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The use of methylene blue index in mine and tailings planning

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Abstract

Methylene blue index (MBI) is a powerful chemical probe with a strong affinity for the hydrophilic surfaces of clay minerals. This high degree of affinity means it outcompetes the majority of other cations to fill all cation exchange sites and to cover the clay surfaces. As a result of this affinity and coverage the MBI correlates well with other important properties such as required flocculant dose and Atterberg limits. Furthermore, MBI is easy to measure, requires small volumes of sample and can be used to predict the properties of mixtures of streams. These attributes make it an extremely powerful tool in exploration and mine planning as an understanding of the MBI of a core can be then used to predict properties further downstream. This paper will highlight experience with using MBI in various mining industries to predict settling properties, understand flocculant dose, and predict relative liquid limits of different tailings streams. This paper will also highlight how the authors suggest the use be extended to better understand potential tailings and closure risks during the exploration phase of a mine.

Keywords: *methylene blue index, clays, tailings, flocculant dose*

1 Introduction

It is very important to ensure that there is a good understanding of the clays in the geological modelling. This can be achieved by measuring clays in the geological cores. Clays can cause issues in both underground mining (tunnel wall integrity) and surface mining (pit wall integrity). Quite often, the geological model is the main indication to the plant on the type of ore being sent for processing.

The processability of ore and the dewatering and consolidation behaviour of tailings are influenced by the surface properties of their associated minerals, with clay minerals forming an appreciable portion of both tailings and ores. In the context of oil sands, many studies have shown that kaolinite and illite are the predominant clay minerals (Masliyah et al. 2013). However, various other clays such as smectite, chlorite, vermiculite, and mixed-layer clays such as kaolinite-smectite and illite-smectite have also been identified (Kaminsky et al. 2009). This diversity in clay content, types, and particle size distributions introduces challenges when processing oil sands ores and tailings. For example, excess clays inhibit bitumen flotation and deteriorate the froth quality through the mechanism of slime-coating (i.e. attachment of fine clays on bitumen droplet) (Tamiz et al. 2015). In addition, if left untreated, the fine clays remain suspended in tailings for extended periods due to the electrokinetic forces and high water holding capacity, jeopardising tailings reclamation processes (Wang et al. 2023) and leading to the accumulation of more than 1.3B m^3 of fluid fine tailings (FFT) in the oil sands region. Furthermore, the efficacy of polymer-based technologies developed for tailings treatment is directly affected by the type and quantity of clay minerals present (Motta et al. 2018).

Several methods have been developed to estimate the clay activity in soil, including Atterberg limits, coefficient of linear extensibility, cation exchange capacity (CEC), and methylene blue index (MBI). The MBI is the most widely used method in oil sands due its simplicity and accuracy in indicating the activity and specific surface area of clays in ore, froth, and tailings (Li & Xu 2020). Conducting an MBI test requires only

0.2–5 g of sample (depending on the amount of clay present) which is comparable to the amount required for powder diffraction and is significantly smaller than the amount of sample required for Atterberg limit tests. Furthermore, the test is inexpensive (CAD 100/sample or less), can be performed without specialised lab equipment, and can be conducted easily by anyone with basic wet chemistry skills. These attributes make it an ideal 'first pass' indicator of clay content and is why every oil sands operator currently measures the MBI throughout their tailings ponds on an annual basis and most operators also include MBI data in their mining block models. However, despite its widespread use, there remains a degree of confusion as to what MBI is and how it can actually help planners and engineers cope with the challenges of excess clay.

MBI estimates the clay activity by measuring the amount of methylene blue (MB) cations required to cover the total available surface of the negatively charged clay particles in a sample. In its aqueous state, MB is a strong cationic dye, $C_{16}H_{18}N_3S^+$, with a molecular structure presented in Figure 1 (Cenens & Schoonheydt 1998). The dimensions of an MB molecule are 17 Å × 7.6 Å × 3.25 Å, and it is estimated to cover a surface area of approximately 130 $Å^2$. Consequently, the sample's surface area can be calculated by assuming that each titrated molecule covers 130 $Å^2$ according to Equation 1 (Hang & Brindley, 1970):

$$SA = MBI \times A_m \times 6.02 \times 10^{-2} \tag{1}$$

where:

SA = surface area of the sample in m^2/g.

MBI = methylene blue index in meq/100 g solids.

A_m = area per MB molecule (130 $Å^2$).

MB adsorbs onto anionic clay surfaces via two mechanisms:

1. Cation exchange within the alumino-silicate lattice which is the predominant mechanism.
2. Attraction of Van Der Waals forces or chemisorption (hydrogen bonding) with the silanol (SiOH) and aluminol (AlOH) groups of the alumino-silicate lattice (Yukselen & Kaya 2008).

