Impact of lime treatment on tailings dewatering and cap water 1 qualityunder an oil sands end pit lake scenario 2

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#### Abstract 9

Oil sands tailings have a limited ability to dewater and can also release toxic water, so finding a 10 cost-effective tailings reclamation strategy has been challenging. End pit lakes (EPLs) are being 11 12 examined as one potential approach to reduce fluid fine tailings (FFT) inventories for the oil sands industry. This study investigated the effect of adding lime (650 to 4000 ppm) on FFT dewatering 13 and improving cap water quality under the simulated EPL scenario. The results illustrated that a 14 high lime dose of 3500 ppm achieved the highest FFT water recovery, decreased the cap water 15 alkalinity at the end of the experiment (after 90 d), and increased the possibility of cation exchange 16 (at day 0). In contrast, the degradation of petroleum hydrocarbons was slightly enhanced at low 17 lime dose of 650 ppm in comparison to high doses. In addition, the 650 ppm dose resulted in 18 minimal change in the microbial cell counts at day 90, compared with high lime doses that resulted 19 20 in a large reduction in the cell counts. Comparisons of toxicity of the FFT pore water after lime addition exhibited higher toxic effects for lime dosages >1600 ppm. Nevertheless, at all lime 21 doses, low cap water toxicity (i.e., <1.0 Toxicity Unit; TU) at day 90 was attained (e.g. for the 22 23 3500 ppm lime dosage, toxicity was reduced from 0.83 TU (day 14) to 0.58 TU (day 90)). The low water toxicity for the cap water (day 90) can be ascribed to the reduction of cap water pH over 24 time due to the dissolution of atmospheric carbon dioxide into cap water. In this study, the 25 significant implications for the use of lime treatment to improve water quality in EPLs were 26

highlighted. We provided an initial understanding of how lime treatment could benefit the longterm success of FFT remediation within EPLs as self-sustaining aquatic ecosystems in the final
reclaimed landscape.

Keywords: End pit lake (EPL); Oil sands tailings; Fluid fine tailings (FFT); Dewatering; Lime;
Cap water quality

## 32 **1. Introduction**

Large volumes of fluid fine tailings (FFT) and oil sands process-affected water (OSPW) are 33 generated from surface mining of oil sands in northern Alberta. The continuous storage of the 34 massive quantities of FFT in engineered tailing ponds (Allen, 2008b; Clemente and Fedorak, 2005; 35 Siddique et al., 2014) requires oil sands operators to use various reclamation strategies to achieve 36 reclamation obligations. Several remediation approaches (e.g., composite tailings, atmospheric 37 drying, etc.) have been studied, however these methods have drawbacks that hinder their 38 39 applications. Most of the proposed alternatives are either energy-consuming and costly (e.g., centrifugation; (Wang et al., 2014) or need a large amount of land (e.g., drying requiring freeze-40 41 thaw cycles (Proskin et al., 2012) (Allen, 2008a; Board, 2012; Hyndman and Sobkowicz, 2010; 42 Vajihinejad and Soares, 2018; Wells and Riley, 2007). Thus, more economical and effective 43 solutions are being investigated to incorporate FFT into oil sands mine closure landscapes 44 (Famakinwa et al., 2018). Water-capped tailings technologies such as end pit lakes (EPLs) are one of those potential approaches to reduce FFT inventories in tailings ponds for subsequent 45 46 reclamation (Kabwe et al., 2018; Zubot, 2010).

47 EPLs are engineered water bodies created in the post-mining pit by pumping freshwater 48 and OSPW on top of the FFT (Zubot, 2010). EPLs are predicted to slowly develop into self-

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49 sustaining aquatic ecosystems (Hrynyshyn, 2012; Zubot, 2010) and act as permanent features in 50 the final reclaimed landscape (Kabwe et al., 2018). In the first field scale demonstration of pit 51 lakes, Base Mine Lake (BML), the FFT was not treated before being deposited into the lake, and 52 the FFT is settling by self-weight consolidation (natural dewatering through gravity settling; 53 (Dompierre, 2016). The FFT (with initial solids content around 10% solids (wt./wt.) as first 54 deposited into tailing ponds) comprises fine clays suspended in water, and the clay particles slowly 55 settle over decades (MacKinnon, 1989).

There are key concerns and potential challenges relevant to the environmental development 56 of EPLs (COSIA, 2012). For instance, during the slow settling of FFT, the released pore water 57 contributes OSPW-derived constituents to the EPL cap water (Dompierre et al., 2016a), including 58 the chemical flux of constituents of potential concern (COPCs) (Dompierre and Barbour, 2016; 59 Lawrence et al., 2015). The presence of COPCs (e.g., major inorganic salts, dissolved organic 60 compounds [DOC], naphthenic acids [NAs], and petroleum hydrocarbons [PHCs]) are potentially 61 62 toxic to aquatic microorganisms (Dompierre et al., 2016a; McQueen et al., 2017; Samadi, 2019; 63 White, 2017; White and Liber, 2018).

