and suspension of fines in the water phase. Figure 1.1 is a refined microstructure model proposed by Takamura (1982).

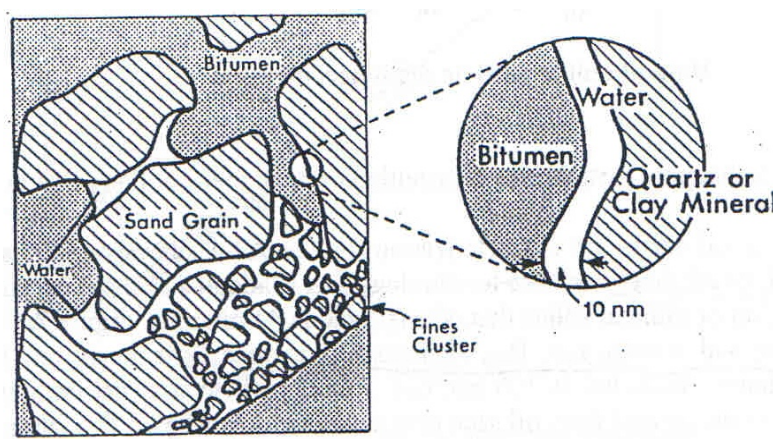


Figure 1.1 A refined schematic structure model of Athabasca oil sands (Takamura, 1982).

1.2 Surface Mining

Many different methods for recovery of bitumen from oil sands ores have been invented and developed or are still under development. Two major techniques are currently employed to recover bitumen: in *situ* thermal recovery and surface mining. Although about 82% of Alberta oil sands are buried so deep that only in *situ* recovery techniques are applicable, at present surface mining operations are still the major players in Canadian oil sands industry. In 2007, Alberta produced 209.9 thousands (10^3) m^3/d of crude bitumen from all three regions: Athabasca, Peace River and Cold Lake, with surface mining accounting for 59 per cent (ERCB, 2008).

A typical surface mining operation includes several operation steps. First the overburden has to be removed with trucks and shovels. Then oil sands ore is mined with electric or hydraulic shovels and transported from the mine face to crushers by trucks. Following various forms of slurrying, initially tumblers were employed to prepare and condition the water/sand slurry, but now pipelines have replaced tumblers to transport and condition the oil sands slurry from the mining area to the extraction site, which is generally referred to as "hydrotransport". Then the conditioned slurry is flooded with warm water and aerated bitumen is separated in the form of bitumen froth from the slurry in a primary separation vessel (PSV). Bitumen froth is treated using diluents (usually naphtha or paraffinic solvent) to remove water and solids entrained in bitumen froth. Finally the bitumen is sent to upgrading unit to be upgraded into light synthetic crude. A generic flow diagram of surface mining oil sands operation is shown in Figure 1.2 (FTFC, 1995).

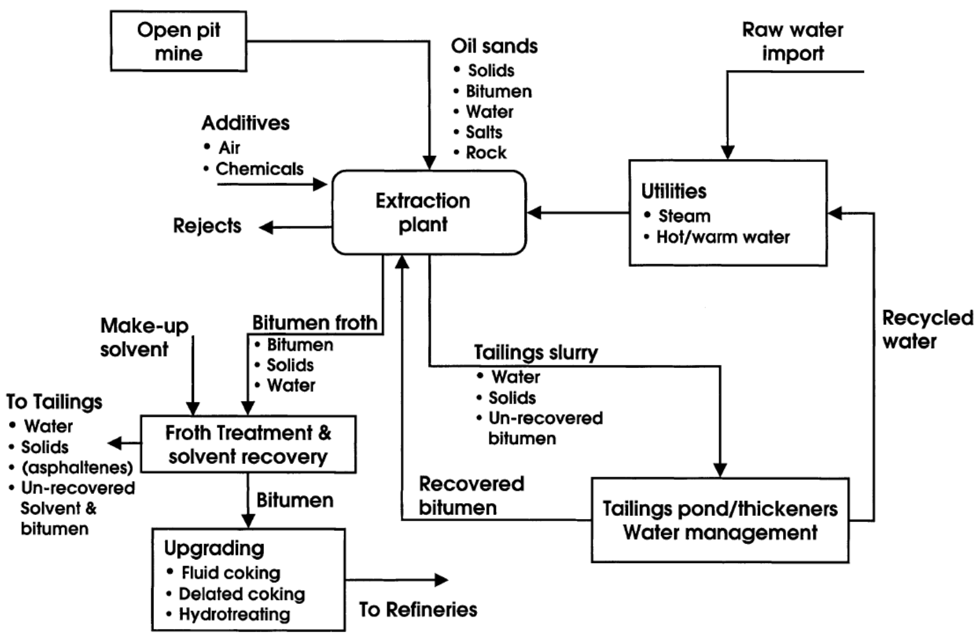


Figure 1.2 A generalized scheme for oil sands processing (Masliyah, 2006).

Current surface mining methods are almost exclusively based on Clark hot water extraction (CHWE) process (Clark and Pasternack, 1932). Following the pioneering researches conducted by Dr. Carl Clark and his colleagues in the 1920s and subsequent tests at laboratory-, bench-, and field-scales, hot water and caustic addition technology, i.e., CHWE process, was invented and developed to separate bitumen from Athabasca oil sands reserves. The CHWE process used hot water and caustic addition to liberate and separate bitumen from oil sands ores at temperatures around 70–80 °C. It was commercialized first by Suncor Energy in 1967. Since then, it has become the foundation of current oil sands processing technology for surface mining operations in Canada. To save energy and minimize the impacts on environments, the industry has changed to operate at lower temperatures by hydrotransport. Now oil sands mining operations employ warm water extraction (WWE) and low energy extraction (LEE) processes (Masliyah, 2006).

1.3 Bitumen Extraction

1.3.1 Extraction Stages

The basic steps of bitumen extraction comprise ore preparation (involving lump crushing, mixing with water and chemical addition), slurry conditioning (concurrent bitumen liberation, bitumen-bitumen coalescence and bitumen aeration), and separation of aerated bitumen aggregates from the slurry (flotation of bitumen-air bubble aggregates to the top of the slurry as bitumen froth) (Masliyah, 2006). It was postulated that the water-based extraction processes involve the following stages as shown in Figure 1.3 (Masliyah, 2006): i) oil sands lump ablation; ii) bitumen liberation or separation from a sand grain; iii) aeration of liberated bitumen; iv) flotation of aerated bitumen to the top of the slurry.

Ablation of Oil Sands Lumps The ablation process starts when oil sand lumps are mixed with hot water and are subjected to mechanical shear in tumblers, mixing boxes, rotary breakers and hydrotransport pipelines (Masliyah, et al., 2004). In the lump, bitumen acts as the glue among oil sands. When exposed to hot water, the outer layer is heated, causing significant decrease in bitumen viscosity, hence it can be easily sheared away by the turbulence in the slurry. As a result, a fresh lump surface becomes exposed to the surrounding warm environment. This process repeats itself until the whole oil sand lump is ablated.

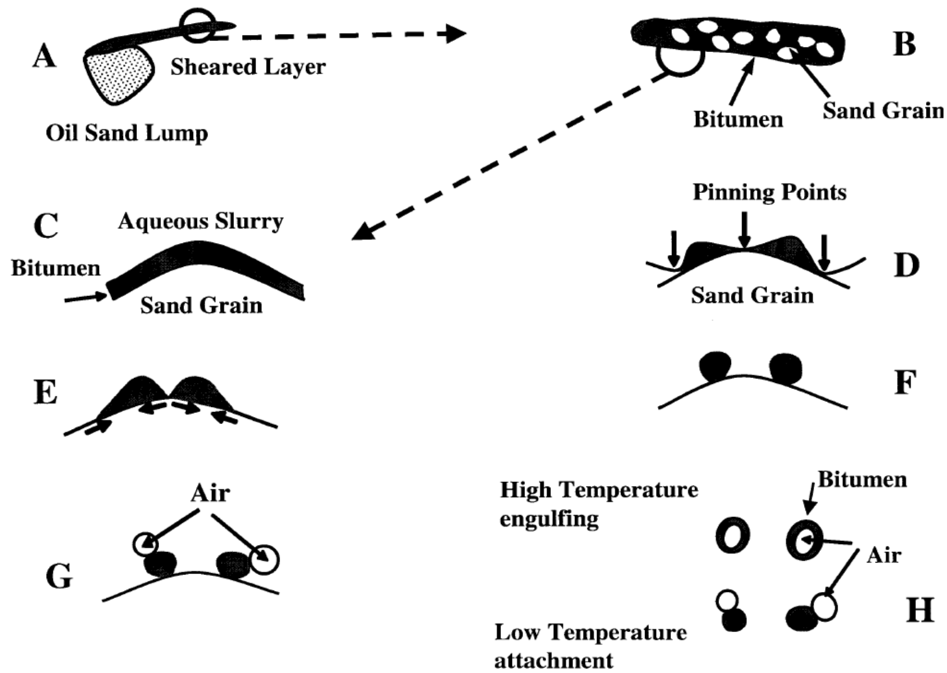


Figure 1.3 Conceptual stages for bitumen liberation and aeration (Masliyah, 2006).

Liberation of Bitumen from Sand Grains It is conceived that liberation step consists of bitumen thinning (stage D), the formation of a pin-hole (i.e., pinning) (stage E), and then the formation of bitumen droplets on the sand grains (stage F) (see Figure 1.3). Masliyah (2006) also mentioned that the shear action in the digestion unit helped the liberation of bitumen within the sand pores.

The liberation rate depends on the balance between forces pulling the bitumen away from the sand grains and forces of bitumen adhering to these grains. Masliyah, et al. (2004) reported that these forces were influenced by the process temperature, mechanical agitation, chemical additives and interfacial properties. All these factors govern how strongly the bitumen adheres to the surface of solids, thereby determining the bitumen recovery from the oil sands. According to the surface thermodynamics, the energy required to detach bitumen from sand grains can be expressed as

$$\Delta G_{bs} = \gamma_{bw} + \gamma_{sw} - \gamma_{bs} \tag{1-1}$$

where ΔG_{bs} is Gibbs free energy of bitumen liberation (J/m^2); γ_{bw}, γ_{sw} and γ_{bs} are interfacial energies of bitumen-water, solid-water, and bitumen-solid (J/m^2), respectively.

Equation (1-1) indicates that the possibility of bitumen liberation depends on three interfacial energies. As the surface of solids is more hydrophilic, the affinity of solids for bitumen decreases, therefore a high bitumen-solid interfacial energy. This leads to a more negative ΔG_{bs}, which is beneficial to bitumen liberation. Thus the hydrophilic solid surface facilitates the bitumen liberation.

Aeration of Bitumen The bitumen once disengaged from a sand grain tends to coalesce to form bitumen droplets and then be aerated with air bubbles available in the slurry (see stages G and H in Figure 1.3). At low temperatures (e.g. < 35 °C), bitumen drops just attach to air bubbles. However, at high temperatures (e.g. >45 °C), bitumen tends to spread along the surface and engulfs the air bubbles (Masliyah, 2006). Similar to the bitumen liberation, the associated bitumen aeration energy depends on three interfacial energies and is expressed as

$$\Delta G_{ba} = \gamma_{ab} - \gamma_{bw} - \gamma_{aw} \tag{1-2}$$

where ΔG_{ba} is the Gibbs free energy of bitumen aeration (J/m^2); γ_{ab}, γ_{bw} and γ_{aw} are the interfacial energies of air-bitumen, bitumen-water, and air-water interfaces (J/m^2), respectively.

Equation (1-2) implies that the possibility of bitumen attaching to air bubbles also depends on three interfacial energies. As bitumen and air bubbles are inherently hydrophobic, their affinity leads to a decrease in their interfacial energy, which in turn increases the tendency of bitumen aeration.

Flotation of Aerated Bitumen In this step, usually taking place in PSV, bitumen is separated from most of the mineral solids and water (Kasperski, 2001). Due to the buoyancy effect the aerated bitumen floats to the top of the gravity separation vessel and is consequently recovered as bitumen froth. Bitumen recovery and bitumen froth quality depend largely on the size and density of the aerated bitumen drops (Friesen, et al., 2004b).

1.3.2 Bitumen Recovery

Bitumen recovery is referred to either primary or overall bitumen recovery. For clarity, in the present study, bitumen recovery is defined as the percent of bitumen in froth collected to the total bitumen in the feed, expressed as

$$B\% = 100 \times B_{froth} / B_{ore} \tag{1-3}$$

where $B\%$ is the bitumen recovery (%); B_{froth} the mass of bitumen reported to the bitumen froth (kg); B_{ore} the mass of bitumen in the initial ore (kg).

The overall bitumen recovery for surface mining operations is about 90%. Bitumen recovery strongly depends on ore quality and operating conditions involved in mining, extraction and froth treatment. In a review of bitumen recovery technology, Flint (2005) cited that percentage of bitumen losses from mining activities was 2–3%, primary bitumen extraction 6–7%, and froth treatment 2%. The major causes of bitumen loss include instable operation, ore variation, water chemistry, mechanical reliability, and improper slurry conditioning. To improve bitumen recovery, all these factors should be addressed collectively.

1.3.3 Bitumen Froth Quality

Ideally the solids in the slurry do not report to bitumen froth because of the thermodynamic interfacial properties: hydrophilic surface of solids, hydrophobic surfaces of air bubbles and bitumen drops. But in the real extraction process water and fine solids always report to froth. The primary bitumen froth consists of bitumen (about 60%), water (about 30%) and solids (about 10%). In laboratory–scale tests, the bitumen froth quality strongly depends on the specific variables used in the extraction step.

Froth quality in terms of bitumen to solids mass ratio (*B/S*) deteriorates possibly due to the entrainment and entrapment of solids in water and bitumen droplets, therein being carried to froth (Masliyah, 2006). Slime coating of bitumen droplets is also responsible for low bitumen froth quality and bitumen recovery (Kasongo, et al., 2000). In the oil sands slurry, montmorillonite clays in the presence of high concentration of divalent metal ions initiate a strong attraction between the bitumen and montmorillonite particles, leading to coating of bitumen by clay particles. This would prevent bitumen-bitumen coalescence and bitumen-air attachment (Zhou, et al., 1999; Kasongo, et al., 2000; Masliyah, 2006). Revealed by zeta potential distribution and AFM measurements, fine solids from poor processing ores tend to coagulate on bitumen surface (Liu, et al., 2004a). Such a hetero-coagulation between bitumen and fines renders the bitumen surface less hydrophobic, thereby causing two adverse effects: low bitumen aeration efficiency and high fines reporting to bitumen froth, leading to a poor bitumen froth quality.

Solids might be able to attach to air bubbles and bitumen partially because the surfaces of some fine solids could become hydrophobic due to adsorption of surfactants (Shaw, et al., 1994; Masliyah, et al., 2004). If the surfaces of fines have an affinity with hydrophobic bitumen and air bubbles, the fines can competitively adsorb to air bubbles and bitumen drops. This also causes deterioration of bitumen froth quality.

1.4 Objectives

Oil sands slurry conditioning is a key step to bitumen recovery from oil sands using water-based extraction technology. But slurry conditioning is not understood well (Friesen and Dabros, 2004a). In industrial oil sands operations, initially the slurry conditioning was designed to be accomplished in a tumbler. The slurry conditioning starts when crushed oil sands lumps are mixed with hot water and steam to prepare the slurry. Since the hydrotransport technology was introduced to oil sands surface mining operations, the conditioning process has been designed to be accomplished in slurry hydrotransport pipelines. In laboratories, research on oil sands extraction has been carried out by using BEU (batch extraction unit) (Schramm and Smith, 1989; Shaw, et al., 1996; Schramm, et al., 2003), LHES (laboratory hydrotransport extraction system) (Wallwork, et al., 2004), laboratory pipelines (Sanders, et al., 2007), glass jars (Flynn, et al., 2001), glass cells (Friesen, et al., 2004b) and pilot facility (Malysa, et al., 1999). In these studies, effects of operating variables such as temperature, slurry flow velocity, air injection rate, and chemical addition on processability of oil sands ores were investigated. Since the presence of air during slurry conditioning is important to bitumen recovery (Flynn, et al., 2001), one question would be what is the effect on bitumen recovery if the air is prevented from entering into the slurry during the conditioning stage?

The main objective of this thesis was to investigate and document the effect of slurry conditioning on bitumen recovery of oil sands ores from Athabasca deposits. Warm water extraction process was used to extract bitumen from these samples. The first goal of this project was to detect the effect of major conditioning variables on bitumen recovery and bitumen froth quality of ores tested with the LHES, and then to understand their effect on bitumen liberation and aeration through the analysis of information obtained by visualization. The second goal of the research was to investigate the effect of conditioning using Denver flotation cell on bitumen recovery and bitumen froth quality. Three types of oil sands ores, covering a wide range of processability from good to transition and to poor processing ores, were used in this study. The specific objectives of this thesis are as follows.

Tests using the LHES The effect of slurry conditioning time, temperature, slurry flow velocity, air injection rate, and slurry density on bitumen recovery and bitumen froth quality was studied using the LHES loop. Three types of Athabasca oil sands ores were used. This study aimed at a better understanding of the bitumen liberation and aeration in oil sands flotation conducted in a laboratory-scale extraction loop.

Tests using a Denver flotation cell Oil sands flotation tests were carried out in a Denver flotation cell at different conditions. The effect of operating variables such as slurry conditioning time, temperature, slurry turbulence, and air injection rate on bitumen recovery and bitumen froth quality was investigated using a good and a poor processing ores. The results were compared with those obtained using the LHES.

1.5 Thesis Structures

Chapter one provides a brief introduction to Alberta oil sands, bitumen recovery methods, bitumen extraction, slurry conditioning, the objectives, and the organization of the thesis.

Chapter two reviews the literature on oil sands slurry conditioning, laboratory extraction systems, and the effect of temperature, air injection, time and slurry turbulence applied to the conditioning step on bitumen extraction.

Chapter three establishes test procedures for conditioning experiments to be conducted on the LHES. The experimental results and discussions are presented to illustrate the effect of temperature, air injection, slurry conditioning time, and slurry flow velocity on bitumen recovery and bitumen froth quality.

Chapter four describes slurry conditioning tests conducted in a modified Denver flotation cell. The effect of conditioning variables on bitumen recovery and bitumen froth quality is analyzed and documented.

Chapter five summarizes the conclusions of present study.

Chapter six provides some recommendations for future work.

Chapter seven lists the literature.

2. Literature Review

Kasperski (2001) and Masliyah, et al. (2004) reviewed the broad aspects of knowledge on bitumen extraction from Athabasca oil sands ores using water-based, surface mining methods. Many factors have influences on bitumen liberation, aeration, and flotation during extraction. They include water chemistry (pH, ions, surfactants), ore processability (solid wettability, oxidation, dehydration, and content of water, fines, clays and bitumen), and operating conditions (temperature, slurry density, air addition, shear strength and slurrying equipment) (Sanford and Seyer, 1979; Sanford, 1983; Bichard, 1987; Kasongo, et al., 2000; Schramm, et al., 2003; Ding, 2006). The importance of slurry conditioning has been emphasized. The complexity of slurry conditioning is also recognized. In this chapter, a literature review on the aspects related to oil sands slurry conditioning will be given, including laboratory extraction equipment and roles of temperature, air addition, residence time and slurry turbulence.

2.1 Laboratory Testing Equipment

Over the last three decades, laboratory scale extraction systems of the CHWE process have been evolved from mineral processing units (Grant, 1977; Sanford, 1979; Sanford and Seyer, 1979; Mckay, 1980; Luthra, 2001; Wallwork, 2003; Friesen, et al., 2004b). BEU (Batch Extraction Unit) was developed to study the continuous commercial process of bitumen liberation from sand grains, bitumen aeration and bitumen flotation (Sanford and Seyer, 1979). It has been widely used in bitumen extraction studies at laboratory bench scale. Beaker or jar tests were also used to study oil sands processability (Bichard, 1987; Flynn, et al., 2001; Jha, 2007). In order to simulate the pipeline hydrotransport process, the LHES (laboratory hydrotransport extraction system) was developed (Wallwork, 2003). Friesen, et al. (2004b) established a method for oil sands conditioning study using a small glass cell and video. These types of equipment and extraction testing methods have their own advantages. In the present study, BEU (or Denver flotation cell) and LHES devices were used and hence reviewed below. The choice was based on their advantages and availability in our laboratory.

2.1.1 Denver Flotation Cell

A modified bench-scale Denver flotation cell has been employed for studying parameters affecting bitumen extraction at laboratory scale (Kasongo, et al., 2000; Zhou, et al., 2004b; Jha, 2007). The schematic of a laboratory Denver flotation cell is shown in Figure 2.1 (Jha, 2007). The 1-liter Denver flotation cell has a jacketed wall connected to a temperature-control water bath through a pair of tubes. This enables the slurry temperature to be kept constant at a desired value during the flotation test. The slurry in the cell is stirred using an impeller at a given speed (rpm). The stirring speed can be adjusted to vary slurry shear strength. During bitumen flotation, air at a controlled rate (mL/min) is added into the lower section of slurry through the center stand pipe hosting the impeller shaft. Sanford and Seyer (1979) claimed that highly reproducible bitumen recovery data (~ ± 2%) could be obtained using the BEU, which is a modified version of Denver flotation cell. The reproducibility of bitumen recovery in these tests was typically better than 98% (Masliyah, 2006). The reproducibility of bitumen recovery and froth compositions was determined by a standard BEU procedure (Sanford and Seyer, 1979) to extract a rich ore (Schramm, et al., 1998; Stasiuk and Schramm, 2001). The variability of primary bitumen recovery was ± 5% absolute (Schramm, et al., 2003). Ding (2006) mentioned that a modified Denver flotation cell could detect the effect of operating parameters on bitumen recovery at a higher sensitivity than BEU tests at temperatures below 50 °C. The errors were just within 4% absolute.

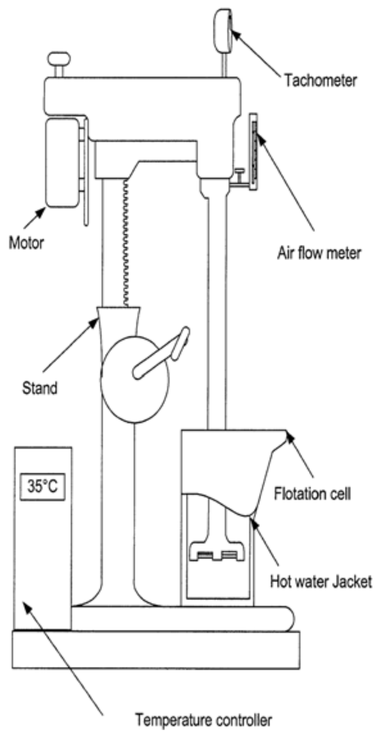


Figure 2.1 Schematic of a laboratory Denver flotation cell (Kasongo, 2000).

2.1.2 Laboratory Hydrotransport Extraction System (LHES)

To simulate the hydrotransport pipelines used in low energy bitumen extraction, a LHES was developed at the University of Alberta. It has been employed to assess oil sands processability at laboratory scales (Wallwork, 2003; Li, et al., 2005). The schematic configuration of LHES is shown in Figure 2.2 (Wallwork, 2003). Initially the loop was made of a 3-meter long glass pipe with a 17 mm inner diameter. The flotation/loading container was in the shape of a wedge end as shown in Figure 2.2. The slurry is circulated inside the loop by a progressive cavity pump. The slurry temperature was controlled to a desired value by a thermal circulating bath and a double-pipe heat exchanger. The unit incorporated a bubble visualization tube (Luba tube) inside the flotation/loading container. The Luba tube and a CCD video camera (L) allowed the investigation of bubble loading and bitumen aeration during the oil sands flotation. A second video camera (J) was used to record the view of flowing slurry in the pipe for studying bitumen liberation kinetics. Bitumen recovery and bitumen froth quality data were obtained by analyzing the bitumen froth collected from the container (Wallwork, 2003).

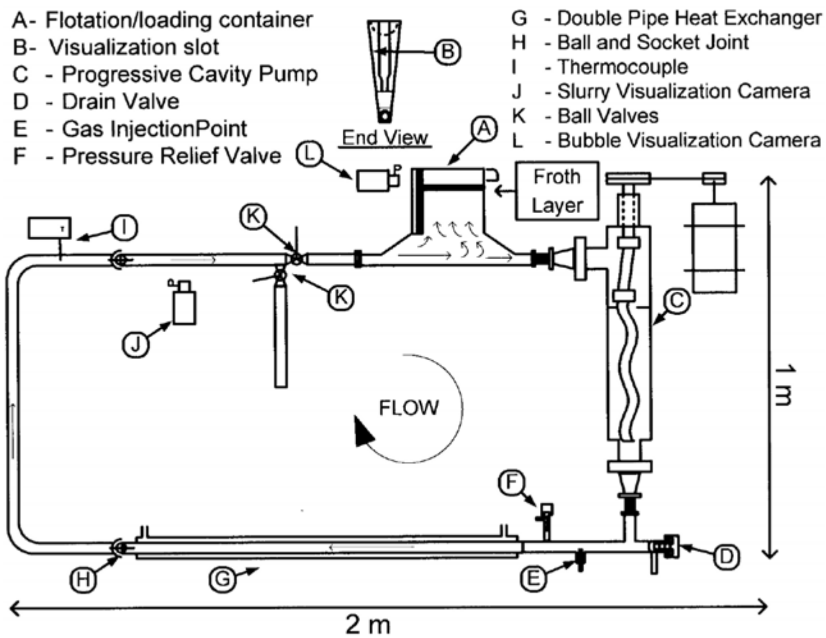


Figure 2.2 Schematic of the laboratory hydrotransport extraction system (LHES) (Wallwork, 2003).

Wallwork, et al. (2004) pointed out that this equipment was quite different from commercial hydrotransport pipelines used for oil sands conditioning. In extraction tests conducted on the LHES, a) all water was added at the beginning and no flood water was added after the conditioning step; b) air was continuously injected into the slurry and escaped from the slurry through the top of the flotation/loading container, thereby the ratio of air to slurry by volume remained relatively constant; c) aerated bitumen was continuously separated from the slurry through the loading container and collected; d) slurry was continuously circulated in the loop rather than flowed once through the pipeline.

The above characteristics of LHES allowed us to study the effect of operating parameters such as temperature, air addition, chemical addition, density and slurry flow velocity on the processability of various types of ores. It offered new analytical advantages to better understand oil sands processing characteristics (Wallwork, et al., 2004). After its initial tests to establish the LHES viability, some changes were made to the LHES. The loop then consisted of a steel pipe having an inner diameter of 25.4 mm except for the part on which the slurry visualization camera was installed to take videos. The flotation/loading container was changed to a cuboid shape with a rectangle end view. The arrangement of the bubble visualization system was also changed to enhance the quality of videos captured (Li, et al., 2005). In extraction tests, the Luba tube was inserted into the slurry at a slight angle rather than vertical, and process water was pumped into the Luba tube from the top end at a well controlled slow flow rate.

2.2 Slurry Conditioning

Slurry conditioning, or mixing of ore, water, and/or air prior to bitumen recovery in a primary separation vessel (PSV), is an essential step in the extraction of bitumen from oil sands. The purpose is to liberate bitumen from the oil sands matrix and then to allow coalescence of bitumen drops and attachment of liberated bitumen to air bubbles so that large, aerated drops can be recovered easily (Shaw, et al., 1996; Friesen, et al., 2004b). The conditioning process conceptually includes the steps of lump size reduction, bitumen liberation and air attachment (Figure 1.3 A to H). The liberation step starts with the formation of a three-phase contact point, followed by the bitumen recession along a sand grain surface and completed by the bitumen disengagement from the sand grain. The liberation step is controlled by bitumen viscosity and interfacial properties that are influenced by temperature, the presence of surfactants and clays, and water chemistry of the slurry. The liberated bitumen droplets need to attach to air bubbles. Air attachment takes place when bitumen droplets and air bubbles contact with each other. The aeration of bitumen is controlled by the wettability and interfacial properties of bitumen and air in water. The interfacial properties are affected by fine solids and/or clays attached to the surfaces of bitumen and/or air bubbles. The type and concentrations of fine solids, clays, electrolytes and pH values of the slurry all contribute to determining aeration efficiency. It is quite likely that bitumen liberation from the outer layer of a bitumen lump takes place simultaneously with the already sheared bitumen-sand layers. Air entrainment or engulfing can assist bitumen liberation (Masliyah, 2006). During the conditioning, the degree of shear in the slurry plays an important role for all stages from lump size reduction to aeration of bitumen.

Traditionally the conditioning is completed in a tumbler. Recently, oil sands hydrotransport (Maciejewski, et al., 1993) as an important technological advance has been employed in commercial operations of water-based bitumen extraction from Athabasca oil sands. The flow diagram of ore preparation and slurry conditioning using a hydrotransport pipeline is shown in Figure 2.3 (Masliyah, et al., 2004). The slurry conditioning of oil sands occurring in the pipeline remained poorly understood (Söderbergh, 2005). In either case, it is expected that conditioning is sufficient to ensure good bitumen recovery in PSV. Well-conditioned slurry yields high bitumen recovery in the flotation step.