Therefore, the adsorption process of MB can be described as a two-phase process according to (Jacobs & Schoonheydt 2001). The first phase is the instantaneous adsorption phase, which is independent of clay type and involves a rearrangement of MB molecules in the hydration sphere of the clay platelets, expressing the high affinity of clay minerals for MB cations. The second phase is the MB distribution phase which involves the breakdown of MB aggregates and displacement of water molecules from the clay surface (i.e. adsorption on edges and external surfaces before migrating to the interlayer regions if they are accessible). The distribution of MB molecules depends on the balance among the MB–surface interactions, MB–MB interactions, and interactions among clay particles. The strength of MB's interaction with clay surfaces is affected by the colloidal nature of the suspension and the clay characteristics such as charge density and charge distribution. Clay minerals with tetrahedral substitution and a large basal surface area (e.g. vermiculites and illite) exhibit a strong MB–surface interaction, retaining MB molecules on the surface. Conversely, clay minerals with octahedral substitution (e.g. montmorillonite) and small clays with a large edge surface (e.g. kaolinite) show a weaker MB–clay interaction, with MB–MB interactions and interactions among clay particles dominating, causing MB molecules to remain in the hydration layer of the particles, thus remaining mobile and not in direct contact with the surface (Bergaya et al. 2006; Jacobs & Schoonheydt 2001). Additionally, Greathouse et al. (2015) investigated the MB adsorption on the basal surfaces of kaolinite using both quantum and classical molecular simulation approaches. Their findings revealed that MB demonstrated a weaker adsorption energy, indicating MB monomer adsorption is influenced more by strong interfacial forces than by the hydration properties of the dye. In short, the adsorption of MBI is complex but it does absorb on every clay surface and absorbs more strongly on the surfaces known to be most problematic in tailings and flotation processes (illitic/smectitic clays).

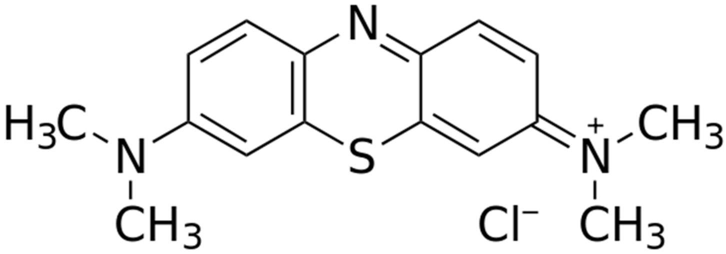


Figure 1 Structure of methylene blue (MB)

2 Use of methylene blue index as pain index

The attribute of adsorbing to every clay surface but more strongly on smectitic clay surfaces means that MBI can be a particularly effective ‘pain’ index to quantify challenges in solid/liquid separation. In other words, elevated MBI values indicate greater process difficulty or ‘pain’ in the process, and conversely, lower values suggest a less challenging process.

One way that this technique can be used in the initial building of the mine block model has also been seen that different clays are located in different areas of the mine. Therefore, operations may be able to manage the clay interference in operations in one section of the mine. But when the shovels move to a different area of the mine, the types and quantity of clays may change and without understanding their activity, then plant operations can struggle, and recovery can suffer. Typical exploration geology will measure the valuable resource (i.e. bitumen content, copper content etc.) by various techniques but good measurement of the clay fractions can be hard to obtain. The oil sands experience shows that MBI is a cheap technique, easily compatible with other core analysis tools and provides an indicator of where clays may be problematic.

Table 1 shows the typical MBI values observed in different oil sands deposits along with standard clay minerals such as kaolinite and bentonite. Typically, oil sands ores are characterised by bitumen content and percentage of the solids <44 microns (grade and fines content). Work by Yang & Sedgwick (2014) and Liu et al. (2004) demonstrated that while these are important variables, the MBI provides an added indicator which is particularly important when differentiating ores with higher fines. Specifically, they found if the MBI of the ore was higher than 1.5 meq/100 g, one always ended up with poor processing ore whereas the same grade & fines with an MBI <1 meq/100 g would typically process well and be less sensitive to changes in water chemistry.

Another indicator of how MBI can work as a pain index is related to the typical rule of thumb in soil mechanics, which indicates that soils with more than 30% clay or more than 1% bentonite/swelling clay will be dominated by the clay size fraction (Mitchell & Soga 2005). This rule of thumb can be hard to apply in practice when soils often have a mixture of different types of clay minerals. Interestingly the expected MBI for a deposit containing 30% kaolinite or a deposit containing 1% bentonite works out to be the same – an MBI of ~1 meq/100 g. Even more interestingly is ~1 meq/100 g is where the observed change in processability and water chemistry sensitivity is observed in oil sands.

With these experiences in mind, the authors suggest that using MBI to evaluate ores and potential foundation sites for dams/dykes might help highlight when/where clays may be an issue. Areas with an MBI >1 meq/100 g should then be evaluated in more detail to determine whether the material is indeed clay dominated and whether there may be impacts of clay content on the process/dam design.