Earlier studies investigated the influence of vertical mixing (Lawrence et al., 2015) and 64 have used a numerical modeling approach to simulate the movement of COPCs (Dompierre and 65 Barbour, 2016) and identify the key mechanisms controlling mass transport in FFT layers within 66 EPLs. Similarly, White and Liber (2018) monitored the concentrations of inorganic salts in the cap 67 water of BML over three consecutive years and assessed the corresponding toxicological risk of 68 elevated concentrations of inorganic constituents such as sodium (Na<sup>+</sup>) and bicarbonate (HCO<sub>3</sub><sup>-</sup>) 69 on EPL cap water toxicity (White, 2017; White and Liber, 2018; 2020). The studies reported a 70 sufficient level of Daphnia's populations within the BML surface water with a promising 71

72 improvement trend of BML surface water quality over time (White and Liber, 2018; 2020). However, this was constrained with the less sensitive toxicity endpoints samples used in the study 73 (White and Liber, 2018) that could necessitate further investigations to claim the development into 74 a robust EPL aquatic ecosystem. Several research groups have looked at the ability of the 75 indigenous microbial community in BML to remediate COPCs (Bowman et al., 2016; Bradford et 76 al., 2017; Richardson et al., 2020; Yu et al., 2019). While investigating methanogenic activity, 77 Samadi et al. (2019) reported an enhancement in FFT dewatering with the addition of 78 hydrocarbons and nutrients due to biogenic gas production. Also, Poon et al. (2018) reported a 79 80 decrease in the turbidity of the EPL cap water through pH reduction by inducing CO<sub>2</sub>. However, that study was focused on improving cap water clarity by enhancing the clay flocculation in BML. 81

Because FFT takes a long time (decades) to settle (Masliyah et al., 2011), coagulation and 82 flocculation were implemented to enhance the dewatering process (Famakinwa et al., 2018; Wang 83 et al., 2015; Wang et al., 2014). Promising results in the acceleration of tailings dewatering were 84 85 reported using lime or calcium hydroxide  $[Ca(OH)_2]$  as a stand-alone coagulant or in combination with gypsum (CaSO<sub>4</sub>·2H<sub>2</sub>O) or polymers (Baillie and Malmberg, 1969; Beaty and Lane, 1983; 86 87 Caughill et al., 1993; Chalaturnyk et al., 2002; Ewin et al., 1981; Hamza et al., 1996; Lorentz et 88 al., 2014; Matthews et al., 2002). Lime addition caused an increase in pH and provided divalent calcium ions (Ca<sup>2+</sup>) that improved FFT coagulation (Chalaturnyk et al., 2002; Tate et al., 2017). 89 90 To date, chemical treatment, particularly lime addition, has not been thoroughly and 91 comprehensively explored in EPL scenarios. Moreover, the effect of the slow dewatering of FFT on the development of EPLs is still unclear (Risacher et al., 2018) and should be investigated 92 93 further.

Based on previous studies, lime addition can be hypothesized to accelerate the dewatering
of FFT through cation exchange. The increase in pH resulting from lime addition can reduce
methanogenesis in the FFT layer, and therefore reduce the chemical flux of ions and organic
constituents to the water cap and enhance the cap water quality in EPLs.

Consequently, the key goal of this study is to examine the impact of the addition of various lime doses on FFT dewatering and cap water quality in EPL configurations. The specific objectives are as follows: a) to investigate the feasibility of lime treatment to enhance FFT dewatering in EPLs; b) to evaluate the influence of adding different dosages of lime on the quality of cap water and FFT pore water in EPLs; c) to elucidate the role of cation exchange on the reduction and removal of organic fractions in FFT (DOC and PHCs); and d) to examine the variation of water chemistry (alkalinity, pH, and cations), bacterial cell counts, and toxicity toward *Vibrio fischeri*.

105 **2. Materials and Methods** 

#### 106 **2.1 Materials**

107 FFT was collected from an active oil sands tailings pond in northern Alberta. Hydrated lime [Ca(OH)<sub>2</sub>] was provided by Graymont Inc. (Calgary, Alberta, Canada). FFT and lime were 108 stored in sealed buckets at 4 °C until use. Raw synthetic OSPW was prepared in 15 L deionized 109 water with: 10.80 g NaHCO<sub>3</sub>, 4.35 g Na<sub>2</sub>SO<sub>4</sub>, 8.70 g NaCl, 0.75 g KCl, 2.10 g MgCl<sub>2</sub>·6H<sub>2</sub>O, and 110 111 0.75 g CaCl<sub>2</sub>·2H<sub>2</sub>O. The specifications for Raw synthetic OSPW were provided by Graymont Inc., 112 and mimics the solute concentrations in Albian OSPW (Mahdavi, 2014; Poon, 2019). The Raw synthetic OSPW didn't contain any organics to better understand the movement of COPCs from 113 the FFT layer to the cap water. 114

