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Factors Affecting Bitumen Recovery From Oil Sands

By

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Abstract

The oil sands deposits of northern Alberta are providing more than 15 percent of Canada's petroleum requirement. To meet this demand, the modified Clark Hot Water Extraction Process (HWEP) is used to recover bitumen from surface mineable oil sands. The critical step in the extraction of bitumen from oil sands is its separation from the particulate mineral components during a conditioning step. Traditional, simple ore characterisation methods, such as measurements of fines (< 45 μm) and clay (< 2 μm), are not always effective in predicting operational problems in this process.

The current bitumen separation process relies on the surface characteristics of the solids components. When the particles are water-wet, the transfer of mineral particles into the aqueous phase occurs easily and released bitumen separates readily because of its relatively lower density. However, the presence of oil-wet particles complicates the situation by decreasing the buoyancy of the bitumen phase. The problem of sludging in the Primary Separation Vessel (PSV) of the HWEP can also hinder bitumen separation and potentially eliminate bitumen recovery altogether. This problem is encountered when coarser mineral particles and bitumen are entrapped in the process slurry under non-segregating conditions.

Several ore components that can potentially effect processability have been previously identified, namely ultra-fine clays and organic rich solids (ORS). In this thesis the use of ultra-fines (<0.3 μm) and ORS to predict ore process problems has been explored. It was found that the ORS content of an ore is more appropriate for predicting absolute bitumen recoveries than the more commonly used fines and clay values. Critical ultra-fines and cation concentrations are shown to trigger the sludging process. However, these two components are difficult to measure and their use as a characterisation tool would probably be too expensive for general application.

In view of this problem, a simple, inexpensive evaluation procedure was developed to identify anomalous ores. This involves using a jar test to provide a qualitative assessment of the segregating-non segregating boundary conditions for bitumen separation. More extensive characterisation and testing can then be performed on problem samples.

Sommaire

Les dépôts de sable bitumineux du nord de l'Alberta génèrent présentement plus de 15% des besoins en pétrole du Canada. Pour faire face à cette demande, le «Procédé d'extraction par eau chaude » (PEEC) est employé pour recouvrer le bitume des sables bitumineux retrouvés en surface. L'étape critique du procédé d'extraction du bitume des sables est sa séparation des particules minérales durant l'étape de conditionnement. Des méthodes simples de caractérisation des minerais, telles la mesure des particules fines (< 45 μm) et des argiles (< 2 μm) ne se sont pas montrées particulièrement efficaces pour prédire les problèmes opérationnels de ce procédé.

Le procédé courant de séparation du bitume est dépendant des caractéristiques de la surface des composants. Quand les particules sont mouillées par l'eau, le transfert des particules minérales dans la phase aqueuse s'effectue facilement et le bitume libéré se sépare facilement à cause de sa faible densité relative. Cependant, la présence de particules mouillées par l'huile complique la situation en abaissant la «flottabilité » de la phase bitumineuse. Un autre problème, la formation d'un gel dans le contenant de séparation primaire du PEEC peut aussi affecter la séparation du bitume et, potentiellement, empêcher la séparation. Ce problème est souvent rencontré quand les particules minérales relativement grosses et le bitume se retrouvent emprisonnés à l'intérieur de la suspension ce qui produit des conditions ou il n'y a pas de séparation.

Plusieurs composants du minerai qui peuvent affecter la processabilité ont déjà été identifiés, tels que les particules ultra-fines et les solides riches en matières organiques. Cette thèse explore l'utilisation possible des mesures de particules ultra-fines et des solides riches en matières organiques pour prédire les problèmes pouvant être rencontrés. Il a été trouvé que le contenu en solides riches en matières organiques d'un minerai est

plus approprié pour prédire le taux absolu de recouvrement du bitume que les données sur les particules fines et les argiles. Des concentrations critiques en particules ultra-fines et en cations semblent déclencher le procédé de gélation. Cependant, ces deux composants sont plus difficiles à mesurer et leur utilisation comme méthode de caractérisation serait probablement trop dispendieuse pour des analyses routinières.

Après considération de ce problème, une procédure simple et peu coûteuse a été développée pour identifier les minerais «difficiles ». Cette méthode implique l'utilisation d'un test qui mène à l'évaluation qualitative pour déterminer les conditions limites de «ségrégation – non-ségrégation» de la suspension de minerai. Une caractérisation plus exhaustive peut ensuite être faite sur les échantillons «problématiques » pour obtenir une évaluation plus complète.

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Nomenclature

ARC	Alberta Research Council
BIT/ORS	Bitumen to Organic Rich Solids Ratio
BEU	Batch Extraction Unit
CGC	Critical Gelation Concentration
CWAT	Cold-Water Extraction Process
DLVO	Derjaguin, Landau, Verwey, Overbeek
HWEP	Hot Water Extraction Process
IOCC	Insoluble Organic Carbon Content
k	Boltzmann Constant
LUBB	Liberated-Unliberated Bitumen Balance
MFT	Mature Fine Tailings
NMR	Nuclear Magnetic Resonance
ORS	Organic Rich Solids
PSV	Primary Separation Vessel
QMA	Quantitative Mineral Analysis
R	Separation distance
T	Absolute temperature
TEM	Transmission Electron Microscopy
TIOM	Toluene Insoluble Organic Matter
V_A	Van der Waals attractive force
V_{max}	Maximum Van der Waals attractive force
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

Greek Symbols

π	pi

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Chapter 1

Introduction

1.1 General

The Canadian oil sands deposits have been well known since the first European explorers discovered bitumen seeping from the banks of the Athabasca River. The explorers, at that time, could not have predicted that this resource would be one of the largest in the world. The Athabasca region represents the biggest oil sands deposit in Alberta with an estimated in-place reserve of 869 billion barrels of bitumen; it covers approximately 46 800 square kilometers (Fine Tailings Fundamentals Consortium, 1995). Of the estimated total, 60 billion barrels are recoverable using surface mining techniques.

By 1988, the Athabasca oil sands were providing more than 15 percent of Canada's oil requirement (Fine Tailings Fundamentals Consortium, 1995). With the decline of conventional oil reserves, this percentage has inevitably grown. Consequently, this resource will prove to have increasing economic importance in Canada's petroleum future.

1.2 Geological Aspects

Geology has long been known to play a major role in the processability of oil sands. As a result, the geological aspects of Alberta's oil sands have been well documented. It was determined that the recoverable bitumen in the Alberta deposit occurs in unconsolidated sandstones of the early Cretaceous era (ca. 110 million years). Sediments were brought to the deposit by two mechanisms. In the first, an ascent in the Rocky Mountain area. provided sediments from the west that had previously been under the sea. Secondly, the erosion of the Canadian Shield provided sediments from the east (Shaw et al, 1996).

In this period, most of the sediments were deposited in a fluvial environment, although some occurred as marine deposits in northern Alberta. Fluvial sedimentation is defined as the transport and deposition of overland flows of sediment-filled water, i.e. by rivers (Finkl, 1984). Alternatively, marine sedimentation is the deposition of mineral particles eroded from continents and the biochemical precipitates from seawater. Typically, marine sediments consist of sands, silts and clays that are carried into the sea by water run-off, wind, and ice.

After the deposition of fluvial and marine sediments, the Boreal Sea shifted to the south, causing the deposition of glauconitic sandstones. After this, an inflow of sediments of volcanic origin caused the Boreal Sea to migrate to the north and northwest, producing continental-type deposition in certain areas. However, marine conditions dominated for a prolonged period in most regions. Eventually, a union of the Boreal Sea from the north and the Gulfian Sea from the southwest covered most of Alberta. Subsequently, the marine upper Cretaceous sediments were deposited.

The combination of these events has resulted in oil sands that are a mixture of different sediments covered by formations of varying thickness that do not contain oil, see Figure 1.1. Each deposit in Alberta has its own distinct depositional characteristics. The Athabasca region's distinct shallow aspect has proven to be beneficial because the ore there is recoverable by surface mining techniques. The extractable bitumen is mostly accumulated in the McMurray Formation, which lies over limestones and under marine shales. The McMurray Formation is a drainage basin filled with different sediments deposited over time. At one stage, the sea flooded, leaving behind estuarine deposits. These are sediments containing materials deposited in estuaries where flow was hindered. After this, fluvial deposits were left when the sea receded. Upon reviewing the area, it is evident that most of the sediments are the result of estuarine phases, with increasing marine influence over time. Owing to these repeated events, a range of depositions can be observed today. Consequently, the oil sands in this deposit have great diversity in their composition and properties (Shaw et al, 1996).

It is clear that oil sands deposits are diverse because of different sedimentary environments. For this reason, there are varying amounts and sizes of mineral components in ores from the various zones. Generally, the predominant mineral is quartz, present as grains, with small amounts of feldspar, mica flakes and clays (Shaw et al, 1996). The clays consist of kaolinite, illite and chlorite in different ratios. The oil sands also have varying porosity, ranging from 25 to 35 percent. The high porosity results from a low amount of mineral cements causing low consolidation and strength in the sediments. The oil saturation of oil sands is quite variable and can be as high as 18 w/w% with corresponding water contents of about 2 w/w%. As the oil level decreases the amount of water increases correspondingly.

It is not obvious why each deposit has its own distinct oil type and degree of saturation. To understand this issue, it is useful to explore the origin of the oil. Most researchers agree that at some point the oil was mobile and was able to migrate into the sediments. After this, the hydrocarbon was altered in-situ by a combination of several processes. It has been suggested that the lighter components may have evaporated or migrated away from the bulk oil. It is also possible that the lighter components may have been partially extracted by the formation water. Moreover, it has been suggested that oxidation may have occurred at shallow depths. Finally, it is also possible that anaerobic bacteria have degraded the lighter components. These mechanisms would increase the molecular weight and viscosity of the oil (Shaw et al, 1996). Any combination of these events would result in the heavy hydrocarbon, namely bitumen, visible today.

1.3 Bitumen Separation Process

Current bitumen separation processes for surface mined oil sands are based on the pioneering research of Dr. Karl Clark of the Alberta Research Council. The basis for his work originated from the observation that oil sands solids were primarily water-wet. He believed that formation water, naturally present in the oil sands, forms a film around the particles, thereby preventing the oil from adhering to the surfaces of mineral solids (Clark, 1944). The Hot Water Extraction Process (HWEP) was developed to take advantage of this oil sands characteristic. Ore is mixed with water and heat is used to lower the density of bitumen with respect to the slurry, allowing a simple settling step to be used to separate bitumen as a light froth at the surface.

Before ore can be processed in the HWEP, it must be recovered using surface mining techniques. Depending on the deposit, a certain amount of overburden material must be removed to access the oil sands ore; typically this amounts to 30 m of barren material. Then, large draglines are used to remove about 60 m of oil sands in an open pit operation. At Suncor's plant, the sands are mined by bucketwheel equipment that load the oil sands directly onto conveyors for transport to the separation plant. At Syncrude's site, draglines and bucketwheels combined with truck and shovel methods are used, see Figure 1.2. With the introduction of more mobile equipment, flexibility in mining becomes more feasible. Because the mining operation is carried out in several areas at once, problem material, identified in advance through a comprehensive core drilling programme, can be blended with better ores to reduce processing problems.

During mining, draglines dig from the bottom of the deposit upward, collecting a cross-section of oil sands and clay bands in one vertical sweep. The different ores are mixed, to a certain extent, during loading and moving to the plant. At this point oil sands is slurried with warm water in a tumbler into which pressurized steam is injected to raise the temperature to about 80°C, see Figure 1.3. Sodium hydroxide is used to bring the slurry pH into the range 8.0 to 8.5 to promote bitumen detachment from the mineral solids. This process also causes a large fraction of the clays, occurring naturally in the feed, to be dispersed throughout the pulp mixture.

The HWEP is used to recover bitumen from all types of oil sands, however the diversity of oil sands plays an important role in processing. The fines content (defined as the fraction of mineral matter less than 44 μm) has traditionally been used to characterize feed to assess its processability. Generally, high-grade oil sands containing greater than 12 w/w% bitumen have lower fines content than lower-grade material. Typically, the larger the amount of fines, the lower the recovery of bitumen in the HWEP. However, in some cases this general rule is not the case. Consequently, other ore characteristics must be involved.

Following the conditioning step, the slurry is screened to remove rocks and balled clay-rich wastes. The rejected solids, approximately 5 percent of the original oil sands, are conveyed out of the extraction plant for disposal. This slurry is then diluted with more water to a composition of about 7 w/w% bitumen, 43 w/w% water, and 50 w/w% suspended solids, which is transported to the primary separation vessel (PSV). In this vessel bitumen droplets float to the top because of the density differential at the process temperature. The finer particles and some bitumen are removed as a middlings slurry from the center of the vessel. Primary tailings, comprising coarse particles and un-recovered bitumen are withdrawn from the bottom of the vessel as a concentrated suspension. The middlings stream is transported to the secondary separation vessels, where flotation is used to remove more bitumen as a secondary froth. The rejected suspensions from each process unit are combined with the primary tailings and pumped to a tailings pond. Coarse solids are settled out to form beaches or dykes to contain the remaining fine solids suspension that flows to the center of the pond. After prolonged settling some relatively clear water is separated for recycle while the fine solids form a sludge, or Mature Fine Tailings (MFT), containing a large proportion of the process water. Bitumen froth is processed to remove air, water and solids before being upgraded to useful products.

1.4 Microstructure of oil sands

Takamura has summarized the evolution of the microstructure model of oil sands (Takamura, 1982). Before the commencement of commercial bitumen separation operations in 1967, Cottrell proposed a model for the microstructure of oil sands in terms of interactions between water, bitumen and mineral particles. He concluded that a water film of uniform thickness surrounds the sand grains while clay particles are suspended within the water phase, see Figure 1.4. Moreover, the water film is surrounded by a bitumen layer, which fills the voids between the individual sand grains (Takamura, 1982).

Later, a modified model was proposed in two independent studies by Mossop and Morgenstern. In this updated model, all the sand grains are in direct contact with one another, while the clay particles adhere directly to the sand grain surfaces (Takamura, 1982). This theory contradicts Cottrell's earlier hypothesis that the clay minerals are suspended in the water layer.

These updated models assume that the thickness of the water film is uniform. Also, the proposed models are only applicable to high-grade oil sands. Takamura has developed a more refined structural model that takes other factors into consideration. In this concept the sand grains occupy approximately 65 percent of the total pore volume. In high-grade oil sands, 10-15 percent of the pore space is occupied by formation water, while the remainder is occupied by bitumen. It was shown that water exists as pendular rings, with a thickness below 15 nm, at the contact points between the sand grains (Takamura, 1982). Low-grade oil sands contain more fines (<44 μm) than the richer ores. These fine particles exist between the coarse sand particles as clusters saturated with water. Consequently, an increase in fines content results in an increase in this so-called formation water.

Zajic proposed a conflicting description for the microstructure of oil sands. His conclusions were based on an electron microscopy study of freeze-fractured samples. In this technique samples are rapidly frozen so that water forms a glassy solid rather than crystals. The samples are then fractured using a sharp object; the fractured surface is coated with platinum and carbon, after which the sample is melted and removed. The platinum mask is then examined using transmission electron microscope (Zajic et al, 1981).

After applying this technique, electron micrographs of oil sands samples were examined. It was concluded that bitumen was present as a water-in-oil emulsion with a water droplet size usually less than 0.5 microns. The presence of water-in-oil emulsions is in addition to water wetted or pendular bonded sand grains.

1.4.1 Organic Rich Solids

A more recent study by Kotlyar has added to the complexity of the suggested structure (Kotlyar et al, 1988). It has long been recognized that oil reservoirs are not totally water-wettable and this observation is now believed to apply to oil sands. Hydrophilic inorganic minerals, such as clays, may have their surface properties changed by adsorbed organic matter. This creates a problem when oil is recovered by hot or cold water displacement processes. To understand this issue, an investigation was initiated to examine the influence of non-petroleum organic matter on bitumen recovery from different grades of oil sands (Kotlyar et al, 1988). Kotlyar used a cold-water agitation test (CWAT) to separate oil sand solids into three fractions on the basis of the amount of insoluble organic carbon content (IOCC) associated with the inorganic solids. It was determined that solids enriched with humic matter, with an IOCC of approximately 36 percent, were present in association with bitumen. On the other hand, solids with an IOCC of 5 percent or less were found in the aqueous suspension. A comparison of H/C and O/C atomic ratios for the adsorbed organic matter indicated the same origin as kerogen type III. Some of these solids were believed to report with bitumen froths in the extraction process because their surfaces are, more or less, naturally oil wetted (Kotlyar et al, 1998b).

Previously, it was generally accepted that the sand component of the ore was completely hydrophilic. Kotlyar concluded that fine solids can exist individually or as sand grain sized aggregates of smaller sand or clay particles bound together by organic and inorganic cementing agents. These clusters can range in diameter from a few micrometers to the size of coarse sand grains. Because of its high organic matter content this aggregated fraction of oil sands has been designated organic rich solids (ORS). The organic content in these solids is mainly humic matter, a dark coloured, colloidal organic material present in soils and sediments. This toluene insoluble organic matter (TIOM) can exist as free particles, as a surface layer adsorbed on individual particles or as a binding agent in the porous aggregates. The presence of humic matter provides potential sites for sorption and fixation of bitumen to the particles on both internal and external aggregate surfaces. In this work, bitumen bound to particle surfaces is referred to as "unliberated" bitumen. On the other hand, "liberated" bitumen is the hydrocarbon phase separated from the inorganic matrix by interstitial water.

Organic rich solids may play an important role in the extraction process because of their potential to report with the bitumen froth, the primary tailings or the PSV middlings, depending on their size and density. Small aggregates may report with the froth along with the liberated bitumen. Some of these particles are discarded to the centrifuge tailings during the froth cleaning process while others remain with the bitumen and adversely affect its quality. Coarser, higher density aggregates will not float with the bitumen fraction. Instead, they will remain with the middlings or settle with the primary tailings along with their associated bitumen. The behaviour of this component can therefore play a significant role in bitumen recovery.

1.5 Clay Minerals

Fine particles and particularly clays, have long been considered a significant factor in oil sands processing. Consequently, it is important to understand the behaviour of this component. The term "clay" can be used in several different contexts depending on the field of study. For example, in colloidal chemistry, the term "clay" refers to crystalline material. However, in soil science the term "clay" is often used for the soil fraction with particle diameters less than 2 μm. In this context the clay fraction of the soil also includes other minerals in the same size range (Moore, 1996). For the purpose of this discussion the term "clay" will refer to crystalline material.

In essence then, there are two distinct types of clay minerals. The first is an amorphous material found in soils derived from volcanic activity. These solids are small particles with very low crystalline structural order. The second type is represented by crystalline minerals, typical of the clay in oil sands. They resemble plate-like particles comprised of one or more unit layers. Crystalline clay units are composed of two-dimensional sheets of silicon-oxygen tetrahedra (oxygen linked to silicon) and two-dimensional sheets of aluminum- or magnesium-oxygen-hydroxyl octahedra (hydroxyls or oxygen linked to aluminum or magnesium), see Figure 1.5 (van Olphen, 1991). In the Athabasca oil sands the major clay mineral contributor with this structural characteristic is kaolinite.

1.5.1 Interaction of Colloidal Clay Particles

The properties of colloidal clay dispersions have been the subject of many studies. Colloidal clays carry a net negative charge over the basal surfaces at all pH values above two (Swartzen-Allen et al, 1973). A basal surface is the plane of a unit layer. Accordingly, basal spacing is the distance between the plane of one unit layer and the next unit layer.

Because colloidal clay minerals have a net negative charge, the net force acting between the particles is repulsive. Consequently, when suspended in pure water, clay particles move freely as separate entities, i.e., they form a stable suspension or sol. The system becomes unstable when the repulsive electrostatic charge is neutralised and the attractive force between all the atoms of one particle and all the atoms of another particle. commonly known as the van der Waals attractive force, comes into play. This attractive force results from the behaviour of the electrons surrounding a nucleus. In essence. the electrons surrounding a nucleus do not form a uniform envelope. For this reason. each atom reacts like a fluctuating dipole, resulting in charge fluctuations in the atoms. These fluctuations create a force of attraction between the atoms (van Olphen, 1991).

For two atoms, this attractive force, V_A, is inversely proportional to the seventh power of the separation distance or the sixth power of the attractive energy. However, it is important to note that the expression for the attractive force is different for platelets. Because the clay minerals in oil sands are in this form, another expression for V_A must be examined. For platelets, the attractive force is inversely proportional to the second power of the separation distance R and can be expressed as

$$V_A \quad \alpha \quad R^2 \qquad (1.5.1\text{-}1)$$

The balance of forces governing the stability of colloidal clay suspensions was first described by Derjaguin, Landau, Verwey, and Overbeek in 1941. It is commonly known as the DLVO theory. It states that the net force between colloidal particles is the combination of two opposing forces: the van der Waals attractive force and the electrostatic repulsive force. In addition, this theory demonstrates that there must be enough ions in the suspending phase to exactly balance the charged colloidal surfaces to produce an electrically neutral system.

The result of this interaction between ions and charged surfaces in an aqueous suspension is a diffuse double layer that can be described as an accumulation of counter-ions around the particles. Counter-ions have an opposite charge to that of the particle surfaces, while co-ions have the same charge as that of the particle surfaces. This accumulation occurs because the counter-ions are electrostatically attracted by the oppositely charged surface (van Olphen, 1991). In addition to this accumulation, the counter-ions are also inclined to diffuse away from the particle surfaces to a zone of lower concentration. Thus, the concentration of the counter-ions near the particle surfaces is high and decreases with increasing distance. While the counter-ions accumulate around the surfaces of the particles, the co-ions are electrostatically repelled by the identically charged surface. For this reason, there exist only a small number of co-ions close to the particles. It is commonly stated that the counter-ions are adsorbed while the co-ions are negatively adsorbed.

A schematic representation of the DLVO theory is shown in Figure 1.6. Here, a primary minimum in potential energy is seen close to the particle surface owing to the van der Waals attractive force. The potential energy then increases until it reaches a maximum, V_{max}. At this point, the electrostatic double layer produces its highest repulsive barrier. For aggregation to occur, the potential energy of this barrier must be less than 10kT, where k is Boltzmann's constant and T is absolute temperature (Popiel, 1978). The repulsion becomes less as the counter-ion concentration increases. Addition of ionisable salts to the suspending medium causes the electrostatic double layer to be compressed, allowing particles to move closer together and thereby eventually permitting the van der Waals attractive force to overcome the electrostatic repulsion. However, if the repulsive energy barrier is larger than 10kT, then the particles will not be able to approach closely enough to form aggregates. When the particles are even further apart, a secondary minimum is seen in the DLVO diagram. This occurs when the attractive force is unchanged and the repulsive potential is reduced; the interaction is not strong enough to allow particles to combine instantaneously.

Even though the van der Waals attractive force and the repulsive electrostatic force play major roles in controlling the interactions of colloidal clays, the hydration repulsion force should not be ignored. This hydration force is important at short-range (less than a few nanometers) interaction of particles. It is known that a thin layer of water a few molecules thick is strongly adsorbed at clay particle surfaces. Consequently, particle-

particle interaction is also controlled by the ease with which the water can be desorbed from these surfaces (van Olphen, 1991).

1.5.2 Structure Formation in Clay Suspensions

Flocculation, or agglomeration of clay particles is the precursor to the important phenomenon of gelation to be discussed in Section 1.5.3. Flocculation can be described as the process of aggregation between finely divided particles suspended in a liquid medium (van Olphen, 1991). The process begins when particles can approach closely enough to overcome the repulsive electrostatic energy barrier between them allowing van der Waals forces to come into play. Eventually, enough individual particles combine to form more complex structures called flocs.

As discussed earlier, flocculation can be initiated by the addition of a soluble salt to a clay suspension. Adsorbed cations from the salt neutralize the negatively charged particles so that the electrostatic repulsive force is no longer able to counteract the van der Waals attraction. First, small flocs are formed by the aggregation of individual clay particles. With time, these flocs begin to bridge together to create larger clusters. Eventually, all the flocs are linked together to form one single floc, called a spanning cluster. Because the particles are unable to flocculate further, a weak, gel-like structure is formed. Such structures exhibit thixotropic behaviour in which a gel re-forms after being destroyed by the application of shear.

To understand the fundamentals of gelation, it is important to visualize the interactions of the clay platelets, which make up the gel network. Three different modes of particle association may occur: face-to-face, edge-to-face and edge-to-edge. The different modes of association are shown in Figure 1.7.

Each mode of association will result in a different type of floc structure. A face-to-face association leads to a dense construct while edge-to-face or edge-to-edge association will lead to expanded three-dimensional structures. In the latter situations, the result is voluminous flocs that hold a large amount of water. Edge-to-face interaction produces the cardhouse structure while edge-to-edge association yields ribbons or sheets.

1.5.3 Introduction to Ultra-fine Clays

As mentioned in Section 1.3, the fines content of oil sands has been traditionally considered an important ore characteristic. However, in some cases, this measurement is not sufficient to predict oil sands behaviour. Based on this and earlier work on MFT it is now believed that ultra-fine clay particles (<0.3 μm) have a significant effect on oil sands processability and should be taken into consideration as a primary ore property (Kotlyar et al, 1992).

Numerous studies have been conducted to determine the properties of ultra-fine clay particles and their gel-forming capabilities. In particular, the gel-forming characteristics of colloidal solids present in MFT was investigated (Kotlyar et al, 1992). In this research, samples of MFT from oil sands extraction plants were examined to determine the cause of their stability. From initial observations it was apparent that the samples were thixotropic gels. Ultra-centrifugation was used to separate the samples into different particle size fractions, which were then resuspended in recycle water from the tailings pond. It is important to note that this water contains enough salt to induce flocculation of the particles. The degree of gel formation was determined visually as a function of concentration for each size fraction. A critical concentration of ultra-fines needed to form a gel was identified. The study showed that this critical gelation concentration (CGC) decreased with increasing centrifugation speed, or decrease in particle size. A critical gelation level occurs when the clay flocs begin to interact with each other to form a structured system, showing hindered settling. This phenomenon was associated with particles less than 0.4 μm in diameter. Further research has shown that the size fraction < 0.3 μm is most significant. The CGC for these ultra-fine particles was found to be only 2.5 - 3 w/w%. This size fraction can account for nearly all of the water-holding capacity of the tailings. The ultra-fine floc structure provides a support medium that entraps coarser solids to produce a non-segregating suspension.

