Unsaturated soil properties of centrifuged oil sand fine tailings

John Owolagba and Shahid Azam
Environmental Systems Engineering, University of Regina, Regina, SK, Canada

ABSTRACT
The main objective of this study was to determine the unsaturated soil properties of oil sand fine tailings after centrifugation, that is, at a solids content of 60% (void ratio = 1.5). The soil water characteristic curve plotted using the degree of saturation resulted in an air entry value of 1000 kPa. Likewise, the shrinkage curve showed a J-shaped pattern with most of the volume decrease occurring during normal shrinkage. With increasing suction, the unsaturated hydraulic conductivity decreased by several orders of magnitude from a saturated value of 2×10^{-10} m/s.

RESUME
L'objectif principal de cette étude était de déterminer les propriétés des sols non saturés de sable bitumineux. Après centrifugation, c'est-à-dire pour une teneur en solides de 60% (taux de vide = 1,5). La courbe caractéristique de l'eau du sol tracée en utilisant le degré de saturation a donné une valeur d'entrée d'air de 1000 kPa. De même, la courbe de rétrécissement a la forme de la lettre « J », avec la plus diminution de volume survenant au cours du rétrécissement normal. Avec l'augmentation de succion, la conductivité hydraulique non saturée a diminué de plusieurs ordres de grandeur à partir de sa valeur saturée de 2×10^{-10} m/s.

1 INTRODUCTION

For over four decades geotechnical engineers have struggled to improve the dewatering capability of oil sand fine tailings due to the combined effect of ore geology, resulting in the formation of kaolinite, illite and smectite clay minerals and extraction process characterized by high temperature and caustic pore fluids. Conventional tailings management in containment facilities results in segregation of the fines from the coarse fraction such that the entire coarse and about one-half of the fines forms the dykes and beaches whereas the remaining fines suspended in water flows into the pond. After a few years the fines consolidate to about 30% – 35% solids to form mature fine tailings (MFT), the properties of which are governed by water chemistry, clay mineralogy and residual bitumen and are difficult to dewatered if allowed to settle naturally (Scott et al, 2010). The main operational challenge facing oil sand tailings management is the separation of water from the fines thereby converting the latter to a semi solid that can be reclaimed as soil. The ever increasing volume and slow densification of this loose and toxic material generated at a rate of 0.25 m3/barrel of crude oil (Mikula et al., 1996) is estimated to reach 1×10^9 m^3 by 2014 thereby requiring large storage areas. To contain the environmental footprints, current regulatory guidelines specify performance criteria for MFT reduction and the formation of geotechnically stable deposits within a short time frame. Hundreds of natural, physical, chemical and physiochemical processes have been investigated with varying degrees of success at the conceptual, research, pilot scale and commercial scale levels. Centrifuge technology is being adapted as part of the long-term management of oil sand fine tailings. The process involves application of acceleration several times that of gravity to the polymer modified tailings. The method has been tested at the pilot-scale and has been found to dewater the tailings stream up to 60% solids content (Devenny, 2010).

The main objective of this study is to determine the unsaturated soil properties of centrifuged oil sand fine tailings which are necessary to evaluate the natural dewatering processes when the material is deposited on the ground under a carefully designed deposition scheme. These unsaturated properties were achieved by conducting a comprehensive research program as follows: geotechnical index properties for material classification and reported change in material state due to centrifugation, soil water characteristic curve to evaluate dewatering due to desiccation, shrinkage curve to understand effect of volume changes during suction application and hydraulic conductivity to investigate flow rate under both saturated and unsaturated condition.

2 RESEARCH METHODOLOGY

The mature fine tailings sample was obtained from a mine site in Northern Alberta and was handled in accordance with the ASTM Standard Practice for Preserving and Transportation of Samples (D5079-08). The sample (20L bucket) was stored in the Geotechnical Testing Laboratory at the University of Regina at 20.5°C.

