

Effects of polymer dosage on dewaterability, rheology, and spreadability of polymer-amended mature fine tailings

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Abstract

One of the technologies now undergoing trials at the operational scale in the oil sands industry to promote dewatering of fine tailings is in-line polymer mixing followed by air drying. Examples of these technologies include Shell's atmospheric fines drying (AFD) and Suncor's tailings reduction operations (TRO). Fine tailings (mature fine tailings or cyclone overflow tailings) are mixed with polymer to promote clay particle aggregation. The polymer is injected and mixed within a few metres of discharge points. The proper concentration of polymer and the optimum mixing intensity influence both dewatering processes (initial water release, desiccation, and consolidation) and the spreadability of the tailings over the disposal area. This optimisation significantly affects the initial water release by increasing floc formation, and of course minimises operational costs by keeping the polymer dose as low as possible. In this study, the optimum polymer dosage was determined to be 725 g/ton. Using higher dosage significantly affects the dewaterability due to consolidation. Scanning electron microscopy also showed the presence of free flocculants at higher polymer dosages. It is believed that the presence of free ionic flocculent may increase the negative charges which subsequently diminish the dewaterability potential. Also, in the practical polymer dose range of 600 to 1200 g/ton, the rheological properties of the tailings were measured using different rheometry techniques, slump tests and analytical calculation using flume test. The compatibility of yield stresses obtained in the laboratory with the field measurement of yield stresses confirms that mixing conditions used in the laboratory were representative of field mixing conditions.

1 Introduction

Oil sands tailings consist of water, sand, silt, clay, and residual bitumen that are produced as a by-product of the bitumen extraction process. Mature fine tailings (MFT) are the most problematic portion of the tailings, which has very poor consolidation and water-release characteristics. MFT is the suspended fine fraction (i.e. clay) of tailings with solid concentrations of 30-40% and low hydraulic conductivity that is produced after several years of sedimentation. Therefore, conventionally deposited MFT is not sufficiently effective for the reclamation of tailings impoundments in a reasonable time.

Recent regulations (e.g. Directive 74) have mandated that oil sands tailings achieve set strength criteria within specific times after deposition. These regulations have accelerated development of a range of technologies to improve dewatering rate and strength gain in oil sand tailings. New technologies such as composite tailings, centrifugation, in-line thickening, and the combination of these techniques with thin-lift deposition or rim-ditching have been developed over the decades to improve the consolidation and dewaterability, and consequently permit reduction of the volume and footprint of the MFT deposits (Munoz et al., 2010). One of the more promising technologies that has the lowest cost and requires relatively low energy input is in-line mixing of flocculants with MFT (in-line thickening) followed by thin-lift deposition to promote post-deposition dewatering through desiccation (evaporation, drainage, and/or freeze-thaw) and consolidation (McKenna et al., 2012; Demoz and Mikula, 2012). Figure 1 illustrates the in-

line thickening operation at Shell Canada's Muskeg River Mines where a rapid mixing of polymer occurs in a 5.2 m pipeline. As shown in Figure 1, dewatering of the tailings starts rapidly during the deposition.

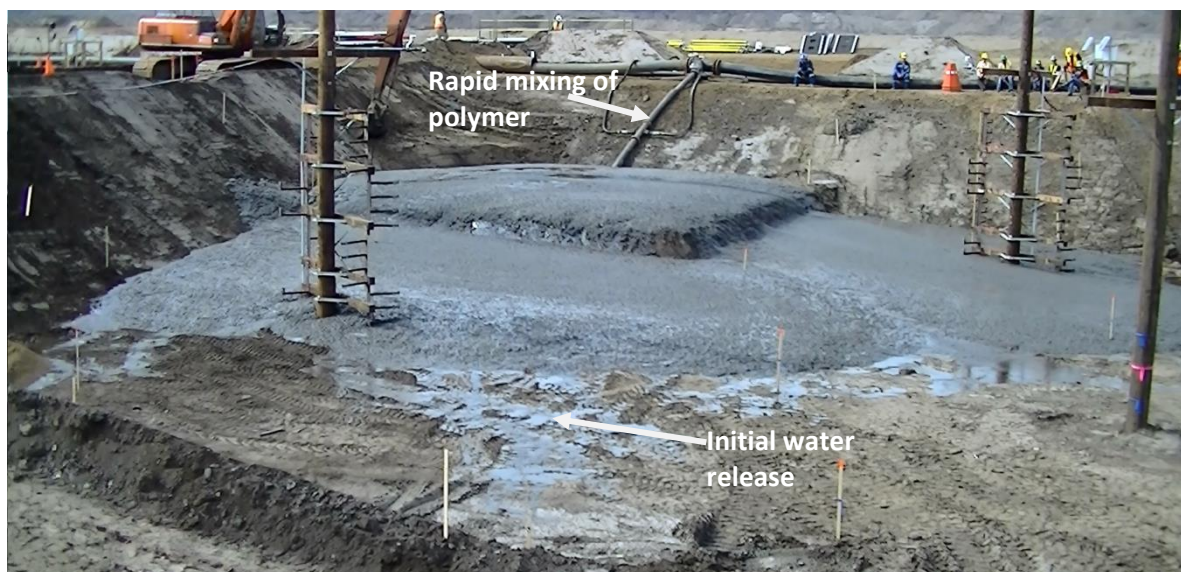


Figure 1 In-line thickening operation at Muskeg River Mines (Shell Canada Energy)

Flocculation is the process of aggregating dispersed fine particles, and usually involves two distinct processes of charge neutralisation and floc growth (Rattanakawin and Hogg, 2001). Flocculants suppress inter-particle repulsion and promote clay particle aggregation, hence enhancing the particle settlement. Formation of larger flocs is preferable for faster sedimentation which results in faster initial water release. However, the floc breakage rates increase with increase in floc size and agitation intensity. Hence, the dewatering performance of a given flocculant also depends on the mixing conditions such as the duration and intensity of agitation (Rattanakawin and Hogg, 2001; Demoz and Mikula, 2012).

