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Article:

Impact of Fugitive Bitumen on Polymer-based Flocculation of Mature Fine Tailings

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Abstract

In bitumen recovery from oil sands, a percentage of the bitumen is lost to tailings. The effect of fugitive bitumen on fines settling and consolidation in tailings ponds remains controversial. In the current study, the settling performance of mature fine tailings (MFT) in response to flocculant addition was considered by studying MFT of varying bitumen content. Bitumen content in the MFT was adjusted by controlled removal of bitumen using a Denver flotation cell. The initial settling rate of flocculated MFT was observed to increase with decreasing bitumen content from 0.45 wt.% to 0.18 wt.%. A further reduction in bitumen content was found to dramatically decrease the settling rate of flocculated MFT. Such behavior seems counterintuitive since the polymer flocculant was found to have a greater affinity for 'clean' surfaces (Al_2O_3) than for bitumen contaminated surfaces, as measured by quartz crystal microbalance with dissipation (QCM-D), which would predict a further increase in settling rate of flocculated MFT with decreasing bitumen content. The reduction in settling rate below a critical bitumen content is thought to result from selective removal of hydrophobic solids, since washing of untreated MFT with toluene is shown to significantly improve settling of flocculated solids. The current

study confirms the use of flotation as a viable option to control MFT bitumen content and improve the settling rate of flocculated MFT.

Keywords: Oil sands, mature fine tailings, bitumen, and flocculation.

Introduction

The oil sands deposits in Northern Alberta, Canada constitute one of the world's largest oil reserves (Masliyah et al., 2004). For several decades bitumen has been recovered from the oil sands via a hot/warm water extraction process. During extraction the oil sands ore is mixed with hot water and agitated to promote bitumen liberation/detachment from the sand grains. Often, to maximize bitumen recovery, caustic (typically sodium hydroxide) is added to the slurry water as a process aid to raise pH. Increasing pH modifies the slurry chemistry by: i) releasing natural surfactants from bitumen (Schramm and Smith, 1985), ii) modifying electrical surface potentials of bitumen and solids (Masliyah et al., 2004), iii) reducing the divalent ion concentration, and iv) decreasing solids hydrophobicity (Hall and Tollefson, 1982). Beyond critical caustic loading, processing conditions become unfavorable, reducing overall bitumen recovery. Typically, optimum recovery is achieved in the pH range 8.5 − 9.0.

Flotation of bitumen forms a bitumen froth containing approximately 60% bitumen, 30% water and 10% solids by weight. Typically, the froth accounts for around 90% or greater bitumen recovered from the oil sands ore. Unrecovered bitumen is lost with the coarse solids removed through the bottom of the primary separation vessel and the fine middlings which are subsequently reprocessed by flotation to recover residual bitumen. Eventually the two streams are combined and discharged to tailings ponds. Coarse sands deposit rapidly, while the fines and clays settle slowly. Over an extended period of time, fine tails form a viscous, gel-like

suspension that resists consolidation. Such material is referred to as mature fine tailings (MFT) which contains approximately 30% solids by weight and remains stable for several decades (Camp, 1977).

Methods for treatment of MFT to improve settling and consolidation have received significant attention in recent years. Several patents and publications describing techniques to enhance tailings consolidation are available in the open literature. The proposed approaches can be summarized as: i) composite/consolidate tailings (CT) process, ii) CO_2 enhanced CT process, iii) flocculation-assisted centrifugation, iv) freeze thaw dewatering, and v) flocculation-assisted thin lift/drying (Sobkowicz, 2011, Powter et al. 2011). The use of polymer flocculants has been considered in detail. While improving the settling of fines/clays at an optimum dosage, the addition of polymer does not significantly improve consolidation without additional mechanical forces (Alamgir et al., 2012). Novel approaches using stimuli-sensitive polymers (Long, et al., 2011), and hybrid-polymers (Wang et al. 2010) have been considered, but remain somewhat inadequate to address the ongoing requirements for tailings management. Alamgir et al. (2012) considered a two-step flocculation-assisted filtration process to produce trafficable filter cake/deposits from MFT. Through appropriate dilution of MFT and the addition of Al-PAM (cationic organic-inorganic hybrid polymer), MFT was transformed into a stackable cake by flocculation-assisted thickening and the subsequent filtration of the sediment. While such studies represent a significant advancement in tailings treatment, little consideration has been given to the role of fugitive bitumen.