Further research was carried out to investigate gel formation of ultra-fines using Nuclear Magnetic Resonance (NMR) techniques. This approach looked at clay-water interactions at a microscopic level and provided information on gel structure and the sol to gel transition, or the dispersed to flocculated state of particles in suspensions (Ripmeester et al, 1993). For these experiments size fractionated samples of ultra-fines were suspended in water doped with D_2O. The solids were also examined using Transmission Electron Microscopy (TEM). Some of the particles were hexagonal, but most had irregular shapes and were very thin e.g., comprising only one unit layer for the finest fraction. Particle sizes decreased from 200 nm, for a sample settled at 500 gravities, to 20 nm for samples settled at 91 000 gravities.

When the ultra-fines suspensions were allowed to gel before being placed into the NMR's strong magnetic field, the spectrum exhibited no fine structure, even after several days (Ripmeester et al, 1993). When the samples were shaken before being placed into the magnetic field the spectra showed peak splitting, characteristic of a well-dispersed particle suspension. As the suspension was allowed to gel outside the magnetic field the splitting disappeared. These results show that gels can be produced in both orientated and random configurations (Ripmeester et al, 1993). An orientated configuration is formed when the particle suspension is placed in the magnetic field before gel formation. The random configuration is formed when the particle suspension forms a gel and is then placed in the magnetic field. It has been speculated that ultra-fines from oil sands associate as sheets or ribbons.

More recently, a study was performed to determine the effect of particle size on the flocculation behaviour of ultra-fine clays in salt solutions (Kotlyar et al, 1998a). Again, samples were separated at different centrifugation speeds. Transmission electron microscopy showed that the particle thickness decreased from 8 nm for the largest (270 nm) down to 1 nm for the smallest particles (60 nm). This research also used photo correlation spectroscopy to study the flocculation kinetics of the particle fractions. It showed that particle interaction is largely dependent on water chemistry. The interaction between dissolved salts and ultra-fines is completely reversible. When particles are suspended in a salt free solution, the particles exist as a stable suspension, or sol. At low concentrations of NaCl (<5 mM), particle repulsion forces are similar to the particles' kinetic energy. While particle-particle interaction is limited, because the particles must still overcome a repulsion barrier, a slow growth in floc size nevertheless occurs. With an increase in salt concentration (10 mM), the energy barrier is more easily overcome and the aggregation rate increases. At a salt concentration of 20 mM there is little resistance to particle-particle interactions and flocs grow rapidly. In the presence of higher salt concentrations more voluminous flocs are quickly formed by diffusion limited aggregation (Fine Tailings Fundamentals Consortium, 1995).

Floc structure was also found to be dependent on particle size and particle number concentration. In the same study, the smaller particles formed larger flocs than the coarser sizes. For a certain volume concentration, the particle number concentration increases with decrease in particle size (Kotlyar et al, 1998a). As the number of microflocs per unit volume increases the probability that they will collide and adhere to each other is greater. In summary, smaller particles are capable of forming larger, less dense flocs, with a concomitant increase in water entrapment.

An important observation from this work is that flocculation rate increases rapidly with salt concentration and that under the right conditions gelation can occur within minutes. This has important implications in the application of the HWEP to certain ores.

1.6 Objectives

Many problems associated with the HWEP occur in the PSV. The result is usually a loss of bitumen recovery or, in extreme cases, complete process breakdown. Traditional ore characterisation procedures have not been completely effective in predicting these problems. Typically, expensive laboratory trials and pilot tests must be carried out to identify intractable ores. Previous work identified the role played by clay ultra-fines and organic rich solids in mature fine tailings formation. This research has been carried out to determine the role of these components in bitumen separation has been investigated.

In water based extraction processes the critical step in the separation of bitumen from oil sands' solids is the transfer of mineral particles into the aqueous phase. When the particles are water-wet, the transfer occurs relatively easily, however, the presence of organic rich solids (ORS), i.e., solids whose surfaces are strongly associated with toluene insoluble organic matter (TIOM), complicates the situation. The first objective of this research project was thus to establish a correlation between bitumen recovery and the ORS content of oil sands from different lithological facies. This information is then used to develop oil sands processability criteria.

In addition, another problem in the primary separation vessel results from PSV sludging. In this situation coarser mineral particles and bitumen are entrapped in the process slurry as a result of non-segregating settling. Under these circumstances little or no bitumen froth is generated. The second objective is thus to determine the factors involved in PSV sludging and establish a process parameter envelope to avoid this situation.

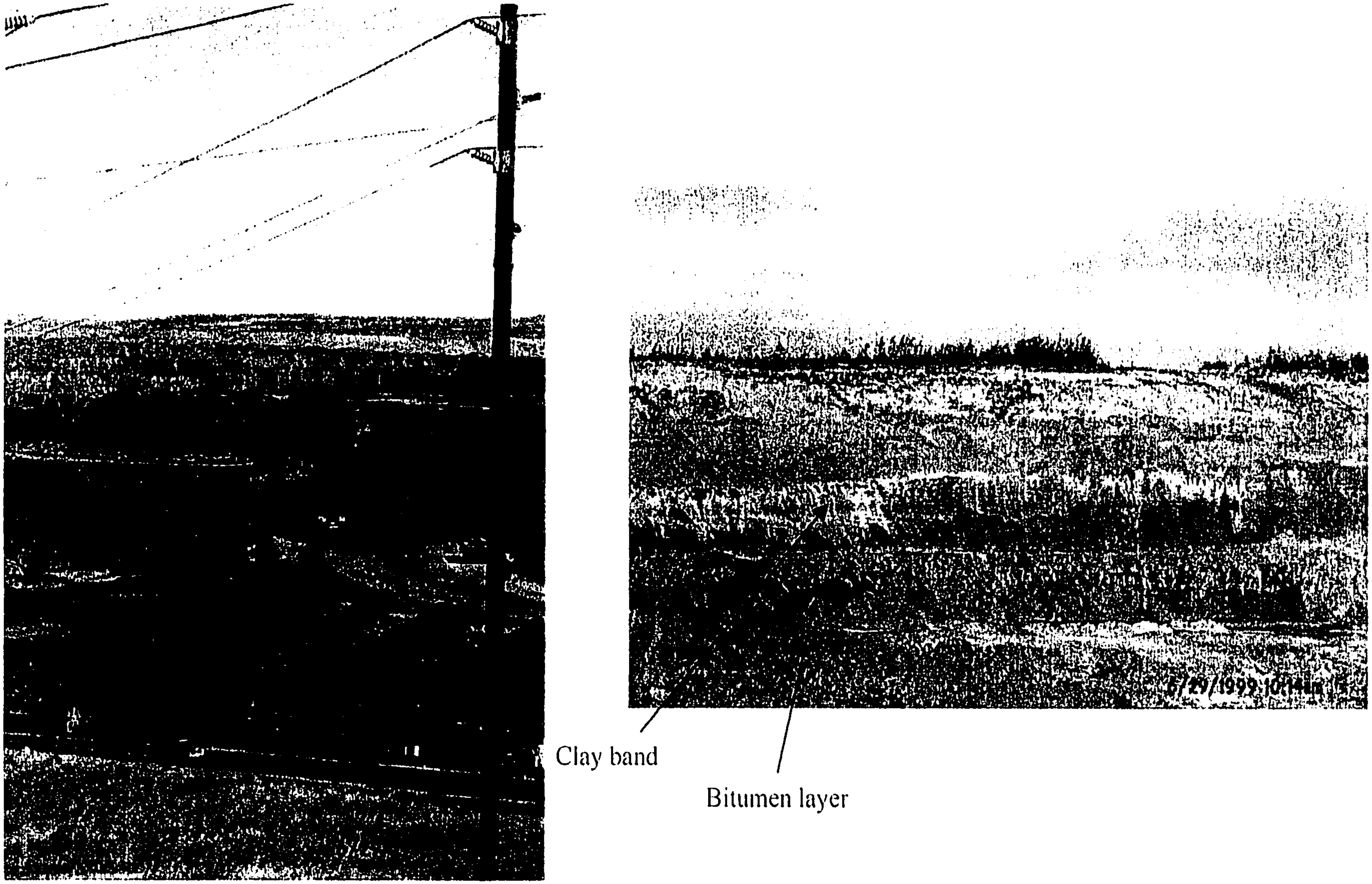


Figure 1.1: Oil sands mine with layers of clay and bitumen of various thickness.

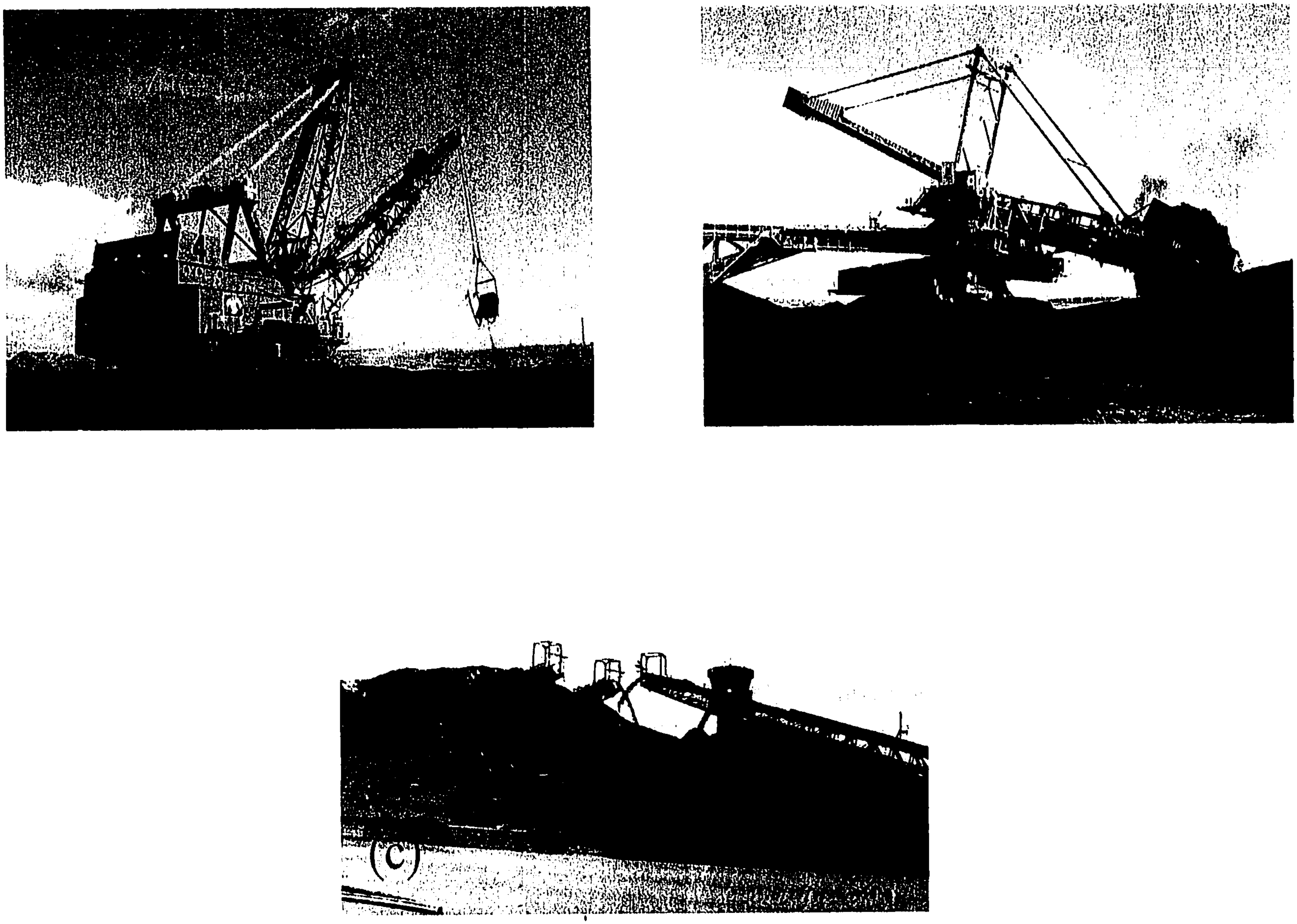


Figure 1.2: Mining operation at Syncrude's site: (a) dragline, (b) bucketwheel, (c) conveyor system to feed pocket.

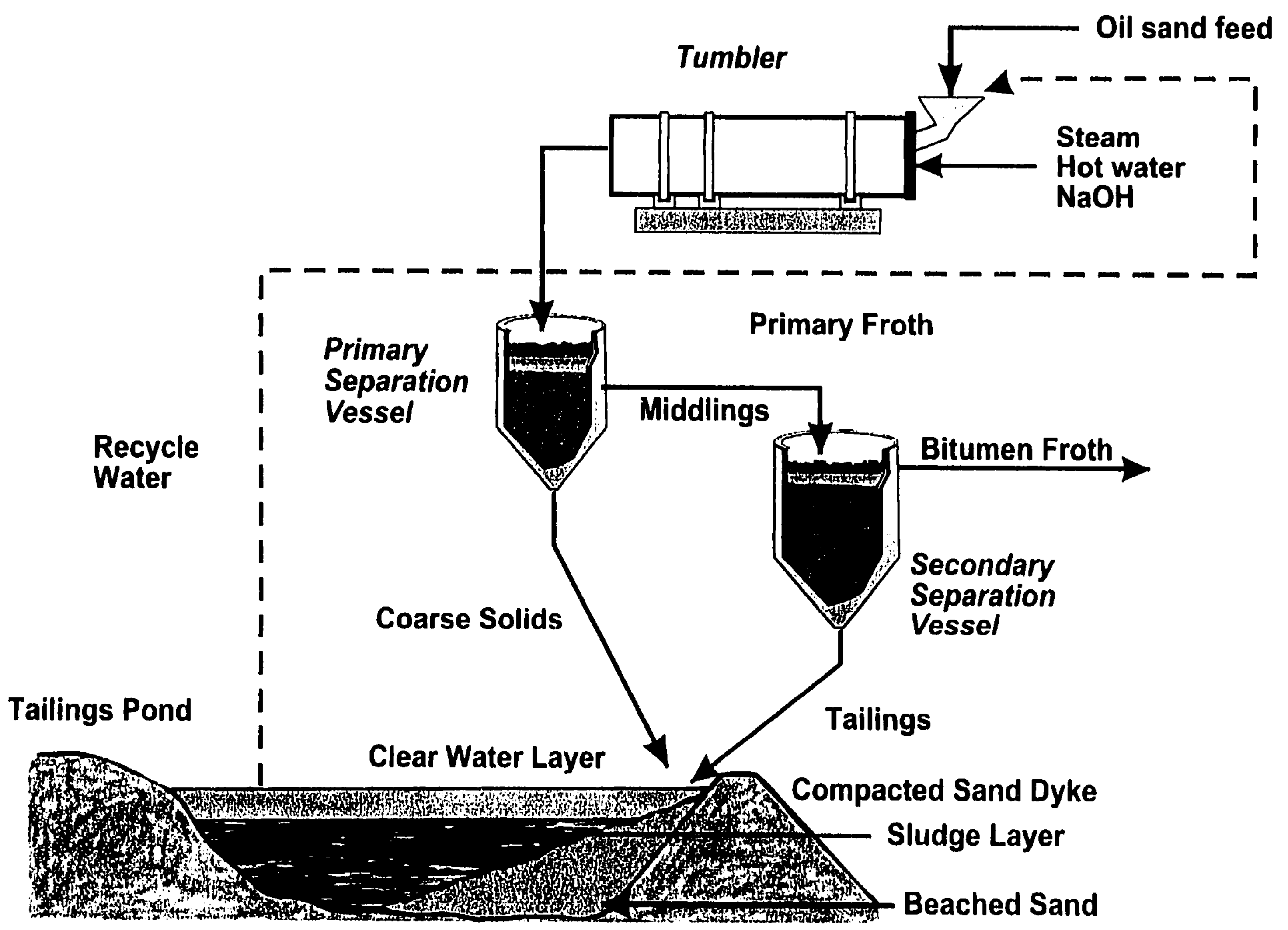


Figure 1.3: Schematic of Hot Water Extraction Process.

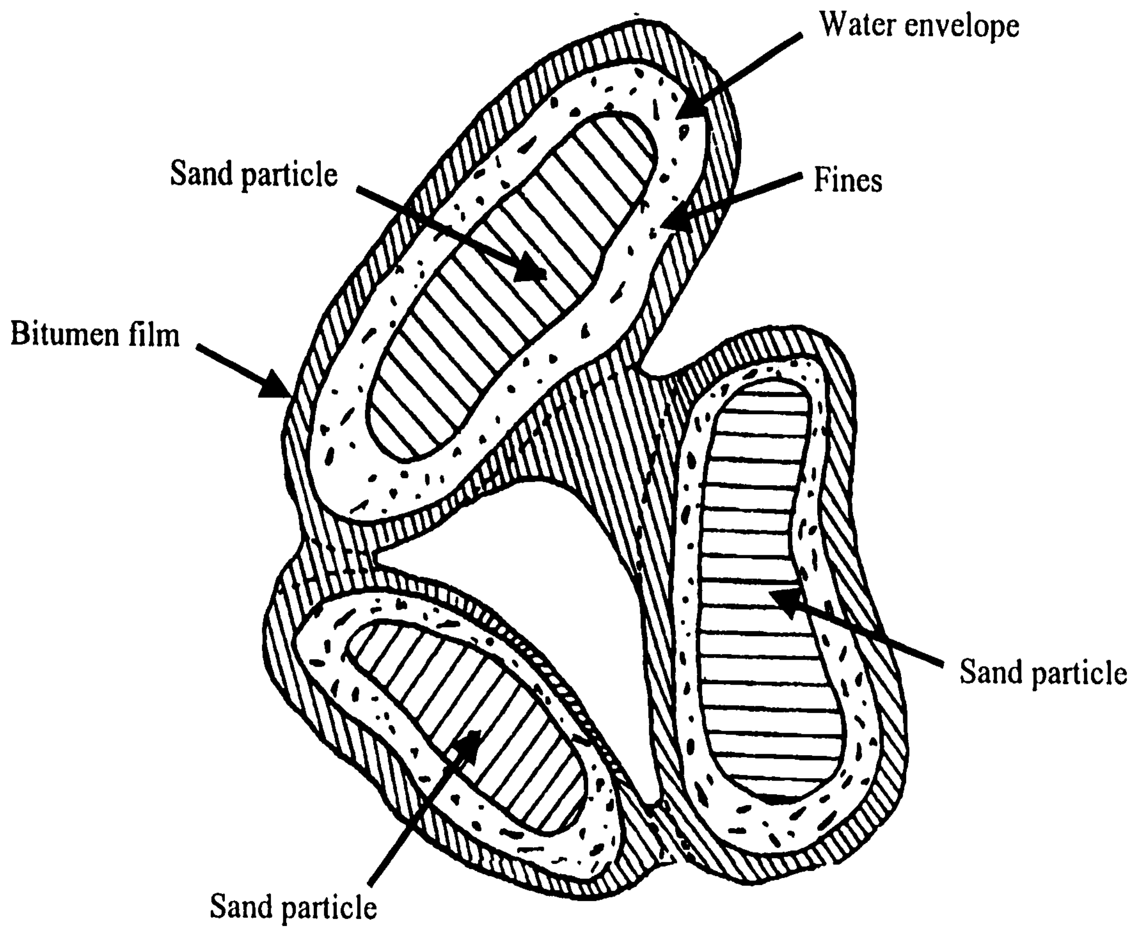


Figure 1.4: Microstructure of oil sands (taken from Takamura, 1982).

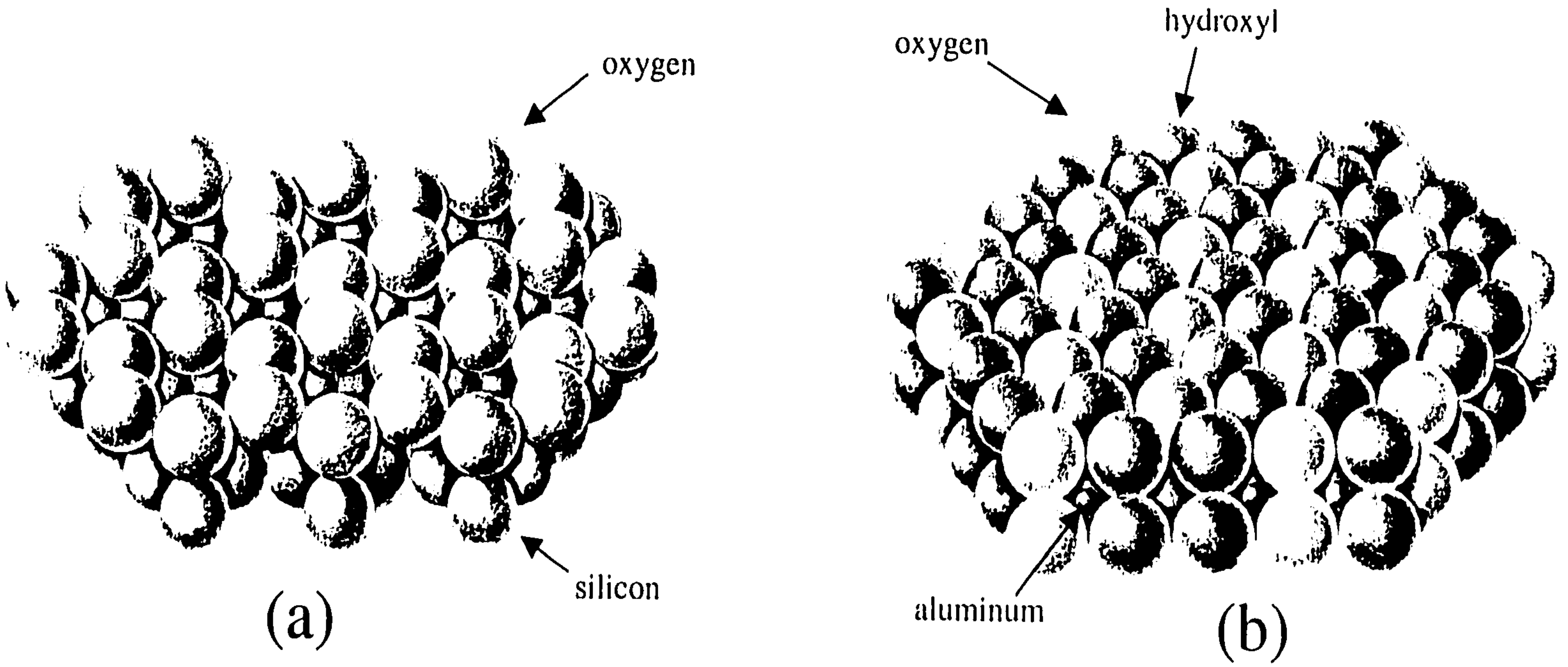


Figure 1.5: (a) Schematic of silicon-oxygen tetrahedral (b) aluminum-oxygen octahedral (taken from van Olphen, 1991).

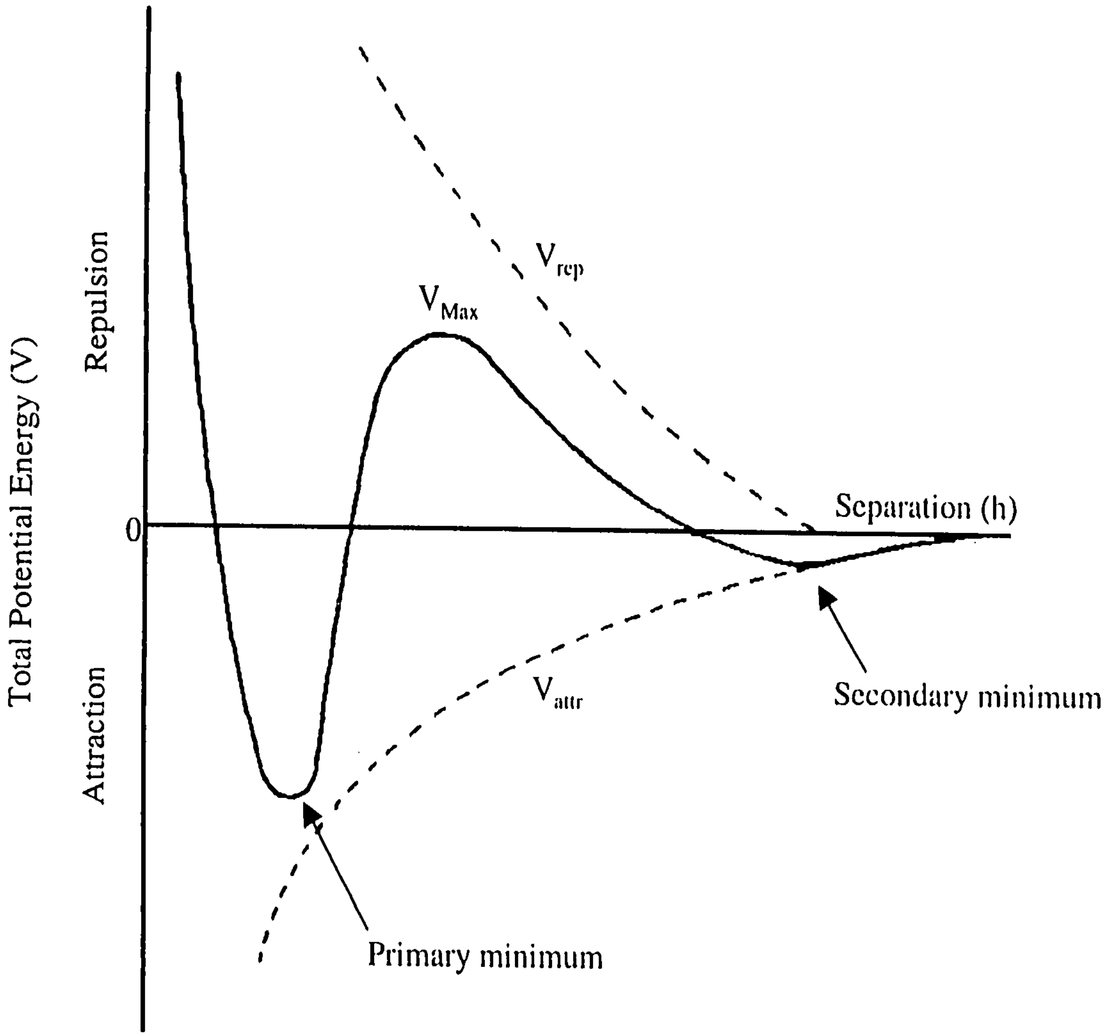


Figure 1.6: Schematic of the DLVO theory (taken from van Olphen, 1991).