Unlike inorganic coagulants (e.g. alum, gypsum), the use of an organic polymer as the flocculant preserves the chemistry of captured release water and therefore does not impede use of release water in the extraction process (Munoz et al., 2010). However, it is crucial to understand the optimal polymer dosage and mixing condition as over-mixing results in shearing and destruction of floc structures, and a reduction in water release. Most of the studies that have evaluated the water release behaviour of polymer-amended MFT were performed in a short period of time in which the sedimentation is the main dewatering mechanism (Watson et al., 2012; Mohler et al., 2012). However, in this study, the dewaterability is characterised over 13 days to also consider the effect of consolidation, as consolidation has been observed to become dominant over sedimentation after one week for different treatments of oil sand tailings (Caughill et al., 1993). Knowledge regarding the rheology and flow behaviour of the flocculated MFT is necessary to maintain control over the thickness of the amended tailings, which in turn influences the efficiency of the different post-deposition dewatering processes. This paper presents experimental research to examine the dewaterability of the flocculated MFT at different polymer dosage and mixing time. Different tests such as slump, flume and rheometry techniques were performed to characterise the flow behaviour of flocculated MFT at different flocculant dosages. The obtained yield stresses and spreadability data are compared with the field data to evaluate the closeness of the laboratory mixing performance to the actual field mixing conditions. The flow characteristics data generated in this study will also provide a useful database for the numerical simulation of polymer-amended tailings flow. Other studies are ongoing on the influence of polymer dosage on the desiccation behaviour of MFT (Bajawa and Simms, 2013).

2 Materials and methodology

2.1 MFT and polymer

MFT used in this study were obtained from Shell Canada's Muskeg River Mine. MFT were thoroughly mixed with the bleed water released during the transportation to bring them to their initial condition. The raw MFT had an initial solid content of 35.5% and sands to fines ratio of 0.27. The geotechnical properties and particle size distribution of the tailings are shown in Table 1 and Figure 2.

Table 1 Geotechnical properties of raw MFT

Property	Value
Specific gravity	2.50
D ₁₀ , D ₅₀ , D ₆₀ (microns)	0.8, 6.4, 11.1
Cu (D ₆₀ /D ₁₀)	14.1
Liquid limit (%)	83
Plastic limit (%)	28.7
Hydraulic conductivity (cm/s)	4.3×10^{-6}
Fines content (<44 μm , %)	78.7

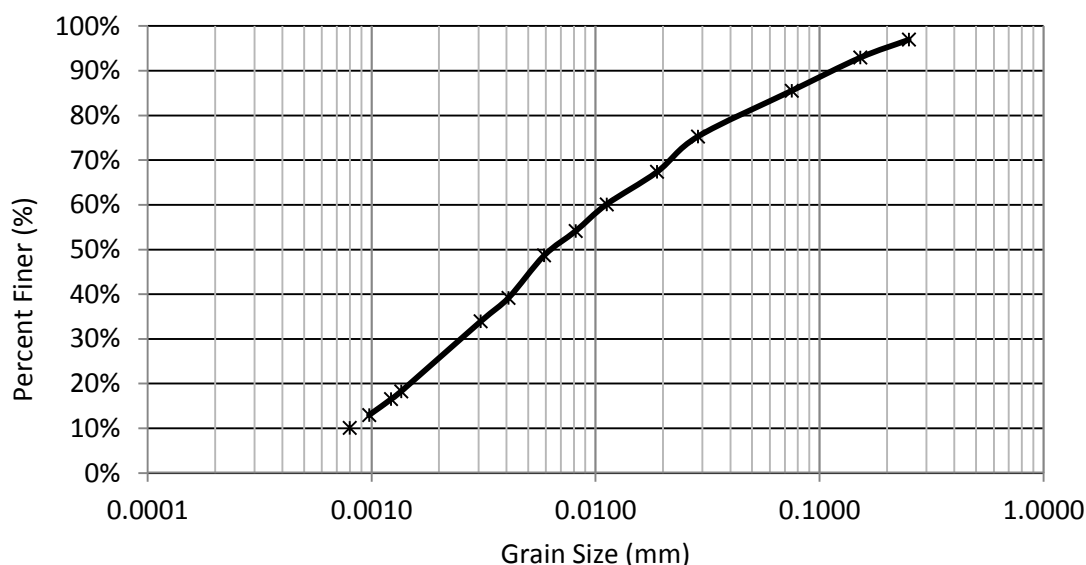


Figure 2 Particle size distributions of raw MFT from hydrometer and sieve analyses

An anionic polymer, A3338, was used as the flocculant for fast dewatering of MFT samples. Solutions of 0.4% (w/w) were prepared by adding 4 g of polymer to 996 g of reclaimed water. Reclaimed water was also received from the Muskeg River Mines. Flow curve of the flocculant prepared at concentration of 0.4% (w/w) is shown in Figure 3. According to Figure 3, the flocculant solution classifies as a pseudo-plastic or shear-thinning fluid in which viscosity decreases with increase of shear rate and the flow curve becomes linear only when the shear rates reach high values. The material showed a yield stress of 0.1 Pa, a consistency index of $1.79 \text{ Pa}\cdot\text{s}^n$ and a Power index of 0.38 when fitted with the Herschel-Bulkley Model.

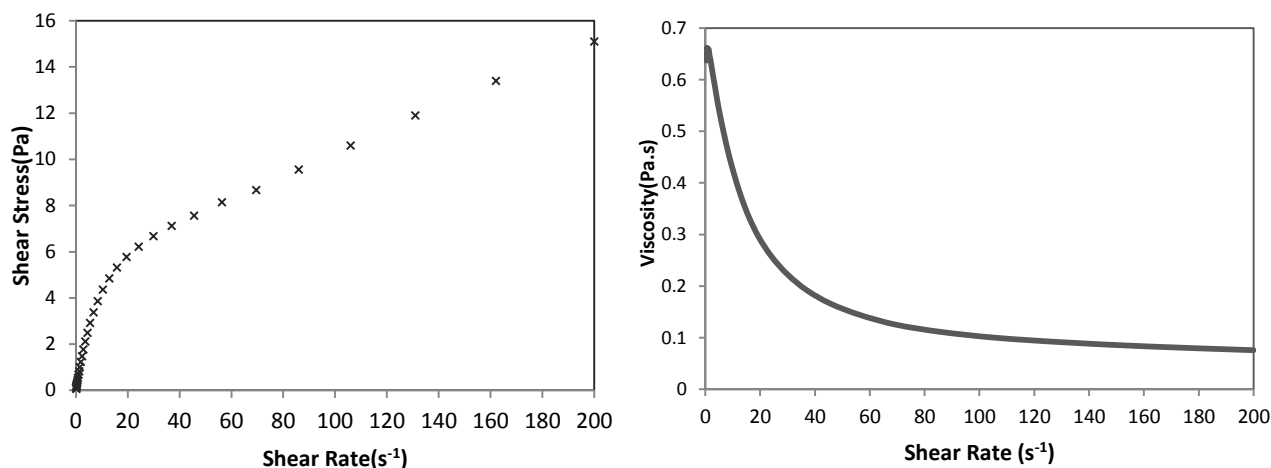


Figure 3 Flow curve for A3338 polymer prepared at concentration of 0.4% w/w (left), viscosity profile with shear rate (right)

2.2 Preparation of flocculated mature fine tailings

In this study, the following procedure was used for making flocculated MFT. First, the MFT was mixed thoroughly with the bleed water to create a well mixed suspension of solids then a four blade impeller with a radius of 8.5 cm was immersed in 1,800 g of MFT. After setting the mix speed at 250 rpm, the 0.4% flocculant solution was added and mixing of MFT and flocculant was conducted for 10 seconds. The flocculant solution was mainly directed near the impeller during mixing. Rheology tests on the flocculated MFT were conducted 30 minutes following mixing. The delay is used in testing protocols on field samples obtained from Shell's AFD trials. Due to sensitivity of flocculated MFT to time and shearing, the above procedure was also followed to obtain permeability and rheological characteristics of the tailings in the field. Comparisons between field and laboratory measurements are presented later in this paper.