Several studies have shown that the ultrafine solids in MFT have a high propensity towards gel formation. These solids in the presence of multi-valent cations promote the formation of large, porous aggregates that rapidly settle to form sediment beds. However, the large aggregates retain

substantial amounts of water and resist consolidation (Kotlyar et al., 1995). Structuring of such aggregates and gelation are more pronounced for solids contaminated by organic matter such as humic matter (Majid et al, 1990). It is believed that hydrophobic interactions between bitumen-contaminated particles help to bridge neighboring particles, contributing to network strength and gelation (Angle et al. 1993, Kessick 1979). The associated and free bitumen in MFT has been shown to decrease hydraulic conductivity (Scott et al., 1985, Suthaker et al., 1996), blocking the fluid pores, impeding water release and hindering consolidation. While research shows improved settling by removal of bitumen (Majid et al., 1991), there remains conflicting discussion with the presence of fugitive bitumen being identified as a binding agent to flocculate fines and increase the settling rate (Scott et al., 1985).

The role of fugitive bitumen on flocculation performance of MFT has not been considered. Indirectly, Long et al. (2006) using single-molecule force microscopy studied the adsorption of a partially hydrolyzed, high molecular weight, anionic polyacrylamide (HPAM) on sand, clay and bitumen surfaces. The probability of picking up HPAM strands by AFM tip was significantly lower on the bitumen surface, indicating fewer HPAM molecules being adsorbed on bitumen, as compared with both clay and sand. The measured desorption force was considerably lower on a bitumen surface than on a clay surface. Such selectivity of HPAM for different surfaces would suggest polymer performance to deteriorate with increasing bitumen loading in MFT. The objective of the current study is to understand the role of fugitive bitumen on the flocculation and settling performance of MFT. Bitumen content in MFT is varied by controlled removal of bitumen using flotation or solvent washing.

Materials and Methods

Samples of raw MFT and process water were obtained from Syncrude Canada Ltd. The composition of the raw MFT was determined by Dean-Stark analysis to contain 1.4 wt.% bitumen, 31.7 wt.% solids and 66.2 wt.% water. MFT samples were blended vigorously and diluted to 10 wt.% with process water prior to their use. The polymer flocculant used throughout the study was AF246 (Hychem Inc., USA), an anionic, high molecular weight hydrolyzed polyacrylamide polymer. In advance of flocculation experiments 1000 ppm (1 g/L) polymer stock solution was prepared in deionized water and agitated for 12 h. All stock solutions were used within 24 h.

Control of bitumen content:

Protocol 1 by Denver cell flotation of MFT: 800 g of diluted MFT was preconditioned at 1200 rpm for 5 min. Flotation air was then introduced at a rate of 150 mL/min. The flotation time was varied to yield samples with varying bitumen content. The composition of the resultant samples was determined by Dean-Stark analysis. It should be noted that after flotation the solid content of the diluted MFT could be considered unchanged due to a limited amount of fine solids along with the slurry water being carried into the froth. Figure 1 shows the final bitumen content of the diluted MFT as a function of flotation time.
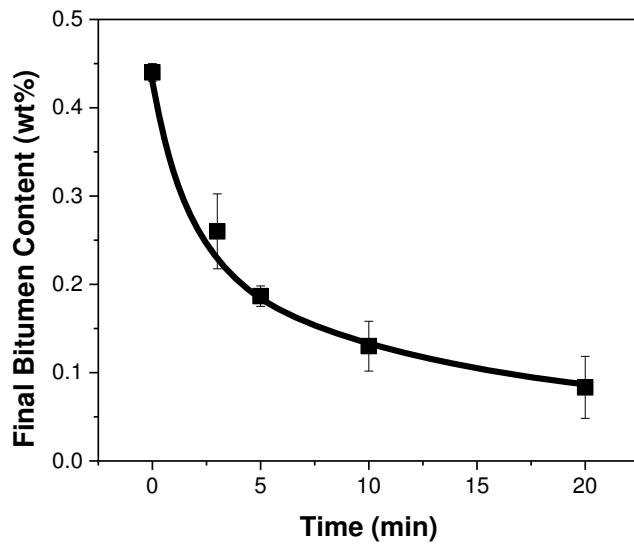
Figure 1: Bitumen content in MFT as a function of flotation time.

Protocol 2 by MFT blending: Low bitumen content samples obtained by protocol 1 were blended with diluted raw MFT (bitumen content ~ 0.45 wt.%). The reason for blending is to achieve a desired bitumen content to differentiate the effect of selective removal of solids by flotation on MFT flocculation and settling. To ensure effective blending the combined MFT sample was mixed at 500 rpm for 1 h using an overhead mechanical stirrer with a 4-blade, 2-inch width impeller. The solids content in the mixed sample remained at 10 wt.%.

