Efficiency of Urea Solutions in Enhanced Oil Recovery

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ABSTRACT: We examine the applicability of urea solutions as a novel cost-effective chemical for enhanced oil recovery processes. Two sandpack flooding experiments were conducted using 5 and 10 wt % urea solutions. Another flooding experiment was also carried out using the same sandpack with fresh water and used as a reference. Supporting experiments such as interfacial tension (IFT), viscosity of water in oil (W/O) emulsions, total acid number (TAN), and Fourier-transform infrared (FTIR) spectroscopy were conducted to confirm the generation of in situ surfactants by reacting urea solutions with the naphthenic acids in bitumen and evaluate their impact on the oil recovery. The analyses of FTIR, IFT, TAN, and viscosity measurements support the generation of in situ surfactants that leads to the formation of stable water in oil emulsions and hence a more stable displacement front resulting in higher oil recovery.

1. INTRODUCTION

Global reserves of bitumen and extra heavy crude oils are three times higher than the conventional crude oils with 10 trillion barrels of bitumen around the world.¹ Steam-assisted gravity drainage (SAGD) and cyclic steam stimulation (CSS) are the most common techniques that have been utilized to recover the bitumen. These recovery techniques are associated with high energy consumption for steam generation, excessive water treatment, and greenhouse gas emissions. To overcome these challenges, co-injection of solvents with steam has been suggested.³ Hydrocarbons such as pentane and hexane are examples of the solvents that have been proposed to reduce the required steam in the expanding solvent steam-assisted gravity drainage (ES-SAGD) process.³ Recovery processes such as CSS and SAGD mobilize viscous bitumen through viscosity reduction by heating,³ while in solvent-aided methods, both heat and dilution effects are utilized to mobilize the bitumen. To the best of our knowledge, only a few studies have examined factors other than viscosity reduction to improve the recovery of bitumen using ES-SAGD processes.⁷ Urea is a potential candidate that can synergize the reduction in bitumen viscosity and sweep efficiency improvement.⁹ Several chemicals such as surfactants, alkaline, and ionic liquids have been proposed in the literature to decrease the interfacial tension and change the rock wettability toward more water-wet, thereby improving the sweep efficiency of crude oil.¹⁰–¹⁸

Urea solution has a basic nature due to the presence of amino functional groups in the urea molecular structure enabling it to react with naphthenic acids found in bitumen and generate in situ surfactants.¹⁹ Urea melts at 133 °C and starts to decompose to ammonia and isocyanic acid (HCNO) at 152 °C, which is much lower than usual SAGD temperature.¹⁹,²¹ However, at the steam chamber condition of SAGD, above 200 °C, the urea solution partially decomposes into ammonia and carbon dioxide.⁷ As a result of the decomposition at high temperature, urea combines the advantages of ammonia and carbon dioxide. Ammonia also dissolves strongly in water condensing from steam to generate a basic solution of ammonium hydroxide (NH₄OH), which in turn reacts with naphthenic acids found in bitumen to generate in situ surfactants.²² By virtue of in situ surfactants, water in oil (W/O) emulsion is generated, and thus, the sweep efficiency of the process is improved through the reduction of viscous fingering as a result of a more stable displacement front. Water in oil (W/O) emulsions formed through an alkaline environment are usually more viscous compared to fresh water in the SAGD process, leading to the suppression of viscous fingers formed through the flooding process.²³ Moreover, flooding the reservoir with alkaline solutions change the rock wettability toward more water-wet due to the presence of polar compounds in alkaline solutions, which are adsorbed to the rock surface containing different charges.²³ In addition to the generation of in situ surfactants, ammonia

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effectively reduces the viscosity and the total acid number (TAN) of acidic crude oil making it more valuable and desired for refining.\textsuperscript{22,25,26} According to ASTM D664, the total acid number is the milligram of potassium hydroxide (KOH) that should be added into the solution to neutralize the acidic compounds found in 1 gram of the crude oil.\textsuperscript{27} Carbon dioxide is another important product resulting from the decomposition of the urea solution at the SAGD steam chamber conditions. Carbon dioxide easily dissolves in bitumen and improves oil mobility due to the viscosity reduction, oil swelling, and lowering of the interfacial tension with formation brine.\textsuperscript{8,28,29} Furthermore, carbon dioxide is a non-condensable gas at the conditions of the steam chamber that can reduce heat loss to the overburden formation leading to recovery improvement.\textsuperscript{7,30}

The objective of this study is to investigate the capability of urea solution to improve the sweep efficiency of bitumen via altering the rock wettability toward water-wet and/or reduction of IFT. Sandpack flooding experiments were conducted to investigate the efficiency of urea solutions in the recovery of bitumen. The overall goal of this work is to investigate the generation of in situ surfactants through the reaction of naphthenic acids found in bitumen with urea solutions and improving our understanding of the underlying mechanisms.