Mixtures of MFT and flocculant were prepared using different dosages and mixing times. This study evaluated amended tailings with polymer dosages of 600, 725, 850, 1,000 and 1,200 g/ton, while varying the mixing time between 5 and 20 seconds. The performance of flocculation processes are usually evaluated using settling rate, supernatant turbidity or sediment compressibility (Hogg, 2000). In the current study, the release water and total solids of supernatant were measured as an indication of settling rate and supernatant turbidity, respectively. For this purpose, the flocculated MFT were poured in 500 mL graduated cylinders to a height of 0.3 m and covered to prevent evaporation. Although water release determinations of field samples are typically conducted within a day or two, in this study measurement of released water was performed for longer time (i.e. 13 days) to investigate the dewaterability potential due to consolidation as well. Total solid concentration measurement of released water was performed on day six to evaluate the supernatant turbidity. Solid concentration was measured by oven drying of 15 ml of released water collected from the columns.

2.3 Rheology and test procedures

In this study, the rheology of the tailings was investigated using an Anton Paar Physica Rheometer with a vane fixture, as well as by using the slump test. The vane fixture was made of four thin blades with the blade length of 4 cm and diameter of 1.1 cm. The theory and description of these techniques are presented in the following sections.

2.3.1 Rate controlled mode (stress growth)

In the stress growth technique, a low and constant shear rate is applied to the vane and the torque is measured as a function of time. The torque is then converted to shear stress. The peak in the torque-time

curve is related to the yield stress of the material. The stress can be calculated from the measured torque through:

$$T_m = \frac{\pi D_v^3}{2} \left(\frac{H}{D_v} + \frac{1}{3} \right) \tau_y \quad (1)$$

Where:

- T_m = measured torque.
- D_v = diameter of the vane.
- H = length of the vane.
- τ_y = shear stress.

Four distinct stages can be identified in the stress–time profile as illustrated in Figure 4. An initial linear region followed by a nonlinear behaviour, a maximum stress and finally the stress decay region where the fluid disintegrates completely and starts flowing like a viscous liquid (Liddell and Boger, 1996). The linear region corresponds to the elastic deformation.

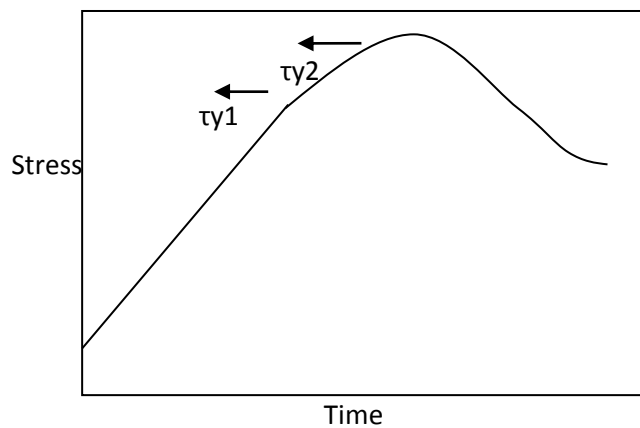


Figure 4 A typical stress-time profile in a rate controlled mode (after Liddell and Boger, 1996)

2.3.2 Stress controlled mode

A constant stress is applied to the vane in increasing or decreasing steps and the strain is measured as a function of time. Below the yield stress, the strain approaches a constant value, whereas it increases indefinitely with time when the stress exceeds the yield stress. In order to simulate the stress history experienced by tailings as they are flowing down the beach, the samples were sheared for duration of time, and then the stress was gradually decreased until the material stopped flowing and no change of strain was detected with time.

2.3.3 Slump test

Yield stress was determined from the slump tests using the cylinder method of Palshias et al. (1996) with a cylinder height of 10 cm and diameter of 5 cm. Dimensionless slump was then related to yield stress by the following equation:

$$\tau_y = \rho g H \left(\frac{1}{2} - \frac{1}{2} \sqrt{s'} \right) \quad (2)$$

Where:

- τ_y (kPa) = yield stress.
- s' = dimensionless slump and is defined as measured slump over the height of the cylinder.

H (m) = the height of cylinder.

ρ (kg/m³) = bulk density of sample.

2.3.4 Flume test

For simulating the depositional behaviour of thickened tailings under laboratory conditions, a flume apparatus with a length of 243 cm, and width of 15.3 cm was used. A set volume of flocculated MFT was poured at a distance of 20 cm above the flume through a funnel and the length of the flow and the depth were measured at different locations (every 5 cm) after the stabilisation of flow. The variability in yield stress was then calculated using lubrication theory for Bingham fluid. Lubrication theory, a simplified form of the momentum and continuity equations, has previously been used (Simms, 2007; Henriquez and Simms, 2009; Mizani and Simms, 2010) to model the steady state and transient flow of yield stress fluid. The steady state profile of a Bingham fluid on a flat surface and inclined surface can be determined through:

$$\tau_y(x - x_0) = \frac{\rho g}{2}(h^2 - h_0^2) \quad (3)$$

Where:

P = the density.

τ_y = the yield stress.

H = the height of the free surface and is measured vertically as:

$$\ln(1 - h') + h' - h'_0 = x' - x'_0 \quad (4)$$

Where θ is the angle of inclined surface and,

$$h = h' \left(\frac{\tau_y}{\rho g \sin \theta} \right) \quad (5)$$

$$x = x' \cot \theta \left(\frac{\tau_y}{\rho g \sin \theta} \right) \quad (6)$$

2.4 Scanning electron microscopy (SEM)

Scanning electron microscopy (Vega-II XMU VPSEM, Tescan) capable of using variable pressure and wet operation was used for SEM imaging in this study. The images were collected at the scanning speed of 148 μ s/pixel and a working distance of 6-8 mm. SEM was operated at acceleration voltage of 20 kV using a cold stage to freeze the samples during the observation. The freezing was performed to prevent excessive water withdrawal during the observation under the vacuum condition of the SEM chamber.

