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# Efficiency of Ionic Liquid 1-Ethyl-3-Methyl-Imidazolium Acetate ([EMIM][Ac]) in Enhanced Medium Oil Recovery

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## Abstract

Chemical flooding is one of the most efficient methods for Enhanced Oil Recovery (EOR). This study demonstrates the efficiency of mixing different concentrations of Ionic Liquid (IL), 1-Ethyl-3-Methyl-Imidazolium Acetate ([EMIM][Ac]), with Weyburn brine to improve a medium oil recovery, Weyburn oil, from an unconsolidated sand pack sample at room conditions. Effects of Slug Size (SS), IL + brine slug initiation time, and combining IL with alkali on the Recovery Factor (RF) were investigated. This study showed that the optimum concentration of ([EMIM][AC]) was 1000 ppm and the most efficient injection time of the chemical slug was at the beginning of the flooding procedure (as secondary flooding mode). In addition, it was proved that the potential of injecting a slug of IL + brine is much better than that of introducing a slug of alkali + brine. Besides, the combination of IL and alkali (AIL) resulted in better RF than injecting either of them alone. Finally, the Surface Tension (SFT), pH, wettability alteration, and viscosity of the displacing phases were measured.

## Keywords

[EMIM][Ac], Medium Oil, IL Concentration, Slug Size, Injection Time

## 1. Introduction

It is crucial to increase oil production from existing reservoirs due to the lack of new oil reservoirs discovered around the world as well as the high cost of exploration and reduction in reservoir drive mechanism [1]. Therefore, most researchers focus on studying new enhanced oil recovery (EOR) techniques. Many

(EOR) techniques had been proven to increase oil recovery after reservoirs had lost their natural energy [2]. Recently, chemical enhanced oil flooding (CEOR) methods have received significant attention to improve oil recovery using surfactants flooding, polymers flooding, alkalis flooding, and alkaline-polymer-surfactant flooding [3] [4]. Chemical flooding plays a significant role in reducing interfacial tension, changing the rock wettability and improving the sweeping efficiency by increasing the viscosity and increasing the mobility ratio [5] [6]. However, the application of surfactant flooding is limited due to high toxicity, high cost, low efficiency in harsh environments of high salinity, and adsorption on formation rock which causes formation damage [7]. Due to the increase in oil demand, the necessity of finding new chemicals as alternatives to surfactants is growing.

Today, injecting ionic liquids (IL) has become a pivot application in the oil industry to increase oil recovery. There are many ionic liquid types such as organic salts which have a melting temperature lower than 100˚C [8]. Additionally, ILs that are used as demulsification to desalt water and salts resulting from crude oil and water emulsions [9]. Compared to other commercial chemicals, ionic liquids have many advantages such as their commercial availability, solvent stability, non-corrosiveness, recyclability, and low toxicity [10]. Many studies presented the extraction of more than 90% of bitumen from oil sand by using several IL (imidazolium base) mixed with non-polar solvents [11] [12] [13]. Hezave *et al.* (2013), have reported the effects of using-dodecyl-3-methayl-imidazolium chloride ([DMIM][Cl]) at different temperature for enhanced oil recovery; they found that ([DMIM][Cl]) was able to reduce dynamic interfacial tension between one of Iranian oils and high salinity formation brine to improve oil recovery [14]. Moreover, increasing ILs concentration in salt water resulted in an increase in oil recovery factor from sandstone and carbonate reservoirs [15]. On the other hand, Tunnish in 2016 reported that IL 1-Ethyl-3-Methyl-Imidazolium Acetate ([EMIM][Ac]) mixed with different synthetic brines was more efficient to extract pelican heavy oil from sand pack column when mixed with low salinity aqueous solution. The results showed that the reduction in interfacial tension between the oil and displacing mixtures was marginal [16]. Moreover, Pereira *et al.* studied the effect of different ILs brine solutions on oil recovery; the highest recovery was obtained by 1-ethyl-3-methyl-imidazolum tosylate ([$C_2$MIM][OTS]) which was able to extract up to 88% trapped oil after 4 pore volume (PV) IL solution was flooded into core sample [17]. However, selecting IL and its optimum concentration to improve oil recovery depends on reservoir rock types and reservoir fluid properties.

In this research, experimental measurements were used to study the ability of the IL 1-Ethyl-3-Methyl-Imidazolium Acetate ([EMIM][Ac]) to improve medium oil recovery. Different ([EMIM][Ac]) concentrations mixed with formation brine using different slug size were flooded into an unconsolidated sand pack at room conditions in different scenarios. Moreover, the effect of injecting alkali into sand pack was investigated. Also, the effect of IL on SFT, pH, wettability alteration, and displacing phase viscosity were investigated for the ([EMIM][Ac]) mixtures and their impact on chemical EOR recovery mechanisms.