Protocol 3 by solvent washing of MFT: Diluted MFT was washed in excess toluene to remove all toluene-soluble organics. A schematic of the cleaning procedure is shown in Figure 2. MFT was mixed with equal volumes of toluene [A] and agitated overnight [B]. The mixture was centrifuged at 3000 rpm for 5 min [C]. The supernatant containing dissolved bitumen was decanted and replaced with fresh toluene. The process was repeated until the supernatant appeared clear [D]. A portion of the solids was observed to partition between the organic and

aqueous phases. These solids are believed to be bi-wettable as described by Kotlyar et al. (1995). To ensure complete removal of toluene these solids were separated, dried and re-dispersed in a small amount of process water to maintain the solids content of diluted MFT. The suspension was then thoroughly blended following the procedure described in protocol 2 [E].
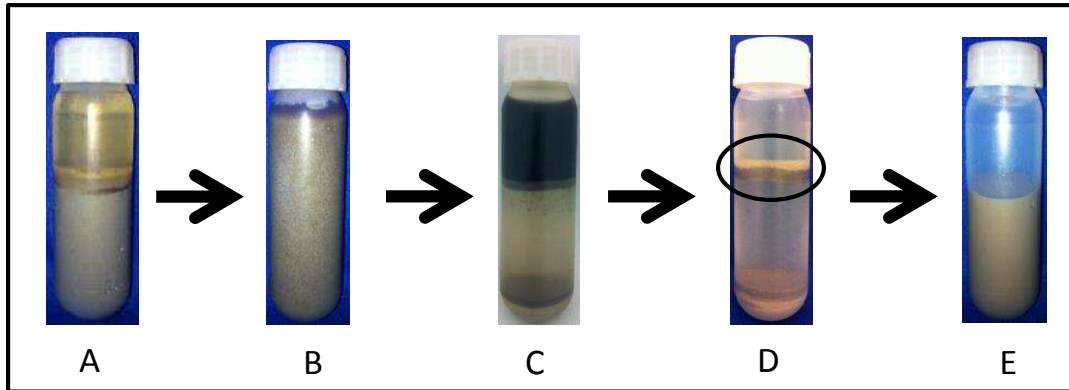

Figure 2: Solvent washing procedure to remove all soluble organic species.

Batch settling: Samples obtained using the above protocols were mixed thoroughly for 1 h prior to use. In a 250-mL beaker fitted with four baffles, 100 g of tailings were placed and mixed at 500 rpm for 5 min with an overhead mechanical stirrer of a 4-blade, 1-inch width impeller, to ensure complete sample homogeneity. The impeller speed was reduced to 350 rpm while 5 g of polymer stock solution was added over a period of 1 min (overall dosage: 50 ppm based on the mass of slurry). Upon completion of polymer addition agitation was stopped and the suspension was transferred to a 100-mL graduated cylinder. The cylinder was sealed and inverted 5 times to evenly distribute the suspension within the cylinder. The "mud-line" height was recorded as a function of time, with the initial linear settling regime of the settling curve being used to calculate the settling rate.

Floc size: A model S400A Focused Beam Reflectance Measurement (FBRM$^®$) probe (Mettler Toledo, USA) was used to measure floc growth after polymer addition. This technique uses a focused laser light (laser wavelength 780 nm) to generate a focal spot that is rotated around a circular path. Intersection of the laser beam at the focal spot by particles generates distinct pulses of the reflected light. The pulse width multiplied by the scan speed (laser rotation speed) is translated into a particle chord length. The probe counts thousands of particles per second and creates a chord length histogram/distribution (Kail et al., 2009). A chord length distribution is similar, though not identical to a particle size distribution. A chord length can be the distance between any two points on the edge of a particle and would therefore always be less than or equal to the particle's diameter. However, it has been reported that the mean square weighted chord length is similar to the mean particle diameter obtained using other methods, such as laser diffraction (Heath et al., 2002). For the flocculation study, samples were prepared following the protocol described previously in batch settling (mixing maintained at 350 rpm) with the FBRM$^®$ probe being positioned at 80° to the horizontal in the 250-mL beaker at the start of each run. The FBRM ® laser scan speed was fixed at 2 m/s and the ''coarse'' detection mode was used for all experiments.