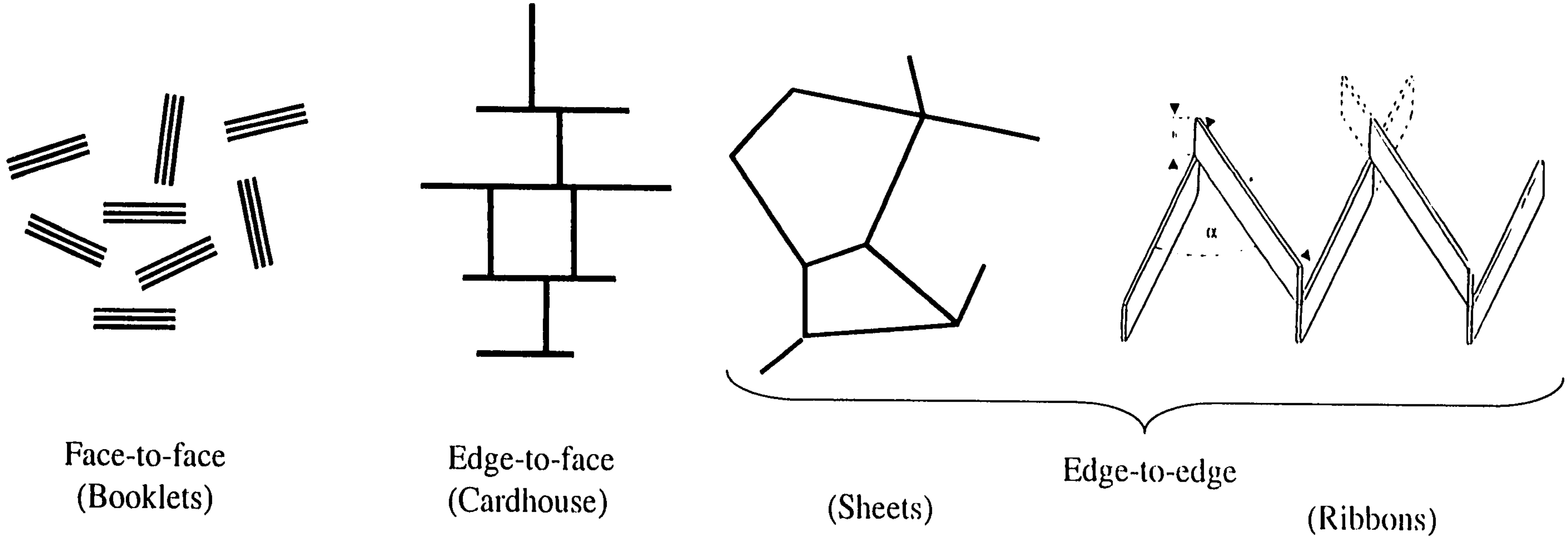


Figure 1.7: Various modes of clay particle association.

Chapter 2

Experimental Procedure

2.1 General

Syncrude Canada Ltd. (Edmonton Research Centre) supplied five oil sands samples in May 1997. Three of these oil sands were core samples from their 1997 core drilling program. Two more samples were supplied from an ore processability study conducted in the 4th quarter of 1997 and the 1st quarter of 1998.

In 1999, Syncrude Canada Ltd. (Edmonton Research Centre) submitted an additional eight samples from their North Mine: four samples from an estuarine environment (two oil sands and two waste units) and four samples representing a marine depositional environment (two oil sands and two waste units). Waste units are clay bands within the oil sands deposit containing virtually no bitumen. Shell Canada Ltd. provided twelve oil sands through the Alberta Research Council (ARC), including eight estuarine oil sands, two fluvial and two marine oil sands. Sample identification and brief geological descriptions of these samples are given in Table 2.1.

2.2 Oil Sands Characterisation

Over many years, the oil sands industry has developed standard methods to assess ore processability. It has become apparent that the nature and properties of oil sands feed has a significant influence on process efficiency. In this research both traditional and novel ore characterisation techniques have been compared for the evaluation of process predictability.

2.2.1 Sample handling

The samples from each site were handled in the same manner. After core sampling in the field, the oil sands were placed in large plastic bags and shipped to the respective Research Centre in 20 L steel pails. Once received, the samples were homogenized to provide representative subsamples. A Quadro Comil™ fitted with a ¼ inch screen was used for this purpose. After the entire sample was put through the Comil, it was placed on a large plastic sheet on a lab bench and thoroughly mixed using a coning and quartering method. The various subsamples were collected at this time and placed in plastic bags before shipment to NRC in Ottawa. Test material for this thesis work was selected randomly from the subsamples.

2.2.2 Bitumen, Water and Solids Content: Soxhlet Extraction-Dean and Stark Separation

The basic method commonly used for oil sands analysis is the Soxhlet Extraction-Dean and Stark procedure to determine the bitumen, water and solids content (Syncrude Analytical Methods, 1979). A representative oil sands sample is weighed into a paper thimble and placed into the Soxhlet extractor attached to a heated, solvent reservoir. The sample is separated into bitumen, water and solids by continuous reflux with toluene.

Condensed solvent and distilled water are separated in Dean and Stark traps and the volume of water collected in the side arm is measured. The distilled solvent is continuously recycled through the extraction thimble until colourless. The dissolved bitumen is collected in the reservoir. The resulting toluene solution of bitumen is concentrated in a RapidVap evaporation system at 80°C for approximately 8 hours. The extracted bitumen free oil sands solids are dried in a forced draft oven at 110°C overnight to remove residual toluene, cooled and then weighed.

2.2.3 Fines and Clay Content

Alberta Research Council (ARC) and Syncrude Canada Ltd. supplied results for fines and clay contents for the samples tested in this work. At Syncrude these components were determined using a laser diffraction method; clay contents for the same samples were also measured at AGAT Laboratory, Calgary. For the Shell samples, ARC used a hydrometer procedure and methylene blue adsorption measurement for fines and clays determination.

2.2.4 Wet Sieving

Particle size analysis was performed on Soxhlet extracted solids from the ore samples. The solids were removed from the thimbles and placed in a 100 mL glass jar. Because Soxhlet extracted solids may still be associated with bitumen that is difficult to remove they were thoroughly washed with fresh aliquots of toluene. Toluene was added to the solids and the sample shaken for 10 minutes using a high intensity Spex Mixer (Spex Industries, Scotch Plains, N.J.). The sample was then centrifuged for 10 minutes at 1500 gravities. After the cleaning process, the sample was dried overnight in a forced draft oven at 110°C.

After drying, sodium pyrophosphate (60 g of 0.1 w/w% solution) was added to a sample of extracted material and the mixture again shaken on the Spex mixer for 10 minutes to disperse the solids. Sieves with mesh sizes of: 212, 150, 75 and 45 µm were used for particle sizing. After the sample was separated, each fraction was collected in a tared 100 mL glass jar and dried in the forced draft oven at 110°C. The amount of each fraction was determined gravimetrically by difference. The results were checked by performing a mass balance closure.

2.2.5 Carbon Content

Total carbon contents of bitumen free samples were determined on dry, bitumen free fractions from wet sieving. The measurements were made using a LECO CR-412 carbon analyser. This instrument uses IR absorption to measure the carbon dioxide produced during complete combustion of the sample. A correction for decomposition of carbonate was determined from measurements on acid digested samples.

2.2.6 Mineralogical and Leachable Ions Composition

ARC used their Quantitative Mineral Analysis (QMA) method to determine the mineral composition of the extracted solids from the Shell samples. This method combines X-ray Diffraction (XRD) and X-ray Fluorescence (XRF) analysis. AGAT Laboratory also used XRD to evaluate the mineral composition of the Syncrude samples. ARC and Syncrude Canada Ltd. also provided results for leachable ions for the samples submitted. For this analysis Soxhlet extracted solids were placed in a paper thimble and were refluxed with water. The water was then analysed by Atomic Adsorption for leached cations and anions.

2.3 Oil Sands Fractionation

Oil sands can be separated into well-defined components based on their physical characteristics. The fractionation techniques developed in this work are based on earlier work by Kotlyar et al, 1988 and 1992.

2.3.1 Ultra-fines Determination

This method is used to determine the amount of ultra-fine solids (<0.3 μm) in oil sands ores. The separation technique is based on differential settling of particles. First. distilled water, or 0.1 w/w% sodium pyrophosphate solution (60 g), was added to each oil sands sample (10 g). Sodium pyrophosphate is a strong dispersing agent that deflocculates clays thereby facilitating the determination of ultra-fines. Use of this reagent is necessary for oil sands exhibiting flocculating characteristics, i.e., those with a high salt content.

After vigorous shaking on a Spex mixer for 10 minutes, the mixture was centrifuged for 1 hour at 200 gravities. This treatment causes each mixture to separate into a top layer of bitumen with strongly associated solids, a middle layer comprising an aqueous suspension of ultra-fines and a bottom sediment of coarse solids. The ultra-fines suspension was pipetted off and ultra-centrifuged for 1 hour at 366 000 gravities. Any bitumen associated with the sedimented ultra-fines was removed by repeated washing with fresh aliquots of toluene; the clean solids were dried overnight at 120°C. This procedure provides an ultra-fines content on a largely salt reduced and bitumen free basis. The same method was applied to determine the ultra-fines contents of Batch Extraction Unit (BEU) middlings streams, see Section 2.4 for BEU details. In these cases a sufficient amount of concentrated sodium pyrophosphate solution was added to the samples to give a concentration of about 0.1 w/w% in the aqueous phase.

2.3.2 Organic Rich Solids Determination

This method determines the amount of organic rich solids (ORS) in oil sands ores. The separation of ORS from bulk oil sands exploits the biwettable character of this component. Because of their dual wettability. ORS concentrate at water/oil interfaces and are consequently easily separated from solids remaining in the bulk water phase. With minor modification the method can also be used to determine ORS in process streams, such as PSV middlings etc.

The first step in this procedure is to dissolve most of the bitumen from an ore sample by vigorously mixing with toluene. A representative sample of ore (10 g) is weighed into a jar and toluene (≈ 80 g) is added. The sealed jar is then shaken on a Spex mixer for 1 minute, followed by centrifugation for 30 minutes at 1 500 gravities. A pipette is used to remove as much as possible of the supernatant bitumen solution, which is then discarded.

Distilled water (≈ 60 g) is placed in a tared jar (Note: for more difficult ores a 0.1 w/w% solution of sodium pyrophosphate can be used) and clean toluene (≈15 g) is added. The sample is shaken on a Spex mixer for 10 minutes and, if necessary, centrifuged for 5 minutes at 200 gravities. Organic rich solids form an emulsion-like phase at the water-solvent interface. This layer is removed by pipette and stored in another 100 mL tared jar. Then, more toluene (5 g) is added to the original jar and the suspension shaken gently by hand to separate any occluded emulsion. This additional material is removed and combined with that in the second jar. This procedure (excluding Spex mixing and centrifugation) is repeated until no more emulsion forms, i.e., the toluene layer remains clear. This may take at least four repetitions.

The phases are separated and the toluene suspension of ORS is washed repeatedly with fresh aliquots of water to remove any hydrophilic particles. To do this, distilled water (≈ 60 g) is added to the combined ORS emulsion in the second jar. The sample is then shaken by hand and the layers allowed to separate. The top layer containing the ORS is pipetted off into a third 100 mL tared jar. This is repeated until no more solids pass into the water layer. The third jar now contains the cleaned ORS.

The ORS fraction is then washed repeatedly with fresh aliquots of toluene to remove any remaining bitumen. Toluene (≈80 g) is added to the ORS in the third jar and the mixture is shaken on a Spex mixer for 5 minutes, followed by centrifugation at 1 500 gravities for 10 minutes. The supernatant toluene is removed from the sedimented solids with a pipette. This procedure is repeated until the toluene layer is colourless; usually at least four repetitions are required.

Finally, the sample of ORS is dried overnight in a forced draft oven at 110 °C to remove toluene. After drying, the sample is weighed and its concentration in the original ore sample calculated.

ORS determination is difficult for waste samples because their particle size is much smaller than that of oil sands. For this reason the ORS emulsion layer is not easy to remove quantitatively.

2.4 Batch Extraction Unit Tests

Bitumen separation tests were carried out in a Batch Extraction Unit (BEU) based on a standard design supplied by Syncrude (Syncrude Analytical Methods, 1979). This small-scale extraction vessel is used to evaluate new process aids, process variables and for determining the processability of different oil sands samples. This is a practical, standard hot water separation unit that is used in conjunction with a test procedure developed over many years of optimisation. Although, the method is believed to provide a good indicator for process problems, its usefulness is limited by the fact that it can only be operated at a higher slurry dilution than that used in commercial operation. A schematic representation of the BEU unit used in this work is shown on Figure 2.1. The extraction vessel is designed with jacketed walls to provide constant temperature during operation. Air is added through the impeller shaft permitting aeration of the slurry during ore conditioning. After testing, the bitumen depleted slurry can easily be removed through a valve at the bottom of the cell.

Normally, tests are made using recycle water from the plant tailings pond. In this work, a synthetic pond water, with a composition based on typical recycle water at the Syncrude plant, was prepared for use as the slurry medium for both conditioning and flotation phases of these tests. A synthetic pond water was used to avoid confounding problems associated with the solids and organic matter present in actual pond water. Table 2.2 compares the composition of synthetic and actual recycle water.

Sodium hydroxide is normally used to optimise bitumen recovery in BEU test work. This practice was not adopted in this work because the objective was to establish base line data to allow the natural processability of various oil sands types to be compared. By using sodium hydroxide, bitumen recovery is enhanced in all cases, possibly masking essential differences in behaviour.

To begin a test, hot water is circulated at 82°C through the cell jacket to provide constant temperature during operation. Synthetic pond water at 90°C and a selected oil sands sample (500 g) are used for the test. At this point, the ore sample is added to the cell, along with synthetic pond water (150 mL). The stirrer is set to 10 rev/s and the air supply adjusted to 7 mL/s. This conditioning step continues for 10 minutes, after which the air supply is turned off and synthetic pond water (900 mL) at 82°C is added to the cell. Mixing continues for an additional 10 minutes at 10 rev/s without air.

After 10 min, the mixing is stopped and the primary froth is skimmed off using a specially designed spatula. The remaining material is mixed for 5 minutes at 13 rev/s with the air supply at 3.9 mL/s. A secondary froth is collected in the same manner as the primary froth. The residual material is drained off through the bottom valve into a 2L plastic container. After 1 hour the aqueous suspension containing any unsettled solids (desanded middlings) was decanted from the settled solids layer (coarse tailings).

2.4.1 Stream Characterisation

Each BEU test produces the four process streams, shown on Figure 2.2. All streams were analysed for ORS (see Section 2.3.2 for procedure) and bitumen content, while only the middlings and tailings were analysed for ultra-fines content (see Section 2.3.1 for procedure). Settling experiments were performed on each middlings sample (see Section 2.4.1.2).

2.4.1.1 Bitumen Content

Prior to analysis, froths and tailings were homogenised by mixing with a spatula; the middlings were shaken for 5 min on a Red Devil paint shaker. Bitumen content of the different fractions was determined by extraction with toluene. Bitumen from the froth samples (10g) was extracted with toluene (80g); bitumen from the middlings (30g) and tailings (30g) was extracted with a lesser amount of toluene (50g). Each mixture was shaken for 5 min on a high intensity Spex Mixer, followed by centrifugation for 30 min at 1 500 gravities.

Bitumen concentration was determined using a Varian UV/Vis spectrophotometer, model DMS90, at a wavelength of 530nm with a 10 mm path length cell. Calibration curves for bitumen determination were constructed using desolventised bitumen separated from the corresponding oil sands by Soxhlet Extraction. Individual curves are necessary because each bitumen sample is slightly different; a sample calibration curve is seen on Figure 2.3.

2.4.1.2 Settling Experiments

2.4.1.2.1 Middlings

Middlings from each BEU test were mixed on a Red Devil paint shaker for 5 minutes, after which 90 mL were decanted into 100 mL cylinders. Because the water used in BEU experiments contained weakly dispersing bicarbonate ions, settling of all middlings was extremely slow. To facilitate the process, NaCl solution was added to all suspensions to produce a concentration of 50 mM. This amount of salt was sufficient to induce rapid settling, allowing information on final sediment volumes to be obtained in weeks rather than months. The process was followed to provide information on final sediment volumes for tailings production prediction.

2.4.1.2.2 Ore Ultra-fines

A settling procedure similar to that performed on BEU middlings was used with ultra-fine suspensions separated from all twenty-five oil sands. To prepare the suspensions distilled water was added to an oil sands sample pre-weighed into a glass jar. The mixture was agitated for 10 minutes on a spex shaker and centrifuged for 1 hour at 200 gravities. This procedure produced a suspension of mostly ultra-fine solids. The suspension (90 mL) was transferred to a 100 mL graduated cylinder and concentrated NaCl solution was added to give a final concentration of 50 mM. After settling was complete, the equivalent final volumes, or volume percentages of ultra-fines in the sedimented tailings was calculated.

2.5 Jar tests

Because of the complex nature of the characterisation procedures a simple test was developed to allow a rapid, qualitative assessment of the processability of oil sands ores. Sufficient distilled water was added to an oil sands ore in a 100 mL, screw capped, glass jar to give a ratio of either 0.7 to 1 or 2 to 1. These ratios cover the range normally used in full-scale operation. To determine the effects of salt the Na^+ or Ca^{2+} content of the slurry water was adjusted within the range likely to be encountered under actual process conditions. The samples were shaken and allowed to stand for a few minutes. Each sample was then examined visually to provide a qualitative assessment of slurry segregation.

Adjustments to the cation content of the slurry water were made with either sodium chloride or calcium-fortified water prepared from suspensions of gypsum in distilled water. In the latter case a gypsum suspension was mixed on a reciprocal shaker overnight, centrifuged at 5 000 revs/min for 1 hour to remove free solids and then analyzed for dissolved Ca^{2+}.

Table 2.1: Geological description of oil sands ores.

Sample ID		Depositional Environment
1	1-OS-E1	Estuarine
2	1-OS-E2	Estuarine
3	1-OS-C1	Continental
4	2-OS-E3	Estuarine
5	1-OS-E4	Estuarine
6	3-OS-E5	Estuarine
7	1-OS-E6	Estuarine
8	1-OS-E7	Estuarine
9	2-OS-E8	Estuarine
10	3-OS-E9	Estuarine
11	1-OS-C2	Continental
12	3-OS-MT1	Marine Transition
13	1-OS-E10	Estuarine
14	3-OS-M1	Marine
15	1-OS-E11	Estuarine
16	1-OS-E12	Estuarine
17	2-OS-M2	Marine
18	3-OS-MT2	Marine Transition
19	1-OS-M3	Marine
20	2-OS-M4	Marine
21	1-OS-M5	Marine
22	2-W-M2	Marine
23	2-W-M3	Marine
24	2-W-E8	Estuarine
25	2-W-E3	Estuarine

Table 2.2: Comparison of ionic composition of synthetic water for BEU tests with typical pond recycle water.

Water Sample	**Ion Concentration (ppm)**						**pH**
	Na^+	**K^+**	**Ca^{++}**	**Mg^{++}**	**Cl^-**	**HCO_3^-**	
Synthetic pond water	500	0	0	0	250	915	8.8
Pond recycle water*	460	9	5	3	280	1100	8.4

* Submitted by Syncrude Canada Ltd. and analyzed at ICPET/NRC 1995

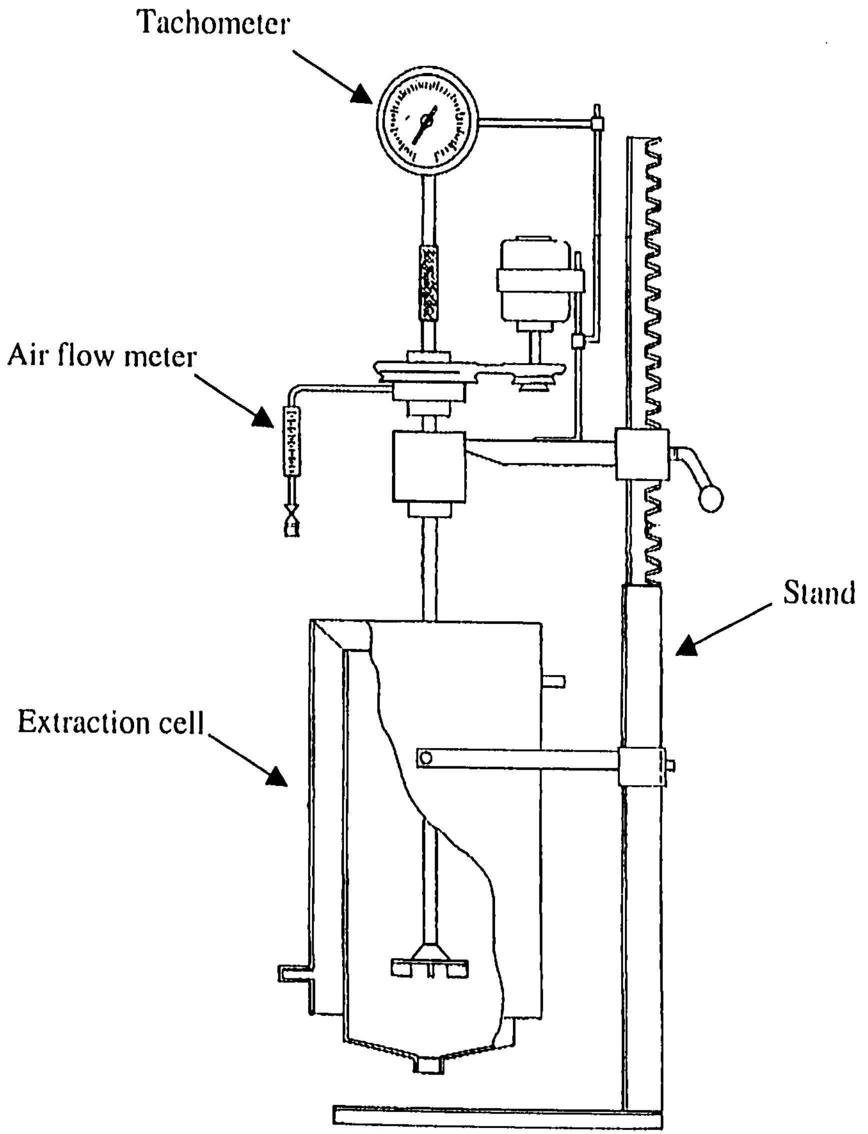


Figure 2.1: Schematic of Batch Extraction Unit (taken from Syncrude Analytical Methods, 1979).

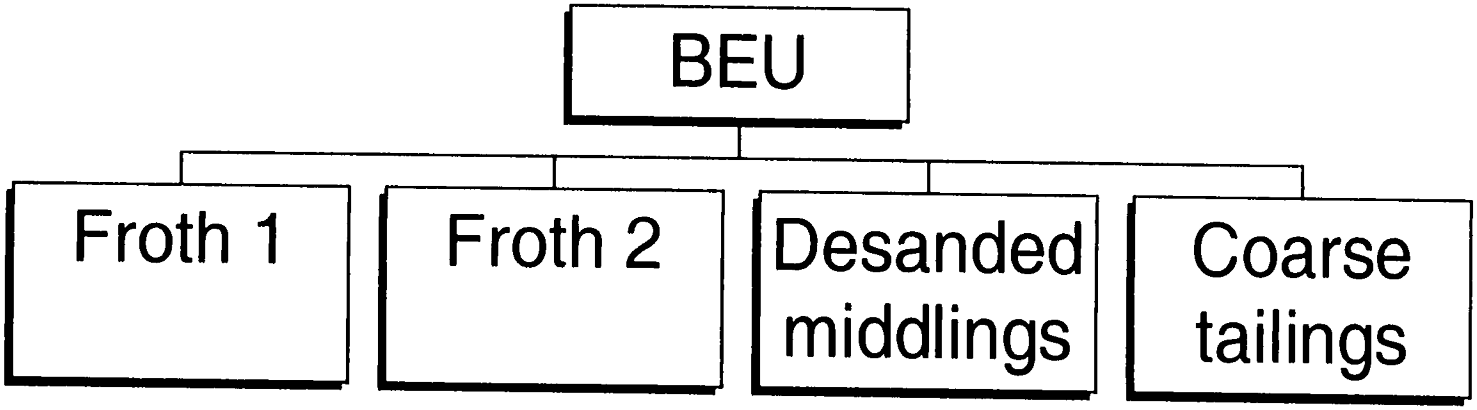


Figure 2.2: Process Streams from Batch Extraction Unit Test.

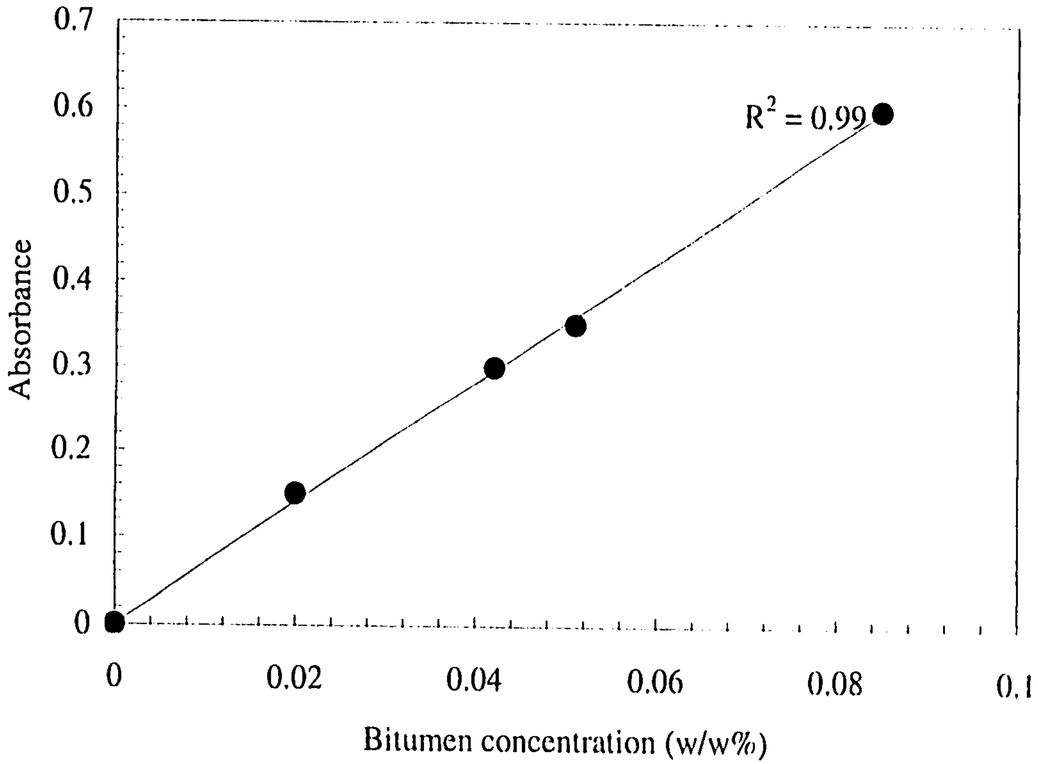


Figure 2.3: Sample bitumen calibration curve.