### 115 **2.2 Experiment**

Four different lime dosages (650, 1600, 3500, and 4000 ppm on a wet weight basis) were used. Lime slurry was prepared by adding hydrated lime to Millipore ultrapure water (18.2 M $\Omega$ cm) to achieve a 5% solids slurry (wt./wt.). Each lime dose was added to 1 kg FFT in a glass beaker and mixed with a mechanical agitator at moderate speed (1200 rpm). The initial solids content of the raw FFT was 32.8%. To achieve 50% solids content, both control (no lime) and lime-treated FFT were compressed in an air pressure filter (OFITE; Bench-Mount Filter Press with Hose and Regulator; #140-31) at 100 psi.

Experiments were conducted in 1 L glass columns, where 500 ml FFT (50% solids content 123 [wt./wt.]) was placed at the bottom of the column and covered with 500 ml Raw synthetic OSPW. 124 All experiments were conducted at ambient conditions (23°C) using uncovered columns in 125 126 triplicate, except for the 4000 ppm treatment which had both covered and uncovered conditions. This step was conducted in order to determine the effect of atmospheric  $CO_2$  on the cap water 127 quality and toxicity and to highlight any discrepancies between covered and uncovered conditions. 128 129 This can be also attributed to the plateau reached at pH 12.5 regardless the increase of lime dose 130 (> 3500 ppm) that requires better understanding about the potential influence of any adverse 131 impact of overdosing.

To better understand the effectiveness of lime addition on FFT dewatering, % water recovery (WR) was used as a comparable index in our assessment. The WR was calculated as follows:

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$$WR\% = \frac{\text{Volume of pore water released at day 90}}{\text{total initial volume of FFT}} \times 100$$

The released water from FFT (FFT pore water) at day 0 was collected using the pressure filter, while FFT pore water at day 90 was collected from the FFT samples by centrifugation at 8,000 g for 40 min. FFT pore water was collected in 50 ml plastic tubes and stored at 4 °C before further analyses. For cap water analysis, samples were collected directly from the columns every two weeks. The initial FFT and final treated FFT samples at day 90 were collected directly from the columns and stored at  $-20^{\circ}$ C in 50 ml plastic vials for DNA extraction. For PHC analysis, FFT samples were stored at 4 °C until further analysis.

## 143 **2.3 Chemical analysis**

Water samples were analyzed for cations including Ca<sup>2+</sup>, Na<sup>+</sup>, K<sup>+</sup>, and Mg<sup>2+</sup> using a 144 Thermo iCAP 6000 series Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-145 OES, ThermoFisher). A Mettler Toledo DL53 (Mississauga, Canada) was used to measure 146 alkalinity with 0.02 N H<sub>2</sub>SO<sub>4</sub> as a titrant, and pH values were measured using an Accumet® 147 Research AR50 (Fisher Scientific, Lenexa, Kansas). DOC was measured using a TOC analyzer 148 (Shimadzu TOC-LCPH, Japan) and a non-purgeable organic carbon method (Brown et al., 2013). 149 Prior to any analysis and unless otherwise specified, liquid samples were filtered with 150 polytetrafluoroethylene (PTFE) syringe filters (Fisher Scientific; 0.22 µm for cation analysis; 0.45 151 µm for DOC analysis). For DOC analysis, all water samples were diluted 10× using ultrapure 152 153 water.

PHCs were analyzed based on the Canada-Wide Standard for Petroleum Hydrocarbons in soil (CCME, 2008). Fractions were grouped as follows: F1 (C6–C10), F2 (C10–C16), F3 (C16– C34), F4 (C34–C50), and F4G-SG (>C50; (CCME, 2008). According to the Dean Stark extraction method (the industry-accepted method) the F4 and F4G-SG fractions were categorized with

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158 respect to bitumen content (Dean and Stark, 1920). F1 fractions were non-detectable in all samples 159 and were measured by purge and trap gas chromatography (Column: DB1 0.25 mm ID; 1.0 µm film; 60 m length). Other fractions (F2, F3, F4, and F4G-SG) were analyzed by Maxxam Analytics 160 (Edmonton, Alberta, Canada). Further details on PHC fractions analysis can be found in the 161 Canada-Wide Standard for Petroleum Hydrocarbons in Soil (CCME, 2008) and elsewhere (Yu et 162 al., 2018a). To determine the statistical differences between the treatment conditions, p-values 163 were calculated using a two tailed t-test in Microsoft Excel. The F-test was initially used to 164 determine the variance among sample pools whether it is equal or unequal. The difference between 165 166 the treatment conditions is considered significant if p-value < 0.05.

