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# Temperature Effects in the Conditioning and Flotation of Bitumen From Oil Sands in Terms of Oil Recovery and Physical Properties

L.L. Schramm, E.N. Stasiuk, H. Yarranton, B.B. Maini  
University of Calgary

B. Shelfantook  
Syncrude Canada Ltd.

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## ABSTRACT

*Batch extraction tests show that, for Athabasca oil sands, the water-based conditioning/flotation process can be adjusted from 80 to 50 °C conditions without substantial changes in optimal process aid addition level or primary oil recovery obtained. When the process temperature is further reduced to 25 °C however, an order of magnitude reduction in primary oil recovery is obtained, suggesting that one or more key process variables have undergone a substantial change. Our studies with process additives suggest that several key physical properties undergo major changes, including bitumen viscosity, interfacial tension, and interfacial charge. If these are addressed then comparable optimum primary oil recoveries can be achieved under all of 25,*

*50, or 80 °C conditions. This is a significant result in terms of identifying the key mechanism(s) by which good primary froth recovery can be achieved. It is shown that the interfacial property changes in particular are consistent with the expected thermodynamic conditions necessary for efficient bitumen separation and flotation.*

## INTRODUCTION

Oil sands are unconsolidated sandstone deposits containing bitumen, which is chemically similar to conventional crude oil, but has a greater density (a lower API gravity) and a much greater viscosity. Because sediments were brought in to the Athabasca deposit area from different sources and at different times, the oil sands occur as a mixture of sediment types, overlain by varying

thicknesses of non-oil bearing formations<sup>(1,2)</sup>, so that a diverse number of distinct depositions can be discerned<sup>(2,3,4,5)</sup>. Accordingly, the oil bearing sands have great variability in their compositions and properties and while in oil sand processing the general principles of mineral flotation apply, oil sand composition and structure, and their variations, have a great impact on the way the flotation must be operated.

The hot water flotation process for oil sands is a separation process in which the objective is to separate bitumen from mineral particles by exploiting the differences in their surface properties. The slurry conditioning process involves many process elements, including ablation, mixing, mass and heat transfer, and chemical reactions leading to the separating of bitumen from the sand and mineral particles. Adopting the water-wet model for Athabasca oil sand one has that a thin aqueous film already separates the bitumen from the sand; this separation needs to be enhanced. Disengagement of bitumen from solids will thus be favoured if their respective surfaces can be made more hydrophilic, since a lowering of surface free energy will accompany the separation. The phase separation is enhanced by the effects of mechanical shear and disjoining pressure.

Although there are many variables, including water addition ratios, mechanical energy input levels, chemical addition levels, temperatures, and residence times, process efficiency is more sensitive to some variables than to others<sup>(6,7,8,9)</sup>. Early studies led to the identification of base (NaOH) addition level as the preferred process variable<sup>(6,10,11,12,13,14)</sup> and it was shown by Sanford<sup>(15)</sup> that NaOH addition level could be controlled in response to fines level in the feed. Processibility, the relationship between primary bitumen (oil) recovery and process aid (NaOH addition) for a given oil sand varies with oil sand composition and forms a partial means for categorizing oil sands. The commercial extraction/flotation plants are controlled using empirical relations involving oil sand grade and fine solids content information<sup>(6,16)</sup>. Despite optimizing for each quality of feed, oil recoveries become progressively poorer with decreasing grade of oil sand<sup>(17)</sup>. Improved empirical correlations are continually being discovered for low grade and other, anomalous, oil

sands<sup>(18)</sup>. Further mechanistic information could be used to develop improved process aids, process controls, and even alternate processes.

The main role of the base (e.g., NaOH) is to produce (saponify) natural surfactants from the bitumen<sup>(16,19)</sup>. The natural surfactants predominantly consist of aliphatic carboxylates having hydrocarbon chains of at least 5 carbons (typically C<sub>15</sub> to C<sub>17</sub>) and aliphatic sulfonates having hydrocarbon chains of at least 5 carbons<sup>(20)</sup>. Misra, Aguilar, and Miller<sup>(21)</sup> have made a similar identification of paraffinic carboxylate surfactants as the principal surfactant type released in the processing of Utah tar sands. Only a small fraction of the NaOH added in processing reacts to produce the natural surfactants; while the major portion (ca. 90%) reacts with minerals (to produce mostly bicarbonate)<sup>(22,23,24,25)</sup>.

The impact of the natural process surfactants arises due to their adsorption at surfaces and interfaces, by which they alter surface electric charges and interfacial tensions<sup>(23,24,25,26,27)</sup>. In the conditioning process, under suitable alkaline conditions, both ionization of functional groups at the bitumen surface<sup>(28)</sup> and adsorption of the natural anionic surfactant molecules at the bitumen/aqueous interface<sup>(25,26,27)</sup> occur. Addition of NaOH in the process increases the concentrations of surfactant in the aqueous phase, which in turn increases the extents of surfactant adsorption at all of the aqueous phase interfaces present in the system: gas/aqueous, bitumen/aqueous, and solid/aqueous. The adsorption of anionic surfactant molecules directly affects the surface electric charges of dispersed bitumen droplets, gas bubbles, and fine solid particles. These surface charges are always quite negative under reasonable processing conditions. The surface charge on the solid particles reaches a plateau with increasing surfactant concentration, and the surface charges on bitumen drops and gas bubbles reaches a maximum and thereafter decrease<sup>(29,30,31)</sup>. The same trends have been independently confirmed by Hupka and Miller<sup>(32)</sup> and Drelich *et al.*<sup>(33)</sup>. The ionization of surface groups and adsorption of charged surfactants cause increased electrostatic repulsion which increases the disjoining pressure in the aqueous film separating the bitumen and solids. Increased disjoining pressure together with the

applied mechanical and thermal energy cause the separation of bitumen from solids. A correlation between maximum negative Zeta potential on bitumen droplets and optimum processing conditions for maximizing primary bitumen recovery<sup>(26)</sup> has been shown to scale-up for on-line optimization of Syncrude's continuous pilot-plant<sup>(29)</sup>.