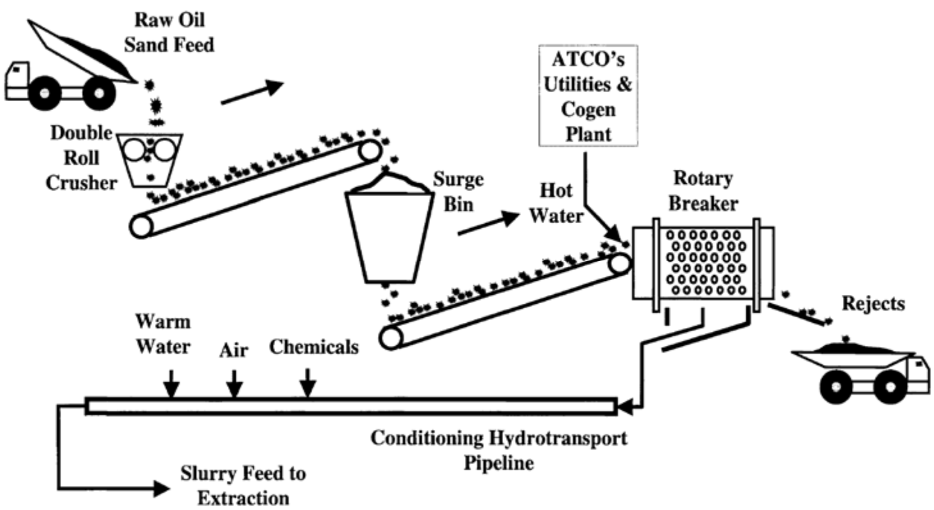


Figure 2.3 Ore preparation and slurry conditioning (cited by Masliyah, et al., 2004).

The replacement of high temperature tumbler conditioning with low temperature hydrotransport pipeline conditioning makes the bitumen recovery and froth quality more sensitive to operating variables and ore characteristics including clay content, water chemistry, aeration, bitumen drop sizes, etc. For conditioning at low temperatures, the slurry conditioning time becomes very important since it can impact both the bitumen recovery and bitumen froth quality. Insufficient conditioning would result in both poor froth quality and poor recovery (Wang and Mikula, 2002). Over conditioning of slurry would cause the formation of too large bitumen drops thereby decrease their aeration efficiency (Friesen, et al., 2004b).

2.3 Effect of Temperature

Much attention has been paid to the possibility to reduce the slurry temperatures in oil sands surface mining operations since its first commercialization in 1960s. Initially a temperature higher than 70 °C was employed. Subsequently, in order to reduce energy intensity (cost) and to decrease emission of greenhouse gases, the industry has been shifted towards operating at a lower temperature and a caustic-free process. Since around 2000, Syncrude and Suncor have adapted lower temperature process in their new operations. Both Steepbank and North Mines operate at about 50 °C. And Syncrude's Aurora Mine initially used a temperature as low as 25–30 °C but then increased to 35 °C to ensure a robust and reliable operation of bitumen recovery. Albian Sands Muskeg River Mine operates at 40–45 °C. The plant experience and laboratory tests have shown that the operating temperature of extraction plays a significant role in bitumen separation. It has strong effect on bitumen liberation, bitumen aeration and bitumen flotation, therein on bitumen recovery and bitumen froth quality (Long, et al., 2007). To choose a proper operating temperature depends on many factors such as oil sand lumps residence time, availability of excess energy from upgrading facilities, water recycling and so on (FTFC, 1995; Masliyah, 2006).

Studies on bitumen liberation showed that the rate of bitumen liberation increased significantly with increasing operating temperatures (Luthra, 2001; Wallwork, 2003). Luthra (2001) found that bitumen liberated at a much faster rate at 50 ºC (the rate constant *k* was 68.8 min^{-1} for a good processing ore and 12.4 min^{-1} for a poor processing ore) than at 35 ºC (*k* was 10.3 min^{-1} for the same good processing ore in comparison to ~0.7 min^{-1} for the same poor processing ore). Measurement of contact angle of bitumen droplets (Basu, et al., 1996) and bitumen recession from a model silica glass surface (Masliyah, et al., 2004) indicated that high temperatures could enhance bitumen liberation.

The overall bitumen recovery rate can be expressed by a first-order kinetics equation as follows

$$R_t = R_\infty [1 - \exp(-kt)] \tag{2-1}$$

where R_t and R_∞ are the bitumen recovery at time t and the ultimate bitumen recovery, respectively; and k is the bitumen recovery rate constant (min^{-1}).

Other studies also showed that the rate of bitumen recovery from oil sands ores was enhanced at higher temperatures (Zhou, et al., 2004a). For a good processing ore, the recovery rate constant increased from 0.15 min^{-1} at 25 ºC to 0.98 min^{-1} at 50 ºC. For the poor processing ore, the rate constant increased to 0.383 min^{-1} at 50 ºC from 0.084 min^{-1} at 25 ºC. These studies indicated that the rate constants of bitumen recovery from oil sands were much lower at extraction temperatures below 35 ºC (Long, et al., 2007).

At temperatures higher than 50 ºC bitumen recovery was not significantly affected by operating temperatures (Bichard, 1987; Schramm, et al., 2003; Stasiuk, et al., 2004). However, a sharp decrease in bitumen recovery was observed, particularly at temperatures lower than 35 ºC (Long, et al., 2007). Temperatures in the range of 25–50 °C also had a strong effect on bitumen recovery and bitumen froth quality (Bichard, 1987; Ding, 2006; Schramm, et al., 2003). Bichard (1987) reported that for a poor processing oil sands ore bitumen recovery was reduced from about 90% at 37.8 °C to about 40% at 26.7 °C. Ding (2004) reported that for a good processing ore the bitumen recovery dropped from about 88% at 35 °C to about 30% at 25 °C. In the study of Schramm, et al. (2002), the primary bitumen recovery was found to reduce from 88% at 50 °C to 8% at 25 °C for a transition ore. These results imply a critical temperature range within which bitumen recovery increased dramatically with increasing temperatures. Actually Long, et al. (2007) plotted out a critical temperature range from 25 ºC to 35 °C as shown in Figure 2.4.

Over this critical temperature range the viscosity of bitumen was observed to increase sharply with decreasing temperature (Hepler, et al., 1994). Thus, viscosity was considered as a main contributor to the dramatic reduction in bitumen recovery when process temperature was decreased from 50 to 25 °C (Schramm, et al., 2003). Long, et al. (2005) found that the adhesion force between bitumen and fines/clay in process water decreased with increasing temperature until a critical temperature between 32 °C and 35 °C, above which the adhesion force disappeared. The depressed adhesion forces were beneficial to both bitumen liberation and aeration, which explains why bitumen recovery increases sharply with increasing temperature from 25 °C to 35 °C. From their study, Long, et al. (2005) suggested that bitumen extraction should be operated at a temperature above the critical temperature range.

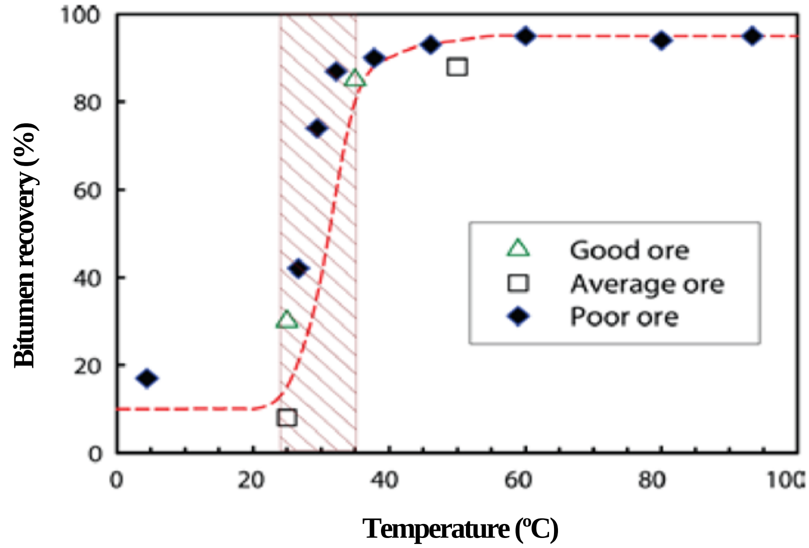


Figure 2.4 Effect of processing temperature on bitumen recovery in bench scale tests (Long, et al. 2007).

Operating temperatures also influences bitumen froth quality. Bitumen froth obtained from extraction at lower operating temperatures (10 °C ~ 30 °C) contained more solids and water than bitumen froth obtained at higher operating temperatures of about 80 °C (Lam, et al., 1995). Using different types of oil sands ores, Bichard (1987) and Hupka, et al. (1987) tested bitumen extraction at different temperatures. Their results showed that bitumen froth quality deteriorated at reduced operating temperatures. The results from Bichard's study (1987), shown in Figure 2.5, indicated a similar critical temperature range from about 45 °C to 55 °C to ensure the quality of bitumen froth obtained from a poor processing ore. The bitumen froth quality was better at temperatures higher than 55 °C than that at temperatures lower than 45 °C. A model study showed that the increased solids content in froth obtained at low extraction temperatures might be a result of the increase in the viscosity of bitumen at low temperatures (Masliyah, 2006). At low temperatures, the extent of air bubble engulfment by bitumen was reduced and the adhesion of bitumen to silica particles was high (Kasperski, 2001).

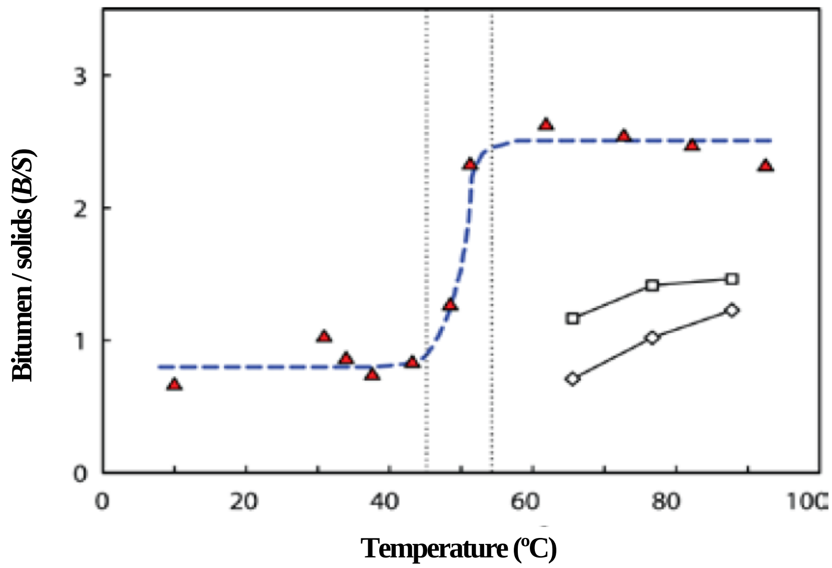


Figure 2.5 Effect of temperature on bitumen froth quality measured by bitumen to solids mass ratio (*B/S*) (Bichard, 1987). The filled triangles represent a poor processing ore.

2.4 Effect of Air Injection

To achieve high bitumen recovery, availability of air during conditioning and flotation is very important (Flynn, et al., 2001; Friesen, et al., 2004b; Wallwork, et al., 2004; Sanders, et al., 2007). Studies have shown that at low (<50 °C) process temperatures air injection could improve bitumen recovery (Luthra, 2001; Mankowski, et al., 1999; Wallwork, 2003). Sources of air for aeration could be air trapped in the ore (indigenous air), dissolved in the process water, entrained into slurry during slurrying, or direct addition to the slurry in a controlled way. Many claims on the need for air were made: i) the dissolved air in process water was sufficient for bitumen flotation (Mankowski, et al., 1999); ii) indigenous air was the smallest contributor; iii) excess air must be introduced into the slurry (Flynn, et al., 2001); and iv) entrained air during slurrying was required. However no agreements were reached on the effect of such factors as the amount of air required, source of air available in the slurry, timing and modulus of air injection and size of air bubbles on bitumen flotation (Kasperski, 2001; Friesen, et al., 2004b).

In jar tests with a limited air to slurry ratio, Flynn, et al. (2001) found that the indigenous air contributed only very small part to bitumen recovery. Excess air must be added into the slurry through either slurrying or direct addition. This was in line with the studies conducted in loops (Wallwork, 2003; Sanders, et al. 2007) and in Denver flotation cell (Zhou, et al., 2004a; Masliyah, 2006). Wallwork (2003) and Zhou, et al. (2004a) found that the bitumen recovery improved with the addition of air. In the tests with a loop (Wallwork, 2003), the bitumen recovery increased with increasing the amount of air injected and flotation time (Figure 2.6). Sanders, et al. (2007) found that the indigenous and incidental air injection of about 2% (by volume) yielded very poorly conditioned slurry, and 5% (by volume) of extra air injection resulted in a well-conditioned slurry. The favorable amount of air in the LHES was found to be around 1: 200 (less than 1% by volume) rather than 5% found in a 100-mm loop (Sanders, et al., 2007).

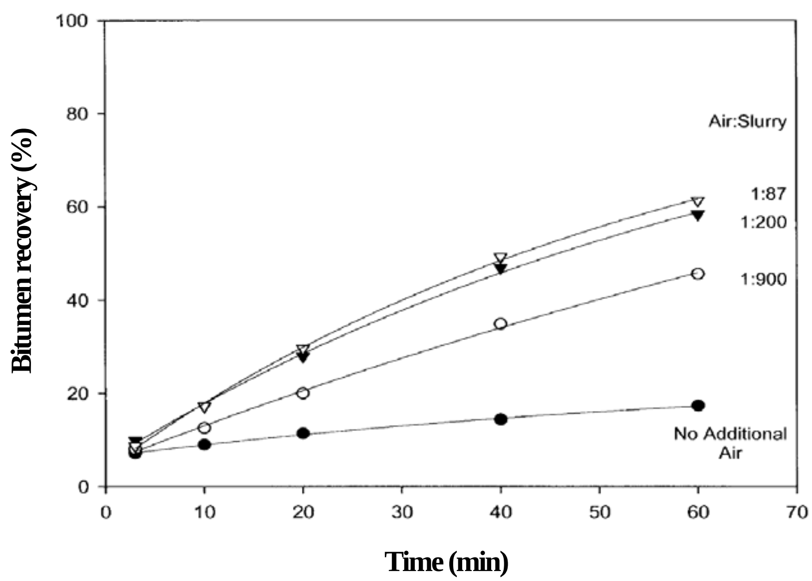


Figure 2.6 Effect of air injection on bitumen recovery of high fines ore, extracted at 35 °C using the LHES loop (Wallwork, et al., 2004).

Flynn, et al. (2001) also claimed that the timing of air injection was not important. To the contrary, Friesen, et al (2004b) demonstrated that the timing of air injection was crucial as stated: “if the bitumen drops are allowed to become too large before air is injected, the efficiency of air-bitumen attachment would be too low for good aeration to occur". The latter claim was supported by the slurry conditioning tests with a 100-mm pipeline loop (Sanders, et al., 2007). The preliminary results indicated that a sufficient amount of air must be available early in the conditioning process.

Bubble size also affects bitumen-bubble attachment. However the effect of bubble size on bitumen aeration is less conclusive. It was claimed that bitumen droplets tended to attach to air bubbles that were roughly the same diameter as the droplets themselves (Malysa, et al., 1999). Higher bitumen recovery could be possible if the size of air bubbles matched the size of bitumen droplets (Wang and Mikula, 2002). Others believed that the smaller the bubbles, the better chance the aeration of bitumen droplets (Clark, 1944; Gu, et al., 2004). Clark (1944) reported that small bubbles attached to bitumen more effectively than large bubbles (in millimeter size range). This observation was justified by numerous theoretical analysis and experimental measurements in minerals flotation systems (Yoon and Luttrell, 1989; Yoon, 1993; Dai, et al., 2000). Gu, et al. (2004) examined the attachment of hydrogen bubbles (0.01 mm to 0.1 mm in diameter) to a bitumen droplet of 1.5 mm in diameter in deaerated tap water. They found that the smaller the bubble sizes, the shorter the induction time for the attachment. When bitumen drop was bigger than 0.3 mm, no attachment was observed in their study.

Bichard (1987) demonstrated that the manner by which the air was added to the process affected bitumen extraction. Wallwork, et al. (2004) and Sanders, et al. (2004) suggested that air be injected into the slurry at different locations along the pipeline rather than at a single point. Such an aeration scheme might be able to improve bitumen recovery. In commercial operations where conditioning was completed in a tumbler or in laboratory research where extraction was carried out in a vessel open to the ambient, the source and the amount of air was not under quantitative control. This is because indigenous air existed in the ore and process water, and air was entrained into the slurry by turbulent slurrying. For example, Friesen, et al (2004b) added no air intentionally in the extraction tests. But sufficient air was entrained into slurry with a reasonable level of stirring in the glass cell.

A question remains on the effect of conditioning variables on bitumen recovery if no air is added during the conditioning step but followed by injection of air into the slurry. Flynn, et al. (2001) reported that it was not important if no air was available at earlier stage of conditioning as long as excess air was added later on. However, Wang, et al. (2002) and Sanders, et al. (2007) showed that if air was not introduced into the slurry during conditioning, many large bitumen droplets would form in the slurry. It implies that the absence of air during conditioning might lead to formation of large bitumen drops and thereby low bitumen recovery.

2.5 Effect of Conditioning Time

Extraction tests have shown that the bitumen liberation, aeration and overall recovery are a function of extraction time. Basically, the longer the conditioning time, the more completed bitumen liberation from sand grains, the more bitumen aerated with air bubbles, and the higher overall bitumen recovery (Basu, et al., 1996; Luthra, 2001; Wallwork, 2003; Masliyah, et al., 2004). In the tests conducted on the LHES, Wallwork (2003) obtained results as shown in Figure 2.7. The degrees of bitumen liberation increased with time until the liberation was completed. The degree of bitumen liberation from sand grains was a function of the slurry conditioning time and temperature. When the conditioning stage lasted 60 minutes, the liberation was nearly identical for all the temperatures and ore types employed.

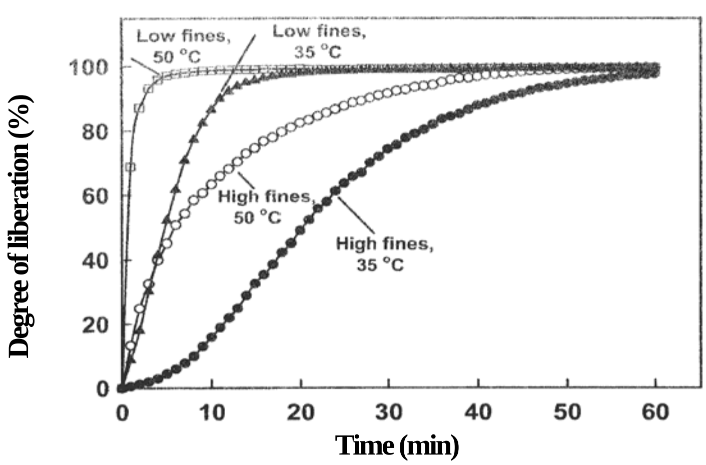


Figure 2.7 Effect of temperature and fines content on bitumen liberation from sand grains (Wallwork,

et al., 2004).

Similar trends for bitumen recovery (see Figure 2.8) were also found by tests conducted in the LHES (Wallwork, 2003) and in Denver flotation cell (Zhou, et al., 2004a).

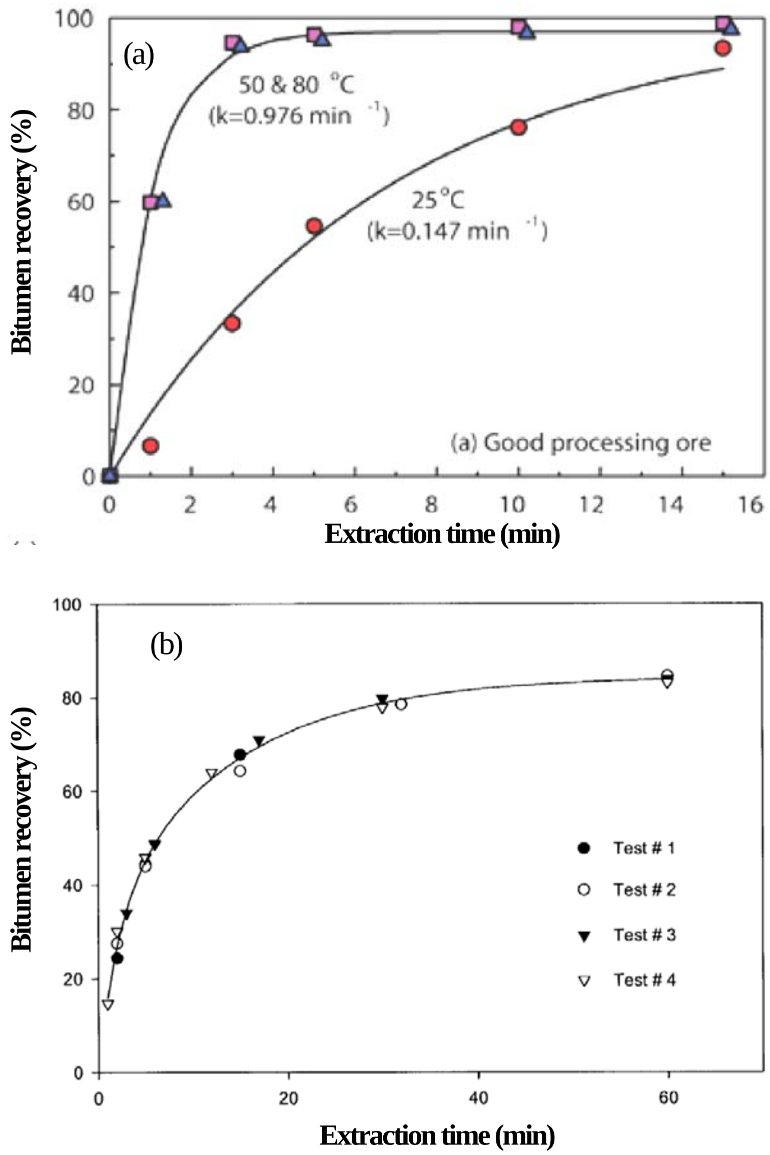


Figure 2.8 Bitumen recovery as a function of extraction time at 50 °C, using (a) a Denver flotation cell (Zhou, et al., 2004b) and (b) a LHES loop (Wallwork, et al., 2004).

These aforementioned tests with bitumen liberation and recovery were conducted at respective specific slurry conditioning time. In these cases, the results could be understood from their flotation kinetics. The longer the flotation time is, the more complete the bitumen liberation and the higher the bitumen recovery is.

Since bitumen recovery is known to be a function of many factors such as slurry shear rates, slurry conditioning time, temperature, ore processability, water chemistry, air bubble size and volume fraction, etc., extensive research effort has been devoted to studying a proper slurry conditioning time. Schramm and Smith (1989) varied the slurrying time from 1 to 15 min rather than the standard 10 min as used in the BEU tests. In their study they used a flotation time (no air injection) of 10 min. At both test temperatures of 82 °C and 55 °C, an optimal primary bitumen recovery was achieved at a slurrying time of about 2–3 min if tested at 82 °C or 10 min if tested at 55 °C. Longer conditioning time than the optimal slurrying time led to a decrease in bitumen recovery (Schramm and Smith, 1989). Wang, et al. (2002) claimed that 20 minutes of slurry conditioning time was sufficient for bitumen liberation. Extending the slurry conditioning time to 30 min did not affect the amount of froth collected.

The studies using the LHES (Wallwork, 2003) and glass cells (Friesen, et al., 2004b) showed that the flotation started with the conditioning. A zero slurry conditioning time, therefore, was tested in a few studies. Friesen, et al., (2004b) showed that the bitumen recovery of a good processing ore at 50 °C increased first, and then dropped sharply with slurry conditioning time after 2 min. This could be an indication of over conditioning of this ore, due to the lack of sufficient air for flotation. Microphotographs of the test showed a layer of unaerated bitumen droplets in size of up to 500 µm. This phenomenon was also observed in oil sands conditioning tests conducted in a 100-mm slurry hydrotransport loop (Sanders, et al., 2007). Without air addition to the slurry during conditioning, a layer of unaerated bitumen droplets formed in the sampled slurry. Lack of air and long conditioning time caused some solids to attach to the surfaces of those unaerated bitumen droplets (Dai and Chung, 1996).

The above cited literature review would indicate that to some degree the concept of slurry conditioning time in oil sands extraction is loosely defined and can be confusing to readers. For clarification, the following time terms are defined and used in the present study:

Conditioning time refers to the period of time during which no air is injected into the slurry while being circulated inside the loop or stirred in the Denver flotation cell.

Flotation time refers to the period of time during which air is injected continuously into the slurry while it is being circulated inside the loop or stirred in the Denver flotation cell. This flotation time is in addition to the conditioning time defined above.

Extraction time refers to the entire period of time of a test during which the slurry is being circulated in the loop or stirred in the Denver flotation cell. The ***conditioning time***, the ***flotation time*** and the ***extraction time*** defined herein are schematically shown in Figure 2.9.

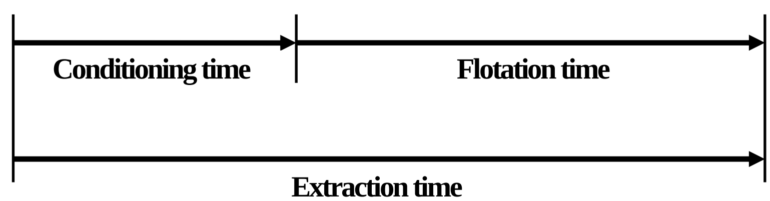


Figure 2.9 Definitions of conditioning time, flotation time and extraction time used in this study.

The extraction time, therefore, equals to the slurry conditioning time plus the flotation time. For example, in a test carried out in the loop, the slurry is conditioned for 10 minutes (i.e., at the beginning of the test, no air is injected into the slurry for a duration of 10 minutes) and followed by air injection into the slurry for 30 minutes while bitumen froth is collected. In this case, the slurry conditioning time is 10 minutes, the flotation time is 30 minutes and the extraction time is 40 minutes (i.e., 10 + 30 = 40 minutes).

2.6 Effect of Shear Rate

The fundamental steps involved in oil sands extraction suggest that higher shear rate of slurry be beneficial to bitumen liberation and aeration due to stronger mechanical energy input and thus higher energy dissipation rate. The average energy dissipation rate equations for turbulent flows given by Hesketh, et al. (1987) for a pipe and by Tsouris and Tavlarides (1994) for a vessel show that a higher slurry flow velocity in a loop or a higher stirring speed in a vessel produces a stronger shear rate and higher energy dissipation rates.

Simulation showed that the size of air bubbles in dense oil sands slurry became smaller with increasing slurry flow velocities (Eskin, et al., 2004). For relatively large bubbles (200 μm) the breakage rate increased faster than the coalescence rate with increasing slurry flow velocity, while for relatively small bubbles both the coalescence and the breakage processes are intensified approximately at the same rate. The larger breakage than coalescence led to an increase in the population of smaller bubbles. This finding implies that stronger shear rates would produce smaller air bubbles and hence enhanced aeration of bitumen droplets. Researchers claimed that smaller air bubbles were in favor of higher efficiency of aeration. This was justified by Sanders, et al. (2004).

On the other hand, the bitumen droplets also became smaller with increasing shear rates in slurry. Friesen and Dabros (2004a) found that if air was not present (not injected nor entrained sufficiently in the ores) during the conditioning, the size distribution of unaerated bitumen drops became narrower and peaked at smaller values as turbulence increased (Figure 2.10). In Denver flotation cell tests, Wang, et al. (2002) found that stirring speeds at 200 and 400 rpm did not produce sufficient agitation of the slurry. Most of the bitumen did not detach from the clay and a bitumen-rich layer formed in the tailings. A stirring speed of 800 rpm was selected to obtain sufficient bitumen release (Wang, et al., 2002). In the experiments carried out in a 25.4-mm loop, Razzaque, et al. (2003) revealed that higher slurry flow velocities produced smaller air bubbles with a narrower size distribution than lower slurry flow velocities due to the stronger shear rates at higher slurry flow velocities. The authors explained that mechanic energy, ε, was a strong function of the slurry flow velocity. Moran, et al. (2000) claimed that larger droplets were in favor of their aeration. It should be pointed out that in the above studies the interactions between fine solids and bubbles (or bitumen droplets) were not taken into consideration.

When clays and surfactants are present the situation can become more complex. It was found that clays could attach to the surfaces of bitumen droplets and air bubbles (Liu, et al., 2004a). The oil sands slurry might become over conditioned and then large bitumen-sand agglomerates may form (Friesen, et al., 2004b).

Since the slurry conditioning step is very important to bitumen extraction from oil sands ores and poorly understood, the main objective of this study was to detect the effect of major conditioning variables on bitumen recovery and bitumen froth quality of ores, tested with both the LHES and the Denver flotation cell.

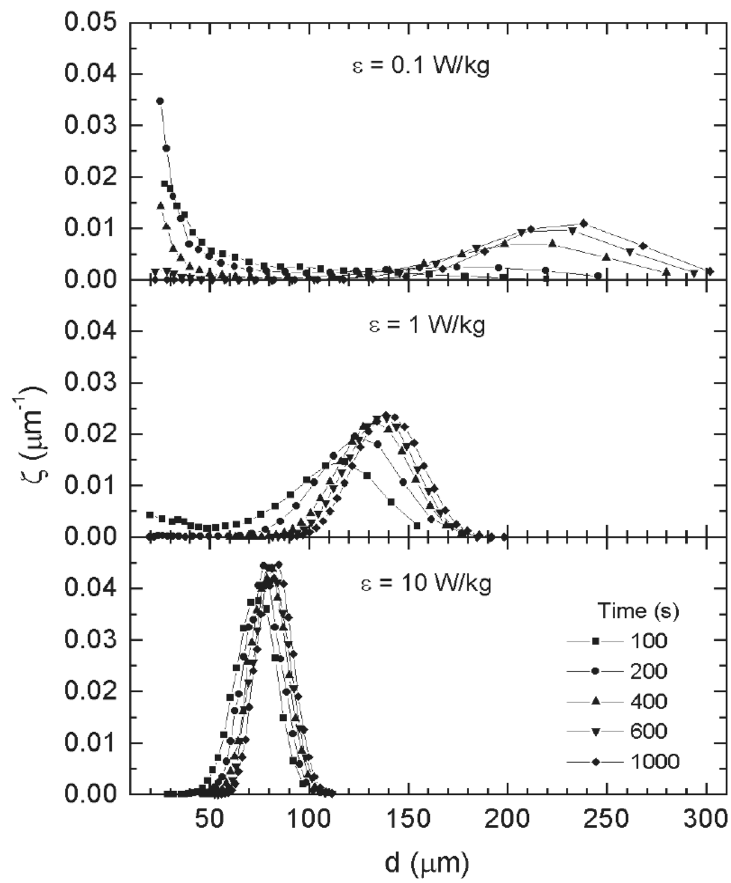


Figure 2.10 Size distribution of unaerated bitumen drops depending on the energy dissipation rate and slurrying time prior to aeration, where ζ is volumetric fraction of drops with specific diameter d (μm^{-1}), ε the average turbulence strength (W/ kg) (Friesen and Dabros, 2004a).

