

THE EFFECT OF FREEZING AND THAWING ON  
THE DEWATERING OF OIL SANDS SLUDGES<sup>1</sup>

by

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**Abstract.** Oil sands processing operations in northeastern Alberta generate  $25 \times 10^6$  m<sup>3</sup> of water-fines mixtures (sludge) per year. The fines settle in several weeks but will not consolidate to more than 35% solids, even over centuries. Freezing and thawing the oil sands sludge led to rapid dewatering. One cycle of freezing and thawing caused 15, 25, 35, and 45% solids sludge to reach 35, 39, 48, and 51% solids content, respectively. Subsequent freeze-thaw cycles, up to a total of three, caused less rapid increases in solids content. The maximum concentration of solids by freezing and thawing, even after repeated cycles, was 60%. The amount of dewatering due to freeze-thaw can be confidently predicted by knowing only the initial solids content. The freezing time for each sludge concentration was monitored to compute proportionality coefficients required to predict freezing depths under field conditions.

Additional Key Words: slurries, tailings, Neuman-Stefan proportionality coefficient, solids concentration.

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### Introduction

The enormous volumes of oil sands tailings generated by two extraction and upgrading plants located near Ft. McMurray, Alberta, pose economical and ecological problems in land reclamation. Approximately 170,000 barrels of synthetic oil per day is produced from 400,000 tonnes of oil sands; the tailings stream amounts to 180 million metric tonnes per year and currently occupies 25 km<sup>2</sup>.

Sludge, a mixture of fines (<22 microns diameter), water, and residual bitumen, is the most difficult component in the tailings stream to handle for reclamation. The sand portion of the tailings stream segregates from the slurry upon deposition at the edge of the tailings pond and is used to build the pond dikes. Thin sludge, at approximately 5% solids content, flows into the pond and settles to a solids content of 20 to 30% within two years. The sludge volume now exceeds 250 million m<sup>3</sup> and is increasing at the rate of 20 million m<sup>3</sup> per annum. Consolidation to 80% solids, representing a firm, stable surface, is expected to take tens of thousands of years if left to mature under natural conditions (Scott et al. 1985).

The most recent research at the Alberta Environmental Centre explores the possibility of using vegetation to dewater relatively thin layers of sludge (<3 m) through evapotranspiration (Johnson et al. 1989). Two species of hydrophilic plants, reed canary

grass (Phalaris arundinaceae) and dock (Rumex occidentalis) have proven successful on a small scale. However, to establish plants quickly and achieve complete dewatering in one summer, a bed of at least 50% solids sludge is needed. Among the alternatives to increase the solids content of oil sands sludge from 30% to 50%, dewatering by freezing and thawing appears to be most efficient and economical.

Freeze-thaw originated as a dewatering technique in temperate climates where biological sludges were concentrated from 3-5% solids to >30% solids (Downes, 1939, Clements et al. 1950, Bishop and Fulton, 1968, Logsdon and Edgerly 1971, Rush and Stickney 1979). Preliminary tests on the effect of freeze-thaw on oil sands sludge caused a large increase in percent solids (Johnson et al. 1989). Since Ft. McMurray has long, cold winters, freezing and thawing could be a low cost, efficient means of dewatering oil sands sludge on a field scale.

To develop a rational engineering procedure for freeze-thaw dewatering, the environmental factors that control frost penetration must be quantified (Martel 1989). The Neuman-Stefan formula for predicting the formation of ice on lakes should also apply to liquid sludges (Reed et al. 1986). This formula requires information on the freezing index of a location (Rush and Stickney 1979) and a proportionality coefficient for the material being frozen or thawed, which depends upon its

thermal conductivity and latent heat. One objective of the research described here is to calculate a Neuman-Stefan coefficient for oil sands sludge.

### Materials and Methods

This freeze-thaw experiment on oil sands sludge was carried out under pilot plant scale conditions: experimental units were barrels with a capacity volume of approximately 60 L; sludge was diluted with water to four solids contents using a rotor attached to an electric drill; the sludge was frozen by placing the barrels inside a walk-in freezer and thawed by removing them from the freezer and letting them stand in a open work space.