## 2. Experimental Work

### 2.1. Materials

In this study, 1-Ethyl-3-Methyl-Imidazolium Acetate ([EMIM][Ac]) with purity of 95 wt% and alkali ($Na_2CO_3$) were supplied from Sigma-Aldrich and used without further purification. The chemical structure of the employed IL ([EMIM][Ac]) is shown in Figure 1. The medium oil sample (API° 30.25) and brine were obtained from Weyburn reservoir formation. The properties of Weyburn oil and brine are presented in Table 1. The properties of the sand pack are shown in Table 2.

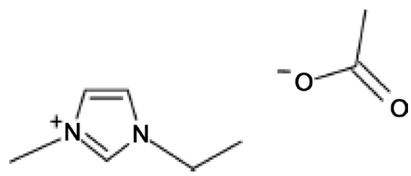

Figure 1. Chemical structure of ([EMIM][Ac]).

Table 1. Properties of brine, oil, and displacing fluids at 21.5˚C.

| Brine composition (Fraction) | | | |
|---|---|---|---|
| **Cations** | | **Anions** | |
| Na | 0.3379 | Cl | 0.571 |
| K | 0.0084 | Br | |
| Ca | 0.0259 | I | |
| Mg | 0.0058 | $HCO_3$ | 0.0091 |
| Ba | 0 | $SO_4$ | 0.414 |
| Sr | 0.0006 | $CO_3$ | 0 |
| Fe | 0 | OK | 0 |
| Mn | 0 | $H_2S$ | |

| | | | | |
|---|---|---|---|---|
| | **Brine $\mu$ (cP)** | 1.017 | | |
| | **Brine $\rho$ (gm/cm$^3$)** | 1.06645 | | |
| | **Oil $\mu$ (cP)** | 15.355 | | |
| | **Oil $\rho$ (gm/cm$^3$)** | 0.87481 | | |
| | **Oil API** | 30.25 | | |
| **Oil SARA fraction (wt%)** | Saturates | 60.3 | | |
| | Aromatics | 24.1 | | |
| | Resins | 10.5 | | |
| | Asphaltenes | 3.15 | | |
| **Displacing fluid (IL + Brine)** | $C_{([EMIM][Ac])}$ | | $\rho$ (gm/cm$^3$) | $\mu$ (cP) |
| | 1000 ppm | | 1.075 | 1.437 |
| | 3000 ppm | | 1.081 | 1.597 |
| | 5000 ppm | | 1.083 | 1.621 |
| **(Alkali + Brine)** | $C_{(Na_2CO_3)}$ | | | |
| | 5000 ppm | | 1.071 | 1.021 |

Table 2. Petro physical properties of sand pack samples.

| PV ($cm^3$) | Ø (%) | K (Darcy) | $S_{wi}$ (%) | $S_{oi}$ (%) |
|---|---|---|---|---|
| 98.5 (±2) | 41 (±2) | 5.2 (±0.5) | 17 (±1) | 83 (±1) |

## 2.2. Measurements

The selected IL ([EMIM][Ac]) for this study was combined at different concentrations with Weyburn brine. The following steps were taken before the flooding process:

1) Prepare the IL mixtures by adding the following amount of ([EMIM][Ac]), (1000 ppm, 3000 ppm, and 5000 ppm), to the brine, the solution is placed on a stirrer (Cole-Parmer Stable Temp Ceramic Stirring Hot Plate) at 120 rpm for 30 - 45 mints.
2) The viscosity of Weyburn oil (15.355 cP), Weyburn brine (1.07 cP), and displacing solutions was measured by using an A Brookfield DV-II viscometer.
3) An Anton Paar DSA 5000 M instrument was used to measure the densities of the aqueous solutions.
4) KRUSS K100 device was used to measure the surface tension of the displacing mixtures using Wilhelmy plate method. Clean the sample vessel and the plate by acetone first and then by clean water before every measurement. Also, the lower edge of the plate is placed straight and parallel to surface of the liquids.
5) The pH of the displacing phases was measured by a Navi pH Meter. An average value was calculated after every third measurement.

## 2.3. Sand Pack Preparation

A sand pack column with a bulk volume of 235.7 $cm^3$ was packed with dry Ottawa sand to prepare an unconsolidated sand pack. The average size of the sand, 40 - 80 mesh, was measured using sieving analysis. The PV, porosity, absolute permeability, and fluid saturations were measured and listed in Table 2.

