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Optimization of closed-cycle oil recovery: a non-thermal process for bitumen and extra heavy oil recovery†

Pushpesh Sharma,^a Konstantinos Kostarelos[✉]^a and Mohamad Salman^{ab}

Energy from unconventional resources includes bitumen and extra-heavy oil that represent two-thirds of the known resources in the world. Extra-heavy oil and bitumen are currently recovered using thermal processes having a large carbon footprint and significant environmental impacts on water resources. A novel process is proposed: closed-cycle oil recovery (C-COR). C-COR is a greener alternative to provide energy from these unconventional resources with minimal water consumption. C-COR relies on recovering oil solubilized within a single-phase microemulsion, eliminating the need for viscosity reduction to both mobilize heavy oil or to transport it. Proof-of-concept work was conducted using conventional phase behavior experiments with extracted oil and surfactant formulations to develop a surfactant formulation for oil recovery using C-COR. As a part of process development and scale-up, we conducted flow experiments presented in this paper. We learned that a high degree of surfactant adsorption, which negatively impacted the C-COR process, resulted at low pH levels. These findings required modifying traditional static batch tests (phase behavior studies) using actual oil sand instead of the extracted oil. These unorthodox tests revealed that surfactant adsorption caused low oil solubilization and that alkali can be used to reduce adsorption, improving oil solubilization. In addition, unique flow experiments were designed to optimize the delivery and recovery process and are presented in this paper. The unique batch tests and flow experiments were conducted using oil sands from Canada to optimize the process. The proposed optimized approach would employ intermittent flow (soaking) that would result in the fastest recovery of about one-third of the OOIP, followed by continuous injection to recover an additional 10% OOIP, ending with thermal enhancement to recover another 25% OOIP for a total of 61%.

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Introduction

Energy from unconventional resources includes bitumen and extra-heavy oil that represent two-thirds of the known resources in the world.¹ The majority of these deposits are found in Canada and Venezuela. The western Canada sedimentary basin is estimated to contain 1.73 trillion bbls of bitumen.² and the Orinoco oil belt in Eastern Venezuela is the largest deposit of extra-heavy oil in the world, with 1.9 trillion bbls of original oil in place.³

The currently-used recovery methods for bitumen and extra heavy oil are open-pit mining, and thermal methods such as steam-assisted gravity drainage (SAGD) and cyclic steam stimulation (CSS). A fourth method called cold heavy oil production with sand (CHOPS) is a non-thermal recovery method for heavy oil that is suitable only for unconsolidated sands and heavy oil

deposits containing solution gas.³ Open pit mining and the above-ground oil extraction process are criticized for having a large carbon footprint. In addition, the excavation process destroys vegetation while the tailing ponds have the potential to contaminate the soil, both having a concomitant negative effect on the ecosystem.⁴ In 2009, National Geographic sparked an uproar among environmentalists when they published an article featuring a series of photographs showing the environmental impacts of open-pit mining (Fig. 1).³³

Thermal methods such as SAGD and CSS are highly energy and carbon intensive. In addition, they require fresh water for steam generation and the produced water must be treated before re-injection.^{5,6} Other methods that have been investigated include VAPEX, thermal-chemical flooding (TCF) and *in situ* oil upgrading, and several derivatives that incorporate additives (e.g., nanoparticles, ionic liquids, catalysts): all are thermal processes that, while they may be more energy efficient than SAGD, require injection of hydrocarbon solvents such as propane or butane and are criticized in terms of sustainability/environmental acceptability.^{7–10} In summary, these methods are either energy-intensive and/or have significant environmental

^aUniversity of Houston, Houston, TX, USA. E-mail: KKostarelos@uh.edu

^bChevron Technology Center, Houston, TX, USA

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Fig. 1 Photographs showing the effects of open-pit mining of oil sands in Canada. Photo credit: Peter Essick.³³

