



Impact of ozonation on particle aggregation in mature fine tailings



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ABSTRACT

The extraction of bitumen from the oil sands in Canada generates tonnes of mature fine tailings (MFT), consisting of a mineral matrix of sand, clay, and water, which without treatment requires thousands of years to fully consolidate. We assessed the performance of a novel ozonation method designed to enhance the settling of MFT and explored the mechanisms involved. The solid content of MFT obtained from oil sands tailings was adjusted to 1, 3, 5 wt % with water before applying 15, 30, and 60 min of ozonation. MFT settled after a short (15 min) ozonation treatment, resulting in a sample with clear released water on the top and condensed sludge at the bottom. The water chemistry characteristics, particles' surface charge and chemical bonding were measured. Ozonation led to the increased organic acids concentrations in MFT suspension through converting of organic matter from high to low molecular weight, and detaching organic coating on MFT particles. The pH and the concentrations of ions in the MFT suspension were changed significantly, an association of metal ions with MFT particles was promoted, and the surface charges of MFT particles were neutralized. Consequently, the MFT suspension was destabilized and MFT particle precipitation was observed.

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1. Introduction

Alberta holds the largest oil sands reserve and the third largest (next to Saudi Arabia and Venezuela) proven crude oil deposits in the world. The oil sands contain an estimated 2.5 trillion barrels of recoverable bitumen entrained in a mineral matrix of sand, clay, and water (Penner and Foght, 2010). At present, bitumen is extracted from oil sands ore by the Clark Hot Water Extraction (CHWE) (Clark and Pasternack, 1932). This technique requires large amounts of water and produces much slurry waste; approximately 12 m³ of water is used and approximately 4 m³ of slurry waste is generated to produce 1 m³ of crude oil (Mikula et al., 2009). The tailings slurry consists of water, sand, fine particles (silt and clay <44 μm in diameter), residual bitumen (0.5%–5% mass), and naphtha diluents (<1% mass) from the extraction process (Chalaturnyk et al., 2002). Mature fine tailings (MFT) are the remaining materials suspended in the large on-site recycle water ponds after the coarse sand fraction rapidly settles (Penner and Foght, 2010). Greater than 90% of MFT solids are fine clay

particles to which aqueous organic matter is usually attached (Adegroye et al., 2010). It has been shown that the adsorption of organic matter onto clay particles leads to an increase in their negative surface potential and contributes to their colloidal stability; consequently, adhesion of organic matter inhibits the aggregation of particles in an aquatic environment (Chandrakanth et al., 1996; Farvardin and Collins, 1989; Jekel, 1986). As a result of these properties, it is estimated that fine clay will require hundreds of years to reach full consolidation (Headley et al., 2010; Mikula et al., 1996).

The constantly increasing volume of MFT produced by bitumen extraction from the Alberta oil sands is creating great economic and environmental concern because it has become a risk to the health of wildlife and the stability of ecosystems in the area surrounding the oil sands (Chalaturnyk et al., 2002). Many efforts involving physical, chemical, and biological means have been made to consolidate and enable the reclamation of fluid from tailings (Li et al., 2003; Matthews et al., 2002; Mpofu et al., 2004; Proskin et al., 2010; Sworska et al., 2000; Wang et al., 2010). However, the present technologies are environmentally unfriendly or economically unfeasible. For instance, oil sands companies have implemented gypsum to accelerate tailings densification, however, the continuous accumulation of calcium and sulfate ions in the recycled water detrimentally affects the efficiency of bitumen extraction (Redfield et al., 2003). High molecular weight anionic polymers (e.g., polyacrylamides and

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their derivatives) have been used commercially for tailings treatment. However, these organic flocculants form loose flocs with low solid content, and thus produce weak consolidation. Although polymers can enhance the flocculation of fine clay particles, the excessive dosage required may cause restabilization of the clay particles (Mpofu et al., 2004). Moreover, these polymer-based processes often require high operational costs and their environmental implications are still unknown (Pourrezaei et al., 2011). The development of more efficient and effective techniques for MFT consolidation is highly desirable.

Our previous research demonstrated that ozonation is an effective treatment of oil sands process-affected water (OSPW) (Hwang et al., 2013). Through this research, ozone caused rapid settling of the fine particles in the tailings water. After ozonation, OSPW became clearer and a layer of fine particles was generated on the bottom of the container. It has previously been demonstrated that a suitable dose of ozone can assist in particle destabilization and aggregation (Beltran et al., 1999; Chandrakanth and Amy, 1996, 1998; Chandrakanth et al., 1996; Chang and Singer, 1991; Chheda and Grasso, 1994; Liu et al., 2007; Li et al., 2009). Several mechanisms of ozone-assisted particle destabilization and aggregation have been proposed by Reckhow et al. (1986): (1) ozone may release oxidized metal ions such as iron, manganese, and aluminum from organometallic complexes formed on solid surfaces, leading to an in situ production of coagulant; (2) ozonation can lead to an increase in carboxylic acids on solid surfaces, resulting in greater aluminum, magnesium, and calcium associations that enable precipitation of metal humate complexes; (3) ozonation can cause the desorption of stabilized organic matter coatings from particles by converting high molecular weight compounds to low molecular weight compounds, reducing steric and electrostatic repulsive forces; (4) ozone might induce polymerization of natural organic matter, leading to aggregation via bridging reactions; (5) algal/bacterial cell lysis caused by ozone can liberate biopolymers that may act as coagulants (Thanomsub et al., 2002). For decades, ozone-induced particle destabilization mechanisms have been widely investigated (Chheda and Grasso, 1994; Farvardin and Collins, 1989; Grasso and Weber, 1988; Jekel, 1983; Paode et al., 1995; Saunier et al., 1983). Accordingly, it seemed promising that ozonation could be successfully applied for MFT aggregation.