Chapter 3

Results

3.1 Oil Sands Characterisation

Both oil sands and waste units were analysed in this work. The samples were taken from three widely separated areas in the Athabasca region. In addition, samples were selected from different depositional environments. These included oil sands from estuarine, continental, marine and marine transition zones. In the case of four oil sands, samples of the corresponding waste unit were also examined. A numbering system was adopted for easy identification of each sample, e.g., #-XX-X#. The first number identifies the area, the letter designation OS or W, refers to oil sands and waste unit respectively. The third grouping indicates the depositional environment and sample number. For example, 1-OS-E4 represents the fourth oil sands sample, of estuarine type, from area one. In the tables, the samples are conveniently arranged in order of increasing fines content. This format is adopted throughout the thesis.

3.1.1 Bitumen Content

Oil sands were analyzed for bitumen, water and solids content by Soxhlet extraction with toluene. Oil sands analysis for all oil sands samples are shown in Table 3.1. Bitumen contents cover a wide range from 0.2 w/w% for waste units 2-W-M3, 2-W-E8 and 2-W-E3 to as high as 17 w/w% for 2-OS-E3.

3.1.2 Fines and Clay Content

Fines content for area 1 samples were determined by wet sieving at NRC. Clay contents for area 1, and fines and clay content for area 2 samples were measured at AGAT Laboratory, while data for area 3 samples were supplied by ARC.

It is important to point out that even adjacent drill cores from within the same depositional environment may have a different composition. This inherent problem with oil sands research causes problems when measuring oil sands properties. For example, an oil sands sample may have been inadvertently contaminated with adjacent waste band material during mining, thereby significantly altering the fines and clay content of the sample. This occurs because waste materials have such high amounts of fines and clays and is demonstrated with the four waste materials tested in this work where the fines and clay contents were between 65 and 90 w/w%, see Table 3.1.

Of the ore samples, estuarine oil sands 1-OS-E1 had the lowest fines content of 1.2 w/w% while marine oil sands 1-OS-M5 had the highest at 39.9 w/w%. For all samples analysed in this work the fines contents are only roughly proportional to clay concentrations, see Figure 3.1. As usual, a general trend for decrease in bitumen content with increase in fines content is also observed, see Figure 3.2.

3.1.3 Mineralogical Compositions

The mineralogical compositions of oil sands solids are summarized in Table A.1. In all cases the mineralogy of oil sands solids is dominated by quartz, with values ranging from 54 w/w% to 97 w/w%. For samples with lower quartz contents there is a distinctly greater contribution of clays, particularly kaolinite and illite. This observation is most obvious for waste sample 2-W-M3, which has an illite content above 40 w/w%. Marine transition and marine samples 3-OS-MT1, 1-OS-M5 and 2-W-M2 have relatively high amounts of calcite and dolomite with values of about an order of magnitude greater than other samples analyzed. This may be important because of the adverse effects of Ca^{2+} and Mg^{2+} on ore processing. It is also observed that the presence of relatively high amounts of pyrite and siderite differentiates estuarine oil sands 1-OS-E7 from all other samples.

3.1.4 Water Chemistry

Results for leachable ions for all samples are outlined in Table A.2. The data is expressed as ppm, i.e., mg/kg of oil sands. The divalent cation concentrations, Ca^{2+} and Mg^{2+}, in oil sands 3-OS-MT1, 3-OS-M1, 1-OS-E12 and 3-OS-MT2 are much higher than those found in other samples.

Apart from the high concentrations of the divalent cations, the very high sodium levels found in estuarine waste units 2-W-E3 and 2-W-E8 are of particular interest. The values are almost an order of magnitude higher than those found in the adjacent oil sands samples from the same depositional environment.

3.1.5 Ultra-fines Content

Results for ultra-fines content of all samples are shown in Table 3.2. The table includes data for three consecutive separation treatments with distilled water. In general more than 50% of the total ultra-fines are separated in the first treatment. This is an important observation because it relates to the amount of ultra-fines likely to be released during processing. That is, process problems depend not only on total ultra-fines content of an ore but also the percentage of this component easily dispersed during the conditioning step of the HWEP.

Marine oil sands have much higher amounts of ultra-fines than samples from estuarine and continental environments. It is particularly important to note that the ultra-fines contents of waste units from both estuarine and marine environments are also extremely high and represent a greater proportion of the fines content than for normal ores.

Another significant aspect of water based bitumen separation processes is the production of fine tailings. In previous work it was established that the final volume of Mature Fine Tailings depended on the initial concentration of ultra-fines in the middlings from the PSV (Kotlyar et al, 1992). To determine whether this finding is applicable to a wide range of oil sands from diverse sources, all twenty-five samples were subjected to settling experiments. The tests included samples of BEU middlings from mine 3 and ultra-fines separated from ores of mines 1 and 2. The results are summarized on Figure 3.3. Solids' settling occurs until the critical gelation concentration of about 2.5 to 3 w/w% ultra-fines is reached. Consequently, final sediment volumes are roughly proportional to the ultra-fines concentration in the original suspension. Original suspensions containing the critical amount of ultra-fines, or greater, hardly settle at all. The critical gelation concentration is an important parameter because it provides information on tailings volume production for different ores. The amount of tailings produced is an important consideration for mine planning and also for tailings handling and disposal. Before mining begins, a detailed disposal plan must be set-up in order to allocate enough volume in the tailings ponds. Based on the final tailings volume, calculations are used to determine the amount of water available for recycle back to the plant. Thus, it is evident that ultra-fines content is very useful in predicting tailings production.

Another feature showing that ultra-fines content is a crucial parameter for predicting final tailings volume is seen when comparing settling data for BEU middlings versus that of ultra-fines separated from ore samples. Final volumes follow the same trend for both data sets. This shows that ultra-fines content is the ultimate parameter controlling tailings volume.

3.1.6 Organic Rich Solids

The ORS contents for all samples are shown in Table 3.3. For most oil sands tested in this work there is a trend for ORS to increase with fines content. This is not unreasonable as both fines and ORS are associated with a high organic matter content. see Tables 3.4 and 3.5 (Kotlyar et al, 1998b). For example, the results for samples 3-OS-E5 and 3-OS-M1 show that the fines fractions representing only 5 and 12 w/w% of the solids are associated with more than 65% of the organic carbon associated with the bitumen free. oil sands solids.

3.2 Batch Extraction Unit Tests

Because of the extensive work involved, oil sands separation experiments were initially carried out on only five oil sands. These included two estuarine, two marine transition and one marine oil sands. A batch unit similar to that designed by Syncrude Research was used for conducting the experiments. Two other estuarine oil sands, 1-OS-E6 and 1-OS-E7, having similar fines, bitumen and clay contents but markedly different ORS concentrations, see Table 3.3, were separated later for confirmation purposes.

BEU separations were carried out using synthetic pond water and a water to oil sands ratio of 2 to 1. Each test produces four streams, namely: primary and secondary froth, desanded middlings, i.e., original middlings settled for one hour, and coarse tailings.

3.2.1 Froth Streams

Oil sands 3-OS-E5, 3-OS-E9 and 3-OS-MT1 produced more than twice as much primary froth as oil sands 3-OS-MT2 and 3-OS-M1. The values ranged from 30 g to 64 g. Total solids and ORS contents covered the ranges of about 4 to 9 w/w% and 3 to 4 w/w% respectively, see Table 3.6. There appears to be a relationship between the amount of secondary froths and the fines contents of the oil sands, see Table 3.7. Secondary froths contained more solids than the primary froths with amounts ranging from 15 to 23 w/w% for total solids and 8 to 14 w/w% for ORS, see Table 3.7.

Froths were practically free of aqueous ultra-fines, consequently this component is not considered to be a significant factor in froth quality.

3.2.2 Tailings Streams

Table 3.8 shows bitumen, ultra-fines, ORS and total solids contents for the middlings streams. Middlings derived from oil sands 3-OS-M1 had the highest concentration of these components. For all BEU samples a reasonably good correlation was observed between the total solids contents of the middlings fractions and their ultra-fines contents, see Figure 3.4. Middlings sample 3-OS-M1 from a Syncrude pilot plant test did not follow this trend, but instead showed a total solids value of more than 30 w/w% with an ultra-fines content of 2 w/w%. From the plot a sharp increase of total solids content is seen once an ultra-fines concentration of approximately 1.2 – 1.5 w/w% is reached. Total solids increases with ultra-fines contents of the stream possibly as a result of higher middlings viscosity. At a critical point settling is affected to such a degree that no segregation occurs and PSV sludging ensues. In the case of tailings both bitumen and ORS concentrations increase for the sample order 3-OS-E5, 3-OS-E9, 3-OS-MT1, 3-OS-M1 to 3-OS-MT2, see Table 3.9.

In Table 3.10 the ultra-fines content of the middlings and tailings samples are expressed as w/w% of the original oil sands. These results show that the total amounts of ultra-fines found in the BEU tailings streams are close to those determined for the parent ores using three sequential treatments with distilled water, compare Tables 3.2 and 3.10. About 70 w/w% of the total ultra-fines report with the middlings fractions produced in the BEU tests; the remaining ultra-fines are captured by the coarse tailings fraction.

Settling tests were carried out on all BEU middlings generated from the different oil sands using synthetic pond water. For sample 3-OS-M1, non-segregating behaviour was observed, see Figure 3.5. This is undesirable because it indicates that coarser particles and bitumen are trapped within the gel network. If this is the case, bitumen recovery will be reduced, or possibly eliminated. To alleviate this condition, a dispersing agent can be added; the restoration of segregating settling to middlings sample 3-OS-M1 after such treatment is also shown on Figure 3.5.

BEU middlings generated from estuarine oil sands 3-OS-E5 and 3-OS-E9 and marine transition oil sands 3-OS-MT1 and 3-OS-MT2 exhibit differential settling indicating that a gel network is not formed and that bitumen separation will occur normally. A typical result for estuarine oil sands 3-OS-E5 is also illustrated on Figure 3.5. In this scenario bitumen recovery is not hindered and coarse particles settle out.

The bitumen distributions among BEU streams, presented as weight percent of the original oil sands and total bitumen, are presented in Tables 3.11 and 3.12 respectively. The data presented in Table 3.12 shows that bitumen recovery in the primary froths decreases in the order of oil sands 3-OS-E5, 3-OS-E9, 3-OS-MT1, 3-OS-M1 to 3-OS-MT2. The ORS distribution among the same streams, as weight percent of that in the corresponding oil sands and of total ORS, are shown in Tables 3.13 and 3.14.

Table 3.15 summarises the bitumen to ORS ratios (BIT/ORS), calculated from compositional data for the parent oil sands and their BEU streams. These ratios in the feed decrease from about 19 to 5 for oil sands samples 3-OS-E5 through 3-OS-MT2. In the primary froths the ratios are greater, ranging from 18 to 25, while in the secondary froths the ratios are much lower, ranging from 3 to 6. A surprising observation is that the ratios determined for the middlings and tailings streams from each ore are surprisingly similar, having an average value of 2.8±0.7. Two confirmatory BEU tests were performed to verify this observation. Two oil sands of similar fines content but markedly different ORS content were tested. Once again, it was found that the average bitumen to ORS ratio for the combined tailings was close to the previous 2.8±0.7 value, see Table 3.16. These results indicate a possible direct association between ORS and bitumen in BEU tailings.

Table 3.1: Oil sands compositions.

ID	Oil sands composition (w/w% of oil sands)						
	Bitumen	Water	Solids	Fines	Clays	ORS	Ultra-fines
1-OS-E1	13.2	1.7	84.7	1.2	0.2	0.8	0.7
1-OS-E2	16.1	1.5	81.4	2.0	0.4	0.7	0.3
1-OS-C1	11.9	4.1	83.7	4.4	1.2	1.3	0.7
2-OS-E3	17.0	0.5	82.3	4.9	1.6	0.8	0.2
1-OS-E4	14.6	3.1	82.3	5.2	1.4	1.6	0.8
3-OS-E5	11.2±0.7	5.0±0.2	84.1±0.1	5.2±0.4	2.6±0.2	0.6±0.1	0.7
1-OS-E6	12.2	4.3	82.3	5.7	2.1	1.0	0.5
1-OS-E7	10.6	5.5	83.4	6.7	3.4	2.9	1.1
2-OS-E8	14.4	0.6	84.8	9.3	2.5	1.0	0.5
3-OS-E9	12.7±0.7	4.3±0.4	82.8±0.2	10.3±2.3	5.8±0.6	1.0±0.2	1.2
1-OS-C2	9.2	4.3	85.9	12.3	9.5	1.2	1.3
3-OS-MT1	10.9±0.3	8.6±0.7	81.1±0.9	11.7±1.8	5.5±0.8	1.1±0.1	1.0
1-OS-E10	11.4	4.3	83.7	12.1	5.3	1.7	1.4
3-OS-M1	7.4±0.2	7.7±0.1	84.5±0.3	12.7±1.4	8.4±1.7	1.0±0.1	2.9
1-OS-E11	10.9	4.5	84.1	16.0	4.4	1.5	1.4
1-OS-E12	8.7	8.0	82.4	16.6	6.0	1.7	1.8
2-OS-M2	7.0	10.5	82.6	19.0	6.6	2.4	2.5
3-OS-MT2	8.9±0.4	7.7±0.2	84.3±1.7	22.0±1.2	8.9±0.6	1.8±0.2	1.6
1-OS-M3	8.9	5.7	84.1	22.6	10.8	2.7	2.9
2-OS-M4	2.6	8.5	89.0	26.7	8.0	2.9	6.5
1-OS-M5	4.0	11.4	84.6	39.9	20.6	3.7	5.6
2-W-M2	2.8	12.0	85.1	55.3	10.2	5.2	5.1
2-W-M3	0.2	11.5	88.2	69.7	15.9	2.6	22.5
2-W-E8	0.2	8.2	91.6	77.9	18.3	3.2	12.4
2-W-E3	0.2	5.4	94.4	85.0	20.8	3.5	13.2

Table 3.2: Ultra-fines contents of oil sands samples.

ID	Weight percent of oil sands				Ultra-fines removed by 1st treatment (Percent of total)
	Treatment No.			Total	
	1	2	3		
1-OS-E1	0.27	0.24	0.19	0.7	39
1-OS-E2	0.17	0.12	0.01	0.3	57
1-OS-C1	0.39	0.18	0.13	0.7	56
2-OS-E3	0.14	0.04	0.02	0.2	77
1-OS-E4	0.49	0.24	0.07	0.8	61
3-OS-E5	0.40±0.1	0.20±0.01	0.10±0.02	0.7	57
1-OS-E6	0.27	0.19	0.04	0.5	54
1-OS-E7	0.61	0.31	0.18	1.1	55
2-OS-E8	0.33	0.10	0.07	0.5	67
3-OS-E9	0.70±0.08	0.30±0.11	0.20±0.02	1.2	58
1-OS-C2	0.87	0.27	0.16	1.3	67
3-OS-MT1	0.60±0.02	0.30±0.01	0.10±0.02	1.0	60
1-OS-E10	0.83	0.34	0.23	1.4	59
3-OS-M1	2.00±0.5	0.60±0.1	0.30±0.03	2.9	69
1-OS-E11	0.80	0.38	0.22	1.4	57
1-OS-E12	0.92	0.54	0.34	1.8	51
2-OS-M2	2.10	0.37	0.03	2.5	81
3-OS-MT2	0.90±0.05	0.50±0.05	0.20±0.03	1.6	56
1-OS-M3	2.00	0.58	0.32	2.9	69
2-OS-M4	4.70	1.30	0.50	6.5	73
1-OS-M5	3.50	1.30	0.80	5.6	62
2-W-M2	4.10	0.71	0.29	5.1	81
2-W-M3	15.9	4.60	2.00	22.5	71
2-W-E8	8.20	3.30	0.90	12.4	66
2-W-E3	9.10	2.90	1.20	13.2	68

Table 3.3: Organic rich solids contents of oil sands samples.

Sample ID	ORS (w/w% of oil sands)	Bitumen/ORS Ratio for ore
1-OS-E1	0.8	17
1-OS-E2	0.7	23
1-OS-C1	1.3	9
2-OS-E3	0.8	21
1-OS-E4	1.6	9
3-OS-E5	0.6±0.1	19
1-OS-E6	1.0	12
1-OS-E7	2.9	4
2-OS-E8	1.0	14
3-OS-E9	1.0±0.2	13
1-OS-C2	1.2	5
3-OS-MT1	1.1±0.1	10
1-OS-E10	1.7	7
3-OS-M1	1.0±0.1	7
1-OS-E11	1.5	7
1-OS-E12	1.7	5
2-OS-M2	2.4	3
3-OS-MT2	1.8±0.2	5
1-OS-M3	2.7	3
2-OS-M4	2.9	1
1-OS-M5	3.7	1
2-W-M2	5.2	N/A
2-W-M3	2.6	N/A
2-W-E8	3.2	N/A
2-W-E3	3.5	N/A

N/A – not available

Table 3.4: Carbon contents of screened size fractions from oil sands 3-OS-E5.

Size (µm)	**Size fraction (w/w% of total solids) ***	**Carbon (in bitumen free solids)**	
		Percent of each size fraction (w/w)	**Percent of that in oil sands (w/w)**
+212	48.9	0.14	20.9
150-212	21.6	0.05	3.3
75-150	22.3	0.09	6.1
45-75	1.5	0.79	3.6
-45	5.6	3.90	66.1
Total solids	100.0	0.33	100.0

* dry bitumen free oil sands

Table 3.5: Carbon content of screened size fractions from oil sands 3-OS-M1.

Size (µm)	Size fraction (w/w% of total solids)*	Carbon (in bitumen free solids)	
		Percent of each size fraction (w/w)	Percent of that in oil sands (w/w)
+212	18.3	0.12	3.4
150-212	19.8	0.10	3.1
75-150	45.2	0.08	5.6
45-75	1.7	0.80	2.1
-45	15.0	3.70	85.8
Total solids	100.0	0.65	100.0

* dry, bitumen free oil sands

Table 3.6: Concentration of components in Primary Froth 1 from BEU tests.

ID	Amount (g)	Fines (w/w% of oil sands)	Composition (w/w% of froth)			
			Bitumen	ORS	Total Solids	Water
3-OS-E5	64.2	5.2	77.7	3.1	6.0	16.3
3-OS-E9	72.5	10.3	73.9	4.1	7.4	18.7
3-OS-MT1	61.6	11.7	68.3	3.2	9.0	22.7
3-OS-M1	30.0	12.7	60.5	2.5	4.4	35.1
3-OS-MT2	29.8	22.0	63.8	3.8	6.9	29.3

Table 3.7: Concentration of components in Secondary Froth 2 from BEU tests.

ID	Amount (g)	Fines (w/w% of oil sands)	Composition (w/w% of froth)			
			Bitumen	ORS	Total solids	Water
3-OS-E5	10.0	5.2	49.2	14.1	23.0	27.8
3-OS-E9	22.5	10.3	50.6	9.1	20.9	28.5
3-OS-MT1	27.1	11.7	40.4	9.8	20.1	39.5
3-OS-M1	30.0	12.7	35.0	7.7	15.2	49.8
3-OS-MT2	59.4	22.0	35.4	8.0	19.5	45.1

Table 3.8: Concentrations of components in middlings from BEU tests.

ID	Amount (g)	Fines (w/w% of oil sands)	Composition (g/1000 g of middlings)			
			Bitumen	Ultra-fines	ORS	Total solids
3-OS-E5	866	5.2	0. 2	3.0	0. 1	11
3-OS-E9	859	10.3	0. 3	6.0	0. 1	18
3-OS-MT1	866	11.7	1.0	4.0	0. 3	15
3-OS-M1	911	12.7	6.0	12.0	2.0	57
3-OS-MT2	880	22.0	4.0	7.0	1.0	29

Table 3.9: Concentrations of components in coarse tailings from BEU tests.

ID	Amount (g)	Fines (w/w% of oil sands)	Composition (g/1000 g of tailings)			
			Bitumen	Ultra-fines	ORS	Total solids
3-OS-E5	537	5.2	0. 9	2.0	0. 6	749
3-OS-E9	524	10.3	1	4.8	0. 4	751
3-OS-MT1	501	11.7	2	4.0	0. 6	746
3-OS-M1	489	12.7	3	6.1	1.0	741
3-OS-MT2	547	22.0	8	5.5	3	764

Table 3.10: Ultra-fines content of BEU tailings streams.

ID	Ultra-fines content (w/w% of oil sands)	Ultra-fines content of tailings streams (w/w% of oil sands)			Ultra-fines in middlings (percent of total)
		Middlings	Coarse Tailings	Middlings + Coarse Tailings	
3-OS-E5	0.7	0.5	0.2	0.7	71
3-OS-E9	1.2	1.0	0.5	1.5	67
3-OS-MT1	1.0	0.7	0.4	1.1	64
3-OS-M1	2.9	2.2	0.6	2.8	78
3-OS-MT2	1.6	1.2	0.6	1.8	67

Table 3.11: Bitumen distribution among BEU streams as w/w% of original oil sands.

ID	Bitumen in ore	Froth 1	Froth 2	Middlings	Coarse Tailings	Total in BEU streams
3-OS-E5	11.2	10.0	1.0	0.04	0.1	11.1
3-OS-E9	12.7	10.7	2.3	0.05	0.1	13.2
3-OS-MT1	10.9	8.4	2.2	0.2	0.2	11.0
3-OS-M1	7.4	3.6	2.1	1.1	0.3	7.1
3-OS-MT2	8.9	3.8	4.2	0.7	0.9	9.6

Table 3.12: Bitumen distribution among BEU streams as w/w% of total bitumen in original oil sands.

ID	Froth 1	Froth 2	Middlings	Coarse Tailings	Combined recovery
3-OS-E5	89.8	9.0	0.3	0.9	98.8
3-OS-E9	81.4	17.5	0.4	0.8	98.9
3-OS-MT1	76.4	20	1.8	1.8	96.4
3-OS-M1	50.7	29.6	15.5	4.2	80.5
3-OS-MT2	39.6	43.8	7.3	9.4	83.4

Table 3.13: ORS distribution among BEU streams as w/w% of original oil sands.

ID	Froth 1	Froth 2	Middlings	Coarse Tailings	Total ORS In BEU streams
3-OS-E5	0.4	0.3	0.02	0.06	0.8
3-OS-E9	0.6	0.4	0.02	0.04	1.1
3-OS-MT1	0.4	0.5	0.05	0.06	1.0
3-OS-M1	0.2	0.5	0.4	0.1	1.2
3-OS-MT2	0.2	1.0	0.2	0.3	1.7

Table 3.14: ORS distribution among BEU streams as w/w% of total ORS in original oil sands.

ID	**Froth 1**	**Froth 2**	**Middlings**	**Coarse Tailings**
3-OS-E5	51.3	38.5	2.4	7.8
3-OS-E9	56.6	37.7	1.9	3.8
3-OS-MT1	39.6	49.5	5.0	5.9
3-OS-M1	16.7	41.7	33.3	8.3
3-OS-MT2	11.8	58.8	11.8	17.6

Table 3.15: BITUMEN/ORS ratios for BEU streams.

ID	Feed	Froth 1	Froth 2	Middlings	Coarse Tailings
3-OS-E5	18.7	25.0	3.3	2.0	1.7
3-OS-E9	12.7	17.8	5.8	2.5	2.5
3-OS-MT1	9.9	21.0	4.4	4.0	3.3
3-OS-M1	7.4	18.0	4.2	2.8	3.0
3-OS-MT2	4.9	19.0	4.2	3.5	3.0

Note: For middlings and coarse tailings average BIT/ORS = 2.8 ± 0.7

Table 3.16: Total solids and ORS contents of BEU froths for oil sands 1-OS-E6 and 1-OS-E7.

ID	Total solids (w/w% of froth)		ORS (w/w% of froth)		Bitumen/ORS ratio		
	Primary froth	Secondary froth	Primary froth	Secondary froth	Primary froth	Secondary froth	Bulk
1-OS-E6	7.0	15.9	3.0	8.0	13.1	2.8	2.0
1-OS-E7	15.0	19.4	11.3	17.9	2.9	1.1	3.6

Note: Average BIT/ORS = 2.8 ± 0.8

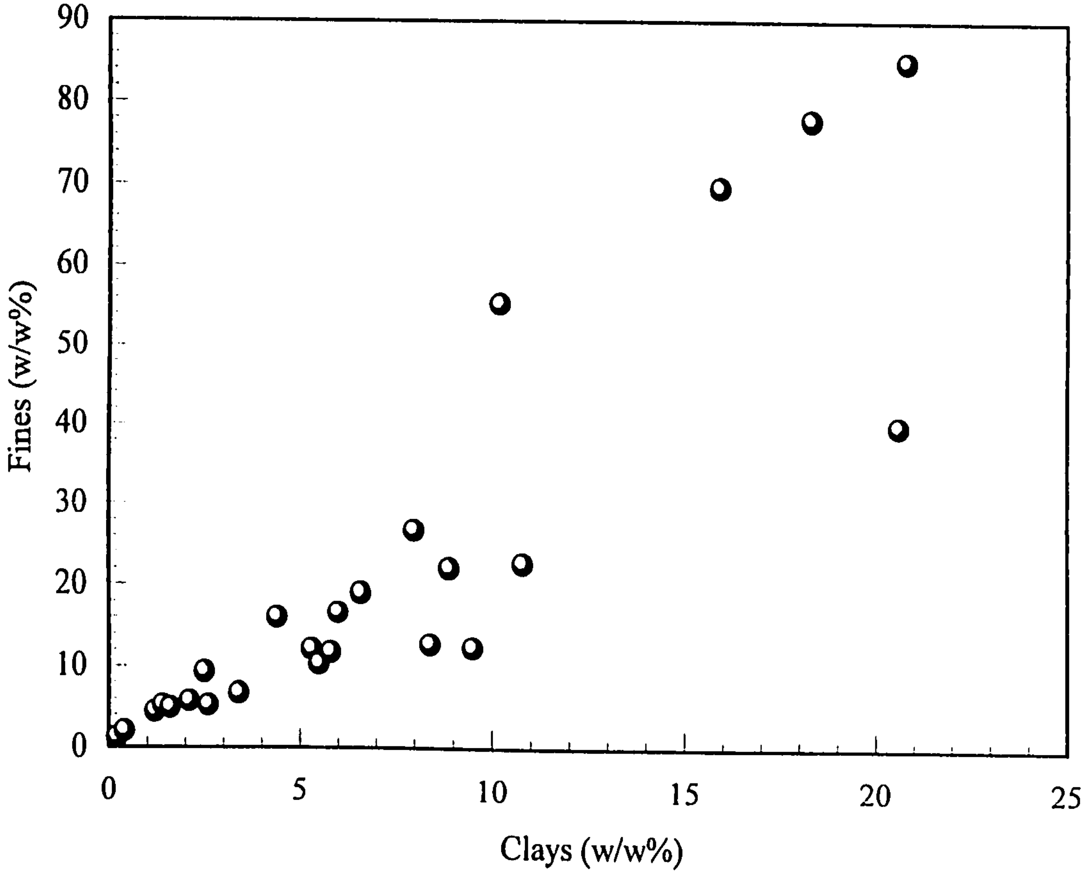


Figure 3.1: Fines vs. clay contents.