Total organic carbon (TOC) analysis: To study polymer flocculation performance, the TOC concentration in the supernatant after batch settling was measured using a Shimadzu TOC-V CHS/CSN Model Total Organic Carbon Analyzer (Shimadzu Corporation, Japan). This method measures TOC by acidifying a sample of water and then sparging to remove purgeable organic and inorganic carbon. The sample undergoes a reaction with a catalyst to produce CO_2 which is then detected by a non-dispersive infrared (NDIR) detector. First, approximately 30 mL of sample obtained using protocol 1 was centrifuged at 14000 rpm for 20 min. The supernatant was

then decanted and the TOC of the supernatant measured to establish a baseline for each sample. After flocculant addition and batch settling, approximately 30 mL of the supernatant was extracted and centrifuged at 14000 rpm for 20 min to remove the remaining solids. The TOC was measured to determine the polymer excess (free polymer) relative to the TOC from the baseline measurement.

Quartz crystal microbalance with dissipation monitoring (QCM-D): Polymer adsorption onto model clay surfaces (silica, alumina and bitumen coated on silica QCM-D sensors) was determined by QCM-D (Q-Sense E4, Sweden). QCM-D is an ultra-sensitive balance in which the resonance frequency of a piezoelectric quartz crystal (5 MHz) is damped through the addition of a mass, at nanogram scale, to the crystal surface. In addition to resonant frequency, crystal dissipation monitored in the absence of a drive voltage provides structural information of the adsorbed polymer layer and its viscoelastic response (Tammelin et al., 2004). Depending upon the nature of the adsorbed layer the added mass can be calculated using appropriate modeling. Sauerbrey equation is commonly used for a rigid adsorbed layer that is distributed evenly and with the mass change smaller than 2% (Ekholm et al., 2002). For a "soft" (viscoelastic) layer, the adsorbed mass generates friction which increases dissipation (Ekholm et al. 2002). For such a system, the Sauerbrey equation is not valid and the Voigt Model for viscoelastic layers should be used (Voinova et al., 1999). Prior to each measurement or bitumen coating, all sensors were sonicated in Milli-Q water and cleaned using UV irradiation (~9 mWcm^{-2} at 254 nm) for at least 10 min, rinsed thoroughly with Milli-Q water and finally blow-dried with ultra-pure nitrogen. To coat a thin layer of bitumen on a QCM-D silica sensor, the silica sensor surface was first hydrophobized using dichlorodimethylsilane (DDMS, Fisher Scientific, USA) by placing a clean silica sensor in a 250-mL beaker with a small, open vial containing approximately 2 mL of

DDMS for 2 min. The beaker was covered with aluminum foil to allow DDMS vapors to deposit on the sensor. The sensor was then heated to 80 ^{o}C under vacuum for 24 h, causing the DDMS to bind chemically to the silica substrate. Using a spin coater (WS-400A-^NPP/Lite, Laurell Technology Corporation, USA) rotating at 2500 rpm, 8 drops of diluted bitumen (5 wt.% bitumen in toluene) were added dropwise onto the sensor surface. The sensor was allowed to rotate at this speed for 50 s, followed by rotation at 4000 rpm for 40 s to ensure an even layer and evaporate any excess solvent.

With the crystal mounted, the resonant frequency of the crystal was monitored to assess its cleanliness. The background solution (0.1 µm filtered process water, pH 8.2) was pumped through the measurement chamber at 0.15 mL/min until a stable baseline was attained. Once stable, the baseline was recorded for approximately 10 min, after which a 500 ppm polymer solution prepared in the same filtered process water was continuously pumped through the measurement chamber. The shift in resonant frequency and dissipation were monitored until steady state readings were obtained. Typically the experiment ran for 30 min.

X-Ray Diffraction (XRD): A Rigaku rotating anode XRD (Rigaku Corporation, Japan) equipped with a copper radiation source and a scan speed of 2°/s over a range of 10° to 110° was used to obtain diffraction patterns of the minerals. MFT solids of varying bitumen content prepared by protocol 1 were separated using Dean Stark extraction and ground to a powder using a mortar and pestle. Quantification of the mineral phases (Rietveld refinement) was obtained with Siroquant^{TM} software (Sietronics, Australia).

