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# HYDROPHOBIC SOLIDS AND STRUCTURE FORMATION IN OIL SANDS FINE TAILINGS

A. Majid and B.D. Sparks

*Institute for Environmental Research and Technology, National Research Council of Canada, Ottawa, Ontario K1A 0R9, Canada*

## ABSTRACT

*Hydrophobic solids, having a range of surface wettability, occur in oil sands fine tailings at both the Syncrude and Suncor plants. These relatively coarse solids appear to play a role in the stability of fine tailings by forming a structure, through inter particle flocculation, as a result of interaction with residual bitumen and naphtha. The removal of these solids, together with residual bitumen, results in a change in settling behaviour and subsequent dewatering of the tailings.*

*A fractionation scheme has been developed that allows the hydrophobic solids to be separated selectively from both Syncrude and Suncor fine tailings samples. The solid fractions separated were characterized by particle size distribution and elemental analyses. Significant differences were found in the distribution of certain elements among the fractions as well as between Syncrude and Suncor samples. These differences are believed to be important in terms of the stability of fine tailings.*

*Two types of solids, each having relatively high organic carbon content relative to the bulk solids, have been identified. In one case the solids were predominantly oil wettable, hydrophobic solids (HPS) while the other fraction exhibited partial hydrophobic, or biwetable, surface characteristics (BWS). Biwetable solids behave similarly to sub-bituminous coals, which flocculate in the presence of immiscible liquids of different polarity. On the other hand, HPS can form compact agglomerates after agitation with a critical amount of a water immiscible, non-polar liquid.*

*The implication of these results is that HPS, together with BWS, could be involved in the stability of fine tailings by forming a flocculated structure through interaction of the dispersed solids with bitumen and naphtha. This flocculation can strengthen the tailings structure by reinforcing the gel networks formed by an ultra fine solids fraction also known to be present in the tailings.*

## INTRODUCTION

The Hot Water Extraction Process (HWEP) used by Suncor and Syncrude to extract bitumen from Athabasca oil sands produces tailings with about 35% more volume than that occupied by the bituminous sands before mining. This

increase is largely the result of water hold-up in the fines fraction from the tailings, arising primarily from the "middlings" treatment circuit. Long term economically and environmentally acceptable operation of this process ideally depends on being able to reduce the tailings volume. Therefore, economical reduction of the water content of fine tailings is of critical interest.

Recently, we have investigated the applications of liquid-phase agglomeration techniques for the removal of residual organics and oil wettable solids from the aqueous tailings streams of bitumen separation plants [1-3]. This treatment results in destabilization of the sludge, which then differentiates into four distinct layers as it settles. A clear water layer forms at the top, followed consecutively by: a clay suspension, a thin dark brown interface layer, and finally a well-compacted sediment. The colloidal clay fraction displays a propensity to form gels which are believed to be largely responsible for the water holding capacity of oil sands fine tailings [4]. The solids in the brown interface layer have a high organic carbon content compared to the bulk solids. This organic matter is insoluble in toluene and has been designated as "strongly bound organic matter" (SOM) [5-11]. These organic rich solids may play a significant role in the formation of fine tailings structure because they are wettable by both oil and water; such solids are known to form strongly flocculated structures.

In this investigation an existing fractionation technique [12] was improved to produce materials having relatively uniform surface characteristics. These fractions were characterized by elemental and particle size distribution analyses. The agglomeration characteristics of the two fractions (BWS and HPS) were investigated by measuring the changes in their sediment volume after agglomerating with a bitumen solution in naphtha [13]. The latter solution is known to be present in oil sands tailings ponds.