#### 167 **2.4 Toxicity Bioassay**

The toxicity of the aqueous samples toward V. fischeri was measured with the Microtox® 168 bioassay test (Osprey Scientific, Edmonton, Alberta, Canada). Briefly, the 81.9% Basic Test 169 protocol was followed by 5 min and 15 min incubation in the Microtox<sup>®</sup> 500 Analyzer (Azur 170 171 Environmental time, (Anderson et al., 2011). The light emission was measured and recorded with MicrotoxOmni software to determine the inhibitory concentration resulting in 20% less light 172 emission ( $IC_{20}$ ) or inhibitory concentration resulting in 50% less light emission ( $IC_{50}$ ) values. 173 There were no significant differences in the toxicity measured between 5 min and 15 min tests, 174 therefore only the 5 min data is described (Miles et al., 2019). Toxicity units, derived from IC<sub>50</sub> 175  $(TU = 100 \div IC_{50})$ , were used to represent high-level toxicity trends (Yu et al., 2018a). In previous 176 studies, 1.0 toxicity unit (TU) or lower is considered completely detoxified (Scott et al., 2008; Yu 177 et al., 2018a). 178

179 Both principal component analysis (PCA) and cluster analysis were employed using Minitab 19 in order to evaluate and highlight any variations between the lime treated conditions 180 and control. Implementing the PCA and cluster analysis in addition to Tukey Pairwise comparisons 181 also supported the determination of clustering, similarity, and significant differences between the 182 treatment conditions (i.e., p-values <0.05 is considered significant different). 183

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## **2.5 DNA Extraction and qPCR analysis**

DNA was extracted from the FFT samples (up to 500 mg of FFT used per sample), using 186 the FastDNA<sup>™</sup> SPIN Kit of Soil (MP Biomedicals) according to the isolation protocol provided 187 188 by the supplier. The extraction was conducted at the start and end of the experiment to examine the initial and final microbial population density for all conditions. Microbial cell counts were 189 determined by qPCR amplification of the RNA polymerase beta subunit (rpoB) gene, utilizing 190 191 rpoB 1698f (5'-AACATCGGTTTGCTCAAC-3') and rpoB 2041r (5'-CGTTGCATGTTGGTACCCAT-3') primers (Brown et al., 2013; Mahdavi et al., 2015; Yu et al., 192 193 2018b). Using a Bio-Rad CFX96 optical reaction module conversion for the C1000 Touch thermal 194 cycler, the qPCR assay was performed on DNA extracted samples from the lime treated/untreated 195 tailings samples. The amplification data were analyzed using Bio-Rad CFX Manager<sup>™</sup> 3.0 software. All standards and samples were analyzed in triplicate. More details can be found 196 elsewhere (Yu et al., 2018b). 197

#### 3. Results and discussion 198

#### **3.1 Influence of lime treatment on FFT dewatering** 199

The effectiveness of lime addition on the FFT dewatering was assessed based on released water volumes, water recovery, and solids content. The released pore water volumes were 21.7, 30.7, 37.7, and 30 ml for the 650, 1600, 3500, and 4000 ppm lime dosages, respectively. Therefore, an increase in the released volume of FFT pore water was observed after lime treatment.

To better compare our study with others, the WR was also used as a comparable index. In 204 general, WR increased at day 90 for the lime-treated FFT samples, as illustrated in Table 1. At the 205 3500 ppm dose, the maximum WR was 7.9%. However, increasing the lime dose to 4000 ppm 206 decreased the WR to 6.4%, implying that there is an adverse effect from lime overdosing. This can 207 be attributed to the overdose effect (i.e., 4000 ppm), which might create an extra strength on the 208 solids and resist the self-weight settling. Tate et al., (2017) reported the same notion that increasing 209 lime dosages could lead to an increase in the yield stress of the FFT (i.e., developing strength by 210 lime addition) and hence can enhance the geotechnical properties of FFT. Another explanation 211 could be due to the premature cracking as a result of the reduction of solids percentage happened 212 213 after lime overdosing (Tate et al., 2017).

Comparing our results with previous studies, the 3500 ppm lime treatment achieved a 214 similar WR (7.9%) to Yu et al. (2018a), who reported 8% WR after 100 d using a biological 215 216 amendment (Ultrazyme). In contrast, Siddique et al. (2014) reported a higher WR of 26% after 90 d using hydrolyzed canola meal (i.e., as an organic substrate). For solids content in our study, the 217 highest increase was also observed at a dose of 3500 ppm (the final solids content of the FFT 218 increased to 51.6% at day 90; Table 1). Despite the increase in the solid content, it can be 219 considered marginal. It is worth to note that the initial solids content had a significant impact on 220 the final WR (or the final volume of released water) where the lowest initial solids content led to 221

a high WR (Yu et al., 2018a). The initial solids content in our study was considerably higher (50%)
than those used by Siddique et al. (2014); 25%) and Yu et al. (2018a); 34.6%).