Table 1 **Typical methylene blue index (MBI) values found in oil sands deposits along with soil containing standard clay minerals**

Deposit	Typical MBI (meq/100 g)	Active surface area (m^2/g)
High grade/good processing ore	0.5	4
Low grade/reasonable processing ore	0.9	7
Low grade very poor processing ore	1.5	12
Pure kaolin deposit	3	23
Typical fluid fine tailings (average)	7	55
Pure bentonite deposit	102	799
Soil with 30% kaolin	1	8
Soil with 1% bentonite	1	8

2.1 Mineral processing

In mineral processing, the effect of clay on recovery is very important. Clays can affect solid–liquid separation in mixing tanks and can decrease the flotation effectiveness. Depending on the flow sheet, these problems can severely impact mineral recovery. Several different unit operations can be impacted by clay rich ore including choking problems in the crushing plant, viscosity problems in the milling and scrubbing plant, and blinding of screens (Figure 2).

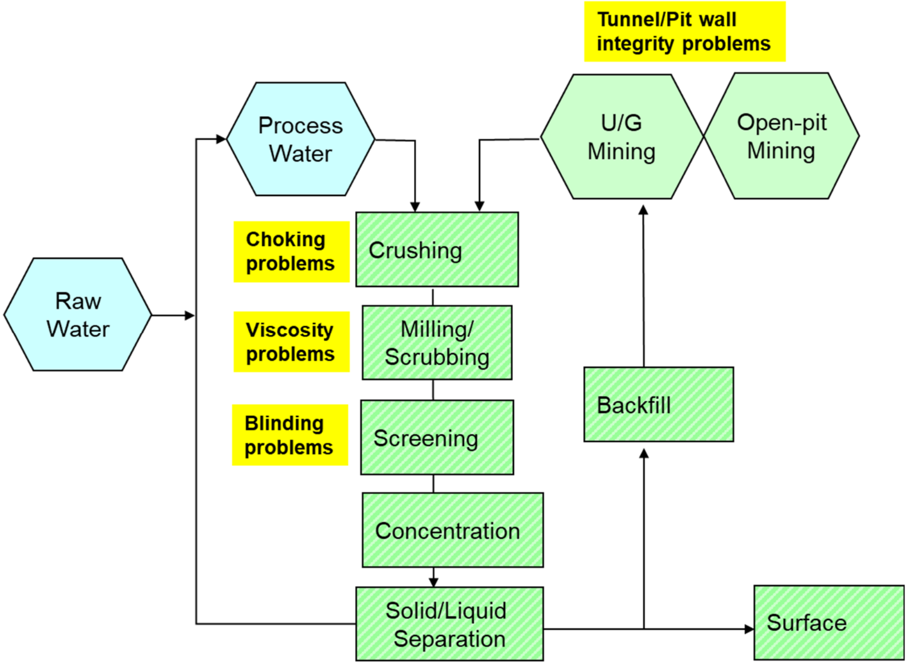


Figure 2 **Effects of clays in mineral processing (solid/semi-solid state)**

After the ore becomes a slurry, clays create different problems in the plant (Figure 3). In the concentration of the ore, there can be separation problems particularly in the area of solid–liquid separation (gravity separation, flotation).

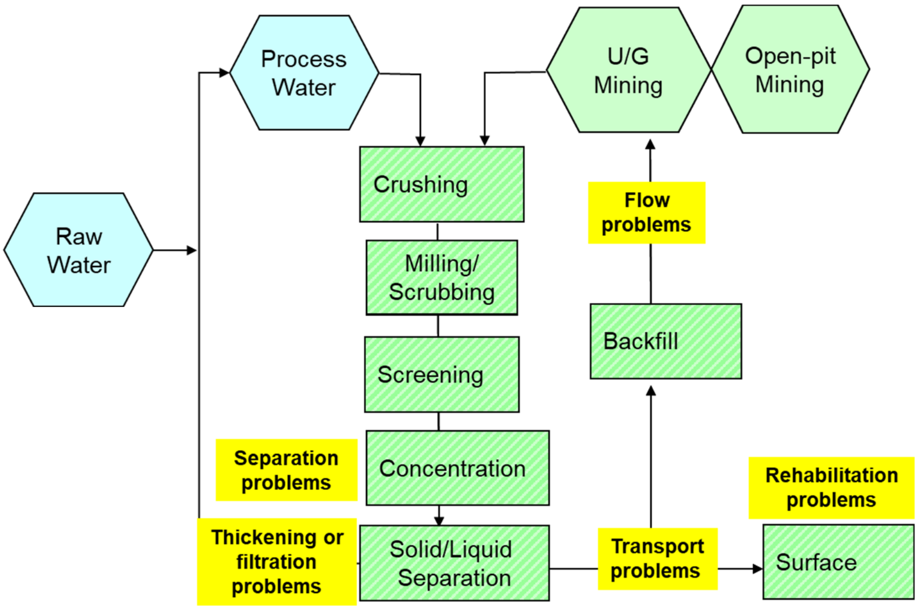


Figure 3 Effect of clays in mineral processing (slurry state)

