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# STRUCTURATION IN POLYMER AMENDED OIL SANDS FINE TAILINGS

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ABSTRACT
Although void ratio-effective stress relationship and hydraulic conductivity-void ratio relationship are the two most important properties that govern the consolidation behaviour of oil sands tailings in tailings ponds, the phenomena of creep and thixotropy in tailings also could affect its compression behaviour significantly. Reporting on ongoing experimental investigations of creep/thixotropic behaviour of polymer amended fluid fine tailings, this paper presents findings from a new batch of experiments conducted using different polymer types and doses, different boundary conditions, and different scales of tests. The experiments included column dewatering tests with pore water pressure measurements, advanced rheology tests, oedometer tests, zeta potential, mercury intrusion porosimetry, and low vacuum SEM and optical microscopy. The tailings exhibited structuration (change in fabric and the associated increase in apparent pre-consolidation pressure) seen in some natural clays.

RÉSUMÉ
Bien que les relations entre volume des vides et contrainte effective ainsi que conductivité hydraulique et volume des vides sont les deux propriétés principales régissant le comportement de consolidation dans les bassins de résidus de sables bitumineux, les phénomènes de fluage et de thixotropie peuvent affecter la résistance en compression significativement. Cet article présente les résultats d 'un nouveau lot d’expériences menées en utilisant différents types et dosages de polymers ainsi que différentes conditions aux limites et différentes échelles d'essais. Les expériences comprennent des essais de déshydratation en colonne avec des mesures de pression interstitielle, des tests de rhéologie avancés, des tests d'oedomètre, la mesure du potentiel zêta, de la porosimétrie par intrusion de mercure, et une analyse par microscopie optique et SEM sous vide. Les résidus présentent un comportement de structuration (modification du tissu associé à une augmentation de la pression apparente de pré-consolidation), ce qui est observé dans certaines argiles naturelles. Les implications de ces phénomènes sur le potentiel d'assèchement des bassins de résidus de sables bitumineux sont discutées.

## 1 INTRODUCTION

Following the mining of oil sands ores and the thermal-chemical-hydraulic separation process of bitumen extraction from the ores, the by-product materials or tailings, which is a mixture of water, sands, fine clays, silts, and residual hydrocarbons, are hydraulically transported from the extraction plant to the dedicated disposal areas or dammed impoundment structures with substantial footprints called tailings ponds (Oil Sands Discovery Centre, 2009). Over the past few decades, the rapid development of oil sands projects in Alberta, and subsequently, a continuous accumulation of fluid fine tailings (FFT) has resulted in the formation of very large deposits of soft tailings material. These tailings ponds now cover more than 250 square kilometers and hold an estimated 1.287 trillion liters of material (Orland, 2018).

To ensure that the environmental impacts of oil sands production are being addressed adequately, current regulations for tailings management as described under the ‘Directive 085: Fluid Tailings Management for Oil Sands Mining Projects’ require the oil sands operators to develop a reclamation plan that spans the life of the project. The goal is to reclaim all the lands affected by the mining operations within 10 years of the end-of-mine life (Alberta Energy Regulator, 2017; Canadian Association of Petroleum Producers, 2017).

Natural dewatering mechanisms such as sedimentation and self-weight consolidation work relatively slowly in FFT. Although the clay-rich fluid fine tailings settle to a solids content of 30% to 35% within a decade of deposition, they do not settle or consolidate significantly thereafter, even for significant dam heights (~80 m), and even after 50 years (Sobkowicz, 2013; Beier et al., 2013).

To make the settling process faster, the oil sands industry is treating the tailings using a variety of methods, often in combination. Some of the improved tailings treatment technologies, such as in-line flocculation, tank thickening, and centrifugation technique, which use polymers to promote flocculation for faster dewatering, have shown considerable success during the field trials. Polymers have been found to allow such technologies to increase solids concentration to at least 50% in short-term, usually within days (Matthews et al., 2011; Wells, 2011). Although attaining a solids concentration of 50% within such a short time is a good progress, this 50% solids concentration is equivalent to a gravimetric water content of 100%, which is still well above the liquid limit of the material. In fact, to meet regulatory standards and to develop a deposit strong and safe enough for reclamation, it is thought that a solids concentration of at least 70% or a gravimetric water content of 43% is required, which is close to the plastic limit of the material (McKenna et al., 2016). Understanding of longer-term dewatering processes such as consolidation and potentially others such as creep bear on optimization of tailings deposition plans to take the tailings to this state.

The consolidation of oil sands tailings involves the slow settlement of the fine-grained tailings material by the release of excess pore water pressure and the development of effective stress over time in response to the self-weight or vertical surcharge from a capping layer (BGC Engineering Inc., 2010). It is a very long and complex process, which is influenced by many natural and external factors, such as compressibility, permeability, etc. Generally, void ratio-effective stress relationship and hydraulic conductivity-void ratio relationship are the two most important properties that govern the consolidation characteristics of fluid fine tailings. However, there are other important factors such as creep and thixotropy. The creep and thixotropic behaviour have been found to affect the tailings consolidation behaviour as they affect both void ratio-effective stress relationship and hydraulic conductivity-void ratio relationship (Jeeravipoolvarn, 2005; Jeeravipoolvarn et al., 2009; Miller, 2010).