After bitumen-solid separation, bitumen-air attachment has to occur. If the interfacial tension between bitumen and the aqueous phase is low enough, then the balance of interfacial tensions in the system will favour filming of the bitumen around the gas bubbles. The thermodynamics of this process are described elsewhere<sup>(30)</sup>. For 80 °C processing one finds that under reasonable processing conditions bitumen will spontaneously attach to and then spread over the gas bubbles, encapsulating them<sup>(30)</sup>. The surfactant properties that most promote this behaviour are a major lowering of bitumen/aqueous interfacial tension with minor lowering of the aqueous phase surface tension. This behaviour is consistent with the action of the kind of natural surfactants identified above. Schramm<sup>(30)</sup> used batch extractions, interfacial property measurements, and microphotography to show, for optimized processing conditions, the spontaneous filming of bitumen around a gas bubble brought into contact with the solution/bitumen interface. Similar observations have been made independently by Miller *et al.*<sup>(17,21,33)</sup>. These aerated bitumen globules are the species that float upwards in the flotation vessels to form froth. Both laboratory studies<sup>(34)</sup> and pilot plant studies<sup>(35)</sup> indicate that under normal (good) processing conditions the bitumen does indeed preferentially encapsulate air bubbles.

There has recently been much attention paid to the possibility of reducing commercial process costs by reducing the slurry temperature. 55 °C could be reached in a continuous process tumbler using only hot water (i.e., no steam addition). Between 80 and 55 degrees, it has been shown that all of the processibility trends are virtually identical, although it was found<sup>(27)</sup> that the lower temperature process is associated with a slightly lower critical surfactant concentration, a slightly lower optimum slurry water addition level, and a longer slurring time requirement. For even lower temperature

conditioning, say 25 °C it will likely be necessary to add diluents to reduce bitumen viscosity and facilitate its separation and subsequent flotation. This is an essential additive in the processing of Utah oil sands, in which the bitumen is extremely viscous and for which kerosene has been recommended<sup>(36,37)</sup>. A blend of kerosene and methylisobutyl carbinol (MIBC, also known as methylamyl alcohol) is the process aid of choice for the so-called "OSLO" extraction process<sup>(38)</sup>.

In the present work we evaluate the processibility of an oil sand at 25 °C using the standard hot water flotation process batch extraction unit (BEU) and test procedure, in which small (0.5 kg) samples of homogenized oil sand are processed.

The long-range objectives of this research are to better understand the fundamentals of cold conditioning process operation in comparison to warm and hot water processing, and to learn how the anticipated high levels of solids and water in the froths produced may be dealt with. This research will provide knowledge and insights that are crucial to the development of new cold (energy efficient) extraction process technology for Athabasca oil sands.

## EXPERIMENTAL

An average-grade oil sand used was sampled in October, 2000, from Syncrude's mine. The oil sand was shipped in two 20L plastic pails and stored in the dark in a freezer to minimize ageing effects, as recommended by Schramm and Smith<sup>(39,40)</sup>. The oil sand from one pail was chopped and homogenized, and sub-samples were sent to Syncrude for oil/water/solids (OWS) and solids particle size distribution (PSD) analyses. The average composition was 10% bitumen, 10% water, and 80% solids having a <44 µm size fraction of 27 %. Methylisobutyl carbinol (MIBC; 4-methyl-2-pentanol) was 99%, obtained from Aldrich. Kerosene was commercial grade, obtained from a local hardware store.

Oil sand samples (0.5 kg) were processed using the standard hot water flotation process batch extraction unit (BEU) and test procedure as described elsewhere<sup>(19,23)</sup>. This test is reproducible and sensitive enough to be useful for evaluating new process aids (chemicals), process

variables, and determining the processibility of different oil sand samples (see reference<sup>(41)</sup>). Sub-samples of the primary and secondary froths from the batch extractions were collected and assayed for bitumen, water and solids using the "Syn crude method" which involved dissolving the bitumen froth in a 74% toluene/26% 2-propanol mixture. Bitumen was determined gravimetrically after evaporating an aliquot to remove solvent and water. Water was determined by Karl Fischer titration on another aliquot. The solids content was calculated by difference. The detailed procedure is reported elsewhere<sup>(42)</sup>. In previous work<sup>(33)</sup> the reproducibility of froth recoveries and compositions was determined by conducting replicate extractions of a rich oil sand. The primary oil recoveries were  $\pm 5\%$  absolute, the oil contents in primary froths were  $\pm 8\%$  and the water contents in primary froths were  $\pm 4\%$ .