## AGGLOMERATION THEORY

Liquid Phase Agglomeration (LPA) is a phenomenon based on selective wetting of the surfaces of finely divided solids in the presence of two immiscible liquids [14]. One liquid is present in sufficient quantity to form a continuous medium for the suspension of particulate solids. The second, or bridging liquid, is present in relatively small amounts and

must preferentially wet the surfaces of the suspended particles. In order to initiate the agglomeration process, there must be sufficient agitation to disperse the wetting liquid throughout the suspending medium and to promote inter particle contact. Surface wettability, the degree of agitation, and amount of bridging liquid, relative to the solids being collected, are the major controlling parameters. Once dispersed, bridging liquid droplets are selectively adsorbed by the solids having the most compatible surface. Inter particle collisions between these wetted solids results in their agglomeration by interaction between the bridging liquid layers on the particles involved.

The bond strength between particles arises from the capillary forces within the bridging liquid at particle junction points. Greater surface curvature of the adsorbed bridging liquid is associated with the smaller inter-particle pores exhibited by finer particles. Consequently, smaller particle sizes produce stronger agglomerates. Another important criterion is the degree of wettability of the solids by the bridging liquid; complete agglomeration is only possible when the solid surfaces are completely wetted. Figure 1 summarizes the agglomerate characteristics associated with different bridging liquid levels.

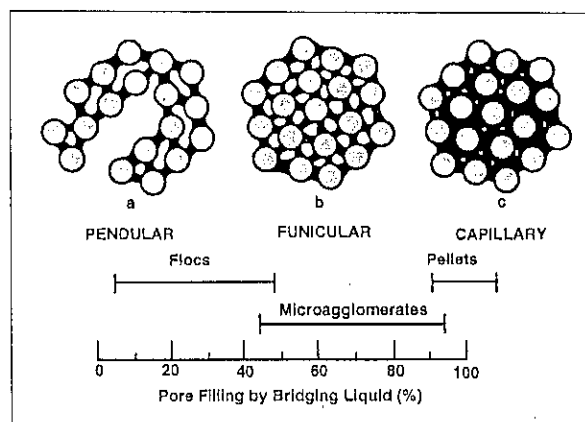


Figure 1. Agglomerate forms.

Amounts of bridging liquid which lead to incomplete surface coverage of the solids limits the number of bonding points between individual particles. This results in the formation of voluminous flocs that entrap large amounts of the suspending medium; the bridging liquid is said to be in the pendular state. In the special case where the bridging liquid does not completely wet the suspended particles, that is, where the contact angle is less than  $90^\circ$ , agglomeration cannot proceed beyond this flocculation stage.

As the amount of bridging liquid increases the adsorbed liquid eventually forms a continuous layer around each completely wettable particle. The system is now in the funicular state. Under these circumstances there is no limit to the number of particle bonding interactions and the flocculated structure condenses to form more compact agglomerates. At this stage the combined effect of capillary forces and agitation produces roughly spherical bodies. However, the bridging liquid volume may still be insufficient to completely fill the internal porosity of the agglomerates. Retention of suspending liquid in the intra-particle pores may remain relatively high.

When the agglomerate pores become saturated with bridging liquid the system is said to be in the capillary state. Under these conditions the agglomerates formed achieve a maximum in density and strength. Final agglomerate size results from an equilibrium between the amount of bridging liquid and the degree and type of agitation. The presence of large amounts of solids, not wetted by the bridging liquid, adversely affects the agglomeration process by interfering with contact between wetted particles. The overall result is a decrease in both agglomeration rate and selectivity of the process.

In the case of oil sands tailings the solids are suspended in water with an immiscible liquid in the form of the bitumen and naphtha lost to the tailings during processing. Mixing can occur during pumping and transport, to the tailings ponds. Consequently, all the requirements are present for agglomeration to occur. Agglomeration of suspended solids, to produce large three dimensional flocs, causes a significant increase in the volume occupied by the particles [13]. In oil sands tailings such structures may interact with other flocculated particles [15] to increase the tailings stability. Change in sediment volume provides a simple index for observing the degree of agglomeration occurring in a suspension of selected components.

