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Chapter 9

Supramolecular Polymer-Surfactant System for Heavy Oil Recovery

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Abstract

Water soluble polymers are widely used as mobility control agents for enhanced oil recovery (EOR). Yet, in harsh reservoir environments (i.e., elevated temperatures and high ionic strength), the applicability of conventional polymers is limited. This issue has been somewhat resolved through the chemical synthesis of polymers having functional moieties such as sulfonic acid groups and/or n-vinylpyrrolidone. Another approach to circumvent expensive chemical syntheses, it is the formulation of supramolecular polymers built via non-covalent and β-cyclodextrin (β-CD) host-guest interactions. In this study, an advanced polymer-surfactant (SAP-AP1) system formulated via the self-assembling of an associative polymer with an anionic surfactant and β-CD was evaluated as a mobility control agent to displace and recover heavy oil (i.e., 2560 cP at 25°C). Displacement tests employing unconsolidated sand-pack systems were carried out at simulated heavy oil reservoir conditions. The experimental results demonstrate that the SAP-AP1 produces a stable viscous displacement front that results in more efficient volumetric sweep, faster reduction of the water/oil ratio (WOR), and incremental oil recovery (e.g., 19% higher incremental oil recovery relative to the baseline polymer). The SAP-AP1 system shows potential for EOR applications at economically favorable conditions.

Keywords: β-CD host-guest complexations, noncovalent bonding, associating polymers, supramolecular systems, enhanced oil recovery, mobility control, resistance factor, residual resistance factor, water-oil ratio

1. Introduction

Worldwide, polymer flooding is extensively applied as a mobility control agent to increase the sweep efficiency of the displacing fluid during enhanced oil recovery (EOR). As stated by

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Perttamo, "[Compared] to conventional waterfloods on a timescale, polymer floods will accelerate the recovery process due to rapid viscosity build-up…. [that] will contribute to a faster and higher oil production. An incremental recovery factor of 5% [of the] original oil in place (OOIP) or more is regarded as a successful polymer application" [1]. Polymer flooding has been historically applied in light and medium gravity oil reservoirs. More recently, it has also been applied successfully in heavy oil reservoirs with oil viscosities ≥1200 cP, which expands the practical applicability of this EOR technique [2–11].

Incremental oil recovery by polymer flooding is induced by the following mechanisms: reduction of the water-oil mobility ratio by means of the increased viscosity of the displacing phase (i.e., injected water) and reduction of viscous fingering, decrease of the water relative permeability due to polymer retention within the formation rock, diversion of the injected water to unswept reservoir regions, improvement of the water-injection profile (i.e., preventing crossflow between vertical heterogeneous layers), and the increase of oil fractional flow [4, 5, 12–21].

Field polymer-flood projects carry high chemical operating expenditures [5, 22]. Therefore, it is vital to carefully select the appropriate polymer chemistry for the specific reservoir characteristics. For instance, the practical applicability of polymer flooding is limited to reservoirs with moderate temperatures (i.e., <90°C) and formation brines containing low concentrations of divalent cations (e.g., Ca^{2+}, Mg^{2+}) to avoid the chemical degradation of the polymer guaranteeing the technical success of the process in a cost-effective fashion [12–17, 19, 23].

Divalent cations (i.e., Ca^{2+}, Mg^{2+}) significantly affect the viscosity of polymer solutions. The bridging effect of divalent cations with the negatively charged moieties (i.e., carboxyl groups) causes viscosity loss due to polymer coiling [12, 13, 15, 16, 23, 24]. Furthermore, the reaction of the carboxyl group with divalent cations causes polymer precipitation and phase separation [14]. Therefore, to compensate for the loss of viscosity, it is necessary to add higher polymer concentrations to the brine solution [12]. Alternatively, the reservoir could be conditioned before polymer flooding by injecting low-salinity water to prevent the mixing of the polymer slug with the high-salinity reservoir brine [16].

Several approaches have been taken to improve the chemistry of polymers to ensure their stability and functionality at elevated temperatures and in reservoir brine containing high salinity and hardness concentration. These polymeric systems have been customized by incorporating specific functional moieties that are covalently grafted onto the polymer structure. The attachment of sulfonic acid groups like allyl sulfonic acid, 2-acrylamido-2-methylpropane sulfonate (AMPS) and/or n-vinylpyrrolidone (n-VP) monomers increases the polymer resistance to hydrolysis and tolerance to high salinity and hardness. Shear stability and viscosifying power of polymers have been advanced by the introduction of hydrophobic groups like n-alkyl (i.e., $\geq C_6$ carbon numbers) acrylamide, styrene, ring structures, large and rigid side groups such as styrene sulfonic acid, n-alkyl maleimide, acrylamide-base long-chain alkyl acid, and 3-acrylamide-3-methyl butyric acid, among others [3, 13, 14, 17, 23, 24].

