Oil Sands Mature Fine Tailings Consolidation Through Microbial Induced Calcite Precipitation

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REPORT SUMMARY

The accumulation of mature fine tailings (MFT) in tailings ponds has been a concern of the oil sands industry and regulators for decades. Previous studies and current practices for fine tailings consolidation focus on the addition of inorganic and organic additives to reduce MFT production and accumulation. These treatment processes are were not sufficiently efficient in reduction of fluid tailings accumulation. In addition, treatments using chemical and polymer additives do not effectively increase the solids content of the settled tailings to more than 50%, which is lower than the required level for the surface trafficability mandated in Directive 074 issued by the Energy Resources Conservation Board. The development of treatment technologies to remove the last remnants of water from the settled tailings is critical. It is desirable to develop more efficient and effective techniques for MFT consolidation to reduce the risk to wildlife and ecosystems in the surrounding area.

In this study, the performance and mechanisms of a microbial induced calcite precipitation (MICP)-assisted MFT settling and consolidation method was assessed. MFT samples of 35 wt% and 60 wt% were treated with MICP. The volume of released water, the solids content of the tailings, and the shear strength of MFT were measured to evaluate the effects of MICP on MFT consolidation. MFT initial settling curves were developed by monitoring the change of the water-solid interface position over time. To investigate the surface interaction mechanisms involved in the process, the calcium concentration of MFT release water was measured and the size and shape of MFT particles were observed by scanning electron microscopy (SEM).

The results showed that although treatment with ureolysis-driven MICP effectively accelerated diluted MFT settling, ureolysis-driven MICP had little capacity to settle solids in raw (undiluted) MFT samples using tested reagents concentrations. In terms of MFT consolidation, our results clearly showed that ureolysis-driven MICP can accelerate raw MFT consolidation, leaving compact sludge with significantly enhanced shear strength within 24 hours of the experiment. Consolidation of the settled solids and settling of the MFT slurry through ureolysis-driven MICP might be achieved through precipitation of calcite on bacteria and solid surfaces, altering surface characteristics of particulate materials, and reducing steric or electrostatic stabilizing effects among particles.

The denitrification-driven MICP did not show promising results in terms of either MFT settling or consolidation. Denitrification may require more time and/or additional nutrient addition to accelerate the denitrification process, and to settle and consolidate MFT.

Ureolysis-driven MICP provides several advantages compared to traditional chemical additives currently used or studied to accelerate MFT consolidation: (1) ureolysis-driven MICP is effective for consolidating MFT from different sources and with different surface properties; (2) although mixing is needed to introduce the bacterial culture to MFT, mixing conditions do not significant impact the effectiveness of MICP-assisted consolidation process; (3) the key sources (calcium and carbonate ions) required to expedite $CaCO_3$ precipitation and MFT settlement are readily available in tailings; (4) the required dosage of urea is much less than that

of other traditional chemical additives; and, (5) $CaCO_3$ precipitation reduces the calcium concentration, potentially enhancing the quality of the recycled water.

For future research, experimental conditions for ureolysis-driven MICP can be improved by optimizing stoichiometric amounts of Ca and urea, bacterial growth conditions and nutrient levels. Field application strategies for urea and bacterial culture should be evaluated. Options for anaerobic calcite precipitation should be tested, particularly processes that utilize endogenous microorganisms. The options of combining MICP to other consolidation processes and to further improve MFT consolidation and strength should be tested and evaluated. Long-term studies on the impact of MICP assisted MFT settling and consolidation on land reclamation should be performed.

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1 INTRODUCTION

Alberta holds the largest oil sands reserve and the third largest proven energy reserve in the world (after Saudi Arabia and Venezuela). The Alberta oil sands reserve contains an estimated 2.5 trillion barrels of recoverable bitumen entrained in a mineral matrix of sand and clay (Penner and Foght 2010). Only about 2% of Alberta's initial resource has been produced to date (Government of Alberta 2009). Annual bitumen production from Alberta oil sands is expected to grow from 1.2 million barrels per day in 2008 to 3 million barrels per day by 2020, and possibly to 5 million barrels per day by 2030 (Giesy et al. 2010, Siddique et al. 2008).

The most commonly used method for producing bitumen from mined oil sands ore is the Clark hot water extraction (CHWE) process (Clark and Pasternack 1932). During the water-based extraction of bitumen from the oil sands, a large quantity of tailings is produced in the resulting slurry. The tailings contain residual bitumen, quartz sands, clays, and silts (Long et al. 2006b). The slurry is transferred to a tailings pond where the coarse sand settles rapidly leaving a stable suspension of fine solids in the middle and a low solids content water supernatant on the top. The aged stable suspension with >30% solids in the middle layer is called mature fine tailings (MFT). It is estimated that without treatment it will take several hundred years to consolidate MFT due to its extremely poor water releasing property (Headley et al. 2010, Mikula et al. 1996). The constant accumulation of MFT requires more and more ponds for storage; the ponds are a health risk to wildlife and disturb ecosystems in the surrounding area. The economic and environmental concerns created by the proliferating tailings ponds containing MFT have become one of the most critical challenges for the oil sands industry and regulators.

1.1 Objectives

Our research is testing feasibility of a novel, low-cost, and environment-friendly microbial induced calcite precipitation (MICP)-assisted consolidation process to increase the shear strength and settling rate of the MFT particles.

The specific objectives of this project were to:

1. Explore the potential application of MICP processes for MFT settling;
2. Explore the potential application of MICP processes for MFT consolidation; and
3. Compare the potential of ureolysis-driven MICP and denitrification-driven MICP to consolidate MFT.

1.2 Report Organization

The proposed theories and experimental design of the MICP-assisted MFT settling and consolidation process are discussed in sections 2 and 3. The performance of MICP on MFT settling and consolidation and the mechanisms involved are evaluated and discussed in section 4. Conclusions of the study are provided in section 5.

2 BACKGROUND

2.1 Mature Fine Tailings (MFT)

Bitumen extraction from oil sands requires approximately 12 m^3 of water to produce 1 m^3 of crude oil (Mikula et al. 2009). Approximately 4 m^3 of slurry waste is generated consisting of a matrix of sand, clay, water, fine particles (silt and clay < 44 μm in diameter), residual bitumen (0.5% to 5% mass), and, sometimes, diluent (< 1% mass) used in the extraction process (Chalaturnyk et al. 2002). MFT are clay-dominated particles suspended in the large on-site settling ponds after the coarse sand fraction rapidly settles (Penner and Foght 2010). Greater than 90% of MFT solids are fine particles to which aqueous organic matter and residual bitumen are usually attached (Adegoroye et al. 2010). It has been shown that the adsorption of organic matter onto particles leads to an increase in their negative surface potentials, contributes to colloidal stability, and consequently inhibits the aggregation of particles in an aquatic environment (Chandrakanth et al. 1996, Farvardin and Collins 1989, Jekel 1986). Without treatment, it is estimated that the fine clay will require hundreds of years to reach full consolidation (Headley et al. 2010, Mikula et al. 1996).

2.2 Existing Technologies for Sludge Settling

Technologies based on physical, chemical, and biological methods have been investigated to improve MFT consolidation and enable reuse or release of the process-affected water (Li et al. 2003, Matthews et al. 2000, Mpofu et al. 2004, Proskin et al. 2010, Sworska et al. 2000, Wang et al. 2010). The advantages and disadvantages of traditional methods of MFT consolidation are described in sections 2.2.1 to 2.2.3.

2.2.1 *Inorganic Additives*

Flue gas desulfurization (FGD) gypsum ($CaSO_4 \bullet 2H_2O$) has been widely used to accelerate tailings densification. The most effective and economically viable gypsum dosages are between 900 and 1,200 g/m^3 (Matthews et al. 2000). However, the continuous accumulation of Ca^{2+} and SO_4^{2-} ions in the recycled water detrimentally affects bitumen extraction efficiency (Redfield et al. 2003).

An alternative option for tailings consolidation is the application of a 50% solution of alum (aluminum sulfate, $Al_2(SO_4)_3 \bullet 14.3H_2O$) at a dosage of about 1,000 g/m^3. Compared with gypsum, alum addition leads to lower salinity and lower pH in recycled water (MacKinnon et al. 2000). However, from operational and reclamation aspects, these inorganic coagulants have an undesirable impact on the resulting water (Redfield et al. 2003).