Results and Discussion

Settling performance: Figure 3 shows the initial settling rates of diluted MFT containing a varying amount of bitumen (Protocol 1) in the presence of 50 ppm flocculant. For the highest bitumen content (0.45 wt.%) the settling rate was measured to be ~2.5 m/h. This value is similar to that previously reported by Alamgir et al. (2012) for a similar polymer (Magnafloc 1011) at equivalent polymer dosage and solids content. With a reduction in the bitumen content to 0.18 wt.% , the settling rate increased to ~7 m/h, indicating an increase in polymer performance. With the bitumen content in the range 0.11 to 0.23 wt.%, the settling rates of MFT in the presence of 50 ppm flocculant are essentially the same in the range of ~ 6 – 7 m/h. The measured variability associated with these experiments most likely results from the inconsistent break-up of large flocs under shear during transfer and inversion of the settling column (sample conditioning). Also with high settling velocities, accurate visual determination of the mud-line becomes increasingly more difficult, thus contributing to an increase in the experimental error. Interestingly, with further bitumen removal to 0.08 wt.%, the settling rate of the resultant MFT after 50 ppm flocculant addition was observed to decrease to ~1.3 m/h, a value lower than the MFT without bitumen removal (0.45 wt.% bitumen) at same flocculant dosage. The substantial reduction in settling rate does not follow the perceived trend of improved settling performance with reduced bitumen content. Further discussion on the reduction in settling rate at low bitumen

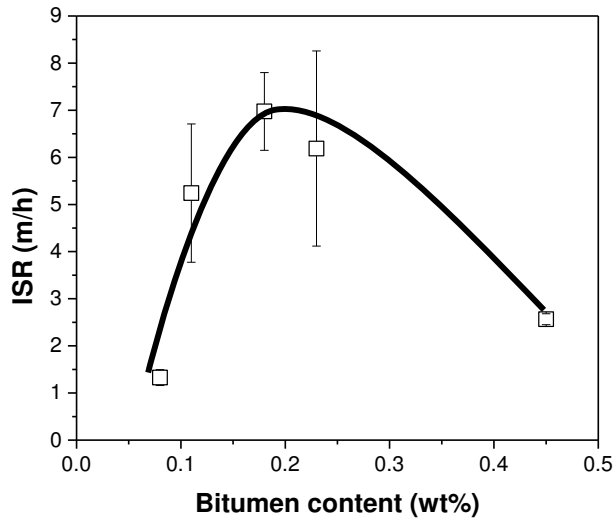content will be provided later in this paper.

Figure 3: Effect of bitumen content on the initial settling rate of diluted MFT with 50 ppm flocculant addition, bitumen content controlled by protocol 1.

To better understand the flocculation performance at different bitumen contents, FBRM® was used to measure in real time the floc size (mean square weighted chord length) under constant shear. Figure 4 shows the growth of flocs (ultimate floc size dependent on floc strength and dynamic shear) formed through the addition of 50 ppm flocculant. These experiments were conducted for three different bitumen contents of 0.08, 0.18 and 0.45 wt.%, which is inclusive of the lowest and highest settling rates shown in Figure 3. Immediately after polymer addition flocs form rapidly, reaching a maximum size within ~50 seconds. The floc size is then observed to decrease, resulting from either restructuring or fragmentation, and eventually reaching a steady-state size within 1 hr. For the MFT of the highest and lowest bitumen content, 0.45 and 0.08 wt.%, the maximum square weighted chord lengths were ~280 and ~300 μm, respectively. A square weighted chord length of ~350 μm was measured for the diluted MFT containing 0.18

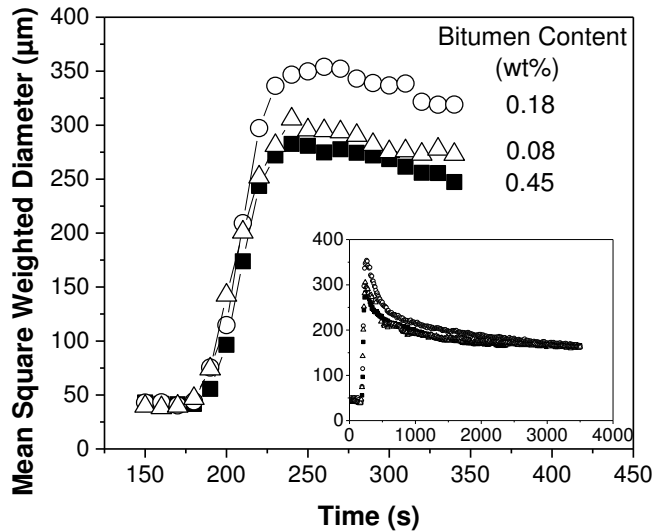wt.% bitumen, in agreement with the higher settling rate shown in Figure 3.

Figure 4: Flocculation kinetics as a function of bitumen content in MFT (protocol 1) with corresponding initial settling rates of ○ : 6.98m/h, △ : 1.33 m/h, and ■ : 2.57 m/h.