A bench scale centrifuge was used to obtain a centrifuged cake with geotechnical properties similar to the field condition (60% solids content). The centrifuge consists of a swing type bucket capable of simultaneously holding four 100 ml sample in a single test run. The MFT samples in the specimen holders were subjected to an angular velocity of 4000 rpm operated at an equilibrium temperature of 20°C in the rotary chamber and allowed to run for 3 hours. The mass in the specimen holders were kept equal to avoid rotary imbalance. The slurry dewatered and the supernatant water was removed from the top of specimen holder and the bottom cake collected. The process was repeated until about 2 kg material was obtained and it was put in a sealed plastic container and stored in an air-tight chamber to preclude evaporation. Sub-samples were retrieved from the cake for subsequent laboratory testing.

2.1 Geotechnical Index Properties

The geotechnical index properties were conducted on the produced cake for preliminary material assessment and for use in subsequent laboratory investigations according to the ASTM test methods as follows: (i) water content (w) by the Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass (D2216-05); (ii) dry unit weight (γd) by the Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method (D2937-10); (iii) specific gravity (G_s) by the Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer (D854-10); (iv) liquid limit (w_L), plastic limit (w_p) and plasticity index (I_p) by the Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils (D4318-10); (v) shrinkage limit (w_s) by the Standard Test Method for Shrinkage Factors of Soils by the Wax Method (D4943-08); and (vi) grain size distribution by the Standard Test Method for Particle-Size Analysis of Soils (D422-63(2007)). The measured data were fitted with a uni-model curve using the following pedo-transfer function, P_p, (Fredlund et al., 2000):

$$p_p = \frac{1}{\ln\left[2.72+\left(\frac{a_{gr}}{d}\right)^{n_{gr}}\right]^{m_{bi}}}\left\{1-\left[\frac{\ln\left(1+\frac{d_r}{d}\right)}{\ln\left(1+\frac{d_r}{d_m}\right)}\right]^7\right\} \qquad [1]$$

The above equation consists of the following fitting parameters: a_{gr}, the initial break point of the curve; n_{gr}, the steepest slope of the curve; m_{bi}, the shape of the fines part of the curve; and d_r, the amount of fines in soil; d, grain size under consideration and d_m, the minimum allowable grain size.

2.2 Soil Water Characteristic Curve

The SWCC was determined according to the ASTM Standard Test Methods for Determination of the Soil Water Characteristic Curve for Desorption Using a Hanging Column, Pressure Extractor, Chilled Mirror Hygrometer, and/or Centrifuge (D6836-02(2008)e2) using MFT cake material of 60% solids initial condition. A unique specimen holder of 26 mm diameter and 16mm height was prepared to accommodate the high volume loss and prevent crack propagation during desaturation as suction application increases. The cake was scooped into the specimen holder and carefully compressed to ensure there are no cavities within the sample it was then transferred on top of a wet porous stone with a wet filter paper placed underneath to prevent fines migration. Predetermined suction values were applied using pressure plate/membrane extractors manufactured by Soil Moisture Equipment Inc. These included the following: (i) a 15 bar pressure plate extractor (Model 1500F1) along with a 0.5 bar porous plate (0675 Series) for 30 kPa suction, a 3 bar porous plate (0675 Series) for 100 kPa and 200 kPa suction and a 5 bar porous plate (0675 Series) for 380kPa, 400kPa, and 530kPa suction; (ii) a cellulose membrane (1041D21) for 1500 kPa, 3000 kPa and 6000 kPa suction. The porous plates and the cellulose membranes were submerged in distilled and de-aired water for 24 hours to expel air bubbles. Thereafter, the specimens along with the retaining ring were placed on their respective porous plate or cellulose membrane and allowed to saturate in water. Next, the excess water was removed and each porous plate or membrane was placed in the extractor for corresponding suction application. For each suction value, the expelled water from the samples was monitored in a graduated burette. When two consecutive readings nearly matched over a 24 hour period, the test was terminated and the sample water content was determined.