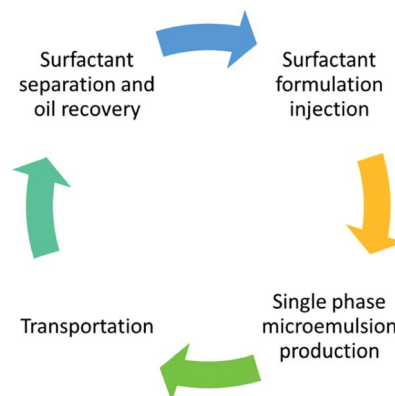


Fig. 2 Conceptual diagram of closed-cycle oil recovery.

impacts. Excepting open-pit mining, the methods that are currently used or are under development rely on mobilizing viscous oil or bitumen. A high-level comparison of these technologies is presented in Table 1.

Alkali-cosolvent-polymer (ACP) flooding, a modification of alkali-surfactant-polymer (ASP) flooding, and speciality chemical flooding, have recently been proposed for heavy oil recovery.^{34–36} The non-thermal recovery methods that are at low technology readiness level (TRL) are derivatives of chemical enhanced oil recovery (cEOR) methods developed for conventional oil reservoirs. These non-thermal approaches have a minimal environmental impact and better energy efficiency but are also focused on mobilizing oil, which has proven difficult due to the high viscosity of bitumen and extra-heavy oil.^{11–26} The potential to unlock these vast energy resources lies in overcoming this challenge while recognizing the importance of the energy-water nexus. Closed-Cycle Oil Recovery (C-COR) is under development as a sustainable, non-thermal method of bitumen/extra heavy oil recovery that relies on solubilizing oil within a single phase microemulsion. The conceptual flow of the C-COR method is depicted in Fig. 2. A schematic of the scale-up for field implementation is rendered in Fig. 3. This

novel approach is designed to solubilize the oil only-not mobilize it. Only single phase microemulsion is produced, which has a lower viscosity being an oil-in-water microemulsion. The produced microemulsion can be transported more easily. After transportation, microemulsion can be separated to recover oil while the surfactant can be re-cycled for re-injection. This is an additional benefit since existing methods require the addition of solvent (diluent) to transport the oil, which adds to the carbon footprint for solvent separation. In brief, the proposed C-COR concept is a low energy approach with potentially lower environmental impacts than the methods proposed to date. Surfactants have been employed in environmental restoration for nearly 2 decades.^{38,40}

A proof-of-concept study for C-COR was published by the authors^{27,28} utilizing coal tar as a model oil, which highlighted the potential of the method achieving 78% oil recovery. In addition, phase behavior and preliminary flow experiments conducted with actual oil sands were conducted by the authors²⁹ to show the potential of the C-COR approach. In that work, the details were presented regarding the formulation selected as the basis for the present work. This paper presents in-depth studies aimed at optimizing the C-COR method

Table 1 Comparison of existing approaches with the proposed C-COR approach^g

Bitumen recovery technology	Thermal (T)/non-thermal (NT)	TRL	Cost/bbl	Environmental impact
Open-pit mining	T	9	Low	High
SAGD ^a	T	9	Intermediate	High
CHOPS ^b	NT	9	Low	Low
CSS ^c	T	9	Intermediate	High
VAPEX ^d	T	6–7	High	High
TCF ^e	T	6–7	High	High
<i>In situ</i> oil upgrading	T	6–7	Intermediate	High
C-COR ^f	NT	3	Intermediate	Low

^a SAGD = steam assisted gravity drainage. ^b CHOPS = cold heavy oil production with sand. ^c CSS = cyclic steam stimulation. ^d Vapor extraction. ^e Thermal-chemical flooding. ^f C-COR = closed-cycle oil recovery. ^g Notes: the table is based on information reported in the literature for each process. Cost per bbl of oil recovered is estimated based on technical recovery efficiency where the TRL of the technology is low and cost data is not yet available.