## EXPERIMENTAL METHODS

### Sample Description

Aqueous tailings from the 17 m level of the Syncrude tailings pond were pumped into 200 L plastic drums [16]. A barrel tipper was used to invert each drum five times before dividing their contents among a number of 5-L plastic jugs. The sub-samples were stored in a cooler at  $10^\circ\text{C}$ . The results presented in this report were obtained on these sub-samples, provided courtesy of R. Schutte of Syncrude Canada Ltd. A sample of aqueous tailings from Suncor pond 2, provided courtesy of the Fine Tailings Fundamentals Consortium was obtained in a 200 L plastic drum. Sub-samples in 5-L plastic

jugs were taken from this drum after mixing it thoroughly with a long iron rod. Before further sub-sampling, each jug was shaken vigorously by hand to ensure complete mixing. The physico-chemical properties of both samples of fine tailings are presented in Table 1.

Table 1. Physico-chemical properties of fine tailings samples

Property	Value	
	Syncrude	Suncor
pH at $25 \pm 0.5^\circ\text{C}$	7.6	8.1
Conductivity (ms/cm)	1.8	1.7
Total solids (wt%)	35.1	29.1
Bitumen [OWS] (wt%)	$1.2 \pm 0.1$	$5.7 \pm 0.3$
Density (g/mL)	1.22	1.20
Solids, $< 44 \mu$ (%)	$99 \pm 1$	$84 \pm 2$

## Separation of Bi-Wetted Solids

Figure 2 shows the sequence of steps used to fractionate the whole fine tailings. In general, a tailings sample (500 g) is gently agitated with stainless steel balls in a polyethylene bottle, rotating about its symmetrical axis. Residual bitumen and hydrophobic solids are collected as a surface coating on the balls. The treated tailings are agitated in a Waring Blender, at 15,000 rpm for 30 minutes. After gravity settling, for another 30 minutes the tailings separate into two distinct layers. A mixed sediment layer of black BWS and coarse inert solids forms at the bottom of the container while finer, clay solids remain in suspension. The clay fraction is decanted off and the sediment mixed with pond water to resuspend any entrapped clay. After gravity settling for another 30 minutes the suspension layer is again decanted off. This procedure is repeated several times until the amount of clay solids remaining in suspension is negligible. Differential settling, in pond water, is then used to separate the more rapidly settling, coarse, inert solids from the less dense BWS. The separated BWS fraction is washed several times to ensure complete removal of the inert solids and then centrifuged at  $500 \times G$ , for 30 minutes, to obtain a wet cake concentrate. A small portion of this cake material was dried at  $110^\circ\text{C}$ , for analysis, while the remainder was used in flocculation tests.

## Separation of Hydrophobic Solids

The bitumen phase separated by collection on stainless steel balls is washed several times, first with pond water and then with distilled water to remove any occluded hydrophilic