EOR polymers are shear sensitive, which is a downside for EOR applications. According to Zaitoun et al. [23] and Sheng et al. [17], shearing occurs within several devices during the different phases of polymer handling and injection process in the high flow rate region close to the

injection well such as in shearing devices during polymer dissolution, during recirculation of the polymer solution through centrifugal pumps, polymer flow through chokes and downhole valves under high differential pressure, and during the flow of polymers at high flow rates through the perforations of the reservoir rock and sand face [25]. The shear degradation of the polymer structure consists of the breakage of the macromolecule chain reducing its molecular weight, size, and viscosifying power. Thus, shear degradation is irreversible [17, 23, 26].

The shear degradability of EOR polymers is directly related to the polymer molecular structure, molecular weight, and chain flexibility. The physics of polymer mechanical degradation is reported in [26]. As indicated by Jouenne et al., "flexible polymer chains have the ability to be extended under elongational flow fields [and the] … stretching of the polymer chains can lead to chain rupture" [27]. For example, xanthan gum, which is a rigid rod-like biopolymer with a double-strand helical structure that aligns in the direction of the flow [26], displays a very high shear resistance because it does not stretch under shearing/elongations forces, which reduces the friction forces on the carbon/carbon backbone. On the contrary, the linear polyacrylamide homopolymer is highly flexible and therefore very sensitive to shear degradation. The shear stability of polyacrylamide is commonly improved by introducing negative acrylate groups to the backbone, since it provides rigidity by means of electrostatic repulsion. Nevertheless, the presence of electrolytes (e.g., Na^+, Ca^{2+}, Mg^{2+}) shelters the negative charges of the acrylate groups inducing the coiling and folding of the polymer chain, which becomes less rigid and more flexible [17, 23]. Then, the stretching of the coiled (i.e., coiled-stretch transition) polymer chain under the influence of shear and elongational forces makes it vulnerable to chain breakage and irreversible shear degradation. Thus, the shear sensitivity of EOR polymers increases with brine salinity [23].

The shear stability of acrylamide copolymers can also be increased by introducing the polymer chain large functional hydrophobic groups such as the acrylamide tert-butyl sulfonate (ATBS) and the n-vinylpyrrolidone (n-VP) as they impart rigidity to the polymer structure [23]. The attachment of hydrophobic groups to the macromolecular backbone of EOR polymers to improve the shear and thermal stability, as well as the tolerance to brines with high salinity and hardness concentration, has been widely recognized. The main benefit of the incorporation of hydrophobic groups is as explained by Perttamo [1]: "the reorientation of the macromolecules due to polar and non-polar, results in [the] formation of hydrophobic associations between de incorporated hydrophobic groups," generating intramolecular and intermolecular associations forming supramolecular aggregates. These polymers are called associating hydrophobic polymers or hydrophobically modified polymers or for short associating polymers [1, 17, 19, 28, 29]. Under shear, these supramolecular aggregates can disassemble due to the reversible disruption of the hydrophobic bonds; therefore, at high shear rates, these systems show a remarkable shear-thinning behavior. As indicated by Dupuis et al. [19], these systems offer several benefits for field applications: "…reduced polymer concentration to achieve a required mobility ratio, extend the range of suitable reservoirs in terms of salinity, and facilitate the mixing, pumping, and injection procedures."

In this chapter, we evaluated the effectiveness of a supramolecular polymer-surfactant (SA-AP1) system as a mobility control agent for displacing heavy oil in high salinity and

hardness concentration. The SAP-AP1 system was formulated via self-assembling driven by β-CD host-guest complexation, divalent cation bridges (i.e., Ca^{2+} or Mg^{2+}), hydrophobic interactions, and hydrogen bonding, among others. The SAP-AP1 system contains 0.75 wt% of an associating polymer (AP1), 0.007 wt% (70 ppm) of an anionic surfactant, and 0.007 wt% (70 ppm) of β-CD prepared in saline solution. Detailed information on the formulation and properties of the SAP-AP1 system is provided in the preceding chapter of this book.

In this chapter, we begin by describing the sand-pack core-flooding displacement test and the properties of the unconsolidated porous media and fluids (i.e., heavy oil and brine) employed. Next, we discuss the viscosifying power of the baseline AP1 polymer and the SAP-AP1 system during flow through porous media by means of the resistance factor (RF). Polymer retention in porous media is also analyzed through the residual resistance factor (RRF). The effectiveness of both polymer AP1 and the SAP-AP1 system in recovering heavy oil is analyzed next. Finally, we discuss the effect of AP1 and SAP-AP1 on the water to oil production ratio (WOR).