As an example, in the oil sands bitumen recovery process, there are several process unit operations that can be affected by high MBI (Omotoso & Melanson 2014). In the primary separation cell (PSC), heat and minimal dilution are used to aid in the gravity settling process. When there is a high clay condition, the viscosity in the PSC can increase (gelling) such that normal gravity settling cannot occur. In this case, it can be very difficult for operations to correct without a loss of recovery. It also means that a greater amount of bitumen ends up in the flotation circuit. However, the clays impede the proper flotation of the bitumen, causing further recovery and often sending bitumen to the tailings streams. These consequences are very similar to other mining processes. MBI has been used to identify these challenges in oil sands, and recently it has been found useful in other industries such as platinum flotation recovery circuits where high concentrations of talc clays accumulate.

2.2 Tailings dewatering

MBI is also useful in providing an insight into the dewatering characteristics of clay dominated streams. There are two challenges in dewatering clays: firstly, their small size which leads to increased surface area and secondly, their non-neutral layer charge on their surfaces. The effect of size can be seen in Figure 4 where spheres of different sizes are assumed to be coated with a one micron layer of water. As shown, the effect of this coating is negligible on particles larger than 20 microns but below 20 microns the surface area of the smaller particles dramatically increases the amount of water held by the particles and thus decreases the solids content.

For clays, this effect is more pronounced as the surfaces typically have a net negative layer charge. This negative charge is typically satisfied by the presence of cations but because these charges are often diffuse and not equal to an integer, there ends up being excess charge that needs to be satisfied. This charge is satisfied by the orientation of the water molecules and thus there is a plane of water around the clays that is heavily influenced by the surface charge characteristics.

Bringing this into a dewatering context, in dilute clay suspensions, the clays dewater like any other non-charged particle but as the water layer becomes thinner electrostatic forces come into play.

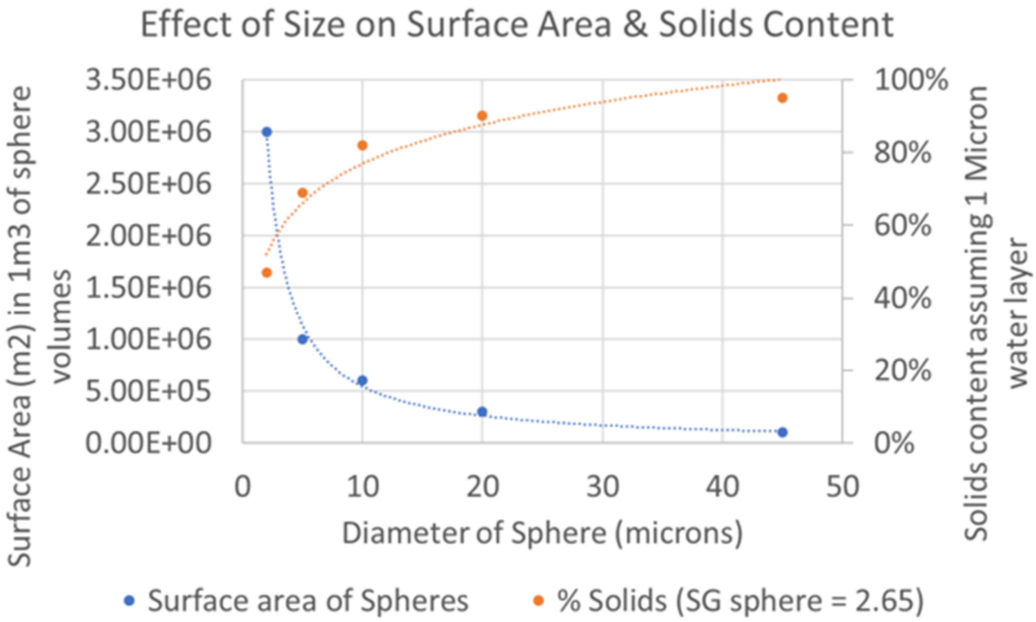


Figure 4 Effect of size on surface area and solids content

MBI provides insights into the water active surface area as shown in Equation 1. The water layer thickness adsorbed on clay mineral surfaces is influenced by both the total water content and the active surface area of clays within a given sample (Dolinar & Macuh 2016). With the assumption of a monolayer of MB adsorbed on all available clay surfaces, the adsorbed water layer thickness can be determined using Equation 2 (Li et al. 2021).

$$Water\ layer\ thickness\ (nm) = (Total\ water\ content\ (\tfrac{g}{g}) \times 10^9 \tfrac{m^2}{m})/(SA\ (\tfrac{m^2}{g}) \times Density\ of\ water\ \left(10^6 \tfrac{g}{m^3}\right)) \qquad (2)$$

Table 2 summarises the tested MBI and liquid limit values of various clay mixtures and corresponding calculated actively clay surface area and water layer thickness. As shown, the actual thickness of the water layer at the liquid limit varies significantly but it is in the order of magnitude of 10 nm. Typical dewatering technology struggles to reach the liquid limit of a material as the yield stress rapidly increases above this point as the behaviour transitions from fluid to solid.