## 2.4. Flooding Procedure

A vertically oriented core holder, with a length of 18.75 cm and an inside diameter of 4 cm, was packed with dry unconsolidated sand. After packing the sand and fixing the caps, the sand pack sample was 100 % vacuumed using a pump until no air bubble came out and then saturated by Weyburn brine to obtain the porosity from the difference between the dry and saturated weight divided by brine density and bulk volume. The column was injected by brine at different injection rates to determine the absolute permeability using Darcy's Law. After taking the petrophysical properties, the core holder was connected to the core flooding system, a conventional core flooding system, vertically and then the sand pack samples was flooded by the medium oil at a rate of 1 cc/min until no water drop came out from core holder outlet. Total displaced brine represents the original oil in place while the remaining represents the irreducible water saturation. In the next stage, the injection rate was held at a constant rate of 2 cc/min, and the core was placed horizontally and flooded by brine and IL mixtures at different scenarios to obtain the optimum concentration, slug size as well as initiation time. Eventually, all the above procedures were repeated in each experiment with fresh sand to maintain the same properties.

## 3. Results and Discussion

### 3.1. Critical Micelle Concentration

Surface tension (SFT) was considered to determine the Critical Micelle Concentration (CMC) of the displacing solutions at room conditions. The CMC is the concentration at which surfactant solutions surface tension could not be reduced further, as the concentration increased [18]. Moreover, ([EMIM][Ac]) has the capability to reduce the SFT regardless of solutions salinity [16]. As shown in **Figure 2**, a noticeable reduction in the SFT was observed, as the concentration of the IL increased from 0 ppm to 1000 ppm. Any concentration greater than 1000 ppm resulted in a slight increase in SFT values. It was observed that 1000 ppm of ([EMIM][Ac]) has the highest potential to reduce the SFT of the aqueous solution. So the concentration of 1000 ppm was considered as the CMC of ([EMIM][Ac]). For alkali ($Na_3CO_2$), as shown in **Figure 3**, 5000 ppm was considered as the CMC.

### 3.2. pH and Conductivity Behaviors of Aqueous Solutions

The pH values of ([EMIM][Ac]) and Weyburn brine mixtures were measured at 21.5˚C. It was found, in another study, that the pH values of the solutions increased with increasing ([EMIM][Ac]) concentration in the mixtures while the effect of temperature was marginal [19]. As expected, pH values decreased with increasing water content. Additionally, the alkali has the same effect on the pH as presented in **Table 3**.