Creep behaviour is different from consolidation in that creep refers to any change that occurs in volume over time and is independent of the effective stress changes. Although creep behaviour is prominent during the secondary compression, creep can also take place during the primary consolidation and contribute to void ratio changes (Jeeravipoolvarn, 2005; Jeeravipoolvarn et al., 2009). Creep behaviour in tailings was observed in the lab-scale experiments when they were conducted over a larger time-scale. For example, in the case of 10-meter tall standpipe tests conducted at the University of Alberta, after a period of 20.6 years, relatively little effective stress development took place with almost no dissipation of excess pore water pressure. However, there was a reduction in void ratio throughout most of the depth of the standpipe with a significant amount of settlement taking place at a uniform rate over time. Such observation supports the idea that creep compression substantially contributed to settlement (Jeeravipoolvarn, 2005; Jeeravipoolvarn et al., 2009). Creep can be explained in terms of viscous deformation that manifests as a time-dependent compressibility curve, and several models in the literature quantitatively reproduce this effect (e.g. Yin and Graham 1994).

Thixotropy is the time-dependent reorganization of clay particles due to electrochemical forces (Mitchell, 1960). Thixotropy is most clearly manifested in the time-dependent strength recovery of clays after remolding at constant density - this has been measured numerous times in oil sands tailings (Jeeravipoolvam, 2005; Jeeravipoolvarn et al., 2009). Similarly, there is evidence to suggest that thixotropy also affects the compression behaviour in FFT, and the rearrangement of the particles leads to a density-independent increase in pre-consolidation pressure, which results in less volume change during consolidation (Jeeravipoolvarn et al., 2009; Miller, 2010).

The creep and thixotropic behaviour demonstrated by the tailings material have also been observed in many natural clays. These clays exhibit a time-dependent behaviour called ‘structuration’, which is typically manifested in a change in microstructure and an associated increase in apparent pre-consolidation pressure. Subsequently, the clays show reduced compressibility, higher yield strength, and higher stiffness (therefore, an increased resistance to the compression) (Locat & Lefebvre, 1986; Delage, 2010). Processes other than consolidation or compaction, such as cementation, delayed compression (creep), and ageing effects (thixotropy) have been found responsible for such changes and the subsequent development of strength and stiffness in fine-grained soils (Locat & Lefebvre, 1986; Burland, 1990).

This paper reports on the creep and ‘structuration’ effects observed in polymer amended fluid fine tailings. Long-term column dewatering tests with pore water pressure measurements were combined with advanced rheological tests, oedometer tests, zeta potential measurements, mercury intrusion porosimetry, low/high vacuum SEM, and optical microscopy to characterize both consolidation and non-consolidation volume change behaviour under the saturated conditions. Based on the findings, the implications of non-consolidation behaviour for the dewatering and consolidation performance of tailings deposits in the longer term are discussed.

## 2 MATERIALS

### 2.1 Oil Sands Tailings

Oil sands tailings samples were collected from a tailings pond in Northern Alberta, Canada, and shipped to Carleton University in Ottawa, Canada. Different laboratory tests and analyses were performed to determine the physical, mineralogical, and chemical characteristics of the raw fluid fine tailings. The initial solids content was 31% and the liquid limit was 60%. The sands to fine ratio (SFR) was 0.25. The clay content obtained from the Methylene Blue Index (MBI) analysis ranged from 28% to 32%. According to the X-ray diffraction (XRD) results, the composition of the clay fraction was 68-72%% Kaolinite and 28-32% Illite. Total Dissolved Solids (TDS) in the pore water collected from the raw fluid fine tailings was 1050 mg/L, electrical conductivity was 1590 micro-S/cm, while the dominant cations were sodium at 340 mg/L. Typical material characteristics of the tailings used are listed in Table 1.

Table 1: Physical properties of the raw fluid fine tailings

| Parameters | Average value |
|---|---|
| Initial solids content (%) | 31 |
| Initial water content (%) | 220 |
| Hydrocarbons content (%) | 1.4 |
| Initial wet density (g/cm$^3$) | 1.20 |
| Initial void ratio | 5.1 |
| Specific gravity | 2.12 |
| Liquid limit (%) | 60.0 |
| Plastic limit (%) | 27.0 |
| Plasticity index (%) | 33.0 |

## 2.2 Polymer stock solution

Polymer A3338 (SNF), an anionic polyamide based flocculent, was used to prepare the polymer amended oil sands tailings samples. In a plastic weighing dish, 4 g of A3338 polymer (for the preparation of 0.4% polymer stock solution) was weighed using an analytical balance (Fisher Scientific, Sartorius AG Germany, LE225D) and decanted into a 1500 mL glass beaker and completed to 1000 mL with deionized water. The polymer solutions were stirred using a jar tester (Phipps and Bird, USA) at 200 rpm for 5 minutes and at 125 rpm for the following 55 minutes. Then the polymer solution was mixed with a hand blender for 10 seconds and left for maturation for 1 hour.

To prepare the polymer amended tailings, a pre-determined volume of the polymer solution was mixed with the fluid fine tailings in a 10-liter pail using a mixer set at 250 rpm and the mixer ran for 20 seconds. Both the mixing speed and the duration were kept at optimal points for dewatering, which were determined based on the findings from a combination of CST tests and settlement tests.

# 3 METHODS

10 cm tall column experiments were conducted using two (2) different boundary conditions: Single-drainage and double-drainage (Figure 1). The short height was chosen to maximize the influence of creep. Typically, a short column height leads to the development of a low effective stress in the deposited tailings material, and the creep is thought to be one of the main mechanisms behind the deformations that occur at lower effective stress (Miller, 2010).