3 Results and discussion

3.1 Dewaterability of flocculated MFT

Flocculation effectiveness can be assessed based on the settling rate tests where the mud height over time is measured in large graduated cylinder (Mohler et al., 2012). The effects of flocculant dosages were investigated by measuring the release water from tailings prepared at different dosages. Figure 5 illustrates the water release as a function of flocculant dosages at different times. Although initially the dosage of 850 g/ton had the highest water release, the dewaterability of the 725 g/ton started to exceed others after six days. This may be explained by the fact that at higher dosage, the floc structure may be stiffer and hence reduce consolidation at this very low stress (5 kPa). Jin et al. (2004) found that in wastewater sludge the dewaterability potential will be reduced by formation of bigger flocs and high viscosity of the material. It is also believed that high flocculant dosage increases the amount of free floc, consequently increasing the negative charges.

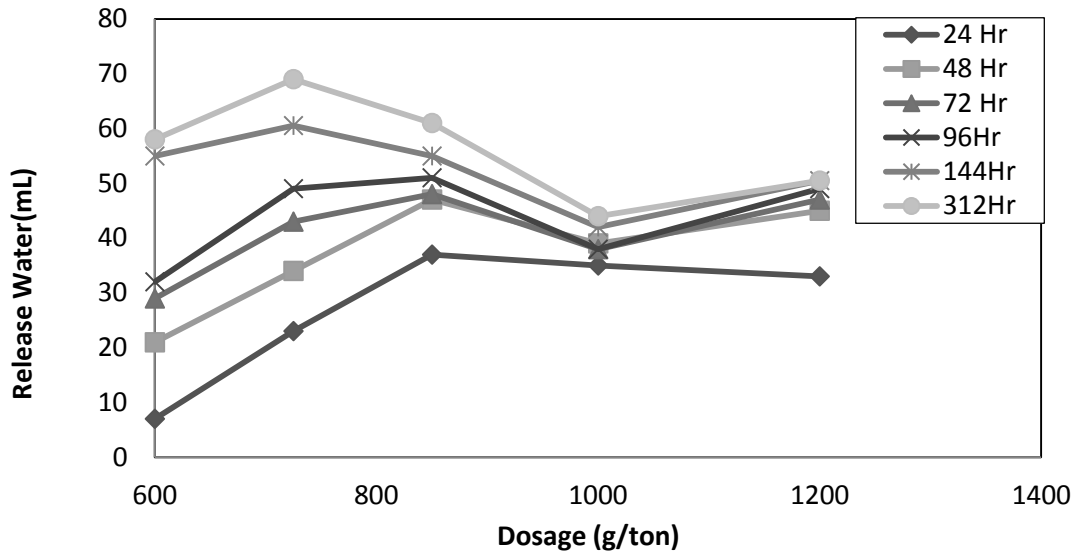


Figure 5 Water release as a function of flocculant dosage and time

Figure 6 shows the total solid concentration in the released water which is a representative of turbidity of the water and usually is used to evaluate flocculation performance. As shown in Figure 6, the clearest water was obtained at the flocculant dosage of 1,000 g/ton. Observation of high turbidity water can be explained by either inadequate flocculant dosage to provide particle neutralisation for all the dispersed particles or improper mixing to break the formed flocs. Hence, it may be interpreted that at flocculant dosages of less than 1,000, the amount of flocculant was not enough to neutralise all the particle charges.

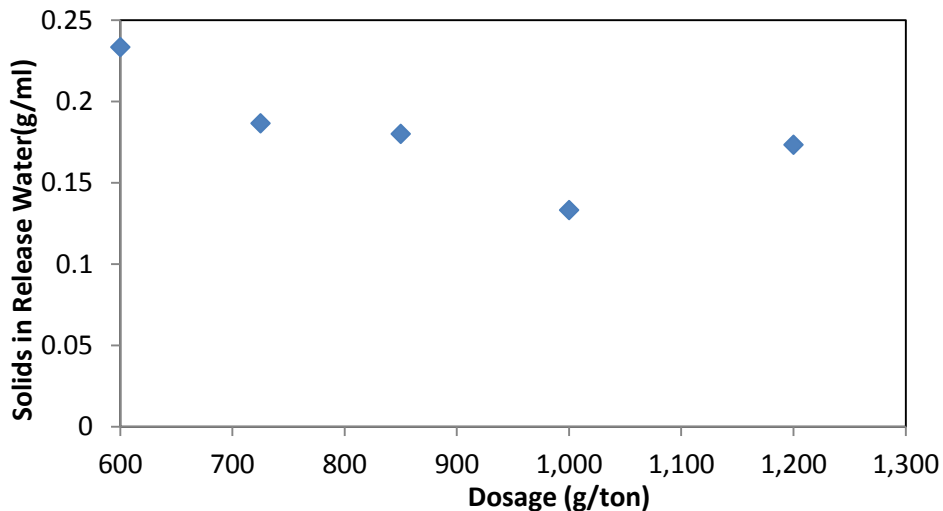


Figure 6 Total solid concentrations in released water as a function of flocculant dosage

Previous studies (Watson et al., 2012) have shown that water release is a function of mixing energy (stirring speed and mixing time). This study was based on single stirring speed of 250 rpm to simulate the field conditions implemented at Shell Canada’s Muskeg River Mine. Therefore, only the effects of mixing time were investigated. Mixing times of 5, 10, 15 and 20 seconds were considered. Water release data in terms of mixing time for floc dose of 725 g/ton, which was deemed to be the optimum dose in terms of water release, are presented in Figure 7. After repeating this test for various flocculant dosages, it was visually observed that mixing times above 10 seconds resulted in collapse of the floc structure causing irreversible floc breakage possibly due to high shearing.

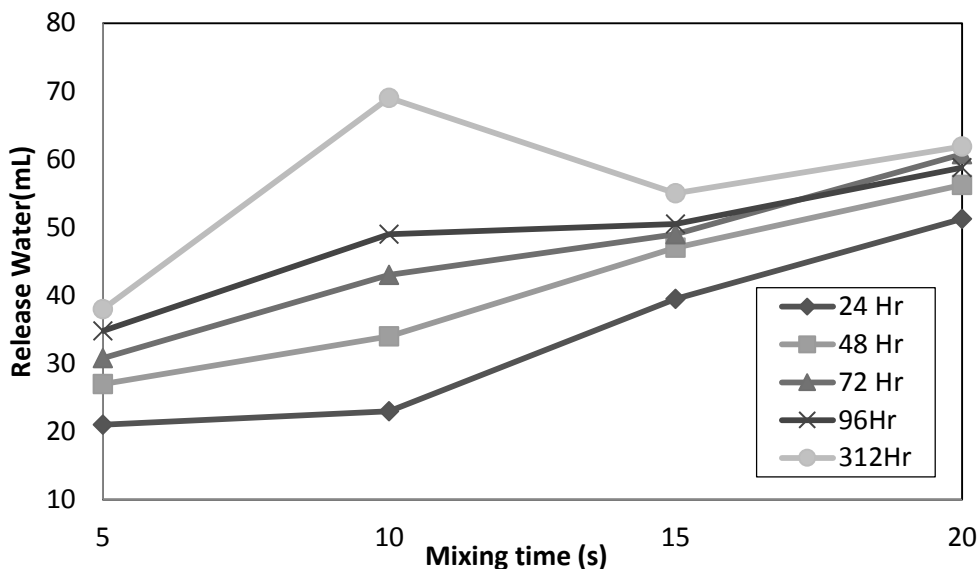


Figure 7 Effect of mixing time at 250 rpm on rate of water release for tailings prepared at 725 g/ton

