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Investigation on the flocculation of oil sands mature fine tailings with alkoxysilanes

Dominic K. Thompson, Fernanda L. Motta*, João B.P. Soares

Department of Chemical and Materials Engineering, University of Alberta, Edmonton, Alberta, Canada

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ABSTRACT

Mature fine tailings (MFT), a mineral matrix of residual hydrocarbons, water, and fine clays, are generated by the extraction of bitumen from oil sands. Finding ways to get MFT to dewater more quickly is critical to improving the reclamation of tailing ponds. Although many methods have been developed to treat MFT, most of them have been rejected for lack of technical or economic feasibility. The aim of this study was to investigate the performance of alkoxysilanes on dewatering and densifying MFT. The performance of five alkoxysilane types was evaluated by capillary suction time (CST), settling percentage, and solids content. Among them, bis(3-trimethoxysilylpropyl)amine (bis-amine) performed best, especially when combined with centrifugation. Bis-amine was then used in an experimental design to study the influence of alkoxysilane dosage and initial solids content on MFT dewatering and densification. The experimental design showed that certain operation conditions produced sediments with low CST (below 20 s), relatively high solids content (over 42 wt%), very low turbidity (about 6.0 NTU) and minimal concentration of solids in the supernatant (0.2 wt%), which facilitates the reuse of the recovered water from MFT flocculation in the oil sands extraction process. In addition to these promising findings, it is important to point out that no calcium was added, and that bis-amine has a lower molecular weight, and therefore is less shear-sensitive than the commercial high molecular weight polymers used to treat MFTs in commercial applications.

1. Introduction

Canada has huge oil sands deposits in northern Alberta, which are estimated to be the third largest in the world. Oil sands represent 97% of Canada's total oil reserves (Olsson, 2012), constituting an important economic benefit, both nationally and globally, that is expected to increase because the global demand for energy is projected to increase by 48% from 2012 to 2040 (U.S. EIA, 2016). Unfortunately, increasing the exploration of oil sands is considered by some to be controversial because of several environmental and health issues associated with oil sands extraction which, added to strict government regulations and economic constraints, have urged industries to increase the speed of tailings land reclamation and water recycling (Shende et al., 2016).

The waste byproduct from oil sands extraction processes, consisting of sand, clays, fine silt, and water (tailings) is sent to tailing ponds for sedimentation. Over time, a top water layer is formed in the tailing ponds, which is recycled back to the extraction plant. The bottom layer eventually forms mature fine tailings (MFT), which has a solids content up to 30% (w/w) with the remainder being water. Further dewatering of MFT may take as long as 100 years (Masliyah et al., 2011). The slow settling of MFT is mainly attributed to factors such as pH (Masliyah et al., 2004), small clay particle size (Boxill, 2011), clay negative surface charges (Bakhtiari et al., 2015), clay gelation (Mercier et al., 2012), clay type (Yin and Miller, 2012), and the presence of residual bitumen in tailings (Klein et al., 2013).

Reducing the volume and increasing the density and strength of MFT are probably the most important environmental challenges faced by the oil sands industry (Salam et al., 2016). Destabilization of the suspended solids in MFT by aggregation is often a necessary first step. Accelerated settling will occur after particle aggregation, followed by densification of the settled solid sediments. The aggregation of fine particles by polymeric and other macromolecular additives (flocculants) is used in many applications both in and outside mineral processing, and has attracted considerable academic interest (Smith-Palmer and Pelton, 2012). Common flocculants include polyacrylamide (PAM), poly(diallyldimethyl ammonium chloride) (PDADMAC), polyethylene oxide (PEO), chitosan-based polymers, (Wang et al., 2014) among others. However, the use of chemical reagents may be detrimental to the quality of the recycle water. In addition, high costs associated with the use of flocculants represent a problem for their

* Corresponding author at: Postal address: 9211 - 116 St, Edmonton, Alberta T6G 1H9, Canada.
E-mail address: lopesmot@ualberta.ca (F.L. Motta).

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commercial utilization. In the case of high molecular weight polymers, high viscosities may compromise their interaction with the large volumes of solids present in the tailings, and shear stresses may break polymer chains and flocs, reducing the flocculation and dewatering efficiency of these polymers. Therefore, it is highly desirable to develop more efficient and effective techniques for MFT consolidation.

This study uses alkoxysilanes as flocculants for MFT dewatering and densification. Compared to commercially available flocculants, such as polyacrylamide (PAM), poly(diallyldimethylammonium chloride) (PDADMAC) and polyethylene oxide (PEO), alkoxysilanes are chemicals with low molecular weight, which make them less shear-sensitive. In addition, besides the low cost of alkoxysilanes, several types of these chemicals are commercial available, which make them an interesting alternative for the flocculants currently used by industries.

Alkoxysilanes can form oligomers or polymers after sequential hydrolysis and condensation reactions in aqueous alcoholic solution; they also bond to other materials, including those with Si-O-Si bonds, such as those present in certain clay minerals, via silicate bonding. It is, therefore, plausible that alkoxysilanes could polymerize and bond with suspended clay particles, in a manner similar to conventional polymer flocculants, although via a different mechanism. Once bonded together by alkoxysilane bridges, the clay particles might undergo flocculation, if they could be brought close together by the tendency of the alkoxysilane polymer to minimize its surface area.

