Application of Water-Soluble Polymer/Biopolymer Combined with a Biosurfactant in Oil-Wet Fractured Carbonate Reservoirs

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reduce permeability in the fracture zones.\textsuperscript{21} The results of a study by Shedid\textsuperscript{22} on the effect of water and polymer injection in fractured carbonate reservoirs revealed that the highest oil production was observed when polymer flooding was used rather than water flooding. Zhang et al.\textsuperscript{23} conducted an experiment to determine the effect of gel particles and the HPAM/Cr\textsuperscript{3+} system on plugging in a fractured oil reservoir. Their results indicated that oil recovery could be increased to 18.5\% by using gel particles, and then adding HPAM/Cr\textsuperscript{3+} to the system led to oil recovery up to 29\%. Similar observations have been reported for the plugging of fracture zones to improve oil production in fractured carbonate reservoirs when polymer gel is used.\textsuperscript{24−26}

Biosurfactant flooding is another important bio-EOR technique. Biosurfactants can reduce the interfacial tension (IFT) between the oil and water phases and also change the wettability state of the matrix from oil-wet to water-wet; hence, water can move easily through the rock pores due to reductions in capillary forces.\textsuperscript{27−29} Recently, Cheng et al.\textsuperscript{30} have studied the effect of surfactant flooding in a fractured core, and their results indicated that the movement of the surfactant through the matrix depends strongly on diffusion so that increasing the injection rate led to increased oil recovery. Spinler et al.\textsuperscript{31} carried out an experiment using a surfactant in chalk cores to enhance forced and spontaneous imbibition. Their results indicated that, even with a low concentration of the surfactant, oil recovery can be improved.

It is suggested that flooding with both the surfactant and polymer (SP flooding) can be used as an effective approach in the EOR process to change the wettability of reservoir rocks as well as plugging high-permeability zones.\textsuperscript{32} Sayed Akram and Mamora\textsuperscript{33} conducted a simulation study of polymer-surfactant injection in fractured carbonate reservoirs, and the results showed that such flooding can improve oil recovery. Since various chemicals for EOR are available to increase oil production in fractured carbonate reservoirs, where each has specific advantages and disadvantages, it is challenging to determine the best chemical EOR methods in the most ideal approaches to fulfill all requirements.\textsuperscript{34,35}

In this study, PAM and XG were selected to represent the polymer and biopolymer, respectively, as potential chemicals for EOR and bio-EOR due to their low cost and ability to decrease water mobility and increase its viscosity.\textsuperscript{36} On the other hand, environmentally friendly biosurfactant rhamnolipid was also combined with those chemicals to enhance the water wetness of oil-wet carbonate as well as the interfacial activities of rock/water/oil interfaces.\textsuperscript{37}

Although a significant volume of research has been carried out on the effects of chemical EOR in production from sandstone reservoirs, there have been few publications relating to chemical EOR in fractured carbonate reservoirs. Water flooding alone cannot be performed in fractured carbonate reservoirs due to a large contrast between fracture and matrix permeability as well as the rock matrix being in a strongly oil-wet state. This research proposes a new approach to solve this problem and reap the benefits of water flooding, where the fractured zones are temporarily plugged with the polymer PAM and biopolymer XG to divert the water into the matrix zones. After that, the wettability state of the strongly oil-wet rock matrix is modified toward a water-wet state using biosurfactant RL following the injection of polymeric solutions. Furthermore, a comparison of the performance of polymer PAM and biopolymer XG is conducted.

2. RESULTS AND DISCUSSION

Before starting core flooding, the polymeric solutions were prepared by mixing the polymer with an appropriate cross-linker. Figure 1 illustrates the cross-linking reactions of PAM-PEI and XG-STMP. The PAM-PEI system produces gels through the transamidation mechanism,\textsuperscript{38} in which the amine nitrogen in the PEI structure reacts with the amide group within the structure of PAM to produce PAM-PEI gelation (Figure 1a).
In the cross-linked biopolymer reaction presented in Figure 1b, the structure of STMP is that of a cyclic triphosphate, which makes it difficult for it to react with XG. Therefore, NaOH was added to break down the cyclic triphosphate within the STMP. After that, the hydroxyl group on the XG chains reacts with the STMP to produce PAM-STMP gelation. After preparation of the cross-linked polymer/biopolymer solutions, their values of viscosity against the shear rate were measured and are presented in Figure 2. As can be seen in this figure, both solutions behave similarly at higher shear rates, while the biopolymer is more viscous at lower shear rates. If we translate this behavior into flow regimes near to and far from the wellbore, it could be concluded that both polymeric solutions would behave similarly near the wellbore with higher shear rates. However, far from the wellbore, the biopolymer would retain more of its gelation properties compared to the polymer, and hence, better performance would be expected from the biopolymer. The impact of temperature on the rheological properties of the chemicals used in this study was reported in previous research by Elyasi Gomari et al. It is interesting to note that the viscosities of both polymer and biopolymer decrease with temperature; however, a greater reduction was reported for XG compared to PAM solutions at higher temperature.

