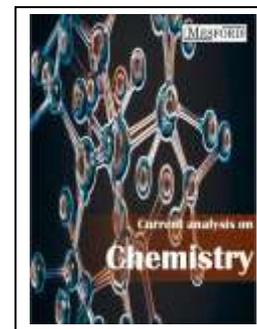


Role of n-Decane in an Alternative Bitumen Recovery Method

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Abstract:

A lower-temperature water/solvent hybrid bitumen extraction (HBE) has been considered as a possible alternative method to the commercially used hot or warm aqueous extraction for mineable oil sands. This communication describes the effectiveness of n-decane (as an example of a high-boiling-point solvent) in the HBE process. Extraction efficiencies of decane-based HBE flotation tests using a batch extraction unit, and the amount of decane ending up in the extracted tailings were determined by gas chromatography. It was suggested that adding n-decane significantly enhanced bitumen recovery at 20°C for three different grades of ore, but was not as effective as additions of lighter solvents such as n-hexane, at the same dosage. Interestingly, the levels of solvent remained in the decane-based HBE tailings for the three ores were much higher than levels obtained when n-hexane was used in the HBE. The relationship between n-decane loss ratio to tailings and bitumen recovery is outlined.

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Decane; Enhanced oil recovery; Flotation; Gas chromatography; Solvent loss; Tar/oil sand.

1. INTRODUCTION

A process based on Clark hot water extraction (CHWE) is the only commercially-used mean to recover bitumen from mined Canadian oil sands [1, 2]. In the CHWE, mined oil sands are blended with hot water, and sometimes caustic, to make a slurry of the oil sands ore at a temperature of 50°C or higher [3]. The slurry is pumped to hydro-transport pipelines, where the complex structure of the slurry undertakes conditioning through mechanical agitation, leading to the breakage of the interactions that hold the sand, bitumen, and water together [4, 5]. Under microscopic scrutiny, slurry conditioning could be deemed as three fundamental elements: Liberation of bitumen from the sand particles, coalescence of bitumen droplets, and aeration of bitumen droplets resulting in the engulfment of bitumen around air bubble [6-9]. The conditioned slurry is then directed to separation vessels, where the aerated bitumen droplets float to the top to form bitumen froth, while solid particles sink in the underflow and are charged as oil sand tailings. Bitumen froth that is far from pure must be 'treated' (i.e. further remove the unwanted water and solids) by adding a solvent such as naphtha or light paraffin, which is referred as froth treatment [10], before bitumen product can be sent to downstream upgrading facilities. The commercial CHWE-based process requires intensive consumption of thermal energy mainly generated by the burning of natural gas, and

creates huge volumes of very stable mature fine tailings, both adding to environmental concerns [11-15].

Recently, a water-solvent hybrid bitumen extraction (HBE) process has been suggested as a possible alternative to commercial CHWE [16-22]. In the HBE, a small part of solvent already used in the current commercial froth treatment is proceeded upfront at a pre-treatment stage of bitumen extraction, after which the remainder of the process is identical to CHWE, with the important exception that it can be run at lower temperature (e.g. 20°C) and without caustic addition [21, 22]. Obviously, there is no significant requirement for heating water and maintaining high-temperature extraction facilities when the CHWE is replaced by the HBE. In addition, HBE requires similar facilities and procedures currently used in the commercial process. Previous studies indicated that, when light paraffinic or aromatic solvents were used, bitumen recovery rates from the lower-temperature HBE were higher than or similar to those accomplished from normal CHWE, without sacrificing bitumen froth quality [21-23].

The underlying mechanisms of the HBE technology were explained in detail elsewhere [7, 8, 16-22]. The observed improvements in extraction performance by adding solvent in the lower-temperature HBE were largely attributed to the positive impact of solvent on the aforementioned three

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essential steps. Soaking of the ore by a solvent dramatically increased both the rate of bitumen liberation and the ultimate degree of bitumen liberation from an ore at an ambient condition [16-19, 22]. The addition of solvent to bitumen led to a noticeable increase in the probability of bitumen droplet coalescence [7, 8, 22], regardless of the source of bitumen. Adding the desired dosage of solvent to bitumen achieved a significant reduction in induction time between bitumen and air bubble, and promoted bitumen aeration [20-22].