Rheological properties were determined over a range of shear rates, at predetermined temperatures, using a Contraves rheometer (Rheomat 15T-FC) and concentric cylinder sensors (Contraves Measuring System MS-AIE) immersed in a thermostat. Microelectrophoresis measurements were made using a Rank Brothers microelectrophoresis apparatus Mark II (Rank Brothers, Cambridge, England) fitted with a rotating prism and video-viewing system. Particle or droplet mobilities were determined at  $25.0 \pm 0.5$  °C, following the procedure of Schramm and Smith<sup>(31)</sup>. The reproducibility of the electrophoretic mobilities was  $\pm 2 \times 10^{-5}$  cm<sup>2</sup> s<sup>-1</sup> V<sup>-1</sup>. Interfacial tensions were determined using a controlled-temperature, capillary displacement differential maximum droplet pressure method (MDPM), as described in detail in reference<sup>(47)</sup>. Droplet-phase liquid flow rates were chosen to achieve droplet periods of 2 to 4 minutes for best results. The capillary orifice diameters used were  $r_1 = 0.127$  mm and  $r_2 = 0.344$  mm and the capillary displacement was set to the value needed to make the calculated interfacial tension independent of density difference.

## RESULTS AND DISCUSSION

The processibility of the oil sand at 80 °C (Figure 1) is consistent with the expected processibility of a fresh, average-grade oil sand sample<sup>(39,25)</sup>. The optimal sodium

hydroxide addition level for maximizing primary oil recovery was 0.036% NaOH (mass of oil sand basis). It has been shown that sodium hydroxide addition levels in excess of the optimum cause the free natural surfactant concentrations to exceed their optimum levels in turn (the critical free surfactant concentrations). Exceeding the critical free surfactant concentrations is associated with reduced disjoining pressures between bitumen and the solids (thus reducing bitumen separation efficiency)<sup>(26,27,30)</sup>, and with increased bitumen/aqueous interfacial tension (reducing the driving force for filming of bitumen on gas bubbles and thus reducing primary flotation efficiency)<sup>(30,43)</sup>; for both reasons, primary oil recovery suffers.

Next the processibility studies were repeated using a lower process temperature, 50 °C, throughout. No other changes were made to the standard batch extraction procedure. The processibility of the oil sand at 80 and 50 °C (Tables 1 and 2) is generally consistent with that found for a similar oil sand (10% bitumen, 24 % fines) that was studied at similar (80 and 55 °C) conditions in earlier work<sup>(27)</sup>. In the present case the optimal sodium hydroxide addition levels for maximizing primary oil recovery at each temperature were slightly higher, and the primary oil recoveries achieved were also slightly higher. For the present oil sand, the NaOH addition level required to optimize primary oil recovery increased slightly, from 0.04 to 0.06%, when the process temperature was reduced from 80 to 50 °C. Previous work has shown that, when the process temperature was reduced from 80 to 55 °C, the NaOH requirement for optimum primary oil recovery decreased slightly or increased slightly<sup>(27)</sup>. The latter work also showed that slight, temperature-induced changes in NaOH requirement could be offset by corresponding changes in the slurry water addition level, although such a change made the process more sensitive to slurring time (at the lower temperature). In the present work, for now, we plan to keep the slurry water level constant while other factors are investigated.

A series of process investigations were conducted at a process temperature of only 25 °C (Table 1). No other changes were made to the standard batch extraction procedure. The optimal NaOH addition level from the 50

°C work, 0.06% (oil sand basis), produced an order of magnitude reduction in primary oil recovery, to only 8% compared with 88% at 50 °C and 84% at 80 °C (see Figure 1). Given such a dramatic reduction in recovery, we decided to retain the optimal NaOH addition level from the 50 °C work, 0.06% (oil sand basis) and pursue other process aids. In Table 1, froth composition estimates only, in terms of the directly measured oil and water contents, for conditions yielding primary oil recoveries of less than 15%. In these cases the froth samples produced were too small to be reproducibly sampled and analyzed accurately, and the solids contents, determined by difference, were significantly in error.

In an attempt to make a substantial increase in the primary oil recovery from the cold water process, we next tested the process aids selected for the "OSLO" extraction process, methylisobutyl carbinol (MIBC) and kerosene<sup>(38)</sup>. These reagents were first tested as slurry water additives, blended, and at low dosages, beginning with 10 µg/g MIBC and 20 µg/g kerosene (µg/g on a mass of oil sand basis), the optimal dosages developed for the OSLO process<sup>(44)</sup>. We next tested the effects of low dosages of kerosene alone. Table 3 shows that the blend had little or no effect while the kerosene alone may have had a slight positive effect on primary recovery. Although these preliminary results are not too encouraging, it is likely that a key factor is the high bitumen viscosity present at such a low temperature. Therefore, we next tried some higher dosages of these chemicals before turning to alternative approaches in our research program.

As shown in Table 1 and Figure 2, the rather spectacular result was that increasing additions of kerosene (supplementing the base 0.06% NaOH additions) caused progressively increasing primary oil recoveries, up to the highest addition level tested, 20,000 µg/g. Even at such a high addition level, the further addition of MIBC caused increased primary oil recovery, although for MIBC there was an optimum addition level of 1,000 µg/g MIBC for the 20,000 µg/g kerosene addition. Clearly, the large additions of kerosene alone were able to bring 25 °C process recoveries up almost the level of the 50 and 80 °C process recoveries (79% for 25 °C, 88% for 50 °C, and 84% for 80 °C). The further

addition of MIBC boosted primary recovery to 98%, even higher than was achieved (using NaOH alone) at the higher temperatures (see Figure 2). This is a very exciting result!