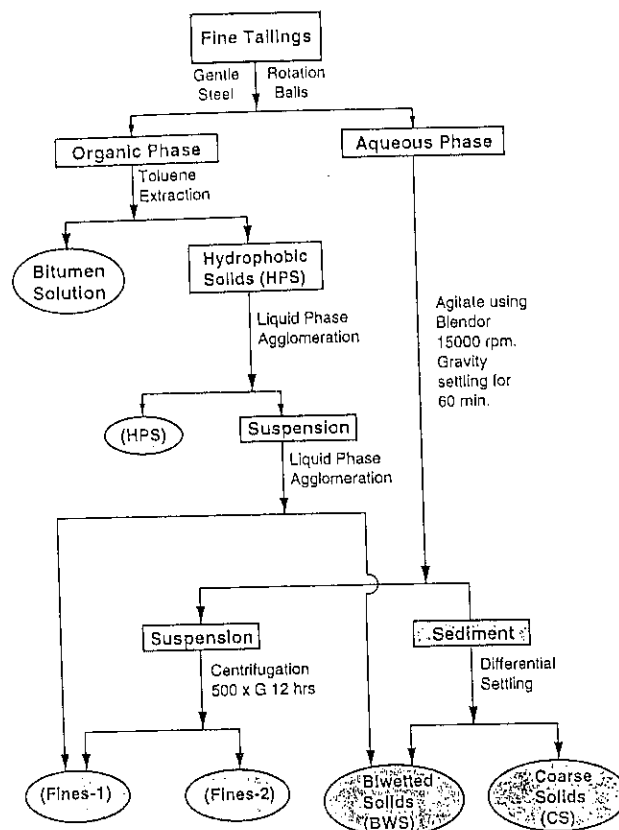


Figure 2. Flow sheet for the fractionation of fine tailings.

solids. The steel balls are separated from the organic matter by washing them with toluene and passing the solution through a 100 mesh screen. The toluene slurry is then centrifuged at  $500 \times G$  for 30 minutes to separate the hydrophobic solids. After decanting the solution layer, the sedimented solids are washed several times with fresh toluene to remove any residual bitumen. After drying at  $110^\circ\text{C}$  the dried, hydrophobic solids are redispersed in distilled water and "purified" by selective agglomeration with Varsol. Spherical agglomerates of HPS are formed that can be separated from the water phase by screening; these solids, after drying at  $110^\circ\text{C}$  were designated as HPS. The under flow from the screen was shaken with additional amounts of Varsol. Inert solids remained in suspension while a biwetted fraction flocculated and settled out as a dark brown layer that could be separated by decantation; no dense agglomerates formed in this case. After drying at  $110^\circ\text{C}$  the settled fractions were designated, BWS.

### Agglomeration Testing

A 2 g sample of centrifuged wet cake, containing about 50 wt% BWS, was transferred to a 50 mL glass jar, using a small amount of pond water. In a series of tests different amounts of 60 wt% bitumen solution in naphtha were added to several slurry samples. Pond water was used to bring the total volume to 15 mL. The jars were sealed with polyethylene gaskets and agitated for five minutes on a Spex mixer. The contents of each jar were transferred to preweighed graduated glass cylinders; pond water washings were used to top up the volume to 25 mL. After the suspensions had been allowed to settle under gravity for 24 hours the sedimentation volumes of each sample were visually estimated by comparison of the solid-liquid interface with the cylinder markings. Little change in volume was observed after only one hour of settling.

In some tests, small flocs floated at the top of the suspension and did not settle. The volume of this fraction was measured separately by transferring the flocs to 10 mL cylinders using a small amount of pond water. The finer flocs were made to settle by gentle tapping or shaking the cylinder and their final volume determined; the main sediment volumes in the 25 mL cylinders were then corrected to include this volume.

The supernatant suspensions from the cylinders were decanted after noting the final sediment volume. The wet sediments were transferred to 100 mL glass jars, and the bitumen extracted with a mixture of 74% toluene and 26% isopropanol. Extracted solids were dried at 110°C to determine the actual weight of BWS. Bitumen was determined gravimetrically after evaporating the solvent in a rotary evaporator. In some experiments the cylinders, containing wet sediments, were dried at 110°C in order to determine the weight of BWS plus bitumen. Corrections were then applied for the weight of bitumen originally added.

### Analysis of Dry Solids

Total carbon was determined using a Leco CR12 carbon analyzer. Organic carbon was redetermined after decomposing any carbonate carbon with dilute hydrochloric acid; carbonate carbon was then determined by difference. Particle size analyses were carried out using a Malvern Particle Sizer M 3.1. Inductively Coupled Plasma Spectrometry (ICP) was used for elemental analyses.

## RESULTS

Table 2 summarizes composition data for the BWS and HPS fractions separated from Syncrude and Suncor fine tailings. Values for the whole, bitumen-free tailings and the coarse, inert sediment are included for comparison. The results indicate that fractions separated under the same conditions exhibit a similar chemical composition. Also, the yields of each fraction are remarkably close for the two tailings samples, indicating that they are a typical component of oil sands ores.