A decrease in settling rate and floc size at low bitumen content would indicate that a small amount of bitumen retained in the diluted MFT is detrimental to flocculation of MFT fines. This is contrary to the findings of Majid et al. (1991) who showed that any amount of bitumen removal resulted in a significant increase in both settling and consolidation of solids. However, it should be noted that the study of Majid et al. was for MFT without flocculant addition, with settling and consolidation performance monitored over weeks to months.

For the hydrolyzed polyacrylamide polymer, approximately 50% of the total mass is organic carbon. With 50 ppm polymer addition, based on total slurry mass, the total organic carbon added to the system is 0.025 g. If no polymer were to adsorb on the fine clays that would correspond to a maximum change in TOC concentration of approximately 25 mg/L. Figure 5 shows the measured TOC after polymer addition, relative to the baseline. Clearly, there is a

dependency on the bitumen content, with the optimum bitumen content resulting in the lowest

increase in the measured TOC concentration (~ 2.5 mg/L). As the settling performance reduces

the increase in the measured TOC concentration is shown to increase. For the lowest bitumen

content (0.08 wt.%) where the initial settling rate remains extremely low, the increase in the

TOC concentration is high, in the region of ~13 mg/L, confirming poor interaction of the

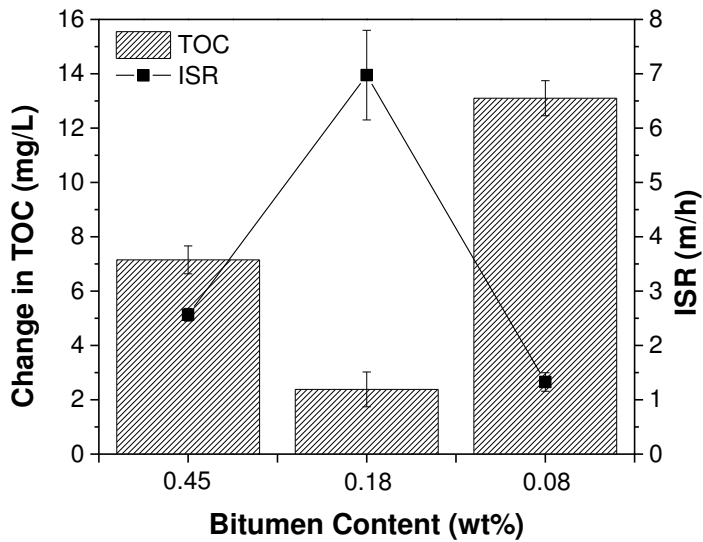polymer with the remaining fines after extended bitumen extraction from the MFT.

Figure 5: TOC increase from baseline and the corresponding initial settling rate after addition of

50 ppm polymer to MFT at pH 8.5, with bitumen content controlled by protocol 1.

A further study to measure polymer adsorption on representative particle surfaces (silica,

alumina and bitumen) was conducted using QCM-D. Figure 6 shows the frequency and

dissipation shifts of the 3rd overtone with continual pumping of 500 ppm polymer solution over

the sensor surface. Preferential adsorption of the polymer was measured on alumina, with little

adsorption on silica and bitumen coated surfaces. Such adsorption characteristics would indicate

that the anionic polyacrylamide interacts with the fines through electrostatic attraction between

negatively charged anionic polymers and positive alumina sites on clay edge surfaces. For

alumina, the resonance frequency (F3/3) of the crystal rapidly decreased to an equilibrium value

of −15 Hz and the dissipation simultaneously increased to ~5 × 10^{-6}, indicating the formation of

a viscoelastic film. Using the Voigt model with frequency and dissipation data of the 3rd and 5th

overtones, a corrected equilibrium mass uptake of ~9 mg/m^2 was measured on the alumina

surface. These polymer adsorption results on both silica and alumina are consistent with Alagha

et al. (2011) who reported a mass uptake on silica and alumina equal to 0 and ~10 mg/m^2,

respectively, for a similar anionic polymer (Magnafloc1011). The QCM-D data confirms

hindrance to flocculation by bitumen-contaminated fine solids, which is consistent with the