The fact that, for 25 °C processing, kerosene additions restored so much of the primary recoveries achieved at 50 and 80 °C suggests that much of the initially poor recoveries at 25 °C were caused by the exponentially<sup>(45)</sup> higher viscosity of bitumen at the latter temperature compared with the former temperatures.

Figure 3 shows the effects on bitumen viscosity and primary and total recoveries of adding kerosene to the slurry. To facilitate comparison the results are shown on a mass of oil sand basis. For the kerosene added to coker feed bitumen results, this involves assuming that all of the added kerosene reports to the bitumen phase in a batch extraction experiment (ignoring only the very small amount that would be adsorbed onto the solids). These addition levels correspond to those from batch extractions. The oil recoveries are those from extractions of this average grade oil sand (always with 0.06% oil sand basis NaOH addition level, conditioned and floated at 25°C). It can be seen that there is a very close correspondence between bitumen recoveries (primary and total) and bitumen viscosity.

The reason this does not show up in 50 or 80 °C extractions is that the bitumen viscosity without diluent is so very much smaller (4,690 mPa.s at 50 °C and 493 mPa.s at 80 °C<sup>(45)</sup>). For comparison, the point marked by the symbol (■) represents, on the same scale, the viscosity of coker feed bitumen at 50 °C (datum from reference<sup>(45)</sup>). Thus, viscosity may be the main contributor to the dramatic reduction in oil recoveries found when process temperature was decreased from 50 to 25 °C. This is reminiscent of some of the U.S. experience gained optimizing the water-based conditioning/flotation process for Utah oil sands, for which the bitumen viscosity is very much higher than for Athabasca oil sands. In the Utah oil sands processing work reported by Drelich *et al.*<sup>(46)</sup>, kerosene was added to the slurry to reduce bitumen viscosity to < 1,500 mPa.s to make it processible at digestion temperatures of 50-60°C.

In the present work, recovery was best restored when 20,000 ppm kerosene was added, an addition level that would have produced a bitumen viscosity of 2,270 mPa.s, at 25 °C, if all of the added kerosene dissolved in the bitumen. This raises the possibility of a bitumen viscosity threshold for good recovery, in the range 2,270 to 4,690 mPa.s. For now, we speculate the existence of such a threshold at a bitumen viscosity value of about 3,000 mPa.s.

We next initiated measurements aimed at quantifying potential changes in appropriate interfacial properties, the aim being to identify the mechanism(s) by which the observed reductions in primary froth recovery were achieved. We first commissioned and validated an interfacial tension technique that is particularly well suited to the study of bitumen/aqueous systems at low temperatures, where the density contrast between the phases can vanish. This technique, the differential maximum droplet interfacial tension method<sup>(43,47)</sup> was employed to measure bitumen/aqueous interfacial tensions from the oil sand extractions described above. We also commissioned a microelectrophoresis apparatus and technique<sup>(31)</sup> and used it to determine the electrophoretic mobilities of dispersed bitumen and fine solids from the same extractions.

The results, for 80 and 50 °C conditions, are shown in Figures 4 and 5. These results show high surface charges on both the bitumen droplets and fine solid particles at the optimum conditions for maximum primary oil recoveries, which is consistent with our previous results<sup>(29,30,31)</sup> and those of others<sup>(32,33)</sup>. These results also show minimum interfacial tensions at the optimum conditions for maximum primary oil recoveries, which is consistent with our most recent research into the variation of primary oil recovery with interfacial tension<sup>(43)</sup>. The latter studies have also shown that minimum interfacial tension appears to be associated with optimum primary oil recovery conditions.

The results for 25 °C conditions are shown in Figure 6. It seems noteworthy that neither the additions of kerosene nor MIBC appear to have had a significant influence on either the bitumen surface charge or the bitumen/aqueous interfacial tension. This is despite the fact that these same

additions had a (positive) order of magnitude effect on the primary oil recovery (Figure 2). This is consistent with the hypothesized role of kerosene in reducing bitumen viscosity, described in previous sections. However, it is not clear why the addition of MIBC has a significant positive effect on primary oil recovery without affecting bitumen viscosity, interfacial charge or interfacial tension.

Czarnecki<sup>(48)</sup> has speculated that the role of the MIBC may lie in the stabilization of gas bubbles in the process. To pursue this line of hypothesis it may be worth carrying out some measurements of aqueous surface tensions and gas/aqueous surface charge.

## CONCLUSIONS

The present results show that the standard conditioning/flotation process can be taken from 80 to 50 °C conditions without substantial changes in optimal process aid level required or primary oil recovery obtained, which is consistent with the results of other published work<sup>(27)</sup>. When the process temperature is further reduced to 25 °C, however, an order of magnitude reduction in primary oil recovery is obtained, suggesting that one or more key process variables have undergone a substantial change. Our initial studies with various additives, beginning with the OSLO process aids, show that at least one of the physical properties undergoing major change is bitumen viscosity, and that if this is addressed then good primary oil recoveries (as good or better than those achieved with the standard process at 50 or 80 °C) can be restored. This is a key result.