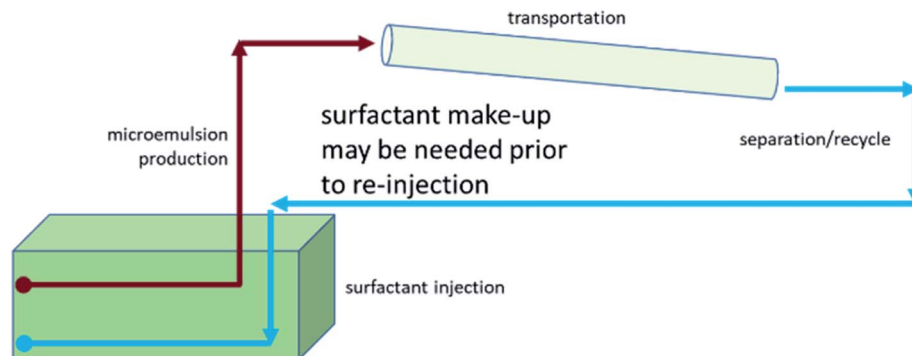


Fig. 3 Flow diagram of the field implementation of closed-cycle oil recovery.

resulting in a significant improvement in recovery and includes unique batch tests and flow experiments.

Results and discussion

Static tests

Two different static tests were performed *adsorption* and *solubilization*. Both tests were uniquely designed to optimize the surfactant formulation for use in dynamic flow tests. Actual Athabasca sand was acquired from Alberta, Canada and the bitumen content of the oil sand was quantified to be 12.8 wt% on average using a Soxhlet extraction procedure with toluene as a solvent (see ESI† for more detail).

Adsorption tests were initially conducted to measure the effect of alkali on surfactant adsorption onto the solids. The surfactant formulation consisted of 0.25 wt% C20-24 IOS, and 0.25 wt% C13 13 PO alkoxy sulfate, with 0.25 wt% 2-butanol for the adsorption static tests.²⁹ The formulation was mixed with 5 grams of clean Athabasca sand (no oil) in centrifugal tubes with a range of alkali (Na_2CO_3) concentrations, while keeping total liquid volume 10 ml. The tubes were tumbled for 3 days and the surfactant concentration in the supernatant was measured by HPLC afterwards (described in the ESI section†). Static adsorption tests revealed that alkali concentration was essential to reduce sand adsorption and improve the performance of the

surfactant formulation. The results are shown in Fig. 4 and in agreement with the literature.³⁷ Static tests were repeated with higher concentrations of sodium carbonate, but the surfactant formulation was not stable above 4 wt% of sodium carbonate possibly due to an effect known as “salting out”.

Solubilization tests were also unique in that they were designed to study the effect of sand particles on the solubilization of bitumen into the microemulsion. The formulation used for the adsorption tests was the basis for these tests: the formulation was varied with respect to the blend ratio of the two surfactants (IOS and sulfate), and two additional co-solvents, isopropyl alcohol (IPA) and tri-ethylene glycol monobutyl ether (TEGMBE) were also studied. Sodium carbonate concentration was maintained at 4 wt%. Glass test tubes were filled with 10 g of oil sand and 10 ml of the desired surfactant formulation. Tubes were sealed, mixed gently, and allowed to equilibrate for 24 hours. The supernatant was collected, analyzed by gas chromatography/mass spectroscopy (GC/MS; see Table SI4†), and demonstrated that as the amount of total surfactant increased, bitumen solubilization increased. The solubilization parameter for oil (volume of oil solubilized/volume of surfactant) was highest with 1 wt% C20-24 IOS and 1 wt% C13 13 PO alkoxy sulfate, 1 wt% TEGMBE, and 4 wt% Na_2CO_3 .

Bitumen solubilization was $60\,000\text{ mg L}^{-1}$ and solubilization parameter was 3. Note that the highest solubilization possible would have been on the order of $128\,000\text{ mg L}^{-1}$ had all the oil within the 10 g of oil sand (12.8 wt% bitumen) been solubilized into 10 ml of surfactant solution. This surfactant formulation was selected for all subsequent flow experiments.

