



# Influence of humic acids on oil sand processing. Part II: Relationship between bitumen extraction, humic acids concentration and power draw measurements on oil sand slurries



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## ABSTRACT

The processability of four oil sand ores was evaluated through batch flotation experiments under different conditions of pH and temperature. The data of bitumen recovery were accurately modeled using a first-order kinetic equation assuming that the total amount of bitumen in the system consisted of two bitumen components, i.e., a fully-liberated bitumen fraction,  $f$ , characterized by a high flotation rate constant ( $k_f$ ), and poorly-liberated bitumen fraction,  $s$ , of a low flotation rate constant ( $k_s$ ) so that  $f + s = 1$ . The role of humic acids naturally present in the ores was basically that of a depressant of bitumen since poor ores contained the highest proportion of humic acids per gram of bitumen. The recovery of bitumen from oil sand ores correlated very well with the absorbance at 520 nm of the alkali extracts produced from the ores. High bitumen recovery was achieved for ores characterized by a low absorbance value of the extract, while low bitumen recoveries were obtained for ores producing a high absorbance value in the alkali extraction test.

Power draw measurements ahead of the bitumen flotation/extraction stage showed that power drawn by slurries of poor processing ores was not affected by pH and temperature, which was attributed to the low amount of bitumen and low degree of bitumen liberation in these ores. In contrast, good ores required more power for mixing under conditions of low pH and low temperature than under conditions of high pH and high temperature. Gradual bitumen liberation at higher temperature and pH improved the dispersion of the slurries and led to lower power requirements for mixing. Additionally, extraction data indicated that bitumen recovery was proportional to the power draw measured during feed conditioning. Poor ores produced slurries of lower viscosities than slurries prepared from good ores. As a result, higher power consumption was required during conditioning of good ores to promote bitumen liberation and to increase bitumen recovery.

Overall, it is possible to assess the processability of oil sand ores by quantifying the occurrence of humic acids in the ores, and to correlate ore processability with the rheology of ore slurries. Although poor ores are characterized by lower viscosities and lower power requirements during mixing, the presence of humic acids in these ores and their depressing action also contribute to lower bitumen recoveries.

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## 1. Introduction

Oil sand ores are composed of bitumen, water, and sand with a typical composition for the Athabasca deposits in Alberta of 4–14 wt.% bitumen, 80–85 wt.% mineral solids, and 2–15 wt.% water (Takamura, 1982). The bitumen extraction process from oil sands is mainly controlled by physicochemical and hydrodynamic variables, with the interfacial properties of the phases involved playing a key role in achieving high bitumen recovery (Masliyah et al., 2004). Unit operations requiring slurry handling are affected by the rheology of treated suspensions,

and in the processing of oil sand ores the hydrotransport stage is strongly influenced by the rheological properties of oil sand slurries.

Gutierrez and Pawlik (2012) used synthetic mixtures of bitumen and fine quartz as well as actual oil sands ores, and studied the effect of variables such as temperature, and pH on the rheological behavior of the mixtures. It is known that particle aggregation is a significant factor in suspension rheology, affecting the internal structure of suspension. In the case of oil sand slurries, the situation becomes more complex due to the presence of bitumen, and the dynamic nature of the bitumen liberation process produces a measurable change in the rheological properties of oil sand slurries (Gutierrez and Pawlik, 2012). Several types of interactions that affect the viscosity of oil sand slurries can be distinguished between the different components of oil sand ores. When sand particles are free of bitumen, the only attractive forces acting between them are van der Waals' forces. In contrast, if sand particles are coated with bitumen, attractive forces existing

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between them include hydrophobic forces generated by the presence of bitumen surrounding the sand. In this case, interactions between bitumen-coated sand particles involve not only the bitumen phase, but also the sand component, thus affecting a large volume fraction of material, particularly when bitumen starts forming a continuous phase in ores of higher bitumen content. As a result, only a combination of high temperature and high pH – conditions that accelerate bitumen liberation from the sand grains – results in a large decrease of slurry viscosity for quartz-bitumen mixtures with a high bitumen content (Gutierrez and Pawlik, 2012).

In general, studies on the effect of rheology on bitumen extraction are very scarce, and the role of humic acids in the rheological behavior of oil sand slurries and in bitumen extraction has not been systematically investigated. In Part I of this contribution, it was demonstrated that the alkali extraction test originally developed to determine the oxidation of bituminous metallurgical coals can also be used to measure the amount of humic acids in oil sand ores. The absorbance of alkali extracts at 520 nm correlated very well with the dimensionless ratio of the fines content ( $-44 \mu\text{m}$  size fraction) to the bitumen content, which also suggests that a relationship exists between the processability of the ores (bitumen recovery) and the absorbance value.

In this Part II of the publication, changes in the viscosity of oil sand slurries due to changes in pH, temperature, and ore quality were studied using power draw measurements. Additionally, power draw measurements were correlated with the bitumen extraction results from the same oil sand ores. Finally, a correlation was determined between bitumen recovery, power draw measurements, and humic acids concentrations in the oil sand samples.