The dew point potentiameter (WP4-T) was used for suction measurement at low water content. The sampling cup was half filled with soil to ensure accurate suction measurement (Leong et al., 2003) by using unsaturated sample from the SWCC pressure plate extractor with a known water content and reduced diameter size due to volume loss in the extractor. The sample was cut and the surface trimmed to 4mm high to fit into the sealed measurement chamber, set at 25oC temperature, through a sample drawer and was allowed to equilibrate with the surrounding air. Equilibration was usually achieved in 10 min to 20 min, as detected by condensation on a mirror and measured by a photoelectric cell. From knowledge of the universal gas constant, R (8.3145 J/mol°K), sample temperature, T (°K), water molecular mass, X (18.01 kg/kmol), and the chamber relative humidity, p/p_o, soil suction was calculated ($\psi = RT/X \ln(p/p_o)$) and displayed on the potentiameter screen. The water content of the soil was measured as described earlier. The entire measured data was fitted with the following equation (Fredlund and Xing, 1994):

$$w = w_s\left[1-\frac{\ln\left(1+\frac{\psi}{h_r}\right)}{\ln\left(1+\frac{10^6}{h_r}\right)}\right]\left[\frac{1}{\left\{\ln\left[2.72+\left(\frac{\psi}{a_f}\right)^{n_f}\right]\right\}^{m_f}}\right] \qquad [2]$$

where: w = gravimetric water content at any specified suction, ψ; w_s = saturated gravimetric water content; hr = residual soil suction; a_f, n_f, and m_f. The equation is applicable to present SWCC using different geotechnical index parameter of water measurement on the y-axis.

2.3 Shrinkage Curve

The shrinkage test was conducted in accordance with the ASTM Standard Test Method for Shrinkage Factors of Soils by the Wax Method (D4943-08) using duplicate samples with known water content and applied suction obtained from previous SWCC test. These data were verified using a digital micrometer for volume measurement after various stages of desaturation before being coated with wax.

The volume of soil specimens was determined using the water displacement method. Each specimen was coated with molten microcrystalline wax (G_s = 0.9) and allowed to cool down at room temperature. After wax solidification, the sample was submerged in a 250 ml graduated cylinder that was filled with distilled water. The water height in the cylinder was carefully recorded using a Vernier caliper before and after sample submersion in the cylinder. A graduated syringe was used to remove the increased amount of water displaced by the sample thereby bringing the water height back to the initial reading. The displaced water mass was determined by weighing the graduated syringe before and after water filling and recording the difference. This quantity was readily converted to water volume representing the volume of the wax-coated soil. The volume of soil was obtained from the difference of volume of the wax coated sample and the volume of wax (mass/0.9). A 7.4% correction was applied to account for the underestimation due to air entrapment at soil-wax interface, as suggested by Prakash et al. (2008). The sample mass was also determined to estimate the bulk unit weight of the soil that, in turn, was converted to the void ratio using basic phase relationships. The entire data was fitted with the following equation (Fredlund and Xing, 1994).

$$e = a_{sh}\left[\frac{w^{c_{sh}}}{{b_{sh}}^{c_{sh}}} + 1\right]^{\left(\frac{1}{c_{sh}}\right)} \qquad [3]$$

a_{sh} = the minimum void ratio (e_{min}), b_{sh} = slope of the line of tangency, (e.g., drying from saturated conditions), c_{sh} = curvature of the shrinkage curve, and w = gravimetric water content. The ratio, $a_{sh}/b_{sh} = G_s/S$ is a constant for a specific soil; where G_s = specific gravity and S = degree of saturation.

2.4 Hydraulic Conductivity

The saturated hydraulic conductivity was chosen based on falling head permeability experimental data not presented in this paper. The unsaturated hydraulic conductivity function (k_r) was estimated using the Fredlund pedo-transfer function (Fredlund et al., 1994) in the soil data base software by SoilVision Systems Limited.

$$k_r = \frac{\int_{In(\psi)}^{b} \frac{\theta(e^y)-\theta(\psi)}{e^y}\theta'(e^y)dy}{\int_{In(\psi\ at\ aev)}^{b} \frac{\theta(e^y)-\theta_s}{e^y}\theta'(e^y)dy} \qquad [4]$$

Denoting $\ln(10^6)$ by b, a dummy integration variable by y, volumetric water content by θ, and air entry value by *aev*. Soil water characteristic curve plotted as degree of saturation versus soil suction was used in the estimation as will be discussed latter in this paper.

Table 1: Change in material state due to centrifugation.