For processing at 25 °C, we have thus far observed a very close, inverse, correlation between bitumen recoveries (primary and total) and bitumen viscosity that is not apparent in 50 or 80 °C processing (in the latter cases the bitumen viscosity without diluent is very much smaller). It appears that viscosity is the main contributor to the dramatic reduction in oil recoveries found when process temperature was decreased from 80 to 50 to 25 °C. In 25 °C processing oil recovery was best restored when 20,000 ppm kerosene was added, an addition level that would have produced a bitumen viscosity of 2,270 mPa.s, at 25 °C, if all of the added kerosene dissolved in

the bitumen. This raises the possibility of a bitumen viscosity threshold for good recovery, in the range 2,270 to 4,690 mPa.s. For now, we speculate the existence of such a threshold at a bitumen viscosity value of about 3,000 mPa.s.

For processing at 25 °C conditions together with the addition of high additions of kerosene, the addition of 1000 ppm MIBC caused a further, significant increase in oil recovery. Having found that the additions of neither kerosene nor MIBC appear to have had a significant influence on either the bitumen surface charge or the bitumen/aqueous interfacial tension, it remains unclear why the addition of MIBC is able to have a significant positive effect on primary oil recovery without affecting bitumen viscosity, interfacial charge or interfacial tension. We plan to conduct some further physical property measurements aimed at confirming the apparent viscosity effects of kerosene on oil recovery, and identifying the reason(s) for the MIBC effects on oil recovery. The aim here is to identify the mechanisms by which the observed reductions in primary froth recovery were achieved.

Finally, we note that the addition levels of kerosene and MIBC used in this work are probably impractically high. Therefore, after pursuing some of the specific questions that have been raised from our work to date, it will be important to move to work at more realistic chemical addition levels, which may require changes in the mechanical aspects of the way in which the conditioning and flotation are conducted. The BEU test apparatus may no longer be appropriate for such work.

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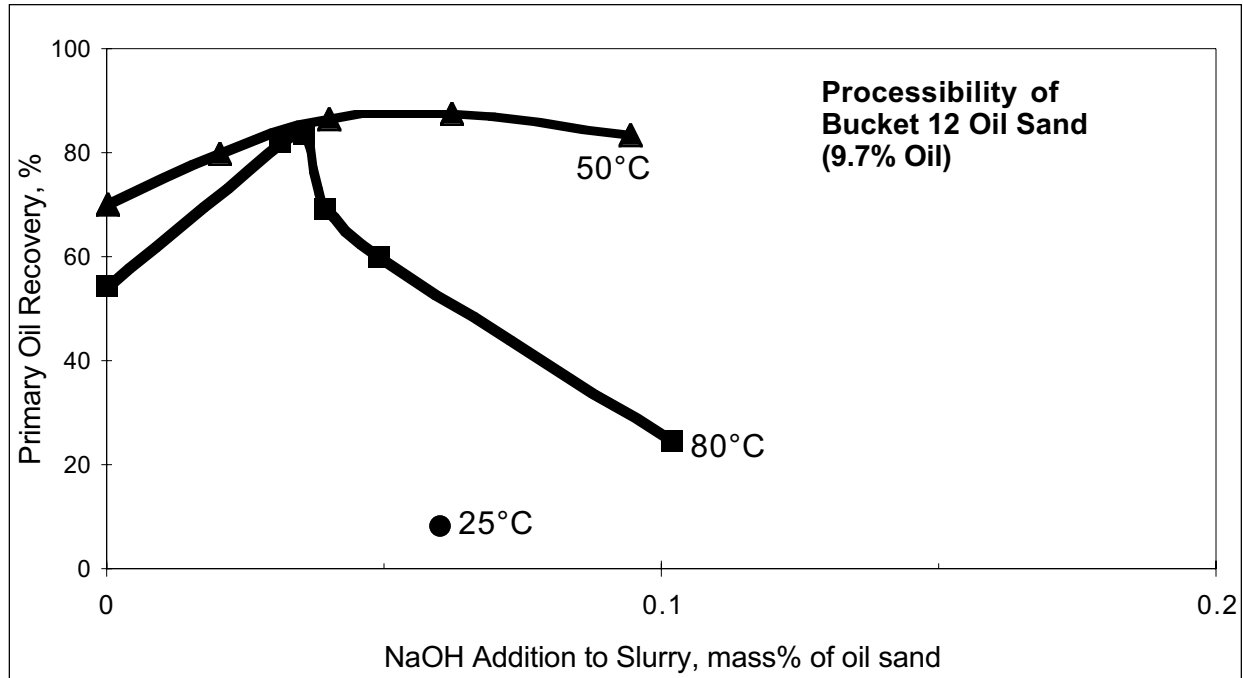
Table 1. Recoveries at Constant NaOH Addition, Using Other Additives, at 25 EC<sup>1</sup>

Kerosene Addition <sup>2</sup> µg/g	MIBC Addition <sup>2</sup> µg/g	Primary Bitumen Recovery %	Primary Froth Quality		
			Oil %	Water %	Solids %
0	0	8	≈60	≈40	nd <sup>3</sup>
20	0	11	≈61	≈37	nd <sup>3</sup>
200	0	15	68	23	8
2,000	0	19	58	35	7
4,000	0	17	53	30	16
6,000	0	22	44	20	36
10,000	0	42	47	28	24
20,000	0	79	52	28	20
20	10	8	≈64	≈36	nd <sup>3</sup>
20,000	100	85	49	34	18
20,000	1,000	98	56	30	14
20,000	10,000	81	60	28	12

<sup>1</sup> All extractions employed 0.06% NaOH Addition (oil sand basis).

<sup>2</sup> µg/g, oil sand basis.

