Evolution of microstructure during desiccation of oil sands mature fine tailings

Évolution de la microstructure en séchage des résidus de sables bitumineux

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ABSTRACT: The coupling between desiccation and consolidation is a process with important implications for the management of soft soils in general and dewatering of fine grained tailings typical of phosphate, bauxite, and oil sands mining in particular. The management of fine tailings can involve the placement of layers that are allowed to desiccate, and then are subsequently consolidated by burial under fresh tailings. While desiccation does densify the material, it also changes both the strength and volume change behaviour of the subsequently consolidated material. This phenomenon is crucial to the oil sands industry, where regulations mandate that tailings achieve a set undrained strength within 1 year after deposition. To understand the interplay of desiccation and consolidation, the evolution of microstructure of oil sand fine tailings are tracked through different drying and consolidation paths using mercury intrusion porosimetry, and non-biased analyses of Environmental Scanning Electron Microscope images. Preliminary results presented in this paper describe the evolution of microstructure in polymer amended tailings during desiccation. The influence of flocculant dose on the microstructure appears to lessen as desiccation progresses, but the final microstructure retains a more open porosity compared to untreated tailings. Résumé

RÉSUMÉ : Le couplage entre la dessiccation et la consolidation est un processus avec des implications importantes pour la gestion des sols mous en général et le séchage des résidus miniers fins typiques du phosphate, de la bauxite, et des sables bitumineux " oil sands" en particulier. La gestion des résidus miniers fins peut comporter le dépôt des couches pour le séchage, et leur consolidation par enterrement sous les résidus frais. Tandis que la dessiccation densifie le matériau , elle change également la force et le comportement mécanique du matériau consolidé. Ce phénomène est crucial à l'industrie de sables d'huile, où les règlements exigent que les résidus atteignent une force non drainée prédéfinie dans un délai de 1 an après dépôt. Pour comprendre l'effet de la dessiccation et de la consolidation, l'évolution de la microstructure des résidus miniers est étudié pour différents chemins de séchage et de consolidation utilisant la la porosimétrie au mercure, et des analyses d'images de microscope à balayage électronique. Les résultats préliminaires présentés dans cet article décrivent l'évolution de la microstructure des résidus modifiés par polymère pendant la dessiccation. L'influence de la dose de floculant sur la microstructure semble diminuer pendant que la progression de la dessiccation, mais la microstructure finale maintient une porosité plus ouverte comparée aux résidus non traités

KEYWORDS: Mature fine tailings, polymer, suction, mercury intrusion porosimetry, SEM, desiccation, microstructure

1 INTRODUCTION

The extraction of oil from oil sands deposit result produces bitumen and tailings. Conventional deposition results in coarse particles (> 74 microns) settling on the beach, while the fine fraction of settles and consolidates extremely slowly, retaining a water content of over 180% after a decade – in this state the tailings are called mature fine tailings (MFT). Due to the extraction process, the clays in the fine fraction are highly dispersed, which results in very low hydraulic conductivity. Because of the volume of MFT produced, impacts include a considerable volume of water is lost to the tailings, and a very large footprint (~200 km^2 for tailings in the FortMcMurray, Alberta area) and dam constructions costs. In order to accelerate restoration and water reclamation form these tailings, the regulator has imposed new rules, that require an increasing inventory of tailings to achieve specific undrained shear strengths at scheduled time after deposition, the first target being 5 kPa after 1 year.

The new regulations have fostered large-scale experimentation with several techniques to dewater and / or strengthen MFT. One technique is to mix an anionic polymer with MFT and re-deposit the amended tailings in relatively thin lifts (Matthews et al. 2011, Wells et al 2011). The mixing is done in the pipeline, only a few metres from the deposition point. This causes aggregation of the clay particles, and results in decreases in water content by settling down to about 100% water content (50% solids), or even greater. To reach the required 5 kPa shear strength, the geotechnical water content must usually be less than 50% water content To achieve this, the material may be deposited in thin lifts to facilitate further dewatering due to evaporation, long-term consolidation, or drainage.. However, the relative contributions of desiccation and consolidation to dewatering are not completely understood, and a better comprehension of the relative effects of each process on subsequent dewatering behaviour could contribute to optimizing the overall dewatering process, especially in terms of required layer thickness, and timing of layer sequencing. This paper presents some preliminary data on the microstructure of polymer amended MFT and how it evolves during desiccation. Data on microstructure is obtained using Mercury Intrusion Porosimetry (MIP) and Scanning Electron Microscopy (SEM).