In single-drainage column experiment, a single lift of polymer amended fluid fine tailings (dosed with 800 ppm anionic polymer) was deposited in two 15 cm diameter transparent plastic acrylic columns with the material reaching a height of 10 cm inside the columns. Two (2) tensiometers (model T5 from UMS) were installed into one of the columns (at an elevation of 1.5 cm and 4.5 cm respectively) to monitor both positive and negative pore water pressures. The columns were kept covered at the top with the help of a tight lid. The space between the lid and the surface of the deposited tailings was closed to prevent the escape of water vapor from the surface.

Like the single-drainage column, a single lift of polymer amended fluid fine tailings (dosed with 800 ppm anionic polymer) was deposited in two 15 cm diameter transparent plastic acrylic columns in the case of double-drainage column experiment. One of the columns was equipped with two (2) tensiometers at the same elevations as the single-drainage column (1.5 cm and 4.5 cm respectively). The only difference between the single-drainage and double-drainage columns is that the non-porous bottom in single-drainage columns didn't allow for any drainage, whereas, the tailings in double-drainage columns were rested on a filter paper that allowed for drainage.

Before the experiments commenced, the capillary suction time (CST) tests were performed to explore the dose-response relationship between the polymer dose and the clay content of the tailings material (Figure 2). CST test is a type of static filtration test that measures the filtration rate using a filter paper as the medium and thus quantifies the dewatering properties. When the CST tests are performed on the samples mixed with different doses of a chemical conditioning agent, the lowest value of CST usually refers to an optimum filterability and dewaterability (Jin et al., 2004). Hence, CST tests are helpful in selecting the polymer dose required for optimizing the dewatering and settling performance of the polymer amended tailings. The results from the CST tests showed that an 800-ppm dose of the polymer performs better than the dose of 600-ppm in terms of dewaterability.

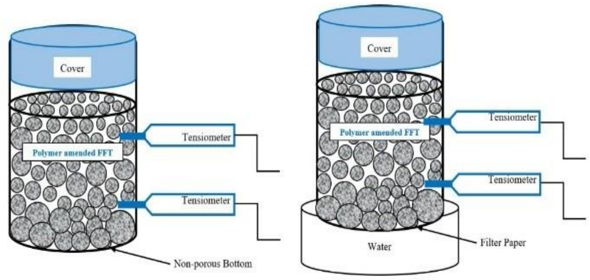


Figure 1: A schematic diagram of the (left) single-drainage and (right) double-drainage column experiments.

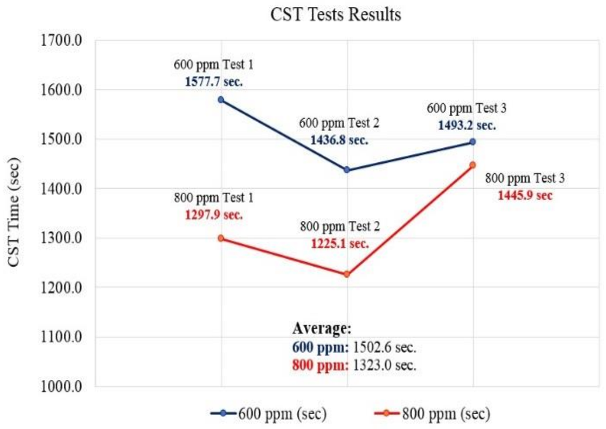


Figure 2. Results from the CST tests conducted on fluid fine tailings mixed with two different polymer doses.

In both types of drainage system, a set of replicate columns was used for measuring the water content at different depths, and the changes in water-solids interface height and void ratio, and for providing samples for other tests and analyses.

Oedometer tests were performed on the samples collected from the replicate columns to determine their void ratio-effective stress relationships and to track the changes in compressibility with time. Tests were conducted on both intact and remolded samples for each sampling period. As it was observed in the case of natural sedimentary clays (Burland, 1990; Delage, 2010), a comparison between the compressibility of the intact and the remolded samples could help in determining the influence of changes in tailings microstructure (arrangement of particles and interparticle bonding) on its compressibility and strength characteristics. A fall cone device was used for measuring the changes in stiffness of the tailings samples based on the measurements of cone penetration.

Advanced rheological tests were performed to examine the mechanical response of the samples to the small-strain shear applied. The changes in viscoelastic properties were tracked using the oscillatory rheometry. First, the sample was placed in a cylindrical sample holder in the rheometer (an Anton Paar Physica MCR301 model rheometer with a vane fixture). The vane fixture was then lowered into the sample, and oscillated at a constant frequency, but at increasing maximum values of stress. This allowed for the determination of the elastic modulus, the linear elastic region, the viscous modulus, and the yield stress (past which viscous behaviour begins to dominate the mechanical response of the material). The elastic modulus is strongly dependent on the tailings fabric (Mizani et al., 2017). A combination of tests was used to measure the recovery of the elastic modulus after shearing. Such thixotropic recovery or strength gain with time is typically prompted by the regeneration of flocs after shearing and manifested in the aging phenomena that occur in tailings at short time-scales and influences the rheological behaviour of tailings in a pipeline or during deposition (Mizani et al., 2017).

For both single-drainage and double-drainage, the scanning electron microscopy (SEM) images were generated using the samples collected from replicate columns. A Tescan Vega-II XMU SEM device was used for the image analysis. Using the cryo-techniques, the samples were frozen at temperatures less than 173 K and then imaged under low vacuum pressure (50 Pa). SEM images helped to observe the changes in size, shape, and arrangement of the flocs with time as dewatering progressed.