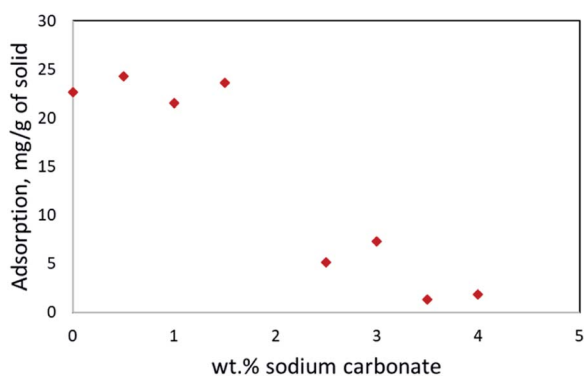


Fig. 4 Surfactant adsorption onto solids with respect to sodium carbonate concentration; note the inflection point at 2 wt% sodium carbonate.

Dynamic tests

Flow experiments were uniquely designed to produce comparative data from five packed columns to optimize the C-COR process. Actual oil sand was used, which is an unconsolidated media and precludes the use of traditional approaches developed for core flooding. As an example, immovable oil (bitumen) occupies a portion of the pore volume at the onset of the experiment so that the porosity cannot be measured. Instead, the bulk water fraction (W_f) denoting the available pore volume for flow was measured in a similar manner as porosity for



consolidated cores. Bulk water fraction is analogous to water saturation in the case of consolidated cores and monitoring the W_f provides a means of quantifying the oil recovery. It is also important to consider that, since the porosity is not known at the start of the flow experiments, bitumen content is not reported in terms of saturation but in terms of mass content. Thus, oil recovery is assessed both gravimetrically and by using W_f (see ESI† for more detail).

A summary of the five test conditions is provided in Table 1; more details of the test setup are provided in the ESI.† The properties of each sand pack measured prior to each dynamic test are similar and listed in Table SI6.† A flow rate of $0.013 \text{ ml min}^{-1}$ (0.274 m per day), vertically upwards, was for all tests except in Test 4 where the rate was doubled. The pressure drop recorded during each experiment was below 0.28 kPa g ($0.18 \text{ m-water per m}$), except for DT 5 that employed a viscosifier where the pressure drop was about 2.76 kPa g ($1.8 \text{ m-water per m}$) on average (see ESI† for more detail). Produced microemulsion was analyzed by GC/MS, and the injection was stopped when bitumen concentration in microemulsion fell below $10\,000 \text{ ppm}$. The selection of this concentration as a stopping criterion was arbitrary; clearly, the field process will reach a point where the produced mass of oil is too low to be continued for economic consideration and the process terminated. In practice, however, that point will depend on economic factors that are not known at this early stage in technology development so a reasonable value was chosen in order to optimize the process.

The results of dynamic tests 1 to 5 are summarized in Table 1. The surfactant formulation exhibited better oil recovery with the addition of sodium carbonate for adsorption reduction compared with tests presented in Sharma *et al.*:²⁹ maximum bitumen concentrations were 3–4 times higher, while oil recovery was between 8–13 times greater. The recovery of original oil in place (OOIP) was calculated using the increase in bulk water fraction and gravimetrically. An interesting observation is made: dynamic test 2 conducted at a slightly elevated temperature of 60°C exhibited the highest recovery, 61% OOIP, but the recovery per PV was not the best. This is an important distinction when factoring costs to decide which of the C-COR process optimizations should be employed. The remaining dynamic tests 3, 4, and 5 achieved roughly the same recovery—on the order of 35% OOIP—with the highest recovery per PV achieved by employing a soaking method. An interesting and unexpected result is noted in terms of recovery per PV of dynamic test 4: a higher flow rate performed well with respect to recovery per PV injected. The various methodologies employed in the dynamic tests indicate that, although the process is affected by dynamic parameters, the main controlling factor is accessibility to bitumen: *i.e.*, macroscopic sweep efficiency of the injected formulation and the solubilized oil. The recovery was constrained by the adverse mobility of the surfactant formulation after solubilizing bitumen (microemulsion). It is helpful, then, to examine each test in more detail to glean useful for scale-up and optimization insight.