Tailings dewatering behavior and the amount of solvent losses to tailings extracted from bench and pilot HBE processes were also demonstrated in our previous publications [24-26]. It was shown that particles in the HBE tailings had higher settling rates, and the HBE tailings suspension was more compressible than those from the normal water-based process [24, 25]. For high-processing ores whose bitumen recovery rates of more than 90% were accomplished using additions of light solvents such as pentane to heptane to cyclohexane in the HBE, the content of solvent remained in the tailings can be lower than the regulatory limit—four volumes solvent per one thousand volumes bitumen recovered—even with no supplemental tailings solvent recovery [23, 26].

From an engineering perspective, it would be desirable to use a solvent or solvent mixture having low vapour pressure (i.e. high boiling point) in order to minimize or eliminate the challenges in controlling solvent losses during the solvent and ore blending stage in the HBE process, when scaled up to a commercial application. Our objective in this communication is to explore the feasibility of using n-decane in the HBE process. n-Decane is chosen mainly as an example of high-boiling-point solvents but also as a single compound whose content can be accurately quantified by gas chromatography (GC). To this end, we examine the processabilities of three ores in decane-based HBE using a batch extraction unit (BEU) and then measure solvent losses to the extracted tailings samples using a chromatographic method. These results will be compared with those generated from the use of a light solvent e.g. n-hexane, to presoak the ore in the HBE.

2. MATERIALS AND METHODS

2.1. Materials

Three oil sand ores from northern Alberta deposits were used and denoted as OS-1, OS-2, and OS-3. The compositions (solids, water, and bitumen contents) and extractability of these three ores were presented in our previous article [26]. OS-1, OS-2, and OS-3 were approximately deemed as low-, medium-, and high-processing ores, respectively.

In order to mimic recycle process water chemistry used in commercial operations, the simulated process water (SPW) used in the BEU tests comprised 1012 ppm Na^+ , 20 ppm Ca^{2+} , 923 ppm Cl^- , 915 ppm HCO_3^- , and 192 ppm SO_4^{2-} in deionized water. The pH was 8.1 ± 0.1 , and the purity of the associated salts from Fisher Scientific) was 99% or better. n-Decane was purchased from Fisher Scientific (ACS reagent grade) and used as received.

2.2. Bitumen flotation protocol

The protocol of decane-based HBE flotation tests was consistent to our previous work [26], and schematically shown in Fig. 1. Firstly, the chosen dosage of n-decane (C10) was blended homogeneously with the oil sand ore by the motion of tumbling in a roller. The dosage of C10 was between 5 and 30 wt% in term of the weight of bitumen in the ore.

After 30 min of rotary blending, approximately 500 g of C10-treated ore was promptly delivered to the BEU pot [27] containing the first amount (200 g) of SPW, resulting in a slurry. A temperature of 20°C was sustained constant by a circulating water jacket. The slurry was agitated at 1200 rpm with no external air input for 20 min; this is so called slurry conditioning. After the slurry conditioning, a second amount (850 g) of SPW water, and air, were introduced simultaneously. The agitation was then reduced to 800 rpm for 15 min during which bitumen was floated to the top, forming the froth layer. The froth was collected in thimbles or containers and then analyzed using a Dean-Stark apparatus to determine bitumen, solids, and water contents in the froth. The percentage of bitumen mass in the collected froth relative to the total mass of bitumen in oil sand ore represents bitumen recovery rate. Once the completion of collecting froth, the slurry suspension remaining in the BEU was discharged as extracted tailings. The extraction tailings samples were then characterized for their n-decane solvent content as described in Section 2.3.