### Characterization of Hydrophobic Fractions

Specifically, the hydrophobic fractions are considerably richer in organic carbon than the tailings as a whole. Another major difference between the hydrophobic fractions and the bulk tailings lies in the high iron content of the former. Values ranging from about 4 to 16 weight percentage were found compared to only 1.2 weight percentage for the tailings solids as a whole. The HPS fractions were associated with the highest iron content. It has been reported that iron plays an important role in the stability of oil sands tailings [17]. Allan and Sanford [18] have hypothesized that ferric iron is

Table 2. Analytical data for various solids obtained from the fractionation of fine tailings (Figure 2)<sup>d</sup>

Analyses (w/w%)	Syncrude tailings						Suncor tailings					
	Blank <sup>c</sup>	Fines-1	Fines-2	CS	BWS	HPS	Blank <sup>c</sup>	Fines-1	Fines-2	CS	BWS	HPS <sup>f</sup>
OC <sup>b</sup>	2.6	6.6	3.2	0.3	6.0	27.6	6.0	3.1	6.4	2.1	12.0	21.1
CO <sub>3</sub> -C <sup>c</sup>	0.3	0.4	0.7	0	2.1	2.3	1.1	4.0	0.9	0.7	0.5	3.7
Total S	0.2	0.5	0.2	0.1	0.6	2.8	0.29	0.6	0.5	0.2	0.8	2.3
Fe	1.2	2.9	NA	1.1	8.7	15.5	2.1	2.6	2.6	2.1	4.0	5.6
Al	11.0	13.6	NA	9.4	11.5	8.0	12.0	13.4	10.8	2.5	7.0	2.9
Ti	0.6	0.2	NA	0.4	2.1	4.6	1.9	0.3	0.4	0.5	0.9	14.1
Av. particle size $\mu$ m)	NA	NA	7.9	11.6	8.1	13	NA	NA	8.2	25.1	24.2	NA
Yield <sup>d</sup>	100	1.8	9.8	23.2	1.7	1.8	100	4.0	3.8	21.0	2.4	2.1

<sup>a</sup> all abbreviations listed on Figure 2; <sup>b</sup> organic carbon; <sup>c</sup> carbonate carbon; <sup>d</sup> w/w% of fine tailings; <sup>e</sup> toluene-extracted solids from whole tailings; <sup>f</sup> results from a previous study; NA, not available

instrumental in binding organic matter (SOM) to the predominant clays in the tailings, thereby affecting their surface properties. Low temperature oxidation of the organic matter indicates that the hydrophobic particles are composites of rutile, zircon, anatase, quartz, mica, and pyrite bound together by organic material having characteristics similar to humic matter [5-11,19]. Figure 3 shows the correlation between organic carbon and iron content for the different fractions. Compared to the Syncrude samples the Suncor fractions gave an excellent first order correlation. This observation is consistent with the fact that the ore deposit at the Suncor mine is more homogeneous than that found on the Syncrude site. The organic carbon and iron content of the BWS fractions is higher than the tailings as a whole, but lower than the HPS fractions.

As a corollary to the high iron and organic content, the hydrophobic components have a lower aluminum content relative to the clay fractions.

## Structure Forming Propensity

An oil phase agglomeration technique was used to study the flocculation characteristics of both BWS and HPS. In this approach the type of aggregate or agglomerate formed by the solids is critically dependent on the amount of bridging liquid used, the degree and type of agitation, and the wetting properties of the material [13-15]. The progress of agglomeration was monitored by following the gravity settling of samples produced after suspensions of the HPS and BWS fractions, in pond water, were agitated with different amounts of bitumen solution.