Carbon dioxide was also tested to assist tailings densification (Zhu et al. 2011). It was found that lowering the pH with CO_2 caused the MFT to coagulate. It is commercially applied by CNRL. However, the low solubility of CO_2 in saline water limits the CO_2 sequestration in oil sands tailings.

Our recent studies evaluated the feasibility of applying ozonation to accelerate MFT settling (Liang et al. 2014). High ozonation of MFT leads to the production of low molecular weight organic acids, causing reduced pH, enhanced ionic strength of the MFT suspension, and neutralized MFT surface charges. Even a low ozone dosage was sufficient to settle diluted MFT (up to 5% solids content). However, it is a challenge to apply this process to MFT with >5% solids content (Liang et al. 2014).

2.2.2 *Polymer Additives*

High molecular weight anionic polymers (e.g., polyacrylamides and their derivatives) have been used commercially for tailings treatment (Sworska et al. 2000). Strong adhesion forces were detected between fine solids in aqueous solutions in the presence of partially hydrolyzed polyacrylamide (HPAM) which acted as a flocculant (Long et al. 2006a). More recently, hybrid $Al(OH)_3$-polyacrylamide (Al-PAM) was shown to be even more effective than HPAM in assisting filtration of MFT (Alamgir et al. 2012, Wang et al. 2010). Although hydrolyzed polyacrylamide (HPAM) can enhance the flocculation of clay fines, the environmental implications of polymer addition are still unknown (Pourrezaei et al. 2011) and may be harmful (Mpofu et al. 2004) since acrylamide monomer is a neurotoxin and potentially carcinogenic. In addition, polymer-based processes incur high operational costs.

Farkish and Fall (2013) applied super absorbent polymer (SAP) to increase shear strength of MFT with initial solid content of 40%. Under optimized conditions, the shear strength of MFT was increased to about 1 kPa after 24 hours mixing with 0.5 wt% SAP (about 10,000 mg/L). However, the performance of SAP could be significantly reduced by changing the MFT pore water chemistry of medium such as ionic strength and pH (Farkish and Fall 2013).

2.2.3 *Microbial Assisted MFT Consolidation*

Microbial assisted MFT consolidation has been explored by other research groups, who mainly focused on the potential applications of methanogenesis for biodensification of MFT. It has been demonstrated that methanogenesis can accelerate MFT settling through changing the MFT pore-water chemistry and the surface properties of suspended clays (Arkell 2012, Li 2010). However, methanogens are slow growers, and carbon source addition (e.g., acetate, glycerol) is often needed to support/stimulate the growth of these microorganisms. As well, methane is a potent greenhouse gas and companies would be responsible for additional methane emissions.

2.3 Microbial Induced Calcite Precipitation

Microbial induced calcite precipitation (MICP) is a microbial-catalyzed process that, in the presence of excess calcium ions, leads to the precipitation of carbonates to form calcite ($CaCO_3$) in situ (DeJong et al. 2010). The formation of calcite on the surface of microbial cells leads to cohesive bonding within the soil particle matrix.

Several mechanisms have been proposed for MICP. For example, a change in environmental conditions during urea degradation promoted by urease-producing bacteria can induce $CaCO_3$ precipitation. The bacteria act as nucleation sites to facilitate the precipitation of $CaCO_3$ on bacterial cell surfaces (Ferris et al. 1987).

Chemical reactions associated with ureolysis-driven MICP are listed in Equations 1 to 6 below:

$$CO(NH_2)_2 + H_2O \rightarrow NH_2COOH + NH_3 \quad (1)$$

$$NH_2COOH + H_2O \rightarrow NH_3 + H_2CO_3 \quad (2)$$

$$2NH_3 + 2H_2O \leftrightarrow 2NH_4^+ + 2OH^- \quad (3)$$

$$H_2CO_3 \leftrightarrow HCO_3^- + H^+ \quad (4)$$

$$HCO_3^- + H^+ + 2OH^- \leftrightarrow CO_3^{2-} + 2H_2O \quad (5)$$

$$Ca^{2+} + CO_3^{2-} \leftrightarrow CaCO_3 \quad (6)$$

During ureolysis-driven MICP, urease catalyzes the hydrolysis of urea to form ammonium (NH_4^+) and hydroxide (OH^-) ions (Equations 1 to 3). The generated hydroxide ions shift the equilibrium of dicarbonate species (H_2CO_3) toward bicarbonate (HCO_3^-) and carbonate (CO_3^{2-}) (Equations 4 and 5). $CaCO_3$ is precipitated in the presence of sufficient calcium ions (Equation 6). Carbon dioxide dissolved in water precipitates calcite through a series of reactions, as shown in Equations 4 to 6. $CaCO_3$ precipitation engendered by urease producing bacteria is governed by four factors: (1) the presence in solution of a significant concentration of calcium (Ca^{2+}), (2) the presence of dissolved inorganic carbon, (3) alkaline pH, and (4) the availability of nucleation sites e.g., bacterial cells

During denitrification-driven MICP, denitrifiers consume the carbon source and nitrate, leading to the production of nitrogen and carbon dioxide as shown in Equation 7. $CaCO_3$ is then precipitated in the presence of sufficient calcium ions.

$$5CH_3COO^- + 13H^+ + 8NO_3^- \leftrightarrow 10CO_2 + 14H_2O + 4N_2 \quad (7)$$

MICP has been successfully applied to solidify loose soil and to cement porous media in many geotechnical projects (Montoya et al. 2013, van Paassen et al. 2010). For example, previous studies have shown that the application of MICP increased the shear strength and stiffness of soil by 100 times, i.e., the effective vertical stress of cemented sands could be increased from 1 kPa to around 100 kPa (DeJong et al. 2010, Montoya et al. 2013). In a laboratory sand column experiment, cementation was induced over a distance of 5 m by injecting bacteria and reagents sequentially (Harkes et al. 2010). Additionally, a large-scale (100 m^3) experiment demonstrated that MICP could be used to significantly improve the stiffness of granular soils in biogrouting-ground applications (van Paassen et al. 2010).

Under suitable conditions MICP can conceivably be applied to precipitate suspended fine particles in MFT, settle the MFT slurry and strengthen the settled solids. Calcium and carbonate ions, the key ions required to expedite MICP and MFT settlement, are readily available in tailings ponds (Liang et al. 2014), providing a unique opportunity to test the possibility of applying MICP to consolidate fine tailings. Calcite precipitation can also reduce the salinity of release water, improving the quality of the recycled water.

3 METHODOLOGIES

3.1 Materials

Two MFT samples having a solids content of 60 wt% (Sample A) and 35 wt% (Sample B) were obtained from two oil sands companies and used throughout the experiments. The MFT samples were sealed and stored at 4 ℃. *Sporosarcina pasteurii* (ATCC® 11859)[1], a urea-hydrolyzing bacterium, was used in the ureolysis-driven MICP experiment. The role of *S. pasteurii* in ureolysis-driven $CaCO_3$ precipitation and its potential role in a number of diverse applications has been widely studied.

Halomonas halodenitrificans (ATCC® 13511) has been shown to precipitate calcium carbonate (Martin et al. 2013). Therefore, *H. halodenitrificans* was also applied to test its ability to solidify MFT through denitrification-driven MICP.

3.2 Bacteria Cultivation

S. pasteurii were cultured in an ammonium-yeast extract medium (ATCC 1376: 0.13 mol/L Tris buffer (pH 9.0), 10 g/L $(NH_4)_2SO_4$, and 20 g/L yeast extract) according to the supplier's instructions. The cultures were streaked onto ammonium-yeast extract medium agar plates and incubated at 30 °C for 48 hours. A single colony from the fresh agar plate was transferred to 150 mL ammonium-yeast extract medium broth in a 500 mL Erlenmeyer flask. The *S. pasteurii* culture was shaken at 150 rpm at 30 °C in a shaker incubator (New Brunswick Scientific Co., New Jersey) for 48 hours.

A nutrient agar medium with NaCl (ATCC 87: 23.0 g/L nutrient Agar, 60.0 g/L NaCl, pH 7.0 to 7.4) was prepared to culture *H. halodenitrificans*. Cultivation of *H. halodenitrificans* was similar to that of *S. pasteurii*, except that the temperature was 26 °C. Cells were harvested by centrifugation at 8,000 g at 4 °C for 10 minutes. The growth medium was decanted and the pellet was resuspended in deionized (DI) water. Centrifugation was repeated to remove traces of growth medium.