3. Tests with Laboratory Hydrotransport Extraction System

3.1 Introduction

The laboratory hydrotransport extraction system (LHES) was designed and constructed to investigate the process occurring in hydrotransport pipelines employed in commercial warm water extraction operations. It was equipped with video systems that allow the visualization on brightness of the bitumen slurry (to estimate liberation rate) and visualization on rising air bubbles and aerated bitumen droplets. The LHES was configured to allow the accurate control of slurry temperature, slurry density, slurry flow velocity, air injection rate and duration, and froth collection time (Wallwork, 2003). These characteristics of the LHES are suitable for studying processability of oil sands ores and effect of operating parameters on ore processability.

The primary objective of this study was to investigate the effect of slurry conditioning in the LHES loop on bitumen recovery and froth quality. To achieve this purpose in a simple but effective way, process water from Aurora Mine of Syncrude Canada Ltd. was used without the addition of chemical aids. A batch extraction method developed by Wallwork (2003) was adapted with minor modifications. The conditioning parameters chosen for this investigation included slurry temperature, slurry density, slurry flow velocity, slurry conditioning time, and air injection rate and duration. Three different oil sands ores were used to cover a wide variety of ore processability: good processing, poor processing and transition ores. The experimental techniques, the results and discussion are presented in this chapter.

3.2 Experimental

3.2.1 Materials

Oil sands ores A good processing ore (AL711) from Albian Sands was selected and shipped in December, 2006. The transition (Syn704) and poor processing (Posyn) ores were received from Syncrude Canada Ltd. in April, 2007 and October, 2002, respectively. All three oil sands ores were shipped in 20 L plastic pails tightly sealed. Each individual ore was chopped, homogenized, bagged (600 grams per bag) and stored in a closed freezer at–29 °C in dark to minimize aging effect, as recommended by Schramm and Smith (1987a; 1987b). The content of oil sands ores was analyzed using Dean Stark method (Bulmer, et al., 1979). The bitumen, water, solids and fines content are shown in Table 3.1.

Table 3.1. Compositions (wt%) of oil sands ores used in this study.

Ores and codes	**Bitumen**	**Connate water**	**Solids**	**Fines***
Good Processing, AL711	12.3	2.7	84.7	13.4
Transition, Syn704	9.7	6.2	85.0	25.5
Poor processing, Posyn	6.1	7.0	88.0	43.0

*defined as percent solids less than 44 μm in the solids of an ore.

Process water The process water from Aurora Mine of Syncrude Canada Ltd. was used in extraction tests on the LHES. It was shipped in tightly sealed plastic pails, 20 L each in volume. The process water was analyzed with atomic absorption. It contained 23.3 ppm calcium, 15.0 ppm magnesium, 20.1 ppm potassium and 582 ppm sodium. The concentration of other anions is shown in Table 3.2. The pH value of the process water was 7.2 measured using a portable pH meter (OAKTON EUTECH Instruments, pH 110). Unless otherwise stated, all experiments were conducted using the Aurora process water.

Table 3.2. Electrolyte concentration (ppmw) in the Aurora process water at pH=7.2.

Ions	Ca^{2+}	Mg^{2+}	K^{+}	Na^{+}	Cl^{-}	NO_3^{-}	SO_4^{2-}	HCO_3^{-}
Concentrations (ppmw)	23.3	15.0	20.1	582	400	2.03	112	664

3.2.2 Experimental Set-up

As per the review in Chapter 2, the laboratory hydrotransport extraction system (LHES) allowed the simulation of and the investigation into commercial oil sands operations using slurry hydrotransport pipelines and warm or low temperature water-based bitumen extraction processes (Wallwork, 2003). The schematic of the initial LHES configuration is shown in Figure 2.2. The LHES consisted of a circulating pipeline, known as loop including a single tube heat exchanger with an attached water jacket, a progressive cavity slurry pump, a separation vessel for loading the feed and collecting the froth, and valves for sampling and cleaning. The slurry pump from the Weatherford Artificial Lift System (Sejo Leopoldo-RS, Brazil) was chosen in such a way that the average flow velocities in the pump were much less than that in the pipe section of the loop. Compared with a centrifugal pump the progressive cavity pump imported very low shear forces to the slurry. As a result, the impact of the pump itself on slurry conditioning was minimized. Any changes in the hydrodynamic conditions of the system and their effect on bitumen extraction could, therefore, be confidently attributed to the shear conditions created by the slurry turbulence in the loop rather than those created by the pump. The pump was driven by a 3 HP variable AC motor drive that allowed precise control in speed. The flow rate inside the loop was controlled by the speed of the motor drive. The temperature of slurry inside the loop was controlled by a thermal circulating water bath connected to the heat exchanger (Wallwork, 2003). For accuracy, a thermocouple was used to measure and monitor the slurry temperature. The temperature set point of thermo water bath was adjusted based on the slurry temperatures monitored. Two video camera systems were mounted to the loop for collecting information on bitumen liberation from the sand grains and bitumen aeration, respectively.

For better performance, the following four minor modifications were made to this system.

A) To improve the observation of bitumen aeration, a peristaltic pump was connected to the upper end of Luba tube (Li, et al., 2005).

B) The glass loop was replaced with steel pipe except for a 30 cm long glass section for the slurry visualization by a video camera system.

C) The loading and froth-collection container (see Figure 2.2 A), functioning as a vessel for loading oil sands and separating aerated bitumen aggregates, was replaced with a vessel composed of two cuboid-shaped cylinders, one above the other. The upper part was a transparent glass frame with a steel trough for loading oil sands and water and collecting froth. The lower part was narrower in width and connected within the loop as a section.

D) A rectangular steel plate called a flap was installed inside the lower part of the container in such a way that it allowed the lower part of the container to be closed to the upper part in conditioning step of the tests, and to be opened in air injection step of the tests for bitumen froth collection.

The modified LHES loop (see Figure B.2 in Appendix B for full details) was used in the present study.

3.2.3 Procedures

Oil Sands Analysis The content (water, bitumen, solids and fines) of each oil sands ore was analyzed by Dean Stark methods (Bulmer, et al., 1979). See Appendix A for full details of the Dean Stark method. A schematic of the Dean Stark device is shown in Figure 3.1. The analysis was based on the following fact: a) high solubility of bitumen in toluene; b) higher vapor pressure of water and toluene than bitumen; c) negligible solubility of water in toluene and bitumen; and d) complete retention of solids and fines in the thimble.

One bag of oil sands ore (600 g) was taken from the freezer and thawed for 2 hrs at room temperatures. Around 50 g of thawed oil sands were precisely weighed and placed into a pre-weighed dry thimble (Whatman) of 100 mL capacity. 200 mL of dry toluene (Aldrich) was placed in the extraction flask. The thimble containing the oil sands sample was hung inside the flask. The completely assembled device is photographically shown in Figure B.1 of Appendix B.

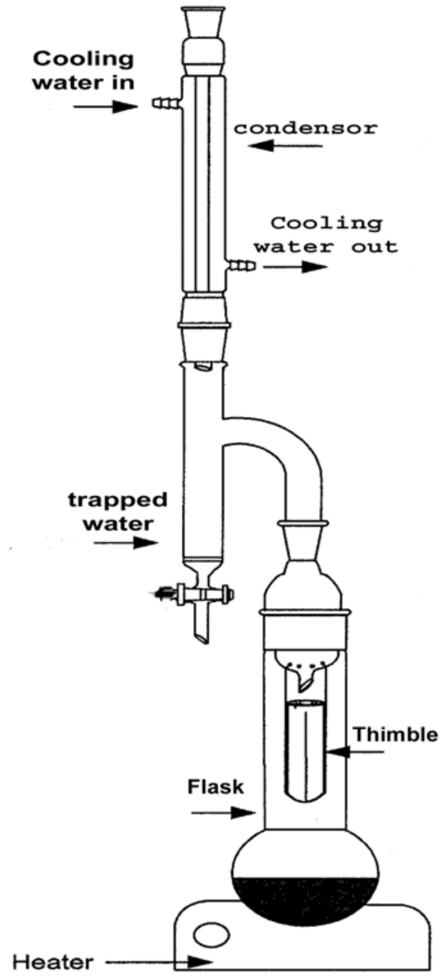


Figure 3.1 A schematic of the Dean Stark device (Bulmer, et al., 1979; Jha, 2007).

The bitumen in the sample was extracted into the refluxing toluene. Water in the sample was evaporated with toluene and then condensed and collected in the side tube. The refluxing of toluene was stopped after the toluene dripping from the thimble became colorless. The water trapped was collected and weighed to obtain the water content in the oil sands sample. The bitumen in toluene solution was cooled down at room temperature and then transferred from the extraction flask to a 250 mL volumetric flask. Additional toluene was added to 250 mL mark of the flask. After compete mixing, 50 mL of this solution was taken and centrifuged at 3000 rpm for 20 minutes to force out the solids in the solution if present to settle down. Then 5 mL of this solution was taken by a pipette and spread on a piece of pre-weighed Ø15 cm filter paper (Whatman). The solution-soaked filter paper was hung in a well vented hood to evaporate toluene in ambient air for 15 minutes. The filter paper with sampled bitumen was weighed, which allowed calculation of bitumen content in the oil sands sample, assuming negligible bitumen evaporation. The thimble together with the solids was dried in a low level vacuum oven at 105 °C overnight. The solids content was determined then. Furthermore, the solid was screened using a 45 µm sieve to separate the solids into two fractions. The -45 µm size fraction was weighed to determine the fines content in the oil sands sample. For each oil sands ore, three sub-samples were analyzed to obtain the content of each ore. The bitumen, water, solids and fines content was calculated using Equations (3-1)–(3-4), respectively.

$$Bit\% = [50 \times (\text{mass of paper with bitumen} - \text{mass of paper}) / \text{sample mass}] \times 100 \quad (3\text{-}1)$$

$$W\% = [\text{mass of water} / \text{sample mass}] \times 100 \quad (3\text{-}2)$$

$$S\% = [\,(\text{mass of thimble with solids} - \text{mass of thimble}) / \text{sample mass}] \times 100 \quad (3\text{-}3)$$

$$F\% = [\text{mass of fines} / (\text{mass of thimble with solids} - \text{mass of thimble})] \times 100 \quad (3\text{-}4)$$

Slurry Conditioning For each bitumen extraction test, unless otherwise stated, 1000 g of thawed oil sands sample was used. 5.5 L measured Aurora Mine process water was loaded into the LHES loop while being circulated. The process water was heated to and kept at 1 °C higher than the experiment temperature. The process water in the loop was pumped backwards for 5–10 seconds to drive off any air that might be accumulated on the top inside the pump. The pump drive was then switched forwards to drive the process water flow clockwise. While the process water was being heated and flowing in the loop, the two video camera systems were prepared for recording. The thimbles and jars were numbered and pre-weighed to be ready for froth collection. Through the loading-separation container, the weighed oil sands sample was loaded into the loop where it was slurried and conditioned. After the completion of sample loading (about 10 s), the flap inside the separation container was switched to the closed position, while the temperature was being adjusted to the set value for the experiment. At the same time the timing of slurry conditioning started. Note that no air was added during conditioning.

Bitumen Flotation After the oil sands slurry was conditioned for a set period of 0, 5, 10, 20, or 30 min, the flap inside the loading-separation container was switched to the open position and air was immediately introduced into the slurry through a stainless steel needle of size 180 mm X 4 mm (Fisher Scientific). The air injection rate was measured and controlled at a desired value. The air injection timing started at the very moment air was injected. The liberated bitumen droplets were aerated and the aerated bitumen aggregates floated to the top of the slurry in the separation container to form a froth layer. Froth samples were collected separately over incremental flotation time into pre-weighed 100 mL thimbles (Whatman) sitting in pre-weighed 200 mL glass jars.

Determination of Bitumen Liberation To obtain information on bitumen liberation from the sand grains online, a slurry visualization system mounted on the LHES loop was used, as shown in Figure 2.1 and Figure B.2 of Appendix B. The video system consisted of a video camera and a pair of strobe lights controlled by a computer. The high-speed black-and-white digital video camera was mounted just in front of the glass section of the loop. Two strobe lights were set up to provide sufficiently bright light for the camera to capture high quality images of the flowing oil sands slurry inside the loop. During each test starting from the slurry conditioning, 15 frames of images per second were acquired and digitalized online into grayscale bitmap images at a resolution of 640 by 480 pixels. The grayscale for each pixel was evaluated by in-house developed program on a desktop computer. The gray level (or brightness) of each pixel was scaled from 0 to 255. It was assumed that a pixel with a gray level higher than a threshold value was considered as an oil sands grain that bitumen did not liberate. The computer compared the gray levels of the pixels with a set threshold value (assumed at 100) and summed the number of pixels that had grayscale below the threshold value into an excel file. This quantity could be used as a measure of the number of bitumen-covered oil sands grains. Based on its initial value (N_0) and the value (N) at time t, the degree of bitumen liberation, $L\%$, was evaluated by Equation (3-5) (Wallwork, 2003).

$$L\% = 100 \times (N_0 - N) / N_0 \qquad (3\text{-}5)$$

where N_0 is designated as the number of black pixels at time t_0 and N the number of black pixels at a given time *t*. The bitumen liberation rate could be obtained as a function of extraction time.

When the recorded data were processed to obtain the degree of bitumen liberation, the pixels of 900 images were averaged to obtain a statistically representative *L*% value. It should be noted that this approach remains semi-quantitative as it has not been proven from vigorous cross testing.

Visualization of Bitumen Aeration To determine the bitumen aeration, a second charge-coupled device (CCD) camera was used with a graduated Luba tube mounted in the LHES (Wallwork, 2003; Li, et al., 2005). The shutter speed of the CCD was set at 0.01 ms. The view area of a lens connected to the CCD was adjustable to as small as 3 mm X 4 mm. The Luba tube with one end connected to a peristaltic pump was filled with the clear Aurora process water and inserted at an inclined angle into the slurry in the loading and froth-collection container. Inside the Luba tube, the bubbles and aerated bitumen aggregates floated up and glided along the upper inner surface, allowing the CCD video system to capture much clear images of sliding aggregates within its view depth. The CCD and the lens were adjusted to a proper position. When bitumen flotation started (i.e., air injection to the loop started), a small counter current flow of process water was pumped into the Luba tube from the top end to suppress the entrainment of dark slurry and to slow down the rising velocity of bitumen aggregates entering the Luba tube. This arrangement kept the content inside the Luba transparent for recording clear images. The digital video of the flotation process was recorded 15 frames per second by a computer. Wallwork (2003) described detailed instructions to record the video and then to get photographs of the bubbles and bitumen aggregates from the videos recorded. This visualization system allowed the detailed view of bitumen attachment to air bubbles (or engulfed bubbles) and loading of bitumen on the air bubbles during the air injection step.

Froth analysis The procedure used for the analysis of bitumen-water-solid content in bitumen froth was similar to that used for oil sands content analysis (see details of Appendix A). The percentage of bitumen, water and solids was calculated using Equations (3-1)–(3-3). The quality of bitumen froth was measured by mass ratios of bitumen to solids (*B/S*) and bitumen to water (*B/W*) using Equations (3-6) and (3-7).

B/S = mass of bitumen in bitumen froth /mass of solids in bitumen froth (3-6)

B/W = mass of bitumen in bitumen froth /mass of water in bitumen froth (3-7)

3.3 Results and Discussion

3.3.1 Repeatability of Test

Oil sands extraction tests using a BEU showed that operating temperatures lower than 50 °C led to inconsistent results (Sanford and Seyer, 1979). The repeatability of the LHES system was previously tested using a good processing oil sands ore. A good repeatability of bitumen recovery within ± 4% absolute was confirmed by operating the LHES at 50 °C and a slurry flow velocity of 2 m/s (Wallwork, et al., 2004). To establish the repeatability of tests with the modified LHES, extraction tests were conducted at a lower temperature of 35 °C using a transition oil sands ore (Syn704), containing 9.7% bitumen, 6.2% water and 25.5% fines (-44 µm). In each test, 1000 g of oil sands were extracted with 5.5 L of Aurora process water. The slurry was conditioned for 5 minutes in the loop without closing the flap prior to the continuous introduction of air at 200 mL/min. To validate the data, control tests were run on a second LHES system at Syncrude Research Centre (Edmonton). The results and operating conditions are shown in Figure 3.2. Runs 1 and 2 were carried out at UA loop and run 3 was conducted at Syncrude. The bitumen recovery data (Figure 3.2 (a)) obtained on UA loop were highly repeatable within an absolute error of ± 2%.

The froth quality data measured by B/S ratio (Figure 3.2 (b)) was also repeatable within an error of ± 0.1 absolute. The comparison of data obtained on the two LHES systems shows, to some degree, good agreement in both bitumen recovery and froth quality. Further tests using Syn704 ore were carried out at 45 °C using the UA LHES. For clarity, the results are shown in Figure D.1 and Figure D.2 of Appendix D. The test results overall show that the results of bitumen recovery and bitumen froth quality obtained using the LHES tests were highly repeatable.

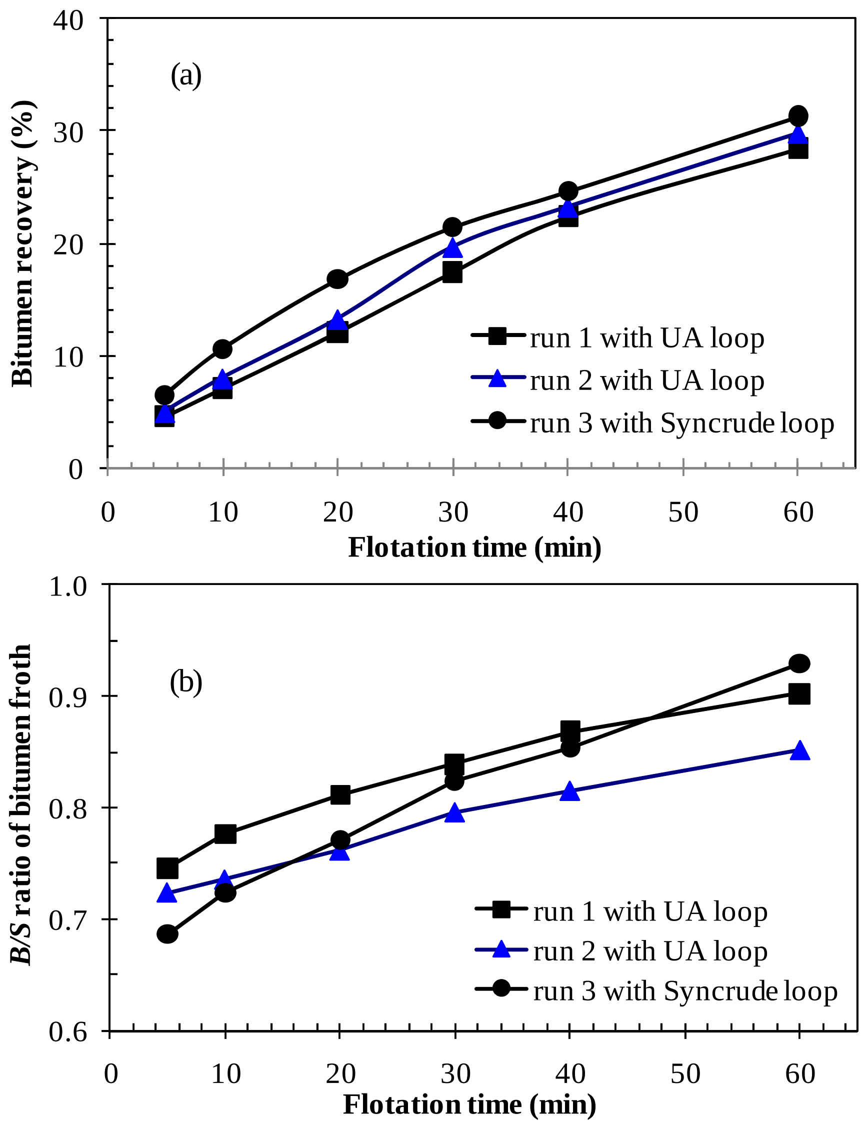


Figure 3.2 Repeatability test of the LHES: (a) bitumen recovery and (b) *B/S* ratio of bitumen froth, using 1 kg transition ore (Syn704) at temperature of 35 °C, air injection rate of 200 mL/min, slurry flow velocity of 3 m/s and conditioning for 5 minutes.

3.3.2 Effect of Conditioning Time

Bitumen liberation rates Bitumen cannot be recovered unless it is liberated from oil sands. The effect of conditioning time on bitumen liberation of all three ores AL711, Syn704 and Posyn was tested at slurry temperature of 45 °C and slurry flow velocity of 3 m/s. The bitumen liberation rates (Figure 3.3) show that the bitumen liberation rates of the good and the transition ores increased with conditioning time. This is in a similar trend as demonstrated by Wallwork (2003). Figure 3.3 also shows that the liberation rate of the poor processing ore was very low and did not change significantly with increasing conditioning time. It implies that the bitumen in the poor processing ore was hard to liberate from the sands, thereby leading to low bitumen recovery. This is to be confirmed in next sections of this chapter.

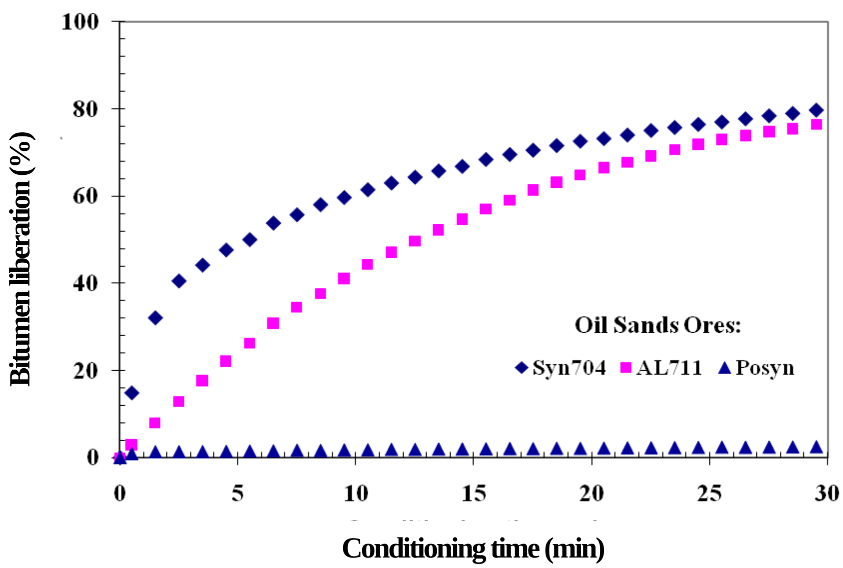


Figure 3.3 Bitumen liberation rates versa conditioning time for transition (Syn704), good (AL711) and poor (Posyn) processing ores, respectively, tested in the LHES. Experimental conditions: 45 °C and 3 m/s slurry flow velocity, no air injected (i.e., 0 min flotation time).

Bitumen recovery and B/S Oil sands slurry conditioning is an important step for bitumen recovery. The purpose of slurry conditioning is to liberate bitumen from the oil sands matrix and then to allow coalescence of bitumen drops so that bitumen drops at suitable size can be aerated when air bubbles are available (Shaw, et al., 1996; Friesen, et al., 2004b; Sanders, et al., 2007). Insufficient conditioning or over conditioning slurry would lead to low bitumen recovery and poor froth quality (Wang, et al., 2002; Friesen, et al., 2004b). The oil sands slurry conditioning, i.e., bitumen liberation, was influenced by such factors as slurry conditioning time, shear rate, temperature, water chemistry, air availability and the processability of the ore itself. When all other parameters are optimized, question remains whether the slurry conditioning time is an essential controllable variable to an appropriate slurry conditioning. To answer this question, extraction tests using transition oil sands ore Syn704 were carried out by controlling slurry conditioning time to 0, 5, 10 or 30 min, followed by an air injection at 200 mL/min for 60 min. The tests were conducted at a slurry temperature of 45 °C and a 3.0 m/s linear slurry flow velocity. The bitumen recovery and the bitumen to solids ratio of bitumen froth obtained in these tests are shown in Figure 3.4.

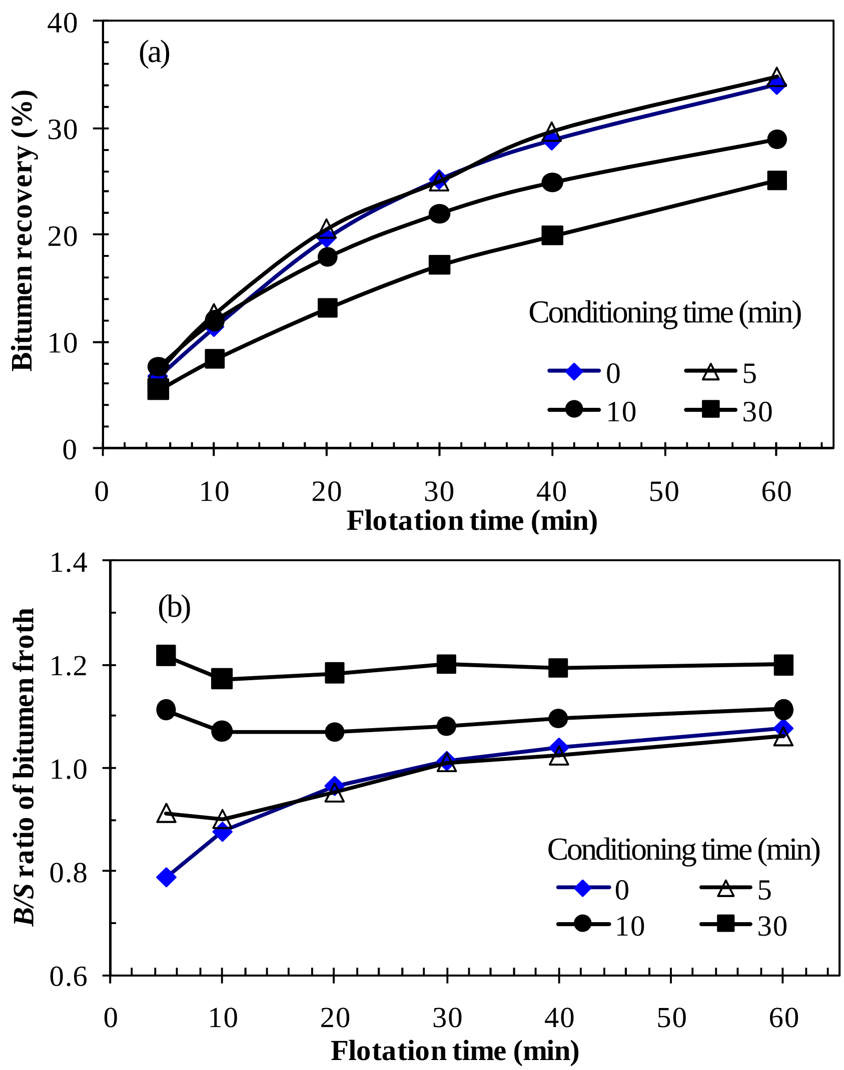


Figure 3.4 Effect of slurry conditioning time on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the transition ore (Syn704) tested in the LHES, using 1 kg ore tested at 45 °C, 200 mL/min air

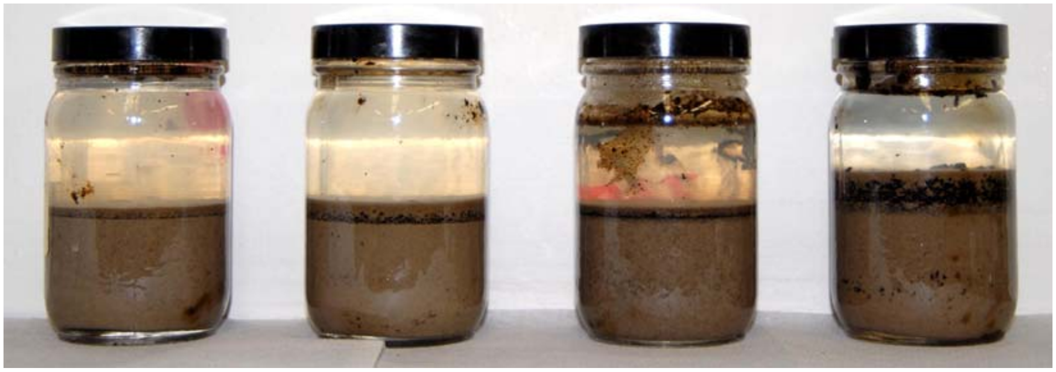
injection rate, and 3.0 m/s slurry flow velocity.