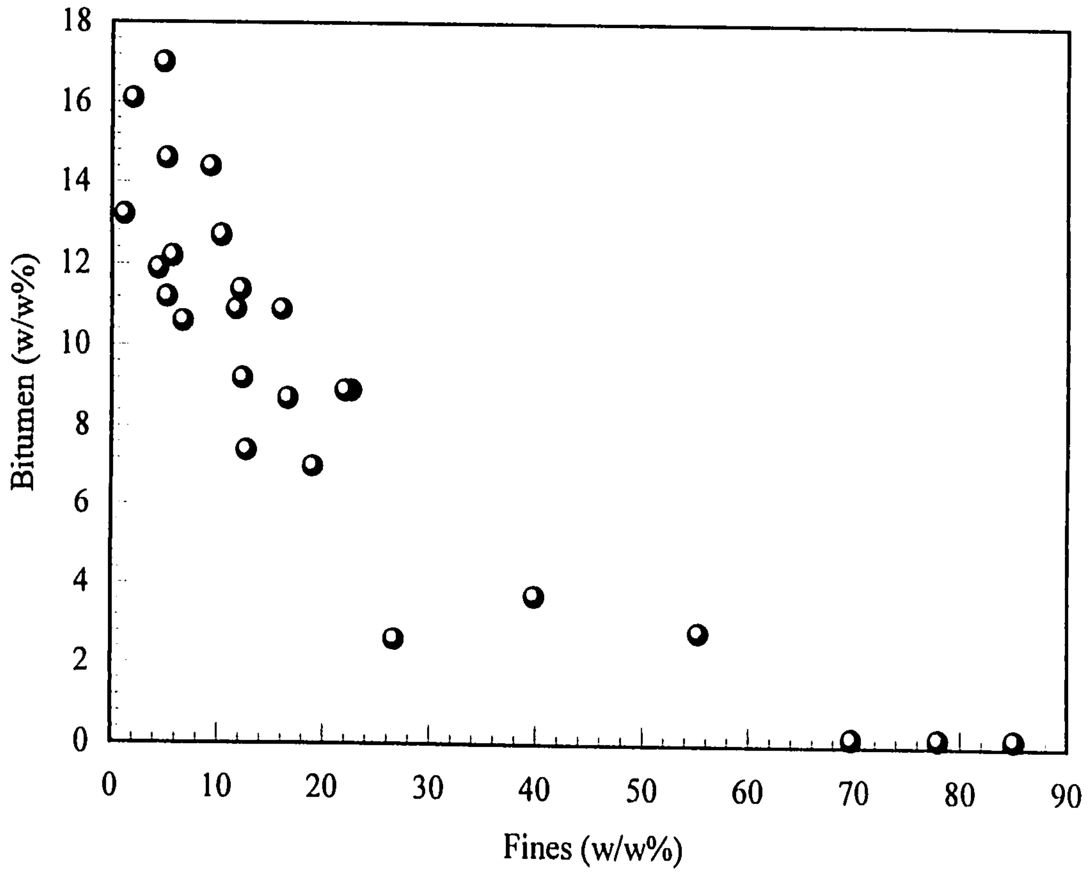


Figure 3.2: Bitumen vs. fines contents.

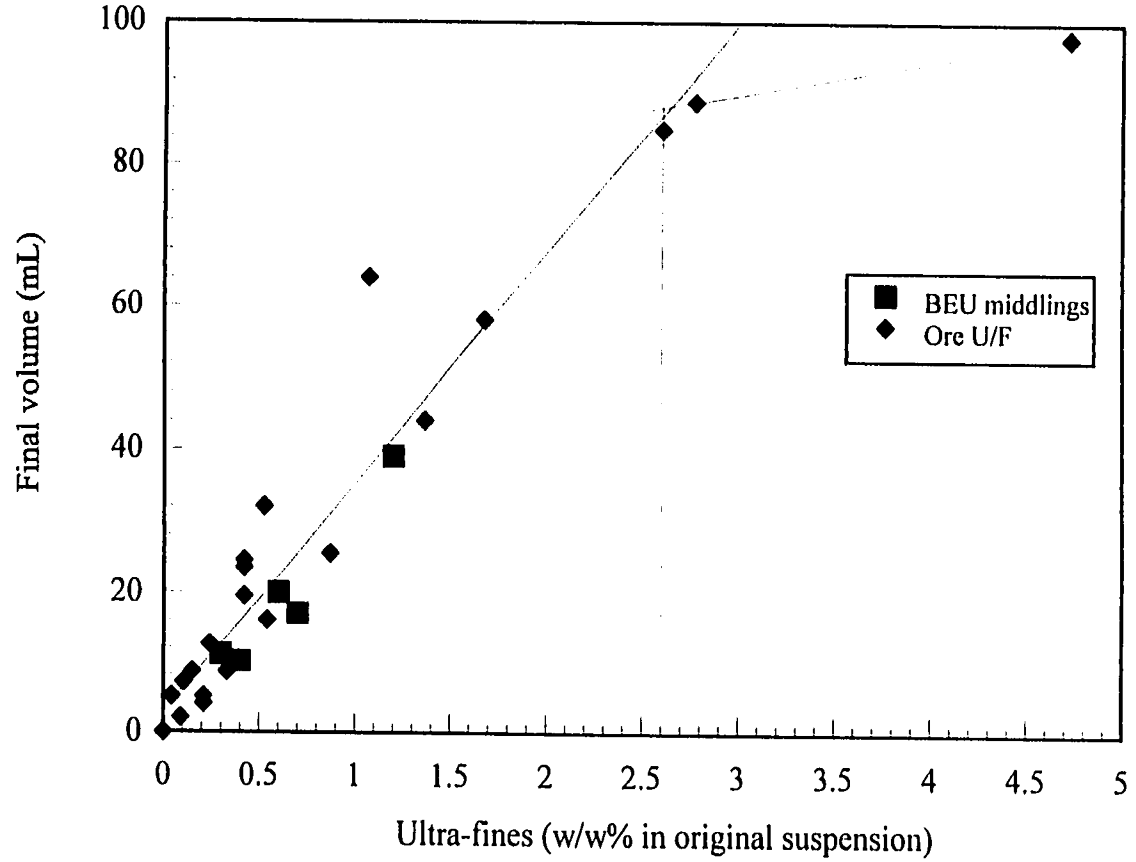


Figure 3.3: Final solids volumes vs. ultra-fines contents of original suspensions of oil sands.

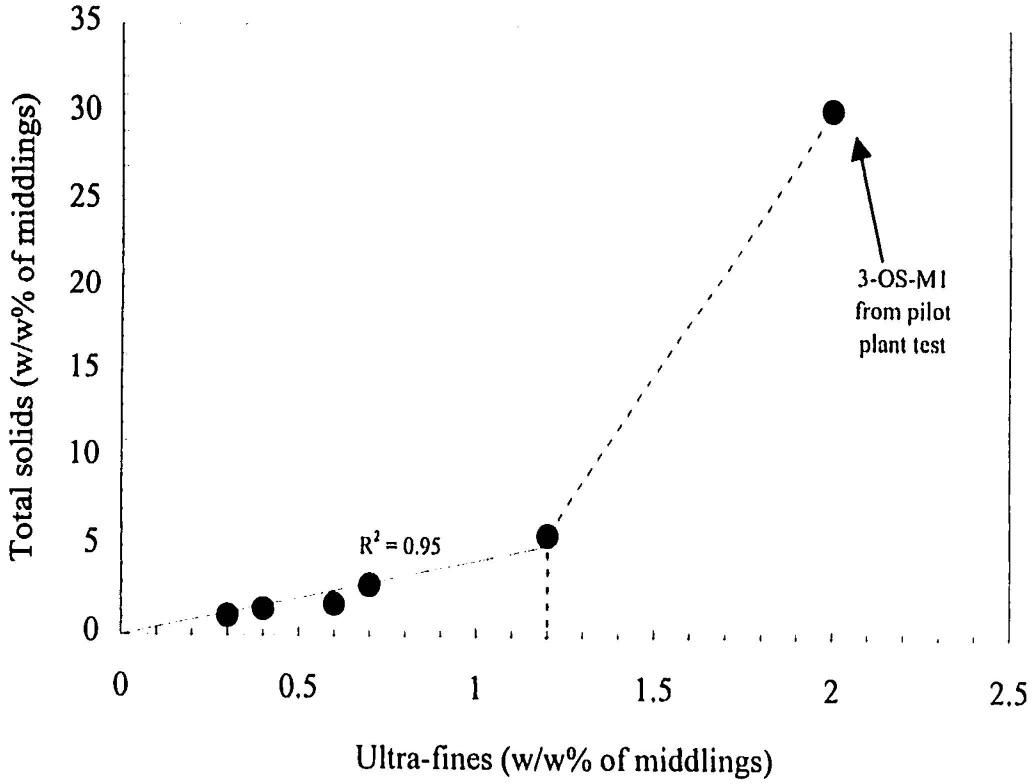


Figure 3.4: Total solids vs. ultra-fines content for BEU middlings.

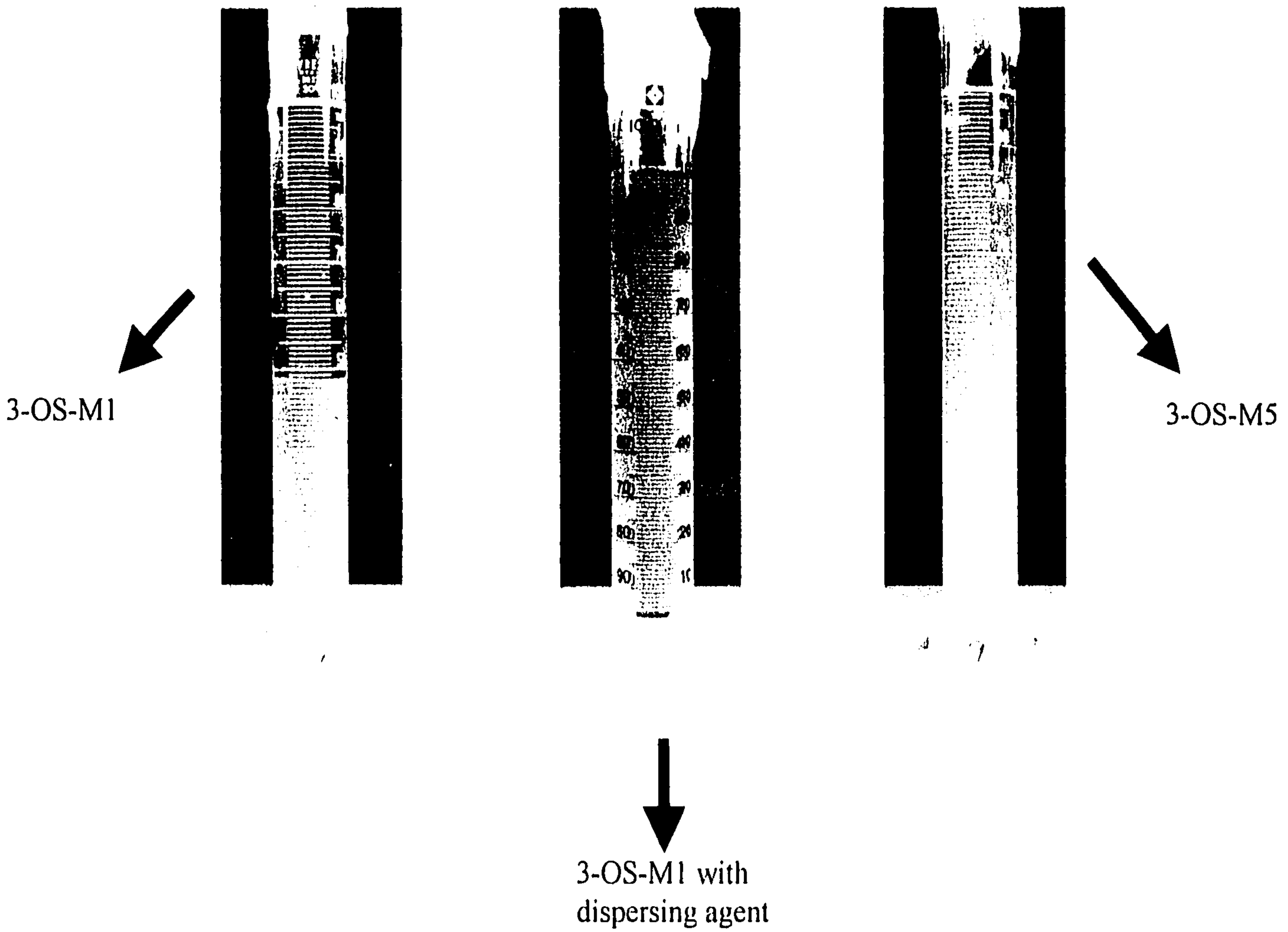


Figure 3.5: Settling test of BEU middlings.

Chapter 4

Discussion

4.1 General

Oil sands properties have been used for many years in an attempt to predict ore processability. In this work, two new criteria have been introduced to improve this important operational practice. Both ORS and ultra-fines content of an ore appear to have a great impact on bitumen separation in the HWEP. In this Chapter the significance of these properties with regard to processability will be explored.

4.2 Effect of Organic Rich Solids

Oil sands solids still contain some organic matter even after exhaustive treatment by multiple extractions with toluene. This carbonaceous material or toluene insoluble organic matter (TIOM), is strongly bound to certain inorganic minerals. It is represented in the most part by humic-like components. In aggregated, organic rich solids (ORS), humic matter and cementitious inorganic materials, such as siderite, iron oxide and calcite, act as binding agents for the clays and other mineral components. Aggregate properties represent a continuum in size, composition, density, porosity and surface wettability (Darcovich et al, 1989 and Kotlyar et al, 1988).

4.2.1 Correlation with current characterisation parameters

ORS were separated from oil sands samples using the method described in Section 2.3.2. The low bitumen content and/or extremely small particle sizes associated with waste units makes the determination of ORS difficult for these samples. While the waste units show the same trend as the other results, they were not included in the data analysis. Figure 4.1 summarizes fines and ORS contents for commercial ores showing normal processing behaviour. The solid line represents the linear regression for the data. While the regression is not particularly good, sample 1-OS-E7 falls well outside the 95% confidence limits represented by the dashed lines.

This anomalous ore has a high content of inorganic cementing agents, siderite and iron oxide, which may produce a larger than normal amount of stable, aggregated fines, see Table A.1. Also the ORS particles in this sample are relatively coarse, see Figure 4.2. This figure compares aqueous slurries of oil sands 1-OS-E6 and 1-OS-E7. In the latter case a distinct layer of coarse, dark ORS and associated bitumen is clearly seen. This material may correspond to so-called 'black sand', a heavy, mineral rich component of oil sands.

BEU separation tests were carried out on seven samples to examine the base behaviour of oil sands in order to confirm that the ORS component is the origin of anomalous behaviour. BEU primary bitumen recoveries were plotted against fines content, see Figure 4.3. Except for a single anomalous data point there is a common trend for recoveries and liberated bitumen to decrease with increase in fines content. While the linear regression line for primary recovery without the anomalous sample gives a correlation coefficient of only 0.81, the regression line including the anomalous sample is much worse at 0.18. This indicates that the behaviour of the anomalous sample is not well described by fines content. Liberated bitumen values are included for reference only.

4.2.2 Correlation with new characterisation parameters

BEU separation tests are a useful method to establish the distribution of ORS among the process streams and, in turn, to establish the effects of ORS content on bitumen recovery. As mentioned in Section 4.2.1, fines were plotted against ORS content of ore samples. Although an approximately linear correlation exists, the correlation was not appropriate for all of the samples tested. It was also noted, after BEU separation tests, that both recovery and liberated bitumen generally decreased with increase in fines content, see Figure 4.3. If recoveries are plotted against ORS contents of the oil sands ores the correlation changes for the better, compare Figures 4.3 and 4.4. The linear regression line for primary recovery, including the anomalous sample gives a correlation coefficient of 0.74 compared to a value of 0.18 obtained when the data is plotted against fines content, see Figure 4.3. While the data is not conclusive, it appears that ORS content may be a better predictor for bitumen recovery than fines content.

The results for two oil sands with similar fines contents but markedly different primary bitumen recoveries are particularly instructive, see Table 4.1. The important point to note is the significant difference in the ORS contents of the two ores. Higher ORS is associated with loss in bitumen recovery and appears to be a better indicator of process performance than fines content.

The ratio of bitumen to ORS in untreated oil sands covers the range from 1 to 23, see Table 3.3. The ratio increases with bitumen content and decreases with higher fines levels, see Tables 3.1 and 3.3. When primary bitumen recovery is plotted against BIT/ORS ratio, a rising trend is seen, see Figure 4.5. Recovery levels off at about 90 w/w% for BIT/ORS values of 20 and greater. Therefore, in order to achieve a primary recovery of at least 75 w/w%, the BIT/ORS ratio in the original oil sands must be more than 8:1. These results indicate that this ratio has potential for use as an "index" in the characterisation of oil sands ores. Much more work needs to be done to confirm this relationship.

Examining the relationship between ORS and bitumen can enhance the understanding of ORS and BEU primary recovery. Ideally, bitumen is separated from the inorganic matrix by a surface layer of interstitial water, allowing it to be easily "liberated" from the bulk mineral component. However, the humic material associated with ORS can adsorb, or bind, bitumen to both internal and external aggregate surfaces. In this work the bound bitumen is considered to be "unliberated"; the amount present is dependent on the concentration of ORS in a given ore. Therefore, an ore with a high concentration of ORS will also have a high amount of unliberated bitumen.

Another important observation was noted when BIT/ORS ratios were calculated for the middlings and tailings. It was found that the ratio was relatively constant (2.8±0.7) for the seven different ores tested under the same operating conditions; other process conditions may give different results. If the bitumen in the middlings and tailings streams is directly associated with ORS it may be considered unliberated. Knowing the BIT/ORS factor and the ORS content of oil sands, the liberated-unliberated bitumen balance (LUBB) can be estimated based on the ORS content of the different oil sands. For oil sands 3-OS-E5, the total ORS in the ore is 0.6 w/w%. The unliberated bitumen is therefore 0.6 multiplied by 2.8, which gives 1.7 w/w%. The liberated bitumen makes up the difference, 11.1 minus 1.7 w/w%, see calculation below.

ORS in ore = 0.6 w/w%

Unliberated bitumen = ORS in ore x (bit/ORS factor)
= 0.6 w/w% x 2.8
= 1.7 w/w%

Liberated bitumen = Total bitumen – Unliberated bitumen
= 11 w/w% - 1.7 w/w%
= 9.3 w/w%

Liberated bitumen values were found to compare well with primary recovery in froth 1 for separations in the BEU, see Table 4.2. Both liberated bitumen and primary recoveries are disproportionately low for sample 1-OS-E7. These observations reflect the fact that high levels of solids (ORS), strongly associated with bitumen, will reduce recovery for any water based separation method relying on density difference. Depending on the size and density of aggregates, these solids will either report with the bitumen froth, the primary tailings or remain with the middlings. If the aggregates are not rejected in the centrifuge tailings, they remain with the bitumen froth and adversely effect its quality. If the aggregates are coarser, with higher density, they will not float with the bitumen fraction. Instead, they will remain with the middlings or settle with the primary tailings, thus reducing bitumen recovery. A snapshot of ORS distribution in the PSV is seen in Figure 4.6. If aeration is introduced into the system, flotation will be enhanced. Bitumen recovery will be greater, but will contain more solids.

From the ORS contents of froths the distribution of unliberated bitumen among BEU streams was calculated as weight percent of total unliberated bitumen and total bitumen, respectively, see Tables 4.3 and 4.4. Combining unliberated bitumen in middlings and tailings provides an estimate of bitumen losses, see bulk column in Table 4.4. Based on these calculations the seven oil sands may be divided into two categories.

"Good": comprising oil sands 3-OS-E5, 3-OS-E9, 3-OS-MT1 and 1-OS-E6 in which losses are about 2 w/w% of total bitumen.

"Poor": comprising oil sands 3-OS-M1, 3-OS-MT2 and 1-OS-E7 where the losses are about 20 w/w% of total bitumen.

This division corresponds quite well with the sum of actual losses to the middlings and tailings in the BEU experiments, see Table 3.12. It is also in accordance with commercial and pilot operating experience.

Table 4.5 shows the distribution of liberated bitumen between primary and secondary froths. Some liberated bitumen reports with the secondary froth with the amounts increasing in the order of oil sands 3-OS-E5 to 3-OS-MT2. This additional bitumen is scavenged by the active aeration used to produce the secondary froth.

4.3 Effect of Ultra-fine Clays

A problem experienced in the full-scale HWEP is the sludging, or gelation, of oil sands slurries in the PSV. In these cases the ORS based prediction criterion developed in Section 4.2 does not hold. When a process upset occurs, segregation of solids and bitumen is prevented and oil recovery is reduced or eliminated.

4.3.1 Correlation with existing characterisation parameters

While the use of BEU tests is helpful in forecasting primary bitumen recoveries, the procedure does have limitations. The primary problem is that the test must be carried out at a much higher dilution than that used in commercial operation, i.e. 2:1 vs. 0.7:1. As a result, this method tends to obscure the possible adverse effects of ultra-fine clays content and water chemistry.

Ultra-fine clays are present in all Athabasca oil sands. These aluminosilicate clay particles have a major dimension of less than 0.3 μm and are no more than a few layers thick. In undisturbed oil sands these clays are naturally flocculated owing to the salts present in the formation water, see Figure 4.7. Until the oil sands is processed these flocs exist in a highly compressed state. When oil sands is slurried with recycle water the flocs are mobilised and released into the aqueous phase. Once free of the constraints imposed by the in-situ oil sands body the volume of flocs expands considerably.

After a conditioned slurry reaches the PSV the behaviour of the mobilised clay flocs is dependent on the following factors: (a) process water to oil sands ratio, (b) ultra-fines content and (c) slurry water chemistry, including contributions from both process and formation water. If the concentrations of flocculating cations and ultra-fines are above critical levels in the process slurry a thixotropic floc network, or gel, will form rapidly (Kotlyar et al, 1998a). A thixotropic gel is a structure that is weak enough to be broken by shearing but reforms when the shear stress is removed. In the relatively quiescent conditions of a PSV the floc domains formed in suspension begin to interact with each other to produce a structured system with a high viscosity, see again Figure 4.7. Coarser mineral particles and bitumen droplets are entrapped in the suspension. In this situation the slurry is non-segregating and little or no bitumen froth is generated. The rate of ultra-fines gelation, or structure formation, is probably the determining factor in the occurrence of sludging. For sludging to occur, the time for gelation must be less than the residence time in the PSV, i.e., of the order of minutes. This possibility has been demonstrated in previous work (Kotlyar et al, 1998a).

4.3.2 Correlation with new characterisation parameters

A plot of ultra-fines versus fines contents for all samples is shown in Figure 4.8. In general, the amount of ultra-fines is proportional to fines concentration but again some significantly anomalous results were noted. Waste units in particular do not follow the same trend and exhibit disproportionately high ultra-fines concentrations, especially sample 2-W-M3, see Table 3.2. Some samples, especially the low-grade marine oil sands, 3-OS-M1, have a disproportionately high ultra-fines content relative to other oil sands, see Figure 4.8. Sample 3-OS-M1 is known to produce PSV sludging in pilot tests carried out elsewhere (Cuddy, 1998). Although this point cannot be statistically identified as an outlier it falls well outside the 95% confidence limits for the regression line for the other data points. Also, it is considered significant because the ultra-fines content is in the same range as the critical gelation concentration identified earlier.

4.3.2.1 Determination of Zone of Operability

While ultra-fines and ORS are better predictors for process behaviour than fines alone they are difficult to measure and the procedures are probably too time consuming for general application. To overcome this problem, a jar test has been developed as a simple evaluation approach to identify problem ores, thus avoiding the time and expense needed for complete characterisation in every case. Once problem ores have been identified, more extensive tests can be performed.

Jar tests allow a visual assessment of segregating, intermediate and non-segregating slurries, see Figure 4.9. In Figure 4.9(a), segregation of particles is seen, i.e., coarse sediment and ultra-fines suspension. A bitumen froth has also separated. In (c), particle segregation is not evident, leaving bitumen entrapped in the mixture. Bitumen, as well as coarse solids are not visible because they are trapped in the floc matrix. Figure 4.9(b), demonstrates an intermediate stage of segregation and non-segregation.

The jar test is also a simple procedure for predicting the zone of operability based on ultra-fines and cation concentrations in oil sands slurries. A series of tests were performed to examine realistic ultra-fines and water chemistry combinations. In respect of the latter, Na^+ and Ca^{2+} are the primary flocculating ions in oil sands ores. The qualitative data obtained from tests with different ores and Na^+ concentrations is presented on a three-dimensional plot, see Figure 4.10. The shaded areas represent the response surface between segregating and non-segregating conditions. Ideally, the process should be operated in the segregating zone.

From these observations it may be deduced that ore slurries containing 1.1 w/w%, or less, of ultra-fines are always segregating regardless of Na^+ concentration. On the other hand, ores containing 2 w/w% of ultra-fines are always non-segregating when Na^+

concentration is above 500 ppm. Intermediate behaviour is observed under other conditions.

Similarly, the effect of ultra-fines and Ca^{++} concentrations on oil sands slurry segregation is presented on Figure 4.11. In this case concentrations of Ca^{++} as low as 40 ppm are enough to cause non-segregation for oil sands containing 2 w/w% ultra-fines.

An important observation in these tests is that the change from segregating to non-segregating conditions can be very dramatic; i.e., only a small change in ultra-fines concentration is sufficient to cause upset conditions when the concentration of cations is high enough.