2. Sand-pack flooding displacement tests

The performance of the polymer AP1 and the SAP-AP1 system as a mobility control agent for heavy oil recovery was determined through routine oil sand-pack displacement tests at simulated reservoir conditions. The heavy oil used in these flooding tests was provided by Husky Energy Inc. (Calgary, AB, Canada) with a viscosity of 68,728 cP at 25°C that was adjusted to a viscosity of 2560 cP at 25°C by dilution with natural condensate produced from the McCully field, Corridor Resources Inc. (Sussex, NB, Canada). The density of the diluted crude oil was 0.954 g/ml at 25°C, the API corrected to 60°F was 15.27, and the interfacial tension (IFT) between the crude oil and the SAP-AP1 system was 0.032 dynes/cm at 25°C. The IFT was determined using a M6500 Spinning Drop Tensiometer manufactured by Grace Instrument (Houston, TX, USA). QUIKRETE® Premium Play Sand® (No. 1113), which is 100% quartz [30], was employed to prepare the unconsolidated sand packs. The sand-grain size distribution was determined by sieve analysis following the procedure described in [31], which conforms to ASTM C136/C136M-14. The sieve analysis indicated that the effective size of the sand, D10, and the uniformity coefficients, D60/D10, were 240 and 2.02 μm, respectively.

A total of four displacement tests were conducted at a temperature of 25°C using a brine concentration of 8.4 wt%. The synthetic brine composition was 6.9 wt% of NaCl, 0.18 wt% of $MgCl_2$, 1.3 wt% of $CaCl_2$, and 0.04 wt% of Na_2SO_4. Two displacement tests were conducted using the baseline polymer AP1 at the optimum concentration of 0.75 wt% (control tests) and two displacement tests were carried out using the optimum SAP-AP1 system which also contained a polymer concentration of 0.75 wt%. Displacement tests were carried out using a DCHH series core holder (pressure-tapped, biaxial-type loading) manufactured by Temco, Inc. (Tulsa, OK, USA). Two CFR series transfer vessels (Temco, Inc., Tulsa, OK, USA) were employed to displace brine, polymer, and crude oil through the sand pack. A Teledyne ISCO Syringe pump, model 100DX manufactured by Teledyne Isco, Inc. (Lincoln, NE, USA), was used to pump the fluids through the transfer vessels. Several PGT-30 series/stainless steel pressure gauges manufactured by Omega (Laval, Quebec, Canada) with an accuracy of 0.5% as a percent of full scale

(FS) were installed at the inlet of the core holder (P1), at the inlet cap to monitor the overburden pressure (P_{OP}), and two pressure gauges along the core holder (P2 and P3). **Figure 1** shows a simplified schematic of the experimental set-up used during the sand-pack displacement tests.

The sand-pack properties, such as pore volume (PV), porosity (φ), and permeability to brine (k) were determined following routine procedures as outlined in [32, 33]. **Table 1** presents the sand-pack properties for each of the displacement tests.

Heavy oil sand-pack displacement tests were carried out following a fluid injection scheme of four stages: heavy oil injection, waterflooding, polymer flooding, and post-polymer waterflooding. All the fluids were injected at a flow rate of 0.98 cm^3/min, which is equivalent to a flow velocity of 0.0116 m/s (0.91 ft./day). During the heavy oil injection stage, oil was continuously injected until the production of brine stopped, which corresponds to a volume of oil equivalent to three pore volumes (3 PV).

Afterward, the oil-saturated sand packs were waterflooding to displace oil by injecting brine (8.4 wt%) at a flow rate of 0.98 cm^3/min until no more oil was produced, which corresponds to a volume of brine equivalent to 6 pore volumes (6 PV). Right after the waterflooding stage was completed, 1 pore volume of AP1 or SAP-AP1 polymer solution was injected at a flow rate of 0.98 cm^3/min to displace the unrecovered or "remaining" oil that was bypassed during the previous waterflooding stage [34]. Finally, a post-polymer waterflooding step was immediately initiated at the end of the polymer flooding stage. A total volume of 6.5 pore volumes of brine (8.4 wt%) was injected at a flow rate of 0.98 cm^3/min. In each of the injection stages, the injection time, pressure readings, and the corresponding volumes of the fluids (brine and oil) produced