Figure 3.4 (a) shows that the bitumen recovery increased with increasing flotation time. This could be understood as a result of no air availability. The more the air bubbles were available and the longer the air was injected, the higher the chances were for liberated bitumen droplets to be aerated and recovered. This finding is well understood in oil sands research. Figure 3.4 (a) also shows that bitumen recovery decreased with increasing slurry conditioning time from 5 min to 30 min. This decrease in bitumen recovery became more significant over a longer flotation time. At flotation time of 60 min, for example, bitumen recovery decreased by about 6% and 10% for the tests conditioned for 10 min and 30 min, respectively. However, the bitumen recovery of tests with no slurry conditioning or conditioned for 5 minutes show a negligible difference over the entire air injection period up to 60 minutes. This might be an indication of over conditioning if conditioning time was longer than 5 minutes

The effect of slurry conditioning time on *B/S* ratio as a measure of bitumen froth quality is shown in Figure 3.4 (b). For the tests with slurry conditioning time of 5, 10, or 30 min, it is very interesting to note a minimum *B/S* value at flotation time around 10 min. With increasing flotation time, *B/S* value dropped initially and then began to increase gradually after 10 min flotation time. In the test without conditioning step (zero conditioning time, i.e., air injection started at the very beginning of the test), *B/S* ratio increased monotonically with increasing flotation time. After the first 10 min flotation, the *B/S* ratio of test with zero conditioning time had little difference compared to the test with 5 min conditioning time. When slurry conditioning time was less than 5 min, *B/S* ratio improved mainly at the early stage of air injection, up to 30 min of air injection. Figure 3.4 (b) also shows an increase in *B/S* ratio with increasing conditioning time. The *B/S* ratio increased by 0.1 from about 1.1 to 1.2 over entire air injection period when conditioning time increased from 10 min to 30 min. After the first 10 min flotation time, the *B/S* ratio kept quite constant up to air injection period of 60 min. This finding indicates that longer slurry conditioning time was beneficial to improving bitumen froth quality. This can be explained by the suppressed entrainment of oil sands in the bitumen froth with increasing conditioning time.

Figure 3.4 (b) shows the improvement of the bitumen froth quality (i.e., an increase in *B/S* ratio) with both longer slurry conditioning time and longer flotation time. This is also in good agreement with the results of previous study that bitumen froth quality improved with extraction time (Kasongo, et al., 2000; Wallwork, et al., 2004). When air bubbles were added to slurry, bitumen droplets were attached to air bubbles and the formed aggregates floated up. At the same time, solids and water together with some undigested oil sands were entrained by floating aggregates and bubbles and reported to froth. The longer the conditioning time, the more bitumen droplets became available for aeration. This explains why the quality of bitumen froth improved with increasing conditioning time. A minimum *B/S* ratio was also observed on each curve of tests with conditioning time longer than 5 min. After this minimum point, the bitumen quality improved with increasing extraction time. This implies a competition between the recovery of aerated bitumen droplets and the entrainment of solids.

The results studying the effect of slurry conditioning time (Figure 3.4 (a)) show that bitumen recovery was negatively impacted if the slurry conditioning time was too long (or air injection was delayed too much). The reduced bitumen recovery might be caused by lack of air bubbles during the conditioning. When no air was added to the slurry during conditioning period, unaerated large bitumen droplets formed from liberated bitumen (Sanders, et al., 2007; Friesen, et al., 2004b). Solids attached to the surfaces of the unaerated bitumen droplets, known as slime coating (Dai and Chung, 1996), therefore leading to deficient bitumen aeration. Actually large bitumen aggregates or lumps were observed in the tailings of the LHES tests. The tailings after 60 min of air injection of corresponding tests in Figure 3.4 were sampled using 200-mL jars after each test. Their photos (Figure 3.5) show a bitumen-rich layer on the top of the settled tailings. The longer the conditioning time, the thicker the bitumen-rich layer and the more the large unareated bitumen aggregates were observed in the tailings. The formation of large bitumen aggregates was also reported by Sanders, et al. (2007) and Friesen, et al. (2004b). In their studies large unaerated bitumen droplets (> 500 μm) and adhesion of bitumen to the stirrer were observed. The formation of large unaerated bitumen droplets could explain the reduced bitumen recovery with longer slurry conditioning time. It is an indication of over conditioning.



(a) (b) (c) (d)

Figure 3.5 Photographs of tailings for tests with conditioning time of (a) 0 min, (b) 5 min, (c) 10 min and (d) 30 min, using 1 kg transition ore (Syn704) at 45 °C, 3.0 m/s slurry flow velocity and 60 min flotation time at 200 mL/min air injection rate in the LHES.

Bitumen aeration To view the bitumen aeration, the good processing ore (AL711) was tested at 55 °C and 4.5 m/s slurry flow velocity in the LHES loop. The slurry was conditioned for 0 min and 30 min, respectively, prior to flotation at air injection rate of 500 mL/min. Just after air was injected into the slurry, microphotographs of the bubbles/aggregates leaving the slurry were taken using the visualization system. The typical photographs are shown in Figure 3.6. In the photograph of case 1 (no slurry conditioning time), many small particles together with air bubbles and bitumen aggregates were observed at the beginning of the air injection period. By contrast, in the photograph of the second case (with slurry conditioned for 30 min), much less small particles were observed. This might provide a possible explanation to the improved bitumen froth quality with longer slurry conditioning time. Wallwork (2003) explained that the bitumen-mineral aggregates rather than fully liberated bitumen were being floated at the beginning of the tests in the LHES loop. This explains the better bitumen froth quality at longer slurry conditioning time.

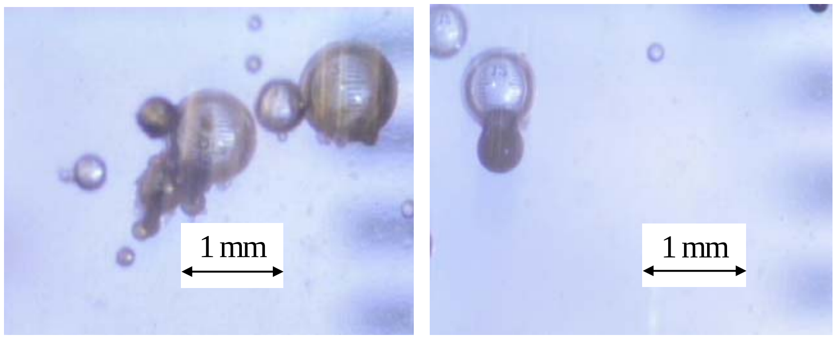
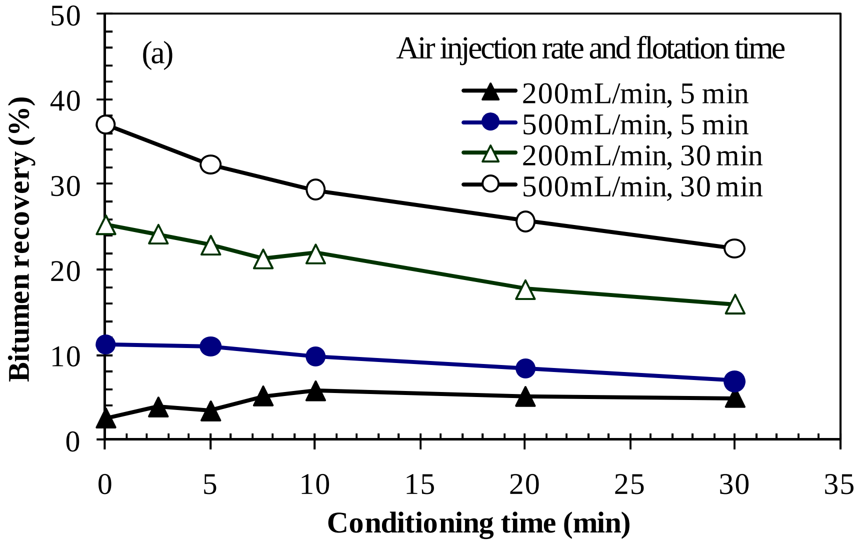


Figure 3.6 Microphotographs of the bubbles/aggregates rising in the Luba tube at air injection, using 1 kg AL711 ore tested at 55 °C and 4.5 m/s slurry flow velocity in the LHES. The slurry was conditioned for 0 min (left) and 30 min (right) prior to flotation at air injection rate of 500 mL/min.

3.3.3 Effect of Air Injection Rate

Availability of sufficient air, indigenous or added, is necessary for effective aeration of liberated bitumen droplets and bitumen recovery (Kasongo, et al., 2000; Wallwork, et al., 2004). A higher bitumen recovery might be expected if more air is available because the attachment of bitumen to air bubbles is probabilistic in the turbulent slurry of sands, bitumen droplets and air bubbles (Eskin, et al., 2004; Friesen, et al., 2004b). It was confirmed that higher air to slurry volume ratio was beneficial to bitumen recovery (Wallwork, 2003). In Wallwork's study a fixed conditioning time of 5 min was employed. In the present study, extraction tests with different slurry conditioning time were conducted to study the effect of air to slurry ratio on bitumen recovery and bitumen froth quality using three types of oil sands ores: good processing, poor processing and transition ores.

The transition ore Syn704 was tested at 45 °C slurry temperature, 3.0 m/s slurry flow velocity, and 200 mL/min and 500 mL/min air injection rates, respectively. Slurry conditioning time of these tests was from 0 to 30 min with flotation time of 5 min and 30 min, respectively. The bitumen recovery and *B/S* ratio are shown in Figure 3.7 (a) and (b), respectively.



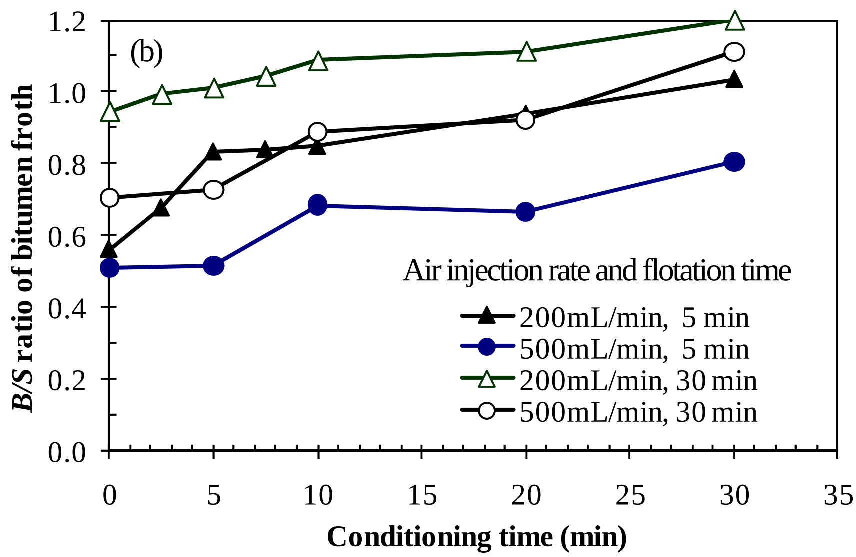


Figure 3.7 Effect of air injection rate on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the transition ore (Syn704) tested in the LHES. Experimental conditions: 1 kg ore tested at 45 °C and 3.0 m/s slurry flow velocity with slurry conditioning prior to flotation for 5 min (solid symbols) and 30 min (open symbols) at an air injection rate of 200 mL/min (triangles) or 500 mL/min (circles).

Figure 3.7 (a) shows that the higher air injection rate yielded higher bitumen recovery for both short (5 min) and long (30 min) flotation time. For tests with 30 min flotation time, bitumen recovery increased by about 10% at 500 mL/min than at 200 mL/min air injection rate with no slurry conditioning time. A corresponding increase by about 7% was achieved when the slurry was conditioned for 30 min. For tests with only 5 min air injection, an increase of about 10% in bitumen recovery was achieved when air injection rate increased from 200 mL/min to 500 mL/min without slurry conditioning. But if slurry was conditioned for 30 min, a smaller increase in bitumen recovery was achieved at the higher air injection rate of 500 mL/min than at 200 mL/min. The improved bitumen recovery at higher air injection rates is in line with the results of Wallwork's study where a fixed slurry conditioning time was used (2003). The increased bitumen recovery with increasing air injection rate could be understood by the increased probability of collision between liberated bitumen droplets and air bubbles at higher air injection rate (Eskin, et al., 2004). Higher air injection rates created higher number of air bubbles in the slurry in the LHES loop therein higher aeration chance for bitumen droplets.

Figure 3.7 (a) also shows that for 30 min flotation time increasing slurry conditioning time from 0 min to 30 min decreased the bitumen recovery significantly from 37% to 23% at 200 mL/min air injection rate and from 26% to 16% at 500 mL/min air injection rate. The reduced bitumen recovery with increasing slurry conditioning time was possibly caused by the delayed air injection as addressed in aforementioned section.

Figure 3.7 (b) shows that increasing conditioning time improved *B/S* ratio of bitumen froth, i.e., higher bitumen froth quality. The improvement appears more significant at lower air injection rate than at higher air injection rate. A possible explanation to the improved forth quality is that the entrainment of solids was reduced at lower air injection rate, thereby less solids reporting to bitumen froth.

The good (AL711) and the poor (Posyn) processing ores were also tested at the same conditions as applied to the transition ore (Syn704). The results of bitumen recovery and *B/S* ratio of bitumen froth are shown in Figure 3.8 for the good processing ore and Figure 3.9 for the poor processing ore. Compared with the results of the transition ore (Figure 3.7), a similar effect of air injection rates on bitumen recovery and bitumen froth quality was observed for both good (Figure 3.8) and poor (Figure 3.9) processing ores. For flotation time of 30 min, bitumen recovery from the poor processing ore (Posyn) increased by around 3% when air injection rate increased from 200 mL/min to 500 mL/min, in contrast to an absolute increase of around 8–12% for the transition (Syn704) and the good processing (AL711) ores. This observation implies that the poor processing ore (Posyn) had less bitumen liberated than the other two ores at a given conditioning and flotation condition. It is very important to note that the bitumen recovery from Posyn ore is much lower than the other two ores for all the conditions testes.

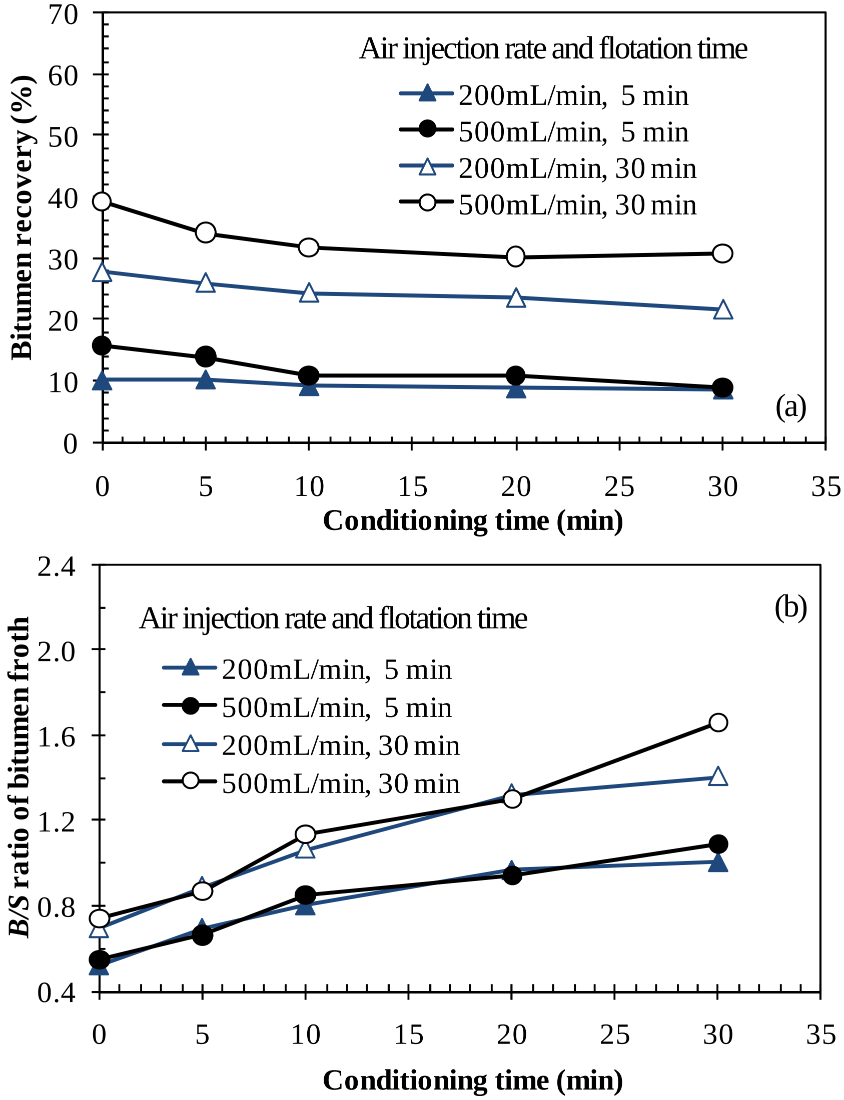


Figure 3.8 Effect of air injection rate on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for good processing ore (AL711) tested in the LHES. Experimental conditions: 1 kg ore tested at 45 °C and 3.0 m/s with slurry conditioning prior to flotation at air rate of 200 m L/min (triangles) and 500 m L/min (circles) for flotation time of 5 min (solid symbols) and 30 min (open symbols).

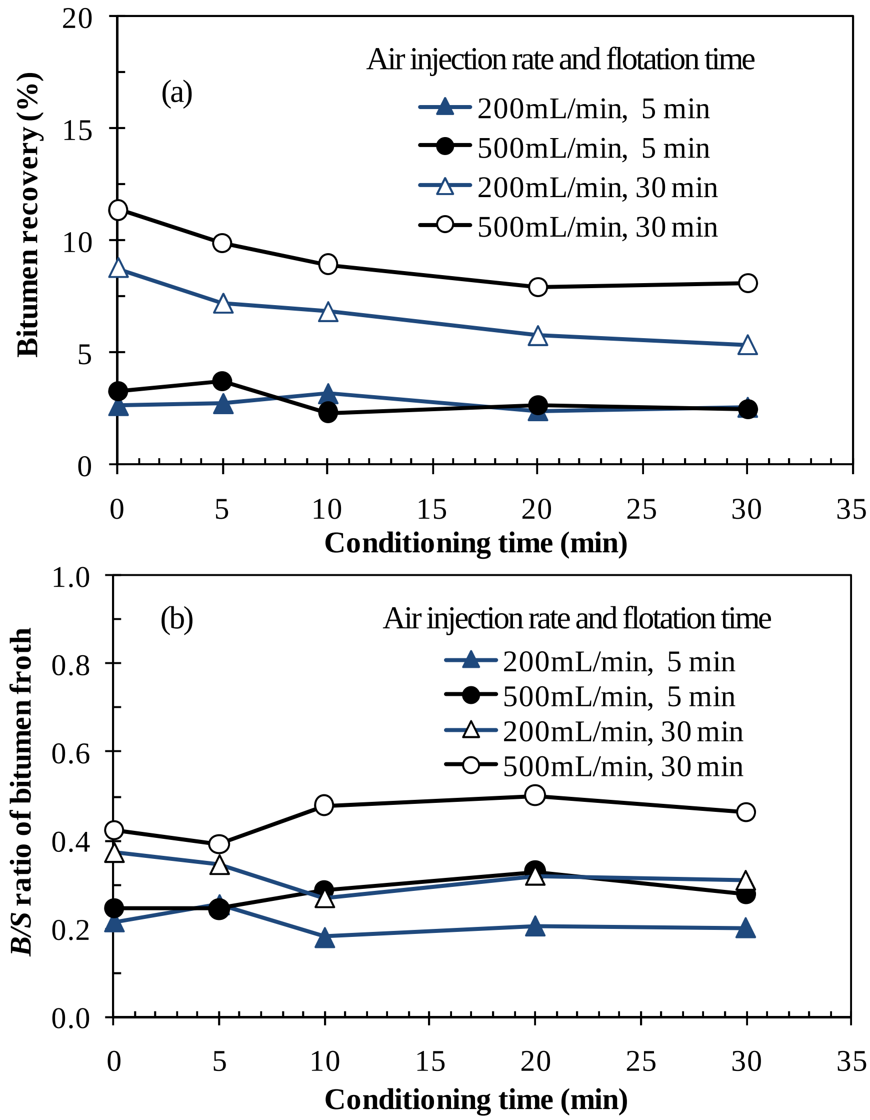


Figure 3.9 Effect of air injection rate on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth using 1 kg poor processing ore (Posyn) tested in the LHES. Experimental conditions: at 45 °C and 3.0 m/s with slurry conditioning prior to flotation for flotation time of 5 min (solid symbols) and 30 min (open symbols) at 200 mL/min (triangles) or 500 mL/min (circles).

Figure 3.7, Figure 3.8 and Figure 3.9 also show that the slurry conditioning time had different effect on both bitumen recovery and *B/S* ratio of bitumen froth for all three ores. With increasing slurry conditioning time, bitumen recovery decreased by around 5–10% for the good processing ore (Figure 3.8), 8–12% for transition ore (Figure 3.7) and about 3% for the poor processing ore (Figure 3.9) for

30 min flotation time, depending on the air injection rates. The bitumen recovery changed little with conditioning time for flotation time of 5 min for all the cases. Increasing slurry conditioning time from 0 min to 30 min increased *B/S* ratio of bitumen froth by about 0.7 for the good processing ore (Figure 3.8 (b)), and by about 0.4 for the transition ore (Figure 3.7 (b)). In contrast, no change in *B/S* ratio for poor processing ore was observed (Figure 3.9 (b)).

3.3.4 Effect of Temperature

Temperature plays a very important role in bitumen liberation from oil sands (Luthra, 2001; Wallwork, 2003), aeration of liberated bitumen droplets (Wallwork, 2003; Masliyah, et al., 2004) and bitumen recovery (Bichard, 1987; Zhou, et al., 2004a). The effect of temperature on bitumen extraction has been comprehensively reviewed by Kasperski (2001), Masliyah, et al. (2004), and Long, et al. (2007). In the current study, effect of conditioning temperature on bitumen recovery and bitumen froth quality is presented. The three oil sands ores were conditioned for 0, 5, 10, 20 and 30 min in the LHES at different temperatures. The effect of slurry conditioning temperature on bitumen recovery and bitumen froth quality was studied.

The results for the transition ore (Syn704) are shown in Figure 3.10 and Figure 3.11 for air injection rates of 200 and 500 mL/min, respectively. The results show that higher conditioning temperatures led to higher bitumen recovery and higher bitumen froth quality (i.e., higher *B/S* ratio). In the case of 200 mL/min air injection rate, increasing conditioning time increased bitumen recovery slightly for 2 min flotation time, whereas decreased bitumen recovery significantly by about 10% at 45 ºC and 8% at 35 ºC for 30 min flotation time (Figure 3.10 (a)). *B/S* ratio of bitumen froth increased with increasing conditioning time at both slurry temperatures (Figure 3.10 (b)). For both short (2 min) and long (30 min) flotation time, *B/S* ratio increased with temperature and conditioning time. It is evident that higher temperatures enhanced conditioning, improving both bitumen liberation and bitumen froth quality. However, longer conditioning time yielded lower bitumen recovery due to delayed aeration of bitumen in the absence of air. As a result, the formation of increasingly larger bitumen droplets led to difficulties in their recovery.

In the case of 500 mL/min air injection rate for 30 min flotation time, with increasing temperatures, bitumen recovery increased by about 11% and 22% when slurry conditioning time was 0 min and 30 min, respectively (Figure 3.11 (a)). Bitumen recovery became less sensitive to conditioning time at higher temperature of 55 ℃ than at lower temperature of 45 ℃. Again higher *B/S* ratio of bitumen froth was observed at higher temperature (Figure 3.11 (b)). At both temperatures, bitumen froth quality improved with increasing conditioning time.

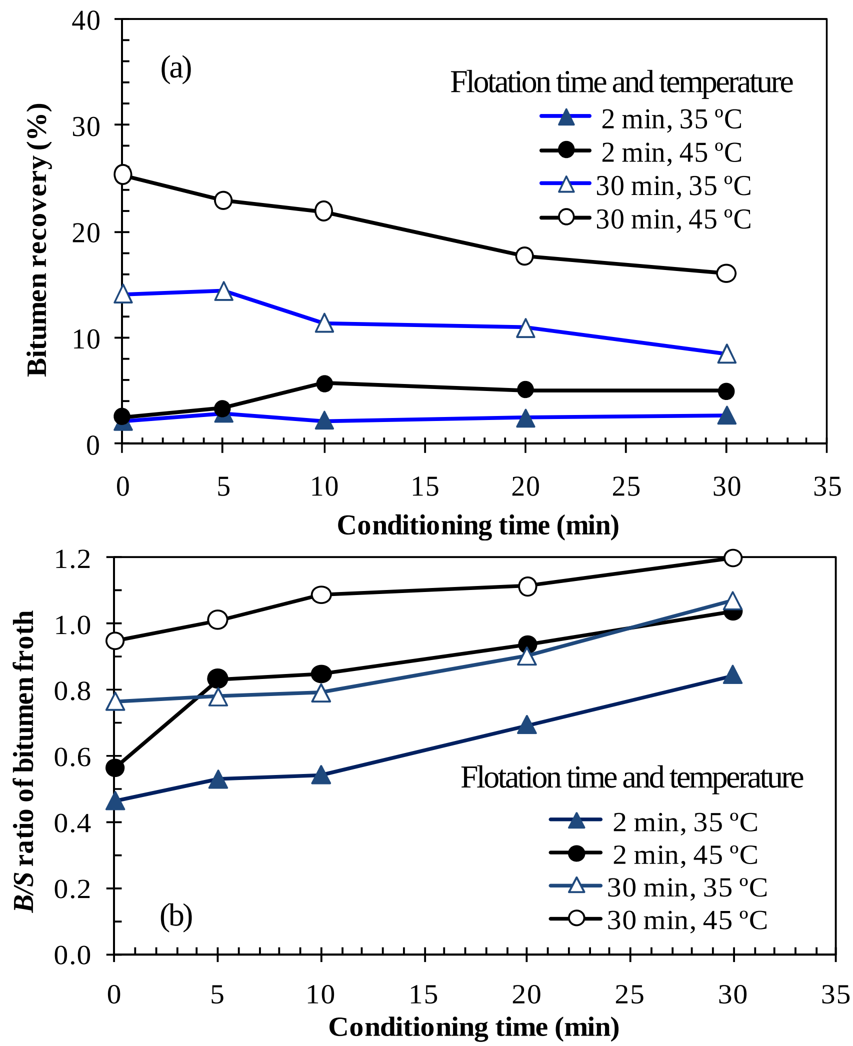


Figure 3.10 Effect of slurry temperature on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for transition ore (Syn704) tested in the LHES, using 1 kg ore tested at 35 ℃ (triangles) or 45 ℃ (circles) and 3.0 m/s with air injection rate 200 mL/min for 2 min (solid symbols) and 30 min (open symbols).

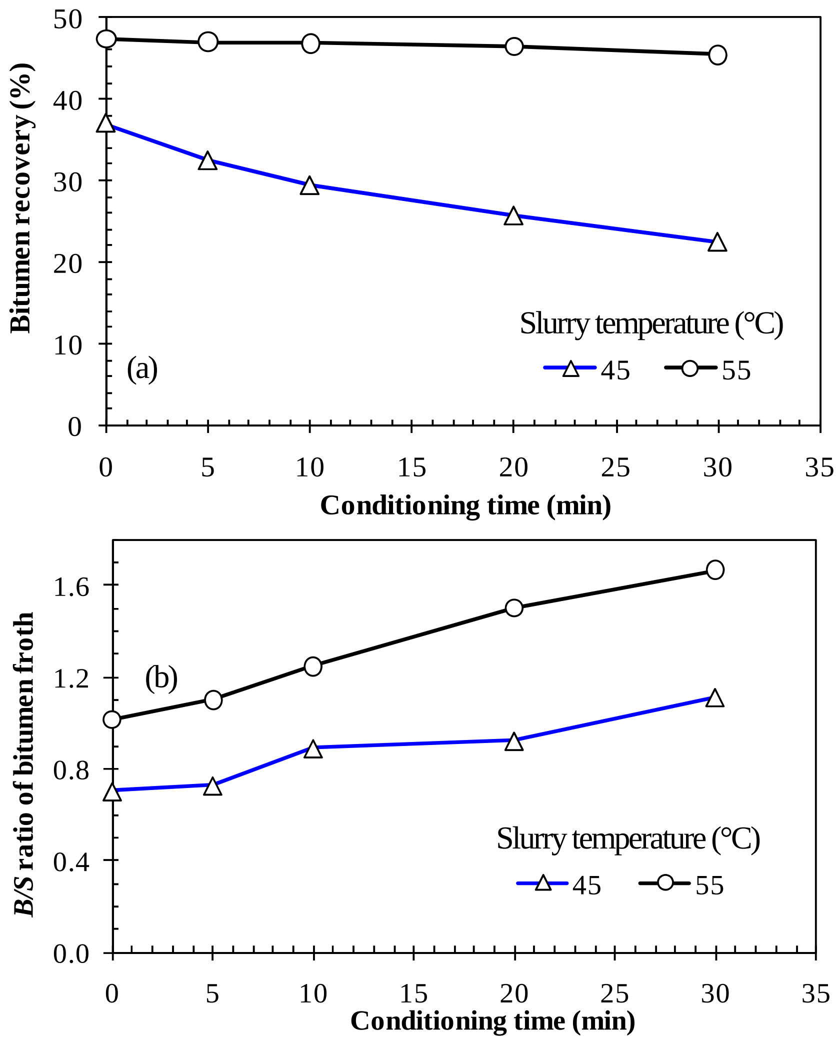


Figure 3.11 Effect of slurry temperature on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the transition ore (Syn704) tested in the LHES, using 1 kg ore tested at 45 °C (triangles) or 55 °C (circles), 3.0 m/s slurry velocity and with 30 min flotation time at 500 mL/min.

The good (AL711) and the poor (Posyn) processing ores were also tested at similar conditions as employed for the transition ore: air injection rate of 500 mL/min, 3.0 m/s slurry flow velocity and temperatures of 45 °C and 55 °C. The results of bitumen recovery and *B/S* ratio of bitumen froth are shown in Figure 3.12 and Figure 3.13. It is evident that a higher slurry temperature and longer flotation time led to higher bitumen recovery and better bitumen froth quality (i.e., higher *B/S*). For the good processing ore (Figure 3.12), increasing conditioning time reduced bitumen recovery slightly at 45 °C and showed negligible effect on bitumen recovery at 55 ºC at both flotation times. *B/S* ratio of the bitumen froth, on the other hand, increased by about 0.4~1.0 at both temperatures. For the poor processing ore (Figure 3.13), increasing conditioning time showed a marginal decrease in bitumen recovery for flotation time of 5 min and significant decrease in bitumen recovery for flotation time of 30 min. Bitumen froth quality improved slightly with increasing conditioning time at both processing temperatures of 45 ºC and 55 ºC.