Electrical conductivity of displacing phases was measured which increased

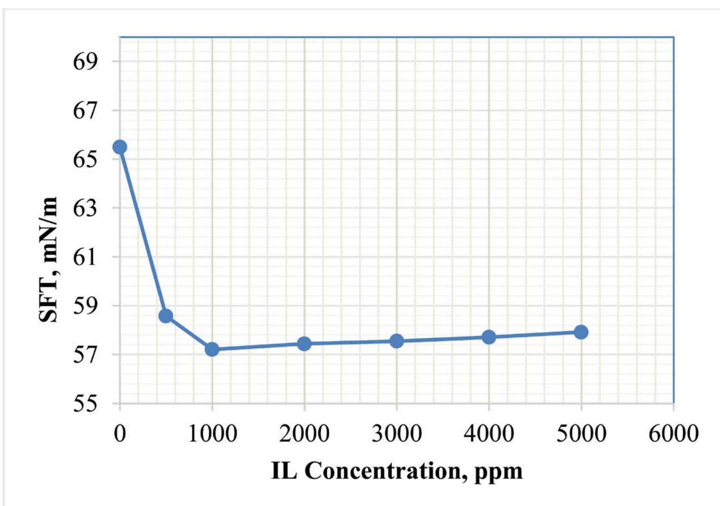


**Figure 2.** Effect of ([EMIM][Ac]) concentration on SFT of displacing phase.

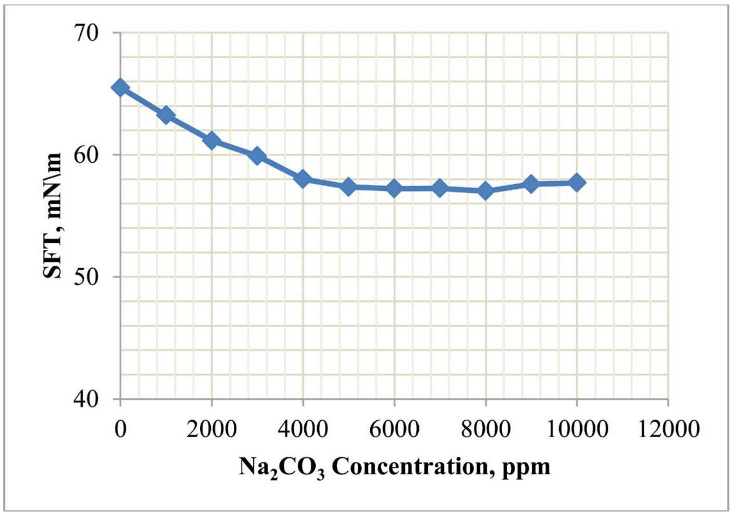


**Figure 3.** Effect of ($Na_2CO_3$) concentration on SFT of displacing phase.

**Table 3.** pH and conductivity measurements of IL and Alkali + brine mixtures.

| Liquid sample | Mixture concentration (ppm) | Conductivity S\m | pH |
|---|---|---|---|
| **Brine** | - | 11.72 | 7.256 |
| **([EMIM] [Ac])** | 500 | 11.65 | 7.255 |
| | 1000 | 09.48 | 7.356 |
| | 2000 | 10.02 | 7.359 |
| | 3000 | 10.51 | 7.361 |
| | 4000 | 11.01 | 7.378 |
| | 5000 | 11.23 | 7.396 |
| **($Na_2Co_3$)** | 5000 | 10.56 | 7.346 |

with increasing ([EMIM][Ac]) concentration. Similar to the CMC values that obtained from conductivity as function of tributylmethyl phosphonium dodecylsulfate concentration which was in good agreement with the CMC that attained from surface tension measurements [20]. The CMC point obtained from surface tension measurements for ([EMIM][Ac]) + brine solutions was in a great agreement with that obtained from conductivities values versus ([EMIM][Ac]) concentrations.

### 3.3. Effect of ([EMIM][Ac]) Concentration on the RF

After preparing the sand pack sample in each experiment, the rock properties were measured, as presented in Table 2. In order to obtain the optimum ([EMIM][Ac]) concentration of displacing phase, three sand pack flooding experiments were performed with three different concentration (1000 ppm, 3000 ppm, and 5000 ppm) and compared with injecting brine alone into sand pack. The flooding process was divided into three stages. First, the sand pack was flooded for one pore volume by formation brine (as secondary recovery mode) followed by one pore volume of IL mixtures. Finally, the sand pack sample was flushed by brine for one pore volume. As shown in Figure 4, the produced oil