The objectives of this study were to: (1) flocculate MFT with five different alkoxysilanes in order to compare their behavior, and screen those with better performance. These tests were conducted in an undiluted MFT sample, which is an already dense suspension of clay particles. This is a hard test for any flocculant, but a relevant one for the oil sands industry; and (2) carry out an experimental design aiming to optimize and understand the role of solids content of MFT and silane dosage on the dewatering and densification of MFT using the best alkoxysilane selected in the screening part of this investigation.

2. Materials and methods

2.1. MFT characterization

Mature fine tailings used in this research were kindly provided by Imperial Oil. A Dean-Stark apparatus was used to quantify its bitumen, solids, and water contents by refluxing toluene in a Soxhlet extractor. Condensed toluene and co-distilled water were continuously separated in a trap designed to recycle the toluene through the extraction thimble, dissolving the bitumen present in the sample, while the water was retained in the trap for quantification. Pore water from centrifugation of the MFT was used to determine its chemical composition by flame atomic absorption spectrophotometry (AAS) (Varian 220FS). The particles size distribution of the solids was evaluated by laser diffraction using a Mastersizer 3000 (Malvern Instruments).

2.2. Flocculation tests

The bulk MFT sample was stirred to homogenize it, and then 200 g aliquots were collected and added to a 250 mL glass beaker. Each sample was stirred at 500 rpm for two minutes, and then at 200 rpm for 8 more minutes. The diameter of the three-blade impeller was 5 cm, and that of the mixing beaker was 6.5 cm. The stirrer was positioned as close as possible to the bottom of the beaker. Immediately after the stirring started, the allotted quantity of flocculant solution was introduced into the MFT sample.

After the mixing step, the samples were allowed to settle under normal gravity (settling) or under centrifugation. Settling experiments were carried out by pouring MFT/flocculant samples into 100 mL measuring cylinders, which were then sealed and left undisturbed for 72 h. Centrifugation experiments were run by subjecting the MFT/flocculant samples to 910 g-force for 10 min.

Several tests were performed to determine the efficacy of the flocculants used in this work, including settling percentage, capillary suction time (CST), sediment solids content, and supernatant turbidity. Settling percentage was calculated as the ratio (*supernatant water height*)/(*total fluid height*), measured visually using the 1 ml markings on a measuring cylinder. Capillary suction time was measured using a Type 319 multi-purpose CST instrument (Triton Eletronics Limited) with Triton filter paper (7 × 9 cm). Capillary suction time determines how well a sample is dewatered by capillary action. The test works by measuring the time taken for water to move a certain distance along the filter paper after the moist sample is placed on it. Solids content was measured by calculating the ratio (*dry mass*)/(*wet mass*). The mass was measured before and after drying in a fan-assisted oven kept at 60 °C until the samples reached constant weight. Turbidity of the supernatant was measured with a 2100 AN turbidimeter (Hach).

2.3. Screening of alkoxysilanes

Five distinct alkoxysilanes (purchased from Gelest) were tested in this work for their ability to dewater MFT. PDADMAC, a water-soluble cationic and linear polymer, which is effective in coagulating inorganic and organic particles such as silt, clay, algae, bacteria, and viruses (Ou et al., 2014), was chosen as a standard flocculant when comparing with these alkoxysilanes. Their structures, chemical formulae, and average molecular weights are listed in Table 1. For simplicity, we will adopt the following abbreviated names for the alkoxysilanes: **bis-amine** – bis (3-trimethoxysilylpropyl)amine, **3-amino** – 3-aminopropyltrimethoxysilane, **glycil** – (3-glycidoxypropyl)trimethoxysilane, **tetra** – tetramethoxysilane (oligomeric hydrolysate), and **metha** – methacryloxypropyltrimethoxysilane.

Flocculants with longer polymer chains (higher molecular weights - MW) can adsorb onto the surface of several particles at the same time, whereas polymers with lower MW attach to fewer particles (Stocks and Parker, 2006). Inspired by the faster solids settling rates achieved with high MW flocculants, a hydrolysis process, and concomitant oligomerization, was carried out with the alkoxysilanes listed in Table 1 to increase their MW and hopefully improve their flocculation performance. As illustrated in Fig. 1, the hydrolysis of an alkoxysilane molecule forms a silanol molecule that may react with another silanol molecule in a subsequent reaction to form a dimer. Subsequent hydrolysis of *i*-mer molecules followed by condensation reactions will eventually form high MW chains (Brinker, 1988).

The hydrolysis was carried out dissolving 1 mL of pure alkoxysilane in 9 mL mixture of 80% ethanol and 20% water, and holding the reactive mixture at 60 °C for 30 min. This procedure was applied to all alkoxysilanes listed in Table 1, except for **tetra** which was already purchased as an oligomer.

Once the alkoxysilanes were hydrolyzed, flocculation experiments were performed according to the methodology described in Section 2.2. With both unhydrolyzed and hydrolyzed silanes. To determine whether ethanol affected the flocculation of MFT, control tests were performed by adding pure ethanol to MFT, in the absence of alkoxysilanes. Three beakers each containing 200 g MFT were prepared, and flocculation tests were performed with pure ethanol solution. The quantities used in the tests corresponded to the amounts of ethanol solution used in tests using 3000 and 6000 ppm of alkoxysilane solids basis.

2.4. Experimental design

A Central Composite Rotatable Design (CCRD) was used to study the influence of two variables – MFT solids content (wt%) and alkoxysilane dosage (ppm solids basis) – on the CST, settling percentage, sediment solids content, and supernatant turbidity of one alkoxysilane selected during the screening experiments. Each variable was studied at five coded levels (−1.41, −1, 0, +1, +1.41), and the model included twelve runs with four replicates at the center value, as shown in Table 2. Multiple

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