2. MATERIALS

Athabasca bitumen with a specific gravity of 1.004 (°API = 9.44), viscosity of 29 cp at 150 °C and 500 psig, and molecular weight of 513 g/mol was used as an oleic phase through the flooding experiments. The molecular weight of bitumen was measured using the freezing point depression technique. A simulated distillation test (ASTM 7169) and the carbon number distribution of the Athabasca bitumen have been reported in the literature\textsuperscript{31,32} High-purity urea provided by Amresco VWR Chemicals was dissolved in deionized water to prepare urea solutions. A sandpack was prepared and packed with fine glass beads of 120 mesh size to generate the porous medium for the flooding experiments.

3. EXPERIMENTAL WORK

3.1. Sandpack Flooding Setup. Flooding experiments were conducted using a 1D sandpack flooding experimental rig shown in Figure 1. The experimental rig consists of an axial vertical core holder packed with glass beads to prepare the sandpack. A hydraulic hand pump is used to apply a vertical pressure on the sandpack to imitate the overburden pressure. A vacuum pump is utilized to vacuum the sandpack and ensure that there is no air trapped in the sand during the flooding tests. A back-pressure regulator designed and made by a machine shop at the University of Calgary is installed to keep the pore pressure at the desired value. A Quizix pump (QX6000) is used to inject water, bitumen, and urea solutions through imbibition and drainage processes. The accuracy of the injection rate of Quizix pumps is ±0.001 cm\textsuperscript{3}, whereas the maximum flow rate and maximum pressure that can be achieved are 50 cm\textsuperscript{3}/min and 41 MPa (6000 psi), respectively. To save the pump from corrosion, the urea solutions are injected indirectly into the sandpack from an external stainless-steel transfer cylinder. A pressure transducer from Sensotec Company with a range from 0 to 100 psi (∼0.7 MPa) is used to measure the pressure drop along the sandpack during the bitumen recovery process. Pressure drop data is recorded and logged using SOLO 9696 data acquisition. All the core holder, back-pressure regulator, and bitumen accumulator were placed inside a temperature-controlled oven from VWR Company to
control the temperature. A heat exchanger is used for heating the urea solution before being injected into the sandpack to ensure that the solution enters the sandpack at the desired temperature. All fluids produced from the sandpack are collected in graduated flasks to obtain the recovery data as a function of time.

### Table 1. Sandpack Petrophysical Properties

| injected solution          | length, cm | bulk volume, cm³ | pore volume, cm³ | porosity, % | absolute permeability, D | irreducible water saturation after oil flooding |
|----------------------------|------------|------------------|------------------|-------------|--------------------------|-----------------------------------------------|
| water flooding             | 26.0       | 736.4            | 273.2            | 37.1        | 14.1                     | 0.127                                         |
| 5 wt % urea solution       | 26.2       | 742.1            | 268.9            | 36.2        | 11.4                     | 0.089                                         |
| 10 wt % urea solution      | 26.0       | 736.4            | 265.4            | 36.0        | 12.3                     | 0.137                                         |

### 3.2. Flooding Experiment Procedures

The axial core holder was packed with glass beads using the hammering technique to achieve porosity and permeability as close as those in the reservoir condition. Then, the sandpack was confined vertically with 1000 psig (~7 MPa) overburden pressure and flooded with carbon dioxide (CO₂) for 30 min at 50 psig (~0.345 MPa) inlet pressure while the outlet was open to the atmosphere to displace air from the sandpack. After that, the sandpack was vacuumed for 1 h to remove CO₂ as much as possible prior to water imbibition. The sandpack was then fully saturated with distilled water and pressurized to 500 psig (3.45 MPa) using the Quizix pump. Porosity and pore volume of the sandpack are reported based on the amount of the water imbibed into the sandpack. Absolute permeability of the sandpack was calculated using Darcy’s law after measuring the pressure drop through the sandpack at different water flow rates ranging from 1 to 16 cm³/min. The sandpack was brought to 150 °C for 3 h to ensure a thermal equilibrium at the desired temperature and then flooded with hot bitumen to establish the initial oil saturation (S_wi). The flooding with hot bitumen was continued for more than one pore volume (PV) of the sandpack to ensure that no further water is produced. The displaced water was cooled and collected using graduated flasks to measure the irreducible water saturation (S_wir) of the sandpack after eliminating the dead volume of tubes. Finally, the urea solution was injected from the top side of the sandpack at a flow rate of 1 cm³/min and 500 psig (3.45 MPa) pore pressure to recover bitumen from the bottom side. In all flooding experiments, a pressure drop of 3–7 kPa was observed. The flooding process was continued until the effluent fluid was only water. The produced emulsion was collected as discrete samples with volumes ranging from 50 to 100 cm³ based on the abundance of bitumen in the produced emulsion. The produced bitumen volume was then used to calculate the residual oil saturation left in the sandpack.