3.2 Microstructural analysis

Scanning electron microscopy observations were carried out to determine the surface structure of flocculant and morphology and arrangement of flocs in flocculated MFT. Figure 8 shows the morphology of flocculant solution used in this study. The flocculant used is branch type flocculant with fibrous network morphology (Figure 8).

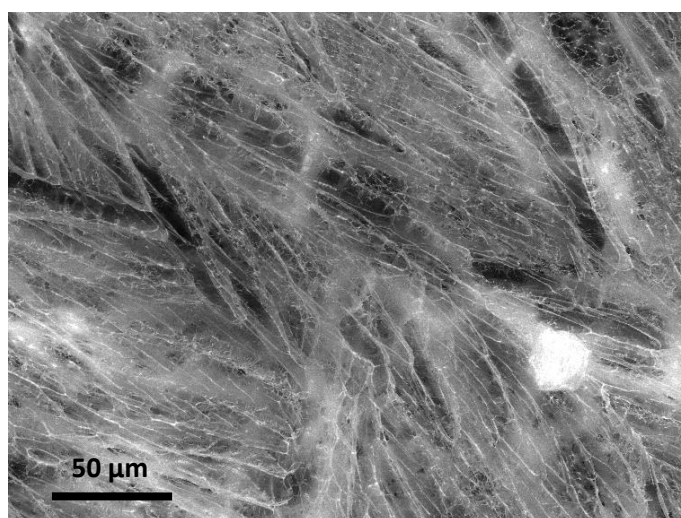


Figure 8 SEM micrograph of A3338 polymer used to prepare flocculated MFT (magnification: 500×)

Comparing SEM micrographs of flocculated MFT with raw MFT in Figure 9 shows that flocculation improved aggregation of particles and resulted in the formation of water channels. The formation of water channels possibly increases the hydraulic conductivity of tailings and improves dewaterability. The higher magnification image of flocculated MFT at 1,200 g/ton dosage in Figure 10 shows the presence of free flocculant particles. Figure 10 also confirms that the presence of free ionic flocculant particles which increased the amount of negative charges was one of the reasons for the reduction in dewaterability potential at high flocculant dosage (Figure 5).

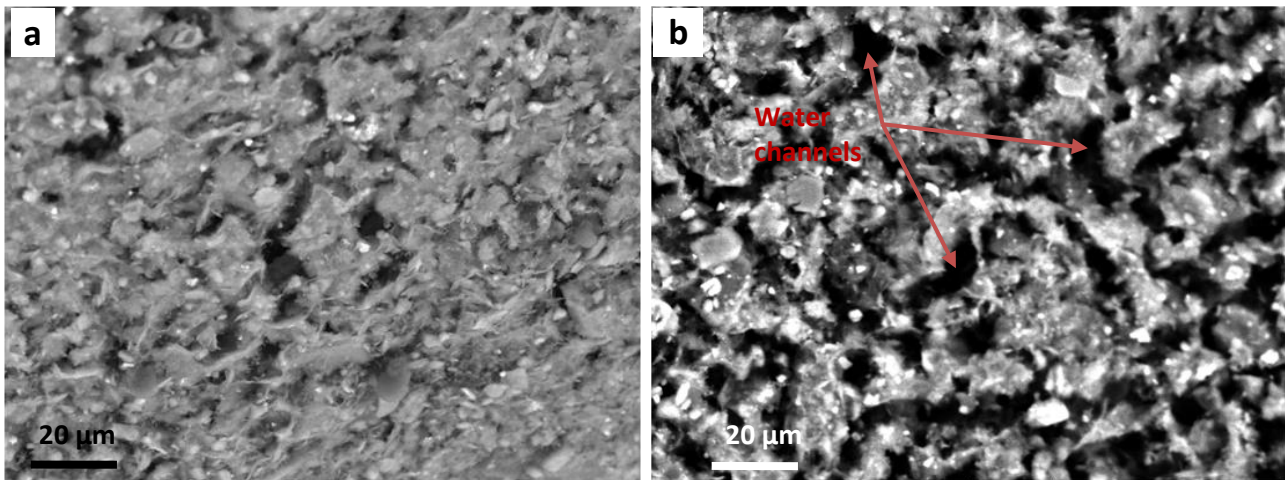


Figure 9 Comparison of SEM micrograph of a) raw MFT with b) flocculated MFT at 1,000 g/ton flocculant dosage (magnification: 1,000×)

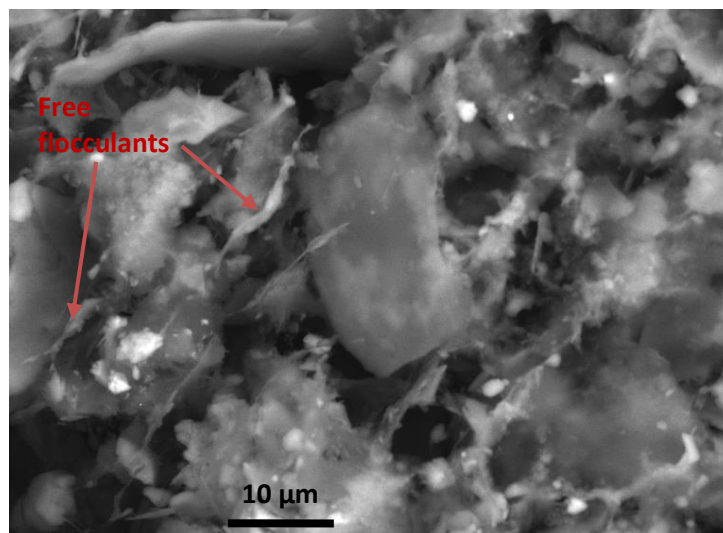


Figure 10 High magnification SEM micrograph of flocculated MFT at 1,200 g/ton showing free flocculant particles (magnification: 2,000×)

3.3 Rheological behaviour

Yield stresses obtained from the various techniques for dosages in the range of 600 to 1,200 g/ton are given in Table 2. As expected, there is an increase in yield stress with increase in flocculant dosage. For the stress growth technique, two different shear rates of 0.1 and 1 s⁻¹ was exerted on the tailings. Figures 11 and 12 show the results obtained from these two sets of tests. It can be seen that the maximum shear stress is a function of shear rate and generally higher values were obtained for the shear rate of 1 s⁻¹. This could be explained by the fact that at higher shear rates not enough time is given for the elastic response and orientation of the network structure. Similar results were observed by Dzuzy and Boger (1983) on red mud suspensions and by Liddell and Boger (1996) on TiO₂ pigment suspension.