3.3 MFT Settling

In the ureolysis-driven MICP experiment, 20 mL of MFT were diluted to 100 mL by adding an *S. pasteurii* suspension, urea, and DI water. The final concentrations of *S. pasteurii* and urea were 10^9/mL and 18 g/L, respectively.

In the denitrification-driven MICP experiment, *H. halodenitrificans* were applied instead of *S. pasteurii* in a similar protocol. In addition, sodium acetate and sodium nitrate (0.3 M each, 25 g/L) were added to the mixture as acetate and nitrate sources.

Bacteria and chemicals in lower concentrations were also examined. The reported concentration was the optimal condition we tested.

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[1] See http://en.wikipedia.org/wiki/Sporosarcina_pasteurii

The MFT mixtures were sealed and thoroughly shaken manually for 1 minute in a 100 mL volumetric cylinder, and allowed to settle for 24 hours. The height of the water-solid interface was recorded occasionally during the settling period and at the 24-hour end point. The extent of MFT aggregation after MICP treatment was evaluated by measuring the volume of release water and plotting 24 hour settling curves. The settling curves were created by plotting the water-solid interface positions over time for the 24 hours of settling.

3.4 MFT Consolidation

MFT consolidation experiments were performed in 1,000 mL beakers to accommodate subsequent shear strength measurements. For the ureolysis-driven MICP reaction, 9 g urea and 5 mL of *S. pasteurii* suspension were added to 500 mL of MFT during mechanical mixing. For the denitrification-driven MICP reaction, 5 mL of *H. halodenitrificans* suspension and 12.5 g each of sodium nitrate and sodium acetate were added to 500 mL of MFT during mixing. The beakers were sealed with aluminum foil and stored for 24 hours at room temperature before the shear strength and pore water chemistry analyses were carried out.

The shear strength of MFT was measured with the vane shear test using a viscometer because the MFT moisture content was high and the estimated shear strength was lower than 1 kPa, the lowest value that the vane shear test can measure. The rotation speed (0.1 rpm) of the spindle and the time interval (1 second) of data printing were defined before each test. The viscometer was connected to a computer to record data. To measure the shear strength of MFT, the spindle attached to the viscometer was inserted in the MFT sample until the spindle was fully submerged. Spindles of different sizes can measure different ranges of shear strength. We selected spindle #73 (height 2.535 cm, diameter 1.267 cm) and spindle #74 (height 1.176 cm, diameter 0.589 cm) which can measure up to 0.1 kPa and 0.96 kPa, respectively. As the spindle rotated at the predefined speed, the reading rose steadily, then began to drop. The maximum value of the viscometer reading was recorded as the maximum percentage of viscometer spring deformation. The shear strength of the MFT sample was recorded as a percentage of the maximum torque of the selected spindle.

3.5 MFT Solids Content and Release/Pore Water Chemistry Analysis

To determine the solids content of MFT, approximately 1 g of MFT was transferred to a crucible and dried at 105 ℃ overnight. When all of the water had been evaporated from the MFT, the crucible was cooled to room temperature in a desiccator and the dried MFT was weighed.

To determine the calcium concentrations in MFT release/pore water, the water was centrifuged at 5,000 x g for 10 minutes to remove suspended particles. The supernatant obtained from the centrifugation process was filtered through 0.45 μm syringe filters to remove remaining particles and titrated against EDTA using a CalVer® 2 Calcium Indicator to determine the calcium concentration.

To obtain a more comprehensive MFT release/pore water profile, samples were analysed by inductively coupled plasma-mass spectrometry (ICP-MS). MFT release water was centrifuged at

5,000 x g for 10 minutes to remove suspended particles. The supernatant obtained from centrifugation was filtered through 0.45 µm filters to remove remaining particles.

3.6 Scanning Electron Microscopy

The size and the shape of MFT fine particles before and after MICP treatment were observed using scanning electron microscopy (SEM) (S-2500, Hitachi, Japan). Prior to imaging, MFT samples were placed on small pieces of 0.22 µm filter paper and fixed in 2.5% glutaraldehyde in phosphate buffer at room temperature for 30 minutes. Samples were dehydrated with 10 minute washes with a series of ethanol solutions (35%, 50%, 70%, 90%, and absolute ethanol) for 10 minutes each, then dried with CO_2 at 31 °C for 15 minutes. The dried samples were mounted on the specimen stub of the microscope specimen holder and sputter coated with gold (Edwards, Model S150B Sputter Coater).

3.7 Statistical Analysis

One-way analysis of variance (ANOVA) was used to validate data differences. Microsoft Excel® software was used to perform ANOVA and results were reported as p-values; p-values of less than 0.05 suggested that differences were statistically significant.

4 RESULTS AND DISCUSSION

4.1 The Effect of MICP on MFT Settling

4.1.1 *MFT After Ureolysis and 24-hour Settling*

Preliminary experiments showed no significant settlement after 24 hours for undiluted MFT samples (Figure 1), indicating the MICP process was not successful for MFT with high solids content (> 35% MFT).

To evaluate the potential application of ureolysis-driven MICP for fresh tailings suspensions, the MFT sample was diluted with oil sands process-affected water (OSPW) or distilled deionized (DI) water to mimic fresh tailings (Alamgir et al. 2012). Our results showed that MFT dilution using OSPW led to fast MFT settling. As a result, we were not able to observe the visual impact of ureolysis-driven MICP on the settling of MFT diluted with OSPW. Similar results have been observed in our previous studies and have been confirmed in oral communications with other research groups studying MFT settling.

The settling of MFT diluted with DI water after ureolysis-driven MICP treatment was studied with various DI water/MFT dilution ratios (Volume $_{\text{DI water}}$: Volume $_{\text{MFT}}$), including 1:1, 2:1, 4:1 and 8:1. We observed that the settling rate of MFT diluted with DI water was significantly enhanced by the ureolysis-driven MICP treatment when the DI water/MFT dilution ratio was greater than three. Consequently, the effect of MICP on MFT settling was studied further only for DI water/MFT volume ratios of 4:1.

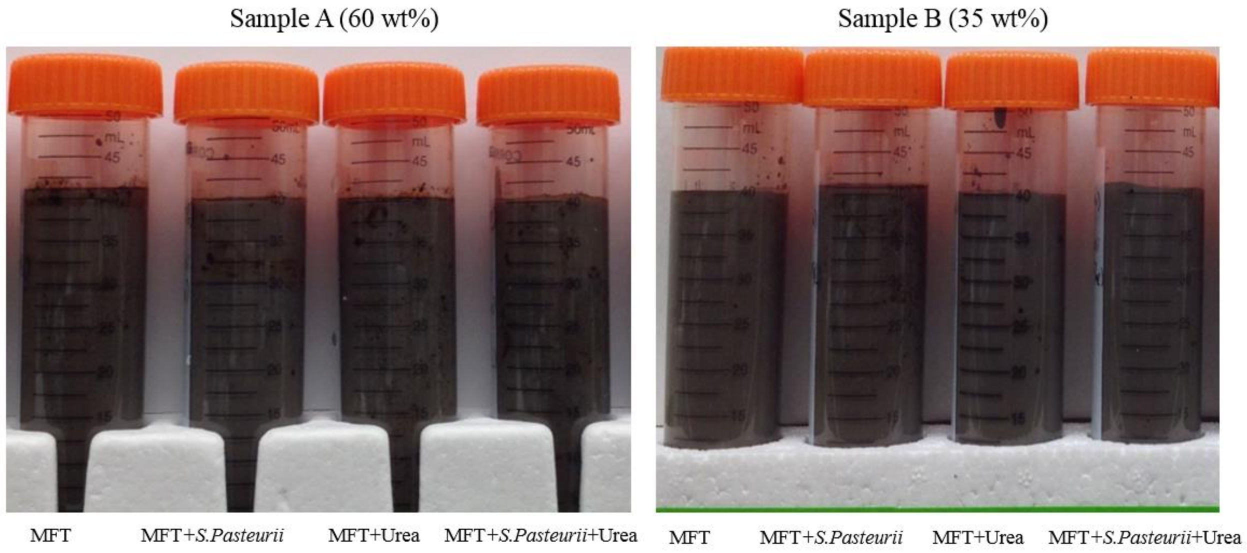

Figure 1. Image of undiluted MFT settling after 24 hours with and without ureolysis-driven MICP.
Additive concentrations: *S. pasteurii* 10^9 cells/mL; urea 18 g/L.

The 24 hour settling results of 4:1 diluted MFT suspensions with and without MICP treatment are illustrated in Figure 2.