4.3.3 Application of Zone of Operability

Based on these tests it can be seen that the ultra-fines content for oil sands 1-OS-E12 falls within the critical range of 1.4 to 2 w/w%, see Table 3.2. Flocculating cations, Ca^{++} and Mg^{++}, with 31 and 7 ppm respectively, are also present in the formation water in amounts sufficient to induce fast, i.e., within minutes, thickening, or gelation, see Table A.2 (Kotlyar et al, 1998a). Cations contributed from the recycle water present in the slurry will accelerate the gelation process. These oil sands properties allow one to predict that, for process water to oil sands ratios of less than 1 to 1, a slurry of this ore will gel rapidly in the PSV. Bitumen and coarser solids will be entrapped in the gel network and practically no segregation of components will occur. The dilution ratio is important because gelation can be prevented if the ratio is high enough.

This prediction is in accord with the observation illustrated on Figure 4.12. The photograph shows oil sands 1-OS-E12 and 1-OS-E6 slurried with distilled water in the ratio of 1 to 0.7. It is obvious that the response of the two samples is quite different with 1-OS-E12 showing no solids segregation or bitumen separation. However, this sludging behaviour of oil sands 1-OS-E12 can be reduced by using a water to oil sands ratio of 2 to 1, see Figure 4.13.

4.3.4 Waste units

Because of mining constraints, waste units with low bitumen content must often be mined as part of the ore. This results in an increase in the fines and clay contents of plant feed. The jar test can be used as an aid to determine whether more selective mining will result in better processability. Hence, the amount of waste material that can be tolerated has been determined for two good ores.

Jar tests were performed using the oil sands, their corresponding waste units and blends of the two to provide information on process performance for the different samples. The behaviour of two estuarine oil sands and their waste units is shown on Figure 4.14. The oil sands have low ultra-fines and flocculating cation contents and consequently show no sludging behaviour, even at a water to ore ratio of 0.7 to 1. Waste unit samples from the same geological environment are heavily enriched in both ultra-fines and flocculating salts, see Tables A.2 and 3.2. Sludging is clearly evident. Even at a higher water to sample ratio of 2 to 1 the waste units still exhibit strongly non-segregating behaviour, see Figure 4.15. Only deflocculation of the ultra-fines by addition of a strong dispersing agent, such as sodium pyrophosphate, at a water to oil sands ratio of 2 to 1, leads to segregation of the waste unit samples, see Figure 4.16. Phosphate anions have the opposite effect to cations and decompress the electric double layer.

Jar tests for both marine oil sands and their waste units at low dilutions are shown on Figure 4.17. The non-segregating behaviour is not surprising considering that the ultra-fines and Na^+ concentrations in these samples are above, or close to, critical levels. Increasing the slurry dilution to 2 to 1 reduces the salt concentrations sufficiently to produce segregation for both oil sands and waste units, see Figure 4.18.

4.3.5 Oil sands-waste units blends

Another series of jar tests were performed to determine the minimum amount of waste material that could be combined with the ore without causing sludging in the PSV. Jar tests indicate that the addition of 20 to 30 w/w% of the corresponding waste material to estuarine oil sands is sufficient to cause sludging. This is an important observation because it shows the importance of mining practices. These tests show that selective mining is important because a concentration of only 20 w/w% waste material can induce gelation, see Figures 4.19 and 4.20.

4.4 Application of oil sands characteristics to processability prediction

A decision tree can be developed to allow logical application of the characterisation tests described above to provide detailed information on oil sands processability. The proposed sequence for decision making is schematically presented on Figure 4.21.

First, a jar test is performed to check for potential separation problems. If the jar test indicates segregation, then normal procedures are followed. On the other hand, if the jar test indicates non-segregation, then the oil sands must be analysed for ultra-fines, organic rich solids and formation water chemistry. In addition the process water chemistry must be known. With this information it is possible to predict whether there will be a sludging problem in the PSV, based on the amount of process water added. If the ultra-fines exceeds 1.4 w/w% of the middlings there will be a sludging problem if the middlings contain at least 500 ppm of Na^+ or 40 ppm of Ca^{2+}; some combination of mono- and di-valent ion concentration will also have the same effect. If flocculating cation concentrations fall below these critical levels then no problem is indicated. The next step is to determine bitumen recovery and final tailings volume.

Based on BEU tests bitumen recovery may be estimated by determining the unliberated bitumen content of the oil sands. This parameter is calculated from an ore's ORS content and the average BIT/ORS factor determined from many middlings and tailings samples produced by BEU tests on different ores under the same separation conditions.

Final tailings volumes are estimated from the amounts of ultra-fines in the PSV middlings. This calculation is based on the observation that under gravity, tailings will settle until their ultra-fines concentration reaches a critical concentration of 2.5 to 3 w/w%, see Figure 3.3. This approach is only possible because coarser particles, trapped in the tailings, occupy spaces within the ultra-fines network and do not contribute to the overall tailings volume.

When the ultra-fines content and formation water chemistry of an ore indicate that PSV sludging is a possibility then measures must be taken to prevent, or moderate, ultra-fines gelation. The options available are limited. Because the problem is caused by critical concentrations of ultra-fines and salts one obvious remediation action is to dilute the slurry. This is typically the route followed in current operating practise. Sufficient water must be added to bring the ultra-fines content to less than 1.4 w/w% in the PSV. Another approach is to prevent gelation from occurring by adding a strong dispersing agent, for example sodium silicate or sodium pyrophosphate.

Once gelation is no longer an issue, bitumen recovery can be estimated using the liberated-unliberated bitumen balance. Using the average value of 2.8 for the BIT/ORS ratio, the amounts of bitumen recovered in the primary and secondary froths can be calculated.

Table 4.1: Amounts of froth and bitumen contents for oil sands 1-OS-E6 and 1-OS-E7.

ID	**Froth (g)**		**Bitumen (w/w% of froth)**		**Bitumen recovery (w/w%)**	
	Primary	**Secondary**	**Primary**	**Secondary**	**Primary**	**Secondary**
1-OS-E6	63.0	26.4	69.7	56.9	71.9	24.6
1-OS-E7	28.3	19.8	52.2	48.9	28.9	17.3

Table 4.2: Liberated and unliberated bitumen contents from BEU tests.

ID	Froth 1 (w/w% of total bitumen in oil sands)	Unliberated bitumen (w/w% of oil sands)*	Bitumen Type (w/w% of total in feed)	
			Liberated	**Unliberated**
3-OS-E5	89.8	1.8	85	15
3-OS-E9	81.4	3.0	80	20
3-OS-MT1	76.4	3.3	70	30
3-OS-M1	50.7	3.0	60	40
3-OS-MT2	39.4	5.4	40	60

* Calculated knowing ORS and bitumen content from spectroscopic method and based on the assumption that unliberated bitumen to ORS ratio is 2.8.

Table 4.3: Distribution of unliberated bitumen among BEU streams.

ID	**ORS (w/w% of oil sands)**	**(w/w% of oil sands)**			**(w/w% of unliberated bitumen in oil sands)**		
		Froth1*	**Froth 2***	**Bulk****	**Froth1***	**Froth2***	**Bulk***
3-OS-E5	0.6	1.2	0.9	0.2	52	39	9
3-OS-E9	1.0	1.8	1.2	0.2	56	38	6
3-OS-MT1	1.1	1.2	1.5	0.3	40	50	10
3-OS-M1	1.0	0.6	1.5	1.5	16	42	42
3-OS-MT2	1.8	0.6	3.0	1.5	12	59	29

* Calculated by multiplying results for ORS in froth 1 and froth 2 by LUBB factor 2.8.

** Tailings + middlings

Table 4.4: Distribution of unliberated bitumen among BEU streams.

ID	**ORS (w/w% of oil sands)**	**w/w% of total bitumen in oil sands**		
		Froth1*[*]*	**Froth 2**[*]	**Bulk**[**]
3-OS-E5	0.6	11	8	2
3-OS-E9	1.0	14	9	2
3-OS-MT1	1.1	11	14	3
3-OS-M1	1.0	8	20	20
3-OS-MT2	1.8	7	34	20

* Calculated by multiplying results for ORS in froth 1 and froth 2 by LUBB factor 2.8.

** Tailings + middlings

Table 4.5: Distribution of liberated bitumen among BEU froths.

ID	**ORS (w/w% of oil sands)**	**w/w% of oil sands***		**w/w% of total liberated bitumen in oil sands**	
		Froth1	**Froth 2**	**Froth1**	**Froth 2**
3-OS-E5	0.6	8.8	0.1	99	1
3-OS-E9	1.0	8.9	1.1	89	11
3-OS-MT1	1.1	7.2	0.7	91	9
3-OS-M1	1.0	3.0	0.6	83	17
3-OS-MT2	1.8	3.2	1.2	73	27

* Calculated knowing total bitumen and unliberated bitumen.

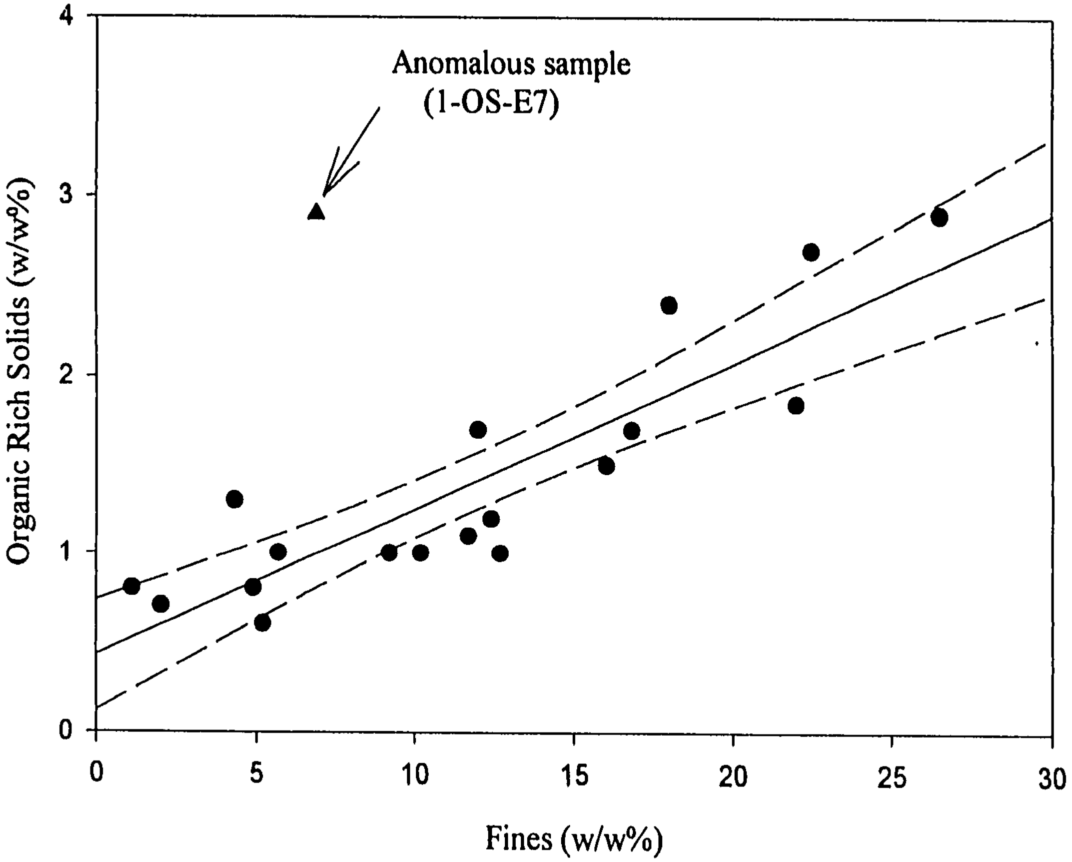


Figure 4.1: Organic Rich Solids(ORS) vs. Fines contents for samples in normal commercial operating range

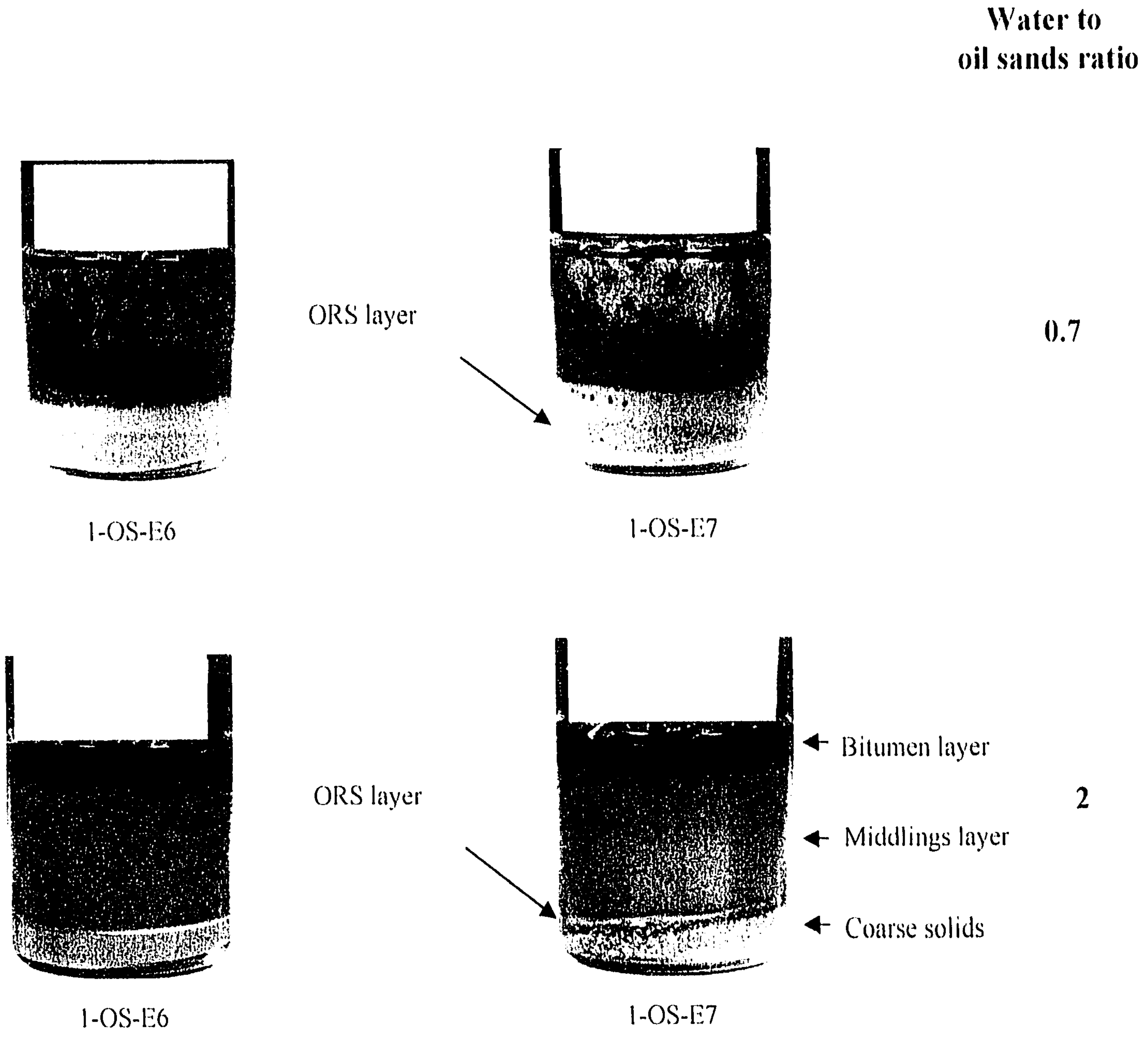


Figure 4.2: Segregation tests.

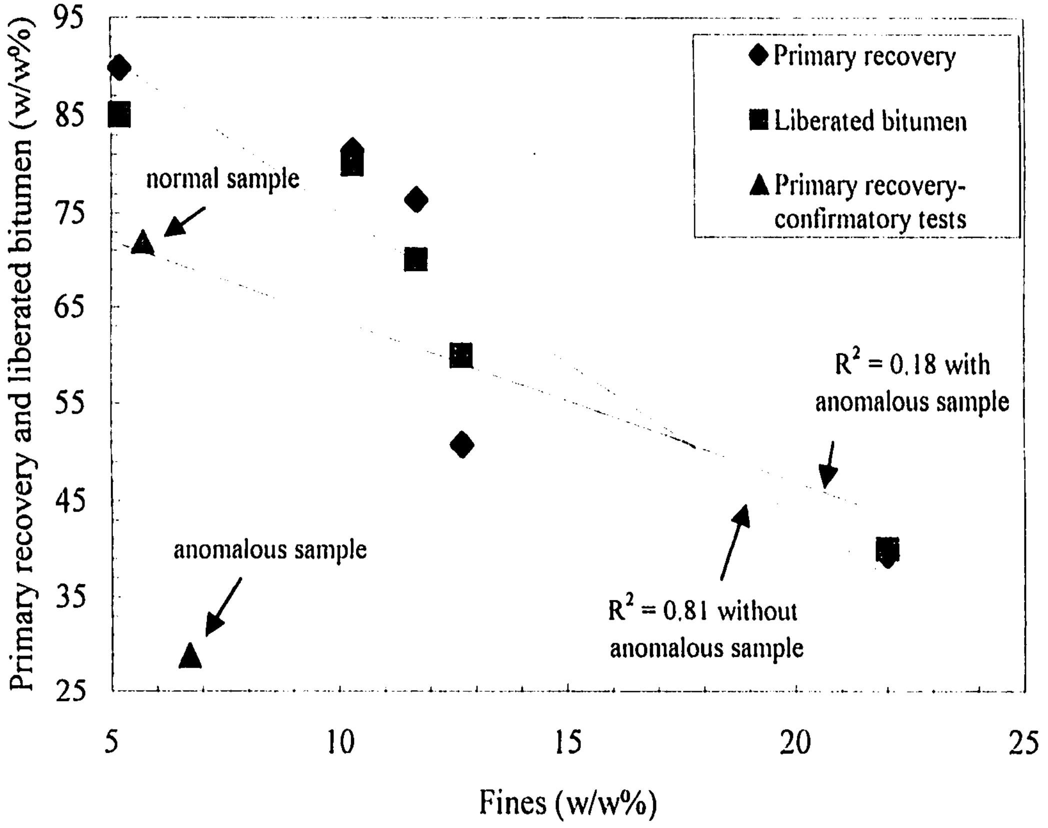


Figure 4.3: Primary recovery and liberated bitumen vs. fines content.

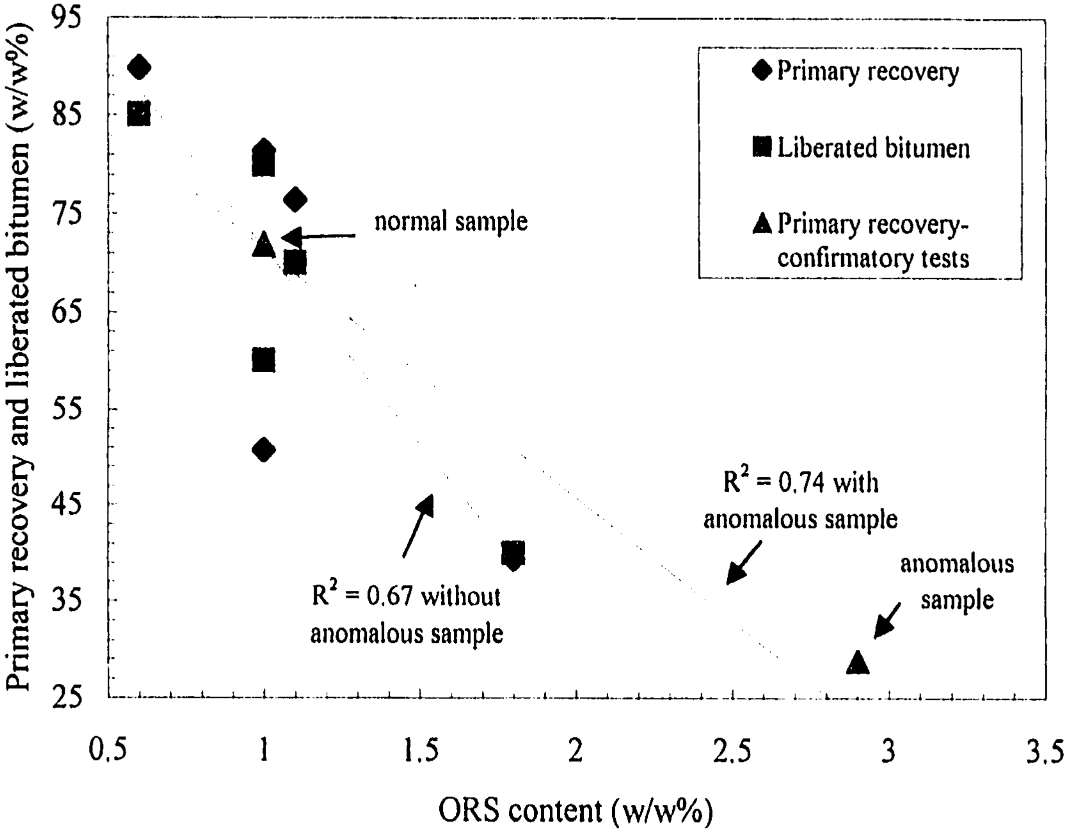


Figure 4.4: Primary recovery and liberated bitumen vs. ORS content.

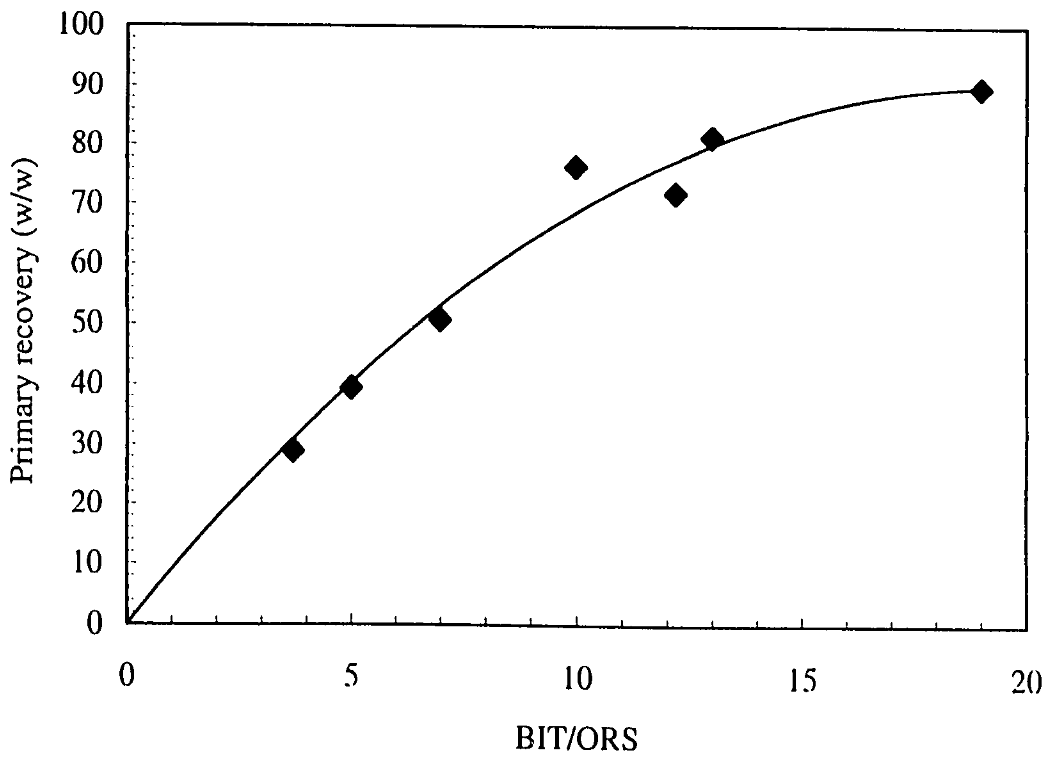


Figure 4.5: Primary recovery vs. BIT/ORS.