<sup>3</sup> Complete froth compositions not reported for primary oil recoveries of less than 15%, see text.



**Figure 1.** Effect of reducing process temperature on oil sand processibility.

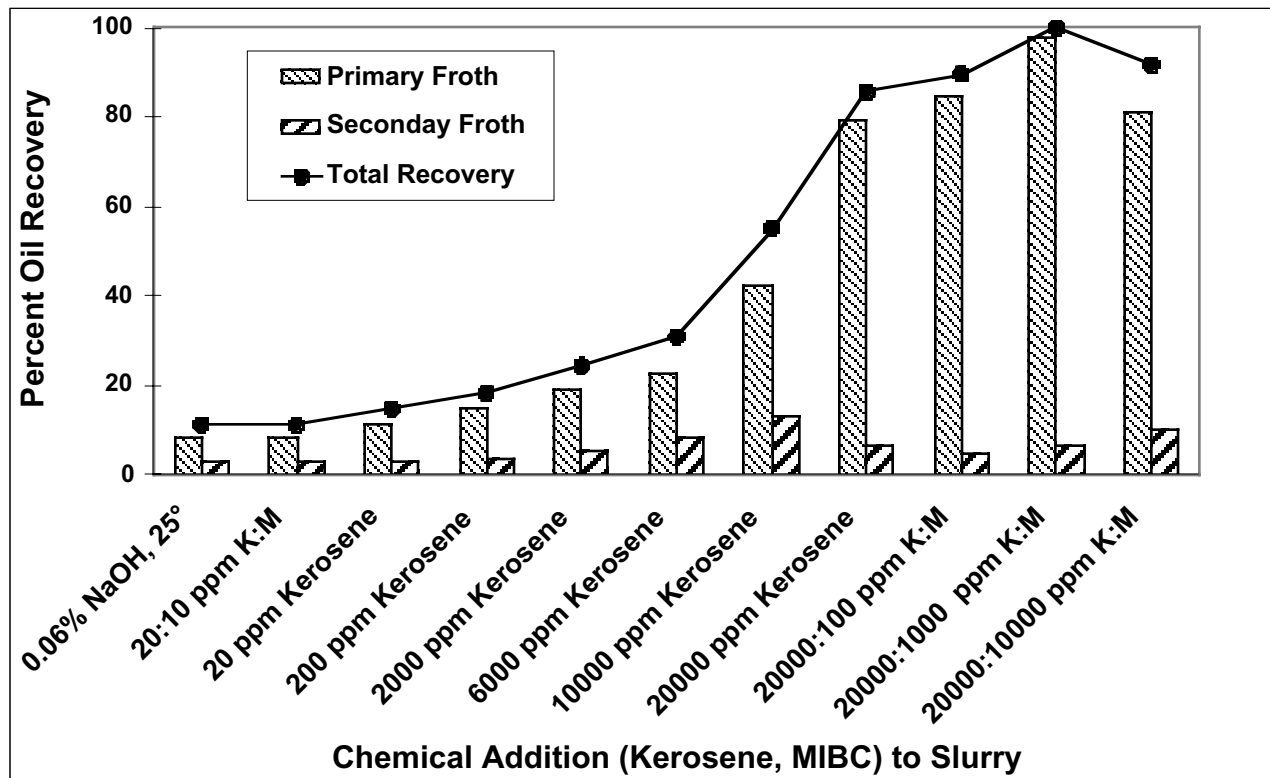


Figure 2. Low temperature extraction results.