1.1 Mercury intrusion porosimetry (MIP):

MIP finds a pore-size distribution for pores ranging from 0.01 up to 100 microns – while this pore-size distribution might not be the true PSD due to pore accessibility and sample preparation issues, MIP data is known to exhibit strong correlations to permeability, consolidation characteristics, and water-retention behaviour (Simms and Yanful 2005, 2004, Romero and Simms 2008) – it appears to give a good quantitative "fingerprint" of microstructure. For compacted clays, it is know that volume change measured by MIP samples is very close to macroscopic volume change. However, for wetter or slurried clays, it has been shown that MIP only measures a fraction of the total porosity, despite use of a rapid freeze drying technique to dehydrate the samples (Sassinan 2011).

Further details on the methodology of MIP are available in many other references, such as ASTM 4404-10, Simms and Yanful (2004) and Romero and Simms (2008), and are not repeated for reasons of space. All samples were prepared by

freeze-drying, by first cooling pentane in liquid nitrogen, then submerging the soil samples (cubes less than 5 mm in all dimensions) by using either a small strainer, or a miniature tray for very wet samples (MFT with no polymer) in the pentane for 1 minute. Soil samples are subsequently dried under vacuum for 1 hour, prior to the the actual MIP test. The porosimeter model was AutoPore IV 9500.

1.2 Scanning electron microscopy (SEM):

We employ backscattered scanning electron microscope (SEM) using a rapid freezing stage (-50 degrees C) before application of vacuum (10^{-3} Pa). Grayscale pixel analyses are used to quantitatively compare SEM images.

1.3 Total suction measurement

Total suction measurements used chilled mirror hygrometer(Wenglor WP4PotentiaMeter). Such hygrometers measure the vapour pressure in porous media, by decreasing the temperature in a confined space with the sample, until water condenses on a mirror. Thus the saturated vapour pressure at this controlled temperature is known, which equal to the vapour pressure at the ambient temperature of the sample. The relative humidity is equated to total suction, based on the well-known Kelvin-Laplace equation. The range of this device is theoretically from 0 up to 500 MPa of total suction, but precision is limited to 0.1 MPa. Sample must be extracted from the porous media and placed in a container for insertion in the WP4 device.

1.4 Material and Experimental set up:

MIP and SEM techniques are applied to oil sand mature fine tailings (MFT) amended with different does of polymer, along with measurement of volume change, desaturation, and total suction in shallow columns (0.30 m in initial height, 0.30 m diameter), exposed to potential evaporation rates of ~ 6 mm /day. Columns were kept on scales, and vertical volume change was estimated by a plumb line dropped on 8 different points on each column. The MFT and polymer were supplied by Shell Canada. The specified MFT is originally at 35 - 40% solid contents (gravimetric water of 135 - 145%). The polymer (Flopam DPR 5285) is mixed into diluted MFT using a paint mixer set to 260 rpm, mixing for 30 seconds – this regime is to reproduce similar mixing conditions to in-pipe mixing that occurs during field trials of polymer-amended MFT deposition at Shell's Muskeg River Mine. Three columns have three different doses of poylmer - 700 ppm of the solid contents, 1000 ppm, 1500 ppm.

2 TEST RESULTS AND DISCUSSIONS

2.1 Basics properties of raw MFT

Table 1 presents some basics characteristics of raw MFT

Table 1: Basics characteristics of raw MFT

Parameter	Value	Parameters	Value
Water contents (%)	14158	SFR	0.1
Solid contents (%)	40%	Liquid limit	45
Density (kg/m^3)	1100-1200	Plastic limit	19
Specific gravity	2.64	Liquidity index	3.96

These values are in close agreement to other studies of MFT, such as in Jeeravipoolvarn (2005).