# 3.2 Variations in water chemistry of FFT pore water and cap water

### 225 **3.2.1 Alkalinity**

As shown in Fig. 1a, pore water alkalinity immediately increased after the addition of high 226 lime doses (day 0; Fig. 1a). For example, after the addition of 1600 or 3500 ppm lime (day 0), the 227 228 alkalinity increased to 2390 mg/l and 5720 mg/l (measured as CaCO<sub>3</sub>), respectively, compared with the control alkalinity of 450 mg/l (measured as CaCO<sub>3</sub>). The increased alkalinity at day 0 229 with the higher lime dose could be attributed to the increase of the hydroxide (OH<sup>-</sup>) concentrations, 230 231 which agrees with Caughill et al. (1993). In contrast, at day 90, the alkalinity sharply decreased at all lime doses, with only a slight change in the control. This could be argued as the pH of the FFT 232 pore water decreased in all the lime-treated columns at day 90 (Fig. S1a). As a result of the 233 234 dissolution of carbon dioxide CO<sub>2</sub> into the cap water; the pH of the pore water started to decrease, 235 and the formation of carbonate minerals, such as CaCO<sub>3</sub>, also reduces the alkalinity (Hrynyshyn, 2012). 236

The alkalinity level of the cap water continuously decreased throughout all the lime experiments as well as in the control (Fig. 1b). However, compared with the control after 90 d (315 mg/l measured as CaCO<sub>3</sub>), the alkalinity level of the samples treated with 1600 ppm and 3500 ppm lime significantly decreased to 170 and 200 mg/l (measured as CaCO<sub>3</sub>), respectively. The slight change in cap water alkalinity in the control experiment (20% decrease) could be attributed to methanogenesis in the FFT layer (Dompierre et al., 2016b; Siddique et al., 2014) that can increase and maintain a stable alkalinity level in cap water. Similar observations were

reported in BML (White and Liber, 2018) where methanogenesis resulted in elevated and high 244 cap water alkalinity. Based on Fig. S1b, the pH of the control FFT decreased below pH 7.5 at day 245 90. This can be attributed to CO<sub>2</sub> production during methanogenesis (Samadi, 2019; Siddique et 246 247 al., 2014) or the contribution of atmospheric CO<sub>2</sub> to decrease the pH. Dissolved CO<sub>2</sub> is converted to carbonic acid through hydration and then dissociates to  $HCO_3^-$  (Li, 2010), which is transported 248 to the cap water through any movement of gas (including CH<sub>4</sub>). To confirm such prospect, further 249 250 investigations are warranted about monitoring of the redox potential for the FFT layer throughout the experiments. 251

An overall decrease in cap water alkalinity in the lime-treated conditions was observed 252 after 90 d, with the highest magnitude of the decrease for the 1600 ppm. For example, the 253 alkalinity decreased after 90 d by 36 %, 58%, and 51% at lime doses of 650, 1600, and 3500 ppm, 254 respectively. The decrease was significantly rapid and sharp in the first 30 d in all lime-treated 255 FFT. A similar trend can be observed for HCO<sub>3</sub><sup>-</sup> concentrations (Fig. S2). There are two 256 257 explanations for this decrease. The methanogenesis process may have been negatively influenced by lime addition which inhibited the methanogens (Nyberg et al., 2011). The changes in the 258 microbial community are discussed later (section 3.3). Alternatively, the change in pH after lime 259 addition could have led to the predominance of  $CO_3^{2-}$  species (MacKinnon et al., 2001). Although 260 261 further studies are needed to confirm the underlying mechanisms, these results reveal the positive 262 role of lime addition to maintain an appropriate cap water quality for the aquatic life (White, 263 2017; White and Liber, 2018). The changing pH is correlated with lime dosage, and Fig. S3 displays the corresponding effect of lime treatment on the pH of cap water with time. 264

### 265 **3.2.2 Cations**

Fig. 2 depicts the change in cation concentrations (Ca<sup>2+</sup> and Na<sup>+</sup>) in the FFT pore water 266 with different lime doses. For the low lime dose (650 ppm; pH < 10), the Ca<sup>2+</sup> concentration was 267 slightly lower than the control (0 ppm lime) at day 0. Similarly, Ca<sup>2+</sup> concentrations were slightly 268 higher in the 1600 lime dose at day 0 compared to the control. An immediate sharp increase in 269 the Ca<sup>2+</sup> concentration at day 0 was observed immediately after the addition of high lime dosages 270 3500 and 4000 ppm (pH > 11.5). At day 90, similar  $Ca^{2+}$  concentrations were observed for all 271 lime doses as well as the control. For lime doses 650 and 1600 ppm, there was no significant 272 change in cation concentration (p > 0.05), while a significant reduction in cation concentration 273 was observed at high lime doses (p < 0.05). The summary of the cation results is presented in 274 Table S1. 275