Dynamic test 1. The concentration profile normalized with the highest concentration of approximately $30\,000 \text{ mg L}^{-1}$ is

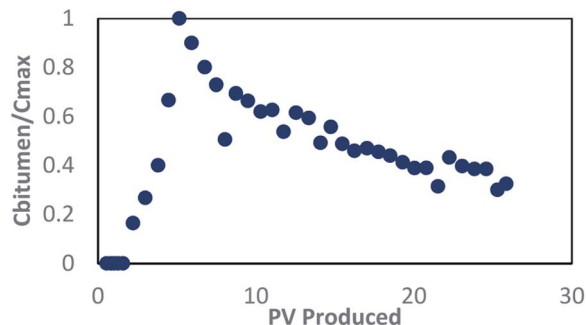


Fig. 5 Concentration profile for bitumen in dynamic test 1; concentrations are normalized by the highest concentration, approximately $30\,000 \text{ mg L}^{-1}$.

shown in Fig. 5. The pressure drop recorded during injection was very low, about 0.28 kPa g on average ($0.18 \text{ m-water per m}$).

The oil recovery was 43.14% OOIP (gravimetric method) before stopping, which was a 10-fold improvement over the flow tests reported by Sharma *et al.*²⁹ that did not address adsorption in the surfactant formulation. The bulk water fraction of the sand pack after the test increased to 22.8%, which was used to estimate an oil recovery of 40.33% OOIP. This value corroborates the value measured gravimetrically. After emptying the sand from the column, it was observed that the sand was lighter in color compared to the original oil sand. The sand near the outlet showed signs that oil recovery occurred more at the periphery of the sandpack, which may indicate possible bypassing (“channelling”) of the injected solution along the sides of the column as the solution moves upwards.

Dynamic test 2. This dynamic test was a replicate of dynamic test 1 with a thermal enhancement at 40°C . A total of 37 pore volumes were injected into the system. The highest bitumen concentration was measured to be approximately $37\,700 \text{ mg L}^{-1}$, marginally higher than dynamic test 1. Fig. 6 depicts the concentration profile during the test, showing that the bitumen concentration remained above the stopping criteria longer, leading to higher oil recovery. From a technical

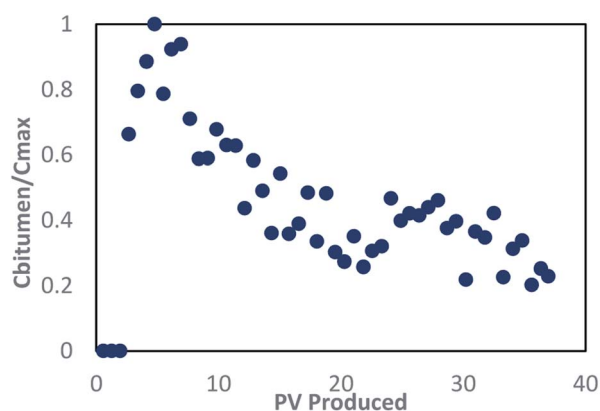


Fig. 6 Concentration profile for bitumen in dynamic test 2, concentrations are normalized by the approximate highest concentration ($37\,700 \text{ mg L}^{-1}$).

perspective, the increased solubilization resulting from thermal enhancement improved performance in terms of oil recovery. Bitumen recovery was 61.07% OOIP (gravimetric method); an increase compared with the base case (dynamic test 1) (Table 2).

The bulk water fraction of the sand pack increased to 27.5% which translates to an oil recovery of approximately 58.12% OOIP recovery. Sand was photographed after removal from the sand pack and, while the sands from the inlet and outlet appeared lighter in color compared to the original oil sand shown in Fig. 7, evidence of flow channelling at the outlet was also observed upon closer examination.