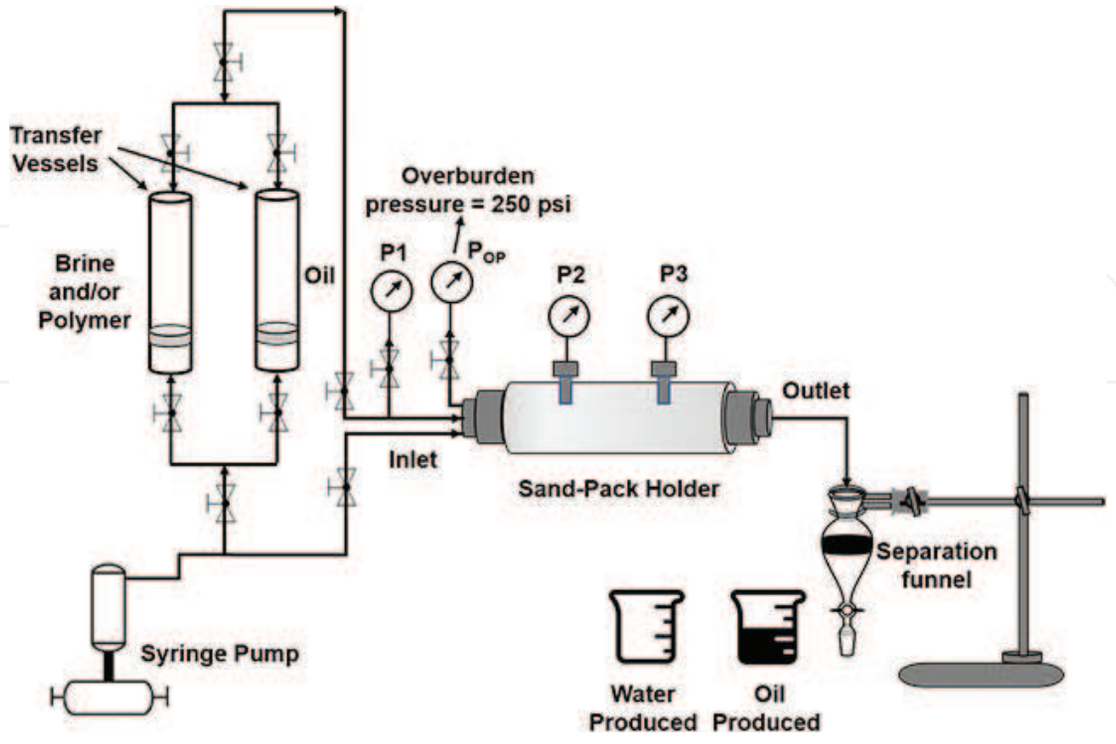


Figure 1. Experimental set-up for sand-pack displacement tests.

Displacement test #	Pore volume [cm^3]	Porosity [%]	Permeability [mD]
Baseline # 1.1	173	24	3085
SAP AP1 # 1.2	177	25	2812
Baseline # 2.1	169	24	2746
SAP AP1 # 2.2	185	26	1758

Table 1. Sand-pack properties.

were monitored. Material balance was applied to determine oil and water saturations, as well as the percentage of oil recovered from each injection stage. More details of the experimental procedure employed during the sand-pack displacement tests are provided in [35].

The results obtained from the displacement tests were analyzed by plotting the resistance factor (RF), the residual resistance factor (RRF), percentage of cumulative oil recovery, ratio of remaining oil saturation over initial oil saturation (S_{ro}/S_{oi}), and water oil ratio (WOR) as a function of volume of fluid injected. The volume of fluid injected was expressed as a fraction of pore volume normalized by porosity and permeability using the capillary bundle parameter [36–39] to compare the displacement tests at the same porosity and permeability reference.

3. Resistance factor and residual resistance factor

Resistance factor (RF) provides information on the effective viscosity of the polymer solution during flow in porous media relative to water before polymer flooding [38]; therefore, RF indicates the effectiveness of the polymer system as a mobility control agent during enhanced oil recovery (EOR) [6, 19, 29, 40, 41]. While, the residual resistance factor (RRF) measures the increased pressure drop across the porous media due to polymer retention (mechanical entrapment and polymer adsorption) [5, 6, 19, 29, 37–44].

Figure 2(a) and **(b)** plots RF as a function of volume of fluid injected expressed as a fraction of pore volume (PV) normalized for permeability and porosity for tests—Baseline # 1.1 and SAP-AP1 # 1.2 and Baseline # 2.1 and SAP-AP1 # 2.2 respectively.