Table 2 The measured methylene blue index (MBI) and liquid limit values for various slurry samples and the corresponding calculated active clay surface area and water layer thickness

Slurry sample	MBI (meq/100 g)[1]	Liquid limit[1]	Solids wt% at liquid limit[2]	Active clay surface area (m²/g)[3]	Water layer thickness (nm) at liquid limit[4]
Centrifuge cake	11.8	64%	61	92.38	6.9
Thickened tailings	5.5	25%	80	43.06	5.8
Bentonite	77.0	656%	13	605.00	10.8
Ball-kaolinite (kaolinite/bentonite mix)	9.8	67%	60	76.72	8.7
Leda clay	6.6	67%	60	51.67	12.9
High clay fluid fine tailings	13.0	48%	68	101.77	4.7

[1] MBI and liquid limit values were tested by NAIT TACEES lab.
[2] Solids% at liquid limit values were calculated based on measured liquid limit values.
[3] Surface area of tailings sample was calculated according to Equation 1.
[4] Water layer thickness was calculated according to Equation 2.

The implications of this order of magnitude are shown in Figure 5. If pure kaolinite slurries (typical MBI < 5 for pure kaolinite streams) were tested in a thickener it would be expected to get thickener underflow densities above 65wt% solids from your dewatering technology. However, if the stream was a ball-kaolinite (mixture of bentonite and kaolinite) dewatered in a similar manner the expected underflow solids content would be ~10wt% lower simply due to the increased surface area of the particles involved.

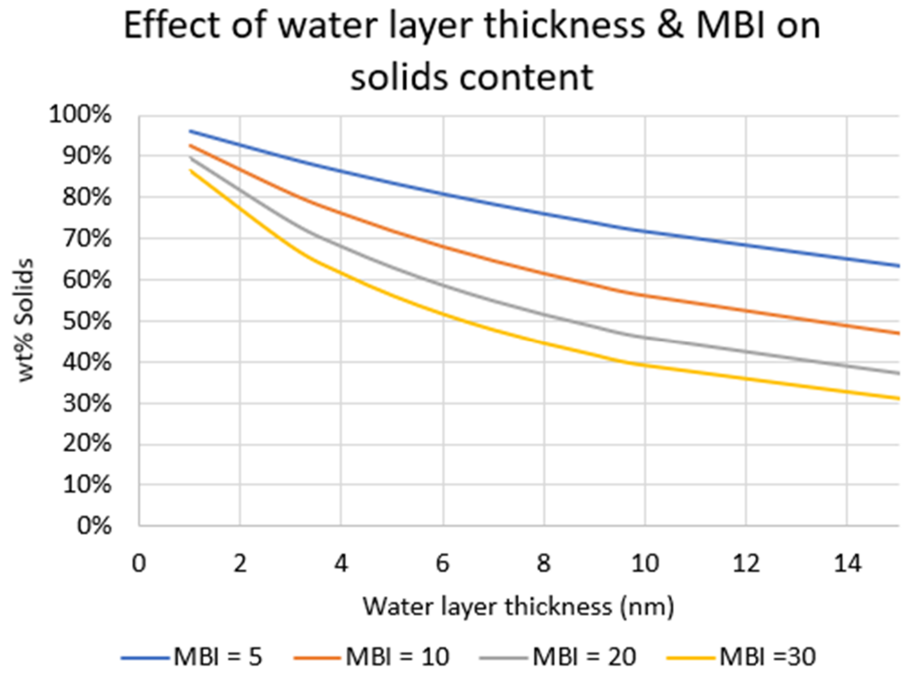


Figure 5 Solids content as a function of water layer thickness at various methylene blue index

Figure 6 shows the box and whisker plot of the MBI found in different tailings from the author's experience. Some mineral tailings, such as copper and platinum, have very tight distributions partially due to their MBI being so small. Whereas the kimberlite, mineral sands and oil sands have higher average MBI and much wider distributions. The range in the MBI value highlights the challenges in operation for these clay rich mineral types as processing material at an MBI of 5 will be very different from processing and MBI of 15 or 20.