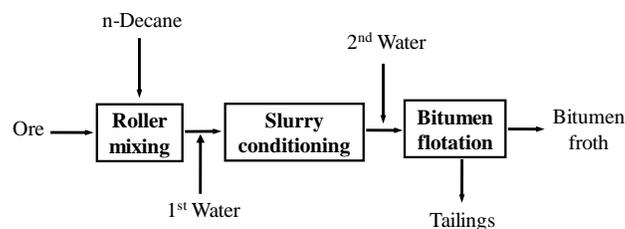


Figure 1. Schematic of decane-based HBE process: n-Decane solvent was distributed evenly to pre-treat the oil sand ore using a roller, prior to slurry conditioning and bitumen flotation.

2.3. Determination of solvent content in tailings

Residual bitumen and n-decane solvent in the tailings sample were separated from water and mineral solids using n-dodecane extraction and then centrifugation. Briefly, about 25 to 35 milliliter of tailings was placed in a 100-milliliter centrifuge tube and the desired amount of n-dodecane was added. The amount of n-dodecane added was about 1 to 2 times the mass of tailings sample, depending on the concentration of solvent residue in the sample. The choice of dodecane instead of the commonly used o-xylene as an extractant is attributed to the basic requirement in gas chromatographic analysis that the extractant must have a higher boiling point than the solute (n-decane in this case). The centrifuge tubes containing tailings and n-dodecane were then shaken in a wrist shaker for about 60 min, ensuring that the tailings and n-dodecane were thoroughly blended. In order to separate the n-dodecane phase from the water and solids phases, the tubes were centrifuged at about 2000 rpm for 30

min. After centrifugal separation, the upper *n*-dodecane phase containing the extracted residual bitumen and *n*-decane solvent was then isolated for further quantification as described below.

Solvent (*n*-decane) content in the upper phase was determined using a GC unit (Agilent Technologies 7890) rigged with flame ionization detection (FID). An example chromatogram is given in Fig. 2. The injector and FID were set at 250°C. The heating profile for the column oven was: initial temperature 30°C, hold for one minute; then linearly ramp to 80°C at a heating rate of 3°C per minute. In the post-run, the column was rapidly heated to 200°C, and maintained for ten minutes to remove the whole *n*-dodecane out of the column. Each compound had its own singular time of retention in the mixture, and the peak of *n*-decane occurred at approximately 14.92 min. The area under the *n*-decane peak was compared with the known value for an internal standard (i.e. *n*-octane) to determine the content of *n*-decane. As *n*-dodecane was baked out in the post run, it is reasonable that the spectrum shown in Fig. 2 had no peak of *n*-dodecane. It is worthwhile to point out that bitumen is an extra-heavy oil, with trifling fraction of hydrocarbons with boiling points lower than 200°C [28-31]. Characterization of background bitumen-in-dodecane solution showed no peaks within the first 17.67 minutes of retention, indicating bitumen possessed no detectable amount of compounds lighter than 180°C. Further details regarding analytical procedures and calculation equations for the GC-FID technique were presented elsewhere [26, 32, 33].

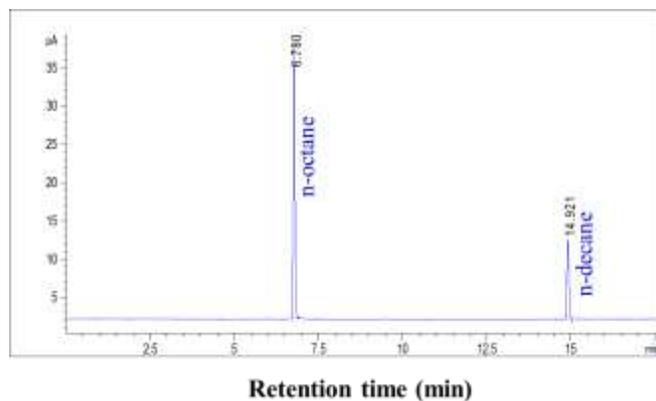


Figure 2. Example of a chromatographic spectrum of *n*-decane and *n*-octane in a mixture of *n*-dodecane-diluted bitumen using a GC-FID.