Immediately after the mixing of polymer, optical microscopy was performed on the samples for tracking the floc development. The technique of optical microscopy could only be used for the relatively wet samples, as beyond a few days the light source could not penetrate the densifying tailings. Tailings were carefully sampled from the surface of replicate columns using a syringe and placed onto the slides using the same syringe. The slides were then imaged using a Nikon Eclipse Ti Optical microscope, at a magnification scale of 200x. The microscope uses backlighting (lighting on the opposite side of the sample from the camera). Zeta potentials of the tailings bleed water were measured during the first 48 hours of mixing to determine the colloidal stability or the status of electrochemical attractions between the charged fine clay particles.

The mercury intrusion porosimetry (MIP) technique was applied to the freeze-dried samples for the pore size distribution measurements. Compressed samples collected from the oedometer tests were also freeze-dried and tested with the MIP technique. The MIP and SEM observation on these freeze-dried samples could help a detailed investigation of the changes in tailings microstructure during consolidation (Delage, 2010).

In addition to the 15 cm diameter transparent plastic acrylic columns, a large transparent plastic column (50 cm X 22 cm X 22 cm) was used for the dewatering test. Fluid fine tailings mixed with 800 ppm anionic polymer was deposited in the column. The column was equipped with three (3) T5 tensiometers at three different elevations (5 cm, 15 cm, and 25 cm respectively) for the measurements of pore water pressure and three (3) 5TE sensors (from Decagon) at three different elevations (5 cm, 15 cm, and 25 cm respectively) for the measurements of volumetric water content, temperature, and electrical conductivity. The test was conducted to see whether the phenomena of creep and thixotropy could be observed when the test is conducted on a larger scale using a greater lift height.

## 4 RESULTS AND DISCUSSIONS

### 4.1 Pore water pressure and water content

In the case of the single-drainage column, the pore water pressure declined rapidly after the deposition took place. Afterward, the pore water pressure became steady and remained at the almost same values till Day 33. Then, the pore water pressure started to decline, but at a negligible rate (Figure 3). To the contrary, the tailings underwent some volume change throughout the experiment with the gravimetric water content declining from 245% to 150% (Figure 4).

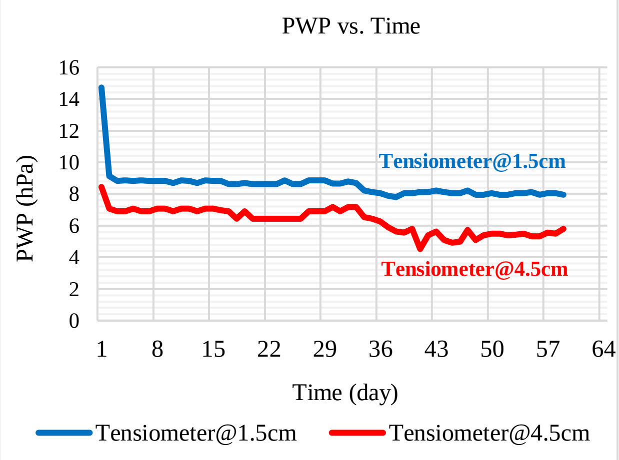


Figure 3. Pore water pressure (in hPa) at 1.5 cm and 4.5 cm from the base of the column (single-drainage column).

Water content was measured at an interval of 1 cm throughout the depth of solids that have undergone sedimentation or consolidation. Figure 4 below shows the water contents measured at the top, bottom, and middle.

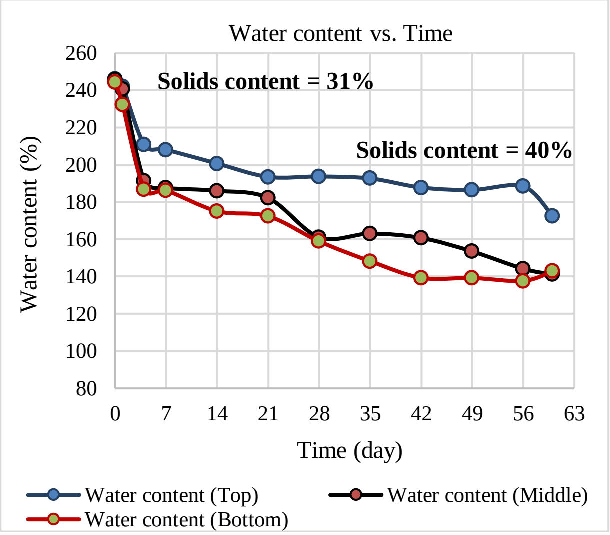


Figure 4. Water contents measured at different depths, from the replicate samples (single-drainage columns).

In double-drainage column experiment, the pore water pressure declined rapidly immediately after the deposition; afterward, it remained at the almost same values till Day 14 and then started to decline gradually (Figure 5). However, the dewatering and the volume change experienced by the tailings throughout the experiment was more substantial as well as more uniform. After 60 days, the water content declined to 100% from the initial water content of 245% (Figure 6).

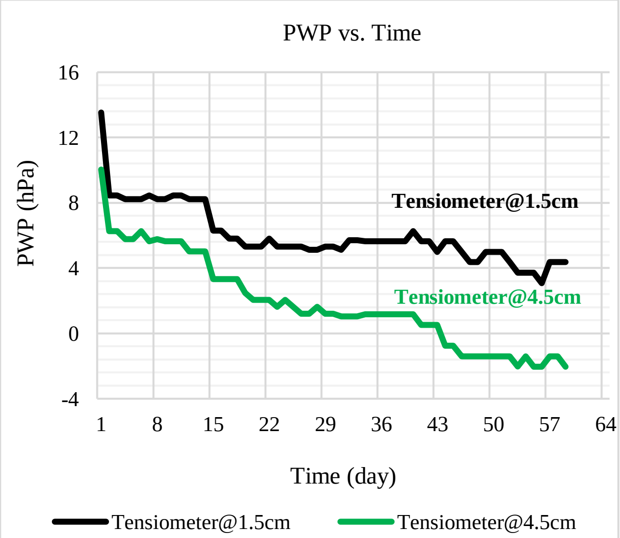


Figure 5. Pore water pressure (in hPa) at 1.5 cm and 4.5 cm from the base of the column (double-drainage column).