Elucidating the influence of pH on changes in cations concentrations at days 0 and 90 can 276 lead to a better understanding to propose the mechanisms involved. Briefly, there are two main 277 phases of  $Ca^{2+}$  availability based on the consumption of carbonate species. In the first phase, as 278 lime is initially added at day 0, (e.g. pH 10, lime dose 650 ppm), the equilibrium of the 279 bicarbonate-carbonate reaction will be shifted toward CO3<sup>2-</sup>. Lime reacts with sodium 280 bicarbonate (NaHCO<sub>3</sub>) through water softening reaction to produce sodium hydroxide (NaOH) 281 282 and insoluble calcium carbonate that precipitate as calcite (CaCO<sub>3</sub>; (Hamza et al., 1996; MacKinnon et al., 2001). This mechanism explained the lower  $Ca^{2+}$  concentrations at day 0 for 283 the 650 ppm lime compared with the control at this pH range (Fig. 2a). This is the dominant 284 285 reaction pathway up to a pH of approximately 11.5 (lime dose <1600 ppm), at which the carbonate species are effectively removed. Accordingly, at this phase, no soluble Ca<sup>2+</sup> is available for cation 286 287 exchange reactions. It is worth noting that the softening reactions are fast to occur before ion 288 exchange mechanisms. The NaOH produced from water softening reacts with the edges of

kaolinite clays, removing hydrogen ions which create new exchange sites that will be occupied 289 by Na<sup>+</sup>. This proposed mechanism can explain the significant reduction in Na<sup>+</sup> concentration at 290 day 0 in the FFT pore water for the 650 ppm compared to control (Fig. 2b). With regards to the 291 second phase (i.e., pH > 11.5, lime dose >1600 ppm), the immediate increase in the  $Ca^{2+}$ 292 concentration at day 0 can be attributed to increased solubility of Ca<sup>2+</sup> from the dissolution of 293 Ca(OH)<sub>2</sub>(MacKinnon et al., 2001; Tate et al., 2016). This provides Ca<sup>2+</sup> ions that can exchange 294 on mineral surfaces. This can be demonstrated where the soluble  $Ca^{2+}$  exchanges and displaces 295 the Na<sup>+</sup> increasing its concentration in the pore water (i.e., Na<sup>+</sup> concentrations are 210, 300, and 296 315 mg/L at 1600, 3500, and 4000 ppm lime dosage respectively). 297

With regards to day 90, we can hypothesize that increasing the pH > 12 (i.e., 3500 ppm 298 and 4000 ppm lime) promote the pozzolanic reaction where soluble Ca<sup>2+</sup> started to react with 299 dissolved silica and alumina from the clay forming hydrated minerals of calcium aluminate and 300 silicate (Tate et al., 2017; Wang, 2017). This can elucidate the reduction of Ca<sup>2+</sup> concentration at 301 day 90 for the pore water. Lane (1983) suggested that the pozzolanic reaction can occur with lime 302 addition at high pH in which a stabilization process can occur through lime addition (i.e., the 303 authors suggested coagulation process as a good description of the lime stabilization in soil and 304 305 tailings). Furthermore, the lime addition can modify particle surface charge and lead to agglomeration and interparticle bonding. In the same sense, the study elucidated the stabilization 306 307 reactions as the pozzolanic reactions and carbonation reactions.

In terms of the cap water,  $Na^+$  concentration was almost stable throughout the experiment at different lime doses (Fig. 3b). In contrast,  $Ca^{2+}$  levels decreased in the cap water with increasing lime dose (Fig. 3a). The change in  $Ca^{2+}$  concentrations confirms that  $CO_2$  is being absorbed by the system to react with alkaline  $Ca^{2+}$ . The Student's t-test results suggested significant differences in Ca<sup>2+</sup> concentrations at day 90 at high lime doses (>1600 ppm) compared with lower lime doses (p < 0.05). Our investigations generally depicted a marginal drop in K<sup>+</sup> concentration for all lime doses throughout the experiment (Fig. S4) that slightly varied with the control. Although this could partially corresponds with the findings of White and Liber (2018), who reported a slight change in K<sup>+</sup> levels in the surface water of BML over three consecutive years, further investigations are warranted to confirm this phenomenon. In summary, confirming cation exchange and pozzolanic reactions occurrence would require a more focused study.

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# **3.3 Variations in bacterial cell counts**

The initial indigenous microbial cell counts are represented by the number found in the 320 control sample on day 0 ( $8.15 \times 10^{10}$  cells/µl), and is used to compare the effect of lime treatment 321 on the microorganisms. The addition of lime resulted in an immediate adverse impact on the 322 bacterial cell counts at day 0 after lime addition, and the microbial count was considerably 323 reduced for all lime conditions (Fig. 4). On day 90, the bacterial cell counts were significantly 324 decreased at high lime doses (pH > 11.5), with the greatest reduction of cell counts occurring at 325 4000 ppm lime under covered conditions (4.41  $\times 10^4$  cells/µl; 98% reduction) resulting from the 326 sharp pH increase. In contrast, at a lime dose of 650 ppm (pH < 10) there was a minimal reduction 327 in the microbial cell counts after 90 d, highlighting the adverse impact of increased pH, 328 particularly pH > 11.5, on the cell counts. Overall, there is significant difference in the microbial 329 cell counts for most of the lime-treated FFT (i.e., lime doses > 1600 ppm; p < 0.05), but no 330 significant difference in the control or low lime-treated FFT (i.e., p > 0.05 for both control and 331 650 ppm lime). 332