### 4. RESULTS AND DISCUSSION

#### 4.1. Sandpack Flooding Experiments

Three flooding experiments including water flooding, 5 wt % urea solution flooding, and 10 wt % urea solution flooding were conducted. The properties of the sandpacks used in these experiments (length, bulk volume, pore volume, porosity, absolute permeability, and irreducible water saturation) are reported in Table 1. In the first experiment, the water flooding was started at an initial oil saturation (S_wi) of 0.873. More than 5.2 pore volumes (PV), 1430 cm³, of fresh water was injected continuously into the sandpack at a flow rate of 1 cm³/min until an effluent water cut of 99% is reached. The results shown in Figure 2 demonstrate that ~73% of the original oil in place (OOIP) was produced at the end of the hot-water flooding process. In addition, the results show an early water breakthrough at 6% of the PV of water injection.

In the second experiment, the sandpack with an initial oil saturation of 0.911 was flooded with 4.37 PV (1174 cm³) of 5 wt % urea solution until a water cut of 93% was achieved. The results reported in Figure 3 show that the breakthrough happened at 12.2% of the pore volume injection as compared to 6% in the first experiment, which indicates a noticeable delay in the breakthrough time. Around 72% of the original oil in place (OOIP) was produced at the end of the flooding experiment.

In the third experiment, 3.93 PV (1044 cm³) of 10 wt % urea solution was injected into the sandpack at an initial oil saturation of 0.863. The results shown in Figure 4 demonstrate breakthrough time at 12.8% PV of injection, which is comparable with the 5 wt % urea solution injection. About 76% of the original oil in place (OOIP) was produced at the

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**Figure 2.** Oil recovery and water cut for the first scenario (hot-water flooding T = 150 °C). (Red-shaded circle) oil recovery factor, (blue-shaded triangle) water cut.

**Figure 3.** Oil recovery and water cut for the second scenario (5 wt % urea solution at T = 150 °C). (Red-shaded circle) oil recovery factor, (blue-shaded triangle) water cut.

**Figure 4.** Oil recovery and water cut for the third scenario (10 wt % urea solution at T = 150 °C). (Red-shaded circle) oil recovery factor, (blue-shaded triangle) water cut.
end of the displacement with a water cut of 91% for the 10 wt % urea solution flooding process.

4.2. Comparison of the Flooding Experiments. Figure 5 presents the oil recovery factor at one and two pore volumes of injection for all the sandpack flooding experiments. The results show that both 5 and 10 wt % urea solutions outperform water flood at 1 PV of injection, while 5 wt % urea solution is more efficient than water flood and 10% urea solution at 2 PV of injection. The results clearly indicate that the recovery of bitumen has improved when the urea solution is injected. One reason behind the additional oil recovery can be due to the generation of in situ surfactants and subsequent emulsification. The emulsification caused by the presence of surfactants leads to the formation of a highly viscous zone leading to a more stable displacement front (less severe viscous fingering) and thus improves the oil recovery. In addition, in situ surfactants generated as a result of the interaction of urea solutions with naphthenic acids found in bitumen at high temperature facilitate the dispersion of water droplets inside the oil phase. In the following section, we have measured the interfacial tension (IFT) of the urea solutions with bitumen, viscosity of W/O emulsions, the total acid number (TAN), and Fourier-transform infrared (FTIR) spectra to further support this observation.