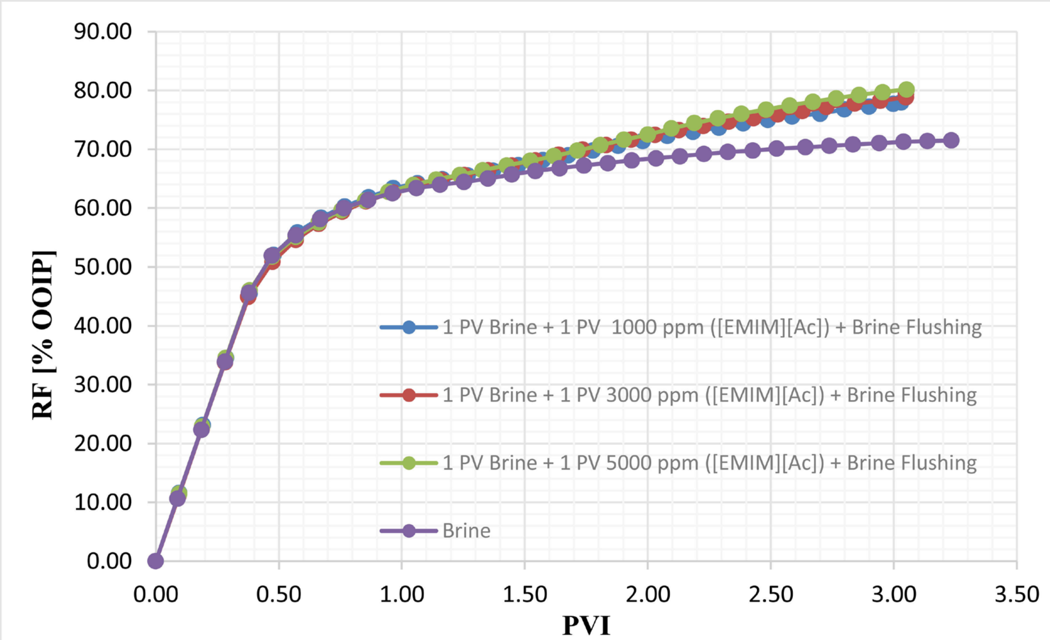


**Figure 4.** Effect of ([EMIM][Ac]) concentration on the RF in tertiary flooding.

was almost identical and the RF values are close to (63% ± 1%) at the end of the first stage. The increase in IL concentration from 1000 ppm to 5000 ppm increased the RF from 77% to 80.64% of original oil in place (OOIP) at end of flooding. Compared to using only water flooding which recovered about 71.17% of the oil in the sand pack sample, the addition of (1000 ppm, 3000 ppm, and 5000 ppm) ([EMIM] [AC]) with the displacing fluid lead to an increase in the oil recovery by 6.57% OOIP, 7.37% OOIP, and 8.77% OOIP, respectively. Finally, as we can see in Figure 4, the increasing on ([EMIM][Ac]) concentration more than 1000 ppm in the mixtures was not efficient to recover more oil economically. Also, the CMC point from SFT and conductivity values were obtained at 1000 ppm ([EMIM][Ac]) + brine solution.

### 3.4. Effect of ([EMIM][Ac]) Slug Size on the RF

Three experiments were performed to select the optimum slug size (SS). In those experiments, the sand pack samples were initially flooded with 1.25 PV of formation brine, followed by different SS (0.5, 1 and 2 PV) of 1000 ppm ([EMIM] [Ac]), and then the samples were flushed with formation brine. As shown in Figure 5, the total obtained RFs of injecting 0.5, 1, and 2 PV SS were 74.85% OOIP, 77.21% OOIP and 77.23% OOIP, respectively. Regarding the RF results, it is obvious that 1 PV of 1000 ppm ([EMIM] [Ac]) is the optimum SS.

### 3.5. Effect of ([EMIM][Ac]) Flooding Initiation Time on Improving Oil Recovery

In order to obtain the appropriate initiation time of the chemical slug, the optimum concentration and slug size of ([EMIM][Ac]) were investigated at three different injection times, as shown in Figure 6. First, the sand pack was initially