We developed a novel ozone-assisted consolidation and remediation technology to accelerate MFT settling and improve the quality of recycled water. The impact of ozone on MFT aggregation and the mechanisms involved were investigated. The specific objectives were (1) to determine the effect of ozone on MFT settling, (2) to measure the impact of ozone on MFT water chemistry, and (3) to elucidate the influence of ozonation on the surface properties of MFT particles.

2. Materials and methods

2.1. MFT settling test

An MFT sample from oil sands sites in northern Alberta, having a solid content of 38 wt%, was used throughout the experiments. Ozone was generated with a laboratory generator (Wedeco GmbH[®] D-32051) using USP-grade oxygen, and was applied to the MFT suspensions. The gas flow rate was 2.38 ± 0.1 L/min with an ozone concentration of 71.14 ± 0.64 mg/L. Prior to ozonation, the MFT sample was diluted with deionized (DI) water to mimic fresh MFT suspensions with solid content of 1, 3, and 5 wt%. Thereafter, samples of 120 mL of MFT suspension were transferred to 1 L jars to allow immersion of the entire diffuser. The samples were then ozonated for four different durations (0 (control), 15, 30, and 60 min). After

ozonation, the MFT suspensions were shaken thoroughly for 1 min, and then transferred to a 100 mL volumetric cylinder to initiate settling. The level of the solid interface was recorded every 15 min for the first 3 h as well as at the end point of 24 h.

2.2. Settling effectiveness

The effects of ozonation on MFT suspensions were evaluated by measuring four parameters: (1) volume of released water, (2) initial settling curves, (3) turbidity of released water, and (4) solid content of the settled sludge. The initial settling curves were created by plotting the positions of the interface between the released water and the settled sludge over time. Afterward, about 20 mL of released water was removed with pipette and transferred to a disposable scintillation vial and the turbidity was measured with an Orbeco-Hellige[®] digital direct-reading turbidimeter. If the volume of released water was insufficient for measurement, a certain amount of DI water was added make up the volume to 20 mL. After removing some of the released water for the turbidity measurement, the remaining released water was removed by pipette and discarded without disturbing the remaining sample. Approximately 1 mL of settled sludge was transferred to a crucible and dried at 105 °C overnight. When all of the water had been removed from the sludge, the crucible was cooled to room temperature in a desiccator and the dried sludge was weighed.

2.3. MFT released water chemistry evaluation

After MFT settling, a sample of the released water was removed by pipette from the top of the settled sludge and centrifuged at 3750 rpm for 10 min to remove suspended particles. The supernatant obtained from centrifugation was filtered through a 0.45 µm filter and the pH of the filtrate was measured. The cations contained in the filtered water were evaluated by an Inductively Coupled Plasma–Optical Emission Spectrometer (ICP-OES) (Thermo Scientific ICAP6300) and the anions were assessed by a Dionex DX 600 Ion Chromatography (IC) system. The acid extractable organic fraction (AEF) concentration in the MFT released water was measured by Fourier Transform infrared spectroscopy (FT-IR) following procedures discussed previously (Hwang et al., 2013).

2.4. Surface characterization of MFT fine particles

MFT suspensions were prepared and ozonated as described above. The MFT suspensions were centrifuged at 3750 rpm for 10 min to obtain solid-free released water. To measure the zeta potential of the MFT particles, a small amount of settled sludge was added to the solid-free released water to generate a solution with 1–5 mg particles/mL. Zeta potentials of MFT particles before and after ozone treatment were measured by a Malvern[®] Zetasizer Nano-ZS. X-ray photoelectron spectroscopy (XPS) was applied to determine the surface chemical bonding of MFT particles. To perform the XPS experiments, several droplets of well-mixed MFT suspension before and after the treatment were dried at 60 °C for 48 h on a piece of aluminum foil. Room-temperature XPS experiments were performed using a Kratos Axis spectrometer with monochromatized Al K_α ($h\nu = 1486.71$ eV).

2.5. Statistical analysis

One-way analysis of variance (ANOVA) was used to validate data differences. Microsoft Excel[®] software was used to perform ANOVA and results were reported as *p*-values; *p*-values of less than 0.05 suggested that differences were statistically significant.

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