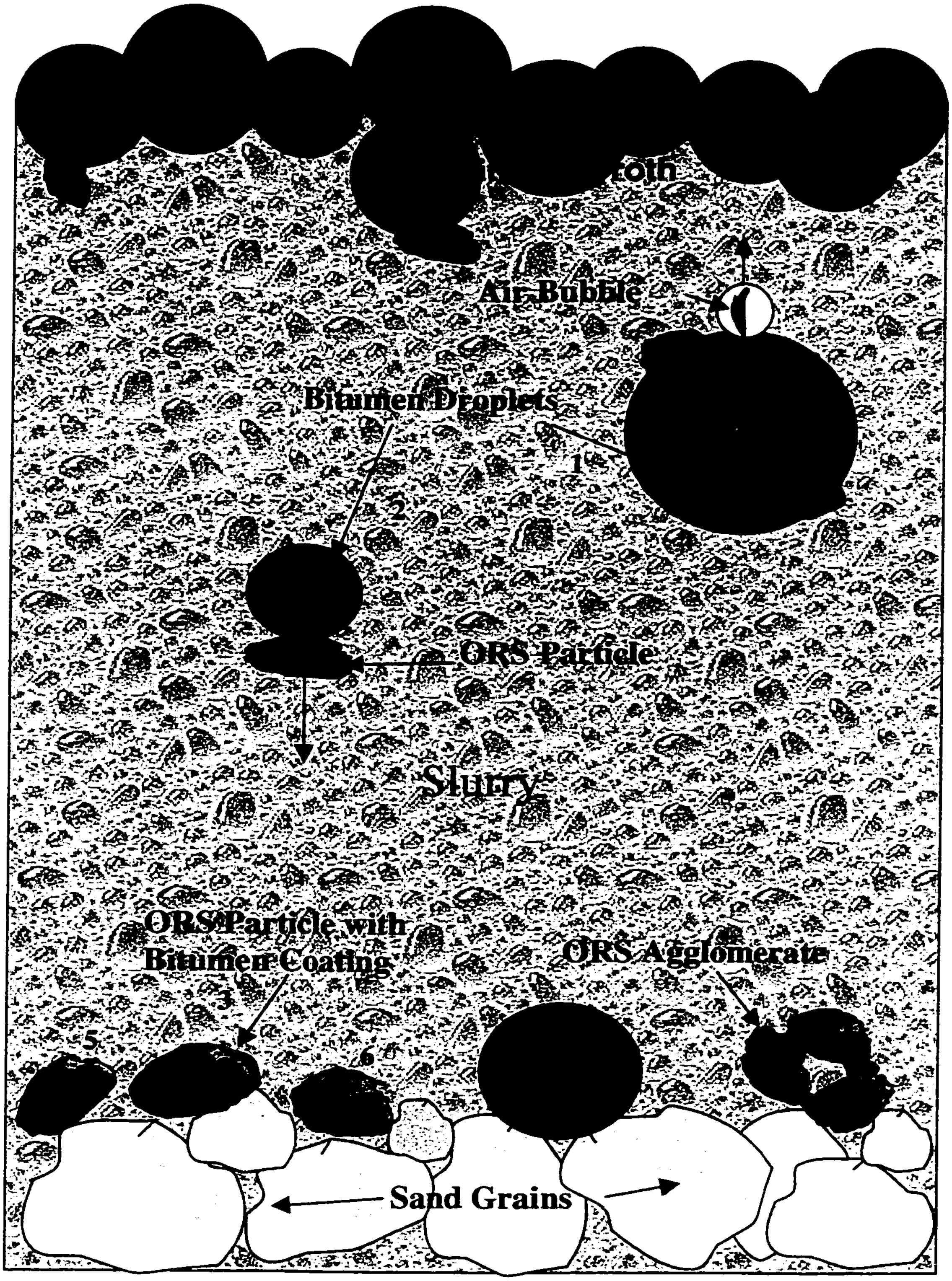


Figure 4.6: Distribution of ORS and bitumen in PSV

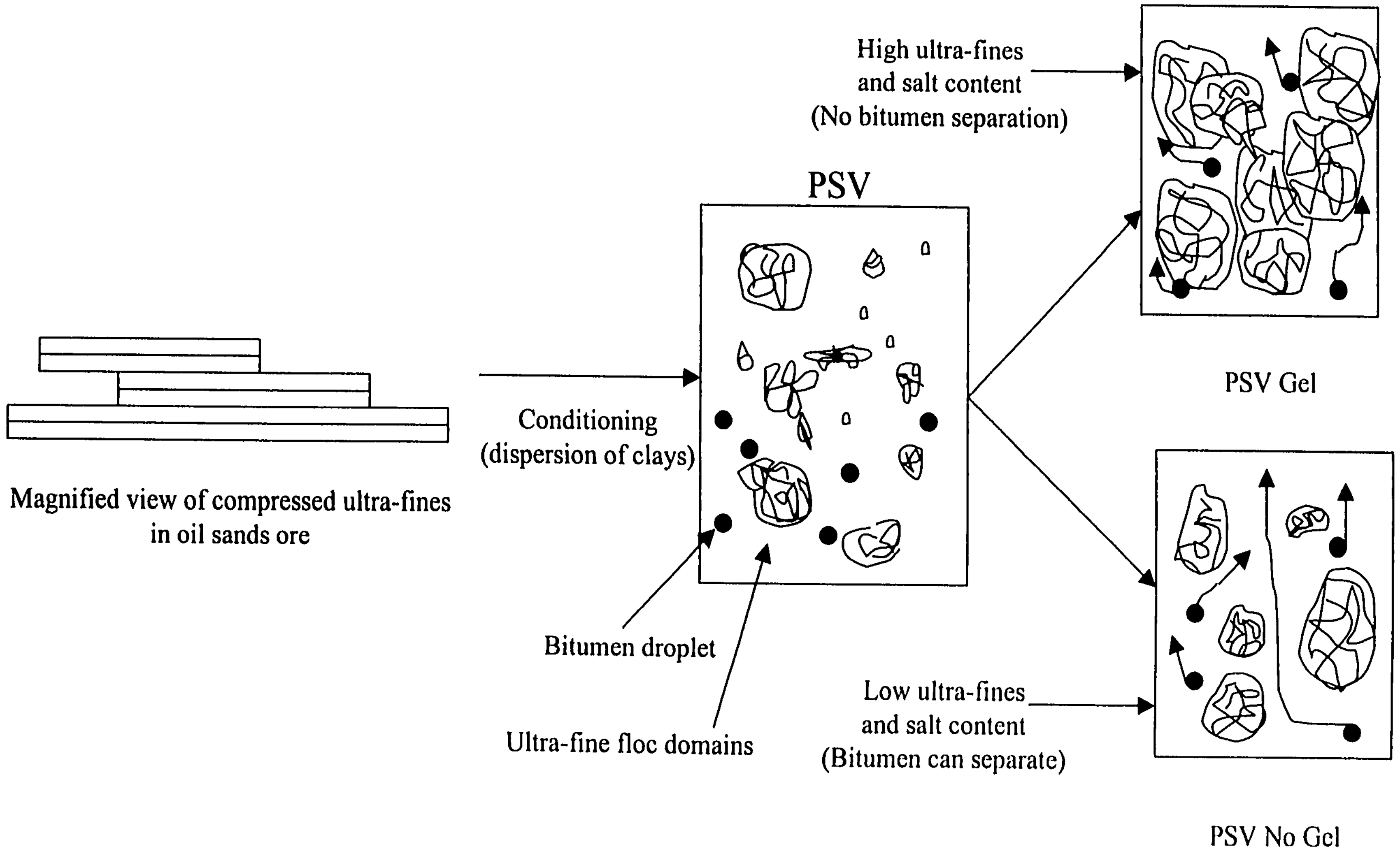


Figure 4.7: Effect of Conventional and Ultra-fine Clays on Performance of PSV.

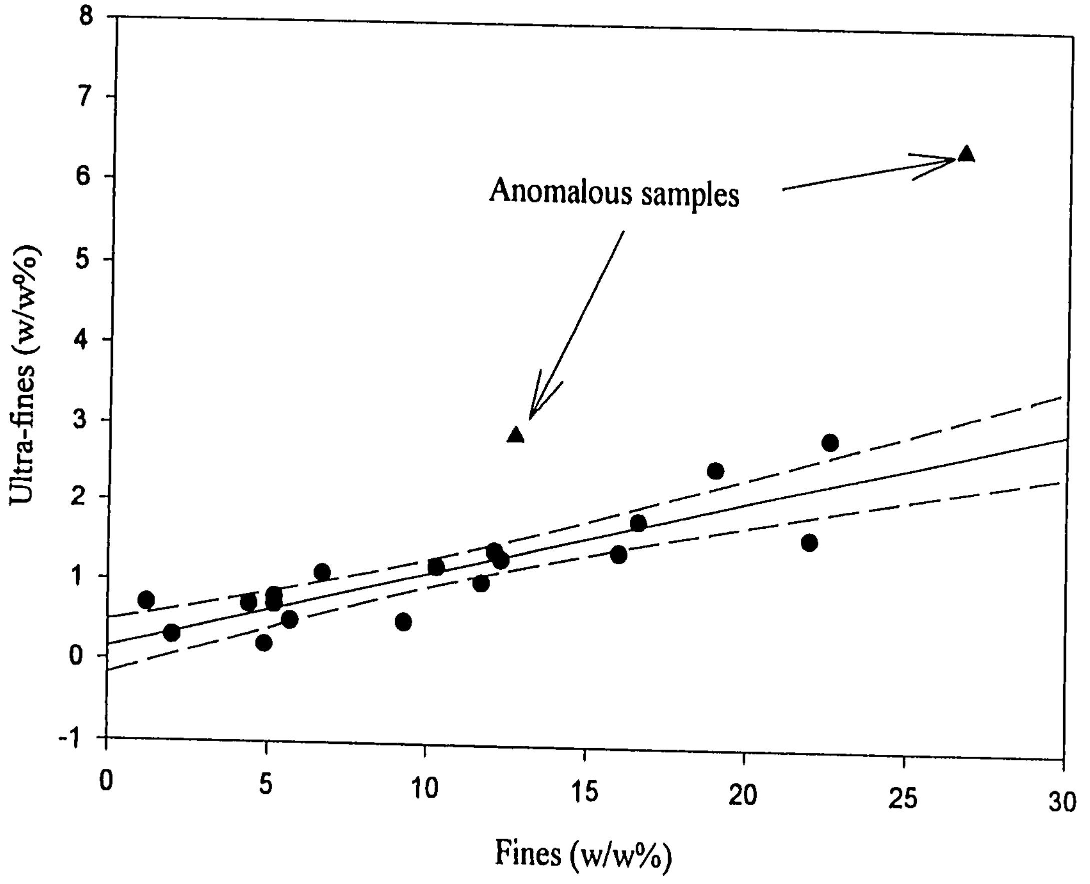


Figure 4.8: Ultra-fines vs. Fines contents for samples in normal commercial operation range

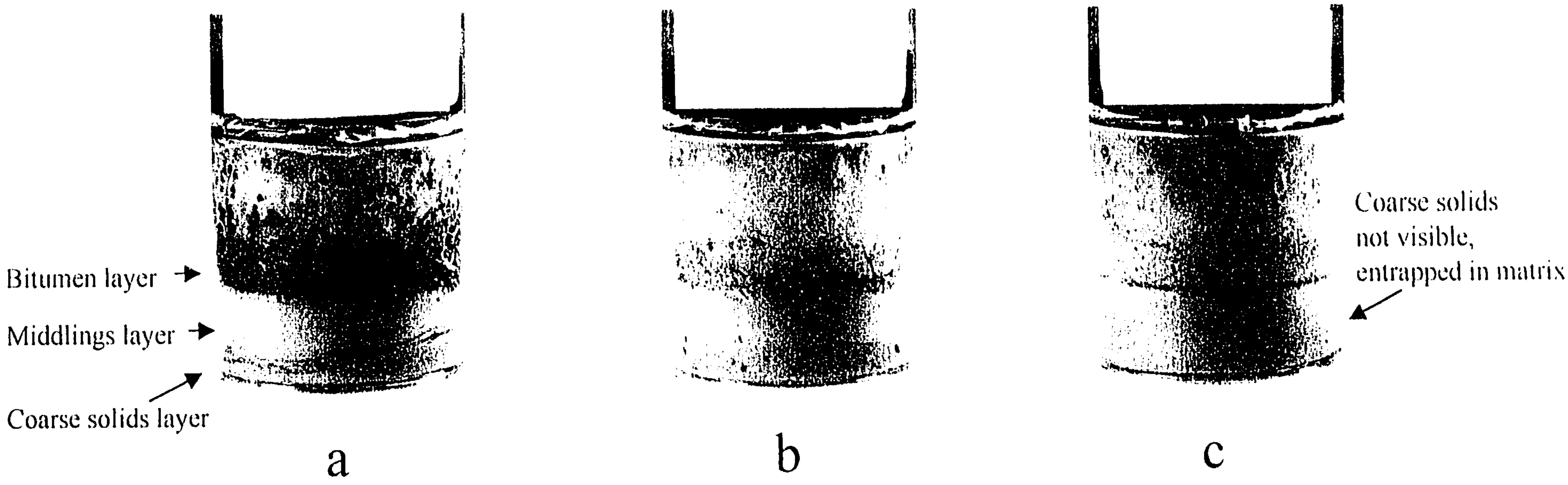
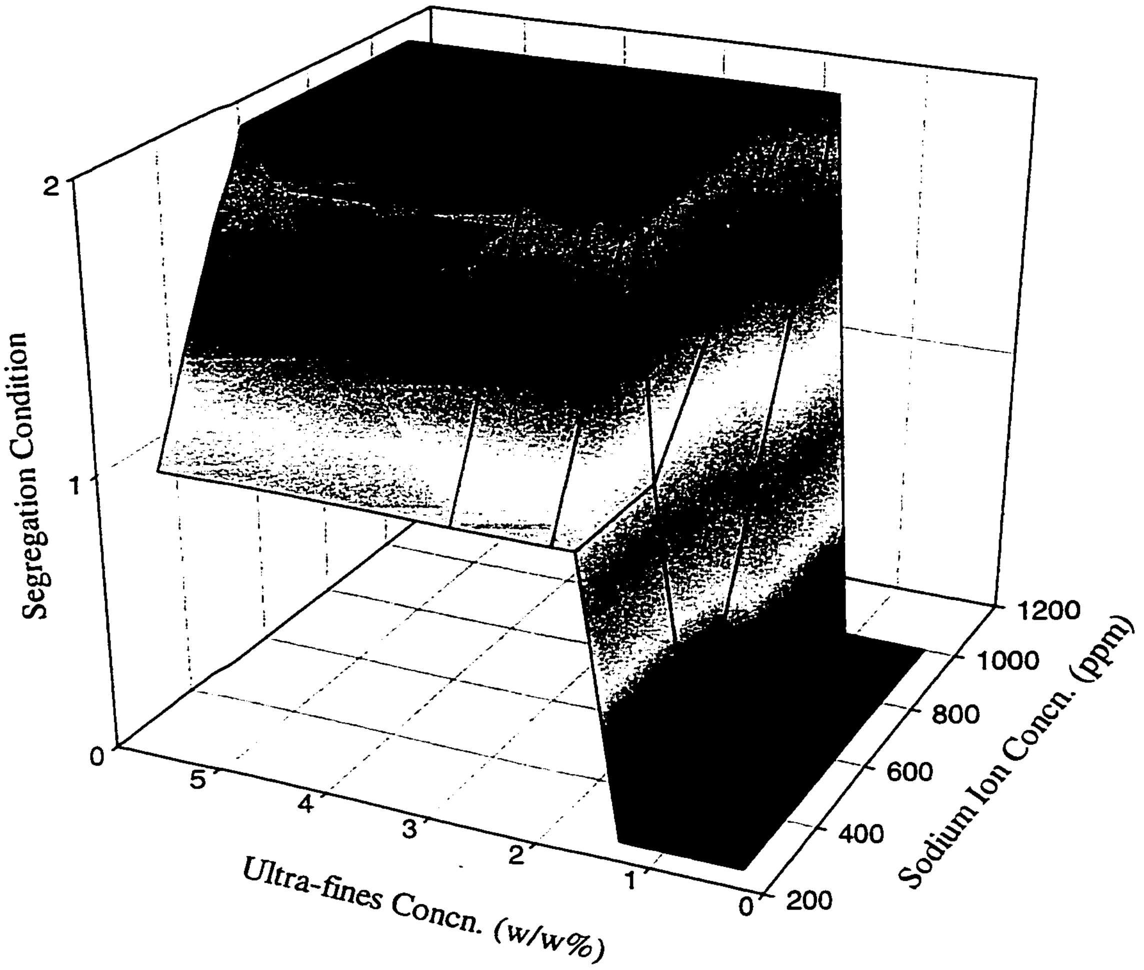
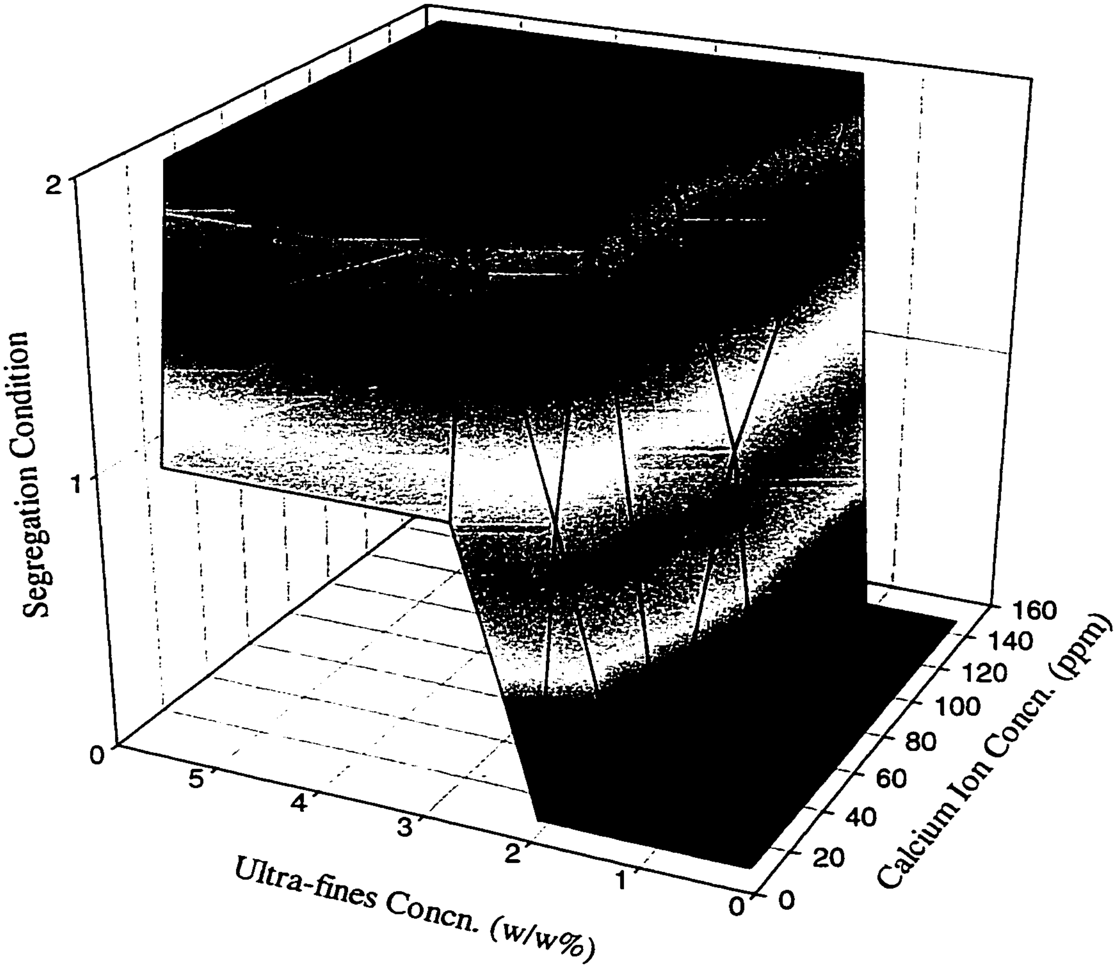


Figure 4.9: Segregating, intermediate and non-segregating systems.



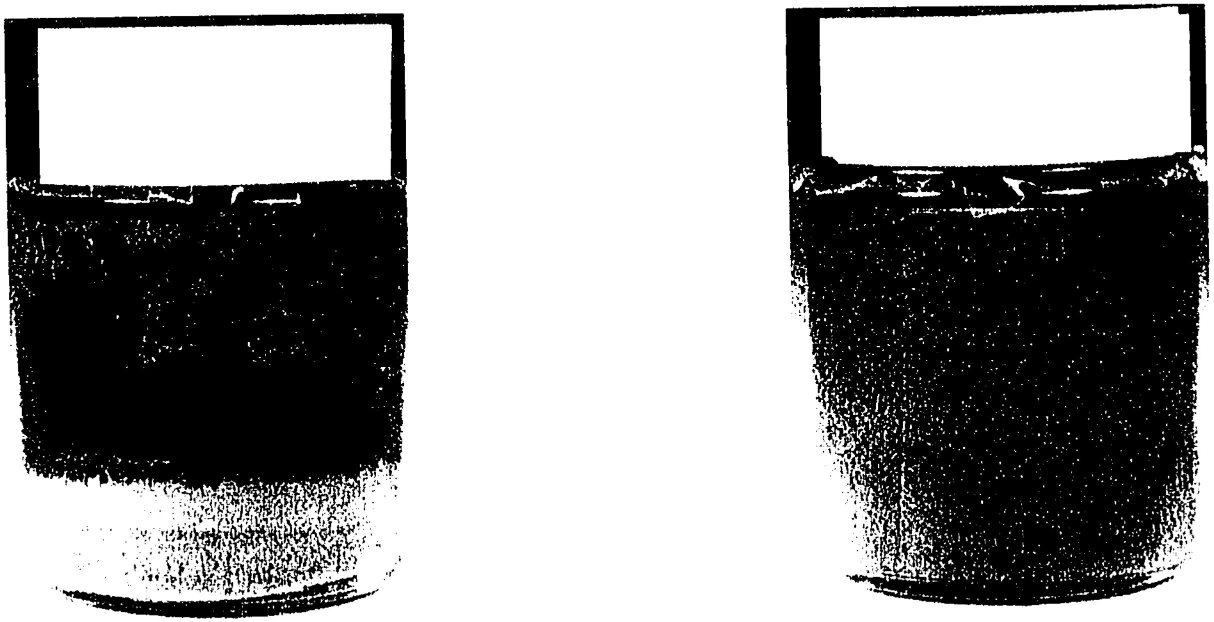
Segregation Conditions:
0 = segregating
1 = intermediate
2 = non-segregating

Figure 4.10: Response Surface for Ore Segregation.
Effect of Ultra-fines and Sodium Ion Concentrations



Segregation Conditions:
0. Segregating
1. Intermediate
2. Non-segregating

Figure 4.11: Response Surface for Ore Segregation.
Effect of Ultra-fines and Calcium Ion Concentrations

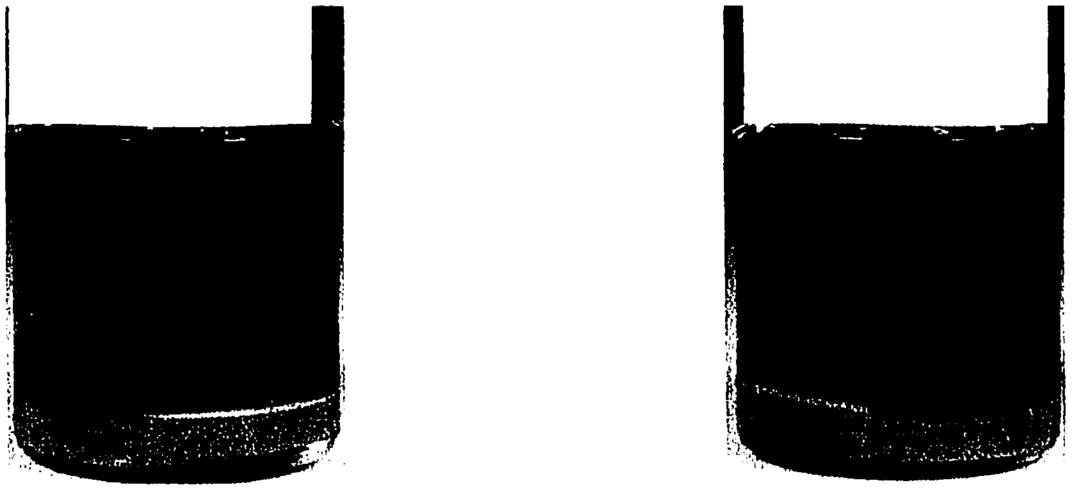


1-OS-E6

1-OS-E12

Water to oil sands ratio 0.7

Figure 4.12: Segregation and non-segregation comparison.



1-OS-E6

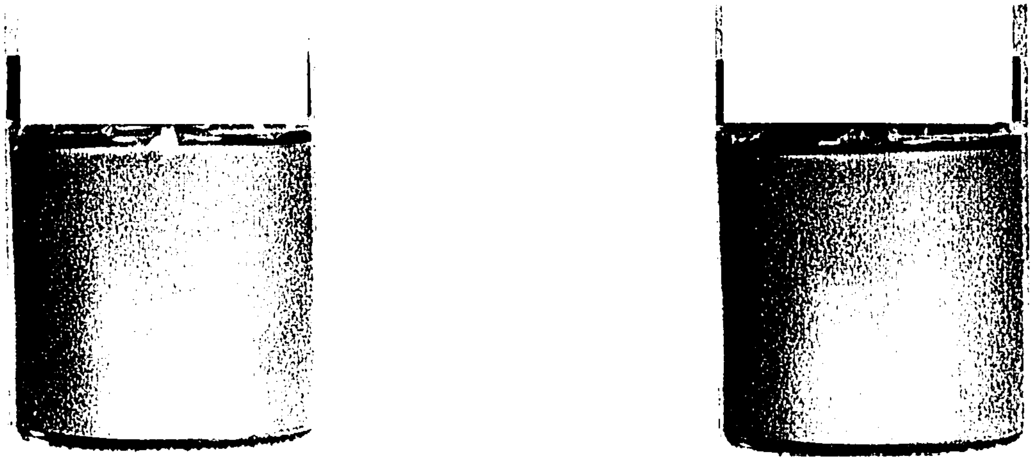
1-OS-E12

Water to oil sands ratio 2

Figure 4.13: Segregation test at high water to oil sands ratio.

2-OS-E3

2-OS-E4

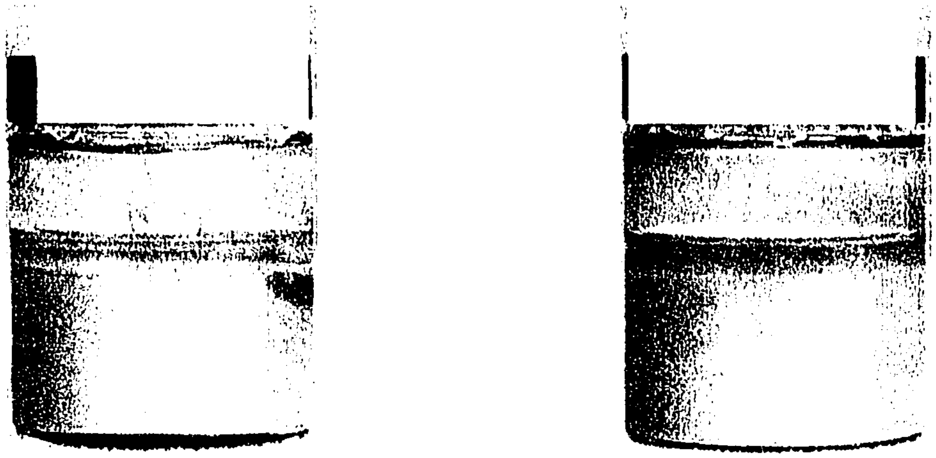


2-W-E3

2-W-E4

Water to oil sands ratio is 0.7

Figure 4.14: Estuarine oil sands and waste units.

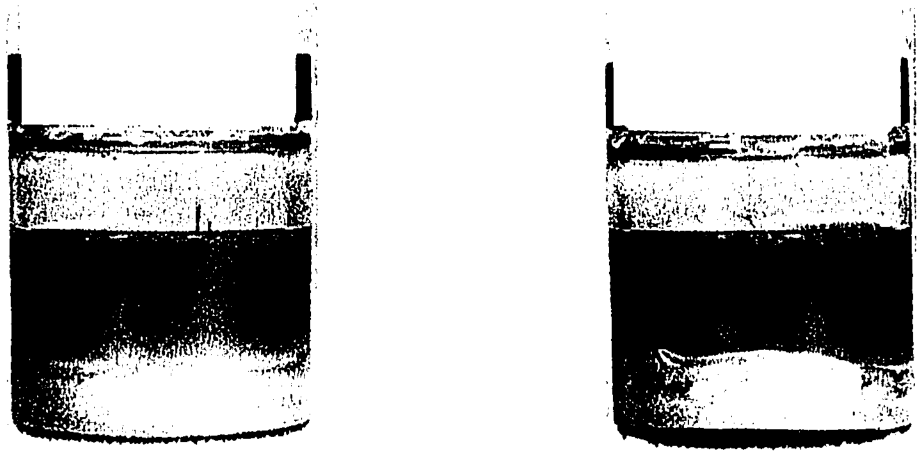


2-W-E3

2-W-E4

Water to oil sands ratio is 2

Figure 4.15: Estuarine waste units.