Our findings correspond with previous studies that showed a similar adverse impact of a rapid increase in pH (pH > 12) on bacterial cell membranes, which inhibited their growth (Burns and Gremminger, 1994; Nyberg et al., 2011; Wong et al., 2001; Wong and Selvam, 2009). Furthermore, the addition of lime (pH 11) was demonstrated to effectively inactivate pathogens in municipal biosolids (Bennett et al., 2003).

## **338 3.4 Impact of lime treatment on organic fractions**

To determine the impact of lime treatment on organic fractions, we characterized both 339 340 PHCs and DOC. Fig. 5a shows the concentrations of PHC fractions after different lime treatments 341 at day 90 compared with the initial concentration in untreated FFT (control) at day 0 and day 90. 342 The greatest reduction in PHC concentrations is observed at 650 ppm lime, with removal 343 efficiencies of 54%, 57%, 60%, and 66% for F2, F3, F4, and bitumen, respectively. However, for lime doses of 1600, 3500, and 4000 ppm, the PHC reductions were minimal compared with the 344 reduction in the control sample after 90 d. The high bacterial cell counts in the sample treated 345 with 650 ppm lime could explain the better degradation of PHCs compared with the degradation 346 in the high lime doses (>1600 ppm). 347

As stated earlier, at a lime dose of 650 ppm, Ca<sup>2+</sup> reacts with carbonate and precipitates as CaCO<sub>3</sub> (Hamza et al., 1996; MacKinnon et al., 2001). In comparison to control, both F4 and bitumen fractions (i.e., the heaviest fractions of PHCs) might complex with calcite precipitates (CaCO<sub>3</sub>) and then settle down. Nevertheless, the F2 fraction (i.e., demonstrated slight reduction, Fig. 5a), which is the highly volatile fraction of PHC (Brickner, 2013), seems to solubilize from the bitumen and dissolve in the FFT pore water. Our findings highlight the negative influence of high doses of lime on microbial cell counts that subsequently affect the degradation of the PHCs. Various studies have reported the effectiveness of various microbial communities such as hydrocarbon-degrading microorganisms, sulfate-reducing bacteria, and iron-reducing bacteria on the degradation of PHCs (Allen et al., 2007; Pandey et al., 2009). Similarly, Yu et al. (2018a) suggested that the hydrocarbons present in tailings serve as a carbon source for the indigenous microorganisms. These findings can explain the reduction of the PHC fractions in the control columns after 90 d.

The variations in DOC concentrations in the pore water throughout the experiment are 361 illustrated in Fig. 5b. No significant change was observed between day 0 and day 90 at 650 ppm; 362 however, at lime doses of 1600, 3500, and 4000 ppm, a significant increase in DOC was 363 observed. The changes in DOC agree with the minimal decrease in the PHC fraction; in which 364 at high lime doses, bacterial cell numbers were decreased and were not able to degrade either the 365 PHC or DOC. Additionally, during cation exchange when pH > 11.5, the F2 fraction might 366 367 dissolve in the FFT pore water and increase the DOC concentration. This mechanism is similar to one proposed by Fine et al. (1997) that highly volatile fractions of PHCs can dissolve into soil 368 369 pore water or groundwater. The increase in DOC may also be ascribed to the release of organic 370 compounds into the cap water during FFT dewatering (Yu et al., 2018a).

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# **3.5 Toxicity and implications of pH influence**

Table 2 summarizes the impact of lime addition on the toxicity to *V. fischeri* of the FFT pore water (days 0 and 90) and cap water (day 14 and day 90). High lime doses (i.e., > 1600 ppm) caused higher toxicity of the FFT pore water (above instrument detection limit) compared with the lower toxicity in the control and in the 650 ppm lime dose (0.1 and 1.2 TU, respectively). These results are consistent with the negative impact of lime addition (i.e., > 1600 ppm) on bacterial cell counts measured in the FFT.

With respect to the acute toxicity effect of the cap water to *V. fischeri* over time (Table 2), for lime doses  $\leq 1600$  ppm (pH  $\leq 11.5$ ), the cap water at day 14 was non-toxic after lime addition. At high lime doses (3500 and 4000 ppm; pH > 11.5), the toxicity at day 14 slightly increased to 0.8 and 1.1 TU at 3500 and 4000 ppm, respectively, then decreased at day 90 to 0.6 and 0.4 TU for 3500 and 4000 ppm, respectively.