Figure 2 demonstrates that the RF curves of the AP1 polymer and the SAP-AP1 network have similar flow behavior. After the injection of 0.013 PV, the RF values increase continuously with increasing volume of fluid injected until a maximum RF value was reached at about 0.02 PV. Then, the RF values decreased with increased throughput in the sand packs until the RF values leveled off. The RF value plateaued out at an average value of 5.6 for the SAP-AP1 system, while for the baseline AP1 polymer, the RF value plateaued out at an average value of 2.0. Therefore, the SAP-AP1 system offered on average 3.6 times higher effective viscosity within the porous media relative to the effective viscosity achieved by polymer AP1.

The RF-curves eventually reached plateau values for both systems: the SAP-AP1 and the baseline. This may occur due to the dynamic disassembling and reassembling of the non-covalent

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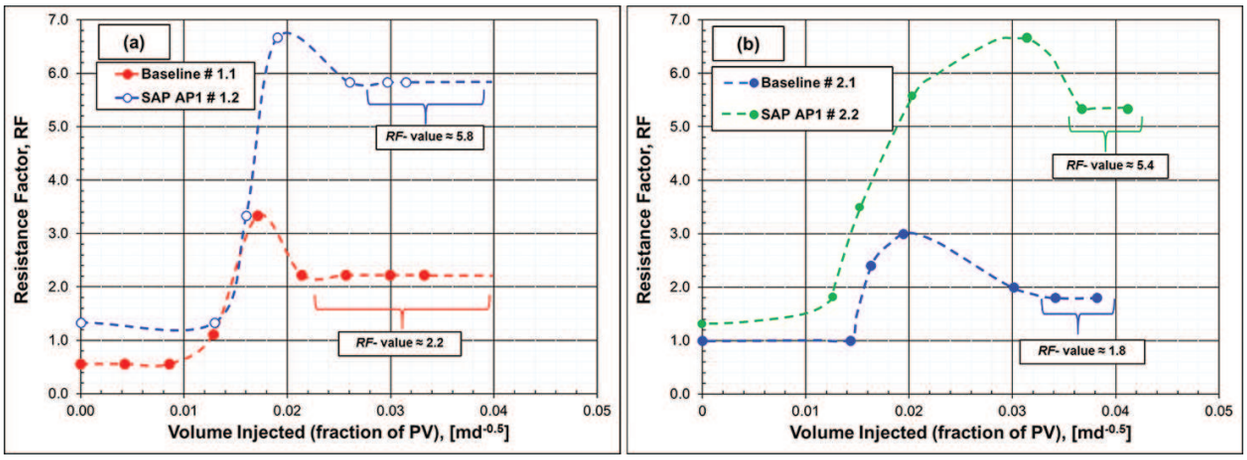


Figure 2. RF versus volume of fluid injected: (a) Baseline # 1.1 and SAP-AP1 # 1.2 and (b) Baseline # 2.1 and SAP-AP1 # 2.2.

intra- and intermolecular interactions (i.e., decomplexation and complexation of host-guest interactions, breaking and reforming of hydrophobic interactions, and hydrogen bonds, among others) under the influence of the shear forces imposed during the flow process. Eventually, equilibrium of the shear forces and the flow resistance forces of the network structures are reached, and the RF curves leveled off. The stabilization of the RF curves also suggests that propagation of the SAP-AP1 systems and the baseline polymers took place along the sand-pack systems. These observations agree with previous research on flow behavior of associating polymers through porous media [28, 38, 44]. Overall, the optimum SAP-AP1 formulation consistently provides higher resistance factors and consequently a better mobility control than the baseline polymer AP1.

The performance of both polymers AP1 and SAP-AP1 in terms of the residual resistance factor, RRF, is presented in **Figure 3(a)** and **3(b)**, which plots RRF values as a function of volume of brine injected for tests Baseline # 1.1 and SAP-AP1 # 1.2 and Baseline # 2.1 and SAP-AP1 # 2.2, respectively.

Figure 3 indicates that the RRF curves for both systems decrease continuously as the volume of brine injected increases that eventually stabilize. The average end RRF value for the