As expected from their surface characteristics the HPS samples showed agglomeration behaviour typical of that for solids completely wetted by the bridging liquid. Figure 4 shows the sedimentation volume of the hydrophobic solids as a function of added bridging liquid; the inset diagram was obtained for the classic barium sulphate/water/benzene system [13]. The comparable behaviour in both cases is characteristic of a progression from flocs to dense agglomerates usually observed after incremental additions of bridging liquid to a suspension in which the solids are completely wetted by the bridging liquid. The amount of bridging liquid required to form agglomerates, rather than flocs, is about 1.5 w/w%, based on the total tailings. This compares to bitumen concentrations of 1.2 w/w% and 5.7 w/w percentage actually found in Syncrude and Suncor fine tailings, respectively. Poor dispersion of this bitumen and interference from non-active solids may prevent dense agglomerates forming. However, flocculation of these particles remains a distinct possibility.

For BWS, the sediment volume increased consistently with bridging liquid addition over the entire range used in this study (Figure 5). In this case no dense agglomerates were formed under any combination of mixing or bridging liquid addition. However the significant increase in sediment volume observed is a good indication that flocculation of the solids has occurred.

## DISCUSSION

The data presented in this report suggest that liquid phase agglomeration can occur in oil sand fine tailings to produce a flocculated structure. The interparticle bonding involved is

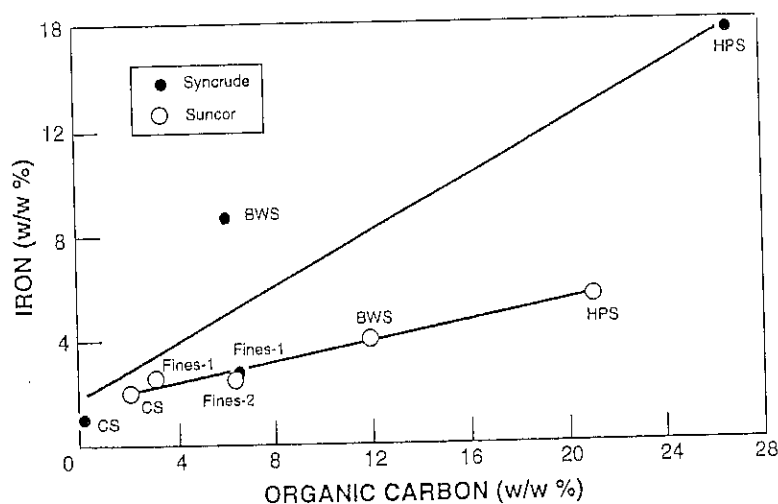


Figure 3. Correlation between organic carbon and iron.

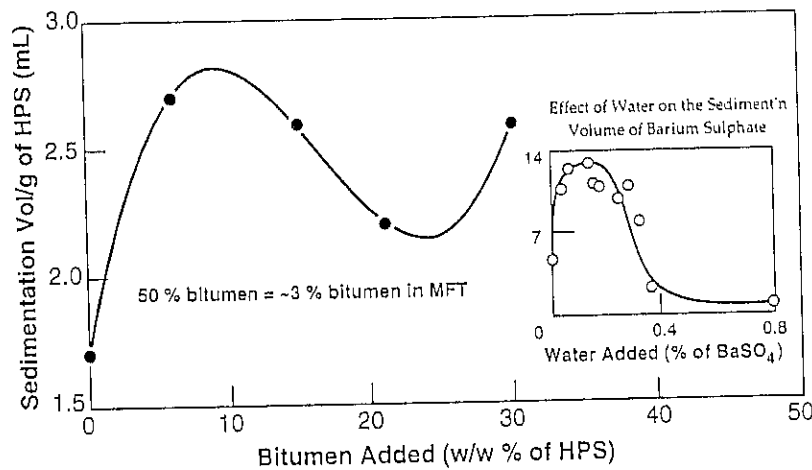


Figure 4. The effect of bitumen on the sediment volume of HPS.