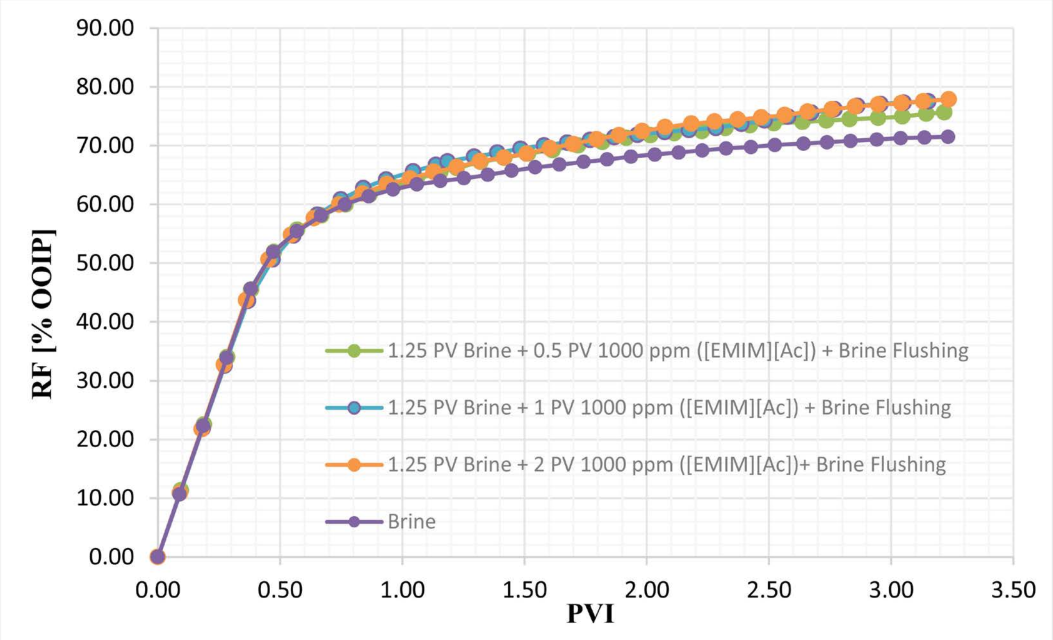


**Figure 5.** Effect of ([EMIM] [AC]) slug size on the recovery factor in tertiary recovery mode.

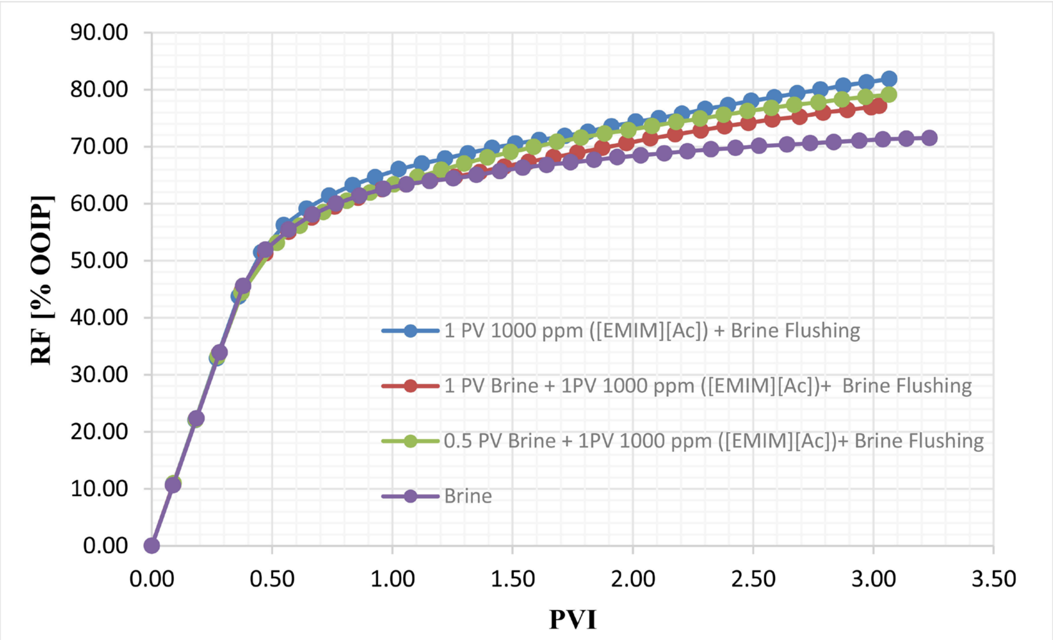


**Figure 6.** Effect of slug initiation time on the RF.

flooded with 1 PV of 1000 ppm ([EMIM][Ac]), and then it was flushed with 2 PVs of formation brine. The results showed that the RF increased from 71.17% OOIP of only brine flooding to 81.31% OOIP. Second, the injection time investigation occurred when the sand pack was flooded with 0.5 PV formation brine; followed by 1 PV of 1000 ppm ([EMIM][Ac]), then finally, the sample was flushed with 1.5 PV of formation brine, as shown in Figure 5. The accumulative RF for this run was 79.01% OOIP, which is less efficient than that of starting the flooding with the chemical slug. The last examined injection time of the chemical slug started with introducing 1 PV of formation brine to the porous medium, followed by 1 PV of 1000 ppm ([EMIM][Ac]), and then flushed with 1 PV of formation brine. The RF was just 76.95% OOIP, which is less than the final RF of the two previous tests. In conclusion, It is obvious that the earlier the injection of the chemical the better the achieved RF.

### 3.6. Effect of Continuous Injection (3 PV) of 1000 ppm ([EMIM][Ac]) on the RF

In this section, the efficiency of injection 3 PV of 1000 ppm on the RF was studied. As can be depicted from Figure 7 depicts the total RF (84.41% OOIP) and it is noticeably better than that of injecting only Weyburn brine. Apart from the economics, the continuous flooding of the chemical mixture is also more efficient than chemical slug and injection.

### 3.7. Effect of Alkalis/Ionic Liquid Slug on RF

One of the upsides of injecting alkali is its ability to react with oil component in order to generate surfactants [21]. Before injection alkali/IL slug, the optimum concentration (5000 ppm) of alkali ($Na_2CO_3$) based on SFT measurements was obtained. Therefore, 1 PV slug with 5000 ppm $Na_2CO_3$ was introduced from the beginning of the flooding process and followed by 2 PVs of Weyburn brine to flush the core sample. In comparison to the results of injection brine alone, an extra 4% OOIP was recovered due to the potential of the added alkali. In addition, combing IL with alkali resulted in much better RF than injection just alkali. When 1000 ppm of ([EMIM][Ac]) was added to 5000 ppm ($Na_2CO_3$) + Weyburn brine slug, the RF was improved from 77.52% OOIP of injecting alkali + Weyburn brine slug alone to 83.95% OOIP, as shown in Figure 8.