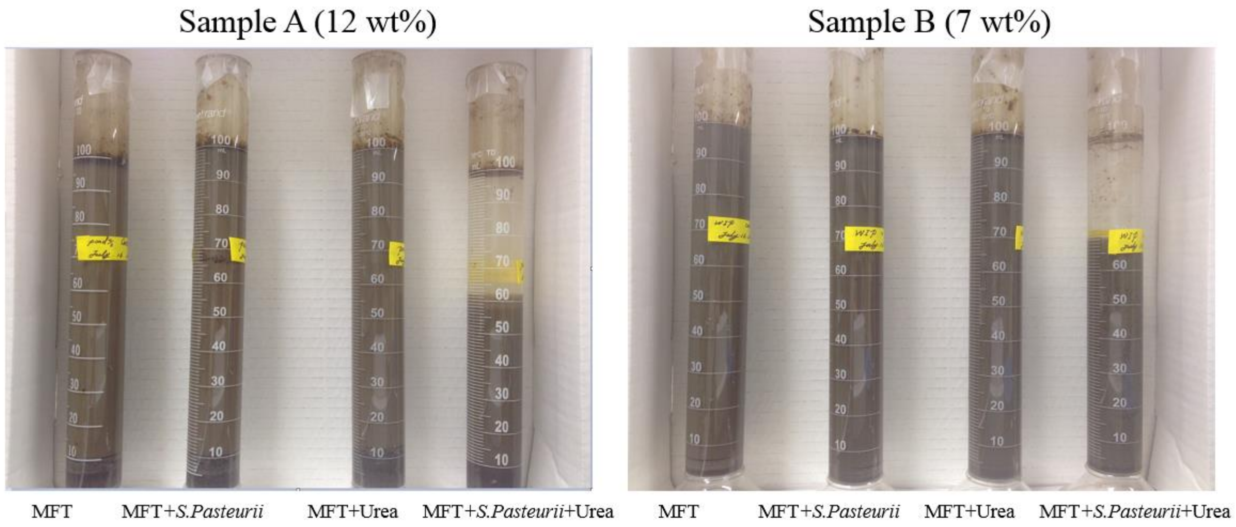

Figure 2. Images of DI water diluted MFT (Vol. = 4:1) after 24 hours settling with and without ureolysis-driven MICP treatment.
Additive concentrations: *S. pasteurii* 10^9 cells/mL, urea 18 g/L.

Figure 2 indicates that the addition of *S. pasteurii* or urea alone did not induce visible settlement of diluted MFT samples A (12 wt% MFT) and B (7 wt% MFT) after 24 hours. When both bacteria and urea were added to MFT, about 40 mL and 30 mL supernatants in MFT samples A and B, respectively, were observed after 24 hours settling. These results indicate that diluted MFT can be settled only by the combined addition of urea and *S. pasteurii* which work together to produce ureolysis-driven MICP.

Figure 3 reveals that MFT treated with ureolysis-driven MICP took some time to settle. This suggests that it takes time for *S. pasteurii* to adapt to the MFT environment before it can produce the urease enzyme, and/or that the hydrolysis of urea and the generation of calcite and other chemical reactions related to MICP take place slowly.

No MFT settling was observed in denitrification-driven MICP. Therefore, MFT settling was studied only for ureolysis-driven MICP.

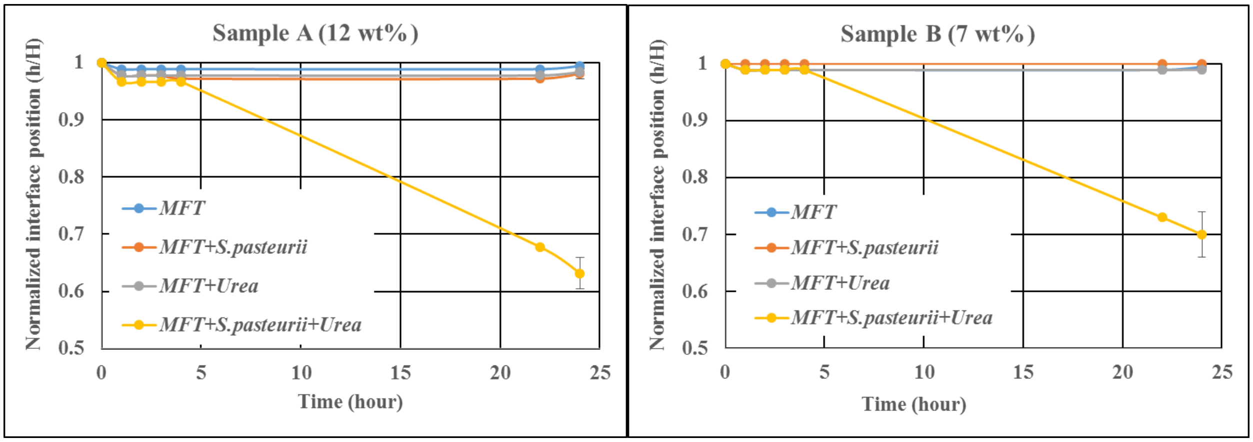

Figure 3. Settling curve of diluted MFT (Vol. = 4:1) with and without ureolysis-driven MICP treatment.
Additive concentrations: *S. pasteurii* 10^9 cells/mL, urea 18 g/L.
Error bars represent standard deviations of triplicated samples.

4.1.2 *Calcium Concentration in MFT Release/Pore Water*

The calcium concentration in MFT release/pore water under different conditions is compared in Figure 4.

The addition of *S. pasteurii* or urea alone did not cause a significant change in the calcium concentration of MFT samples. However, when both *S. pasteurii* and urea were applied, calcium was not detected after 24 hours. These data indicate that calcium was not consumed in MFT samples containing urea or *S. pasteurii* alone; calcium was consumed during ureolysis-driven MICP in the presence of urea and *S. pasteurii*. Diluted Sample A (12 wt% MFT) had higher calcium concentration than Sample B (7 wt% MFT).

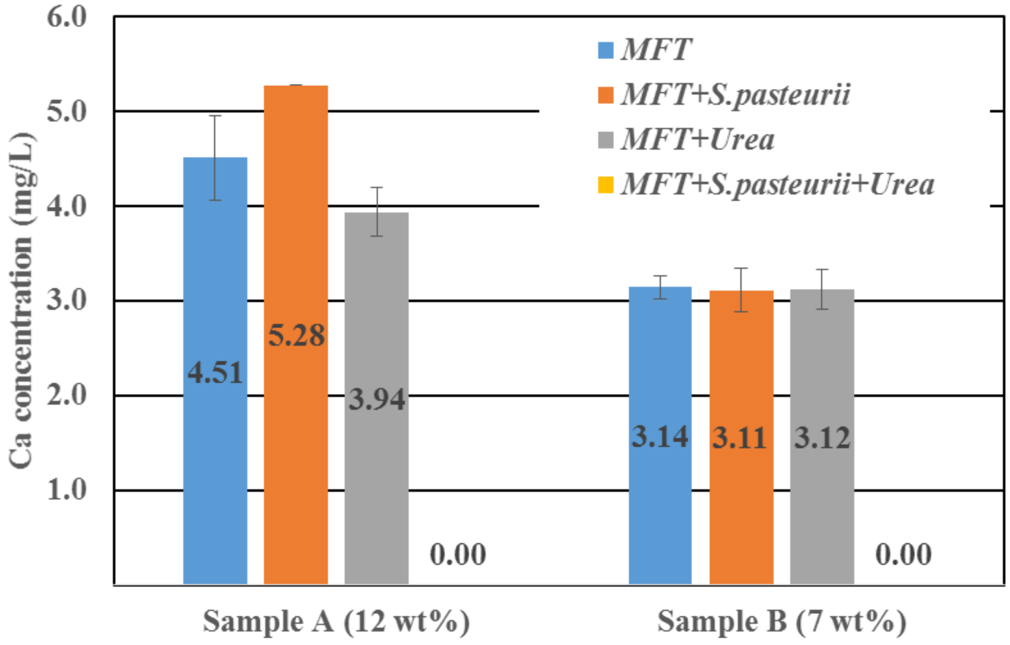

Figure 4. Ca^{2+} concentration in MFT released/pore water after 24 hours settling of diluted MFT (Vol. = 4:1) with and without ureolysis-driven MICP treatment.
S. pasteurii 10^9 cells/mL, urea 18 g/L.
Error bars represent standard deviations of triplicated samples and measurements.