Description	Value	
	As received	Centrifuged
Gravimetric Water Content, *w (%)*	240	63
Solids Content, s (%)	29	61
Dry Unit weight, γd (g/cm^3)	0.36	0.95
Void Ratio, *e*	6.1	1.5
Volumetric Water Content, θ (%)	86	59
Degree of Saturation, S (%)	100	100

* $\theta = (\gamma d/\gamma w)\ w$, $e = (Gs/\gamma w)-1$, $S = wGs/e$, $s = 1/(1+w)$. (Where $\gamma w = 1 g/cm^3$)

Table 2: Summary of geotechnical index properties.

Description	Value	
	As received	Centrifuged
Specific Gravity, *Gs*	2.34	2.39
Material Finer than 0.075 (%)	96	95
Material Finer than 0.002 (%)	52	52
Liquid Limit, w_L (%)	48	41
Plastic Limit, w_P (%)	21	20
Plasticity Index, I_P (%)	27	21
USCS Classification	CL	CL

*$p = w_L - w_P$

3 RESULTS AND DISCUSSION

3.1 Geotechnical Index Properties

Table 1 summarizes material properties before and after centrifugation. The as received MFT had water content, w = 240% and solids content, s = 29%. The dry unit weight, γd was found to be 0.36 g/cm3 corresponding to an initial void ratio, ei = 6.1 and volumetric water content, θ = 86%. After centrifugation, the material dewatered to w = 63% (s = 62%). The γd was found to be 0.95 g/cm3 corresponding to a void ratio, e = 1.5 and θ = 60%.

Table 2 compares the geotechnical index properties of as received MFT and centrifuged MFT. The specific gravity *Gs* measured 2.34 and 2.39, respectively. These values are lower than those of usual sedimentary soils because of the presence of bitumen (*Gs* = 1.03). The slight increase in the *Gs* of centrifuged MFT is attributed to partial removal of residual bitumen during centrifugation. This process did not result in any significant change in grain size distribution as illustrated in Figure 1. Both samples showed about 95% material finer than 0.075 mm and 52% material finer than 0.002 mm.

On the contrary, bitumen release decreased lubrication thereby marginally decreasing the consistency limits. The w_L and I_P were found to be 48% and 27% for as received MFT and 41% and 21%, for centrifuged MFT. Scott et al. (1985) reported similar reduction in consistency limits of different sludges from oil sand extraction plant. Irrespective of the material treatment through centrifuge, both tailings samples classified as inorganic clay of medium plasticity (CL) based on unified soil classification system.

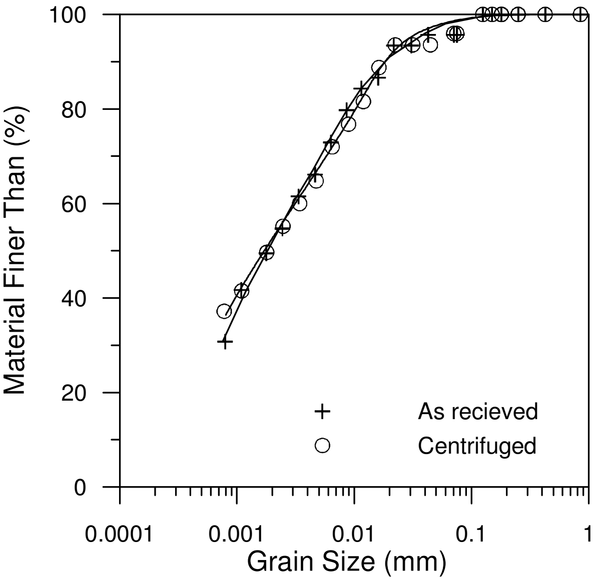

Figure 1. Grain size distribution curve.

3.2 Soil Water Characteristic Curve

SWCC highlights important features of soil when their saturation state is altered. Figure 2 illustrates measured SWCC. The curve comprises of three straight line segments with two inflection points. The initial horizontal line represents no change in material state up to the air entry value (*AEV*) where air first enters into the pore spaces. Thereafter, the second segment known as the transition zone represents a gradual decrease in the water (increase in air). Water migration under the action of capillarity, is the dominant transport mechanism in this zone resulting in a downward slope from the *AEV* up to the residual condition. The third segment is from residual condition to completely dry condition. Water vapour becomes the dominant phase in the transport mechanism and require (10^6 kPa) suction to achieve complete soil desiccation.