2.2 Dewatering / desiccation behaviour of polmer amended MFT

As shown in Figure 1, evaporation proceeds at the potential rate of ~ 6 mm /day, until about Day 10. This corresponds to an average water content of 50%, or a solids concentration of 67%. At this water content, polymer amended MFT will have a peak undrained shear strengths in excess of 5 kPa (Matthews et al. 2011). For this relatively short layer thickness (0.30 m initial height), the drying is quite uniform with depth. Total suction values near the (~5 mm) surface increase above 1 MPa at this point (10 days), correlating with the onset of Stage II drying (actual evaporation declines significantly compared to potential evaporation). As described in Wilson et al. (1997). evaporation declines as a function of total suction at the soil surface, the decine becoming significant for total suctions in excess of 3 MPa.

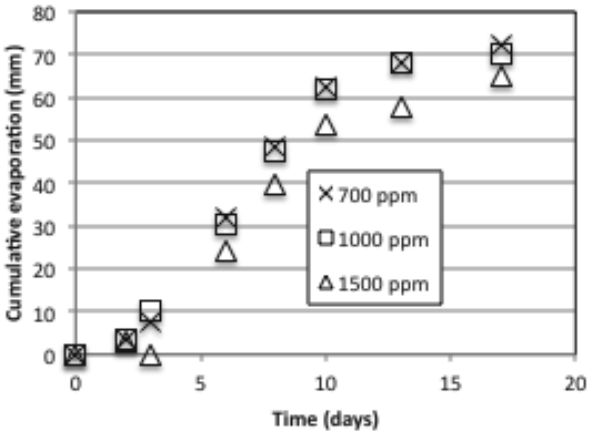

Figure 1: Cumulative evaporation in polymer amended MFT columns

Volume change behaviour is shown in Figure 2 (the shrinkage curve) and in Figure 3 (showing relation between degree of saturation and void ratio). Void ratio and degrees of saturation are based on vertical volume change only. Therefore, the initial degree of saturation are somewhat lower (70% initially) than the true value. This low value is also due to large aggregates formed by the polymer, resulting in some significant macroporosity that drains within the first few hours. Figure 3, however, clearly shows when the air entry value (AEV) occurs and the expected subsequent decreasing rate of volume change. Plotting total suction versus water content data from the same samples, and converting water content to degree of saturation using volume change data, a rough water-retention curve (WRC) can be obtained for the MFT with different does of polymer (Figure 4). Generally, the WRC and the volume change behaviour are very similar between the different treatments, with the exception that

the sample dosed with 1500 ppm of polymer appears to have a higher shrinkage limit. From total suction measurements, the AEV appears to be between 300 and 400 kPa, though this may be biased by osmotic suction.

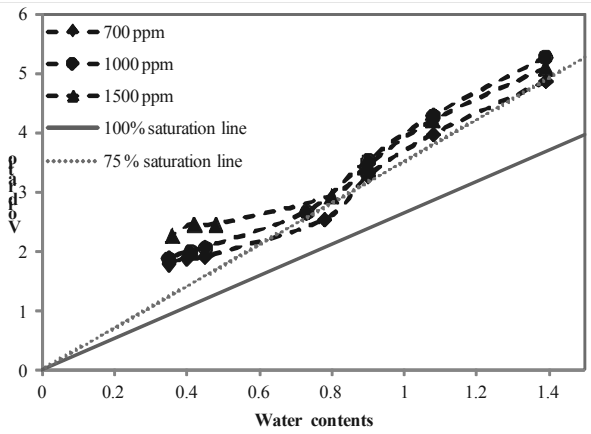

Figure 2: Shrinkage curves for polymer amended MFT

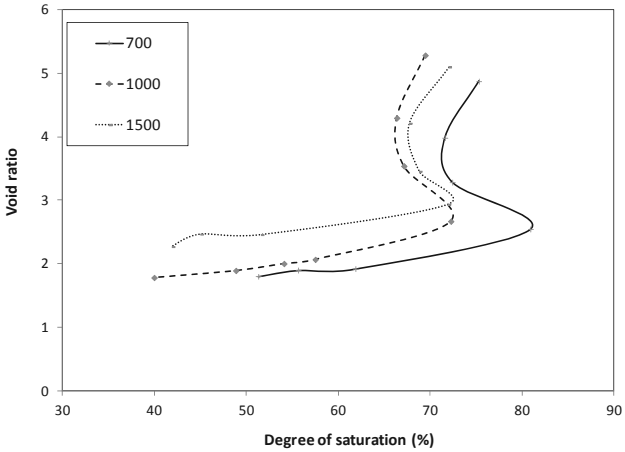

Figure 3: Void ratio and degree of saturation for polymer amended MFT desiccating in columns