observed lower initial settling rate of MFT containing higher content (0.45 wt.%) of bitumen.
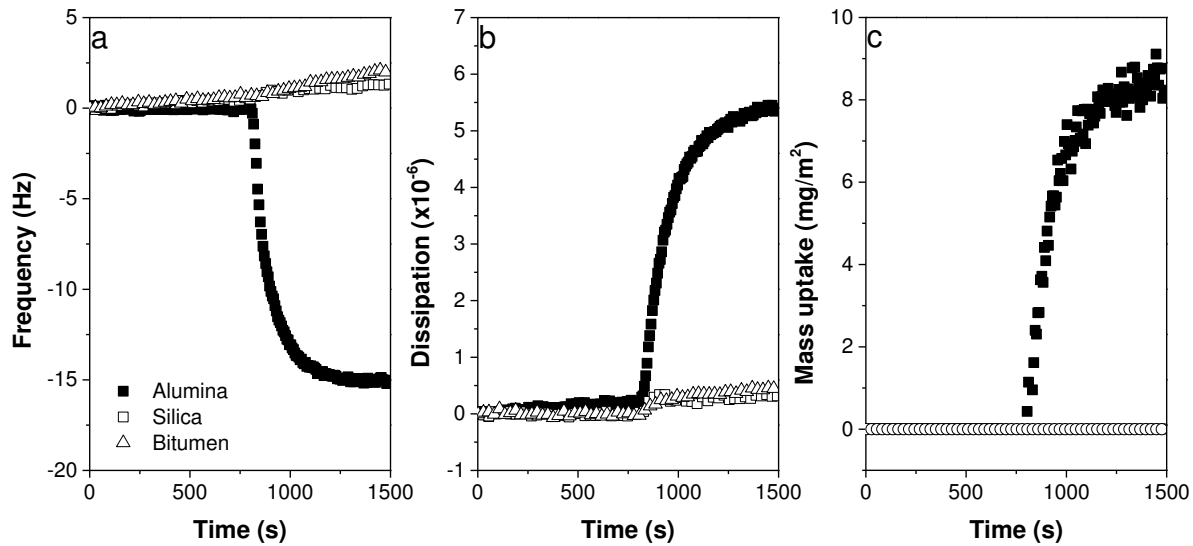

Figure 6: Adsorption of polymer flocculant (AF246) from 500 ppm solutions of pH 8.5 on model

particle surfaces: frequency (a), dissipation (b) and mass uptake (c) as a function of time.

However, contrary to conventional thinking and the QCM-D results, extensive removal of

bitumen from MFT by flotation is shown to correspond with decreased settling rates, smaller

sized flocs, and lower polymer adsorption, as shown in Figures 3, 4 and 5, respectively. To further understand the role of bitumen content on the flocculation of fine solids in MFT, two additional protocols to control bitumen content were considered. Protocol 2 includes blending of MFT after extraction of bitumen (0.08 wt.%, extended flotation) with diluted raw MFT (0.45 wt.%, untreated) to produce an overall bitumen content of 0.18 wt.% [blend composition: 73% of MFT containing 0.08 wt.% bitumen with 27% diluted MFT of 0.45 wt.% bitumen]. Protocol 3 includes solvent washing of diluted MFT (0.45 wt.% bitumen, no flotation) and MFT after bitumen extraction by extended flotation to 0.08 wt.% bitumen to remove any remaining toluene soluble organics. Figure 7 compares the results obtained with samples prepared using protocols 1 and 2 at an equivalent bitumen content of 0.18 wt.%. Clearly there is a substantial difference in the settling rate of MFT, albeit equivalent bitumen contents. The settling rate after 50 ppm polymer addition to the sample prepared using protocol 2 remains low ~1.2 m/h, even though the bitumen content is at the optimum level as determined by protocol 1. Since protocol 2 includes the blending of two poorly settling suspensions, the result may not be unexpected. The result does however confirm that the bitumen content is not a universal criterion for controlling solids-flocculant interactions and hence flocculation. The state of the bitumen in MFT and/or composition of fine solids need to be considered carefully to understand the role of bitumen content in flocculation of MFT.

For samples prepared using protocol 3, one would expect similar settling characteristics as the bitumen is completely removed from the MFT solids. However, a substantial difference in settling performance was observed between the two solvent-washed samples as shown in Figure 7 (triangle symbols). For the solids obtained by directly washing the dilute raw MFT (0.45 wt.% bitumen), the initial settling rate of the MFT with 50 ppm flocculant addition is very high, in the