3. RESULTS AND DISCUSSION

3.1. Effect of *n*-decane on bitumen recovery

Fig. 3 shows the bitumen recovery rates for the three grades of ores in the HBE process at 20°C slurry temperature, using *n*-decane at different solvent dosages to presoak the ore. Note that the amount of *n*-decane was based on the mass of bitumen in the ore. For example, for ore OS-1 containing about 10 wt% bitumen, 5 g decane added to 500 g oil sand corresponded to a decane dosage of 10 wt% on bitumen. As ores OS-2 and OS-3 contained approximately 11 wt% bitumen, 5.5 g decane was needed to achieve the same solvent dosage scaled by bitumen content. As shown in Fig. 3, bitumen recovery for the high-processing ore OS-3 was noticeably increased from 61% (no solvent) to about 81%, when soaked with decane at 7.5 wt% bitumen. A further addition of decane to 10 wt% bitumen increased bitumen

recovery to nearly 88%. For low- and medium-processing ores OS-1 and OS-2, a higher dosage of decane was required to achieve a reasonably high recovery. However, much greater increases in bitumen recoveries for OS-1 or OS-2 than for OS-3 were seen when *n*-decane was added. For medium-processing ore OS-2, for example, *n*-decane at 7.5 to 20 wt% bitumen enhanced bitumen recovery from about 8% to 29%, and further to 89%. A similar enhancement was observed for low-processing ore OS-1, but only when higher doses of *n*-decane (20 to 30 wt%) were added. However, over 30 wt% *n*-decane dosage, based on the bitumen mass in the ore, for any ore grade, would be impractical, and would have elevated operating costs and solvent losses to tailings.

Compared with the bitumen recovery results achieved using water-only extraction at 50°C (CHWE, shown in our previous article [26]), the results shown in Fig. 3 show that adding *n*-decane in the HBE process had a slightly lower (about 5% less) bitumen recovery than CHWE for high- and medium-processing ores, but a significantly higher recovery for the low-processing ore. Compared to light solvents such as pentane, hexane, heptane, and toluene (also shown previously [26]), bitumen recoveries for the three ores presoaked with *n*-decane were lower (about 5 to 20% less) for an equivalent solvent addition, suggesting that *n*-decane is not as competent as a light aromatic or paraffinic solvent in enhancing bitumen extraction efficiencies in the HBE process. This lower performance of *n*-decane in the HBE is possibly ascribed to less competency of *n*-decane on reducing bitumen viscosity in comparison with lighter solvents. Previous studies showed that heavier solvent has a smaller power on viscosity reduction than a lighter solvent of similar structure [34, 35], and the decrease of bitumen viscosity with adding *n*-decane, at an identical dosage, is less significant than that with *n*-hexane addition as an example. Higher viscosity in decane-diluted bitumen than lighter-solvent-diluted bitumen would imply slower bitumen liberation kinetics from sand grains [17-19] and lower coalescence probability of bitumen droplets [8, 22], thus translating into relatively lower effectiveness of decane on increasing bitumen recovery than a lighter solvent [22].

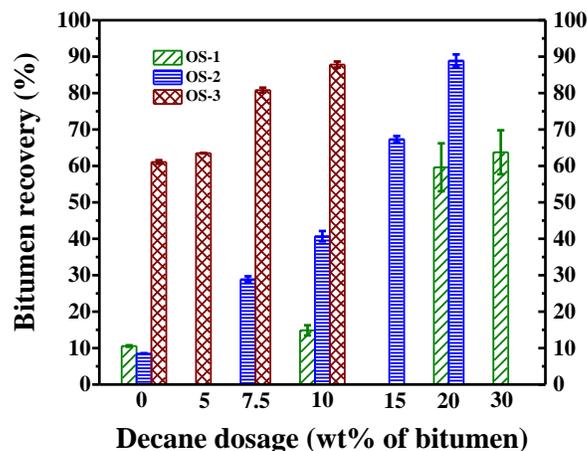


Figure 3. Effect of *n*-decane dosage on bitumen recovery from three oil sand ores using bench-scale HBE at 20°C.