2.1. Effect of Chemical EOR and Bio-EOR on the Permeability of the Fractured-Matrix System. Figure 3 shows the effect of the chemical EOR and bio-EOR on the permeability of fractured-matrix systems for PAM (Figure 3a) and XG (Figure 3b). It should be noted that the average water permeability ($K_w$) of the core samples before fracturing was measured between 12 and 15 mD (see Table 7), while after fracturing it increased to the 100–200 mD range, which can be termed the fracture-matrix permeability to water ($K_{fwm}$). The whole injection into fractured plug was performed in four steps. In the first step, 6 pore volumes of distilled water were injected, and no changes were observed in fracture-matrix permeability. This is due to the high contrast between fracture permeability and matrix permeability, and because the sample is oil-wet, all water will pass through the fracture and no change in fracture-matrix permeability was recorded. Therefore, this step is not presented in Figure 3. In the second step, 6 pore volumes of polymeric solutions were injected, and the fracture-matrix permeability to water was decreased due to the plugging of the fracture by the polymeric gels. In the third step, the fracture-matrix permeability to water was increased after injection of 6 pore volumes of the biosurfactant. The reason

![Figure 2. Viscosities of the cross-linked polymer and biopolymer at 20 ± 2 °C.](image)

![Figure 3. Effect of chemical EOR and bio-EOR on the permeability of the fractured-matrix system: (a) polymer; (b) biopolymer.](image)
could be due to two main mechanisms that can happen by injection of a biosurfactant. The first mechanism is that the biosurfactant had displaced some of the polymeric gels, and the fluid moves faster in the porous and fracture system, the inference being that the polymer had temporarily plugged the fracture zones. The second mechanism is that the biosurfactant causes a reduction in IFT and wettability alteration through the system. In this scenario, the resistance of the fluid can reduce, and this resulted in the increase of the fracture-matrix permeability to water. Finally, the injection resumed by final 6 pore volumes of water where no significant change in permeability was recorded.

Overall, the fracture-matrix permeability decreased after the injection of the cross-linked polymer/biopolymer into the system. It can be seen from Figure 3a that the highest reduction in fracture-matrix permeability was observed for core 3. In fact, there was a decrease of 55.133 mD (35.61%) in fracture-matrix permeability from 154.823 to 99.69 mD for this core. Meanwhile, values of fracture-matrix permeability for cores 1 and 2 dropped by 39.045 and 45.973 mD, respectively. Differences in the effect of the polymer and biopolymer in reducing fracture-matrix permeability may be due to their differences in terms of shear thinning and thickening. The received wisdom in the literature is that, in most cases, polymers and biopolymers display shear thinning behavior at different shear rates. An example from the biopolymers is XG, which demonstrates shear-thinning flow behavior in porous media that is independent of the flow rate. Meanwhile, some polymers such as PAM can have effects that are dependent on the flow rate and may exhibit shear-thickening flow behavior. The reason for this could be that the carboxylic groups in the PAM structure release their molecular chain stretch in water. The subsequent increase in the hydrodynamic radius of the polymer molecular chain in aqueous solution thus makes the solution more viscous in porous media.

Similar results were observed when XG solutions were used (see Figure 3b), but they were not as effective as PAM. According to Figure 3b, the highest reduction in fracture-matrix permeability was observed for core 5, which was of 29.339 mD (18.3%), followed by cores 4 and 6 at 24.346 and 14.926 mD, respectively. Differences in the effect of the polymer and biopolymer in reducing fracture-matrix permeability may be due to their differences in terms of shear thinning and thickening. The received wisdom in the literature is that, in most cases, polymers and biopolymers display shear thinning behavior at different shear rates.

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It should be noted that, after the injection of the biosurfactant and the subsequent injection of water, the fracture-matrix permeability was increased. This may be because the injected fluid had displaced some of the polymer gel, the inference being that the polymer had temporarily plugged the fracture zones.