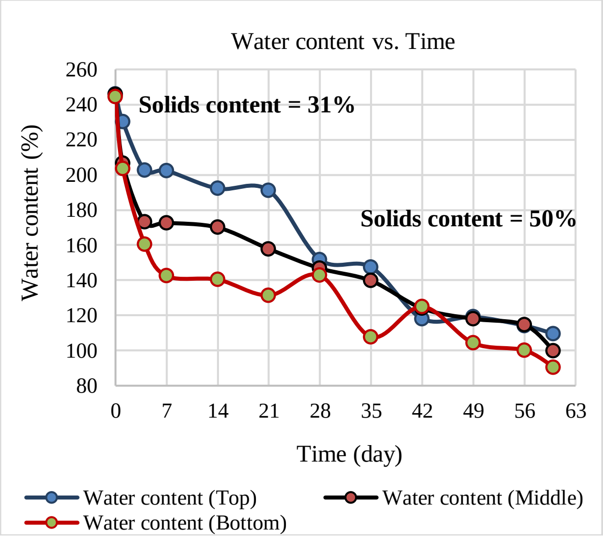


Figure 6. Water contents measured at different depths, from the replicate samples (double-drainage columns).

Figure 6 shows the water content measured at the top, bottom, and middle of the solids settled. The decline in pore water pressure was more rapid with the tensiometer installed at 4.5 cm height. After Day 44, the pore water pressure started showing negative values indicating that the tailings became unsaturated at the top.

In both types of drainage, it was anticipated that the decline in gravimetric water content would stop or become negligible with the pore water pressure dissipation becoming negligible. However, it was not the case in these experiments. Especially, in the case of the double-drainage experiment, the substantial reduction in gravimetric water content was quite a remarkable observation indicating that some processes or mechanisms other than consolidation such as creep contributed to this volume change behaviour.

When comparing the rates of dewatering, the magnitude of both initial consolidation and the subsequent creep compression in single-drainage columns was not so much as in the double-drainage columns. Such difference could be attributed to the different boundary conditions. In longer-term, the double-drainage columns showed rapid and greater settlement in comparison to the single-drainage columns. Although the effective stress in the double-drainage samples was slightly larger (1.2 kPa at the bottom in double-drainage column compared to 0.2 kPa in single drainage) due to the imposed boundary conditions, samples were subjected to the same effective stress, different boundary conditions resulted in different rates and magnitudes of settlement.

## 4.2 Oedometer tests

In the single-drainage experiment, 56-day and 70-day samples were used for the oedometer tests, whereas, in the double-drainage experiment, oedometer tests were performed on the 28-day and 42-day samples. It was difficult to obtain competent samples before 28 days. The compressibility curves for the 28-day and 42-day samples are shown in Figure 7. The 42-day sample was found considerably stiffer in comparison to the 28-day sample. With time, because of the structuration effect, there was an increase in apparent pre-consolidation pressure and a reduction in apparent compression index, which contributed to such stiffness.

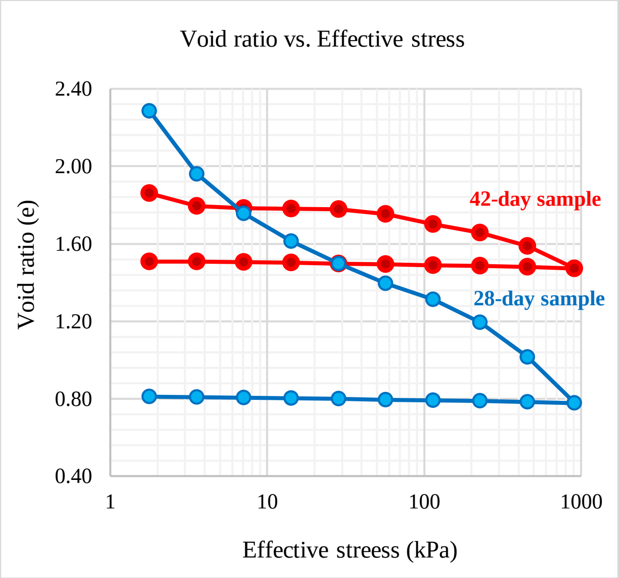
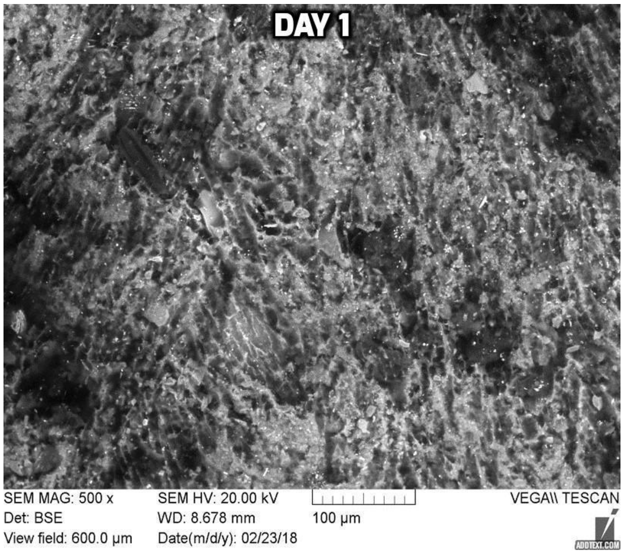
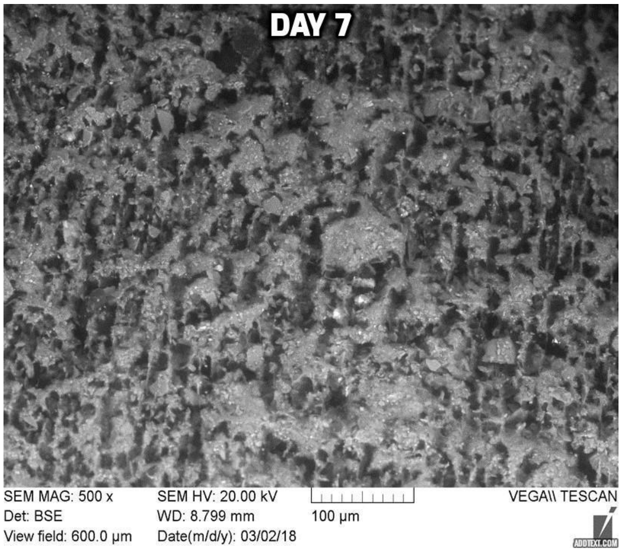