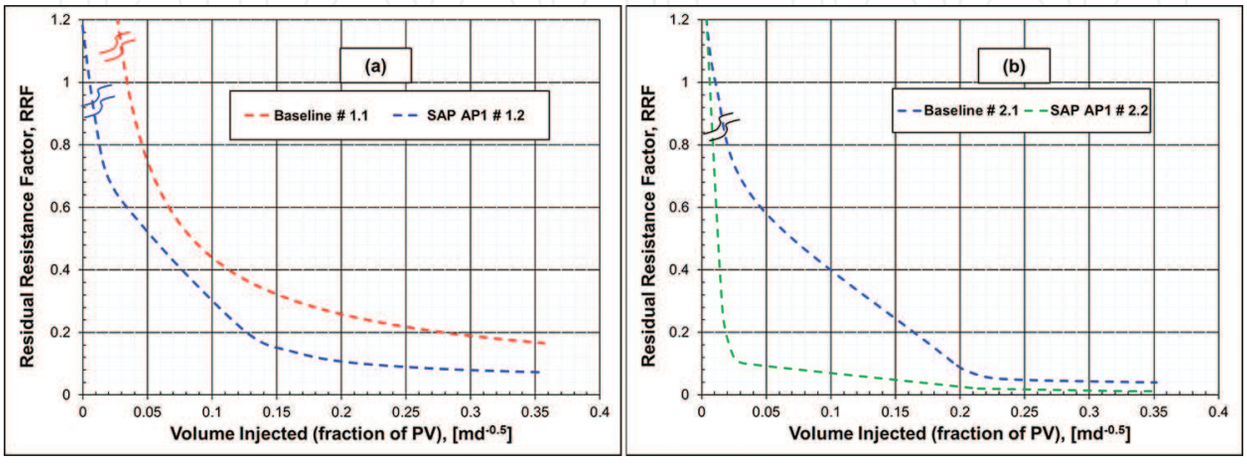


Figure 3. RRF versus volume of fluid injected: (a) Baseline # 1.1 and SAP-AP1 # 1.2 and (b) Baseline # 2.1 and SAP-AP1 # 2.2.

AP1 was 0.5, while the average end RRF value for the SAP-AP1 system was 0.02, suggesting insignificant pore plugging and/or permeability reduction due to polymer retention, which is expected in unconsolidated and/or high permeability porous media [37, 42].

4. Heavy oil recovery

The performance of the SAP-AP1 system and baseline AP1 polymer as mobility control agents for the displacement and recovery of heavy oil is displayed in **Figure 4**, which plots the percent of cumulative oil recovery as a function of volume of fluid injected and flooding stage.

Figure 4 reveals that the average oil recovery during the waterflooding stage was about 30% for all the displacement tests. The combined average cumulative oil recovery produced by polymer flooding and post-polymer waterflooding for the Baseline tests # 1.1 and # 2.1 was 37%, respectively, after subtracting the average oil recovery attributed to the initial waterflooding stage. Whereas, the combined average cumulative oil recovery produced by flooding and post-polymer waterflooding for the SAP-AP1 tests # 1.1 and # 2.1 was 56%, respectively, after subtracting the average oil recovery attributed to the initial waterflooding stage. These experimental observations demonstrate that the SAP-AP1 system produced an additional incremental oil recovery of 19% relative to the baseline AP1 polymer. In this analysis, average values of cumulative oil recovery were used as an alternative to the individual results from each of the displacement tests to provide a conservative assessment of the experimental results in terms of heavy oil recovery. This approach was necessary to avoid the overestimation of oil recovery from polymer flooding and the post-polymer waterflooding stage, because the oil and water separation process after polymer flooding was found to be a difficult and lengthy process, even though several experimental steps were carried out to achieve the most effective separation of water from the produced oil.

The ratio of remaining to initial oil saturation as a function of volume of fluid injected and injection step is presented in **Figure 5** for the baseline polymer and SAP-AP1 system.