Among the three ores (Figure 3.11, Figure 3.12 and Figure 3.13), slurry temperature had similar effect on bitumen recovery and bitumen froth quality. Higher bitumen recovery was achieved at a higher slurry temperature. With increasing conditioning time, bitumen recovery decreased at both temperatures except the cases that the good processing ore (Figure 3.12) and transition ore (Figure 3.11) were processed at 55 ºC, in which negligible change was observed. The increased *B/S* ratio of bitumen froth for the good processing ore was the most sensitive to the increase in conditioning time and the least for the poor processing ore at both temperatures. For example, at 55 °C and for 30 min flotation time the increase in conditioning time from 0 to 30 min led to an increase in *B/S* ratio by 1.2 for the good processing ore (Figure 3.12 (b)), by 0.7 for transition ore (Figure 3.11 (b)), and by only about 0.3 for the poor processing ore (Figure 3.13 (b)). The difference in the effect of conditioning time and temperature on bitumen recovery and froth quality for the three ores might be linked with their bitumen liberation kinetics. The earlier bitumen is liberated from the sand grain, the stronger is the effect of conditioning time on bitumen recovery and froth quality.

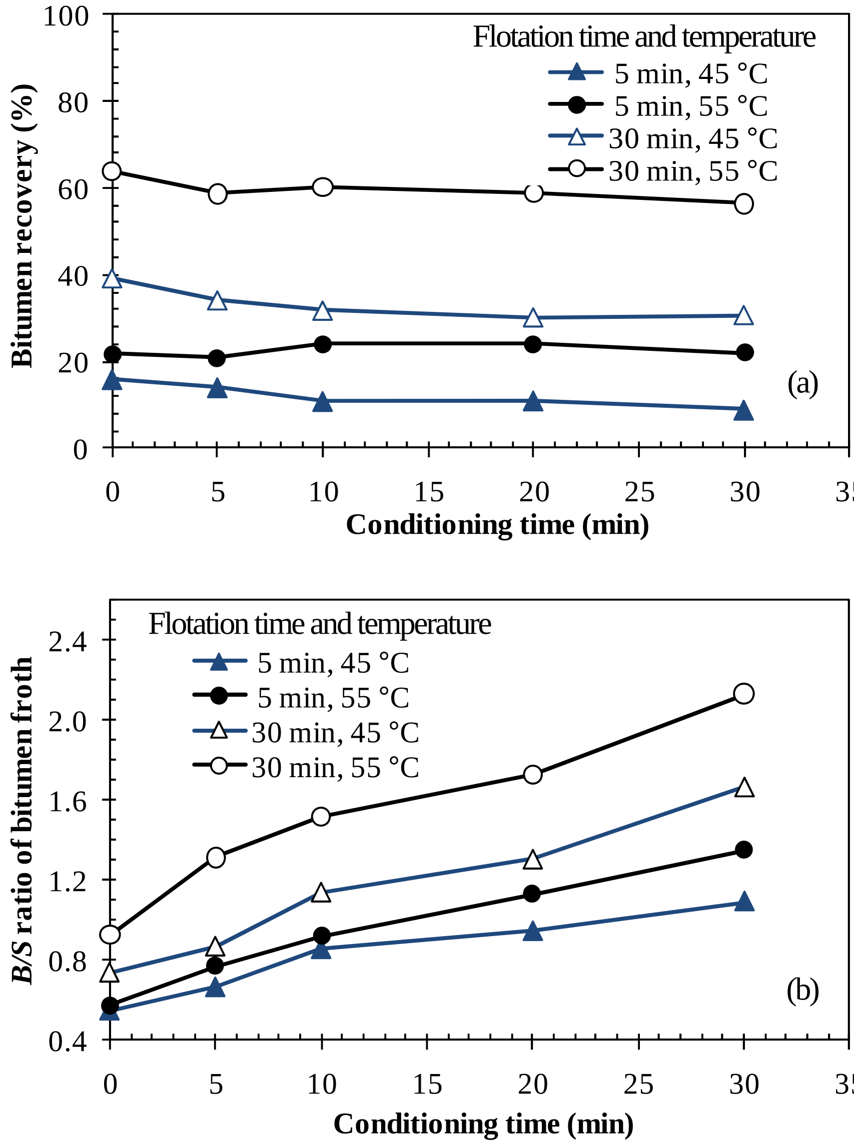
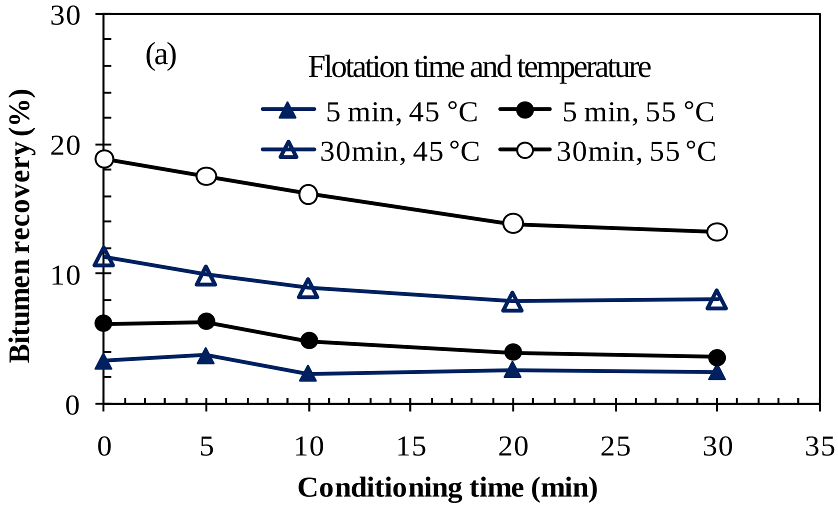


Figure 3.12 Effect of slurry temperature on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the good processing ore (AL711) tested in the LHES, using 1 kg ore tested at 45 °C (triangles) or 55 °C (circles) and 3.0 m/s slurry flow velocity with flotation at air injection rate of 500 mL/min for 5 min (solid symbols) and 30 min (open symbols).



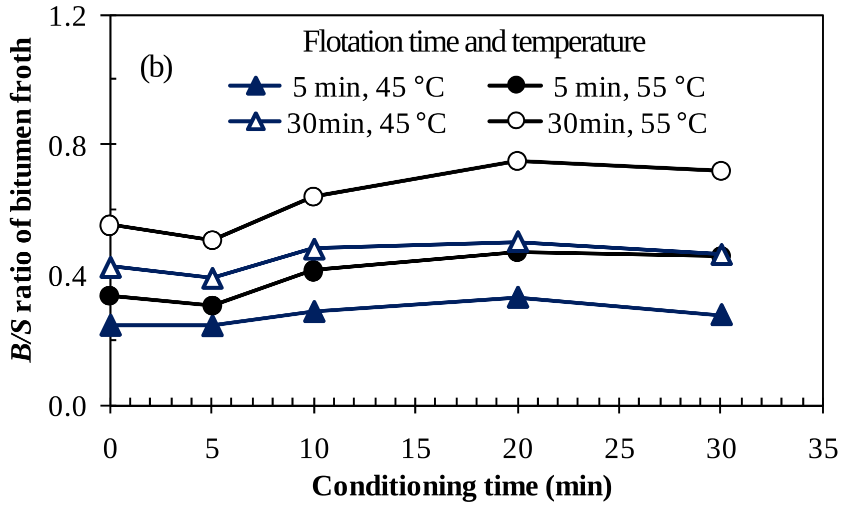


Figure 3.13 Effect of slurry temperature on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the poor processing ore (Posyn) tested in the LHES. Experimental conditions: 1 kg ore tested at 45 °C (triangles) or 55 °C (circles) and 3.0 m/s slurry flow velocity with air injection at 500 mL/min for 5 min (solid symbols) and 30 min (open symbols).

The above tests indicate that bitumen froth for the good processing ore and transition ore was improved at higher temperatures. This is attributed to quicker bitumen liberation and better aeration of bitumen droplets. The quicker bitumen liberation was proven. The improvement of bubble attachment to bitumen at higher temperatures was investigated in current study by using the good processing ore (AL711) tested at 45 and 55 °C separately. The slurry was conditioned for 30 min at 4.5 m/s slurry flow velocity prior to flotation at air injection rate of 500 mL/min. Microphotographs of bubbles and/or bubble-bitumen aggregates rising in the Luba tube were taken using the visualization system. The typical photographs are shown in Figure 3.14. It shows that the surfaces of bitumen aggregates in the test at temperature 55 °C looked cleaner than those at 45 °C. This provides a possible explanation to the improved bitumen froth quality at higher temperatures.

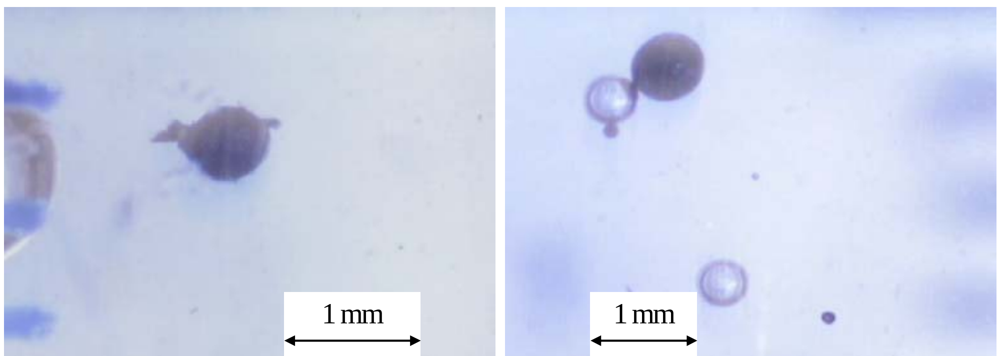


Figure 3.14 Microphotographs of bubbles/aggregates rising in the Luba tube at 1 min flotation time in the LHES using 1 kg AL711 ore tested at 45 °C (left) and 55 °C (right) with 30 min conditioning time, 4.5 m/s slurry flow velocity and 500 mL/min air injection rate.

3.3.5 Effect of Slurry Velocity

Studies have shown that bitumen extraction from oil sands was also enhanced by higher mechanical energy input (Masliyah, 2006). Increasing mechanical energy input to the slurry offset the low viscosity of slurry at low temperatures, thereby improving bitumen recovery (Stasiuk, et al., 2004). The mechanical energy could be identified by the slurry turbulence (Friesen and Dabros, 2004a). The study of Sanders, et al. (2007), using a 100-mm pipeline loop, showed that higher slurry flow velocity produced better oil sands conditioning. The authors contributed the better conditioning results to two critical factors: enhanced bitumen liberation due to increased particle dispersive stresses and enhanced bitumen-air contact frequency. A higher slurry flow velocity also promoted the formation of smaller bubbles and larger bitumen droplets (Razzaque, et al., 2003; Friesen and Dabros, 2004a). In the present study, the effect of slurry flow velocity on bitumen recovery and bitumen froth quality was studied by using the three types of ores.

For the transition ore (Syn704), tests were conducted at slurry temperature of 45 °C and air injection rate of 500 mL/min for a flotation time of 30 min. The effect of slurry flow velocity on bitumen recovery (Figure 3.15 (a)) shows a much higher bitumen recovery by about 45%–50% at higher slurry flow velocity of 4.5 m/s as compared to lower slurry velocity of 3.0 m/s. This observation indicates that slurry flow velocity positively impacted bitumen recovery. This observation is in good agreement with previous results showing a very significant effect of increasing mechanical energy on bitumen recovery (Sanders, et al. 2007). *B/S* ratio of bitumen froth (Figure 3.15 (b)) shows that higher slurry flow velocity was also beneficial to bitumen froth quality. Also show in Figure 3.15 a negative impact of increasing slurry conditioning time on bitumen recovery for both slurry flow velocities, but a positive impact on bitumen froth quality. A decrease in bitumen recovery by about 15% and 8% at 4.5 and 3.0 m/s slurry flow velocities, respectively, occurred when slurry conditioning time increased from 0 to 10 min. Considering a much more significant impact of slurry flow velocity on bitumen recovery, the negative impact of conditioning time on bitumen recovery can be compensated by operating at higher slurry flow velocities with added benefit of better bitumen froth quality. It appears that operating hydrotransport pipelines at high flow velocity is beneficial to both bitumen recovery and froth quality.

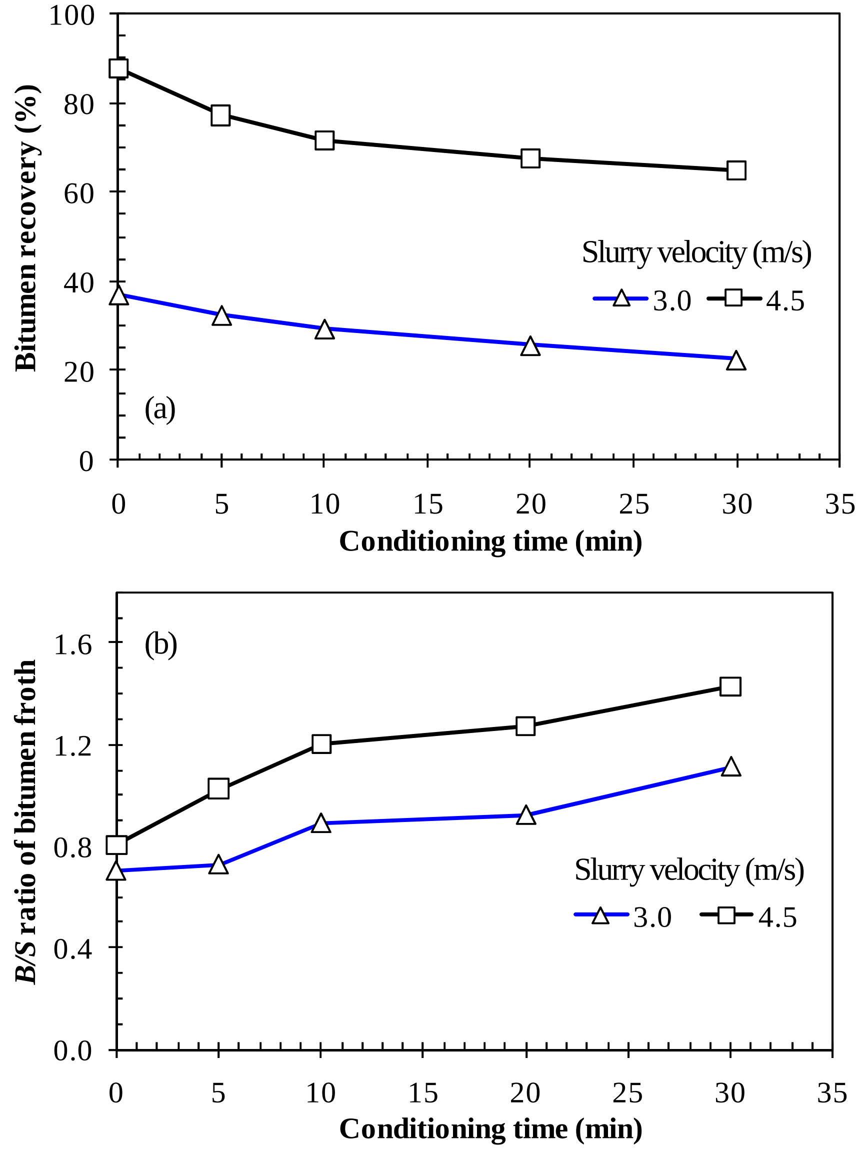


Figure 3.15 Effect of slurry velocity on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the transition ore (Syn704) tested in the LHES. Experimental conditions: 1 kg ore tested at 45 °C and 3.0 m/s (triangles) or 4.5 m/s (squares) slurry flow velocity with air injection for 30 min at 500 mL/min.

For the good (AL711) and the poor (Posyn) processing ores, the results are shown in Figure 3.16 and Figure 3.17, respectively. Figure 3.16 shows that, for a flotation time of 30 min, the bitumen recovery for AL711 ore (Figure 3.16 (a)) increased significantly with increasing slurry flow velocity, especially at lower slurry flow velocity. For example, for the test with zero conditioning time, the bitumen recovery improved from about 40% to 80% when the slurry flow velocity increased from 3.0 m/s to 3.75 m/s. A further increase in slurry flow velocity to 4.5 m/s led to a further increase in bitumen recovery by 10%. When the slurry conditioning time was longer than 5 min the quality of bitumen froth (Figure 3.16 (b)) also improved with increasing slurry flow velocity for a flotation time up to 30 min, more so at higher slurry flow velocities. Figure 3.16 also shows that increasing slurry conditioning time at a given slurry flow velocity reduced the bitumen recovery only marginally, in particular at higher slurry flow velocity. At slurry flow velocity of 4.5 m/s for 30 min conditioning time, for example, *B/S* ratio as high as 2.0 was achieved, in contrast to a *B/S* ratio of 0.9 at zero conditioning time.

For the poor processing ore, the effect of slurry velocity is shown in Figure 3.17. With increasing slurry flow velocity from 3.0 m/s to 4.5 m/s, bitumen recovery (Figure 3.17 (a)) increased significantly by about 16%, but remained relatively low at less than 30%. Figure 3.17 (b) shows only a marginal increase in *B/S* ratio of bitumen froth with slurry flow velocity and/or conditioning time. The highest *B/S* ratio for this ore remained below 0.6. In comparison, increasing slurry flow velocity, the increase in *B/S* ratio of bitumen froth of AL711 (Figure 3.16) and Syn704 (Figure 3.15) was much more significant.

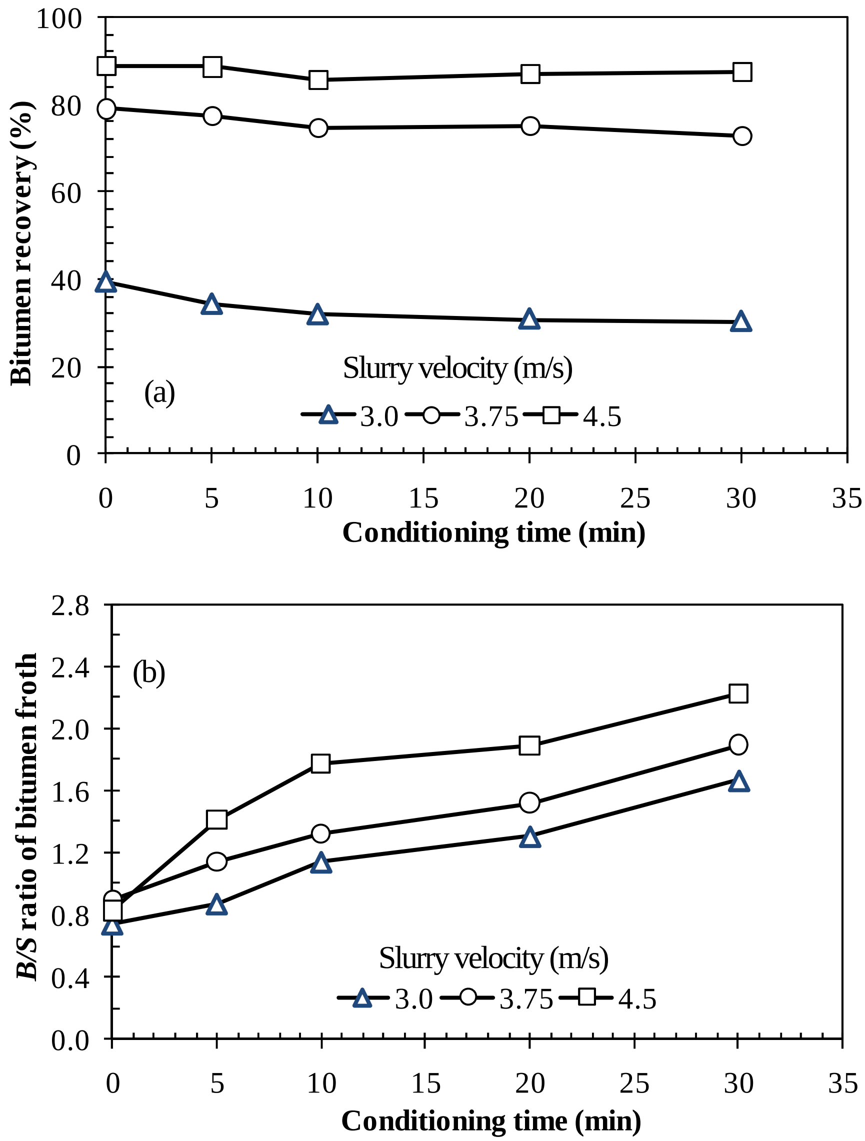


Figure 3.16 Effect of slurry velocity on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the good processing ore (AL711) tested in the LHES, using 1 kg ore tested at 45 °C and 3.0 m/s (triangles), 3.75 m/s (circles) or 4.5 m/s (squares) slurry flow velocity and with 500 mL/min air injection for 30 min.

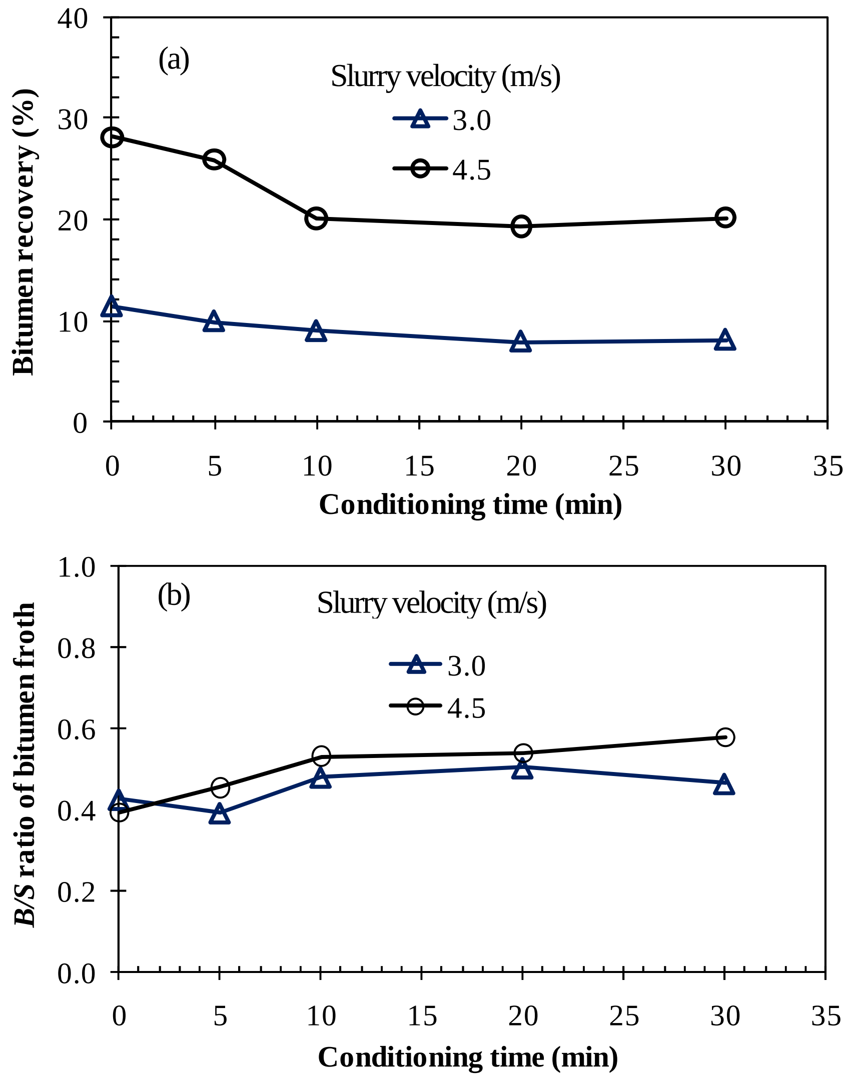


Figure 3.17 Effect of slurry velocity on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the poor processing ore (Posyn) tested in the LHES. Experimental conditions: 1 kg ore tested at 45 °C, 3.0 m/s (triangles) or 4.5 m/s (circles) slurry flow velocity and air injection rate 500 mL/min for 30 min.

For all the three types of ores (Figure 3.15 ~ Figure 3.17), the slurry flow velocity showed positive effect on bitumen recovery and bitumen froth quality. At low slurry velocity, the slurry might suffer from over conditioning if conditioning time is long. At high slurry flow velocity, the negative impact of long slurry conditioning time on bitumen recovery became less significant. This could be elucidated as follows. With no air being added to the slurry during the conditioning step, the longer the conditioning time is , the more unaerated large bitumen droplets are formed in the slurry (Sanders, et al., 2007) and the more solids/fines are attached to the surface of those unaerated bitumen droplets, known as slime coating (Dai and Chung, 1996). For the good processing ore slurry less slime coating on liberated bitumen droplets occurred and the force between coating fines and bitumen might be weaker than that for the transition and poor processing ores (Liu, et al. 2004a; Liu, et al. 2004b). The large slime-coated bitumen droplets got a higher chance to be broken up at strong mechanical energy input (high slurry flow velocity) and be aerated with air bubbles when available.

For comparison, the effect of each conditioning variable on bitumen recovery and *B/S* ratio of bitumen froth of the three different types of oil sands ores, AL711, Syn704 and Posyn, is shown in the same figures. For clarity, these figures are shown in Figures E.1~E.3 of APEENDIX E. The good processing ore (AL711) had the highest bitumen recovery and best bitumen froth quality while the poor processing ore (Posyn) had the lowest bitumen recovery and worst bitumen froth quality. This is expected because they have different fines content and processability.

3.3.6 Effect of Slurry Density

Slurry density might have influence on bitumen recovery and bitumen froth quality. At high slurry density of about 1.6 kg/L, batch extraction test results showed that an optimal addition of slurry water did exist (Schramm, et al., 1989). But bitumen recovery changed little with changing slurry density of both high fines and low fines oil sands ores in the LHES (Wallwork, 2003). Note that the density used was very dilute compared with slurry density encountered in commercial operations or standard BEU tests. In the present study, the density effect was investigated using the good processing ore (AL711).

AL711 ore slurry was diluted to 1.10 kg/L and 1.20 kg/L and tested at slurry temperature of 45 °C and slurry flow velocity of 4.5 m/s. The slurry was conditioned prior to flotation for 30 min at air injection rate of 500 mL/min. The results in Figure 3.18 show that the slurry density had little effect on bitumen recovery and bitumen froth quality with slurry conditioning time from 0 to 30 min.

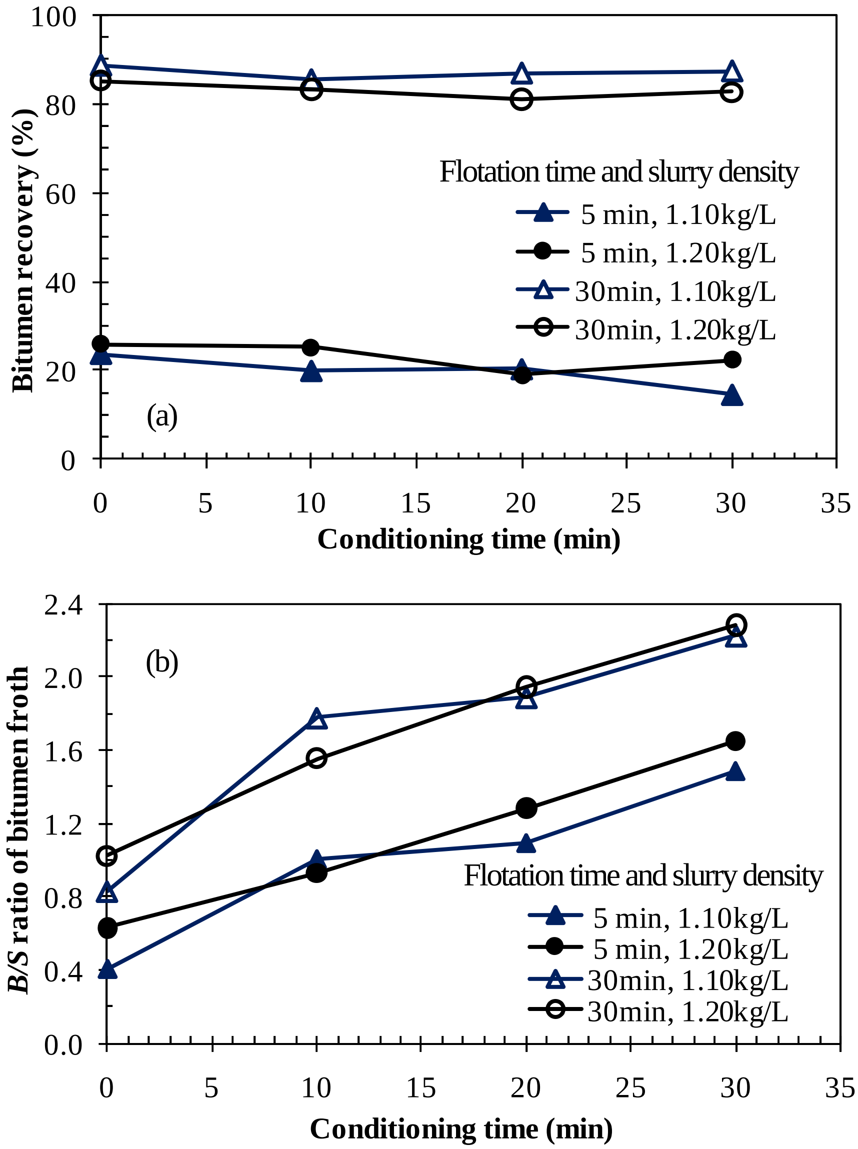


Figure 3.18 Effect of slurry density on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the good processing ore (AL711) tested in the LHES. Experimental conditions: 1.10 kg/L (triangles) and 1.20 kg/L (circles) slurry densities, at 45 °C, 4.5 m/s slurry velocity and air injection rate 500 mL/min for 5 min (solid symbols) and 30 min (open symbols).