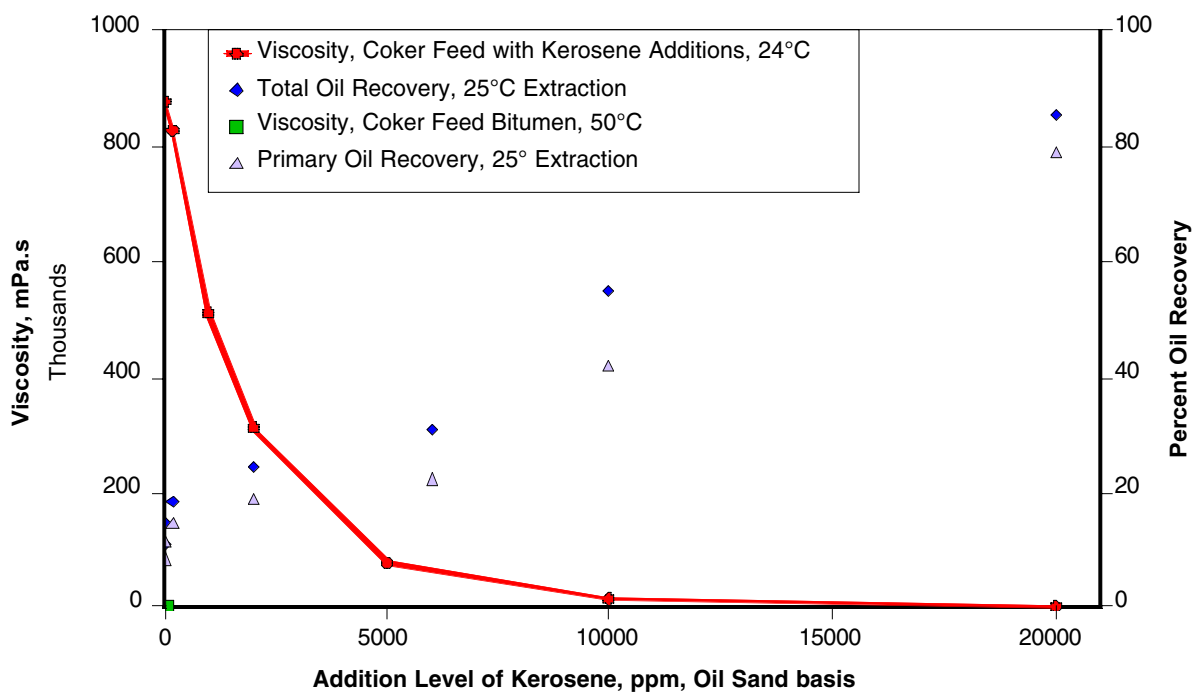
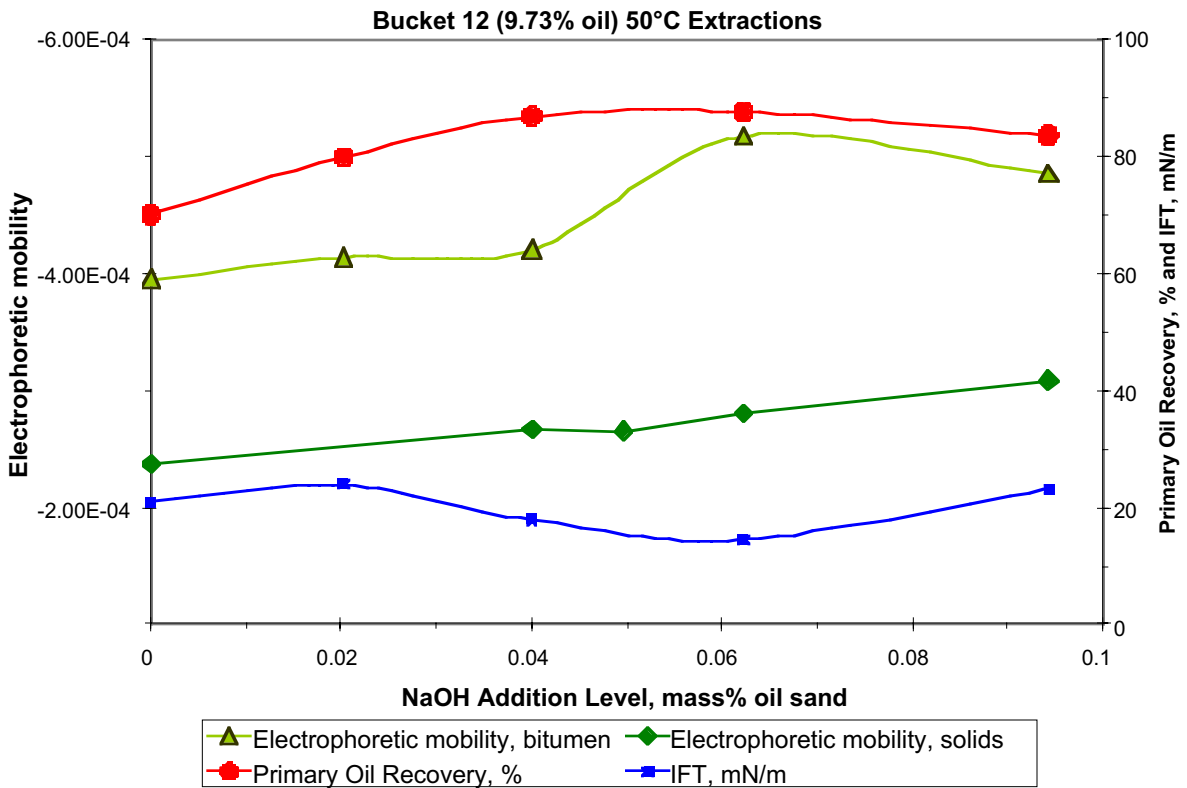
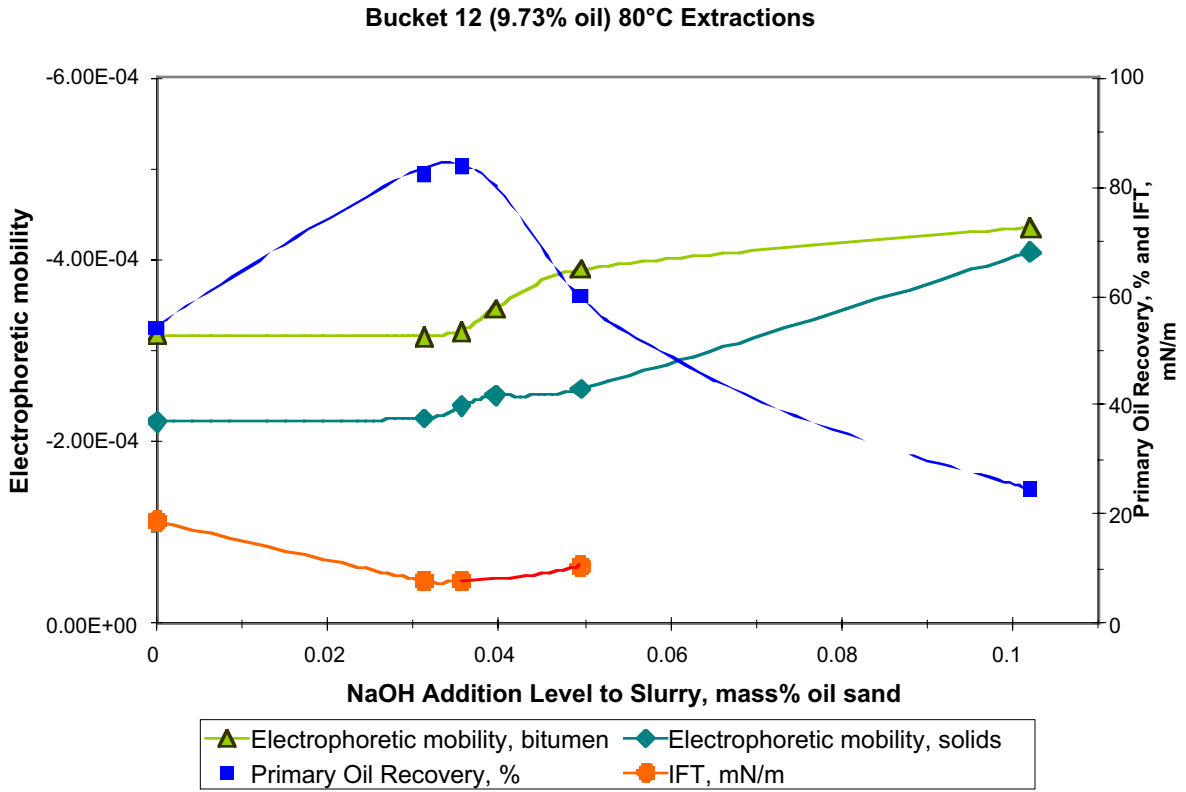