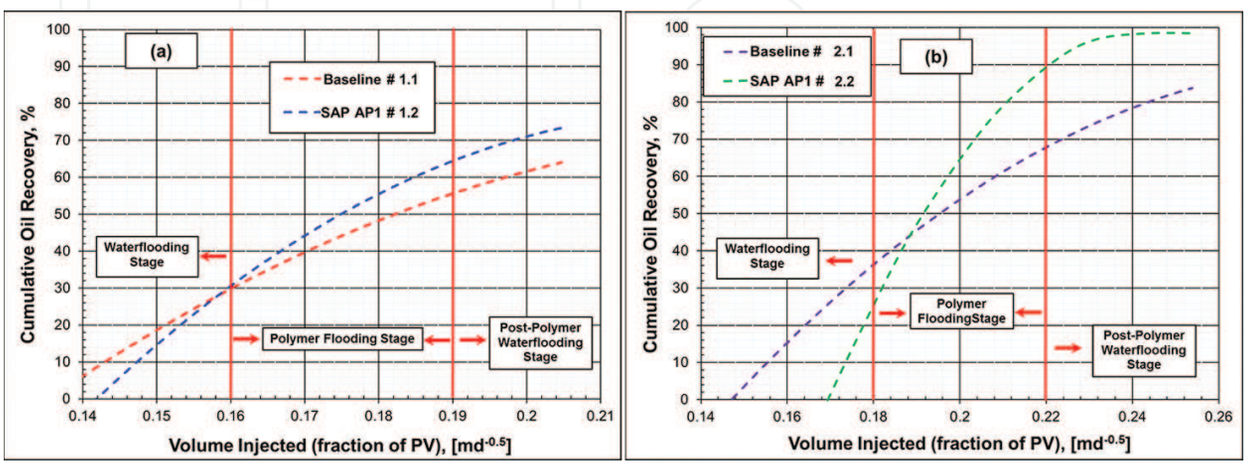


Figure 4. Percentage of cumulative oil recovery versus volume of fluid injected and flooding stage: (a) Baseline # 1.1 and SAP-AP1 # 1.2 and (b) Baseline # 2.1 and SAP-AP1 # 2.2.

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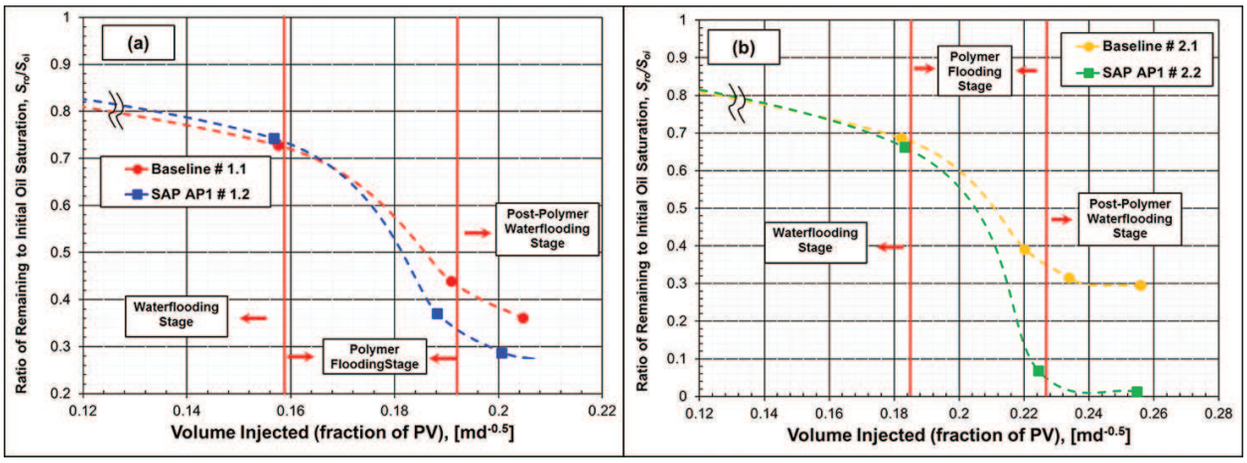


Figure 5. S_{ro}/S_{oi} versus volume of fluid injected and flooding stage: (a) Baseline # 1.1 and SAP-AP1 # 1.2 and (b) Baseline # 2.1 and SAP-AP1 # 2.2.

Figure 5 clearly shows the effect of each of the flooding stages on oil recovery. The polymer flooding stage produced a dramatic decrease of the S_{ro}/S_{oi} ratio that rapidly stabilized during the post-polymer waterflooding stage. The average S_{ro}/S_{oi} ratio obtained from the Baseline tests # 1.1 and # 2.1 was 0.33, while the average S_{ro}/S_{oi} ratio attained from the SAP-AP1 tests # 1.2 and # 2.2 was 0.14.

These experimental results demonstrate that the optimum SAP-AP1 system provided a more efficient mobility control compared to the baseline AP1, which resulted in a more stable viscous displacement and accelerated heavy oil recovery. Furthermore, the low concentration of anionic surfactant contained in the SAP-AP1 formulation reduces the interfacial tension (IFT) of the oil-brine system from 30 [45] to 0.032 dynes/cm. This remarkable reduction in IFT decreases capillary forces, which facilitates the detachment and mobilization of oil during SAP-AP1 flooding [46, 47]. Therefore, the SAP-AP1 system produces incremental oil recovery by the synergistic effect of greater mobility control functionality and by decreasing the IFT of the oil-brine system.

5. Water-to-oil ratio

During waterflooding of heavy oil, "the adverse mobility ratio between the viscous oil and the water induces high-water-cut production and poor sweep efficiency" [2]. Polymer flooding decreases the mobility of the injected water (i.e., augmented water viscosity) reducing the water-cut production levels. **Figure 6** presents the water/oil ratio (WOR) as a function of volume of fluid injected and the flooding stage for the baseline polymer and the SAP-AP1 system tests.