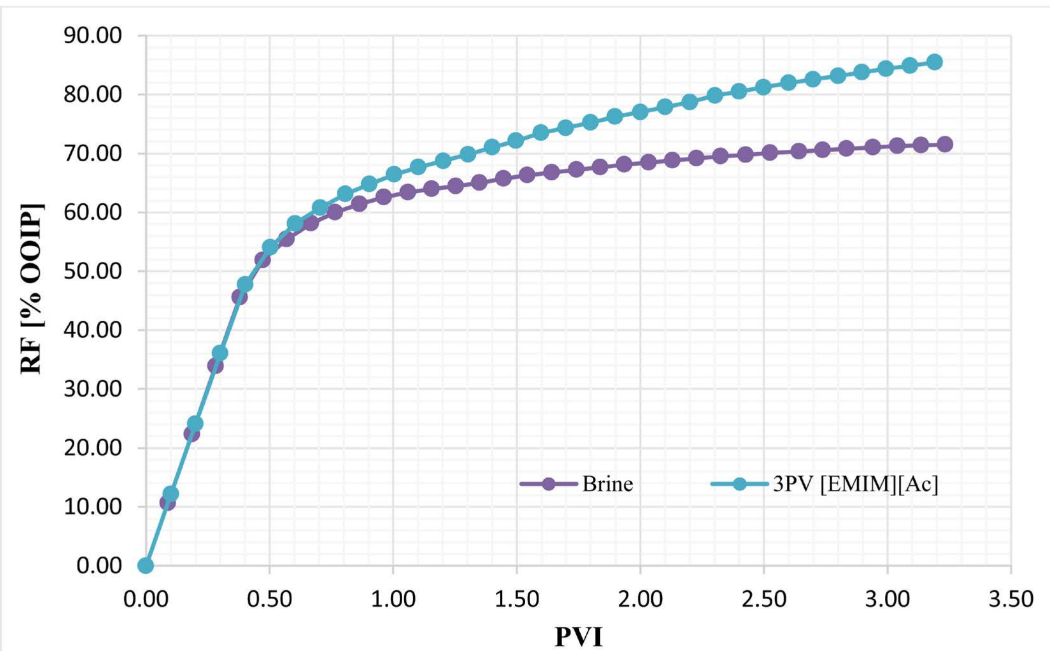


Figure 7. Effect of ([EMIM][Ac]) on the recovery factor in different recovery mode.

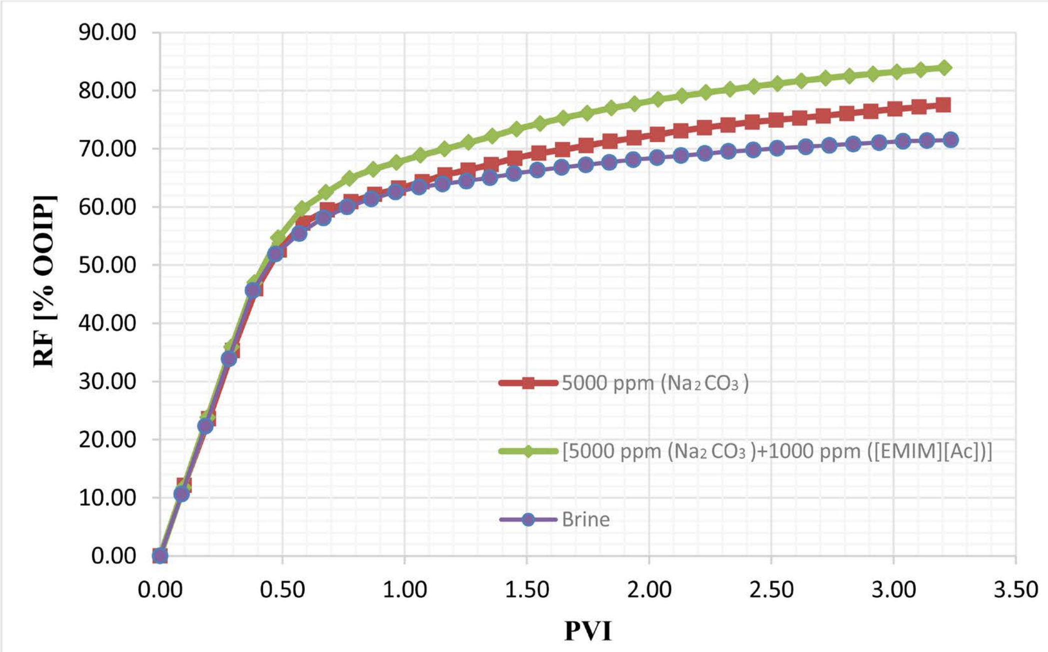


Figure 8. Effect of ($Na_2CO_3$) on the RF in secondary recovery mode.