4.3. Measurements of Interfacial Tension (IFT). Two concentrations of urea solutions (10 and 15 wt %) were prepared to measure the interfacial tension (IFT) with bitumen at 100 °C and 500 psig (3.45 MPa) using a pendant drop tensiometer as shown in Figure 6.37 The tensiometer has a high-pressure, high-temperature isolated cell with a volume of 600 cm³. A Quixiz pump was utilized to inject a small droplet of bitumen from a standard needle into the isolated cell, which was prefilled with urea solution. Two thermocouple probes were used to monitor and control the temperature inside and outside of the isolated cell using a temperature controller. A cell containing water and pressurized by nitrogen was connected into the system to keep the pressure of the system constant at 500 psig (3.45 MPa). The light, digital camera, and software from Rame-hart Instrument Company were used to record video frames and process them to obtain interfacial tension using the Young—Laplace equation.33 The densities of water and bitumen are nearly equal at 150 °C, which makes the IFT measurements using the pendant drop tensiometer very difficult if not impossible. For that reason, IFT measurements for bitumen/water and bitumen/urea solutions using the pendant drop tensiometer were not feasible. However, for the purpose of this study, a qualitative trend of the IFT would allow interpretation of our experimental observations of the bitumen recovery factor. To increase the density difference between bitumen and urea solutions, the IFT measurements have been conducted at high concentrations of urea (10 and 15 wt %) at 100 °C. The IFT measurements were started as soon as the oil drop was formed at the tip of the needle, and the IFT data were recorded at the rate of 1 frame/s for 2000 s. To ensure the reproducibility of the results, each experiment was repeated twice, and the final value was reported as an average of the last 100 s experimental data. The IFT of bitumen and water measured by the pendant drop method at 90 °C has been reported to be 34 mN/m.34 The results shown in Figure 7 reveal that the interfacial tension of the urea solution and bitumen decreases by increasing the urea concentration. This observation supports the fact that urea promotes water and oil emulsification due to the generation of in situ surfactants,34−36 leading to additional recovery of bitumen.

4.4. Measurements of Viscosity of W/O Emulsions. Two W/O emulsions were prepared by stirring 15 g of bitumen with 30 g of 5 and 10 wt % urea solutions for 2 days at 150 °C and 400 rpm. Then, the mixtures were left for two weeks to allow the separation of the two stable phases including W/O emulsion on the top and O/W emulsion at the bottom. Small samples of each W/O emulsion were charged into the pressure cell of an MCR 302 Anton Paar rheometer to

Figure 5. Oil recovery factor of all three flooding experiments at one (left) and two (right) pore volumes injected.
measure the viscosity of emulsions at 150 °C and 500 psig (3.45 MPa). The results shown in Figure 8 reveal that the W/O emulsion treated with 10 wt % urea solution has a higher viscosity compared to 5 wt % urea solution in the typical shear rate of 20−100 s⁻¹. The high viscosity of the W/O emulsion treated with 10 wt % urea solution is attributed to the small droplet size of water inside oil through generation of W/O emulsion. The measured higher viscosity of the 10 wt % urea solution is consistent with the lower interfacial measurements for the same solution. Certainly, viscosities of these emulsions are higher than viscosity of oil in water (O/W) emulsions produced through the water flooding, which are less than 1 cP at the operating condition of our experiments. The high viscosity of W/O emulsions generated through urea solution flooding leads to the suppression of viscous fingering and delays the water breakthrough to 0.12 PV compared to 0.06 for the case of water flooding.

4.5. Measurements of Total Acid Number (TAN). The total acid number of the untreated bitumen was measured to be 3.768 mg/g. To measure the total acid number of the urea-treated bitumen, three samples of the bitumen produced from each experiment (5 and 10 wt % urea solution flooding) were collected separately at different times. The total acid number (TAN) of each sample was measured three times using a Metrohm 848 Titrino Plus instrument, and the results are reported in Table 2. First, 1.5 g of each sample was diluted in