3.4 Conclusions

The effect of slurry conditioning on bitumen extraction from oil sands ores was investigated using good (AL711), transition (Syn704) and poor (Posyn) processing ores. The variables investigated include conditioning time, flotation time, slurry temperature, slurry flow velocity, air injection rate and slurry density. The information gathered in this study shows that the variables of slurry conditioning in the LHES had different effects on bitumen recovery and bitumen froth quality, depending on the operating variables and types of ores. The key results can be summarized as follows:

1) An increase in slurry conditioning time yielded lower bitumen recovery for a long flotation time (30 min) but higher bitumen froth quality regardless of flotation time for all three oil sands ores. However over conditioning could be compensated by higher conditioning temperatures and higher slurry flow velocities. For example, little over conditioning was observed for the good processing and the transition ores tested at 55 °C and 3.0 m/s and for the good processing ore at 45 °C and 4.5 m/s.

2) Bitumen recovery of the good processing (AL711), poor processing (Posyn), and transition (Syn704) ores increased with flotation time. So did the bitumen froth quality.

3) Increasing the air injection rates led to higher bitumen recovery but lower B/S ratio of bitumen froth, i.e., poorer bitumen froth quality.

4) At higher slurry temperatures and/or higher slurry flow velocities, higher bitumen recovery and better bitumen froth quality were achieved for all three oil sands ores.

5) Increasing conditioning time and mechanical energy input to the slurry within the range of this study does not seem to resolve the difficulties of poor processing ores.

4. Tests with Denver Flotation Cell

4.1 Introduction

In Chapter 3, the effect of ore slurry conditioning on bitumen recovery and bitumen froth quality was investigated using three types of oil sands ores tested in the LHES loop. Denver flotation cell has been used as a batch extraction unit to test the processability of oil sands ores (Kasongo, et al., 2000; Zhou, et al., 2004b; Liu, et al., 2004b; Ding, 2006). Its configuration allowed convenient parameter studies and accurate control of slurry temperature, slurry density, slurry stirring speed, slurry conditioning time, air injection rate and time, and water chemistry such as slurry pH. It was suggested that a modified Denver flotation cell be more sensitive to detect the effect of operating parameters on bitumen recovery than traditional BEU for tests conducted at temperatures lower than 50 °C (Kasongo, et al., 2000; Zhou, et al., 2004b).

In this chapter, the effect of the slurry conditioning was studied using the Denver flotation cell. The same process water, and the same good (AL711) and poor (Posyn) processing ores as used in LHES tests were used. In this study, the Denver flotation cell was further modified to study the effect of slurry conditioning on oil sands ore processability. Tests were conducted in a modified Denver flotation cell without addition of chemical aids to the slurry.

4.2 Experimental

4.2.1 Materials

The same good (AL711) and poor (Posyn) processing ores as used in LHES tests were used. Their compositions are given in Table 3.1. The same Aurora Mine process water from Syncrude Canada Ltd. (Table 3.2) was used in the Denver flotation cell tests.

4.2.2 Flotation Apparatus

A modified Denver flotation cell was used in the bitumen extraction tests. The slurry temperature was controlled by using a 1-liter jacketed stainless steel cell connected to a thermal water bath. A stainless steel lid was designed and machined to cover the top of the Denver flotation cell when necessary. The steel lid could slide along the shaft to cover the cell in slurry conditioning step or to open the cell in air injection step. The lid was designed in such a way that when the cell was closed, there was no air gap between the lower surface of the lid and the pulp, i.e., the lower surface of lid touched the pulp when not under being stirred. This design reduced entrainment of air from pulp surface, allowing control of air availability to oil sands slurry during the conditioning step. A schematic of the modified Denver flotation cell is shown in Figure 4.1. A photograph is shown in the Figure F.1 of Appendix F. Air could be introduced into the slurry through the hollow stand shaft. When injecting air for flotation, the lid was lifted to open the cell. The air injection rate was precisely controlled and monitored using a digital flow meter and a needle valve. The stirring speed of the rotating shaft rotor was able to be set to the desired value between 900–3000 rpm.

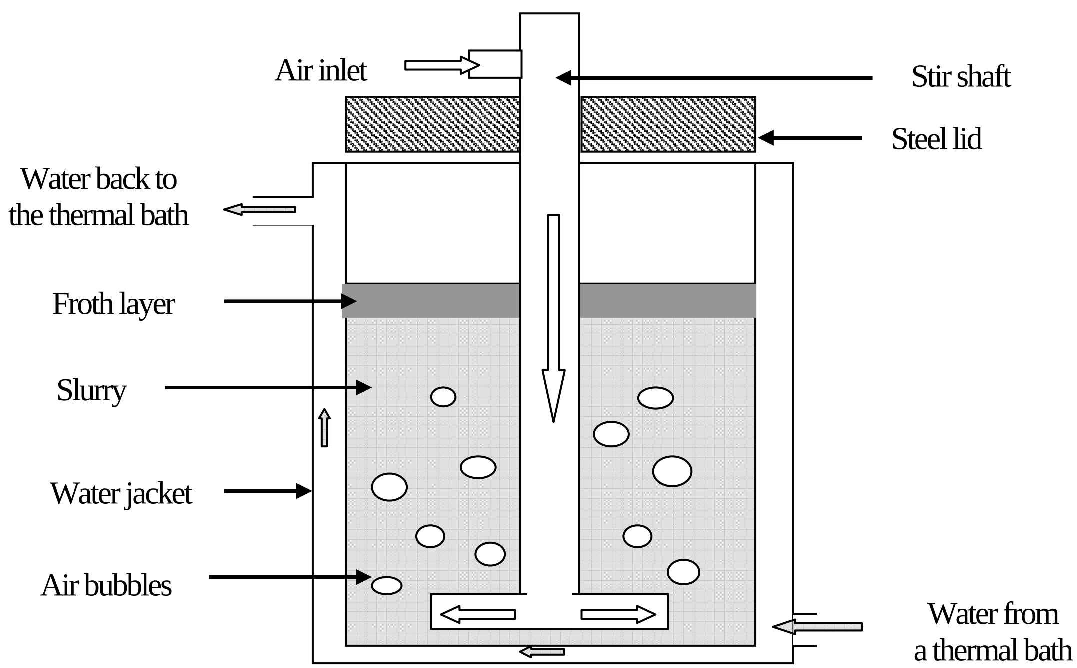


Figure 4.1 A schematic of the modified Denver flotation cell.

4.2.3 Procedures

Before extraction test, the water bath and the temperature control of the Denver flotation cell were pre-tested. Before each test, the shaft and the rotor were cleaned with toluene, n-hexane and then hot water. The stirring speed was set to a desired value.

A bag of oil sands ore was freshly thawed at room temperature. Precisely weighed 300 g oil sands ore were added to 950 mL process water in the 1-liter Denver flotation cell. The water was pre-heated to 4 ºC higher than the test temperature. And the Denver flotation cell was then covered with the cell lid and sealed with duct tape if required. The oil sands ore slurry was stirred at a given speed. After the slurry was conditioned for a given time, recorded as slurry conditioning time, the cell lid was lifted up along the shaft from the cell. Air was then introduced continuously into the slurry at a given flow rate. Under continuous air injection bitumen froth was collected into pre-weighed jars over specified intervals up to 20 min, reported as flotation time.

The bitumen froth collected in the jars was put aside for the analysis on its content of bitumen, solids and water, which was determined using the standard Dean Stark method (see section 3.2.3 and Appendix A for details). The bitumen recovery and bitumen to solids (*B/S*) ratio of bitumen froth were calculated using Equations (1-3) and (3-6), respectively.

4.3 Results and Discussion

4.3.1 Effect of Air Entrainment through Denver flotation cell

It has been shown that the amount of air available to oil sands slurry during conditioning significantly affects bitumen recovery. The studies of Flynn, et al. (2001) and Friesen, et al. (2004b) showed that the amount of air available by entrainment played an important role in bitumen recovery tested in a sealed jar and an open small glass cell. In the present study, oil sands ore extraction tests were conducted to determine the impact of two different modes of conditioning in the Denver flotation tests: a) cell open to air, b) cell covered and sealed with duct tapes. The good processing oil sands ore (AL711) was tested at 45 °C and 1500 rpm. The slurry conditioning time varied from 0, 5, 10, to 30 min. After conditioning, the lid was lifted up and air was injected into the conditioned slurry for 20 min at 150 mL/min. Bitumen froth was collected separately for flotation time intervals of 5 min and 20 min.

The bitumen recovery and *B/S* ratio of bitumen froth are shown in Figure 4.2. Figure 4.2 (a) shows that for both flotation times of 5 min and 20 min a higher bitumen recovery was achieved in the case that the Denver flotation cell was open to air than for the case when it was sealed with the lid and tape during the conditioning step. Compared with the case of cell-sealed conditioning, for instance, bitumen recovery over 5 min flotation time increased by about 5% and 35% with open cell conditioning for 5 min and 30 min, respectively. The increase in bitumen recovery in the case of open cell conditioning was attributed to the additional air available for entrainment during conditioning. However, for 20 min flotation, no significant difference in bitumen recovery was observed between the two cases of open-cell and sealed cell conditioning. The bitumen recovery for both cases was high up to 95%. This implies that in the current Denver flotation cell flotation tests, air availability during conditioning had negligible effect on bitumen recovery provided that a sufficient flotation time (20 min) was available.

The results in Figure 4.2 show no over conditioning for both cases. This is different from the LHES tests where over conditioning was evident with long conditioning time. The strong mechanic energy input in the slurry might be the reason to prevent the slurry from being over conditioned.

Figure 4.2 (b) also shows that *B/S* ratio in the case of open cell conditioning was higher than that of the case of sealed cell conditioning. It implies that more availability of air during conditioning resulted in a better bitumen froth quality. It is also interesting to note a slight higher *B/S* ratio of bitumen froth collected in 5 min than that in 20 min. A possible explanation to this observation could be that the bitumen froth quality deteriorated with the depletion of bitumen as flotation progressed, as often observed in mineral flotation, and known as recovery-grade trade off.

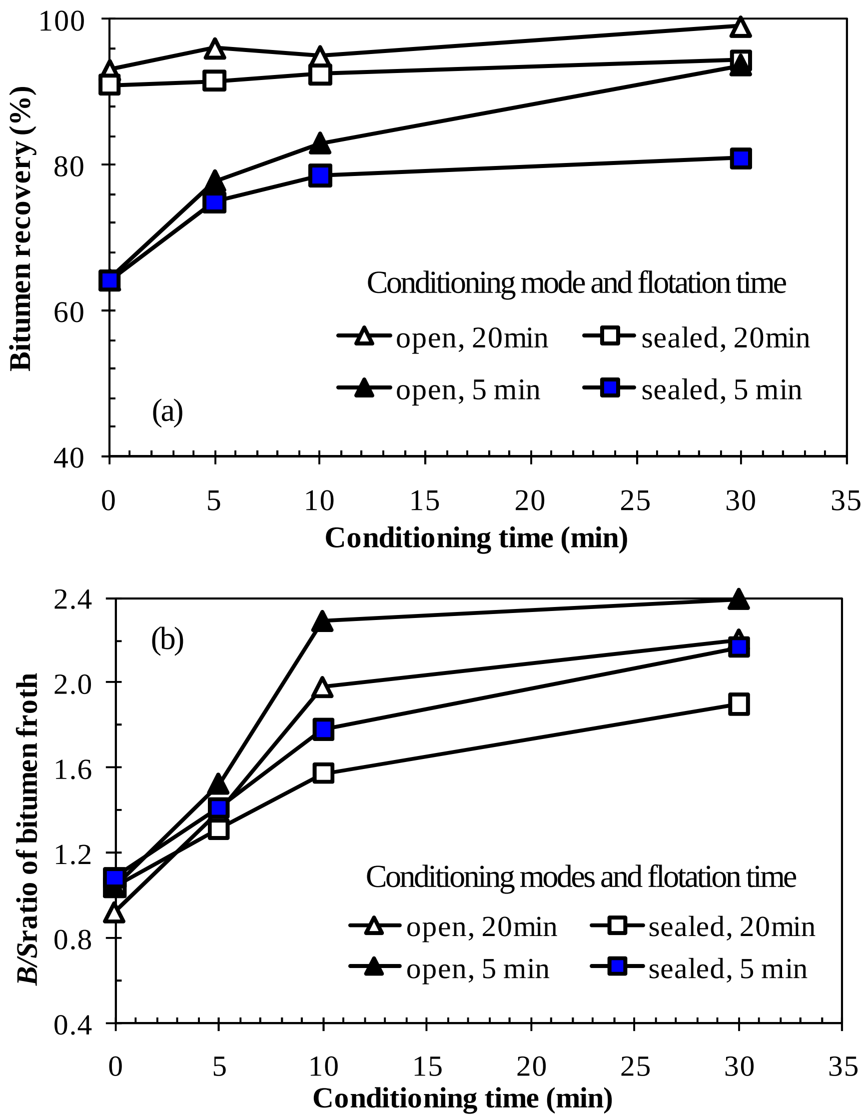


Figure 4.2 Effect of conditioning modes on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the good processing ore (AL711) tested in Denver flotation cell, using 300g ore tested at 45 °C and 1500 rpm with conditioning in open cell (triangles) or sealed cell (squares), prior to flotation at air injection rate of 150 mL/min for 5 min (solid symbols) and 20 min (open symbols).

4.3.2 Effect of Conditioning Time

Using good (AL711) and poor (Posyn) processing ores, the effect of slurry conditioning in the Denver flotation cell on bitumen recovery and *B/S* ratio of bitumen froth was studied. The test was conducted at 45 °C and 1500 rpm for a slurry conditioning duration of 0, 5, 10 and 30 min prior to flotation at air injection rate of 150 mL/min for 20 min. Denver flotation cell was kept sealed during the conditioning step.

The test results for the good processing ore for flotation time of 5 min and 20 min are shown in Figure 4.3. Figure 4.3 (a) shows that, for a flotation time of 5 min, higher bitumen recovery was achieved for longer slurry conditioning time. The bitumen recovery increased from 64% to 80% with increasing slurry conditioning time to 10 min. By contrast, for 20 min flotation time, increasing slurry conditioning time showed a negligible effect on bitumen recovery. The effect of slurry conditioning time on *B/S* ratio in Figure 4.3 (b) shows that increasing slurry conditioning time from 0 min to 30 min increased *B/S* ratio of bitumen froth significantly from 1.0 to around 2.0 for flotation time of both 5 min and 20 min. This observation indicates that longer slurry conditioning time was beneficial to improving bitumen froth quality. With intense mechanic energy input like in the current test, longer conditioning time might help to produce more bitumen droplets at recoverable sizes. When air was added to the well-conditioned slurry, comparatively less sand would be entrained into the aerated bitumen aggregates and less fines would be attached to air bubbles, minimizing their reporting to the froth. As a result, better quality bitumen froth was produced.

The test results of the poor processing ore (Posyn) are presented in Figure 4.4. Figure 4.4 (a) shows that, for 5 min flotation time, the bitumen recovery increased significantly with slurry conditioning time up to 20 min studied. For 20 min flotation time, bitumen recovery shows no significant increase with increasing slurry conditioning time. The highest recovery obtained was less than 75%. It implies that increasing conditioning was not a good solution to recovering bitumen from the poor processing ore.

It is interesting to note that the slurry conditioning time had similar effect on bitumen recovery for both good (Figure 4.3 (a)) and poor (Figure 4.4 (a)) processing ores. Higher bitumen recovery over the test conditions for the good processing ore was achieved than for the poor processing ore as expected. The comparison is shown in Figure G.1A of Appendix G.

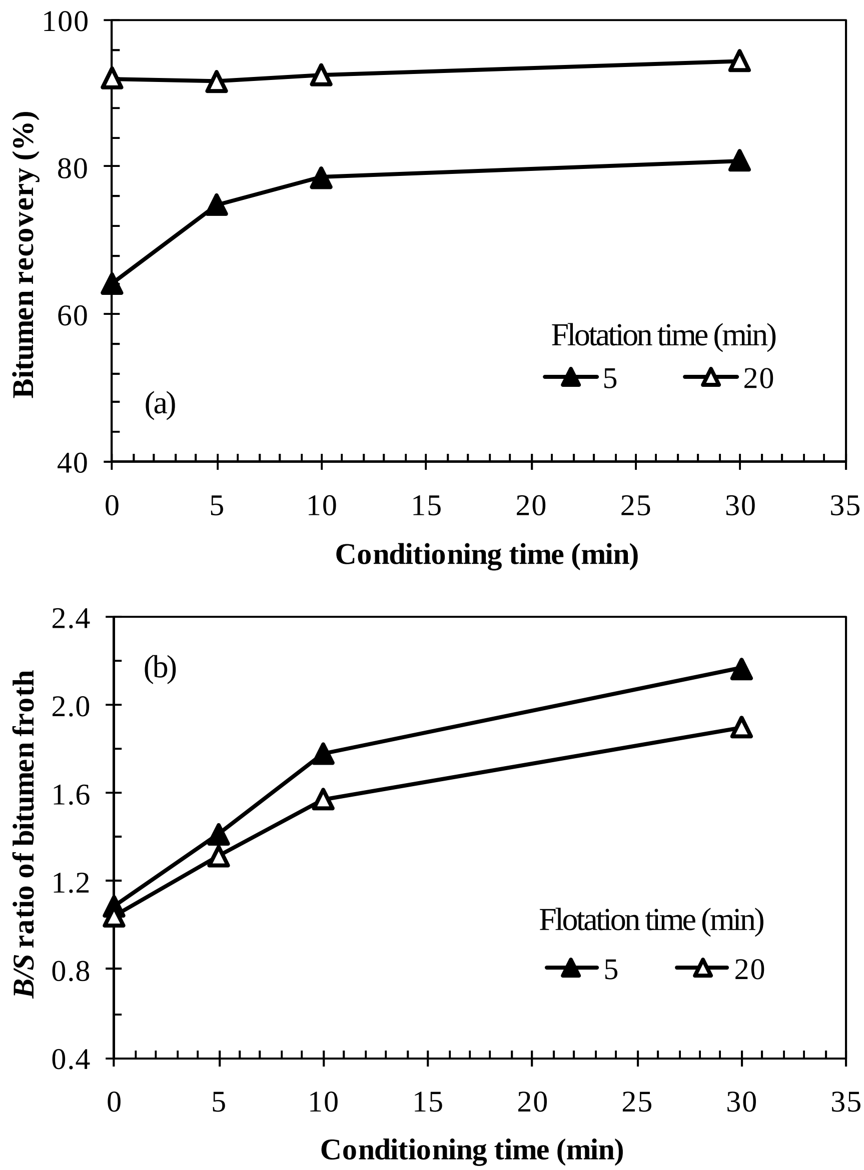


Figure 4.3 Effect of conditioning time on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the good processing ore (AL711) tested in Denver flotation cell, using 300 g ore tested at 45 °C and 1500 rpm and with sealed cell conditioning prior to air addition at 150 mL/min for 5 min (solid triangles) or 20 min (open triangles).

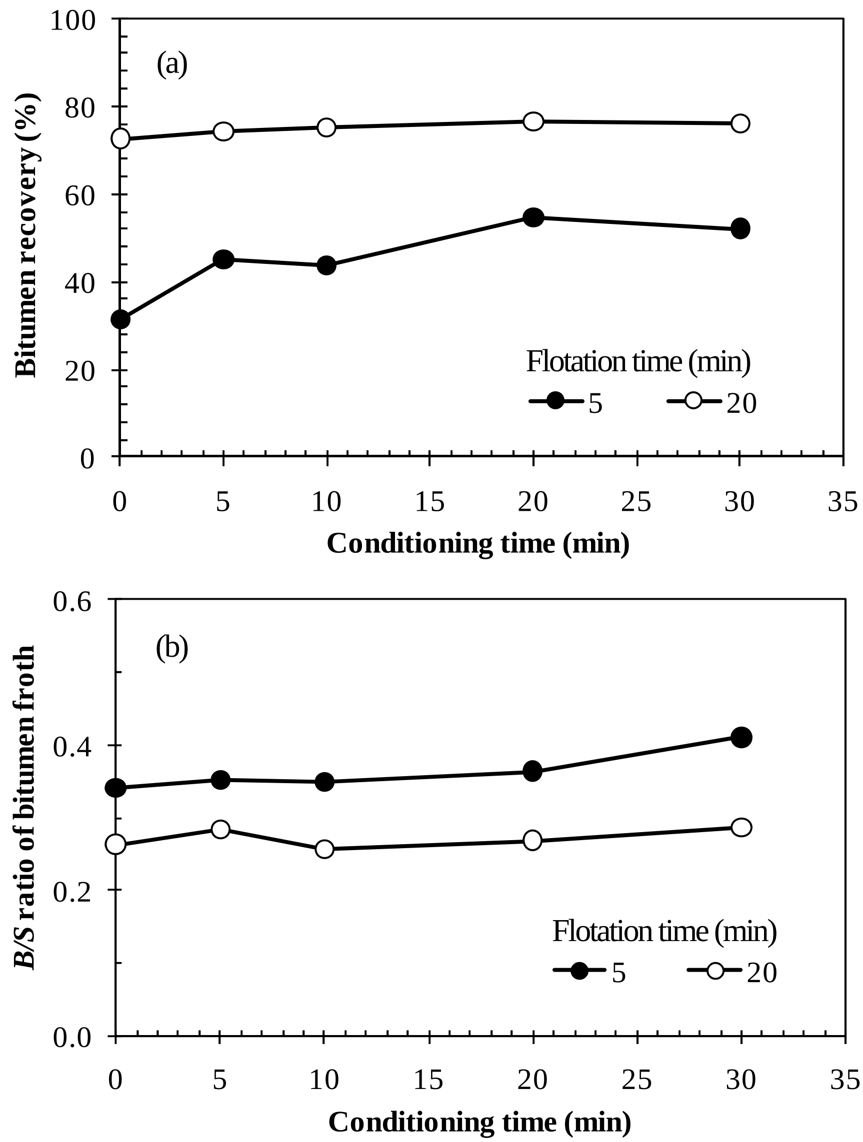


Figure 4.4 Effect of conditioning time on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth, for the poor processing ore (Posyn) tested in Denver flotation cell, using 300 g ore tested at 45 °C and 1500 rpm and with conditioning in sealed cell prior to flotation at air injection rate of 150 mL/min for 5 min (solid circles) and 20 min (open circles), respectively.

Figure 4.4 (b) shows the effect of conditioning time on *B/S* ratio of bitumen froth for the case of poor processing ore tested in the Denver flotation cell. For the poor processing ore, the *B/S* ratio remained almost constant with increasing slurry conditioning time. In contrast, *B/S* ratio of bitumen froth in the case of good processing ore increased with increasing slurry conditioning time (Figure 4.3 (b)). It is important to note that the *B/S* ratio was much lower for the poor processing ore (<0.4) than for the good processing ore (>1.7). The fact that bitumen in poor processing ore is hard to liberate may explain this opposing effect of conditioning time on bitumen froth quality for these two ores. Direct comparison of the effect of conditioning time on bitumen froth quality of the two oil sands ores is shown in Figure G.1B of Appendix G.

Compared with the tests in the LHES loop for both good (Figure 3.8) and poor (Figure 3.9) processing ores, the results obtained in the Denver flotation cell show very different effect of slurry conditioning time on bitumen recovery and bitumen froth quality. In the LHES tests, increasing slurry conditioning time decreased bitumen recovery for long flotation time and little change for short flotation time for both ores. The *B/S* ratio of bitumen froth increased with slurry conditioning time for the good processing ore (Figure 3.8 (b)) whereas no improvement for the poor processing ore (Figure 3.9 (b)). In the Denver flotation cell tests, increasing slurry conditioning time led to slight increase in bitumen recovery for long flotation time but led to significant increase for short flotation time for both good (Figure 4.3 (a)) and poor (Figure 4.4 (a)) processing ores. And *B/S* ratio of bitumen froth improved for the good processing ore (Figure 4.3 (b)) but no change for the poor processing ore (Figure 4.4 (b)). The different results achieved using the two systems could be elucidated as follows. In the Denver flotation cell tests the stronger slurry turbulence produced by higher energy input of mechanical stirring might help to prevent the formation of too large bitumen droplets and to clean bitumen droplets from slime coating, both facilitating bitumen aeration. Therefore, bitumen recovery was higher in Denver flotation cell tests than in the LHES tests. This intense mechanical stirring helps explain why bitumen recovery did not decrease with slurry conditioning time in the Denver flotation cell.

4.3.3 Effect of Air Injection Rate

To investigate the effect of air injection rate on oil sands slurry over-conditioning, both good (AL711) and poor (Posyn) processing ores were tested in the Denver flotation cell. The tests were conducted at slurry temperature of 45 °C and stirring speed of 1500 rpm. The slurry was conditioned for different time prior to flotation for 20 min at air injection rate of 75 mL/min and 150 mL/min, respectively. The Denver flotation cell was covered with its lid and sealed during the conditioning step. The results are shown in Figure 4.5 for the good processing ore and in Figure 4.6 for the poor processing ore.

Figure 4.5 (a) shows that, for the good processing ore of short flotation time (5 min), bitumen recovery increased with increasing conditioning time, although the increase was less significant at higher air injection rate. In general, for a given conditioning time, higher air injection rate produced higher bitumen recovery over a given flotation time. The effect of air injection rates on bitumen recovery became less significant for longer conditioning time (30 min). For 20 min air injection (flotation), the effect of slurry conditioning time on bitumen recovery was negligible for both air injection rates. Again high air injection rate led to slightly high bitumen recovery.

Figure 4.5 (b) shows *B/S* ratio of bitumen froth obtained using the good processing ore. It is evident that *B/S* ratio increases with increasing conditioning time for both air injection rates at two different flotation times. However, higher air injection rates led to slightly lower *B/S* ratio of bitumen froth, especially at longer conditioning time (30 min) and shorter flotation time (5 min). It appears that a higher air injection rate caused more mechanical entrainment of solids in the froth.

For the poor processing ore, the effect of air injection rates on bitumen recovery is shown in Figure 4.6 (a). The results in Figure 4.6 (a) show that, for a short flotation time of 5 min, bitumen recovery at air injection rate of 150 mL/min increased from 32% to about 55% with increasing slurry conditioning time from 0 to 30 min, whereas at air injection rate of 75 mL/min, bitumen recovery was around 30% and less dependent on conditioning time. For a long flotation time of 20 min, no significant increase in bitumen recovery was observed with increasing slurry conditioning time. This is very similar to the case of good processing ore (Figure 4.5 (a)). For a given conditioning time, bitumen recovery increased by about 10–15% with increasing air injection rate from 75 mL/min to 150 mL/min for the poor processing ore (Figure 4.6 (a)).

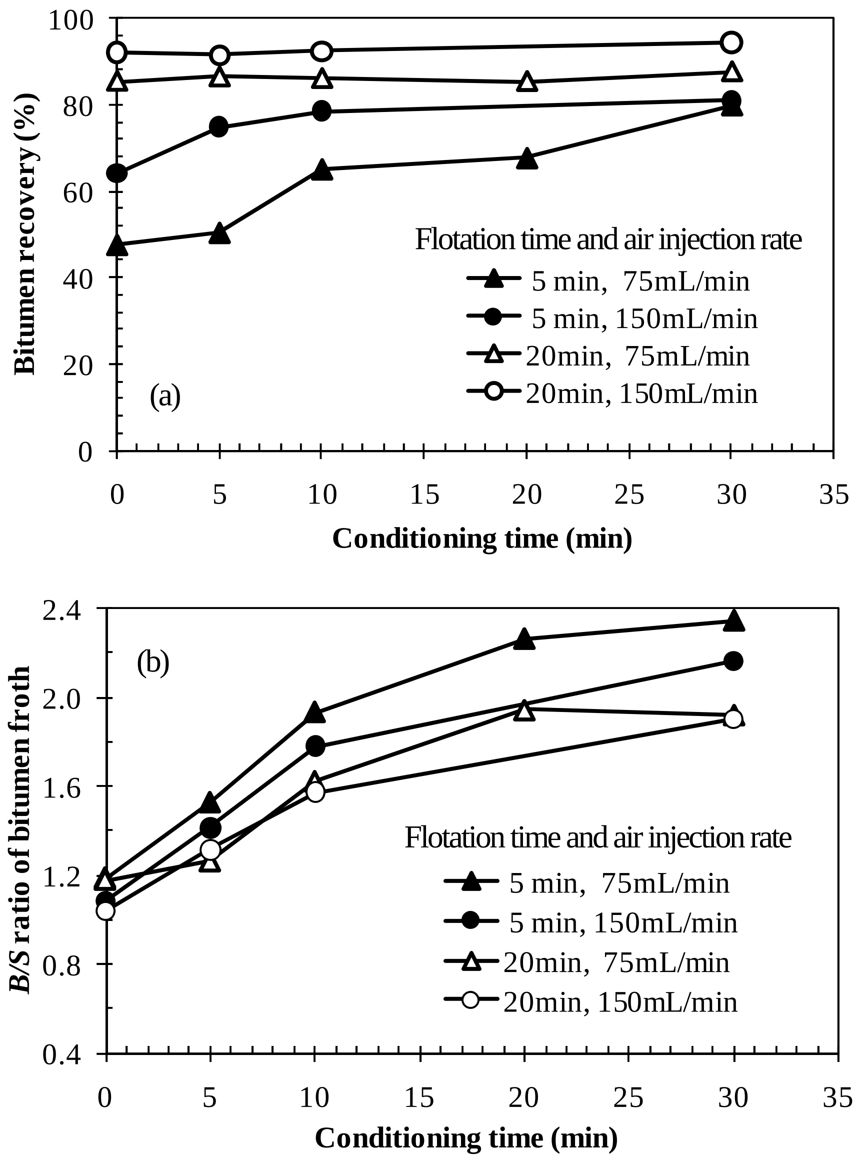
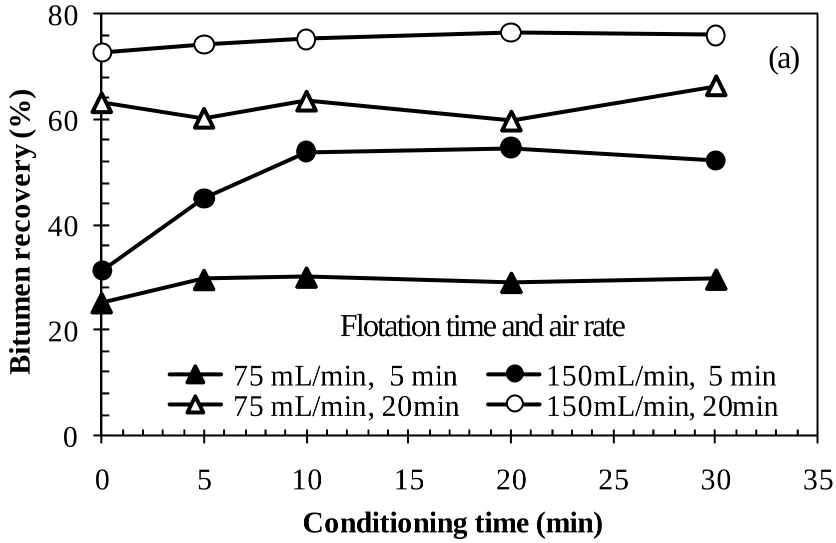


Figure 4.5 Effect of air injection rate on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the good processing ore (AL711) tested in Denver flotation cell, using 300 g ore tested at 45 °C and 1500 rpm with sealed cell conditioning prior to flotation at air injection rate of 75 mL/min (triangles) and 150 mL/min (circles) for 5 min (solid symbols) and 20 min (open symbols), respectively.