4.1.3 *pH of MFT Release Water*

The pH of MFT release water under different conditions is shown in Figure 5. The original pH of diluted MFT was about 8.7 in both MFT samples. The water chemistry was similar in Sample A and Sample B. The weak basic pH of MFT was due to the caustic additions during the oil sands extraction process (Siwik et al. 2000) and redox processes in settled tailings.

The addition of *S. pasteurii* or urea alone did not induce a significant change in pH in MFT samples. After MICP treatment, the pH in MFT samples increased from ~ 8.7 to ~ 9.2. MICP reaction leads to urea hydrolysis and production of ammonia and carbon dioxide. Carbon dioxide is removed through calcite precipitation. As a result, the pH of MICP-processed MFT is increased.

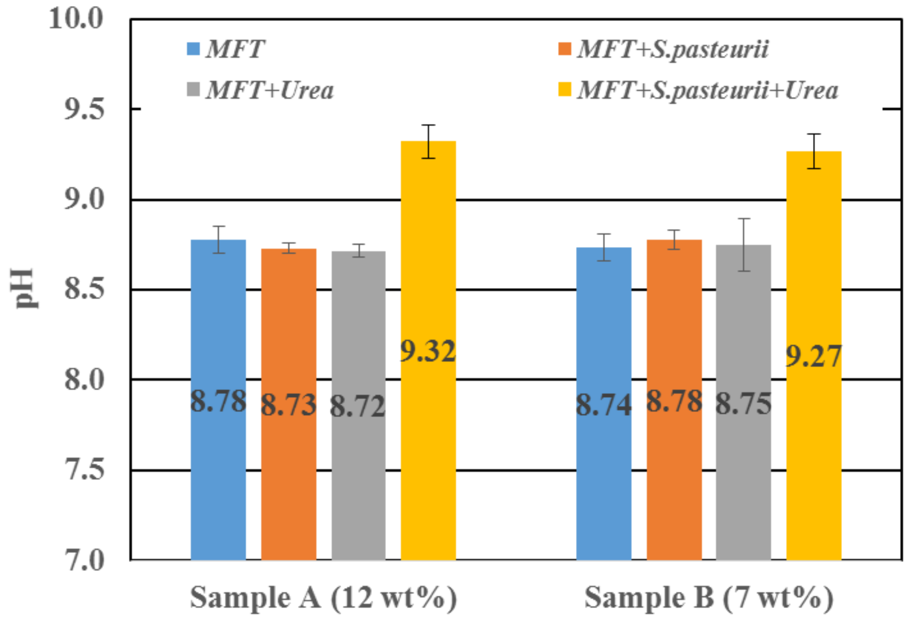

Figure 5. pH of MFT released/pore water after 24 hours settling of diluted MFT (Vol. = 4:1) with and without ureolysis-driven MICP treatment.
S. pasteurii 10^9 cells/mL, urea 18 g/L.

4.2 Effect of Ureolysis-Driven MICP on MFT Consolidation

4.2.1 *Shear Strength of MFT*

In the MFT consolidation experiment, the extent of MFT consolidation was represented by the shear strength of MFT. Shear strength of a material is the internal resistance per unit area that the material mass can offer to resist failure and sliding along any plane inside it.

Figure 6 summarizes the shear strength of MFT in each experimental condition. The shear strengths of the raw MFT samples A (60 wt%) and B (35 wt%) were about 0.02 kPa. The shear strength of MFT did not change significantly after the addition of *S. pasteurii* or urea alone. However, the shear strength of MFT samples A and B increased significantly to 0.79 kPa and 0.5 kPa, respectively, after both *S. pasteurii* and urea were added. Ureolysis-driven MICP increased the shear strength of Sample A more than that of Sample B. This was probably due to the lower water content of Sample A.

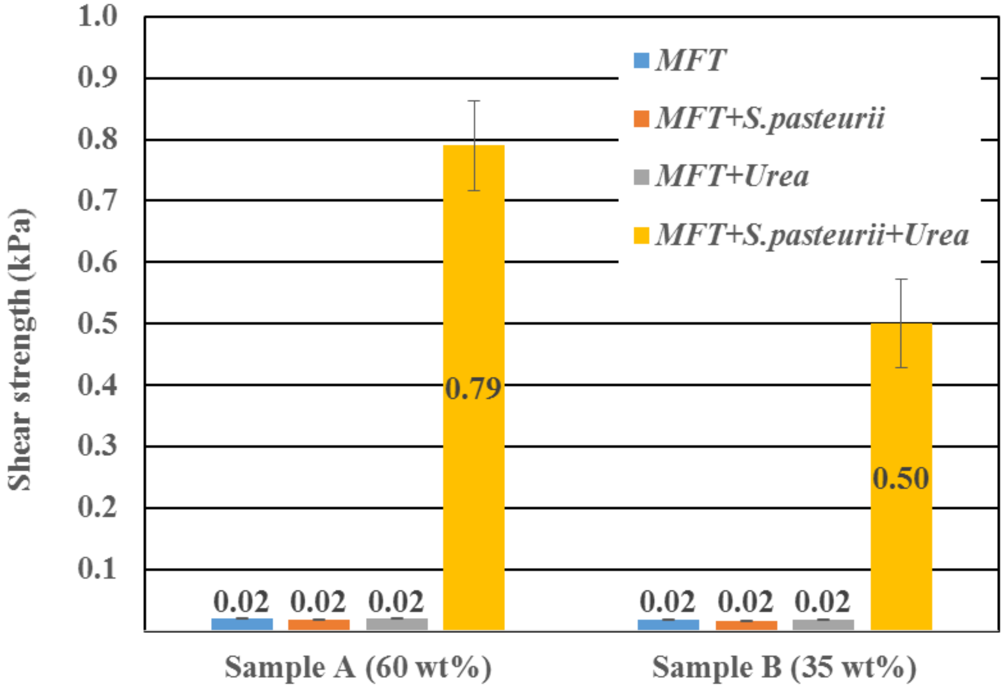

Figure 6. Shear strength of MFT treated for 24 hours with and without ureolysis-driven MICP. *S. pasteurii* 10^9 cells/mL, urea 18 g/L.
Error bars represent standard deviations of multiple samples and measurements.

4.2.2 *Solids Content of MFT*

The MFT solids content was measured to investigate the mechanism involved in the MICP-assisted MFT consolidation. As shown in Figure 7, the solids content of MFT was comparable under different conditions, indicating that calcium precipitation did not lead to an enhanced solids content in MFT. This observation might be attributed to the fact that the original calcium concentration was low (Figure 4). Precipitation of all the calcium present in the pore water would contribute only several milligrams of solid in a 500 mL MFT sample, a quantity of precipitate that would be negligible. Options to further enhance the solids content of settled MFT deserve further studies.

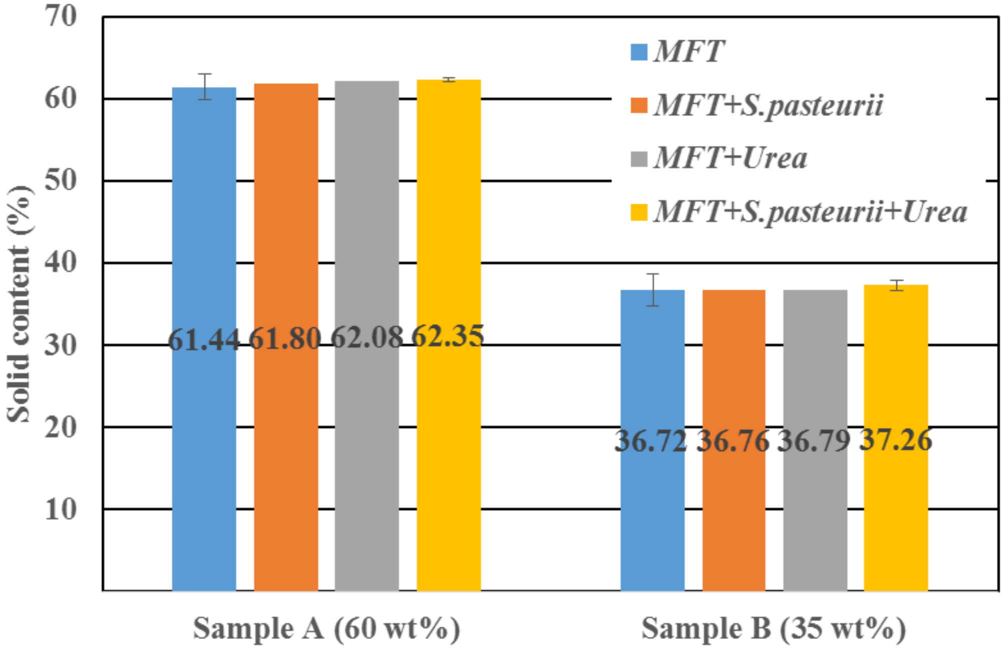

Figure 7. Solids content of MFT treated for 24 hours with and without ureolysis-driven MICP. *S. pasteurii* 10^9 cells/mL, urea 18 g/L.
Error bars represent standard deviations of multiple samples and measurements.