Figure 6 indicates that the average WOR at the end of the initial waterflooding stage for the displacement tests was about 10. As soon as the polymer flooding stage (i.e., baseline polymer AP1 and/or SAP-AP1 system) was initiated, WOR continuously decreased as the volume of polymer injection increased, reaching a minimum WOR value at the end of the polymer flooding stage. The WOR curves in **Figure 6** show that the SAP-AP1 system was more efficient in reducing and controlling the water-oil ratio by providing a faster response and lower average

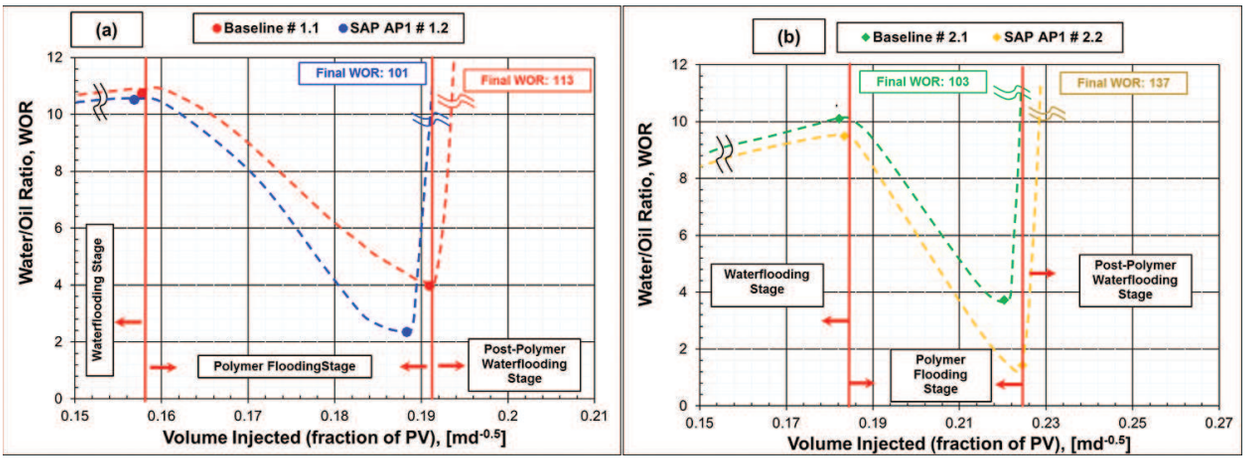


Figure 6. WOR versus volume of fluid injected and flooding stage: (a) Baseline # 1.1 and SAP-AP1 # 1.2 and (b) Baseline # 2.1 and SAP-AP1 # 2.2.

WOR value of 1.9. Whereas, the baseline AP1 polymer rendered a minimum average WOR value of 3.9 at the end of the polymer flooding stage. The WOR curves also show that immediately after the post-polymer waterflooding stage was initiated, an abrupt increase of the water to oil ratio took place that continued until the end of the post-polymer waterflooding stage. As explained by Seright "once brine injection [post-polymer flooding] begins, viscous fingering and [porous media] heterogeneities will quickly lead to severe channeling" [of the water to the production end] [6]. These results demonstrate that the optimum SAP-AP1 system offers superior mobility control functionality relative to the baseline AP1 polymer. The structural strength of the SAP-AP1 system is more effective in generating a stable viscous displacement that promotes a more efficient volumetric heavy oil sweep, a faster WOR reduction, and accelerated heavy oil recovery.

6. Conclusions

We discussed the performance of an advanced supramolecular polymer-surfactant system, SAP-AP1, driven by β-cyclodextrin host-guest complexations as mobility control agent to displace heavy oil (i.e., 2560 cP at 25°C).

Heavy oil recovery displacement tests demonstrated that the SAP-AP1 system shows suitable propagation and low retention in unconsolidated sand-pack systems. The SAP-AP1 system displays superior mobility control efficiency when compared to the baseline AP1 polymer. The higher structural strength of the SAP-AP1 system makes it more effective in generating a stable viscous displacement front that results in a more efficient volumetric sweep, a faster WOR reduction, and accelerated heavy oil recovery. An average additional incremental oil recovery of 19% was achieved relative to the baseline AP1 polymer.

The important incremental oil recovery achieved by the supramolecular polymer-surfactant system is also attributed to the synergistic effect of greater mobility control functionality and decreased interfacial tension (IFT) between the oil-brine system offered by the SAP-AP1 system.

Overall, the SAP-AP1 system offers the potential for increasing heavy oil recovery at economically favorable conditions.

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