2.2. Effect of Chemical EOR and Bio-EOR on Oil Recovery in Fractured Carbonate Reservoirs. Figure 4 illustrates the effect of the chemical EOR and bio-EOR on oil recovery versus pore volume injected into the fractured reservoirs.
carbonate reservoirs. It should be noted that, in the first step, no oil was produced after flooding with 6 pore volumes of distilled water. This indicates that the flow mostly occurred via fracture. This step is not presented in Figure 4.

The polymer or biopolymer has started right after ineffective water flooding, where, as shown in Figure 4, the oil recovery was increased by chemical EOR and bio-EOR. The highest level of oil production was obtained by the polymer and biopolymer flooding of the system, at up to 12 and 6%, respectively. Moreover, an extra 3–4% of oil recovery was achieved by employing the biosurfactant followed by the water flooding of the systems. Use of the biosurfactant leads to improved oil recovery due to the alteration in wettability and reduced IFT between the water and oil phases in porous media. From Figure 4a, the highest improvement in oil recovery of approximately 16% was obtained in core 3 followed by core 1 at 13.75%, while core 2 exhibited the lowest improvement of 11.53%. However, it is noticeable from Figure 4b that the highest oil production obtained by biopolymer/biosurfactant/water flooding was 8.7%, which was approximately half of that achieved by flooding with the polymer/biosurfactant/water system, as shown in Figure 4a. This may be due to the fact that the highest reduction in fracture-matrix permeability was observed when using polymer flooding as opposed to the biopolymer system (see Figure 3). Therefore, it can be concluded that there is a link between fracture-matrix permeability and oil recovery. In fact, as fracture-matrix permeability declines, oil recovery increases, which means that the polymer can plug the fracture and then the injected fluid penetrates into the rock matrix and drives the trapped oil out of the core. Al-Hattali et al. studied the effect of microbial biomass in fractured carbonate reservoirs, and their results revealed that, by using microbial biomass, oil recovery can be increased to 27–30% due to the plugging of fractures. The influence of microbial and water flooding in fractured carbonate rocks was examined by Zekri and El-Mehaideb. Their results indicated that microbial flooding was capable of improving oil recovery in fractured carbonate rocks and altering the performance of the system by plugging a part of the fracturing. A study by Shedid on the effect of the fracture angle on oil recovery by polymer flooding concluded that polymer injection in a fractured reservoir is strongly
recommen... 16% of oil recovery was observed for polymer flooding, followed by biosurfactant flooding, and waterflooding, respectively. As can be seen in the figure, the first pressure buildup was conducted for polymer PAM/biopolymer XG flooding, where the highest differential pressure and oil recovery were observed at 6 pore volumes (DP1). In this step, the differential pressure was stabilized at 9.4 and 5.6 psi for polymer PAM (core 1) and biopolymer XG (core 4), respectively. After that, biosurfactant RL was injected into the system, and the results showed a lower differential pressure in DP2 compared to DP1. It should be noted that, in each step, the pump was started from zero as the solution needs to be changed. Finally, waterflooding was conducted to achieve the ultimate oil recovery. The values of final oil recovery were observed to be 16 and 8.7% for cores 1 and 4, with corresponding stabilized differential pressures at 4.3 and 3.1 psi, respectively. The change in recorded differential pressure clearly indicates the positive impact of polymer/biopolymer-biosurfactant on waterflooding in carbonate fractured reservoirs.

2.4. Contact Angle Measurement as an Indicator of Alteration in Wettability. Tables 1 and 2 show the contact angle measurements of aged core slices before contact with chemical EOR or bio-EOR. Cores 3 and 6 were used to analyze the effect of chemical EOR on changes in wettability. As can be seen in the tables, the average contact angles measured on aged cores were 128.7° for core 3 (Table 1) and 122.6° for core 6 (Table 2), indicating oil-wet systems.

The results for the effect of chemical EOR and bio-EOR on the wettability of oil-wet systems are presented in Tables 3 and 4. The use of chemical EOR, and especially the addition of the biosurfactant across the core sample, reduced the contact angle from 128.7 to 94.8° in core 3 (Table 3), altering the wettability of the rock matrix to a neutral-wet state. The same result was observed to a lesser extent for core 6 (Table 4), where the
The contact angle dropped from 122.6 (oil-wet) to 97.9° (neutral-wet). Based on the SEM images presented in Figures 9d and 10d, some dense deposits of biosurfactant layers have built up at the surface of the rock, and this is responsible for the change in wettability of the oil-wet rock. Several researchers have carried out experiments to alter the wettability of carbonate rock.