Figure 2 gives the SWCC plot. The plot was influenced by γd based on instantaneous volume measurements of the slurry due to substantial volume changes during suction application unlike the gravimetric water content SWCC that is independent of volume changes. The plot shows an *AEV* of 1000 kPa and residual suction of 30,000 kPa at S = 15%.

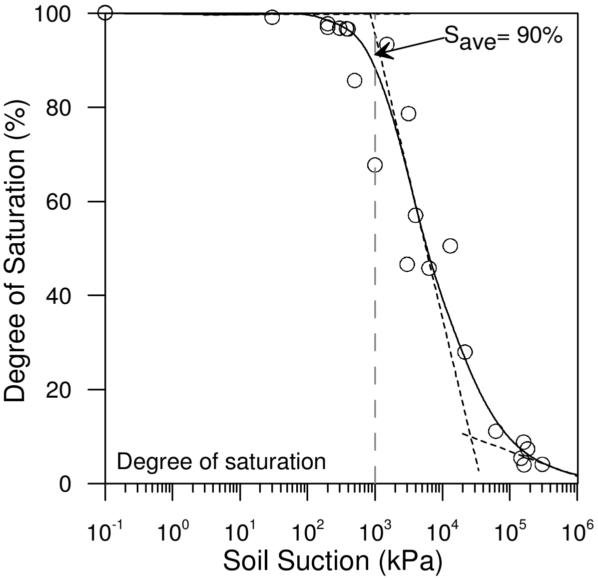

Figure 2: Degree of saturation.

The water in the pores decreases due to drainage through the pore spaces and the average degree of saturation (S_{ave}) reached 90% when most of the pore spaces have been filled with air. Thereafter, the pores compressed and further drainage of water becomes difficult requiring higher suction (10^6 kPa) for air to enter into the matrix. The significantly higher matrix AEV (Figure 2) is associated with gradual decrease in the slurry volume due to increasing suction and this gradual decrease in volume is primarily a result of pore compressibility under capillary action (Marinho, 2005). The use of actual volume measurements better describes the desaturation behaviour of tailings.

3.3 Shrinkage Curve

Figure 3 shows shrinkage curve for centrifuged MFT. The theoretical lines representing the various average degrees of saturation were calculated from basic phase relationship applying G_s = 2.39. The sample was initially completely saturated and subsequently desaturated by applying different suction values. The void ratio and water content corresponding to various applied suction were determined as described earlier in this paper. The data shows a "J" shaped curve in a progressive drying pattern (from e = 1.5 to e = 0.46). The curve comprises of an initially saturated straight line portion up to the w_s (normal shrinkage zone) thereafter, the second horizontal portion from the w_s to completely dry condition (residual shrinkage zone). The curve indicated a gradual decrease in water content in the normal shrinkage zone until a point close to the plastic limit w_p where there is slight deviation from S =100%. Theoretically, S = 100% up to the shrinkage limit where air intrusion in the pore spaces occurs. The slight deviation from theory experienced is attributed to experimental error associated with measurement of precise void ratio e.

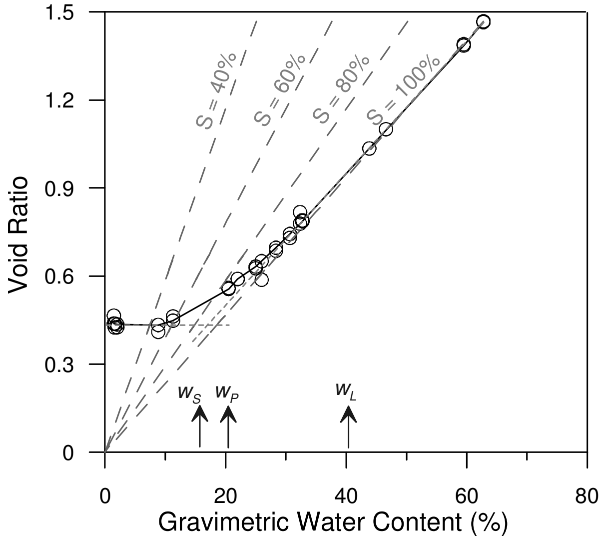

Figure 3: Shrinkage curve.