Dynamic test 3. This test employed a “soaking” or intermittent flow approach to allow more time for surfactant formulation interaction with bitumen. One PV of surfactant formulation was injected (13 h), after which the column was isolated for 13 hours to allow the surfactant formulation soak and the process repeated for a total of 13 PVs. The column temperature was maintained at 20 °C for comparison with the base case. The highest bitumen concentration was approximately 35 500 mg L⁻¹, which is slightly higher than the base case and comparable to the test run at an elevated temperature. Oil recovery was 36.5% OOIP (gravimetric method) and 30.63% OOIP (W_f method). Close examination of sand after the test also led to the same observation that recovery at the inlet is better than the outlet. Although the total recovery in this test was lower than the base case, since the stopping criteria for the test (*i.e.*, the bitumen concentration fell below 10 000 mg L⁻¹) was achieved sooner, the bitumen recovery per PV of the surfactant formulation injection was higher. This indicates that the soaking process achieved more solubilization within the first few soaks.

Dynamic test 4. This test explored the effect of higher inertial forces on oil recovery by doubling the injection rate to 0.027 ml min⁻¹ (0.549 m per day). A total of 15 PVs of surfactant formulation was injected before reaching the stopping criteria. The highest bitumen concentration in produced microemulsion was measured to be approximately 35 000 mg L⁻¹—slightly higher than the base case and comparable to both elevated temperature and soaking cases. The recovery calculated gravimetrically was 36.11% and 30.68% using the bulk water fraction difference method. Thus, the higher flow rate resulted in similar oil recovery to the soaking method.

Visual analysis of the sand revealed patterns similar to the previous tests, where more oil recovery at the inlet than the



Fig. 7 Dynamic test 2 sand pack: top – original sand sample; bottom left – inlet sand sample after test, and; bottom right – outlet sand sample after test.

outlet was observed. Based on test 3 and 4, it was evident that, while dynamic processes are affecting bitumen recovery, the rate and degree of solubilization was not the only controlling factor. The amount of bitumen contacted by the surfactant appears to play a significant role in oil recovery. This observation should not be understated: most studies including ours begin with static testing where dynamic studies show the importance of fluid delivery and achieving good conformance—a measure of the uniformity of the flood front of the injected drive fluid.

Dynamic test 5. The final test was conducted using a visco-sifier to improve the macroscopic sweep efficiency or conformance. The surfactant formulation viscosity was 2.24 cP at 20 °C without polymer (at 1 s⁻¹). Surfactant formulations were prepared with a range of polymer concentrations and the viscosities measured at 20, 40, and 60 °C, over a range of shear rates (see ESI† for more detail). A polymer concentration of 2000 ppm was used to increase the viscosity to 18.8 cP, nearly 8.5 times more viscous than the surfactant formulation alone at a shear rate of 1 s⁻¹.

Note that the presence of polymer in microemulsion interfered with GC measurements of oil concentration so it was not

Table 2 Summary and comparison of dynamic test results

Test number	Description	Flow rate (m per day)	PV injected	% OOIP recovery		Recovery per PV
				Using bulk water fraction	Using gravimetric	
1	Baseline	0.274	26	40.33	43.14	1.77
2	Elevated temp	0.274	37	58.12	61.07	1.65
3	Soaking	0.274	13	30.63	36.5	2.81
4	High rate	0.549	15	30.68	36.11	2.41
5	Polymer	0.274	26.5	26.33	34.9	1.32



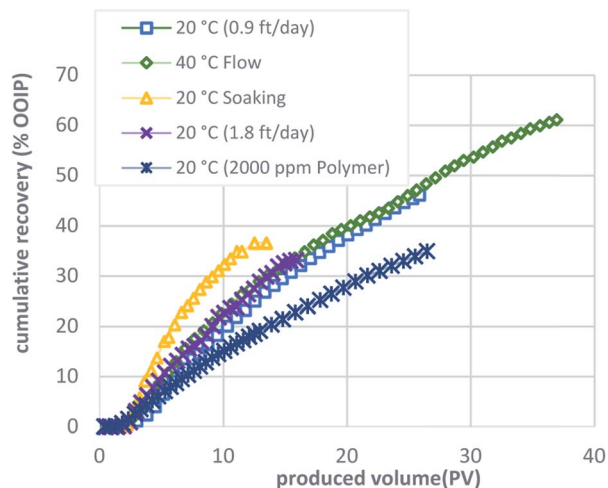


Fig. 8 Comparison of the cumulative oil recovery for the five methods used to implement CCOR. The soaking method resulted in the fastest recovery of over 36% OOIP. Note that each method was terminated once the oil concentration in produced microemulsion.