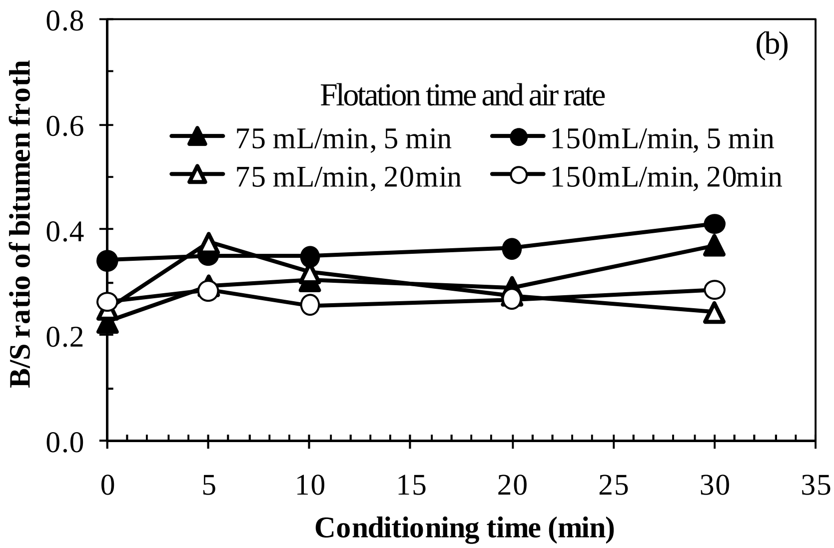


Figure 4.6 Effect of air injection rate on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the poor processing ore (Posyn) extracted in Denver flotation cell, using 300 g ore tested at 45 °C and 1500 rpm with sealed cell conditioning prior to flotation at air injection rate of 75 mL/min (triangles) and 150 mL/min (circles) for 5 min (solid symbols) and 20 min (open symbols), respectively.

Figure 4.6 (b) shows *B/S* ratio of bitumen froth from the poor processing ore. No obvious impact on bitumen froth quality was observed with increasing slurry conditioning time. This is different from the case of good processing ore (Figure 4.5 (b)), wherein the bitumen froth quality (*B/S* ratio) increased with increasing conditioning time. This difference can be explained by the bitumen liberation kinetics. Bitumen in the poor processing ore is harder and slower to be liberated during the conditioning and flotation stages than the good processing ore, thereby, more solids/fines are carried to bitumen froth with bitumen aggregates of the poor processing ore, making *B/S* ratio less dependent on bitumen recovery.

4.3.4 Effect of Stirring Speed

Mechanical energy input to slurry is beneficial to bitumen liberation and aeration (Masliyah, 2006; Stasiuk, et al., 2004; Friesen, et al., 2004b; Sanders, et al., 2007). In this study, tests on the LHES showed that the mechanical energy expressed in the form of slurry flow velocity enhanced the bitumen conditioning thereby the bitumen recovery. The improved bitumen recovery was considered to result from stronger mechanical energy, which enhanced bitumen liberation due to increased particle dispersive stresses, bitumen-air contact frequency (Sanders, et al. 2007), formation of smaller bubbles (Razzaque, et al., 2003; Friesen, et al., 2004b) and breakage of large bitumen droplets (Friesen and Dabros, 2004a). In this chapter, the effect of slurry mechanic energy imported by mechanical stirring on bitumen recovery and bitumen froth quality was tested in the Denver flotation cell using the good processing ore (AL711).

The good processing ore AL711 was tested in Denver flotation cell at 45 °C slurry temperature, 150 m L/min air injection rate and stirring speeds of 950 and 1500 rpm, respectively. The flotation cell was sealed during the slurry conditioning step prior to air injection. The results are shown in Figure 4.7. Figure 4.7 (a) shows that stirring speeds had a strong effect on bitumen recovery. When stirring speed increased from 950 rpm to 1500 rpm, the bitumen recovery increased by about 15% to 20% for 5 min air injection and about 5% to 10% for 20 min air injection. Figure 4.7 (b) shows that *B/S* ratio of bitumen froth obtained at 1500 rpm was higher by about 0.6–0.8 than that at 950 rpm. This means that the stirring speed had a strong positive effect on *B/S* ratio, i.e., bitumen froth quality improved at higher stirring speeds. Figure 4.7 (b) also shows that *B/S* ratio significantly increased with slurry conditioning time. The effect of stirring speed on bitumen recovery and bitumen froth quality observed in the

Denver flotation cell tests is very similar to the effect of slurry velocity in the LHES tests (Figure 3.16). Both tests with LHES and Denver flotation cell showed that the slurry turbulence (i.e., mechanical energy) had a significantly positive effect on bitumen recovery and bitumen froth quality.

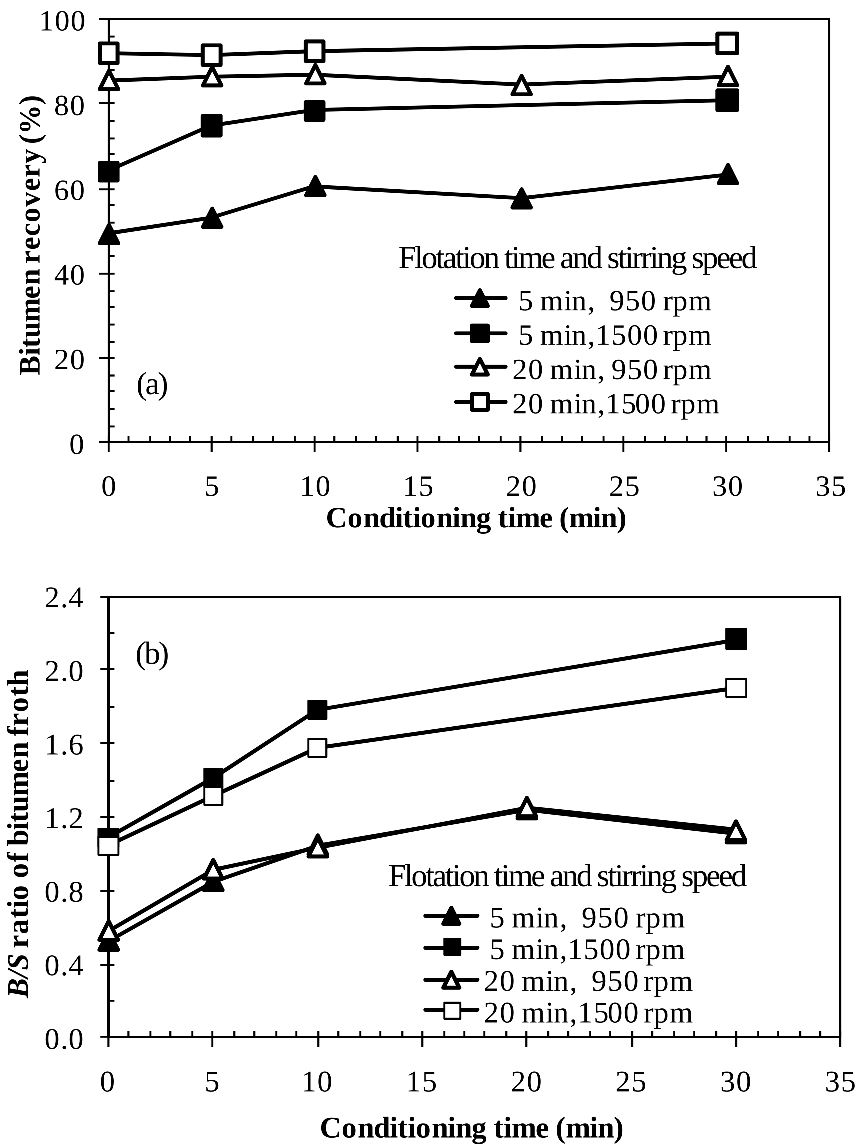


Figure 4.7 Effect of stirring speed on (a) bitumen recovery and (b) *B/S* ratio of bitumen froth for the good processing ore (AL711) tested in Denver flotation cell, using 300 g ore tested at 45 °C and 950 rpm (triangles) and 1500 rpm (circles) with sealed cell conditioning prior to flotation at air injection rate of 150 mL/min for 5 min (solid symbols) and 20 min (open symbols).

4.4 Conclusions

The effects of the flotation time, slurry conditioning time, air injection rates and slurry stirring speed on bitumen recovery and bitumen froth quality were studied in a modified Denver flotation cell using good (AL711) and poor (Posyn) processing ores. The study has shown that these operating variables had a significant effect on bitumen recovery and bitumen froth quality, in particular for the case of short flotation time. The main findings are summarized as follows:

1) Bitumen recovery increased slightly in the open cell conditioning case than the case of sealed conditioning due to additional entrainment of air into the slurry during the open cell conditioning.

2) With increasing flotation time the bitumen recovery increased for both good and poor processing ores, while bitumen froth quality deteriorated slightly for good processing ore and no impact for poor processing ore.

3) Longer sealed cell slurry conditioning time led to a higher bitumen recovery for short flotation time of 5 min and no impact for a long flotation time of 20 min for both good and poor processing ores. Higher *B/S* ratio of bitumen froth was observed only for the good processing ore.

4) Higher air injection rates produced a higher bitumen recovery but similar *B/S* ratio of bitumen froth (bitumen froth quality) for both ores.

5) Stronger mechanical energy input by higher slurry stirring speed was beneficial to bitumen recovery and bitumen froth quality.

5. Summary

5.1 Summary of Tests with the LHES Loop

1) An increase in slurry conditioning time yielded increasingly low bitumen recovery for a long flotation time (30 min). Increasing slurry conditioning time led to higher bitumen froth quality regardless of flotation time for all three oil sands ores. At high temperatures and high slurry flow velocities, the difference in bitumen recovery was not significant for the good processing and transition ores tested.

2) An increase in flotation time yielded higher bitumen recovery and improved bitumen froth quality for all three oil sands ores.

3) Higher air injection rates led to higher bitumen recovery but lower *B/S* ratio of bitumen froth (poorer bitumen froth quality).

4) Both higher slurry temperatures and higher slurry flow velocities were beneficial to bitumen recovery and bitumen froth quality for all three oil sands ores.

5.2 Summary of Tests with Denver Flotation Cell

1) Open cell conditioning yielded a slightly higher bitumen recovery than the sealed cell conditioning. During open conditioning additional air was entrained into the slurry.

2) With increasing flotation time, bitumen recovery increased while bitumen froth quality deteriorated slightly due to the rapid depletion of bitumen in the slurry with stronger mechanic energy input.

3) For a shorter flotation time of 5 min, longer slurry conditioning time resulted in a higher bitumen recovery and better bitumen froth quality for both good and poor processing ores. For a longer flotation time of 20 min, increasing slurry conditioning time had little impact on bitumen recovery but produced slightly better bitumen froth quality for good processing ore whereas no effect for poor processing ore.

4) Increasing the air injection rates led to a higher bitumen recovery but only a slight effect on bitumen

froth quality.

5) Stronger mechanical energy input at higher slurry stirring speed was beneficial to bitumen recovery and bitumen froth quality.

6. Recommendations for Future Work

1) To find an effective way to calibrate the slurry visualization system on the LHES loop. This will improve the accuracy of bitumen liberation data and ensure precise evaluation of bitumen liberation kinetics, thereby providing a better understanding of the effect of conditioning variables on bitumen liberation from sand grains.

2) To consider more variables such as water chemistry and solvent addition. It has been proven that these variables have a strong effect on bitumen extraction from oil sands ores. These variables during the conditioning period may also affect bitumen liberation, bitumen recovery and bitumen froth quality of oil sands ores.

3) To arrange tests with the Denver flotation cell in a way as follows. During the conditioning period, a small amount of air is added into the slurry for some time first, then continue the conditioning without air injection. Froth is collected only in flotation step. These tests may more effectively simulate the industrial oil sands extraction.

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Appendices

Appendix A. The Dean Stark Method

The Dean Stark Method was used to analyze the content of bitumen, water, and solids in a sample of ore or bitumen froth (Wallwork, 2003; Ding, 2006).

Preparation

Dry thimbles (150 mL), filter papers (Ø15cm), cleaned centrifuge tubes (50 mL) and jars (150 mL) in an oven under vacuum for 2 hours at 100 Celsius degree. Store dried materials except jars in dissector until needed. Cool and store jars in air before use. Defrost 1 bag of ore samples in air 2~3 hours before use.

Procedure

1. Number the dried jars, thimbles and filter papers, weigh thimbles and jars to 0.01 g, weigh filter papers to 0.1 mg.

2. Place one thimble in each 150 mL jar.

3. Collect or transfer samples of bitumen froth or ores directly to corresponding thimbles.

4. Place thimbles plus samples in Dean Stark extractors, one sample in each extractor.

5. Add around 250 mL of toluene to each extractor.

6. Connect the extractors as shown in Figure B.1, run the cooling water before heating them.

7. Reflux the extractor without overflowing the thimble for about 3 hours until toluene dripping from thimble is colourless. Collect water from the water trap of the extractor with corresponding pre-weighed jars.

8. Stop heating. Cool the extractor to room temperatures.

9. Add 3 drops of isopropyl alcohol in top of condenser to bring down any water stick to the inner surface of the condense tube of each extractor and collect it with corresponding jars.

10. Weigh the collected water in the pre-weighed jars. Substrate the tare to get the water content of the samples

11. Empty the jars. Disassemble extractor, placing thimbles in original jars and dry overnight in vacuum oven at 100 °C.

12. Remove bitumen/solvent solution to 250 mL volumetric flask, rinsing with toluene and dilute to volume after cool. Be sure not to overfill the flask.

13. Invert flask several times to suspend fine solids and then transfer 50 mL to a clean pre-weighed dry centrifuge tube. Centrifuge at 3,000 rpm for 20 minutes.

14. Using a 5 mL pipette to take 5 mL of each centrifuge supernatants and place on separate pre-weighed and numbered filter papers and dry for 15 minutes in a hood. Weigh the dried bitumen saturated filter papers to ± 0.1 mg.

15. Rinse solids remaining in centrifuge tube with toluene until clean being careful not to dislodge solids. Dry tubes in a vacuum oven at 120 °C for 1 hour and let cool in desiccators then weigh it to 0.01 g.

16. Weigh the dried and cool thimbles with solids in the jars to 0.01 g. Subtract tare to get solid content in the thimbles.

Calculation

17. Calculate the bitumen content of the sample by subtracting the bitumen-laden filter paper from the tare and multiplying by a factor of 50 (due to 250 mL dividing by 5 mL).

18. Calculate the solids content of the sample by taking the mass of the centrifuge tube plus dried solids and subtracting the tube tare, multiply this solid mass by a factor of 5 (i.e., 250 mL dividing by 50 mL, this gives the mass of the fines in the hydrocarbon mixture. Add the mass of the fines in the hydrocarbon mixture to the difference between the thimble plus dried solids and the thimble tare to get the total solids in the sample.

Appendix B. Photos of Dean Stark Device and LHES Loop

Figure B.1 Dean Stark apparatus set-up for oil/water/solids analysis (Wallwork, 2003).

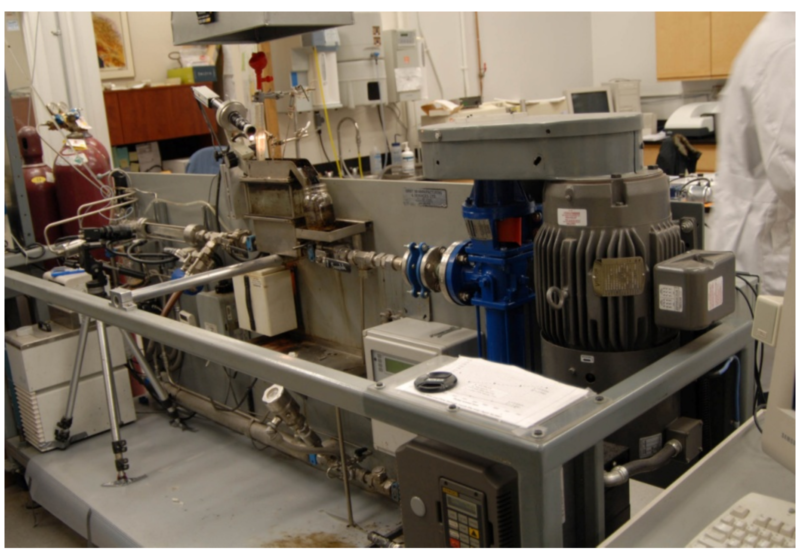


Figure B.2 Photograph of the LHES set-up.

Appendix C. Slurry Visualization Calculation Detail

The slurry visualization was used to estimate the bitumen liberation rate from the sand grains of the oil sands in the slurry (Wallwork, 2003).

Data derived from the visualisation program is scanned for outliers and these outliers are removed from the data set before it is condensed. An outlier is classified as any data point that is more than two standard deviations away from the average of the previous 10 measurements (excluding previously identified outliers). The standard deviation is calculated from the same previous 10 valid data points. From time to time the strobe that illuminates the image fails to fire and results in significantly outlying data that needs to be removed or it would skew the results.

After the data set is purged of outliers it needs to be condensed. The image analysis program analyses 15 images per second, resulting in approximately 54,000 data points in a one hour test. This is too many to plot so the data is averaged into one-minute intervals.

Appendix D. Repeatability of Bitumen Extraction Tests on the LHES Loop

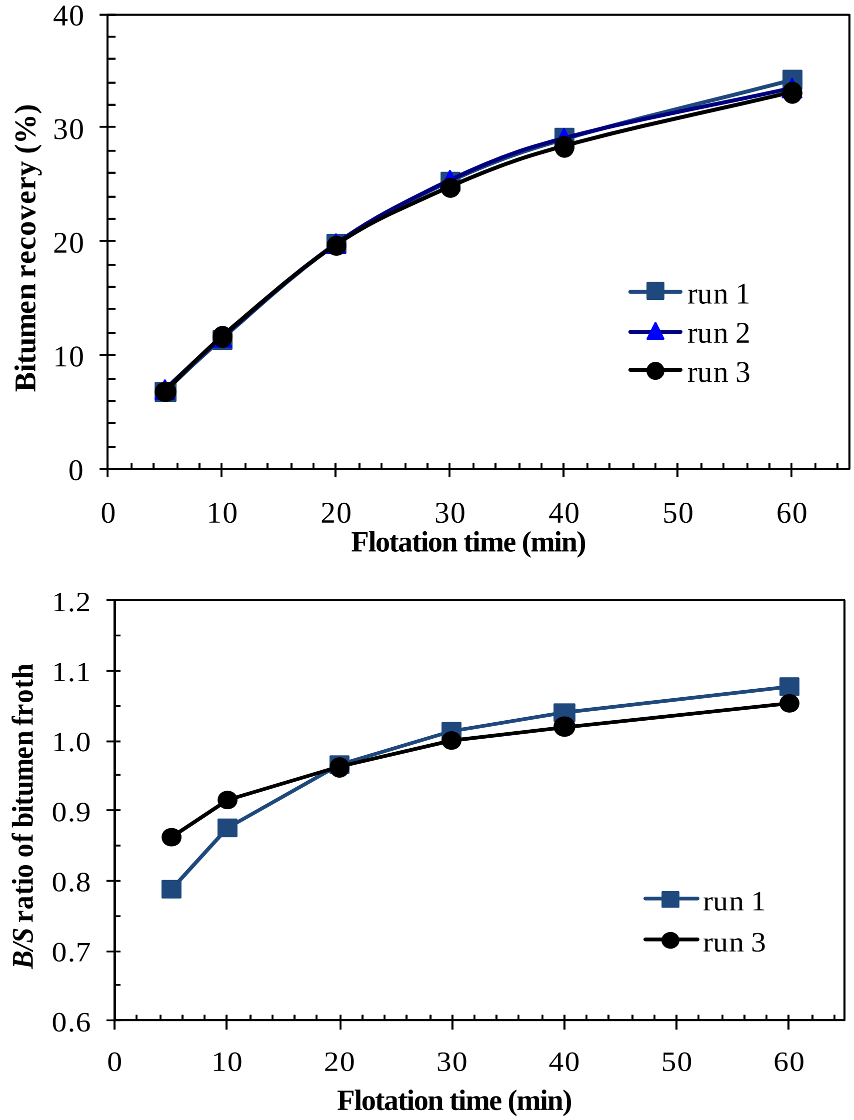


Figure D.1 Repeatability of the flotation tests on the LHES: (a) bitumen recovery and (b) *B/S* ratio of bitumen froth, using 1 kg Syn704 ore, 45 °C, 3 m/s, no conditioning prior to flotation at air injection rate of 200 mL/min.

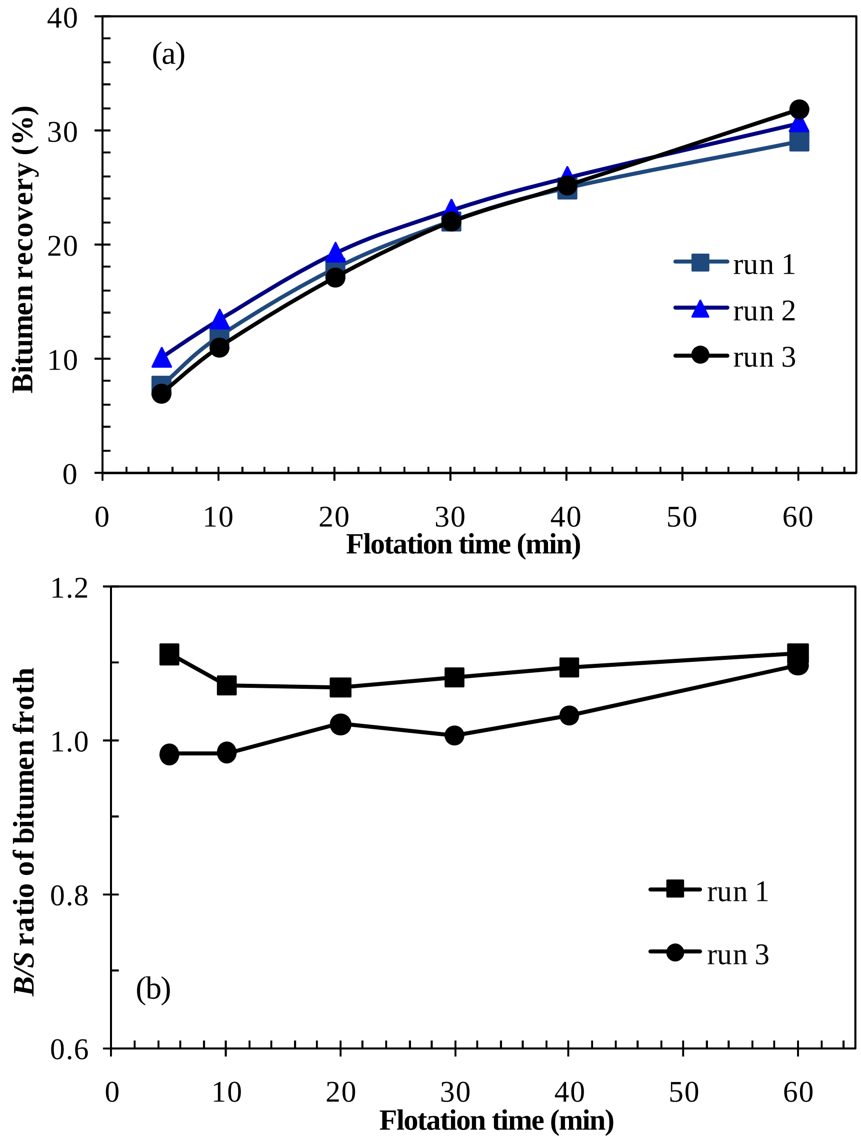


Figure D.2 Repeatability of the flotation tests on the LHES: (a) bitumen recovery and (b) *B/S* ratio of bitumen froth, using 1 kg Syn704 ore, 45 °C, 3.0 m/s, closed conditioning for 10 min prior to flotation at air injection rate of 200 mL/min.

Appendix E. Comparison of Conditioning Effect among the Three Ores Tested in the LHES

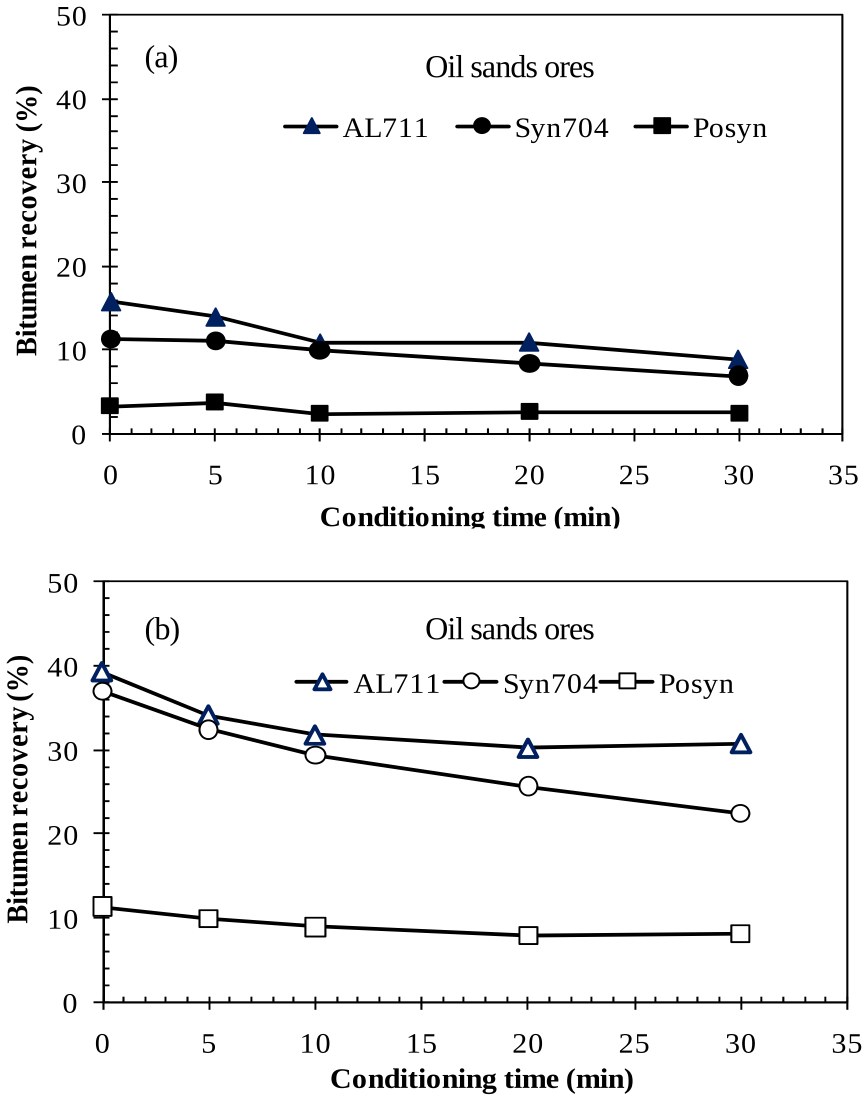


Figure E.1A Effect of conditioning time on bitumen recovery of good (AL711), transition (Syn704) and poor (Posyn) processing ores tested in the LHES at 45 °C, 3.0 m/s and 500 mL/min for flotation time of (a) 5 min (solid symbols) and (b) 30 min (open symbols).