Figure 7. Compressibility curves from the double-drainage column experiment (from 28- and 42-day samples).

## 4.3 Scanning electron microscopy (SEM)

SEM images from the single- and double-drainage column experiments are shown in Figure 8 and 9 respectively.





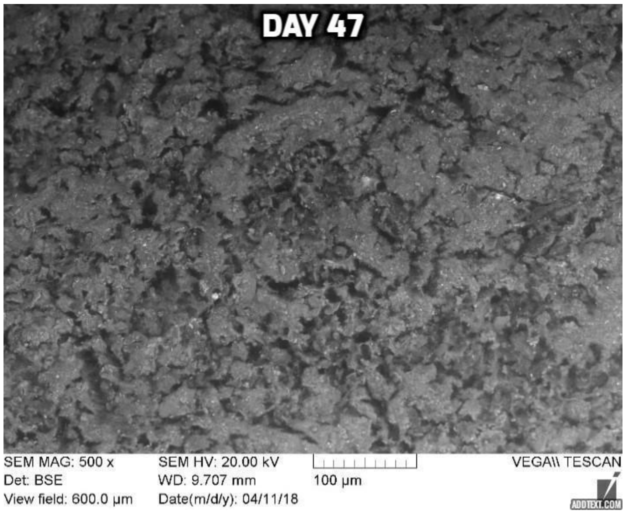
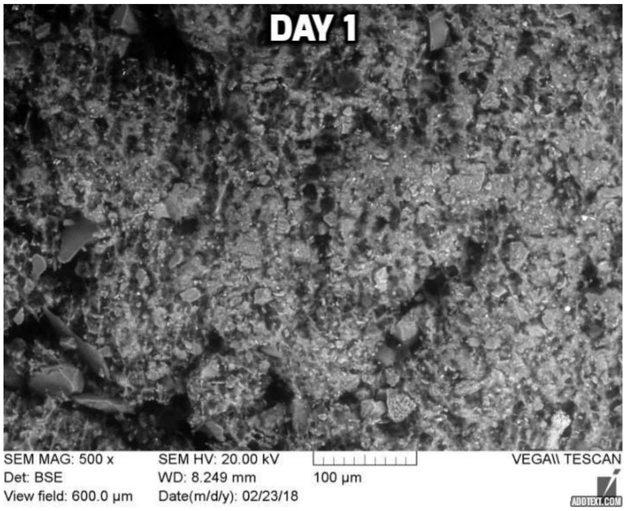


Figure 8. SEM images from the 1, 7, and 47-day single-drainage samples (500X magnified).