4.2.3 *Surface Characterization of MFT*

The shape and size of MFT particles were observed by SEM. Figure 8 shows the representative images of MFT before and after ureolysis-driven MICP treatment. Generally, the size of original MFT particles was less than 50 µm and the particles were uniform in shape. Some large aggregated particles were observed in the MFT after ureolysis-driven MICP in both samples A and B, which was not observed in the untreated samples. There were no significant differences between samples A and B, suggesting that the physical appearance of the particles was similar. We were not able to identify calcite deposits in these SEM images.

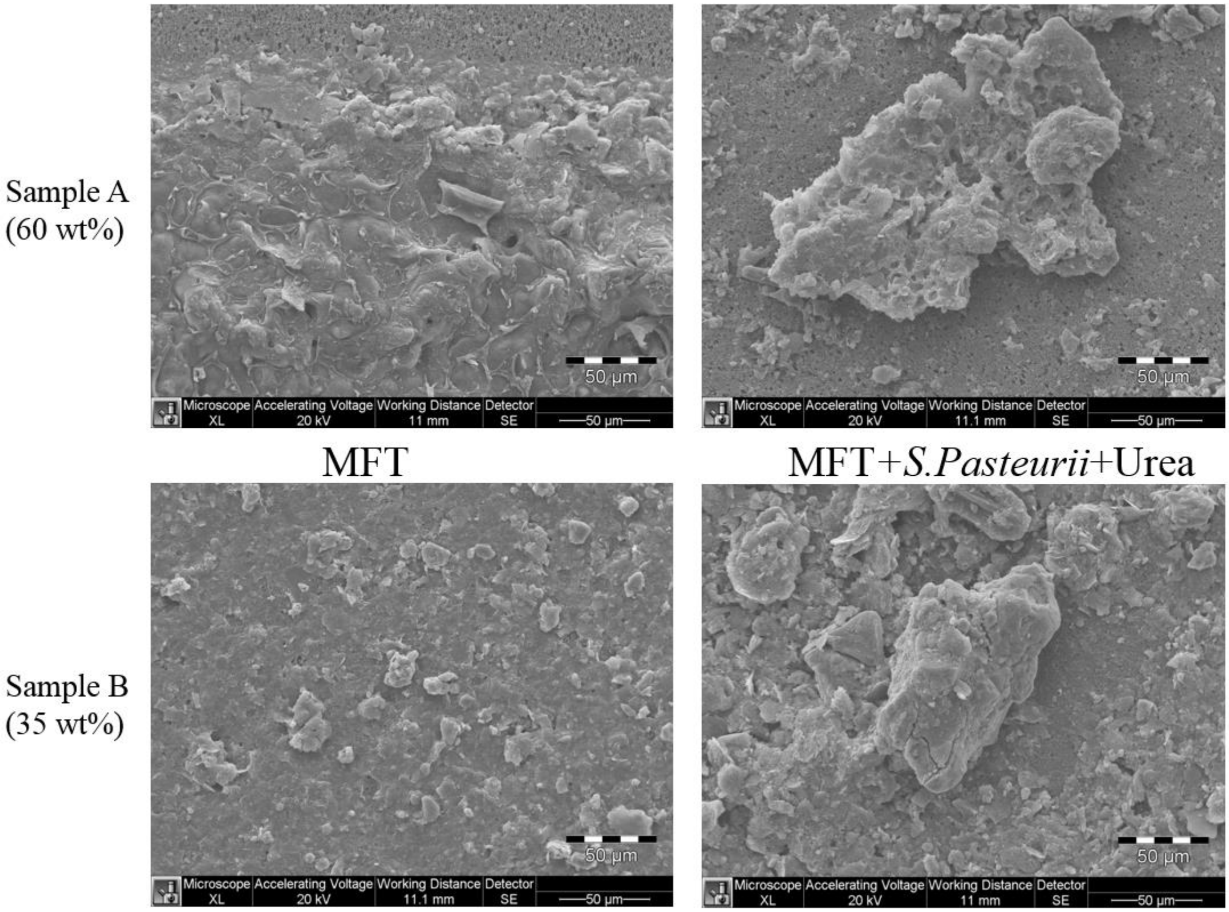

Figure 8. SEM images of MFT before and after ureolysis-driven MICP treatment.

4.3 Denitrification-Driven MICP in MFT

The results of addition of salts in the denitrification experiment on MFT shear strength are shown in Figure 9. The shear strength of MFT was increased from ~ 0.02 kPa to ~ 0.22 kPa in Sample A and from ~ 0.02 kPa to ~ 0.14 kPa in Sample B, by the addition of sodium nitrate (electron acceptor for denitrification) and sodium acetate (electron donor for denitrification), which could (1) stimulate in situ denitrification by tailings endogenous denitrifiers and (2) lead to enhanced ionic strength in the MFT pore water. MFT shear strength did not increase further with application of the external denitrifier *H. halodenitrificans*.

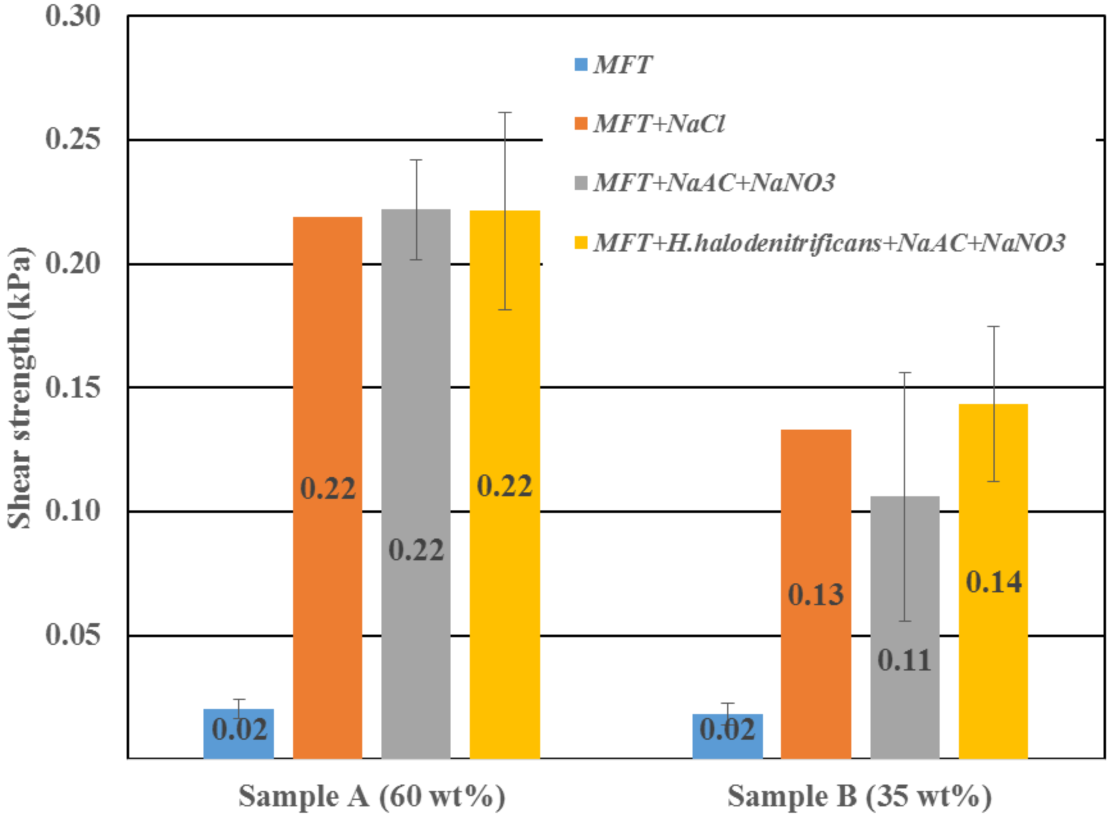

Figure 9. Shear strength of MFT treated for 24 hours with and without denitrification-bacteria. Additives: *H. halodenitrificans* 10^9 cells/mL, sodium chloride 0.6 M, sodium acetate 0.3 M, sodium nitrate 0.3 M.
Error bars represent standard deviations of multiple samples and measurements.