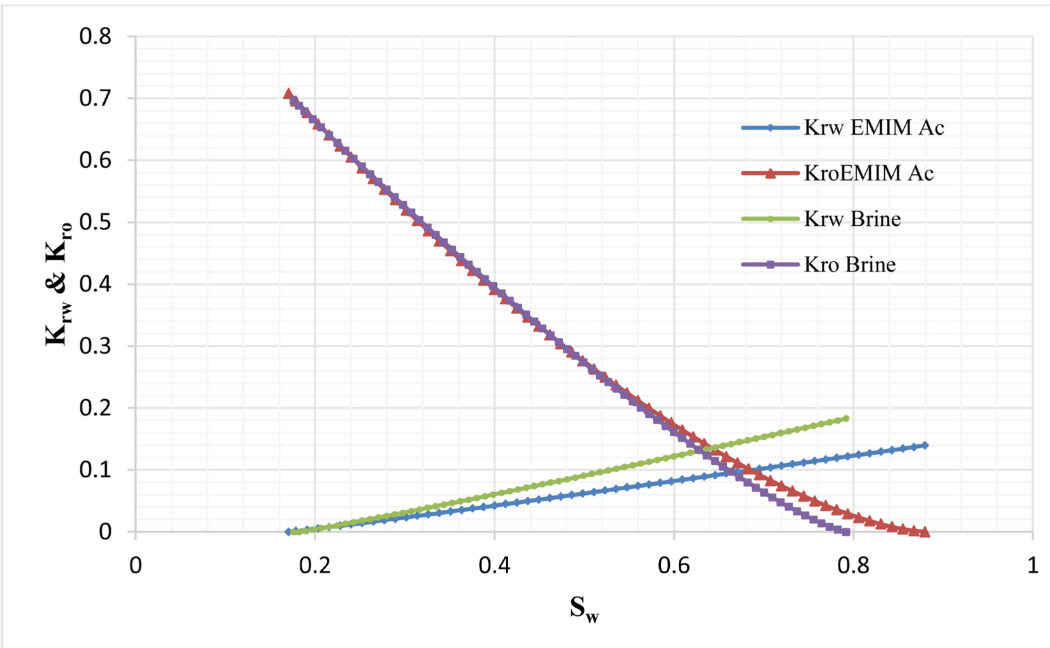


Figure 9. Relative permeability curves of continuous flooding with brine and [EMIM] [Ac] solution.

### 3.8. Relative Permeability Curves

To investigate the effect of ILs on wettability alteration, relative permeability ($k_{ro}$ & $k_{rw}$) curves have been calculated and plotted for two flooding experiments (IL + Weyburn brine flooding and Weyburn brine flooding only) under the same conditions. So, $k_{ro}$ and $k_{rw}$ of 1000 ppm ([EMIM][Ac]) + Weyburn brine and just Weyburn brine were calculated using step by step graphical technique that was explained by Jones and Roszelle (1978) [22]. As shown in Figure 9, the rock water wetness increases and the residual oil saturation decreases for IL slug flooding comparing with brine flooding. These outcomes confirm the enhancement in the RF results in our study.

## 4. Conclusion

This paper studied the application of ([EMIM][Ac]) as an alternative surfactant to increase the medium Weyburn recovery factor from unconsolidated Ottawa sand pack at room conditions. The work began by measuring the surface tension of the ([EMIM][Ac]) IL mixed with brine at different concentrations. The ([EMIM][Ac]) was able to reduce the SFT of the displacing fluid from 65.4 mN/m to 57.2 mN/m and the CMC point was investigated when the ([EMIM][Ac]) concentration was 1000 ppm in the displacing fluid, which was the minimum reduction on SFT and in a good agreement with conductivities values versus ([EMIM][Ac]) concentrations. A series of flooding experiments on Ottawa sand pack samples were done to demonstrate the effect of ([EMIM][Ac]) ionic liquid at different conditions, (concentration, slug size, and initiation time), on oil recovery. From the flooding results, all showed an increase of the recovery factor. Recovery factor was possible to reach up to 84% of OOIP when 1000 ppm ([EMIM][Ac]) + brine mixture injected into sand pack sample as secondary recovery mode either as a one pore volume the flushed by brine or a continuous ionic solution flooding. Moreover, the recovery factor was higher when IL combined with alkali (AIL) flooded at the same condition and selected mode, secondary mode flushed by brine. The relative permeability curves of continuous brine flooding and 1000 ppm ([EMIM][Ac]) + brine flooding indicated wettability alteration toward a slight increase in rock water wet characteristics. The ([EMIM][Ac]) ionic liquid increases the viscosity of the ([EMIM][Ac]) + brine mixtures, which is one of the mechanisms increasing the recovery factor.

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## Nomenclature

$C_{([EMIM][Ac])}$ ([EMIM][Ac]) concentration
$C_{(Na2CO3)}$ ($Na_2Co_3$) concentration
ppm parts per million
PV pore volume
K absolute permeability
$k_{ro}$ oil relative permeability
$k_{rw}$ water relative permeability
$S_{wi}$ initial water saturation
$S_{oi}$ initial oil saturation

Greek Letters

Ø Porosity
$\rho$ Density
$\mu$ Viscosity