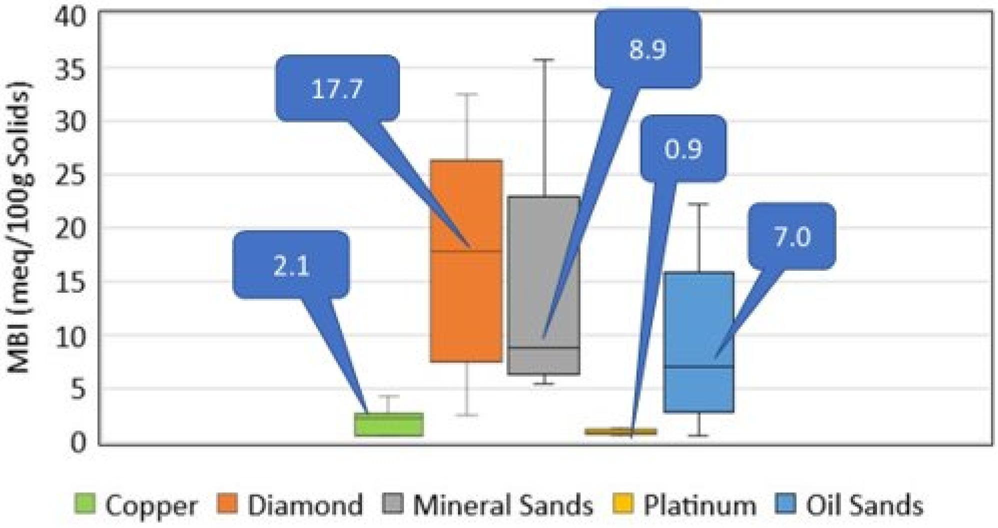
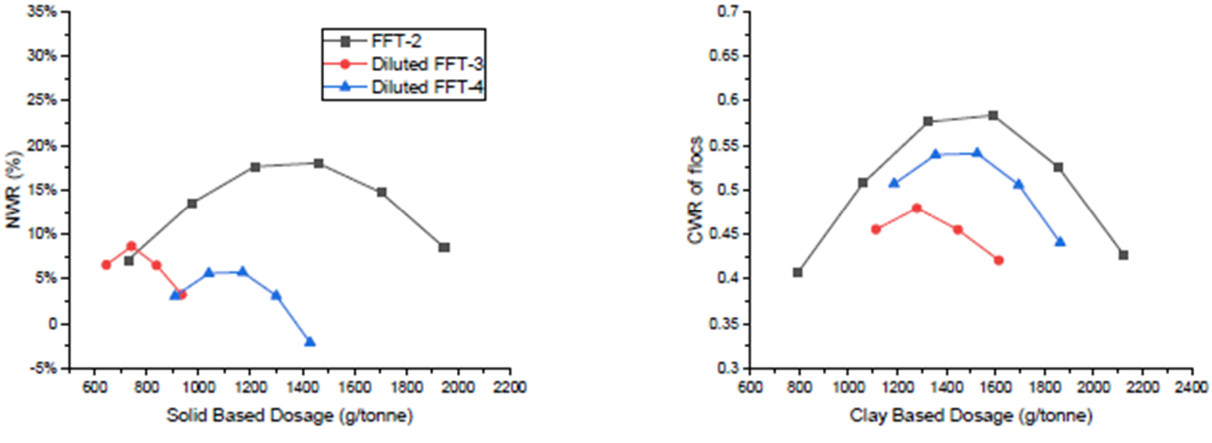


Figure 6 Methylene blue index (MBI) of different tailings types

2.3 Required flocculant dosage

In addition to being able to predict challenges in achieving a particular underflow density, MBI has also been used to normalise the flocculant dosage required between different samples. Experience at Suncor (Li et al. 2021) demonstrates how the required flocculant dosage increases as the MBI of the sample increases as shown in Figure 7. In the figure, you can see that optimal dosage curves can be found using clay based dosages, that better reflect the performance of the polymer. Note that within a given lease boundary, the dosage is approximately normalised by MBI across multiple samples allowing for better predictions of dosage requirements via the mine plan.



(a) (b)

Figure 7 **Dosage curves of different fluid fine tailings represented on (a) a solids basis and (b) a clay basis (from Li et al. 2021). Net water release (NWR) is determined by subtracting the volume of polymer added to flocculate an fluid fine tailings (FFT) from the water volume collected after 24 hours of settling, divided by the initial water volume in the FFT sample before flocculation. Clay-to-water ratio (CWR) represents the ratio of clay-to-water in the flocs after the water has been released for 24 hours**

3 Use of methylene blue index to predict geotechnical behaviour

The acquisition of MBI data is considered essential for achieving a comprehensive geotechnical characterisation of oil sands material behaviour. This involves establishing correlations between the MBI of tailings and key geotechnical properties of treated tailings solids encompassing compressibility, permeability, and both drained and undrained strength. Through these correlations, MBI data becomes a pivotal factor in guiding the geotechnical engineering design of closure landforms (Boxill 2011).