It was noted that the introduction of sodium chloride at the same ionic strength as sodium nitrate and sodium acetate in MFT samples led to the enhancement of MFT shear strength. According to DLVO theory[2], an increase in ionic strength of a solid in a liquid can compress the electrical double layers of the solid particles causing the surface charges of the particles to decrease, encouraging particle aggregation and leading to greater solid shear strength. It can be concluded that ionic strength enhancement was the main contributor to the increase of shear strength in MFT supplemented with sodium nitrate and sodium acetate (with and without external denitrifier *H. halodenitrificans*); and the denitrification process did not lead to the additional consolidation under the conditions studied.

Figure 10 shows that the calcium concentration in MFT samples was not decreased by denitrification-driven MICP, demonstrating that calcium was not precipitated by denitrification-

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[2] See http://en.wikipedia.org/wiki/DLVO_theory

driven MICP. These results help to explain our conclusion that the shear strength of MFT did not increase by denitrification-driven MICP.

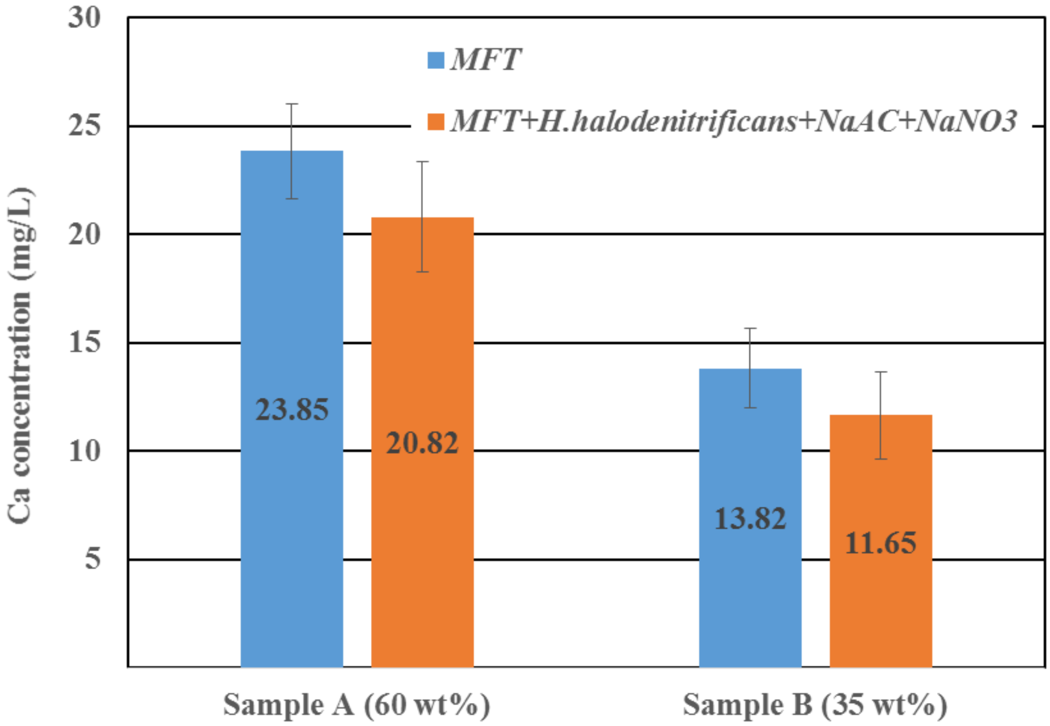

Figure 10. Ca^{2+} concentrations in pore water of MFT treated for 24 hours with and without denitrification-driven MICP.
Additive concentrations: *H. halodenitrificans* 10^9 cells/mL, sodium acetate 0.3 M, sodium nitrate 0.3 M.
Error bars represent standard deviations of multiple samples and measurements.

The failure of denitrification-driven MICP to assist MFT settling and consolidation might be explained by two reasons. First, the reactions involved in denitrification-driven MICP (Equation 7) are slow (compared to the reactions in ureolysis-driven MICP) given that calcium ions in MFT could not be precipitated in 24 hours. Second, either *H. halodenitrificans* could not survive in MFT or the bacteria needed longer than 24 hours to adapt to the chemical environment in MFT.

4.4 Improving the Performance of Ureolysis-Driven MICP on MFT Consolidation

To further evaluate the effect of ureolysis-driven MICP, the shear strength of the MFT samples treated with *S. pasteurii* and urea was measured seven days after the start of the experiment. The shear strength of MFT was observed to be the same 24 hours and seven days after *S. pasteurii* and urea were added to MFT samples.

To increase the MFT shear strength further than the values shown in Figure 6, additional experiments were performed to evaluate options to enhance the ureolysis-driven MICP treatment, i.e., enhanced dosages of *S. pasteurii* and urea were added to the MFT. However, there was no significant improvement in MFT shear strength compared to the shear strength measured for conditions reported in Figure 6.

Since calcium ions were completely removed during the 24 hours of ureolysis-driven MICP treatment, we employed additional calcium ions to MFT samples containing *S. pasteurii* and urea to assist ureolysis-driven MICP. As shown in Figure 11, the calcium chloride addition alone increased the shear strength of samples A and B from ~ 0.02 kPa to ~ 0.24 kPa and ~ 0.16 kPa, respectively. The addition of $CaCl_2$ increased the ionic strength of pore water, enhancing MFT particle attraction. However, the shear strength of the MFT samples treated with $CaCl_2$, *S. pasteurii*, and urea did not show a significant enhancement compared with samples containing *S. pasteurii* and urea. Additional experiments are needed to evaluate the strategies for MFT shear strength improvement with calcium and bacteria addition.

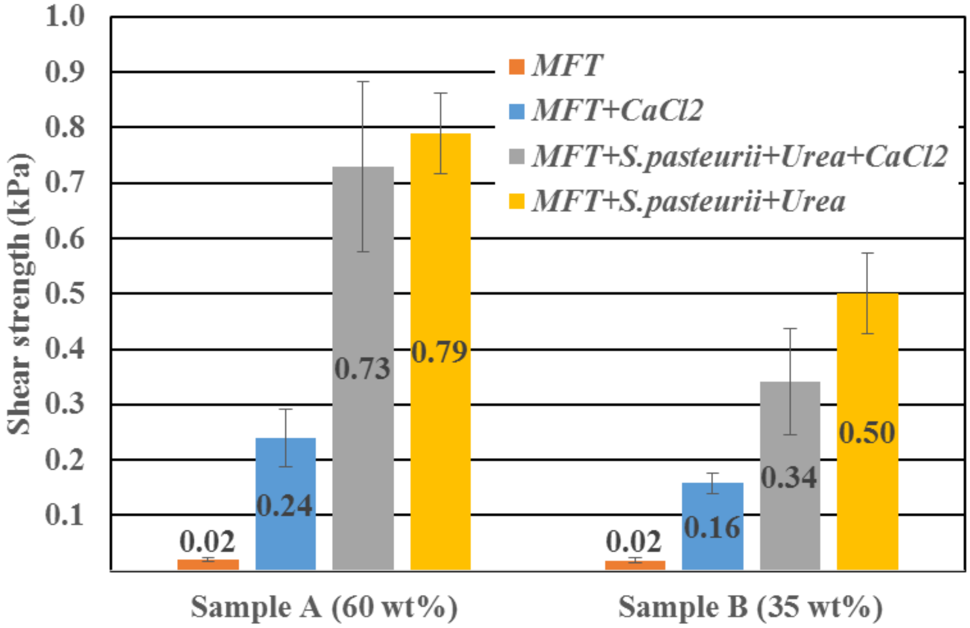

Figure 11. Shear strength of MFT treated for 24 hours with $CaCl_2$, ureolysis-driven MICP, and ureolysis-driven MICP supplemented with $CaCl_2$.
Error bars represent standard deviations of multiple samples and measurements.

Due to the time limitation of this project, we were not able to perform additional optimization of ureolysis-driven MICP for MFT consolidation. However, our results indicate that this process has a potential to enhance MFT strength.