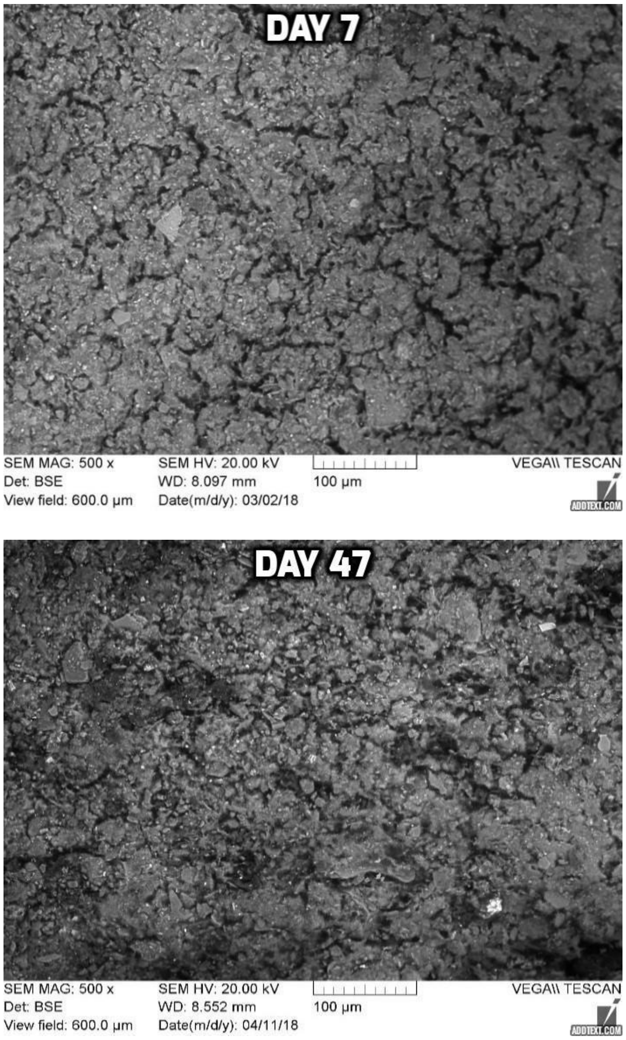


Figure 9. SEM images from the 1, 7, and 47-day double-drainage samples (500X magnified).

In both cases, the sample became relatively less porous and more homogenous as the flocs appeared to grow or coalesce with time. However, the changes in double-drainage samples were more pronounced than in the single-drainage samples.

### 4.4 Oscillatory rheometry

Figure 10, 11, and 12 illustrate the results of the oscillatory rheometry tests performed on the replicate column samples. The x-axis (stress in Pa) denotes the maximum stress during a given oscillation. The maximum stress was successively increased with each oscillation. The resulting deformation in tailings materials was estimated and expressed in terms of the elastic modulus (G') and viscous modulus (G"). G' was calculated from the recoverable strain during each oscillation, while G" was calculated from the measured irrecoverable strain.

The results from the oscillatory rheometry tests demonstrated the changes in the linear elastic range in response to the stresses applied and correlates the changes in material structure and behaviour to these changes in linear elastic range (since the elastic modulus or G' is strongly dependent on fabric). The region where the modulus of elasticity is relatively constant represents the true elastic range of the tailings material and the point where G' reduces and G" becomes dominant represents the yield stress.

In the single-drainage column (Figure 10 and 11), after 42 days, the increase in the linear elastic region was not very prominent. However, with time, as the samples became older, the single-drainage samples showed a considerable increase in their elastic modulus (G') under the same stress applied (increased to 1000 Pa from about 270 Pa). During the same period, yield stress was also increased significantly (from about 150 Pa to 300 Pa).

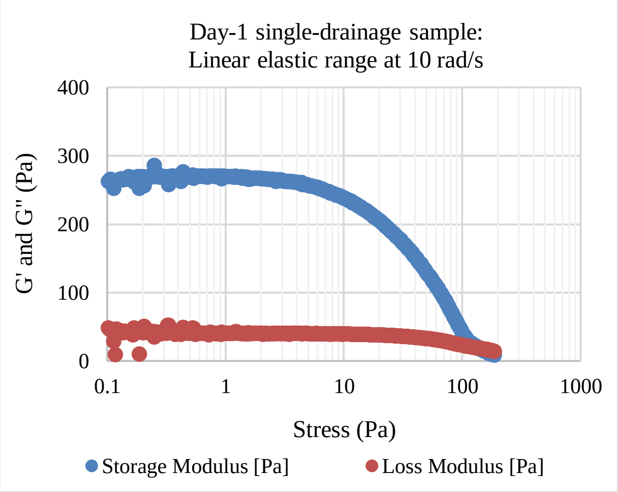


Figure 10. The linear elastic region at 10 rad/s (single-drainage 1-day sample).

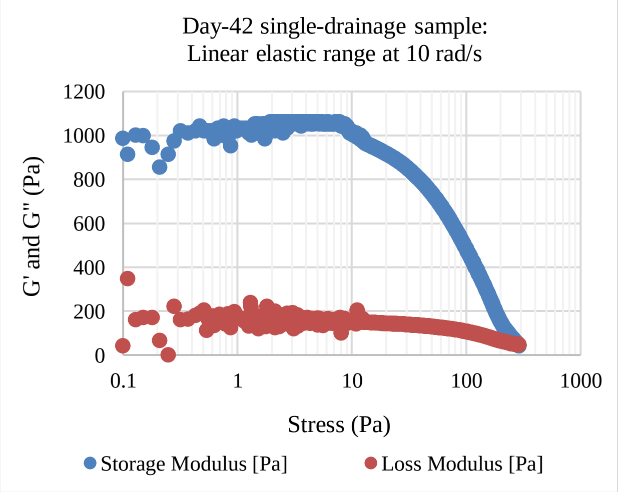


Figure 11. The linear elastic region at 10 rad/s (single-drainage 42-day sample).

A combination of Amplitude sweep tests and Stress growth tests was used to measure the recovery of elastic modulus after the sample had been sheared. In the case of 1-day double-drainage sample, G' was recovered by almost 54% immediately after the shearing, 65% after a resting period of 30 min and 71% after a resting period 60 min. In the case of the 56-day double-drainage sample, G' was recovered by 65%, 89%, and 90% respectively (Figure 12). Therefore, with time, the percentage of recovery increased. Such thixotropic recovery or strength gain with time was an indication of the aging phenomena in tailings.