Figure 9. SEM images: (a) XG-STMP; (b) surface of a rock slice without any chemical EOR; (c) surface of a rock slice with XG-STMP; (d) surface of a rock slice with XG-STMP + biosurfactant.

Figure 10. SEM images: (a) PAM-PEI; (b) surface of a rock slice without any chemical EOR; (c) surface of a rock slice with PAM-PEI; (d) surface of a rock slice with PAM-PEI + biosurfactant.
rocks using surfactants, and they have concluded that biosurfactants such as rhamnolipids are capable of effecting this change. Some biosurfactants have a stronger effect on wettability than others because of their different hydrophilic–hydrophobic balance (HLB). For instance, the HLB of rhamnolipid has been reported to be 9.5 (low HLB), which can alter wettability to a neutral-wet system. However, surfactants with an HLB of 21.27 (representing high HLB) can change the wettability of an oil-wet system toward the water-wet state. Modifications of the wettability of the rock

| droplet       | reading 1 (°) | reading 2 (°) | reading 3 (°) | average contact angle (°) | wettability     |
|---------------|---------------|---------------|---------------|---------------------------|-----------------|
| DW droplet 1  | 128.3         | 129.1         | 128.1         | 128.5                     | oil-wet         |
| DW droplet 2  | 126.4         | 126.1         | 126.8         | 126.4                     | oil-wet         |
| DW droplet 3  | 131.1         | 130.8         | 131.3         | 131.1                     | oil-wet         |

average contact angle for core 3 (°) 128.7 oil-wet

| droplet       | reading 1 (°) | reading 2 (°) | reading 3 (°) | average contact angle (°) | wettability     |
|---------------|---------------|---------------|---------------|---------------------------|-----------------|
| DW droplet 1  | 95.0          | 94.6          | 95.1          | 94.9                      | neutral-wet     |
| DW droplet 2  | 99.1          | 98.3          | 98.4          | 98.6                      | neutral-wet     |
| DW droplet 3  | 91.3          | 90.6          | 90.6          | 90.8                      | neutral-wet     |

average contact angle for core 3 (°) 94.8 neutral-wet

| droplet       | reading 1 (°) | reading 2 (°) | reading 3 (°) | average contact angle (°) | wettability     |
|---------------|---------------|---------------|---------------|---------------------------|-----------------|
| DW droplet 1  | 98.7          | 98.5          | 97.9          | 98.4                      | neutral-wet     |
| DW droplet 2  | 99.2          | 98.8          | 99.0          | 99.0                      | neutral-wet     |
| DW droplet 3  | 95.8          | 96.5          | 96.9          | 96.4                      | neutral-wet     |

average contact angle for core 6 (°) 97.9 neutral-wet

![Figure 11. Oil recovery and differential pressure for cores 1 (a) and 4 (b).](image-url)
matrix may be due to interactions between the carbon components attached to the carbonate surface and the hydrophobic heads of the surfactant.61–65

### 3. CONCLUSIONS

This study has presented an analysis of the effect of polymer, biopolymer, and biosurfactant elements of chemical EOR and bio-EOR on oil recovery performance in fractured carbonate reservoirs. The results show that chemical EOR and bio-EOR flooding can be considered as potentially effective approaches to the improvement of sweep efficiency in fractured carbonate reservoirs. The results indicate that the proposed technique can improve oil recovery, with the highest rates up to 16% being observed using polymer/biosurfactant/water flooding. SEM images show that the polymer and biopolymer were adsorbed physically onto the surface, whereas after biosurfactant flooding no polymer/biopolymer remained on the surface. The images prove that the gels were temporarily plugging the fracture zones, hence reducing fracture-matrix permeability by 18.3–35.61% in porous media and diverting the biosurfactant slug toward matrix zones. The addition of the biosurfactant to the system has been identified as modifying the wettability of the rock matrix from oil-wet to neutral-wet, hence easing the oil flow toward fractures.

### 4. MATERIALS AND METHODS

#### 4.1. Materials. In this study, two types of polymeric solutions, namely, xanthan gum (XG) and polyacrylamide (PAM), were used. Trisodium trimetaphosphate (STMP) and polyethylenimine (PEI) were utilized as cross-linking agents for the biopolymer and polymer, respectively. Water-soluble rhamnolipid was selected as a biosurfactant to study its application in EOR. Stearic acid (0.01 M) dissolved in n-decane was used to represent a model oil resembling crude oil. Table 5 gives information concerning the materials used.