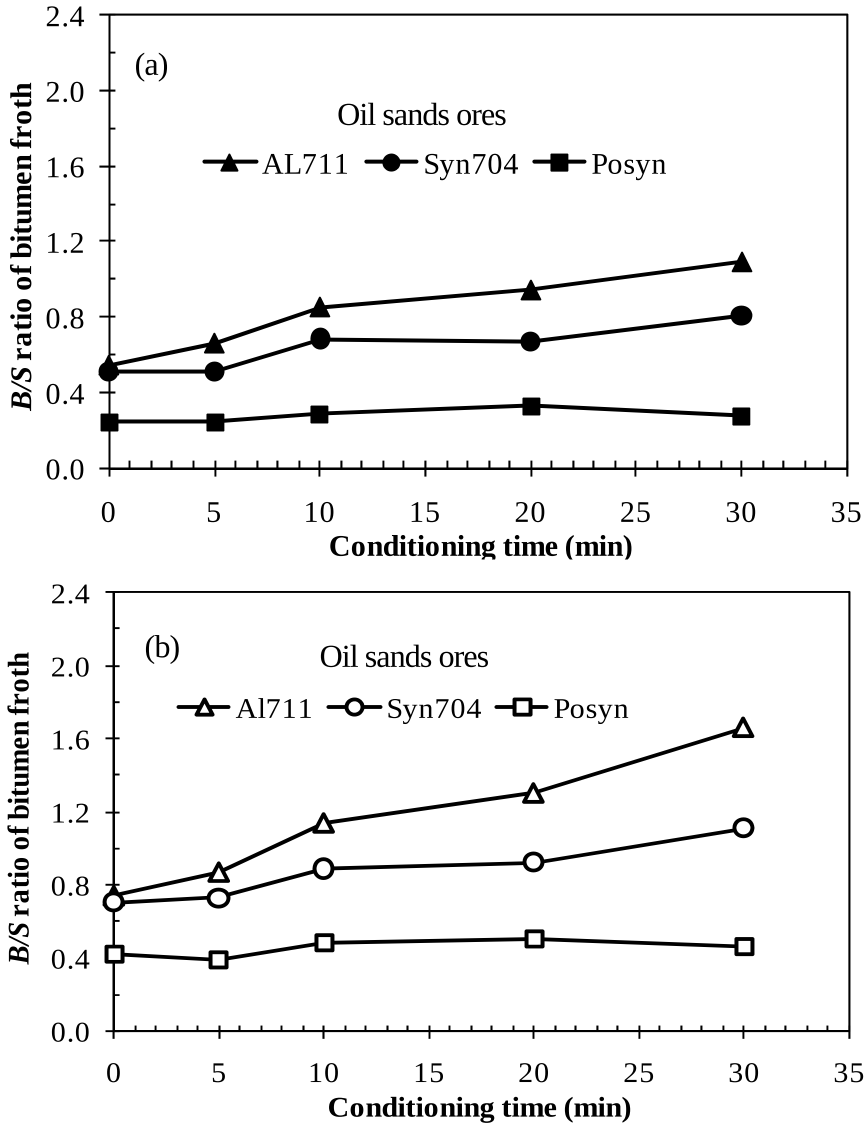


Figure E.1B Effect of conditioning time on *B/S* ratio of bitumen froth for good (AL711), transition (Syn704) and poor (Posyn) processing ores tested in the LHES at 45 °C, 3.0 m/s and 500 mL/min for flotation time of (a) 5 min (solid symbols) and (b) 30 min (open symbols).

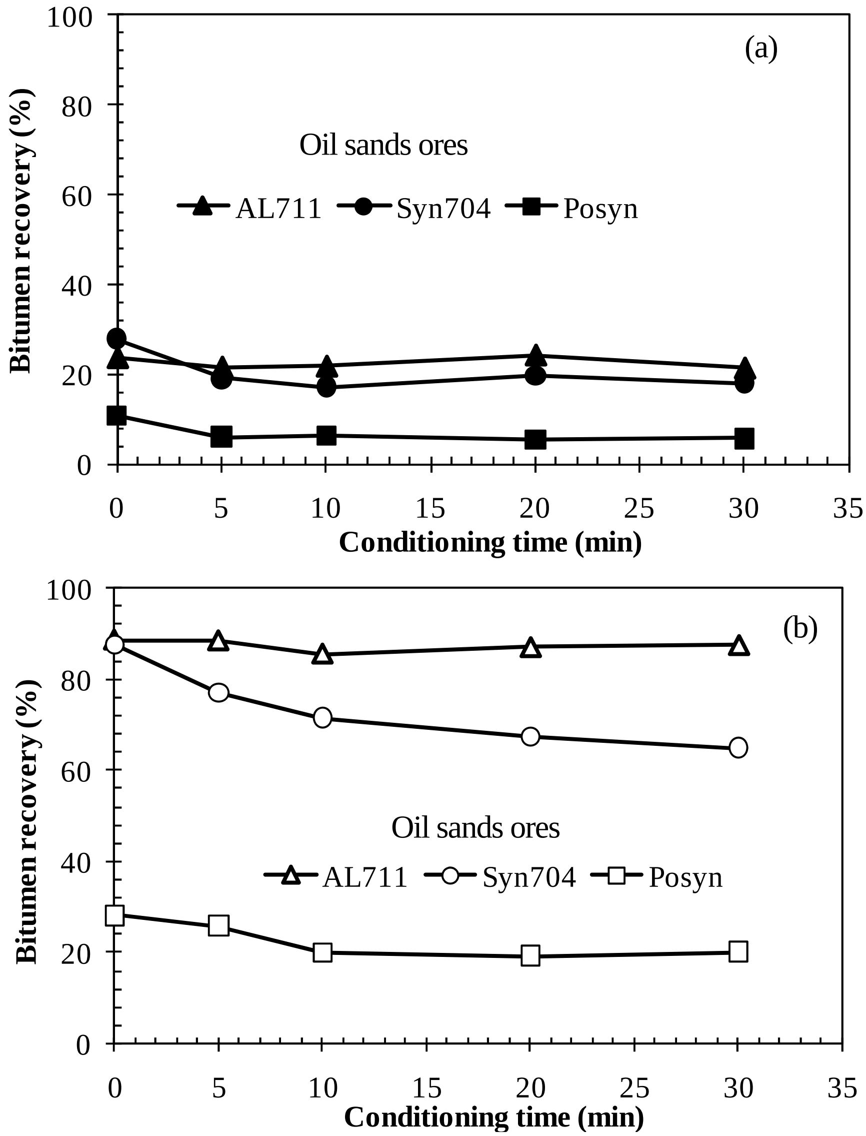


Figure E.2A Effect of conditioning time on bitumen recovery of good (AL711), transition (Syn704) and poor (Posyn) processing ores tested in the LHES at 45 °C, 4.5 m/s and 500 mL/min for flotation time of (a) 5 min (solid symbols) and (b) 30 min (open symbols).

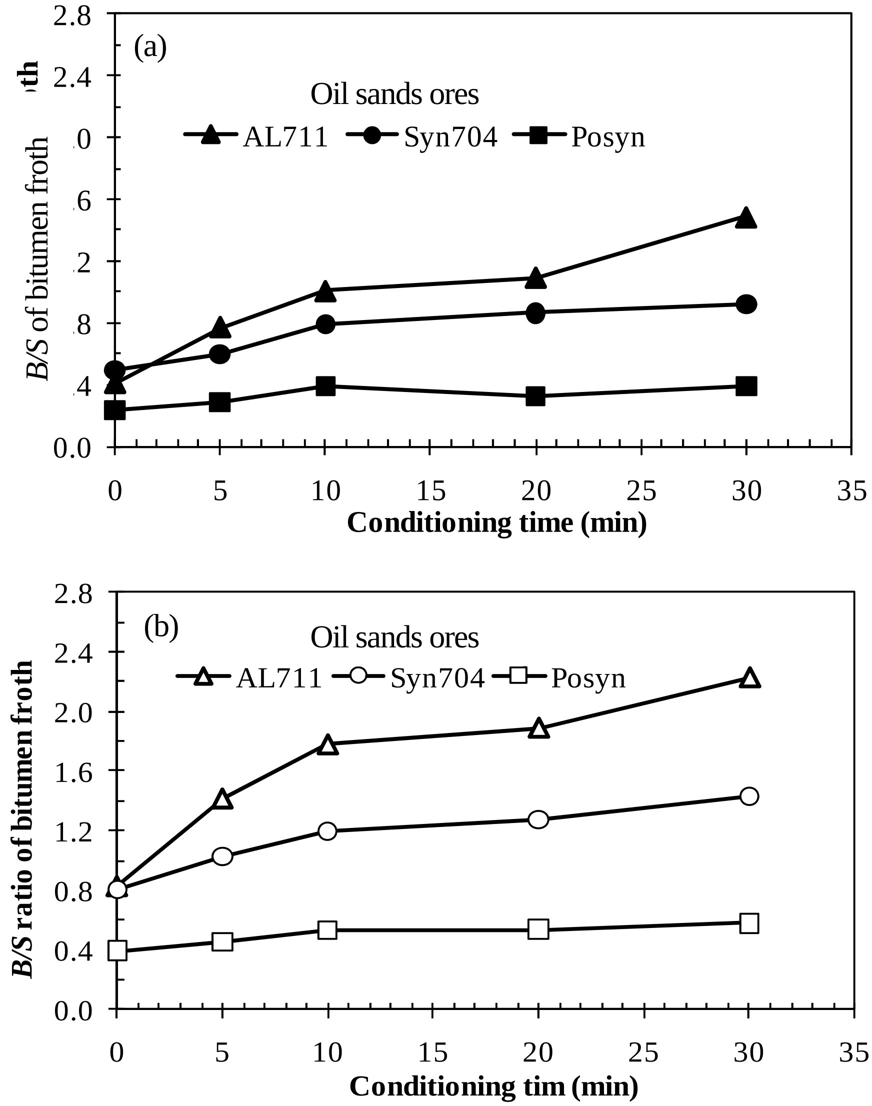


Figure E.2B Effect of conditioning time on *B/S* ratio of bitumen froth for good (AL711), transition (Syn704) and poor (Posyn) processing ores tested in the LHES at 45 °C, 4.5 m/s and 500 mL/min for flotation time of (a) 5 min (solid symbols) and (b) 30 min (open symbols).

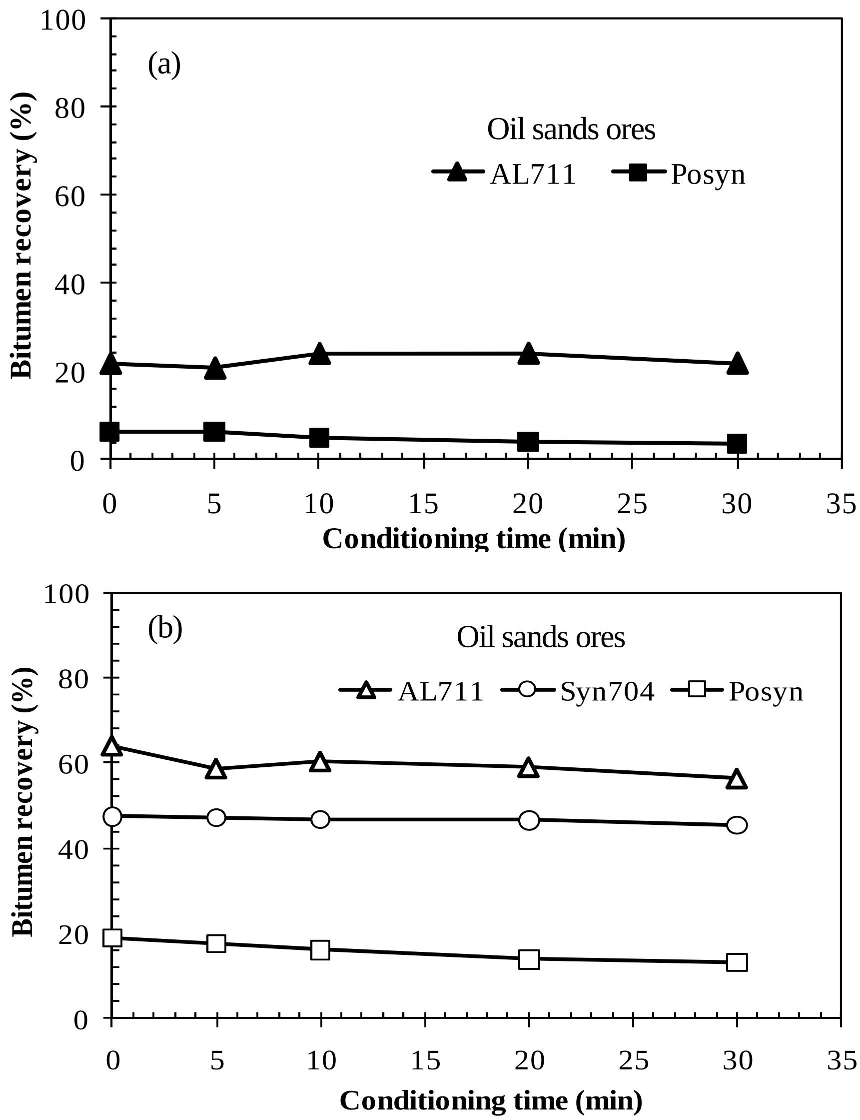
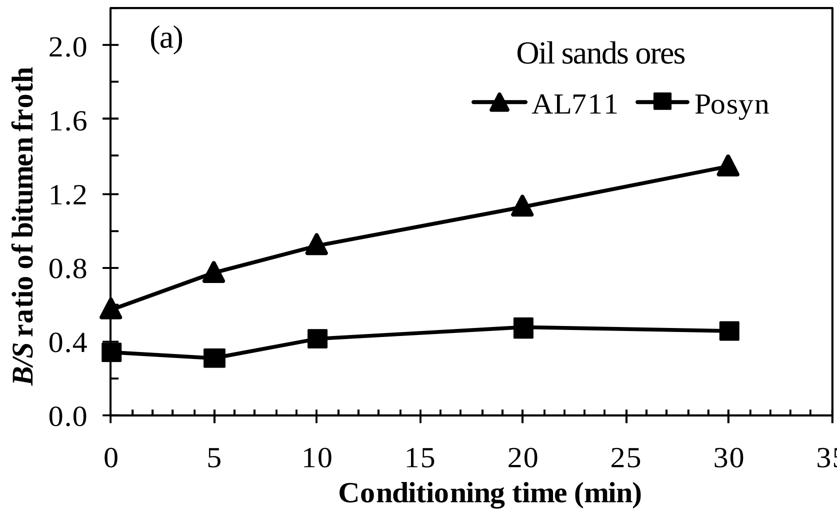


Figure E.3A Effect of conditioning time on bitumen recovery of good (AL711), transition (Syn704) and poor (Posyn) processing ores tested in the LHES at 55 °C, 3.0 m/s and 500 mL/min for flotation time of (a) 5 min (solid symbols) and (b) 30 min (open symbols).



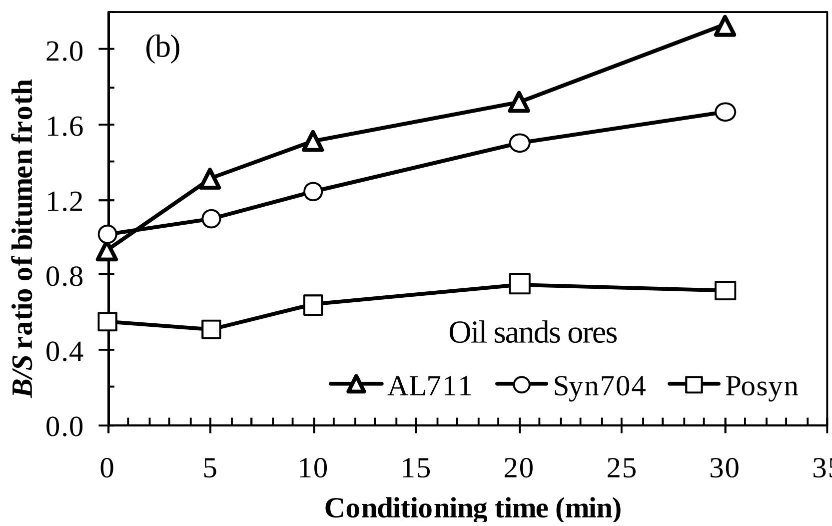


Figure E.3B Effect of conditioning time on *B/S* ratio of bitumen froth for good (AL711), transition (Syn704) and poor (Posyn) processing ores tested in the LHES at 55 °C, 3.0 m/s and 500 mL/min for flotation time of (a) 5 min (solid symbols) and (b) 30 min (open symbols).

Appendix F. Photographs of a Modified Denver Flotation Cell

Figure F.1 Photograph of Denver flotation cell set-up.

Figure F.2 Photograph of Denver flotation cell in use for slurry conditioning.

Appendix G. Effect of Conditioning on Bitumen Extraction for Two Ores Tested with Denver Flotation Cell

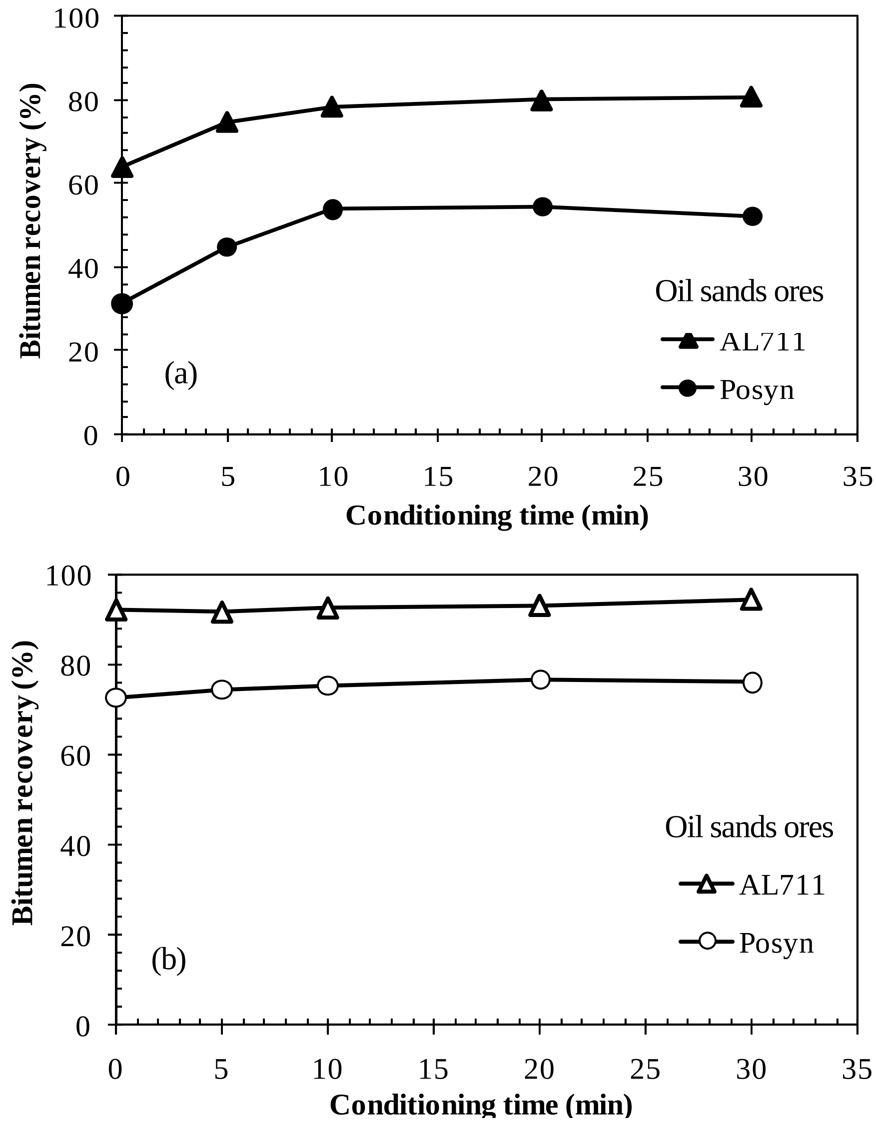


Figure G.1A Effect of conditioning time on bitumen recovery of good (AL711) and poor (Posyn) processing ores tested in Denver flotation cell at 45 °C and 1500 rpm with sealed cell conditioning prior to flotation at air injection rate of 150 mL/min for (a) 5 min (solid symbols) and (b) 20 min (open symbols).

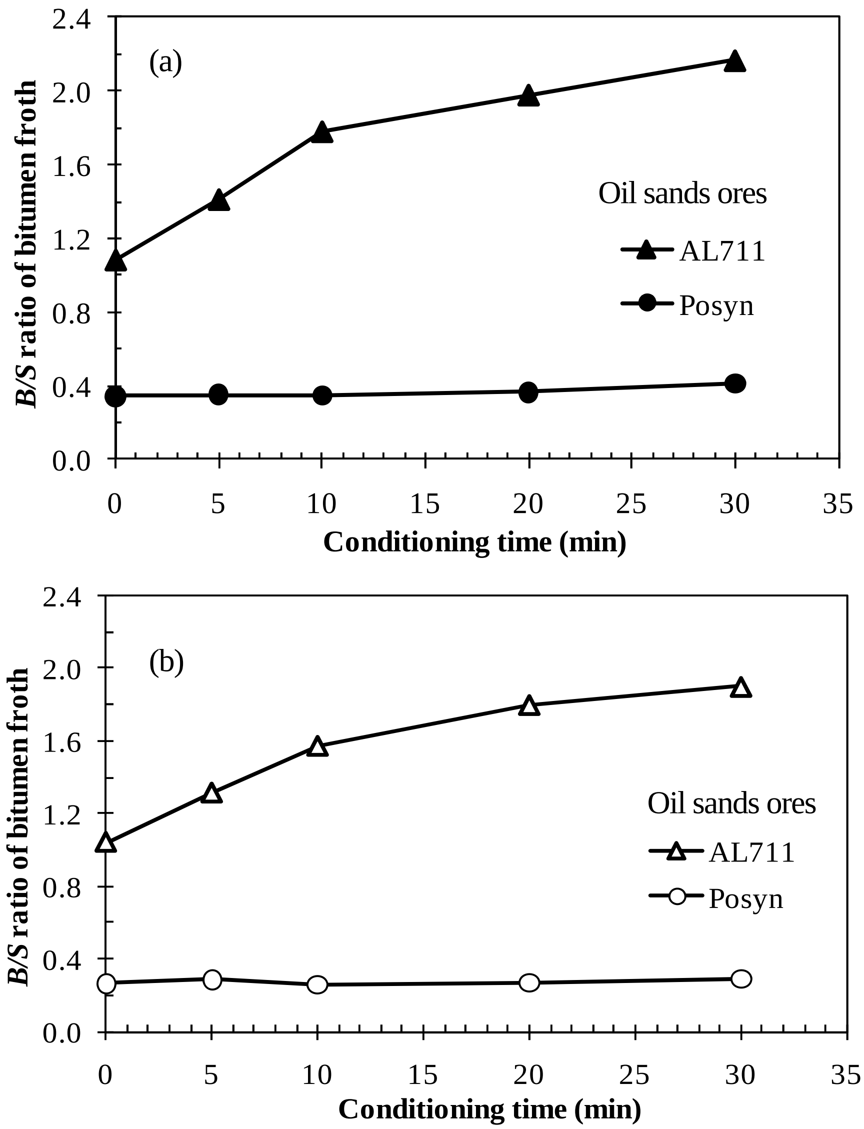


Figure G.1B Effect of conditioning time on *B/S* ratio of bitumen froth for good (AL711) and poor (Posyn) processing ores tested in Denver flotation cell at 45 °C and 1500 rpm with sealed cell conditioning prior to flotation at air injection rate of 150 mL/min (a) 5 min (solid symbols) and (b) 20 min (open symbols).

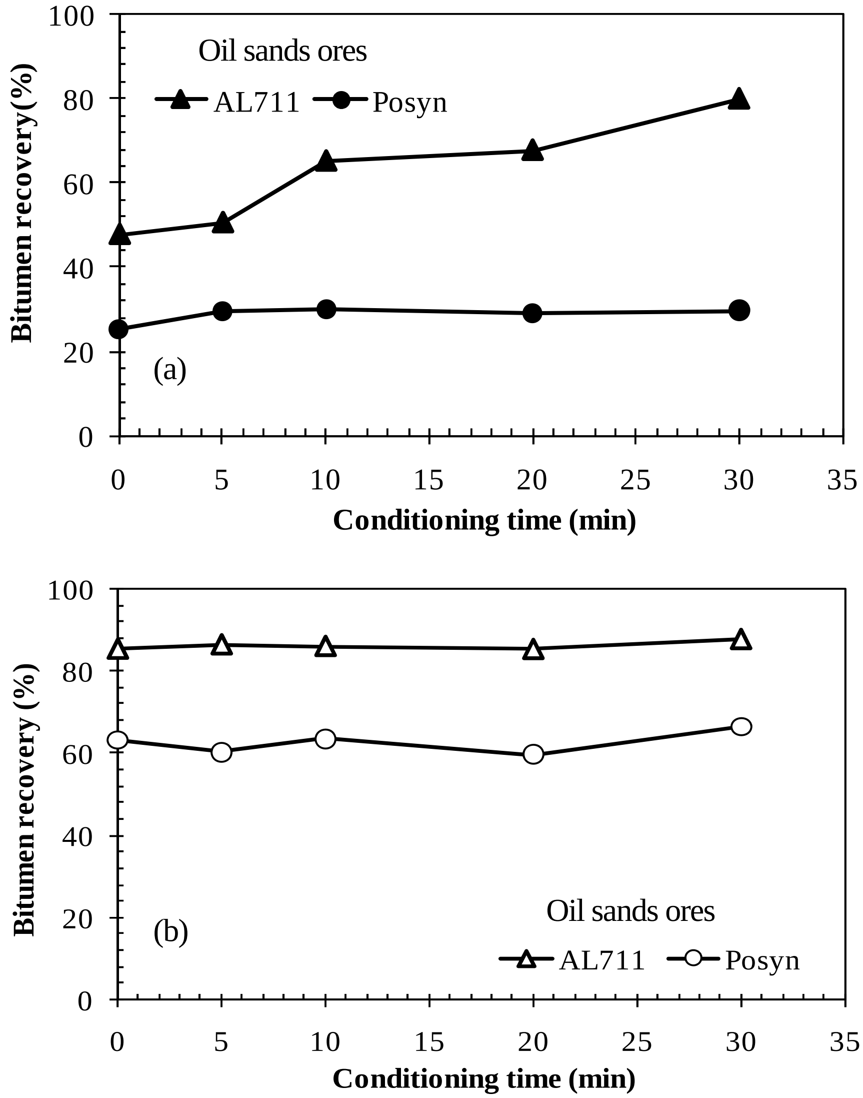


Figure G.2A Effect of conditioning time on bitumen recovery of good (AL711) and poor processing (Posyn) ores tested in Denver flotation cell at 45 °C and 1500 rpm with sealed cell conditioning prior to flotation at air injection rate of 75 mL/min (a) 5 min (solid symbols) and (b) 20 min (open symbols).

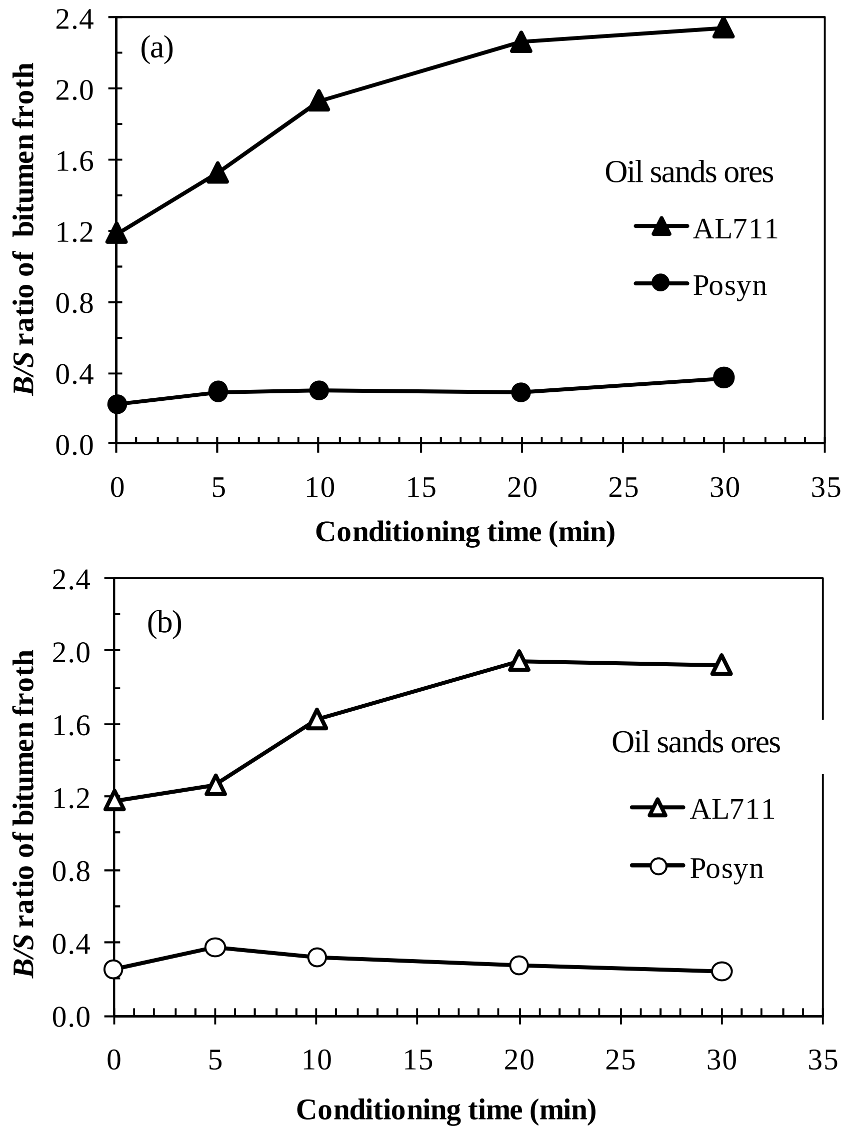


Figure G.2B Effect of conditioning time on *B/S* ratio of bitumen froth for good (AL711) and poor (Posyn) processing ores tested in Denver flotation cell at 45 °C and 1500 rpm with sealed cell conditioning prior to flotation at air injection rate of 75 mL/min for (a) 5 min (solid symbols) and (b) 20 min (open symbols).