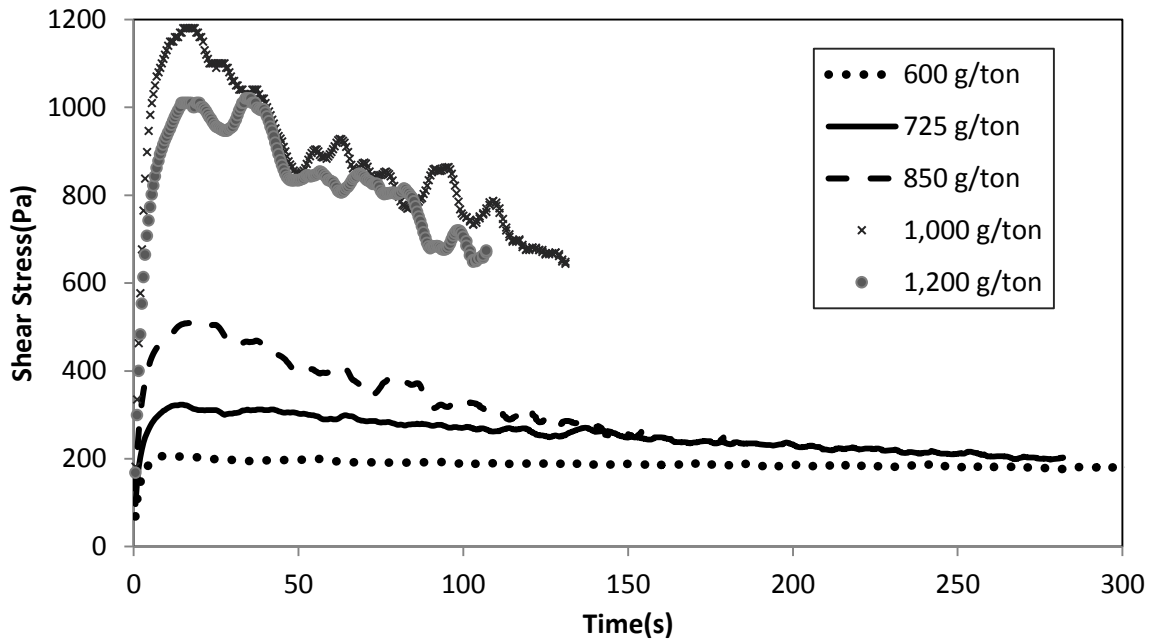


Figure 11 Stress growth diagrams of flocculated MFT at constant shear rate of 1 s^{-1}

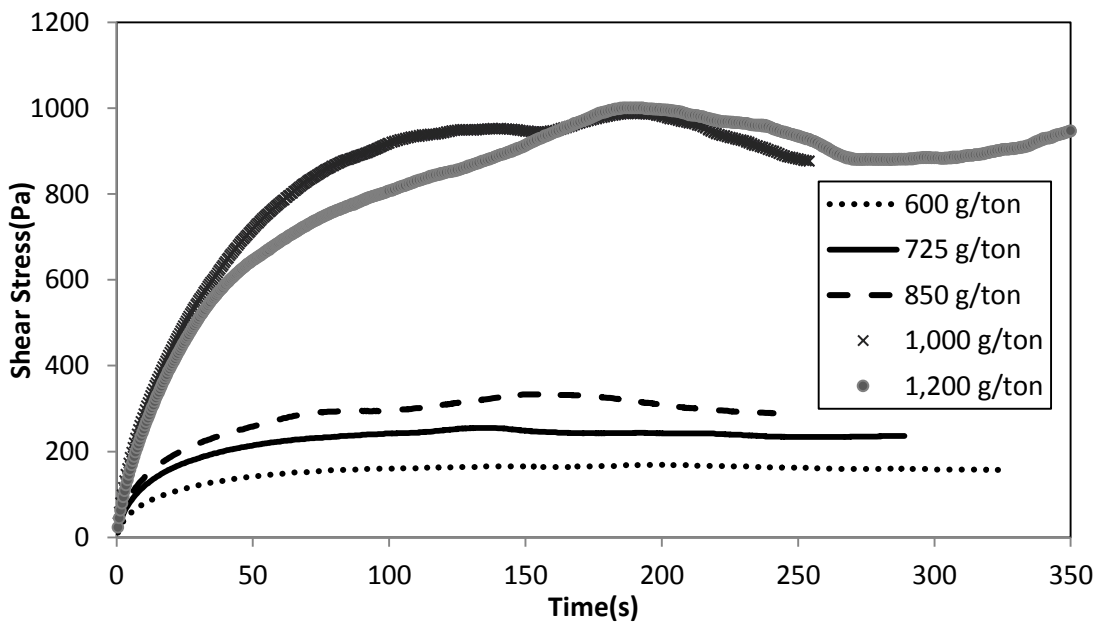


Figure 12 Stress growth diagrams of flocculated MFT at constant shear rate of 0.1 s^{-1}

Table 2 Yield stress measured from various techniques

Dosage (g/ton)	Slump (Pa)	Method of measurement			
		Stress growth		Decreasing shear stress	
		Shear rate(S ⁻¹)	Max stress(Pa)	Starting shear stress (Pa)	Interpreted yield stress (Pa)
MFT	–	0.1	28.8	100	10
		1	28.0		
600	92	0.1	169	250	50–100
		1	207	200	50–100
725	125	0.1	255	450	50–100
		1	323		
850	154	0.1	333	700	50–100
		1	510		
1,000	163	0.1	988	1,000	250
		1	1,020		
1,200	187	0.1	1,000	1,300	–*
		1	1,180		

*Unable to detect yield stress due to high shear intensity and overflowing of tailings out of the measuring cup.

In the stress control mode, tailings were first sheared for duration of time and then the stress was gradually decreased until the material stopped flowing and no change of strain was detected with time. Figure 13 illustrates one of these tests where the stress was decreased from 450 to 5 Pa in 360 seconds. The initial stress value was chosen based on the maximum shear stress gained from the stress growth test (in this case 323 Pa) to make sure that the tailings are sheared. No significant movement of the vane was recorded when the stress reached 50 Pa. Further investigations are underway to see the effects of shearing time and initial shear stress on the yield stress from this method. The graphs of stress control mode for other dosages are not shown for the reason of brevity; however, the yield stress values obtained for various dosages are given in Table 2.