3.1 MBI and Atterberg limits

The geotechnical literatures have extensively documented correlation between Atterberg limits and MBI (Cerato 2001). However, a notable gap exists in published correlations specifically for oil sands. Analysis of published data on oil sands fine tailings, where both Atterberg limits and MBI have been measured, reveals a disappointing correlation. The discrepancy is attributed to the influence of water chemistry and testing methodology on Atterberg limits (Wells & Kaminsky 2015). Data from the paper by Jang & Santamarina (2016) highlights the effect of water chemistry on the Atterberg limits of different clays. Figure 8 displays the liquid limits determined with deionised (DI) water, NaCl brine, and kerosene. These results underscore that pore-fluid characteristics determine interparticle-electrical forces, subsequently influencing the geotechnical behaviour (Jang & Santamarina 2016). Atterberg limits tests, functioning as index tests, yield results best compared when conducted within the same mine, and the test method used to determine Atterberg limits also significantly impacts the results (Gidley & Moore 2013). The ternary diagrams related to the fines in oil sands, as reported by Azam & Scott (2005), have influenced the use of cutoffs for assessing the potential geotechnical impact of clays exceeding 10%.

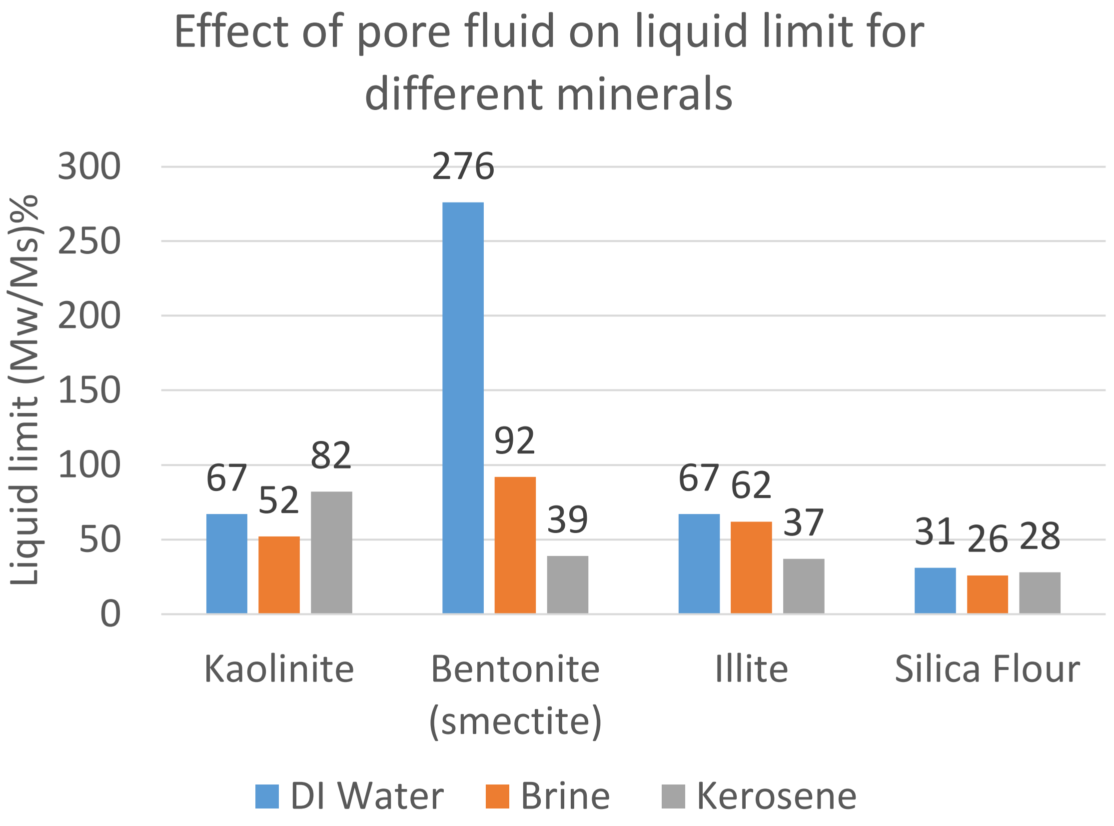


Figure 8 Effect of pore fluids on the liquid limit for different minerals (plotted from data in Jang & Santamarina 2016)

3.2 MBI and segregation

The segregation of tailings can be assessed by examining the sand-to-fines ratio or fines–water ratio, determined through testing the solids content and the fines content of each sediment layer. While the conventional method for determining fines content involves the particle size distribution method using sieves and/or laser detection, an alternative approach is the MBI method. In the presence of bitumen in oil sands tailings, MBI offers a potentially more accurate data representation compared to particle size distribution methods. It has been recognised that the performance of fines and the viscosity of the fines–water matrix are predominantly influenced by clay minerals within the fines. Therefore, it has been suggested that clay content, rather than fines content, be utilised to define tailings segregation (Jeeravipoolvarn et al. 2008).

3.3 MBI and permeability

MBI has been used outside the oil sands industry to characterise the stratigraphy of soil foundations and identify unstable layers (Chiappone et al. 2004). The acknowledgement that the increase of clay content leads to a reduction in tailings permeability positions MBI as a promising tool for predicting the permeability of the tailings deposit (Hyndman et al. 2018). In fact, the MBI method has been used within the oil sand industry to identify low permeability regions within tailings ponds for many years (Lovbakke 2014).