### Experimental Units

The experimental units were constructed by placing an interior metal drum into an exterior polyethylene drum (Figure 1). To simulate natural freezing conditions, where the freezing front progresses from the surface downward, insulation was inserted between the two containers and at the bottom of the inside barrel. A total of 12 experimental units were constructed to allow three replicates of four sludge dilutions.

### Sludge Dilution Mixes

Four sludge dilutions were chosen to represent a wide range of solids contents in the oil sands tailings ponds: 15, 25, 35, and 45% (solids, dry weight basis). These were prepared by

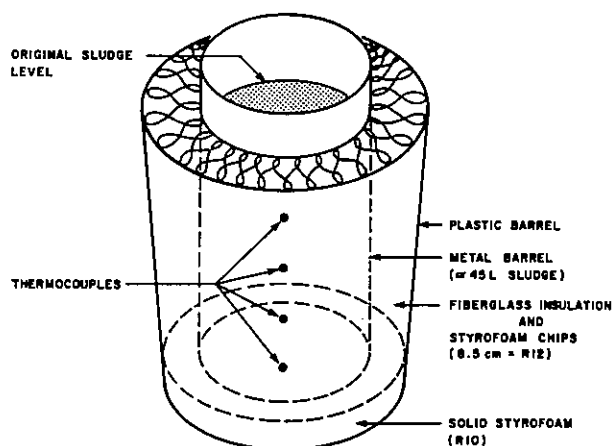


FIGURE 1. EXPERIMENTAL UNIT FOR FREEZING AND THAWING OILSAND SLUDGE PLACED INSIDE AN INSULATED BARREL

sequentially diluting a batch of high solids content sludge with reverse-osmosis, deionized water. After completing each dilution, three experimental units were filled to a depth of 46 cm providing an initial volume of 45 L. Sludge sampling inside the experimental units was conducted prior to initial freezing and after every freeze-thaw cycle. All replicates were sampled at three depths: one-sixth, one-half and five-sixths of the total sludge depth. The sample sites inside the experimental unit were selected randomly, using a random number table where each number corresponded to a grid intersection placed over the sludge surface. The percent solids of the samples were determined by mass measurements before and after oven-drying (105°C for 24 h).

### Freezing and Thawing

The twelve units were placed in a completely random pattern inside the freezer. Unit location

was randomized each time the freezing cycle was repeated. The freezer temperature was kept at  $-24^{\circ}\text{C}$  throughout the experiment. One replicate of each sludge dilution was fitted with four type T thermocouples connected to a Campbell Scientific CR 10 micro-logger (Figure 1). Temperatures were recorded at four depths within each sludge dilution mix. When each thermocouple within the frozen sludge registered a maximum temperature of  $-20^{\circ}\text{C}$ , the freezing period was considered complete. All units were removed from the freezer at the same time for the thaw stage.

Two days into the thaw period the units were covered with lids to prevent moisture loss by evaporation. The thaw period was considered complete once all units registered a minimum temperature of  $20^{\circ}\text{C}$ . The surface bitumen was skimmed off, and the thaw water was removed and measured volumetrically. The experimental units were then sampled for solids contents in the manner described above.

The freeze-thaw process was repeated three times.

#### Freezing Coefficient Determination

The Neuman-Stefan formula allows for the calculation of total freezing depth (Reed et al. 1986):

$$x = m (\Delta T \cdot t)^{1/2} \quad (1)$$

where:

$x$  = depth of freezing [cm],  
 $m$  = proportionality coeffi-

cient depending on the thermal conductivity, density, and latent heat of the material being frozen [ $\text{cm}/(^{\circ}\text{C}\cdot\text{day})^{1/2}$ ]  
 $\Delta T \cdot t$  = freezing index [ $^{\circ}\text{C}\cdot\text{day}$ ],  
 $\Delta T$  = difference between the freezing temperature and average daily ambient temperature [ $^{\circ}\text{C}$ ],  
 $t$  = time period of concern [days].

By rearranging the formula the proportionality coefficient for oil sands sludge can be estimated:

$$m = \frac{x}{(\Delta T \cdot t)^{1/2}} \quad (2)$$

The fourth thermocouple, placed at approximately 8 cm above the bottom of the sludge, was used as the freezing depth to calculate the proportionality coefficient from equation 2. Only data from the first freeze-thaw cycle was used in this calculation.