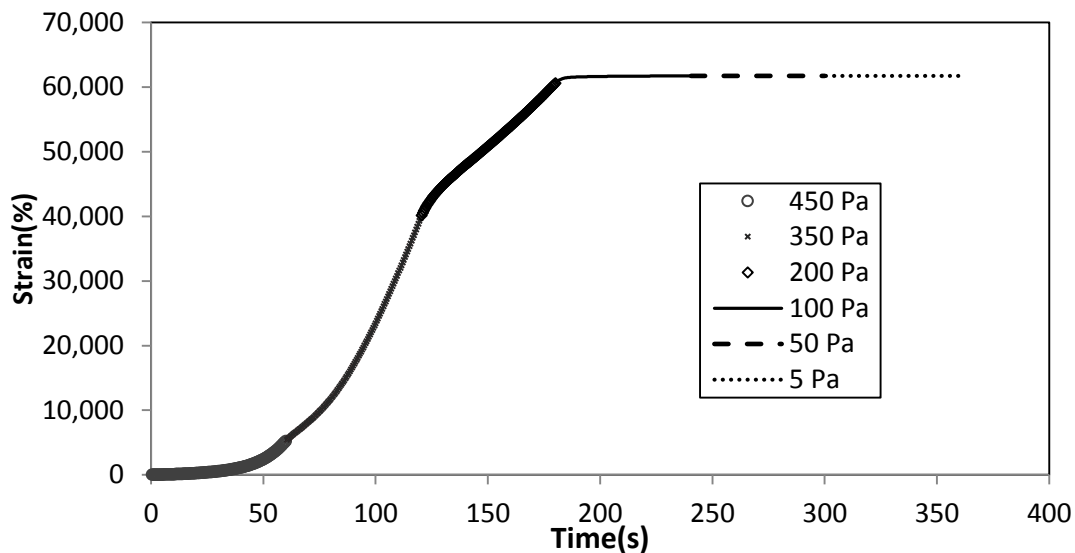


Figure 13 Constant decreasing stresses applied to samples prepared at 725 g/ton floc; each stress level was applied for 30 seconds

From the rheology measurements, it can be seen that a closer agreement is obtained between the yield stress values measured from the slump, and the decreasing shear stress test. That is due to the similarity of these two tests, where the yield stress is actually measured after shearing the material. However, from the constant stress test only the range for the shear stress could be given due to the time consuming nature of the test.

3.4 Spreadability

Figure 14 shows the equilibrium profiles for the tailings deposited at different floc dosage using a funnel. Tailings prepared at the dosage of 600 g/ton had the longest run-out compared to the other dosages. This indicates that the tailings deposited with higher floc dose gained a steeper slope and shorter run-outs upon deposition. This is also consistent with field observations where higher yield stress materials tend to stack closer to discharge points where little or no material reach downstream of the cells. However, to gain a higher storage capacity, not only a steeper slope is required but also uniform footprint coverage is essential. Table 3 shows the yield stress obtained by fitting the lubrication theory equations to the deposited profile.

When it comes to deposition of thickened tailings, the yield stress of interest is the yield stress that characterises where the material stops flowing. Therefore, it is reasonable to observe that the yield stresses obtained from the flume tests lie significantly below the ones measured from the stress growth and closer to the slump and decreasing stress tests.

Table 3 Yield stress obtained from flume test, predicted by Equation (3)

Dosage (g/ton)	Yield stress (Pa)
600	60
725	95
850	104
1,000	110

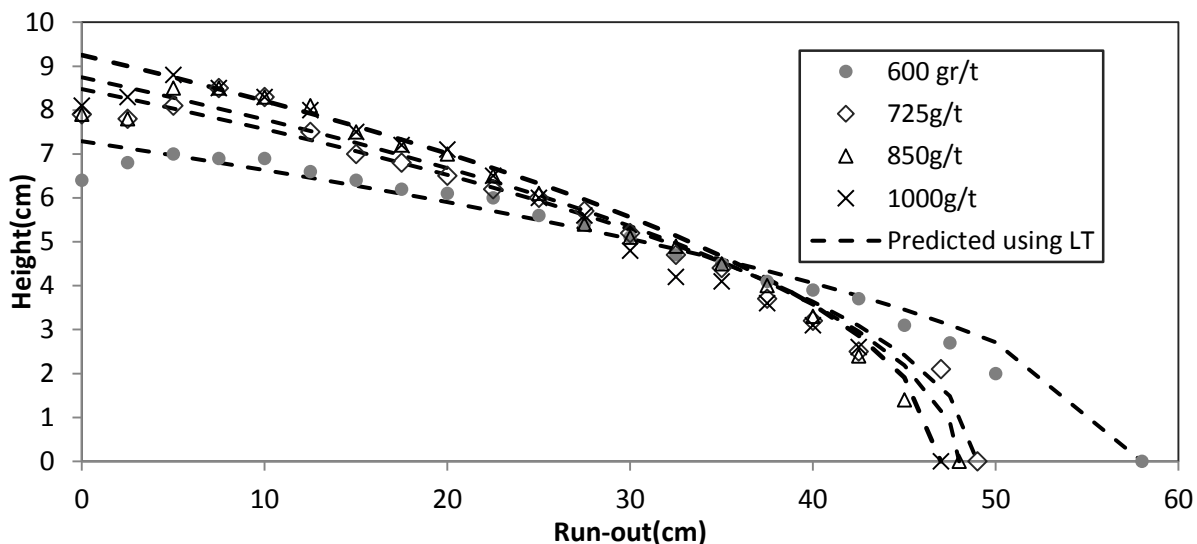


Figure 14 A single layer flow at various floc dosages, all fitted with Equation (4) with the predicted yield stress shown in Table 3

4 Comparison with field data

Figure 15 shows a comparison between the yield stress measured in field during the pilot scale test and the ones measured in the laboratory. Both measurements were conducted using the stress growth method with a shear rate of 0.1 s^{-1} . In this operation the flocculant solution was mixed with the MFT in 5.2 m pipes before deposition into cells. Tailings were deposited at a flow rate of around $900 \text{ m}^3/\text{hr}$ while the flocculant dose fluctuated between 770 and 950 g/ton. Samples were collected at regular intervals and as close as possible to the deposition point. As shown in Figure 15, the yield stresses obtained in the field overlaps well with the yield stresses obtained in the laboratory with the same polymer dosage; hence the mixing time and intensity used to prepare the flocculated MFT in the laboratory was representative of field mixing conditions.