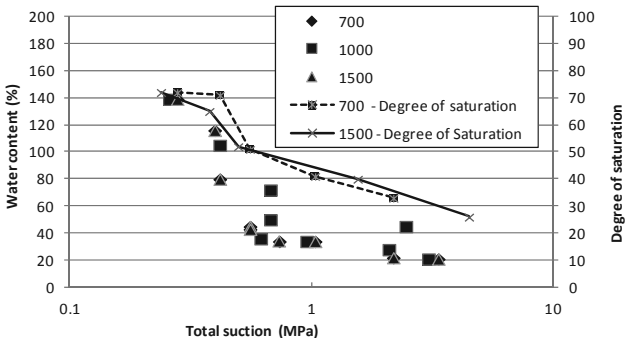

Figure 4: Water-retention curves for polymer amended MFT, from total suction and gravimetric water content analysis on grab samples

2.3 Microstructural analysis of polymer amended MFT

SEM tests were carried out adjusting the magnification of SEM device from 0.1 k V to 5 kV and a high vacuum (10^{-3} Pa) of the vacuum chamber. A cold stage of -50 ^{o}C was applied to all samples for testing. Two samples for each level of polymer dose are shown, for two different water contents of ~100 % and 50% (+/- 5%). Interestingly, while the samples at the higher water content appear to show differences in inter-aggregate porosity, the differences at the lower water content are much less apparent. In the 1500 ppm sample at the higher water content, the shapes of the pores seem more round showing the structure to be more flocculated than that of the other doses.

The appearance of the polymer amended MFT at lower water contents is quite different in comparison to the images for the higher water content samples. At the lower water contents, there is a greater frequency of cracks. However, the difference between samples with different polymer dose is less remarkable than at the higher water content. Grayscale analysis (defining porosity by a range of pixel shade) supports this qualitative judgement.

These results, by themselves, suggest that desiccation does substantially alter the microstructure, similar to other clayey soils (E.G Romero and Simms 2008), and as desiccation progresses the differences in microstructure between samples prepared with different polymer doses become less.

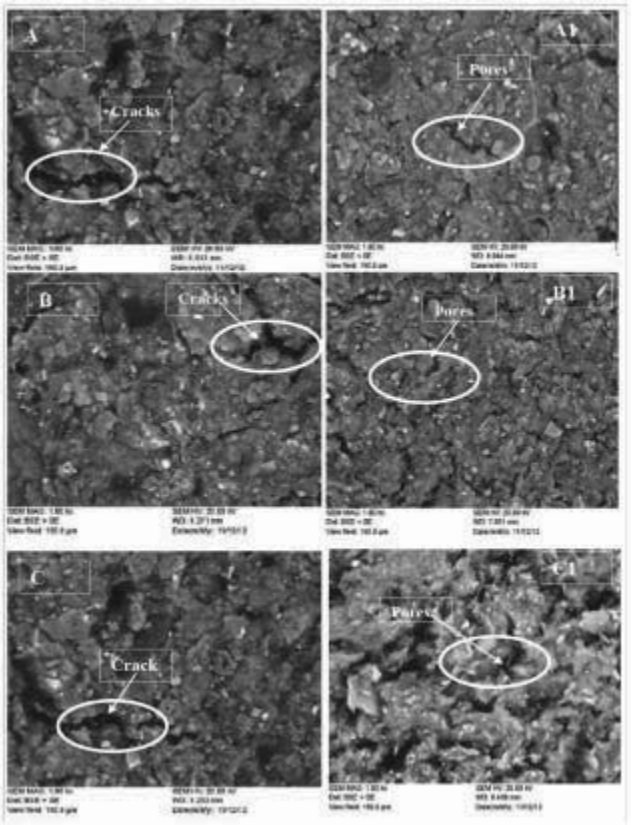

Figure 5: Scanning electron microscopy (SEM) analysis of polymer amended MFT (left; w = 50% for 700, 1000, and 1500 ppm from top to bottom and right; w =103% for 700, 1000, and 1500 from top to bottom. Images are 100 by 100 microns.