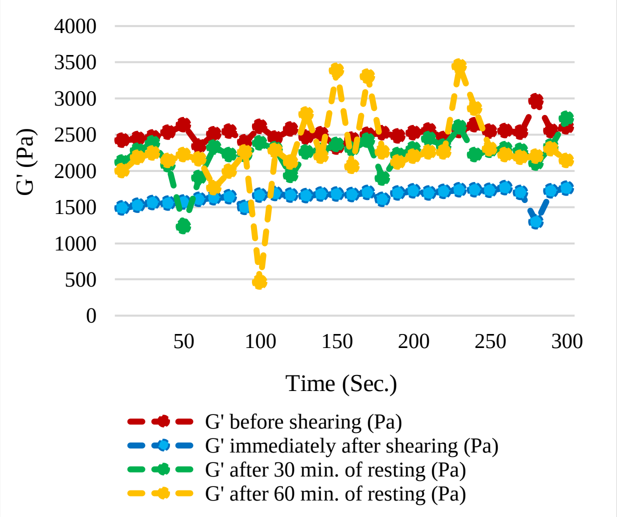
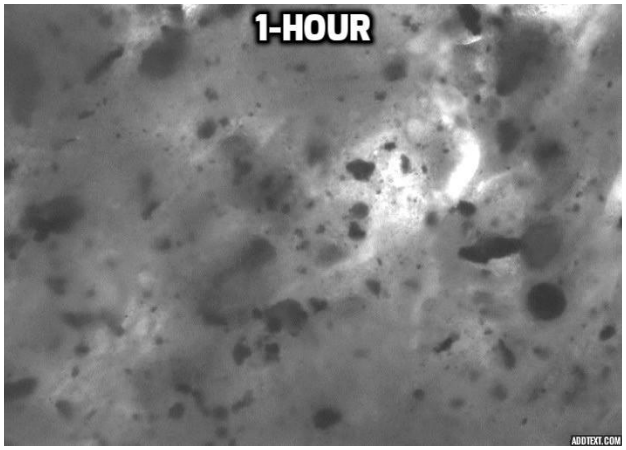
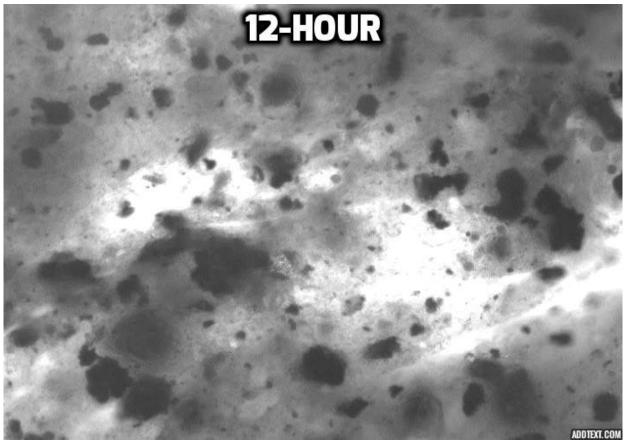


Figure 12. Recovery of G' after shearing (56-day double-drainage sample).

## 4.5 Optical microscopy

In Figure 13, optical microscope images are shown for the polymer amended tailings after 1 hour, 12 hours, and 48 hours of sample preparation. A clear trend can be seen in terms of floc or aggregate size growth.





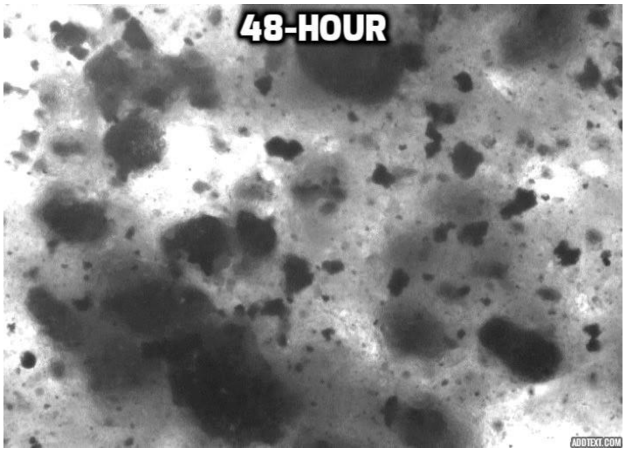


Figure 13. Images from the optical microscopy (Each image is 650 microns wide).

## 5. SUMMARY

The tests result demonstrated that there was considerable deformation after the initial sedimentation and primary consolidation. The solids continued to settle at a uniform rate in both single-drainage and double-drainage columns and the solids content increased to 40% and 50% respectively from an initial solids content of 31%. Also, the tailings showed a ‘structuration’ behaviour which was evident in the changes that took place with time: Compressibility was decreased, yield strength was increased. The rate of dewatering and strength gain was more prominent in the case of double-drainage columns than in the single-drainage columns.

The conventional finite-strain consolidation models use a single function for the estimation of tailings consolidation behaviour, which is again derived from the consideration of two constitutive relationships: Effective stress-void ratio relationship and hydraulic conductivity-void ratio relationship. Typically, these relationships are directly determined from a large strain consolidation test. However, in this experiment, the results from the oedometer tests showed that there were changes in compressibility curves with time. The initial flattening of the compressibility curve at low effective stresses could be due to the development of thixotropic strength which gives the fine tailings an over-consolidation effect (also suggested by Miller, 2010).

The experiments are still in the development stage and expected to generate a range of qualitative and quantitative results that could help in evaluating the implications of creep and thixotropy for the fluid fine tailings dewatering potential and consolidation behaviour over a longer timescale. The consideration of a full range of volume change behaviour including creep and thixotropy could have a considerable impact on the current deposition practices and important implications to the dewatering efficiency of multiple tailings technologies in the oil sands industry, especially with respect to long-term dewatering performance. The findings also complement the ongoing work on modeling creep and thixotropy in oil sands tailings (Qi et al., 2017; Salam et al., 2017).

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