Figure 3. Bitumen viscosity and recovery effects of adding kerosene.



Figures 4 and 5. Physical properties under 80 and 50° extraction conditions.

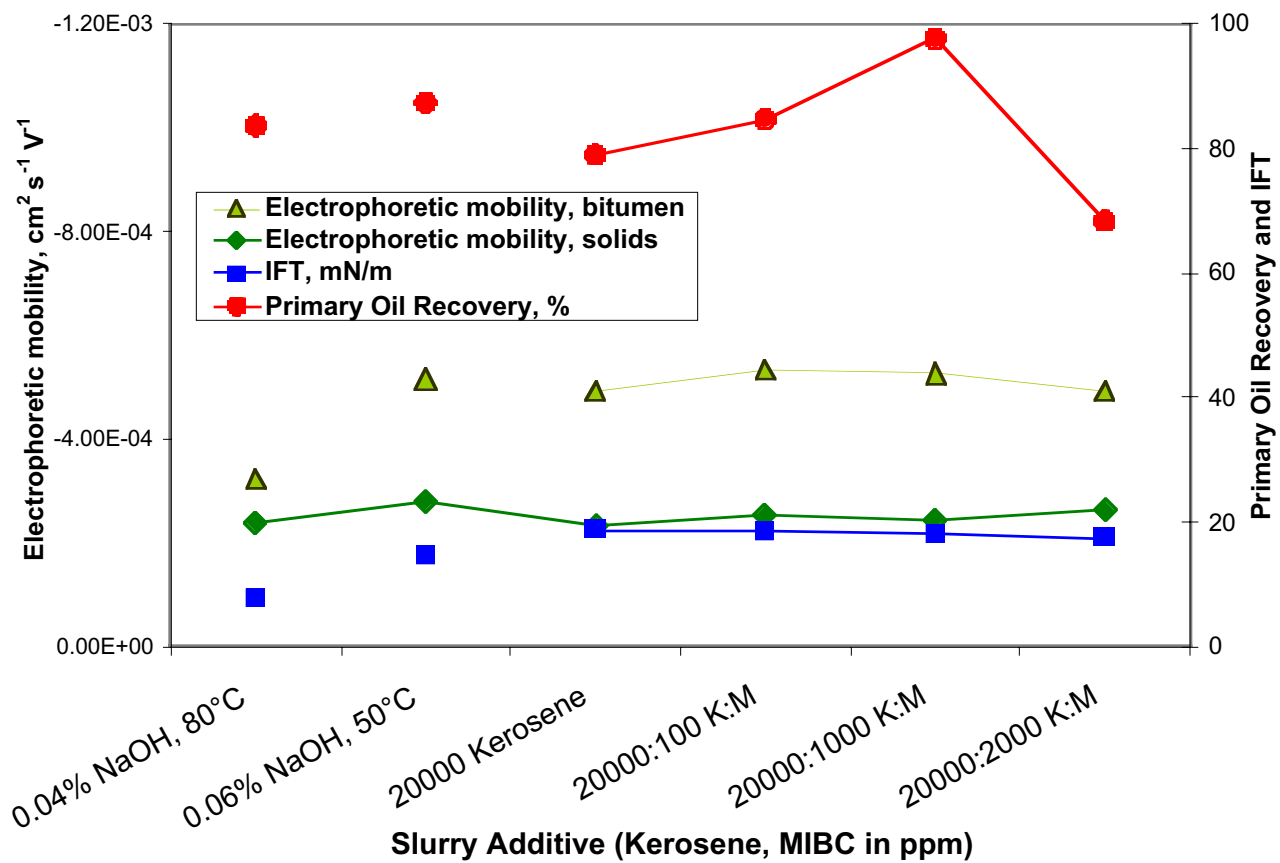


Figure 6. Recovery and interfacial property results for 25 °C processing conditions.