4.5 Effects of Ureolysis-Driven MICP on MFT Particle Size Distribution

Analyses of particle size distribution (PSD) in ureolysis-driven MICP treated MFT were conducted using a standard hydrometer test. The PSD tests followed ASTM D422 (ASTM

2007) for hydrometer analysis. In the PSD test, MFT samples were mixed with sodium hexametaphosphate, a dispersing agent, in water solution in a 1,000 mL cylinder. The falling rate of MFT particles was measured, and back-calculated to the PSD of MFT. PSD tests of untreated MFT and ureolysis-driven MICP treated MFT samples were conducted; the results are shown in Figure 12.

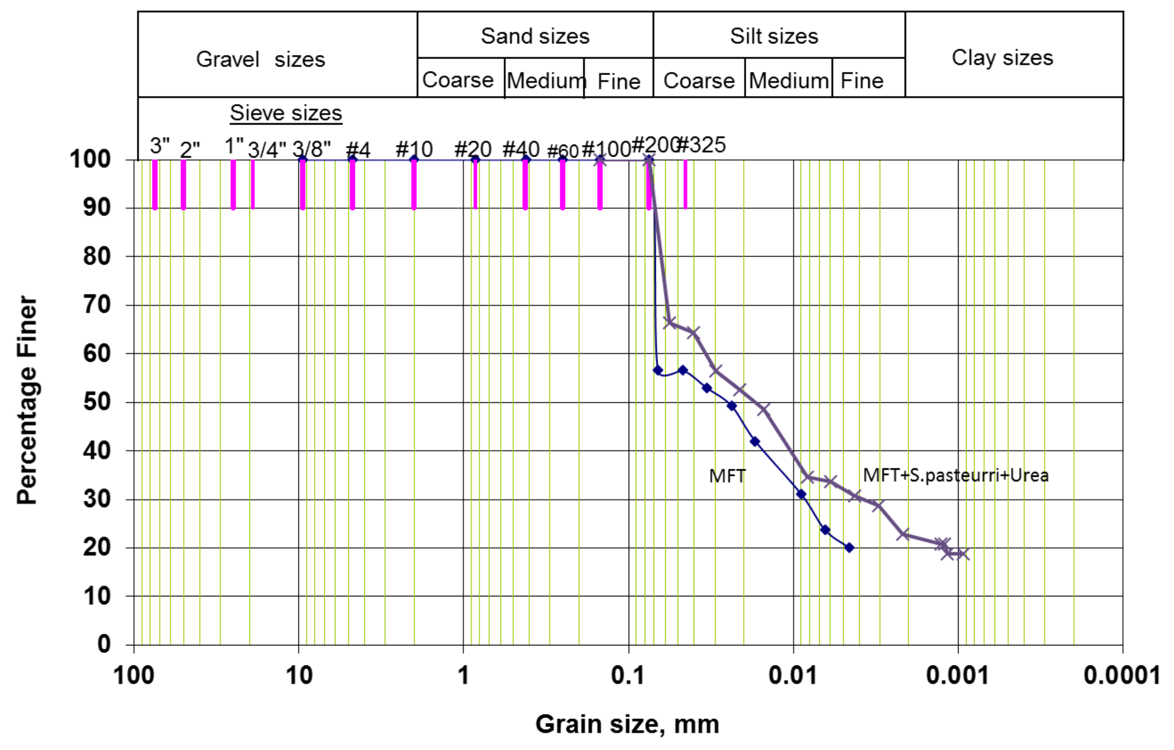

Figure 12. Particle size distribution of untreated and ureolysis-driven MICP treated MFT Sample A (60% solids).

The curves in Figure 12 show that the PSD curve of the treated MFT was shifted to the right of the curve of the untreated MFT. The shift to smaller grain size indicates that the treated MFT has finer particles than the untreated MFT. The change in the PSD curve was not consistent with the expected agglomeration of clay particles due to calcite precipitation. One possible reason is that the added dispersive agent may not be effective in dispersing the untreated MFT due to the water chemistry. Further research could find a more effective dispersive agent or an alternative testing method to determine the PSD of ureolysis-driven MICP treated and untreated MFT samples.

5 CONCLUSIONS

The findings of the experiments performed in this research are summarized below:

- Raw (undiluted) MFT could not be settled by ureolysis-driven MICP. Diluted MFT (Volume $_{\text{DI water}}$: Volume $_{\text{MFT}}$ = 4:1) settling was significantly accelerated by ureolysis.
- Settling of the diluted MFT suspension (Volume $_{\text{DI water}}$: Volume $_{\text{MFT}}$ = 4:1) began at least 5 hours after *S. pasteurii* and urea were added, possibly because *S. pasteurii* needed time to adapt to the MFT environment and/or to produce urease. Calcium ions were not detected in MFT release water 24 hours after combined addition of bacteria and urea, indicating that all calcium had been precipitated.
- No settling was observed within 24 hours in undiluted or diluted MFT treated by denitrification-driven MICP. The calcium concentration in MFT pore water after 24 hours of denitrification-driven MICP did not decrease, indicating that MICP had not occurred.
- Shear strengths of raw MFT samples were significantly increased after ureolysis-driven MICP treatment. The shear strengths increased from ~ 0.02 kPa to ~ 0.79 kPa in Sample A (60% solids content), and from ~ 0.02 kPa to ~ 0.5 kPa in Sample B (35% solids content). Additional calcium, urea, and bacteria did not improve the shear strength of MFT under tested conditions.
- Denitrification-driven MICP did not strengthen MFT under conditions tested in this research.

Directive 074 (Energy Resources Conservation Board 2009) requires oil sands mines to reduce the growth of fluid fine tailings in tailings disposal areas during the life of the mine operations. The Directive further specifies that the fines deposited as a trafficable surface layer must have a minimum undrained shear strength of 10 kPa within 5 years after deposition. In future research, experimental conditions for ureolysis-driven MICP processes should be optimized by optimizing bacterial growth conditions, additives and nutrient levels. Field application strategies for urea and bacterial culture should be evaluated. Options for anaerobic calcite precipitation, in particular processes that utilize endogenous microorganisms, should be tested. The option of combining ureolysis-driven MICP with other consolidation processes should be tested and evaluated. Long-term studies on the impact of MICP-assisted MFT settling and consolidation on land reclamation should be performed.

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7 GLOSSARY AND ACRONYMS

7.1 Glossary

Consolidation

Consolidation is an extreme form of aggregation; it involves a decrease in water content and an increase in solid content of a substance.

Denitrification

Denitrification is the biological conversion of nitrate to nitric oxide or nitrous oxide, and finally to nitrogen gas.

Endogenous

Growing or originating from within an organism.

Flocculant / Flocculation

A flocculant is a substance, often an organic polymer that is used to enhance the aggregation or flocculation of fine particles.

Settling

Settling is the process by which particulates settle to the bottom of a liquid and form sediment.

Shear Strength

Shear strength of a material is the internal resistance per unit area that the material mass can offer to resist failure and sliding along any plane inside it.

Solids Content

The percentage of solids in a liquid mix.

Ureolysis

Ureolysis refers to the hydrolysis of urea.

Viscometer

A viscometer is an instrument used to measure the viscosity of a fluid.

7.2 Acronyms

ANOVA	One-Way Analysis Of Variance
CHWE	Clark Hot Water Extraction
DI	Deionized (water)
DLVO	Derjaguin-Landau-Verwey-Overbeek
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
MFT	Mature Fine Tailings
MICP	Microbial Induced Calcite Precipitation
OSPW	Oil Sands Process-Affected Water
OSRIN	Oil Sands Research Information Network
PSD	Particle Size Distribution
SEE	School of Energy and the Environment
SEM	Scanning Electron Microscopy

7.3 Chemicals and Additives

$Al_2(SO_4)_3 \cdot 14.3H_2O$	Aluminum Sulfate
Al-PAM	Hydrolyzed Polyacrylamide ($Al(OH)_3$)
Ca^{2+}	Calcium
$CaCO_3$	Calcium Carbonate
$CaSO_4 \cdot 2H_2O$	Gypsum

CO_2	Carbon Dioxide
CO_3^{2-}	Carbonate
$CO(NH_2)_2$	Urea
HCO_3^-	Bicarbonate
H_2CO_3	Carbonic acid
HPAM	Partially Hydrolyzed Polyacrylamide
NaAC	Sodium Acetate
NaCl	Sodium Chloride
$NaNO_3$	Sodium Nitrate
NH_4^+	Ammonium
$(NH_4)_2SO_4$	Ammonium Sulphate
OH^-	Hydroxide

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