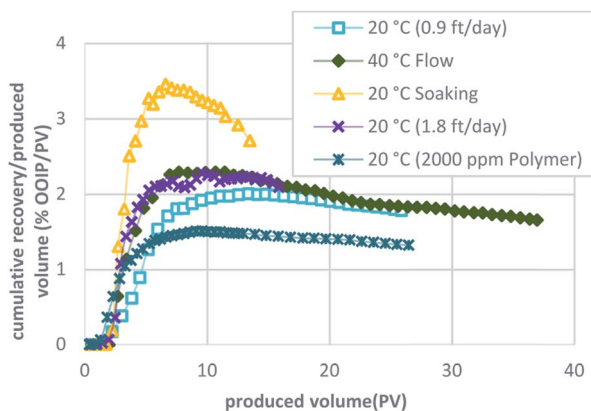


Fig. 9 Comparison of the cumulative oil recovery per produced volume for the five methods used to implement CCCOR shows that the soaking method results in the highest.

possible to use the stopping criteria ($10\,000\text{ mg L}^{-1}$) with certainty. For this reason, the test was stopped after 26.5 PV injection, which is the same throughput for the base case, even though the GC measurement was indicating a concentration value of, above the stopping criteria (approximately $20\,000\text{ mg L}^{-1}$). Oil recovery was lower than the base case, 34.9% OOIP (gravimetric) and 26.33% OOIP (bulk water fraction) using a similar throughput as in the base case. Although a viscosifier was added to improve the conformance of the injected surfactant solution and resulting microemulsion, the sand appeared to have poor overall oil recovery compared with previous tests. The evidence suggests that the addition of polymer to the surfactant formulation may have interfered with the solubilization process and did not improve over the base case. A subsequent static test conducted with the surfactant formulation plus polymer (as used in dynamic test 5) indicated that the amount of solubilized oil measured after 2 weeks of

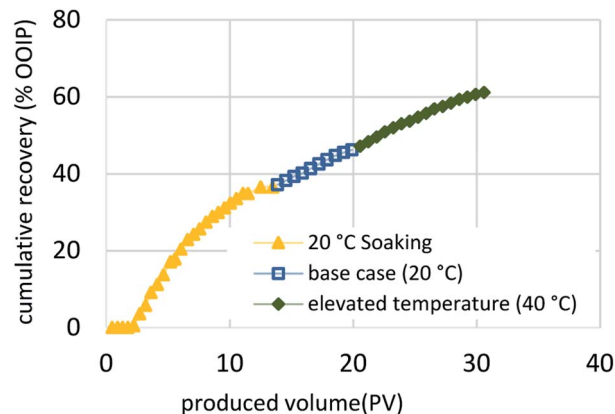


Fig. 10 Cumulative recovery of the proposed, optimized approach begins with soaking followed by continuous flow and ending with thermal enhancement of injected fluids.

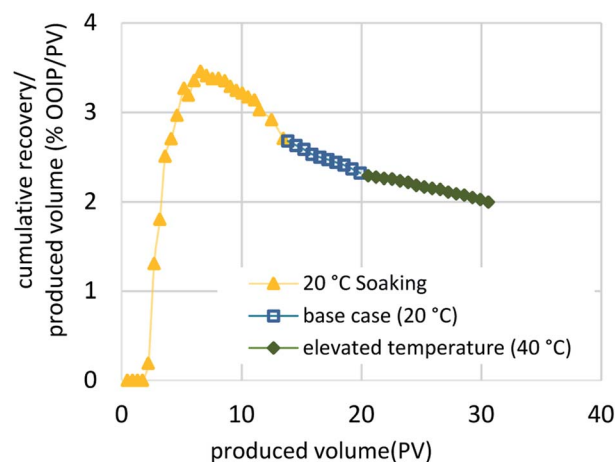


Fig. 11 Cumulative recovery per volume produced of the proposed, optimized approach (soaking, continuous flow, and thermal enhancement).

mixing was lower, corroborating the dynamic test 5 observation that the addition of polymer adversely affected the oil solubilization.