383 Even though the lime treated samples (uncovered conditions after 90 days) in comparison 384 to the control (Table 2), showed a slight increase in toxicity; the toxicity of the cap water for all 385 lime doses did not exceed 1 TU (Table 2). This translates as a nontoxic effect toward Vibrio 386 Fisheri. To confirm our observations, principal component analysis (PCA, Fig. S5) and cluster 387 analysis (Fig. S6) were assessed to pinpoint the influence of lime treatment on cap water toxicity 388 after 14 and 90 days. The PCA data support very minimal variations and high similarity levels 389 (i.e., statistically confirmed as insignificant different at 95% confidence interval and significance 390 level of 0.05) between the toxicity effect of lime treated samples and control samples in cap water.

It is worth noting that our rationale was to monitor the toxicity of the cap water after FFT lime treatment which necessitate using the synthetic OSPW in the experiment without any organics to better understand the movement of COPCs. According to our earlier observations about the adverse effects on WR occurred due to overdosing (i.e., 4000 ppm), we have decided to elucidate the potential influence of any adverse impact of overdosing or discrepancies between covered and uncovered conditions with regards to toxicity. For the 4000 ppm covered condition, a significant increase in toxicity occurred at both days 14 and 90 (14.83 and 16.13 TU, respectively). This can be initially attributed to the significant effect on pH at high lime doses, as shown in Fig. S3. Although at day 14, both of the 4000 ppm conditions (covered and uncovered) had a similar pH ( $\approx$  9.8), the TUs were quite different (1.1 TU for the uncovered condition; 14.8 TU for the covered condition). Therefore, high toxicity is not only related to the pH increase in the covered condition, but also possibly to COPCs moving from the FFT into the cap water. These results highlight the significant impact of atmospheric CO<sub>2</sub> to reduce cap water pH and generate a carbonate buffer in the uncovered conditions.

In summary, the slight increase in toxicity of cap water samples can be related to the movements of COPCs from FFT to the released pore water and cap water. This would necessitate more toxicological investigations to examine the critical concentrations, species, and the environmental impact of lime treatment on live-dead viability assays. This would develop better understanding of the influence of end pit lake surface water on acute toxicity and in vitro assays (e.g. RAW 264.7 mouse macrophage cell line and the response of bone marrow-derived macrophages).

# 412 **4.** Conclusions

While mimicking the EPL approach, this study investigated lime application to control the 413 water quality in EPLs and aid the dewatering of FFT from oil sands operations. In terms of the 414 better performance of WR, alkalinity of cap water, and cation exchange, lime dose of 3500 ppm 415 achieved 7.9% WR despite the relatively high initial solids content of FFT (50% wt./wt.). The 416 417 alkalinity of the cap water decreased after adding lime dose of 3500 ppm, compared with the untreated FFT, promoting and maintaining an appropriate, low alkalinity environment for an 418 aquatic ecosystem in EPLs. Furthermore, the cation concentrations indicate that cation exchange 419 420 occurs at high lime doses (pH > 11.5), which aids in clay settling. However, high lime doses (1600,

421 3500, and 4000 ppm; pH > 10) had a negative impact on microbial cell numbers in the underlying 422 FFT, resulting in minimal reduction of organics (PHC) at high lime doses compared with moderate reduction at a low lime dose (650 ppm). Assessing the toxicity of the FFT pore water after lime 423 424 treatment demonstrated high toxicity effects to V. fischeri because of the increased pH. Nevertheless, the toxicity of the cap water was not affected by the high toxicity of the FFT, except 425 under the covered conditions. This highlights a considerable influence of atmospheric CO<sub>2</sub> on 426 regenerating the carbonate buffer and reducing pH of the cap water over time. As such any pH 427 transport from the FFT will rapidly be neutralized due to the associated impact of dissolution of 428 atmospheric CO<sub>2</sub> into the cap water. Overall, the performance metrics and indicative parameters 429 (e.g. increase the WR% from the FFT, increase the possibility of the cation exchange of  $Ca^{2+}$  with 430 the clay particles, and decrease the alkalinity of the cap water) revealed the role of lime addition 431 432 towards the reclamations benefits within EPLs. Future work is still required to build upon the current findings in order to further optimize the lime treatment and the occurrence of cation 433 exchange. The movement of COPCs into the cap water should be further investigated with a focus 434 435 on chemical characterization of the FFT organic fractions and their associated potential risks toward the aquatic life in the EPLs. This better understanding will infer how to proceed to larger-436 scale and successful applications of lime treatment in EPLs. 437

#### 438 **Conflict of interest**

The authors declare that there is no conflict of interest regarding the publication of thisresearch manuscript.

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451 Writing. Nikolas Romaniuk: Investigation; Methodology, Reviewing & Editing. Mike Tate:

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- 453 Meshref: Formal analysis, Data curation and data visualization, Reviewing & Editing. Bipro R.
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