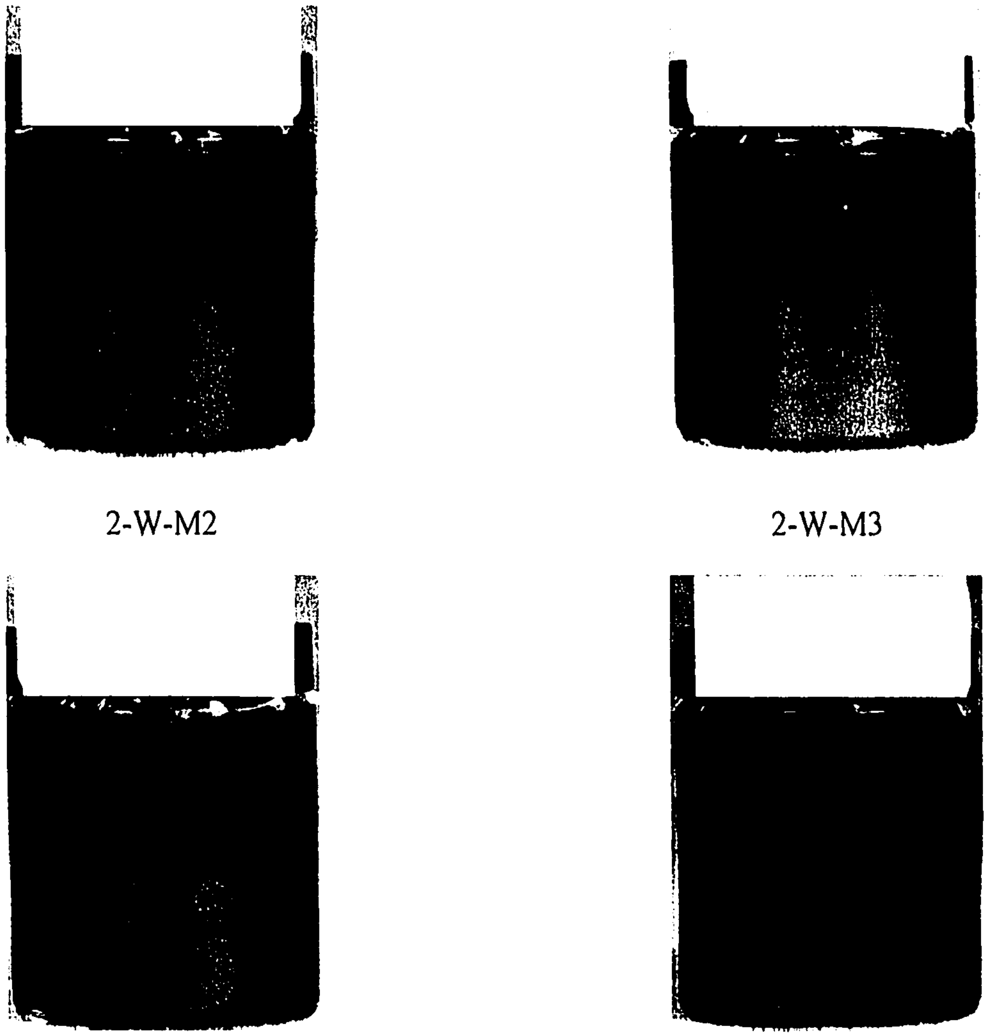


2-W-E3

2-W-E4

Water to oil sands ratio is 2

Figure 4.16: Estuarine waste material in sodium pyrophosphate solution.



2-W-M2

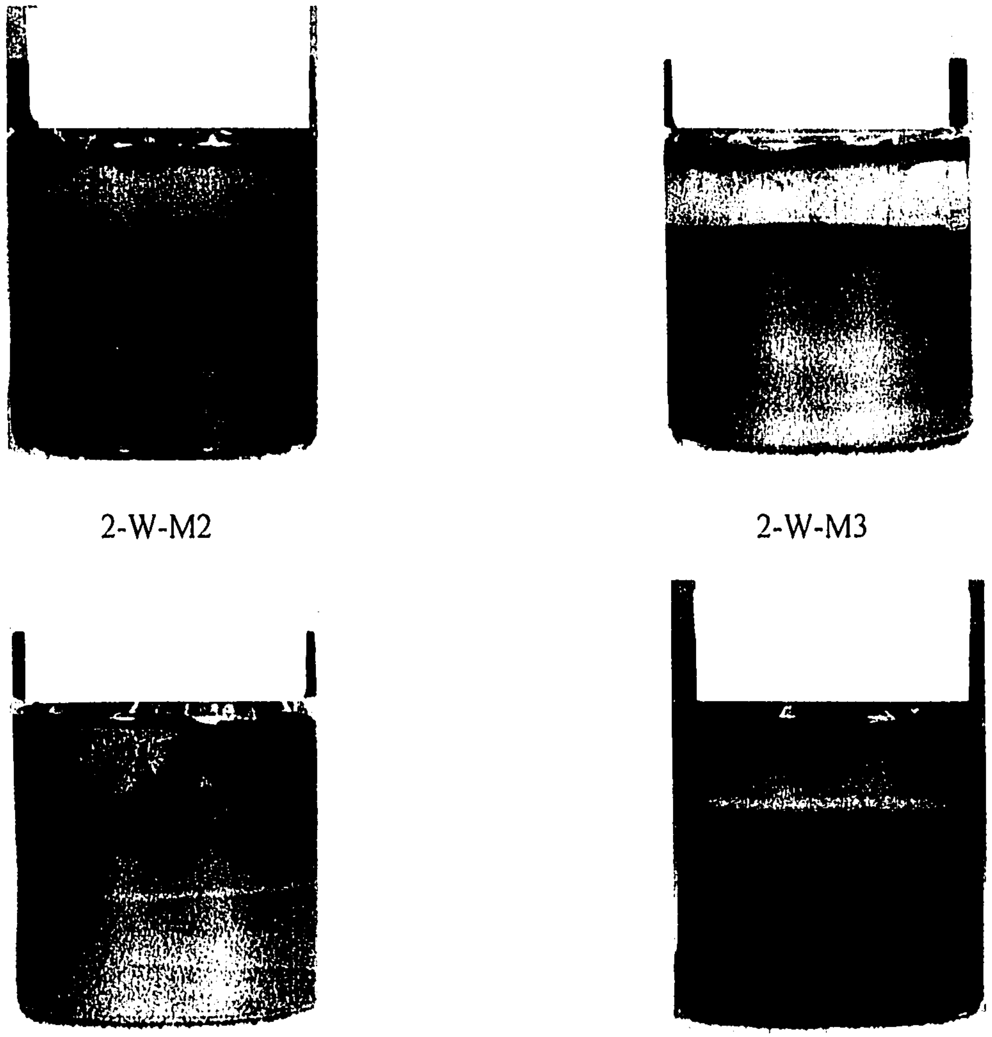
2-W-M3

2-OS-M2

2-OS-M3

Water to oil sands ratio is 0.7

Figure 4.17: Marine oil sands and waste units.



2-W-M2

2-W-M3

2-OS-M2

2-OS-M3

Water to oil sands ratio is 2

Figure 4.18: Marine oil sands and waste units.

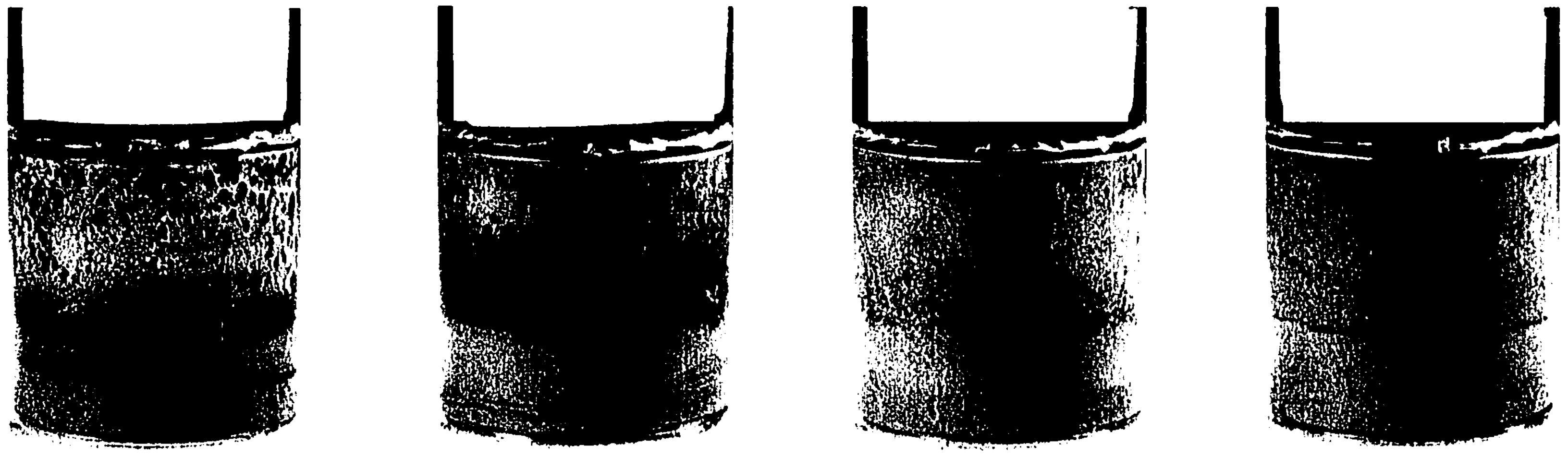
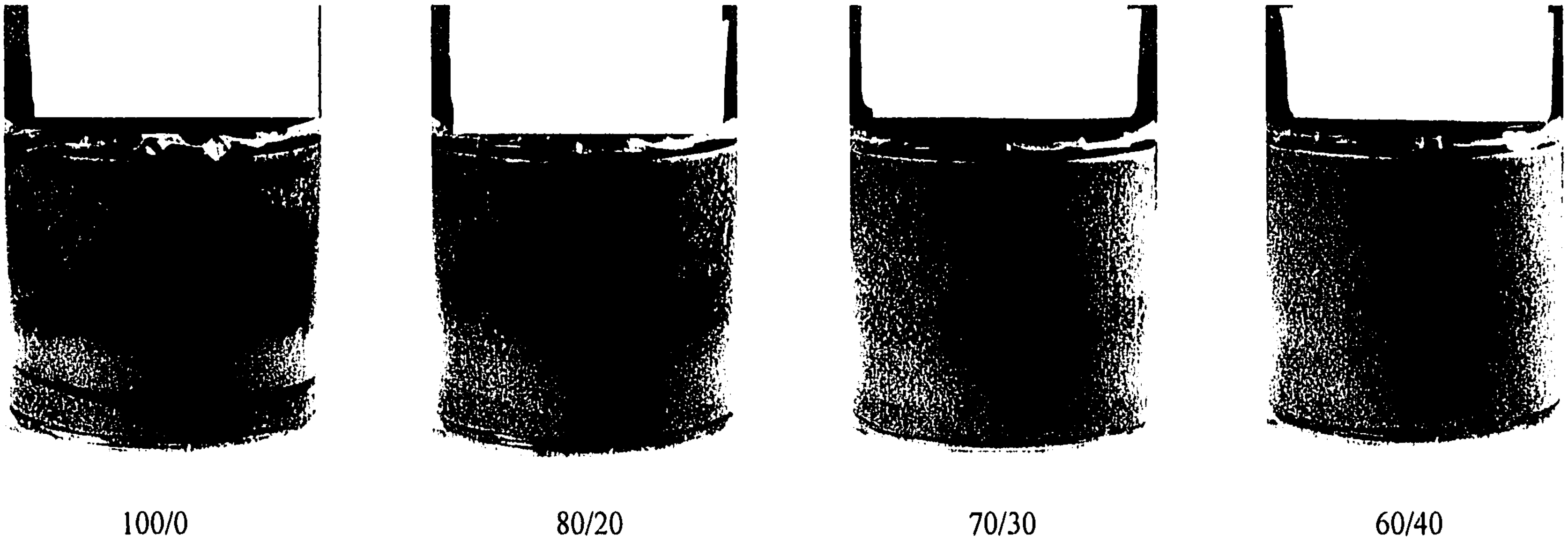


Figure 4.19: Blends of 2-OS-E3 and 2--W-E3.


100/0

80/20

70/30

60/40

Figure 4.20: Blends of 2-OS-E4 and 2-W-E4.

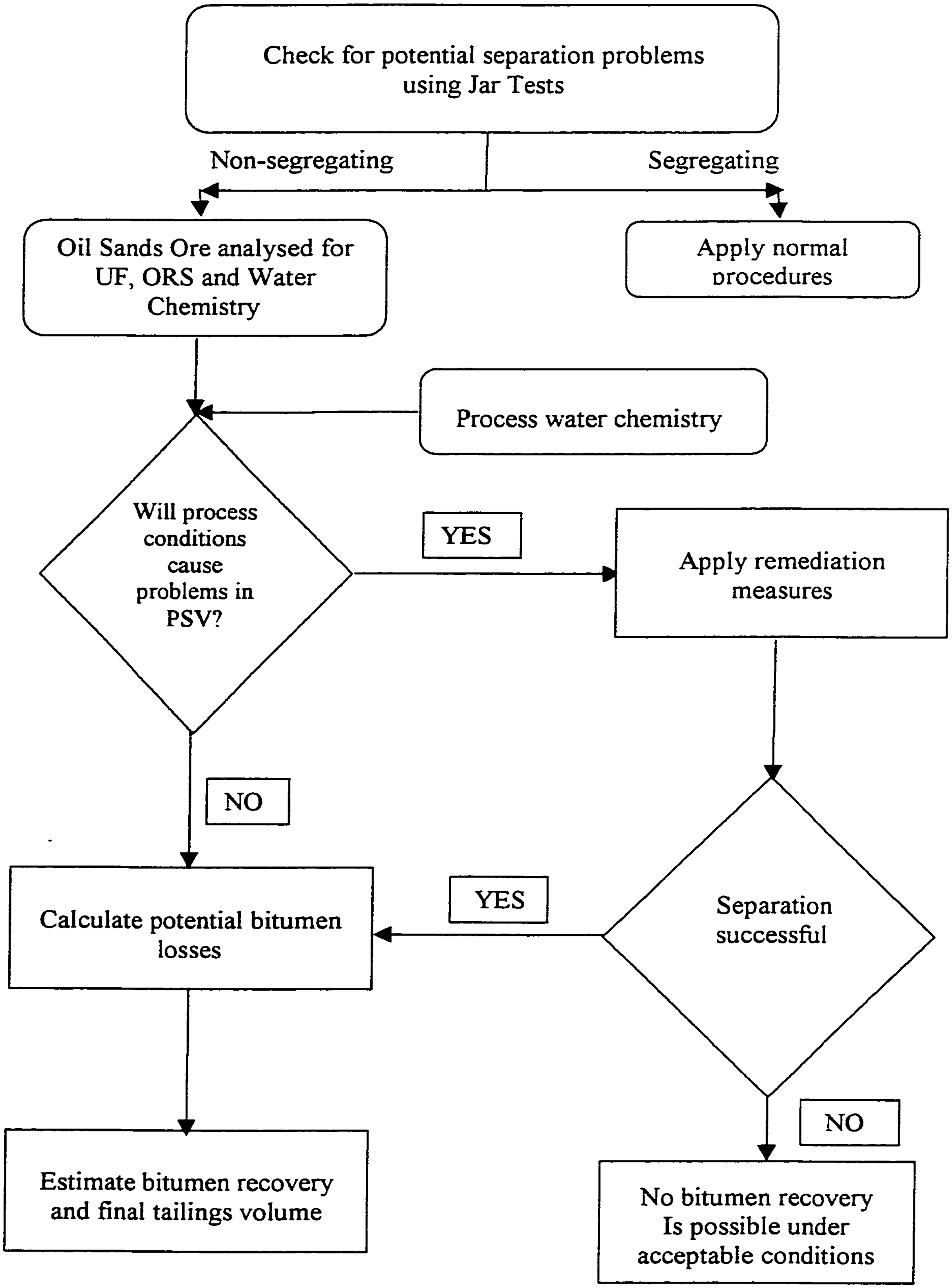


Figure 4.21: Oil Sands processing Decision Tree

Chapter 5

Conclusions

Two new criteria have been introduced to improve the prediction of bitumen recovery in water based bitumen separation processes. The importance of adding ORS and ultra-fines contents of an ore to the list of standard oil sands characteristics is indicated in this work.

ORS have been encountered in all oil sands tested. For most oil sands a roughly linear correlation is observed between fines and ORS contents, however a few anomalous oil sands do not fit this trend. BEU tests were carried out to examine the behaviour of oil sands with different ORS contents. Because of the extensive work involved, complete separation experiments were initially carried out on only five oil sands. There was a common trend for recoveries to decrease with increase in ORS content. Two additional confirmatory BEU tests were then conducted on oil sands from another part of the Athabasca region. The oil sands had similar fines content but extremely dissimilar ORS contents. When BEU primary recoveries were plotted against ORS and fines contents, it was clear that ORS offered a better correlation.

The ability to use ORS for prediction purposes was exemplified further when BIT/ORS ratios were calculated for the middlings and tailings. It was found that the ratio was relatively constant at 2.8±0.7 for all five ores. This surprising observation suggests that bitumen in the tailings is predominantly associated directly with ORS. Knowing this, it is possible to calculate a liberated-unliberated bitumen distribution. A good correlation with BEU primary recoveries with liberated bitumen was observed.

After conducting BEU tests, it was also clear that these tests are not useful in forecasting primary recoveries for ores known to cause sludging in the Primary Separation Vessel (PSV). For example, tests on the same ore demonstrated sludging at the pilot scale but not in the BEU. Because the BEU operates at a higher dilution than a commercial PSV, the role of ultra-fine clays and water chemistry is masked in the former case. Therefore, additional work was required to confirm the role of ultra-fines as an important parameter for oil sands processability. A jar test was developed as a simple evaluation approach to identify problem ores. Once ores are identified more extensive characterization and BEU tests can be performed.

The jar test is also a simple procedure for predicting the zone of operability based on the ultra-fines and cation concentrations in oil sands slurries. A series of tests were performed to examine possible ultra-fines and water chemistry combinations. It was found that ore slurries containing 1.1 w/w%, or less, or ultra-fines are always segregating regardless of Na^+ or Ca^{2+} concentration. Ores containing 2 w/w% of ultra-fines are always non-segregating when Na^+ concentration is above 500 ppm or when Ca^{++} is above 40 ppm.

The jar test can also be used as an aid to determine whether more selective mining will result in better processability. This stems from the fact that waste units with low bitumen content are mined as part of the ore, thereby decreasing the quality of the ore. Jar tests were performed using the waste units, their corresponding oil sands and blends of the two. Results show that the addition of only 20 to 30 w/w% of the corresponding waste material to oil sands is sufficient to cause sludging. A question that remains unanswered is whether the “anomalous” ores are inherently bad or have been unintentionally mixed with waste material.

A decision tree was developed to summarize the logical application of ORS and ultra-fines characterisation tests. This offers a cost-effective and reliable approach to ore evaluation for process predictability.

5.1 Evaluation of Research

This research project was exemplary in establishing the potential of two new criteria for predicting bitumen recovery. However, due to the nature of the samples, some limitations were experienced. Core samples were taken from deposits of different geological origin. One problem with core drilling is that the samples may be different within the same geological region. This would alter the characterisation analysis of this study. Therefore, care must be taken when sampling ores.

Another problem experienced in this study was the characterisation of the waste unit samples. Ultra-fines and ORS were difficult to measure because the samples do not behave as normal ore samples. Along with characterisation problems with waste units, there lies another probing question. Are “anomalous” ores inherently bad or is it the result of mixing with waste materials during sampling?

Also, a limited number of samples were available for this exploratory work. Thus, it was difficult to perform good statistical analysis.

5.2 Recommendations

This research project identified two new criteria for predicting bitumen recovery. The samples used for this work were generated using core drilling methods. It would be beneficial to establish better control methods for sampling, to exclude variation between samples of the same origin.

BEU tests were performed to examine the behaviour of oil sands with different ORS and ultra-fines content, without using any process aids. This was done to establish base line data, which was generated by investigating the natural processability of various oil sands types. In this work seven oil sands were analyzed using a BEU.

The ability to use ORS for bitumen recovery prediction purposes was exemplified when the BIT/ORS ratios were calculated for the middlings and tailings. It was found that the ratio was relatively constant at 2.8±0.7. Future research endeavors should look at solidifying the BIT/ORS ratio by performing BEU tests with ores from different geological regions. This BIT/ORS index could then be used to predict ore processability. Another interesting research project would be to look at the impact that process aids have on BIT/ORS ratios. As well, it would be useful to examine the interaction between BIT/ORS ratios and ultra-fines content of middlings.

Future research studies could focus on understanding the association between bitumen and ORS. This would be beneficial for plant operations, because it may lead to improving recovery by increasing bitumen liberation.

It would also be beneficial to establish a marker for ORS because ORS determination is time consuming. This could be done by establishing a correlation between ORS and carbon content for example. Another potential marker could be heavy minerals because they are concentrated in the ORS component.

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Appendix

Table A.1: Mineralogical composition of solvent extracted solids from oil sands.

ID	Weight percent of solids									
	Albite	K-Feldspar	Calcite	Dolomite	Siderite	Quartz	Kaolin	Illite	Magnetite	Pyrite
1-OS-E1	0.2	1.3	0.1	0.1	N.D.	96.1	0.3	0.5	0.2	0.2
1-OS-E2	0.2	1.6	0.1	0.1	0.2	93.3	1.1	3.5	N.D.	N.D.
1-OS-C1	0.2	1.1	N.D.	0.05	N.D.	93.6	1.7	3.0	0.4	N.D.
2-OS-E3	N/A	1.0	N.D.	N.D.	N/A	94.0	5.0	N.D.	N/A	N/A
1-OS-E4	0.2	1.3	0.05	0.2	1.1	91.9	1.9	3.4	N.D.	N.D.
3-OS-E5	N/A	3.0	N.D.	N.D.	N/A	91.0	6.0	N.D.	N/A	N/A
1-OS-E6	3.3	1.5	0.05	0.1	0.2	90.4	1.0	3.5	N.D.	N.D.
1-OS-E7	0.2	0.1	0.1	0.1	1.7	91.9	2.2	1.8	N.D.	1.9
2-OS-E8	N/A	3.0	N.D.	N.D.	N/A	91.0	6.0	N.D.	N/A	N/A
3-OS-E9	N/A	3.0	N.D.	N.D.	N/A	84.0	1.0	1.0	N/A	N/A
1-OS-C2	0.2	1.5	0.05	0.05	N.D.	90.2	4.6	3.0	0.2	0.2
3-OS-MT1	N/A	2.0	1.0	2.0	N/A	85.0	2.0	3.0	N/A	N/A
1-OS-E10	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
3-OS-M1	N/A	2.0	1.0	1.0	N/A	76.0	10.0	10.0	N/A	N/A
1-OS-E11	0.1	2.4	0.05	0.05	N.D.	87.9	5.1	3.7	0.7	N.D.
1-OS-E12	0.2	2.9	0.04	0.2	N.D.	86.4	6.7	2.9	0.7	N.D.
2-OS-M2	N/A	1.0	N.D.	N.D.	N/A	97.0	2.0	N.D.	N/A	N/A
3-OS-MT2	N/A	3.0	N.D.	N.D.	N/A	80.0	1.0	3.0	N/A	N/A
1-OS-M3	0.1	2.4	0.1	0.4	N.D.	83.9	7.8	4.6	0.7	N.D.
2-OS-M4	N/A	2.0	N.D.	N.D.	N/A	96.0	2.0	N.D.	N/A	N/A
1-OS-M5	0.2	3.5	1.1	0.5	0.3	71.7	14.3	7.8	0.3	0.3
2-W-M2	N/A	1.0	4.0	5.0	N/A	77.0	12.0	N.D.	N/A	N/A
2-W-M3	N/A	N.D.	N.D.	N.D.	N/A	54.0	4.0	42.0	N/A	N/A
2-W-E8	N/A	1.0	N.D.	N.D.	N/A	63.0	19.0	10.0	N/A	N/A
2-W-E3	N/A	2.0	N.D.	N.D.	N/A	63.0	17.0	11.0	N/A	N/A

N.D. – not detected
N/A – not available

Table A.2: Leachable ion contents of solvent extracted oil sands solids.

ID	mg/kg of oil sands (ppm)					
	Na^+	K^+	Ca^{++}	Mg^{++}	Cl^-	SO_4^{--}
1-OS-E1	5	4.1	2.8	1.3	6.1	3.1
1-OS-E2	32.5	5.1	0.5	0.1	26.4	4.1
1-OS-C1	28.1	3.1	0.5	0.2	28.1	5.2
2-OS-E3	184	N/A	0.8	N/A	230	27.0
1-OS-E4	29.9	10.3	5.2	2.8	20.6	4.1
3-OS-E5	170	2.6	2.6	1.7	240.0	94.0
1-OS-E6	6.4	4.2	4.9	1.7	6.4	4.2
1-OS-E7	93.5	10.6	1.0	0.4	37.2	6.4
2-OS-E8	232	N/A	0.2	N/A	293.3	22.3
3-OS-E9	42	7.6	2.4	1.1	6.2	66
1-OS-C2	63.7	9.4	1.2	0.5	12.5	11.5
3-OS-MT1	6	7.7	38.6	4.4	10.2	83
1-OS-E10	33.6	4.3	0.3	0.3	13	4.3
3-OS-M1	270	7.6	31.3	6.9	240.0	510
1-OS-E11	55.5	4.2	0.8	0.3	9.4	8.4
1-OS-E12	8.5	10	30.8	6.5	24.4	5.5
2-OS-M2	413	6.5	9.6	3.8	283.3	380.0
3-OS-MT2	8.0	11.7	29.0	8.0	9.9	110
1-OS-M3	78.3	10.6	0.9	0.5	32.8	7.4
2-OS-M4	407	10.9	10.2	8.5	10.8	667.7
1-OS-M5	162	12.3	1.9	0.9	25.7	17.9
2-W-M2	800	10.9	11.8	6.5	770.0	433.3
2-W-M3	368	109	15.7	83.2	14.7	323.3
2-W-E8	1433	15.7	3.1	3.0	1933.3	31.0
2-W-E3	1403	17.1	2.8	2.4	1933.3	46.0

N/A – not available