#### 4.2. Methods. 4.2.1. Preparation of Solutions. 4.2.1.1. Cross-Linked Polymer. 10000 ppm PAM and 5000 ppm PEI were mixed with distilled water for 2 h at a speed of 1000 rpm.

4.2.1.2. Cross-Linked Biopolymer. 3000 ppm XG was added to a 0.1 M solution of sodium hydroxide and then mixed with 3000 ppm STMP and distilled water for 1 h.
4.2.1.3. Biosurfactant. 500 mg/L rhamnolipid was used. This concentration was considered to be a suitable CMC measurement for applications in EOR by Li et al. 66

4.2.2. Viscosity Measurement. The viscosities of cross-linked polymer and biopolymer under different shear rates in the range of 5.109 to 1021.8 s⁻¹ were measured using a Fann model 35 viscometer.

4.2.3. Core Sample Preparation. Six carbonate cores (Austin Chalk) were washed with toluene for 48 h and then dried in a vacuum desiccator at 70 °C for 24 h. The specifications of the core samples are given in Table 6. After that, a vacuum saturator was used for 48 h to saturate the cores with distilled water to remove air between the grains. Then, the wet weights of the cores were measured to establish the porosities and pore volumes of the samples.

4.2.3.1. Core Flooding Apparatus. Figure 12 illustrates a diagram of the core flooding device. Brine and oil accumulators are attached to an injection pump, which can be set at different flow rates. Inlet and outlet pressures across the core sample are joined at both sides of the core holder. The core sample is held within the core holder, and then overburden pressure can be applied by confining pressure through the cores. Inlet and outlet end plugs allow fluids to be flooded through the core sample.

4.2.3.2. Core Flooding Experiment before Aging of Core Samples. The unfractured water-wet core was inserted in the core holder, and then distilled water was injected at flow rates of 1, 1.5, and 2 cc/min to establish the permeability to water ($K_w$). After that, the core was flooded with sample oil to obtain a value of initial water saturation ($S_{wi}$). At this stage, the oil was injected into the core sample until no distilled water was produced. Then, the permeability to oil ($K_o$) was determined at flow rates of 0.5, 0.75, and 1 cc/min. Finally, distilled water was injected to establish residual oil saturation ($S_{or}$). The results are presented in Table 7.

4.2.3.3. Core Flooding Experiment after Aging of Core Samples. After the evaluation of $K_w$, $S_{wi}$, $K_o$, and $S_{or}$ as described above, the core was cut horizontally as a fractured core at an angle of 180°. Images of an unfractured core and a core fractured at an angle of 180° are shown in Figure 13. The core sample was then placed in a cylindrical box that was dried in a vacuum desiccator at 70°C for 24 h. The required experiments and to gather SEM images of the rock surface for characterization with and without chemical EOR, a Hitachi S-3400 N SEM was used. This device was operated with a BSE detector and an accelerating voltage of 15 kV to achieve high-resolution imaging.

Table 7. Petrophysical Properties of Core Samples before Fracturing

| core sample | $K_w$ | $K_o$ | $S_{wi}$ | $S_{or}$ |
|-------------|-------|-------|----------|----------|
| 1           | 13.032| 2.031 | 29.33    | 38.72    |
| 2           | 14.255| 2.372 | 24.93    | 38.50    |
| 3           | 15.308| 2.897 | 39.50    | 31.14    |
| 4           | 13.826| 4.692 | 17.98    | 44.47    |
| 5           | 13.146| 3.329 | 45.80    | 31.27    |
| 6           | 12.430| 4.901 | 34.82    | 35.71    |

Figure 13. Images of an unfractured core and a core fractured at an angle of 180°.

4.2.4. Contact Angle Measurements. Contact angle measurements were conducted using a Kruss DSA 100 goniometer analyzer at room temperature (20 ± 2 °C) at two time points. The first point followed the 30 day aging of the core sample, and the second was immediately after the final bio-EOR procedure when the core slices had been further aged in the core holder for 48 h after the injection of the biosurfactant and distilled water flooding. These steps were repeated three times for each core sample, and then the average was given as the final contact angle measurement.

4.2.5. Scanning Electron Microscopy (SEM). To carry out the required experiments and to gather SEM images of the rock surface for characterization with and without chemical EOR, a Hitachi S-3400 N SEM was used. This device was operated with a BSE detector and an accelerating voltage of 15 kV to achieve high-resolution imaging.

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Notes

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