3.4 MBI and yield strength

MBI has been used to determine the clay content which, in turn, is used to calculate the clay-to-water ratio (CWR). This ratio is commonly applied in the oil sands industry to predict the strength of a slurry or deposit, given its significant correlation with tailings rheology, especially in clay dominated tailings, as shown in Figure 9. Materials with low CWR and low yield stress exist in a liquid state whereas those falling within higher ranges exhibit plastic to semi-solid state where clay particles rearrange to form a strong structure. It is noteworthy that the correlation provided here is for peak yield strength. However, similar correlations can be developed for remoulded strengths. This proves effective when there is reasonable consistency in water chemistry and effective stress conditions across the deposit. Under such conditions, variations in strength are primarily influenced by the CWR and water layer thickness (Wells & Kaminsky 2015). For oil sands tailings, the assumption of reasonably constant conditions holds true as the sodium absorption ratio is so high and the fluctuations in chemistry don't materially impact the behaviour of the deposit. Similarly, the effective stress conditions remain constant or are easily predictable due to the predetermined tailings deposition methods and deposit design.

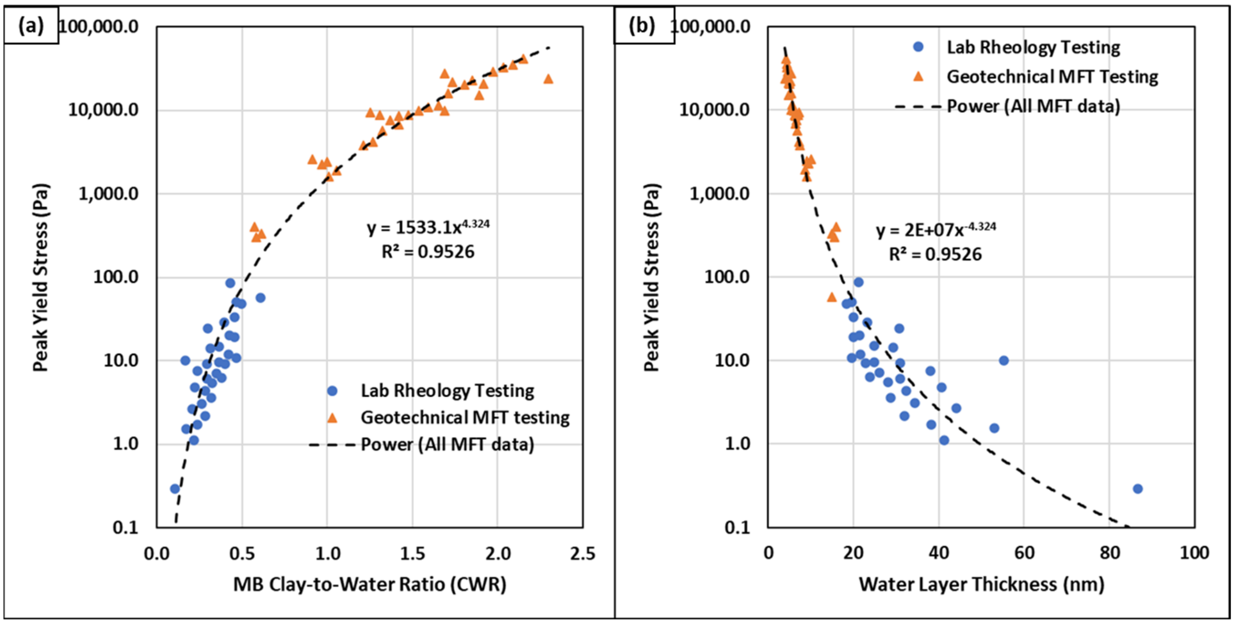


Figure 9 **Peak yield stress as a function of (a) clay-to-water ratio and (b) water layer thickness for samples originated from varying ponds, depths, and years (modified from Wells & Kaminsky 2015)**

3.5 MBI and geochemistry

MBI data, in addition to offering insights into clay mineral surface area, provides valuable information about their potential for cation exchange or chemical reactivity (Boxill 2011). Understanding which deposits have a higher CEC can be invaluable in quantifying potential for absorption/attenuation of contaminants of concern.

4 Conclusion

In this study, the MBI test proves to be highly effective in providing a 'pain index' or initial estimate of various critical properties in mine processing and tailings management. These include, but are not limited to, flotation performance, slurry yield stress, Atterberg limits, dewatering performance, and flocculation dosage. One key advantage of the MBI test is its ease of measurement and cost-effectiveness at multiple stages of the process, utilising small samples, which is particularly advantageous when samples are scarce, such as during initial development. Additionally, the MBI of mixtures of streams can be predicted through a straightforward mass weighted averaging of the individual MBIs of the streams being mixed. Furthermore, MBI excels in identifying problematic clays more efficiently than conventional bulk XRD tests. The authors therefore recommend that MBI be used as a preliminary screening tool when evaluating new ore bodies for exploitation or for better understanding challenges within different areas of mature mines.

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