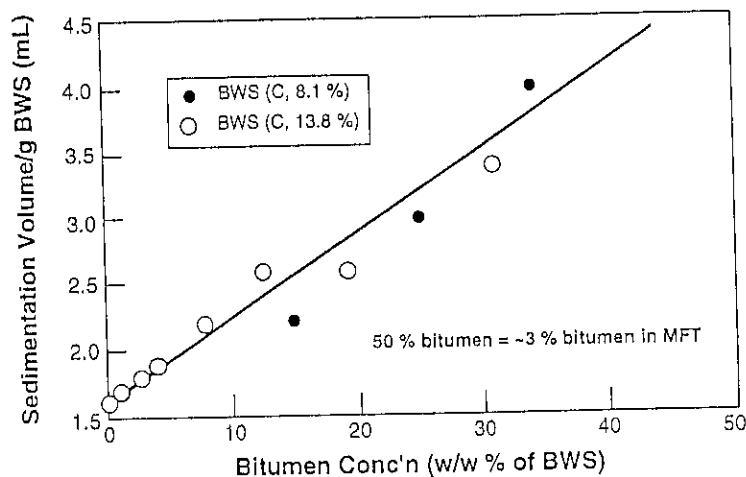


Figure 5. The effect of bitumen on the sediment volume of BWS.

much stronger than double-layer electrostatic interaction or van der Waals forces and is capable of surviving severe mechanical stress. Removal of both bitumen and hydrophobic solids fractions from oil sands fine tailings causes a significant change in the tailings settling characteristics. The treated tailings separate rapidly to form a compact sediment of coarser solids and a suspension of finer clay particles. In comparison the original tailings remain in an undifferentiated state unless subjected to vigorous agitation and then centrifugation.

In other studies we have observed that an ultra fine fraction in oil sands tailings forms strong thixotropic gels [4].

This phenomenon is believed to be the primary cause for the tailings' observed nonsegregating behaviour. However, independent studies on the ultra fines fraction have shown that the gelation process can have a significant time dependency. The fact that no segregation of tailings occurs during the gel forming stage is an indication that another factor is involved to prevent settling of the coarse solids while the ultra fine gel is reforming. The flocculation of hydrophobic solids, being largely time independent and having binding forces capable of withstanding severe mechanical stress, could provide these initial conditions to prevent the sedimentation of coarser solids in whole fine tailings.

Careful removal of the hydrophobic solids from tailings, without any chemical modification, causes the tailings to separate rapidly into fine and coarse fractions. Over time the fines in the upper layer ultimately reform a gel, too late however, to prevent particle segregation. The addition to tailings of a good dispersant, such as sodium silicate, will also cause tailings to segregate after mixing. Sodium silicate is well known to be a good surface wetting agent [20]. Its presence could prevent agglomeration of the hydrophobic solids by changing their surface characteristics to a completely water wettable condition. Flocculation of the solids by the residual oils in the pond would therefore be prevented.

## CONCLUSIONS

A method has been developed that allows the separation of coarse, hydrophobic solids from oil sands tailings. A study of these solids shows that their surface properties cover a wide range of hydrophobicity. One fraction is completely hydrophobic, and is usually directly associated with bitumen. The other component is a biwetted material that preferentially collects at water-oil interfaces. In the latter case the coarser solids can interact to form flocs, while very fine particles may stabilize oil-in-water emulsions. The fractions have compositions characterized by high organic carbon and iron contents relative to the bulk solids found in fine tailings.

Agglomeration tests with the separated fractions have shown that these solids are capable of forming flocculated structures that are strong enough to survive vigorous agitation. On standing these flocs prevent settling of coarser inert solids until reinforcement by the time dependent gelation of an ultra fine fraction also present in the whole tailings. If the hydrophobic solids are removed, by a mild physical treatment, then vigorous agitation results in a system that exhibits rapid segregation of coarse and fine solids. The same effect can be achieved by using a wetting agent, such as sodium silicate, which will alter the hydrophobic nature of the particle surfaces. This change in surface condition will inhibit flocculation by preventing liquid phase bonding between the affected particles.

## ACKNOWLEDGEMENTS

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