Summary and conclusions

The results of this work, combined with the prior “proof-of-concept” work published by Sharma *et al.*,^{27–29} are encouraging and justify an additional investigation. We need to better understand the effects of both the actual sand and the actual bitumen for scale-up using two- and three-dimensional physical and numerical simulation models. From the results described here, we conclude that low surfactant adsorption onto the media is critical for the effectiveness of the C-COR approach. Phase behavior and batch testing conducted without taking surfactant adsorption into account will lead to extremely low recovery in 1-D flow experiments and affect the economics negatively when applied at field scale. Polymer was observed to adversely affect oil solubilization for this formulation. From the

dynamic test results, it is clear that a high degree of oil recovery is possible with proper surfactant selection, even when employing very high flow rates that would be considered an extreme test of the approach. The dynamic flow test were designed to optimize the process and the results indicated that, although the process is affected by dynamic parameters, oil recovery was constrained by the adverse mobility of the surfactant formulation after solubilizing bitumen (micro-emulsion) and is also affected by accessibility to bitumen.

Fig. 8 compares the five methodologies in terms of oil recovery and indicates that a soaking approach yields highest oil recovery at an early stage, while the same formulation injected continuously (base case) or at an elevated temperature (40 °C) yielded the highest oil recoveries overall. This is also apparent in Fig. 9, where the oil recovery per produced volume are shown for the five methodologies. Thus, the proposed optimized approach shown in Fig. 10 would initially employ a soaking method that would result in the fastest recovery of about one-third of the OOIP, followed by a continuous injection to recovery an additional 10% OOIP, ending with thermal enhancement to recovery 25% OOIP more for a total of 61%. Fig. 11 presents the oil recovery per produced volume for the proposed, optimized approach. Both Fig. 10 and 11 are hypothetical and were developed by super-position of the data from the 3 separate tests: this assumes that the conditions within the oil sand pack are identical at the same stage of the experiments. This is a reasonable assumption when considering that the three experiments were conducted using the same initial conditions, employed the same formulation and flow rates, differing only in terms of the implementation: intermittent flow followed by continuous flow followed by elevated temperature. While it is possible that the distribution of bitumen within the sand pack could be slightly different even when the overall oil saturation is the same, the proposed optimized process is logical in terms of simple economic considerations: the initial scheme would use require less (intermittent) pumping to achieve a high recovery before switching to continuous pumping with an associated higher cost, culminating with higher costs to thermally enhance the process.

Additional work on improving the surfactant formulations is strongly suggested. As a part of surfactant selection, environmental acceptability/toxicity should be considered: since the injection of any fluid into the subsurface has a potential of flow from the targeted zone containing oil to an environmentally-sensitive zone (e.g., an aquifer). In general, surfactants have a lower toxicity level and are more environmentally acceptable over solvents proposed for other heavy oil recovery approaches.³⁹ Optimizing the surfactant formulation may positively impact the process economically. A techno-economic and life cycle analysis for the process, comparing it with currently-employed methods such as SAGD and open-pit mining, would also be beneficial at this point. It will also help to identify whether the higher recovery of oil outweighs the additional cost of thermal enhancement. The proposed C-COR process will also require the development of a suitable separation technique before it can be seriously considered for scale-up for a field application. Surfactant recovery processes have not

been well-studied at field scale. Further research to develop an efficient surfactant recovery process would not only help the C-COR approach but would also benefit the scale-up of other surfactant-based processes designed for the subsurface environment. Finally, optimization studies for the field development and well placement are also required as outlined by multiple authors to make it comparable to existing methods.^{30–32}

Author contributions

Pushpesh Sharma: data curation; formal analysis; investigation; methodology; resources; visualization; writing – original draft. Konstantinos Kostarelos: conceptualization; funding acquisition; methodology; project administration; supervision; writing – review & editing. Mohamad Soliman: investigation; writing – review & editing.

Conflicts of interest

There are no conflicts to declare.

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