#### Results

The sludge concentrations before and after each freeze-thaw cycle are given in Table 1. Individual values represent the average of samples taken at three depths within three replicates of each mix. (The exceptions to this are one replicate each from mixes 1 and 2, representing 45 and 37% solids, respectively. In both cases the metal drums failed due to pressures exerted during the freezing cycle).

There were some small differences between the intended and actual solids contents, but the largest variance was only

2.4% (mix 2 was intended to be Table 1. Average percent solids before and after freeze-thaw cycles.

Mix #	Original Solids Content (%)	Solids After Freeze-Thaw Cycle		
		1	2	3
1	45.4	51.3	57.8	60.4
2	37.4	47.9	55.1	59.6
3	26.2	38.7	46.4	52.6
4	16.3	35.4	46.2	49.5

35% and, in fact, reached 37.4%). Overall, the desired range of solids content was achieved.

While each freeze-thaw cycle resulted in a progressive dewatering of each mix, it is apparent that the first cycle was more effective than the second which, in turn, was more effective than the third (Figure 2). And even more noticeable is the effect of initial solids content on the amount of dewatering. Mix 1, starting at 46% solids, had a cumulative increase of only 35% over three freeze-thaw cycles; mix 4, on the other hand, starting at 16% solids finished after three cycles at 49.5% solids, a 200% cumulative increase! The two intermediate dilutions also had intermediate cumulative increases in solids content (Figure 2), indicating a correspondence between initial and final solids content.

The relationship between initial solids content of oil sands sludges and the degree of dewatering through freeze-thaw

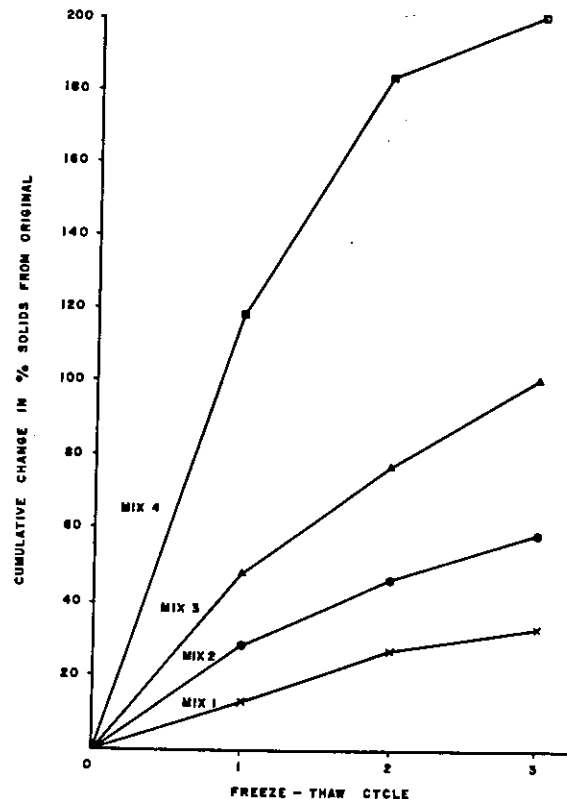


FIGURE 2. CUMULATIVE INCREASE IN % SOLIDS THROUGH 3 FREEZE-THAW CYCLES

cycles is exponential (Figure 3). Low initial solids content results in a large amount of dewatering. As the solids content increases the rate of dewatering drops quickly. The exponential decrease in rate of dewatering is independent of freeze-thaw cycle, which acts only as a determinant of "initial" solids content. The prediction of final solids content of oil sands sludge undergoing freeze-thaw can be confidently made without considering the previous freeze-thaw history of the sample:

$$Y = 365.3 (0.93)^x \quad (3)$$

where:

Y = final solids content [%]  
x = initial solids content [%]

The coefficient of determination ( $r^2$ ) is high - .95. Only 5% of the variation in final solids content is not accounted for by the initial solids content.