3.2. Effect of *n*-decane on solvent loss to tailings

Fig. 4 shows the content of *n*-decane solvent lost to the HBE tailings for three ores at different dosages of *n*-decane in the extraction process. Here, solvent loss is computed as volume

solvent remained in the tailings over 1000 volumes bitumen recovered, and marked as \mathcal{G} . As can be seen, \mathcal{G} depended on the dosage of n-decane used to presoak the ore as well as on the source of ore. For a given ore, \mathcal{G} (as indicated on a logarithmic scale in Fig. 4) was reduced considerably as the dosage of decane was increased in the ranges considered in this study, correlating well with increasing bitumen recovery of the ore in the HBE (see Fig. 3). Furthermore, \mathcal{G} was ore-dependent: At an equivalent 10 wt% n-decane dosage, the solvent loss to the tailings was reduced dramatically in the order of ores OS-1 > OS-2 > OS-3, corresponding to the improving ore processability from low to medium to high.

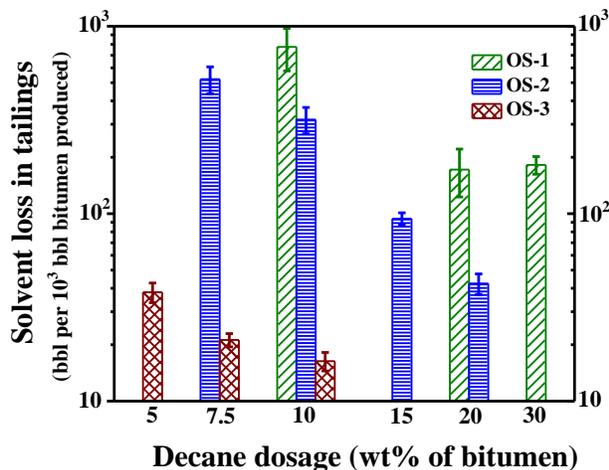


Figure 4. Effect of n-decane dosage on solvent loss in extracted tailings in units of bbl per 1000 bbl bitumen produced for the three ores.

Unfortunately, for all three ores, the amounts of solvent lost to the tailings were much more than the AER (Alberta Energy Regulator) regulated target — 4 bbl (barrels) solvent loss per 1000 bbl bitumen recovered — without further solvent recovery (see Fig. 4). In comparison, as shown in our previous paper [26], solvent losses to the HBE tailings using a light paraffinic solvent, e.g. n-hexane at an equivalent dosage to presoak the three ores, were much lower than those obtained using n-decane. In particular, for high-processing ore OS-3, bitumen recoveries over 90% were achieved by adding 10 wt% or less n-hexane (based on bitumen content). Hexane solvent losses in the tailings were lower than 1.5 bbl/1000 bbl bitumen recovered and were in compliance with the AER regulation without use of a tailings solvent recovery unit [26]. Such differences were mainly attributed to the relatively high bitumen recoveries (or less unrecovered bitumen remaining in tailings), using n-hexane and the same ore, than recoveries using n-decane.

Fig. 5 shows the data converted into solvent loss ratio. Here, the solvent loss ratio is calculated as the ratio of n-decane weight lost to the tailings over the initial mass of n-decane used to pre-soak the ore in HBE i.e. solvent loss ratio = mass of n-decane in the tailings / mass of decane added initially. A trend observed for solvent loss ratio as a function of n-decane dosage was very similar to that seen above (Fig. 4) for \mathcal{G} .

3.3. Correlation of solvent loss with bitumen recovery

To examine the relation of bitumen recovery in governing how much solvent lost to the decane-based HBE tailings, a plot of solvent loss ratio as a function of bitumen recovery percentage is demonstrated in Fig. 6. It is strongly suggested that, in regards to different grades of the ore, there is the same trend that n-decane loss to HBE tailings has an inverse linear correlation with bitumen recovery. The higher the bitumen recovery (i.e. lower bitumen loss to tailings), the smaller the content of solvent residue entrained in the decane-based HBE tailings. Such a correlation between solvent loss ratio and bitumen recovery is in agreement with our previous findings using variety of light solvents such as n-hexane in the HBE [23, 26]. A figure comparing the impacts of using n-decane and n-hexane to presoak the ore on solvent loss ratios and bitumen recovery rates for the three ores in the HBE is provided in the Supporting Information.