## 2. Material and methods

### 2.1. Samples and reagents

Four oil sand ores of varying quality were supplied by Canadian Natural Resources Ltd. Ores 2, 3, 5, and 7 – the same ores as in Part I – were used in these studies, and their characterization was given in Table 1 of Part I.

Sodium chloride (NaCl) was used to prepare background solutions while a 1 M sodium hydroxide (NaOH) solution was used to adjust pH.

**Table 1**  
Parameters of flotation model.

	Ore 2	Ore 3	Ore 5	Ore 7
<i>f</i>				
pH 8.5, 20 °C	18	16	18	16
pH 8.5, 50 °C	35	37	28	18
pH 10, 20 °C	25	14	19	16
pH 10, 50 °C	53	46	41	19
<i>s</i>				
pH 8.5, 20 °C	82	84	82	84
pH 8.5, 50 °C	65	63	72	82
pH 10, 20 °C	75	86	82	84
pH 10, 50 °C	47	54	59	81
<i>k<sub>f</sub></i> , 1/min				
pH 8.5, 20 °C	2.9	2.4	2.1	1.5
pH 8.5, 50 °C	3.4	4.3	2.9	1.7
pH 10, 20 °C	5.6	3.8	2.1	2.0
pH 10, 50 °C	6.2	4.1	2.9	1.4
<i>k<sub>s</sub></i> , 1/min				
pH 8.5, 20 °C	0.08	0.06	0.06	0.03
pH 8.5, 50 °C	0.16	0.13	0.14	0.04
pH 10, 20 °C	0.11	0.08	0.07	0.03
pH 10, 50 °C	0.21	0.16	0.14	0.03

### 2.2. Procedures

The process of bitumen extraction in oil sands processing is in general carried out at solids concentrations around 30 to 45 wt.%. The residence time in the hydrotransport pipeline is around 25 min. Therefore, reliable rheological measurements on these types of diluted slurries under laminar flow conditions over a period of 25 min are very difficult due to particle settling. It was mainly because of the timescale and hydrodynamic conditions of the test that it was decided to use power draw measurements to follow changes in viscosity of these slurries over such extended periods of time. Power draw measurements were based on the torque method using a turn-table setup schematically shown in Fig. 1. The turn-table arrangement consisted of a conditioning vessel (flotation cell) that was placed on a low friction disk that rotated as a result of the torque exerted on the slurry by the impeller. A lever arm was attached horizontally to the base of the low friction disk. This arm was connected through a thin wire to a force gauge that was capable of taking readings every 2 s. Since the force imparted onto the fluid by the impeller caused an equal and opposite force on the lever arm, the gauge reading could be directly related to the force applied to the pulp, and this force could be correlated to the power consumption per unit volume of slurry according to Eq. (1).

$$P_v = \frac{F \times 0.009807 \times L \times rpm \times 2\pi \times 0.016667}{V_s} \quad (1)$$

Where,  $P_v$  is the power input in Watts per cubic meter,  $L$  is the lever arm length in m,  $rpm$  is the shaft speed in rotations per minute,  $F$  is the gram-force readout obtained from the force gauge, and  $V_s$  is the slurry volume in  $\text{m}^3$ . The factor 0.009807 corresponds to the gravitational acceleration divided by 1000 and is used in Eq. (1) to convert the force from grams-force to Newtons, while the factor  $2\pi \times 0.016667$  converts  $rpm$  into  $\text{rad/s}$ . The mixing container used in these measurements was a 2.7 L Denver flotation cell that contained 2.1 L of slurry. The lever arm was 9 cm long, and the slurries were stirred at 800 rpm. A LIGHTNIN LabMaster™ mixer was used as a stirrer, which maintained the mixing speed at a constant value regardless of the slurry viscosities. The control of temperature was achieved using heating straps wrapped around the outside walls of the cell. It was found that the heating straps did not significantly contribute to the total torque readings. It should be recognized that this turn-table design was previously used by Genc (2009) for measuring the power consumption of nickel sulfide slurries with good results. Slurries of the four types of ores were tested through power draw measurements under different conditions of pH (8.5, 10), and temperature (20, 50 °C). pH was adjusted using a 1 M NaOH solution, and slurries were prepared using a 0.01 M NaCl solution. No aeration was applied during conditioning, and the slurries were mixed for 25 min. For tests at 50 °C, slurries were prepared by preheating a background solution in a stainless steel beaker to a temperature higher than desired which, after the oil sand sample was mixed with this background solution, produced slurries at a target temperature.

Bitumen extractability was assessed through batch flotation experiments in a standard Denver flotation machine using the same 2.7 L flotation cell. The ores were tested through flotation tests under the same pH and temperature conditions as in the power draw studies. The temperature was maintained using heating straps in the same way as for the turn-table set-up. The power draw measurements previously described simultaneously served as a feed conditioning and preparation stage for flotation tests. Therefore, it was possible to directly correlate power draw required for conditioning with subsequent bitumen extraction. The conditioned slurries obtained from the turn-table were afterwards re-conditioned in the Denver flotation machine for 2 min at 1200 rpm. After this, the air valve was

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