range of ~9 m/h. However, for the solids obtained by solvent washing of MFT after bitumen removal by extended flotation (0.08 wt.% bitumen before washing), the settling rate was much lower at ~1.8 m/h. Conflicting settling rates between the two solvent washed samples suggest that the extended flotation of diluted raw MFT to obtain the samples of 0.08 wt.% bitumen content using protocol 1 has modified the solids composition of the resultant MFT in such a way that either the remaining particles are less responsive to the added flocculant or particles which could enhance fine particle flocculation, mostly hydrophobic particles have been preferentially removed by the flotation as anticipated.
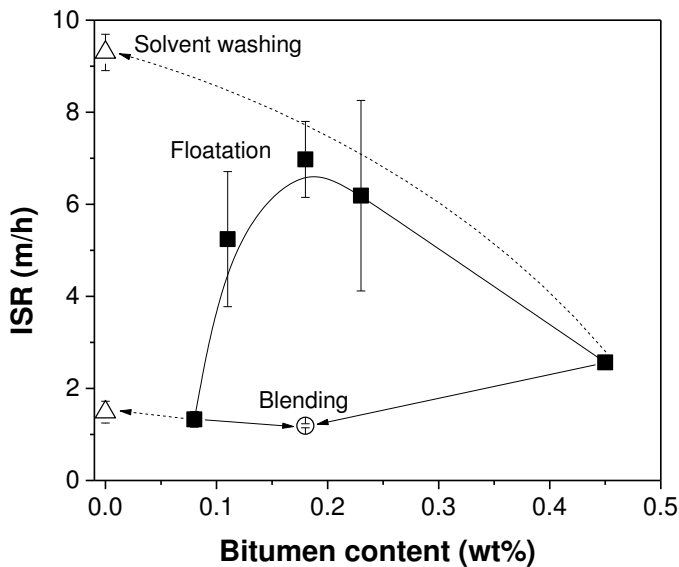

Figure 7: Impact of bitumen content protocol on the initial settling rate of diluted MFT; protocol 1 : ■; protocol 2: ○; protocol 3: △.

Even though there is preferential flotation of the hydrophobic species, measurement of particle concentration by gravimetric method, particle size distribution by light scattering, fines

mineralogy by XRD (Figure 8) and methylene blue index of fines (< 44 µm) suggest that these two tailings samples are similar.
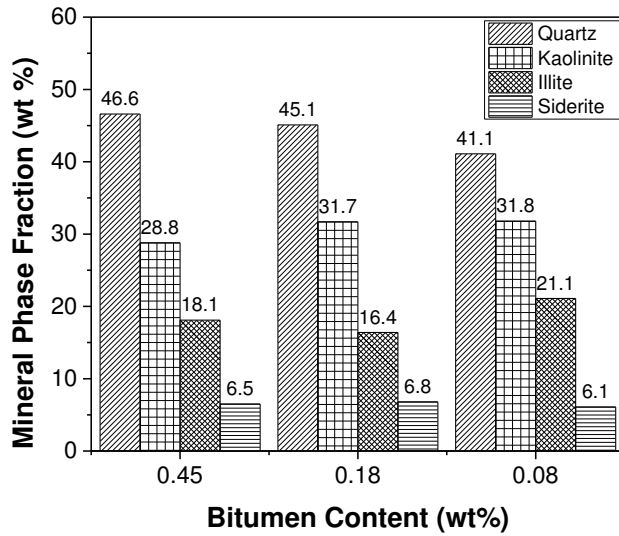

Figure 8: Mineral phases of solids from MFT samples of varying bitumen content controlled by protocol 1. Lower bitumen content indicates longer MFT flotation.

However, the initial settling rate data highlight contrasting behavior which would suggest that the properties of particles removed by flotation and the associated bitumen are the difference in controlling flocculation behavior of solids at very low bitumen content. Research to fully characterize clays and organic matter contamination of these solids and their interactions with flocculant is ongoing.

Conclusions

The major conclusions from this work are summarized as follows:

- The removal of bitumen from MFT by Denver cell flotation was found to improve flocculation and settling performance within the bitumen content range 0.45 – 0.18 wt.%. Any further removal of bitumen by flotation was shown to be detrimental to flocculation of remaining fine solids.

- The anionic polymer used throughout the study was shown to preferentially adsorb on alumina, with little adsorption on silica and bitumen substrates. The polymer interaction with fines is mainly through electrostatic attraction.

- Solvent washing of diluted MFT was shown to increase the settling rate from ~2.5 m/h to greater than 9 m/h. Solvent washing of the MFT after bitumen removal by extended floatation was shown to have a marginal improvement in settling performance, with the settling rate remaining low in the region ~ 1.5 m/h.

- Although removal of bitumen from MFT by toluene washing could significantly improve flocculation and settling of diluted MFT, complete removal of bitumen from MFT by flotation is not recommended as it leads to poor flocculation and slow settling of the resultant MFT.

Acknowledgements

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