| Table 2. Total Acid Number (TAN) of Treated Bitumen at Different Collection Times |
|---------------------------------|-----------------|----------------|-----------------|
| solution                        | collection time, h | TAN measurements (mg/g) | average (mg/g) |
| untreated bitumen                |                 | 3.77            | 3.77            | 3.77            |
| 5 wt % urea solution             | 7               | 1.46            | 1.62            | 1.46            | 1.51            |
| 5 wt % urea solution             | 13              | 1.86            | 2.10            | 1.95            | 1.97            |
| 5 wt % urea solution             | 18              | 2.29            | 2.08            | 2.18            | 2.18            |
| 10 wt % urea solution            | 6               | 2.54            | 2.45            | 2.50            | 2.50            |
| 10 wt % urea solution            | 10              | 1.91            | 1.76            | 1.59            | 1.75            |
| 10 wt % urea solution            | 19              | 1.97            | 2.06            | 1.82            | 1.95            |
10 cm³ of toluene to break the W/O emulsion formed through the flooding process. Then, the solution was washed four times with 40 cm³ of deionized water and centrifuged for 2 min at 4000 rpm to remove acidic water droplets and urea contaminations from the treated bitumen. The arithmetic average of the TAN values as a function of the pore volume injected (collection time) are reported in Table 2. The results shown in Figure 9 reveal a significant reduction in the total acid number of the urea-treated bitumen. The TAN values for the produced bitumen through the urea solution flooding decrease sharply for the first two pore volumes of injection followed by gradual increases over time. The results shown in Figure 9 confirm the reaction of urea solution by the naphthenic acids as evidenced in TAN reduction and formation of stable water in oil emulsion in sandpack flooding experiments. The sharp decrease in the total acid number is due to the sufficient contact and the reaction between the urea solution and the naphthenic acids in bitumen. The small gradual increase in TAN may be attributed to the poor contact between the injected urea solution and bitumen at the later stage of the displacement process that prevents the reaction with the naphthenic acids.

4.6. Fourier-Transform Infrared Spectroscopy (FTIR). The FTIR spectroscopy provides information on the chemical structure and composition of bitumen. Representative FTIR spectra of bitumen samples obtained after the water flooding experiment and reactions with 5 and 10 wt % urea solutions are presented in Figure 10. The FTIR spectra of all samples indicate expected features as evident in the FTIR spectrum of the water flooding sample is around 1700 cm⁻¹. As seen in Figure 10, the carbonyl peak in the water flooding sample is around 1700 cm⁻¹. Interestingly, after the reaction of bitumen with 5 or 10 wt % urea solutions at 150 °C, the carbonyl peak shifts to around 1670 cm⁻¹ and becomes more evident in the FTIR spectra of 5 and 10 wt % urea-treated samples. This is an indication of the formation of amide functional groups and consequently verifies the formation of amide surfactants through the reaction of carboxylic acids found in bitumen with urea in 5 and 10 wt % urea solutions.

5. CONCLUSIONS

Three sandpack flooding experiments were conducted to investigate the efficiency of urea solutions as a novel solvent for solvent-aided thermal recovery processes. Our results indicated that urea solutions positively impact the oil recovery efficiency, highlighting the synergy between the reduction in viscosity of bitumen and generation of in situ surfactants through the flooding process. The results indicated that emulsification at the displacement front leads to formation of an emulsion that has significantly higher viscosity than the aqueous urea solution, leading to a more stable displacement front through the suppression of viscous fingering and consequently higher oil recovery. Interfacial tension measurements revealed a noticeable reduction in the interfacial tension of the bitumen/water in the presence of urea in the aqueous phase solution. In addition, measurements verified a significant reduction in total acid number (TAN) of the treated bitumen. To reveal the source of the IFT and TAN reductions, FTIR measurements were conducted. The analysis of the FTIR measurements indicated that the reaction of carboxylic acids found in bitumen with the aqueous urea solutions led to the formation of amide functional groups and thus the formation of amide surfactants, which confirms IFT and TAN reductions. Comparisons of the flooding experiments revealed that there is an optimum concentration of urea solution that leads to more efficient displacement of oil by urea solutions, which may be due to the effect of urea solutions on the wettability of the sand. Future studies should focus on the wettability alteration of sands due to the generation of in situ surfactants resulted from the reaction of urea solutions with naphthenic acids.

Table 2. Total acid number (TAN) of bitumen treated with 5 and 10 wt % urea solutions at different collection times.

| Pore Volume Injected (PVI) | TAN Ratio |
|---------------------------|-----------|
| 0                         | 1.00      |
| 1                         | 0.75      |
| 2                         | 0.50      |
| 3                         | 0.25      |

Figure 9. Total acid number (TAN) of bitumen treated with 5 and 10 wt % urea solutions at different collection times. (Green-shaded circle) 5 wt % urea solution, (red-shaded triangle) 10 wt % urea solution.

Figure 10. Fourier-transform infrared spectra of bitumen samples obtained after the water flooding experiment and reactions with 5 and 10 wt % urea solutions.

![Figure 10](image)

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Notes
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