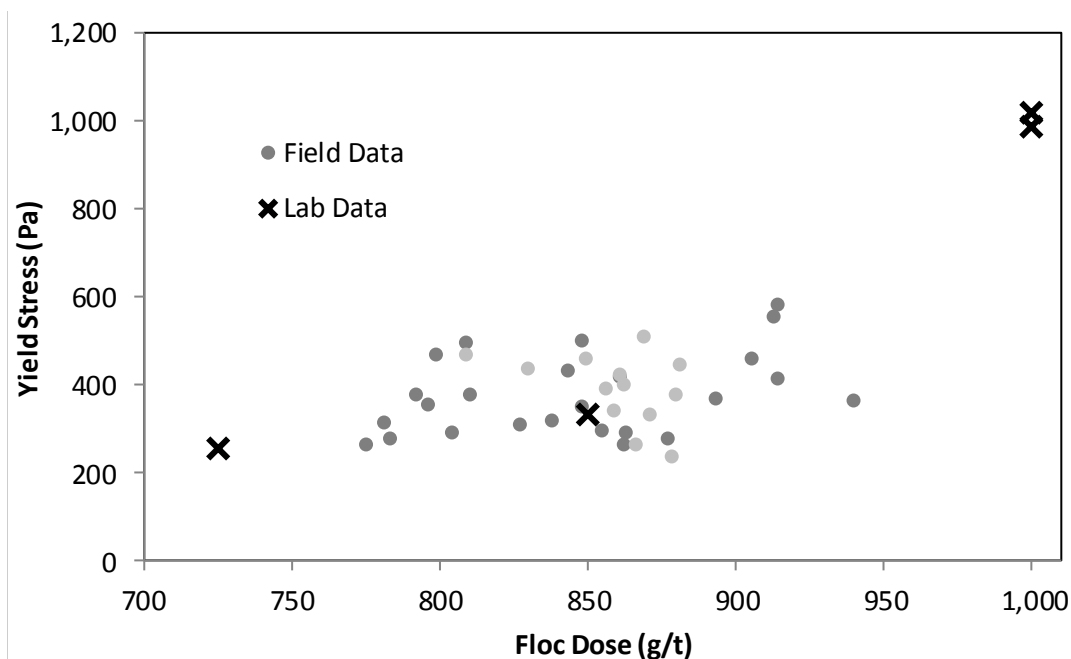


Figure 15 Comparison between yield stress values measured in laboratory and deposition in Muskeg River Mines tailings site during Aug-Oct 2012

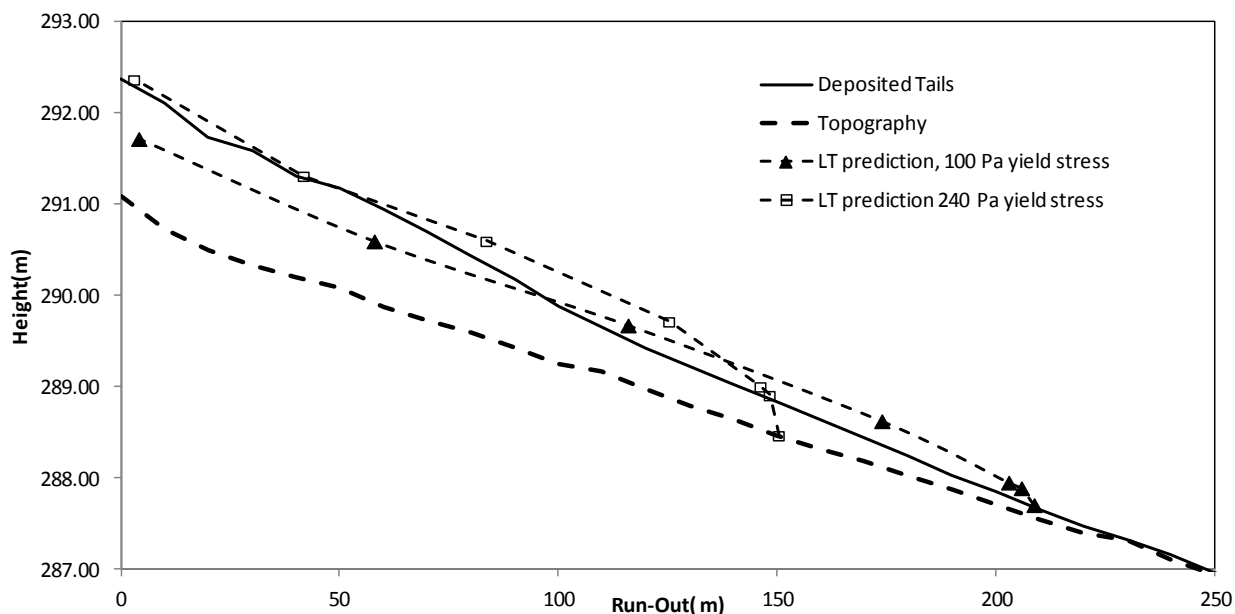


Figure 16 Tailings profile measured at Shell Atmospheric Fine Drying operation; predictions were modelled using lubrication theory (LT)

Figure 16 illustrates the tailings profile deposited at Shell Atmospheric Drying cell during the autumn 2010 programme. The total volume of tailings deposited in this cell was 7,953 m³ with an average slope of 2.1%. The predictions used Equation 4, fixing the volume of tailings but using two different values of yield stress, 100 Pa which is the value represented by the flume tests, and 240 Pa, which is representative of the yield stress for the stress growth tests. While the higher yield stress value best predicts the maximum height of the tailings at the deposition point, the lower yield stress value better predicts the maximum run-out. As the material is highly thixotropic over the timescale required for deposition (five days), it is not surprising that a single yield stress value cannot perfectly capture the shape.

5 Conclusions

Proper concentration of polymer and the mixing intensity both influence the dewatering processes (i.e. initial water release, and dewaterability due to consolidation). In this study, the polymer dosage of 725 g/ton was shown to provide the best dewatering in a 0.30 m layer over two weeks. For samples with doses of 1,000 g/ton, most water release occurred within 24 hours. Using high polymer dosage also showed the presence of free flocculant without attachment to any particles. It was also shown that changing mixing energy significantly affects floc formation, as a higher degree of mixing causes irreversible floc breakage.

The rheological behaviour of flocculated MFT back calculated from the flume test was in better agreement with slump and controlled decreasing shear stress test. This can be explained by the fact that in these tests, the yield stress is measured after shearing the material; whereas, the stress obtained from the stress growth technique actually measures the initiation of the viscous flow. Relatively simple analysis of one field profile shows that while the yield stress corresponding to the slump and controlled stress tests predicted the run-out distance, significant tailings accumulated at higher heights closer to the deposition point. This may be due to the thixotropic behaviour of the material, which certainly manifests over the timescale of deposition (five days). More work is required to characterise the dependence of spreadability on both aging and shearing.

Acknowledgements

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