The data indicated that in the normal shrinkage zone, volume decrease in soil is equal to volume of water lost resulting in a 45° straight line (Ito and Azam, 2012). This reveals that drainage occurs primarily in the normal shrinkage zone whereas in the residual shrinkage zone, air enters into the pores near the shrinkage limit, fills the void spaces accelerating water loss. Water reduction exceeds volume reduction in the residual shrinkage zone and gradually transitions to zero volume reduction beyond the shrinkage limit. The bulk of this volume decrease occurred in the former zone (normal shrinkage zone). The combination Figure 3 and SWCC better explains the behavior of this class of material. The accurate combination is with the degree of saturation plot which shows complete saturation up to the shrinkage limit (air intrusion) occurring at *AEV* = 1000 kPa and after complete air intrusion; the S_{ave} decreased to 90%.

3.4 Hydraulic Conductivity

Figure 4 presents hydraulic conductivity data. The saturated initial hydraulic conductivity at e = 1.5 was determined to be 2 x 10^{-10} m/s based on experimental data not presented here in this paper. Thereafter, the hydraulic conductivity decreased with decreasing void ratio reaching a value of 2 x 10^{-11} m/s at e = 0.5.

The k_{unsat} decreased by several orders of magnitude (10^{-10} m/sec to 10^{-20} m/sec) with increasing suction and closely followed the SWCC presented in Figure 2. As expected, the curve comprises of initial horizontal line up to the air entry value followed by a sharp decline linearly on a downward slope with increasing suction. The *S*-based curve revealed the actual k_{unsat} function for this class of material and better represents the field situation requiring higher suction values for meaningful desaturation

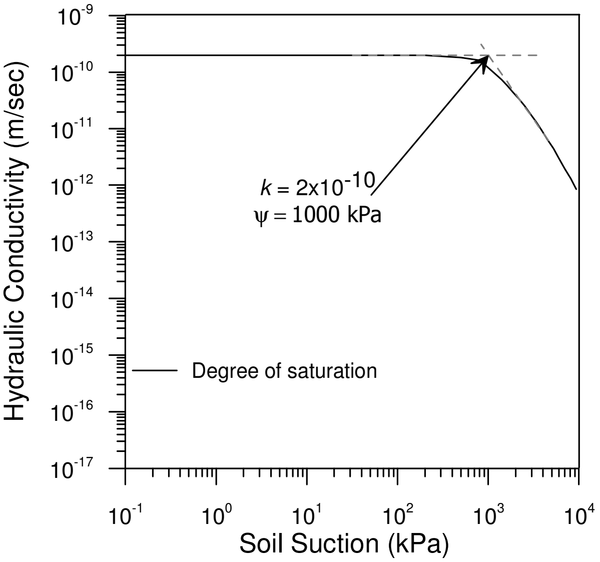

Figure 4: Unsaturated hydraulic conductivity.

4. SUMMARY AND CONCLUSION

Knowledge of unsaturated properties is paramount for geotechnical engineers in developing surface deposition schemes for centrifuged oil sand fine tailings. The main conclusions of this research are summarized as follows:

The investigated material was characterized by fine grained clay of medium plasticity. The moderate liquid limit (40%) and plastic limit (20%) with a low shrinkage limit (15%) indicated a moderate water absorbing and retaining capacity of the clay. The correct way of quantifying SWCC for a high volume change material to model deposit desaturation is degree of saturation plot computed with reference to instantaneous volume of soil thereby accommodating all volume losses as suction increases this plot showed an *AEV* of 1000 kPa. The shrinkage curve showed a “J” shaped curve with the bulk of the volume loss occurring in the normal shrinkage zone. The initial hydraulic conductivity at 60% solid content (e =1.5) was found to be 2 x 10^{-10} m/s. This value decreased with several orders of magnitude (10^{-10} m/s to 10^{-20} m/s) as suction increased.

5. ACKNOWLEDGEMENT

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