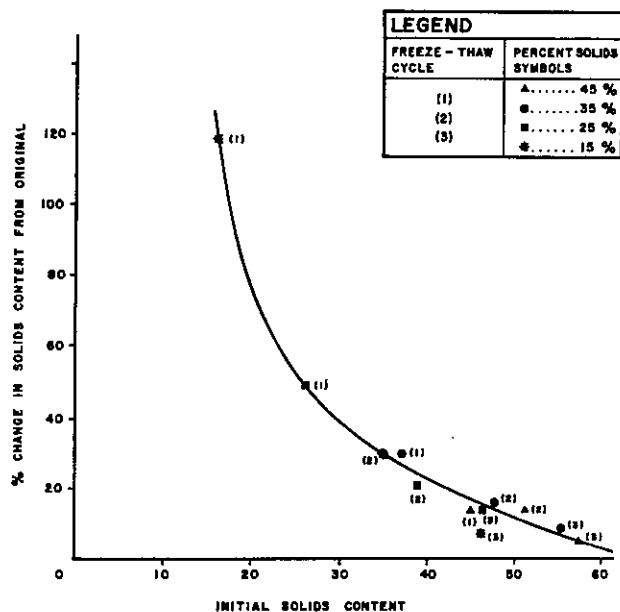


FIGURE 3. CHANGE IN FINAL SOLIDS CONTENT IN RELATION TO INITIAL SOLIDS CONTENT AND FREEZE-THAW CYCLE

The relationship between the initial solids content of the sludge and the calculated Neuman-Stefan proportionality coefficients are given in Table 2. For mixes with solids contents less than 45%, there is a linear relationship between the Neuman-Stefan proportionality constant ( $m$ ) and the initial solids content such that:

Table 2. Neuman-Stefan proportionality coefficients for oils and sludge mixes.

Original solids content (%)	Freezing Time hours	Freezing Time days	Proportionality constant ( $m$ )
45.4	125	5.2	3.41
37.4	177	7.4	2.86
26.2	195	8.1	2.73
16.3	217	9.0	2.59

$$m = 0.0128 x + 0.998 \quad (4)$$

where:

x = initial solids content [%]

The coefficient of determination is 0.996. For the mix with an initial solids content of 46%, the proportionality coefficient is much higher than that which might be predicted by equation 4.

### Discussion

The experimental results confirm previous findings at the Alberta Environmental Centre that the freeze-thaw process increases the solids content of oil sands sludge (Johnson et al. 1989). The mineral fines originating from bitumen extraction processes have less than two percent organic contents (Scott et al. 1985), but act like sewage sludges when subjected to freeze-thaw conditions (Rush and Stickney 1979). The sludge mixtures with the lowest solids content, mineral or organic, dewater to the greatest extent. There is an exponential decrease in the amount of water released

as pre-freezing solids contents increase. Multiple freeze-thaw cycles cause progressive loss of water, but the same exponential relationship between original solids contents and amount of water loss applies. That is, the second and third and more freeze-thaw cycles are acting on mixtures with higher and higher solids contents and, therefore, there is an exponential decrease in the amount of water lost with each cycle.

The oil sands sludges tested in this experiment reach a maximum of approximately 60% solids contents, if subjected to enough freeze-thaw cycles (Table 2, Figure 3). On an operational basis the sludge accumulating at the bottom of the tailings pond on the Syncrude Canada Limited lease reaches a maximum of 35% solids in two to five years; according to equation 3 and Table 2, one winter of freezing and thawing will yield sludge at 45% solids content. This has been corroborated in a recent field experiment conducted in Ft. McMurray (Johnson et al. 1989).

One proposed reclamation scheme for oil sands sludge involves the use of plants to dewater sludge through evapo-transpiration. To successfully establish plants a relatively firm seed bed is needed, corresponding to approximately 50% solids content. One winter will produce the desired seed bed conditions, a sludge surface at 45-50% solids, depending on the depth of sludge frozen, the type of drainage employed, and the amount of moisture lost through

evaporation prior to the establishment of the plants.

The proportionality coefficients for the Neuman-Stefan formula have a linear relationship to the original solids contents. This formula is used to estimate the depth of freezing achievable in a selected time period (Reed et al. 1986) and can now be used confidently to design facilities and treatment programs for oil sands sludges, where the thickness of individual layers and the time periods for freezing are critical constraints. Work is currently underway at the Alberta Environmental Centre investigating a dynamic (multiple layers) freeze-thaw process for oil sands sludges.

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