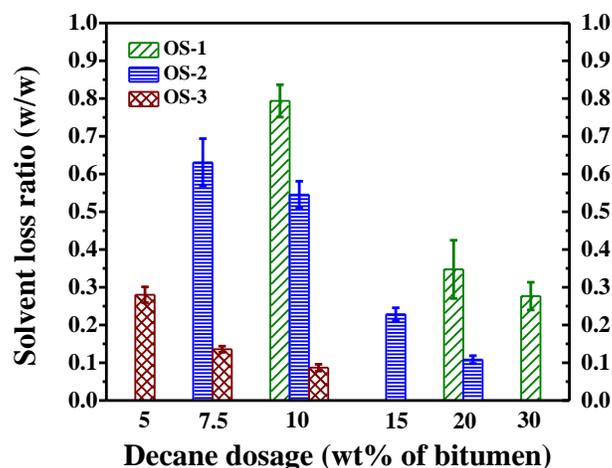


Figure 5. Effect of n-decane dosage on the solvent loss ratio in extracted tailings. Here, solvent loss ratio is defined as the mass ratio of n-decane lost to the tailings over the initial amount of n-decane used to pre-soak the ore in HBE.

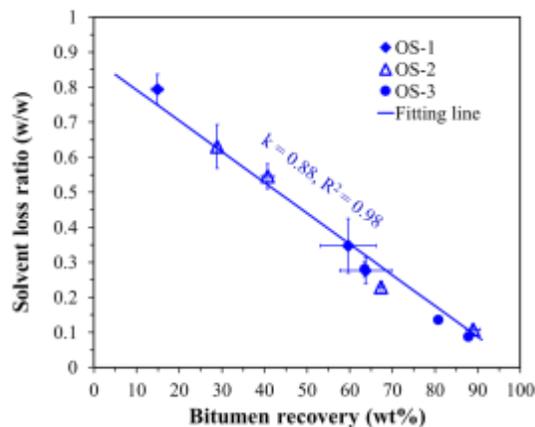


Figure 6. Correlation between solvent loss ratio and bitumen recovery. Experimental data are shown as symbol points for different ores. The line is a linear fitting to experimental data assuming zero solvent loss for 100% bitumen recovery with the slope k and the determination coefficient R^2 .

CONCLUSION

In this work, the use of n-decane in a bench-scale hybrid bitumen extraction (HBE) process was examined. Batch extraction tests showed that when n-decane was used at ambient-temperature HBE, bitumen recoveries were increased considerably for the three ores of different grades as the addition of n-decane increased over the dosage ranges of this study. However, recoveries were lower than those obtained when a light solvent such as n-hexane was used at the same dosage. The amount of n-decane solvent entrained in the extracted tailings were also quantified. Unfortunately, solvent losses to the decane-based HBE tailings were much higher than those obtained by using n-hexane to presoak the ore at an equivalent dosage, and completely failed to comply with AER regulated targets for all three ores without supplemental solvent recovery. Interestingly, n-decane loss ratio to tailings was inversely proportional to bitumen recovery rate. In the next phase, solutions — e.g. tuning water chemistry and the mild increase of operating temperature up to about 30°C, as to decreasing solvent loss to a level below the regulated target shall be addressed, in order to remove this major barrier for using heavy solvents such as n-decane in the HBE technology.

ABBREVIATIONS

ACS: American Chemical Society

AER: Alberta Energy Regulator

bbl: barrel

BEU: batch extraction unit

C10: n-decane

CHWE: Clark hot water extraction

FID: flame ionization detection

GC: gas chromatography

GC-FID: gas chromatography with flame ionization detector

HBE: hybrid bitumen extraction

OS: oil sands

SPW: simulated process water

wt: weight

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CONFLICT OF INTEREST

The authors declare no competing financial interest.

F. Lin and B. Chao contributed equally to this work.

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