Cumulative pore-size distributions (CPSD) from MIP are shown in Figures 6 and 7. Figure 6 shows the CPSD for the three treatments of polymer amended MFT and untreated MFT at the initial water content (140%). As expected, the treated MFT shows much more porosity in the high pore-sizes than untreated MFT. The treated MFT CPSD are very close together, with the 1500 ppm sample showing somewhat larger pore sizes than the other two treatments. Figure 7 shows how the CPSD changes with desiccation, showing the same trend as the SEM images – there is very little difference between the samples desiccated to 100%. This agrees with the relatively small differences in the water retention curve at high suctions – by the time the AEV is reached (about 80 % water content in Figures 2 and 4), the difference in microstructure and relatively small. However, the MIP data does not apparently explain the slightly higher shrinkage limit of the 1500 ppm treatment.

After desiccation to 50% water content, the amended tailings still show a greater porosity in the larger pores, compared to the raw MFT at 140% in Figure 6.

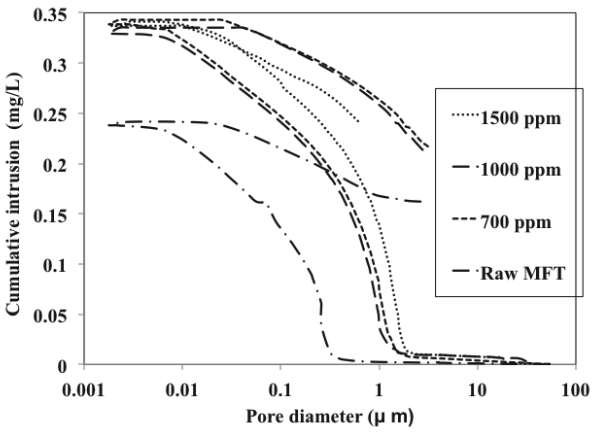

Figure 6 Mercury intrusion porosimetry test results, showing cumulative pore-size distributions (intrusion and extrusion curves) for amended MFT for a water contents of 140% for 700, 1000, 1500 ppm and raw MFT.

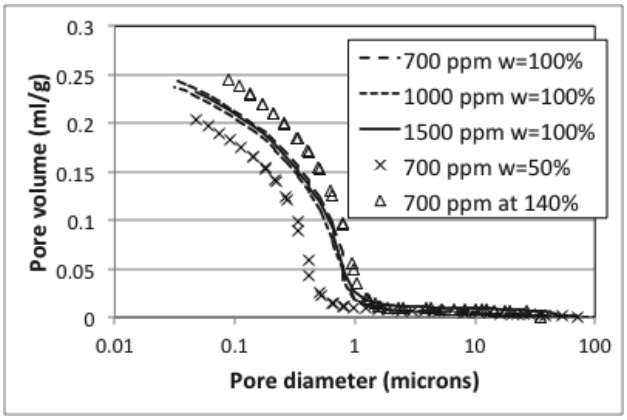

Figure 7 Cumulative pore-size distributions at different degrees of desiccation

3 SUMMARY CONCLUSIONS

Microstructure of polymer amended MFT is studied using MIP and SEM techniques. Both techniques show that the pore-size distributions of the different treatments (polymer doses of 700, 1000, and 1500 ppm) converge with increasing desiccation. This correlates with the very similar water-retention curves of the different treatments, though not with the slightly higher shrinkage limit (58% to 50%) of the 1500 ppm sample. Based on this analyses, it appears that the desiccation behaviour, beyond the initial water release during settling, is relatively insensitive to the range of polymer applied in this study. Ongoing work is examining the consolidation behaviour of MFT treated with different levels of polymer subsequent to different degrees of desiccation. This information is hoped to assist in optimization of this type of technology for tailings operations in the oil sands.

4 ACKNOWLEDGEMENTS

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