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Effect of Concentration on HPAM Retention in Porous Media

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Abstract

This paper investigates the effect of HPAM polymer concentration on retention in porous media using both static and dynamic measurements. Consistent results using these two methods show that different polymer retention behaviors exist in dilute, semidilute and concentrated regions. In both the dilute and concentrated regions, polymer retention is basically concentration-independent. In contrast, in the semidilute region, polymer retention is concentration-dependent. If a porous medium is first contacted sufficiently with dilute polymer solution to satisfy the retention, no significant additional retention occurs during exposure to higher HPAM concentrations. Based on the experimental results, a concentration-related retention mechanism is proposed that considers the orientation of the adsorbed polymer molecules and the interaction between molecular coils in solution. Using this model, we explain why polymer retention does not show much dependence on concentration in the dilute and concentrated regimes. Further, in the semidilute region, we explain how moderate coil interactions lead to mixed adsorbed polymer orientation and magnitude on rock surfaces, and retention becomes concentration-dependent. In field applications of polymer and chemical floods, reduced polymer retention may be achieved by first injecting a low-concentration polymer bank.

Introduction

When water-soluble, high molecular weight polymers are used for enhanced oil recovery (EOR), polymer retention retards propagation into the formation. The presence of the polymer is needed to provide high viscosity and low mobility levels—which in turn are needed to improve oil displacement and sweep efficiency. Consequently, high polymer retention can substantially delay oil displacement and recovery. To illustrate this point, consider the range of polymer retention levels reported in the literature—9 to 700 $\mu\text{g/g}$ (Green and Willhite 1998)—and the range of polymer concentrations used in polymer floods—500 to 3,000 ppm. Given the rock density (ρ_{rock} , 2.65 g/cm^3 for quartz), porosity (ϕ , e.g., 0.3), polymer retention in $\mu\text{g/g}$ (R_{ret}), and polymer concentration in ppm (C_{poly}), Eq. 1 can be used to calculate the delay (PV_{ret} , pore volume delay per pore volume injected).

$$PV_{ret} = [\rho_{rock} (1 - \phi) / \phi] [R_{ret} / C_{poly}] \dots\dots\dots(1)$$

Using this equation and the parameters mentioned above, Fig. 1 shows delay factors. With a very low retention level of 10 $\mu\text{g/g}$ and a polymer concentration of 2,000 ppm, the delay factor is only about 3% of one pore volume (PV). In contrast, for a high retention of 500 $\mu\text{g/g}$ and a polymer concentration of 500 ppm, the delay factor is over 6 PV. For more typical values of 150 $\mu\text{g/g}$ for retention and a polymer concentration of 1,500 ppm, the delay factor is about 0.6. For this latter combination, a 20% difference in retention would mean an extra 12% PV polymer bank needed (if the retention is higher) or not needed (if the retention is lower) to accomplish a given objective. In one 40-acre 5-spot pattern with a height of 20 ft and a porosity of 0.3, 0.12 PV of 1,500-ppm HPAM (costing \$1.5/lb) would represent a polymer cost of about \$176,000.

From another viewpoint, the mass of rock in the above 40-acre pattern is $40 \times 43560 \times (12 \times 2.54)^3 \times 2.65 \times (1 - 0.3) / 0.3 = 6.10 \times 10^{12}$ grams. Given the retention levels of 10, 50, 120, 150, 180, and 500 $\mu\text{g/g}$, and an HPAM cost of \$1.5/lb, the polymer costs required to satisfy the retention requirements of the rock would be \$201,777, \$1,008,883, \$2,421,320, \$3,026,650, \$3,631,980, and \$10,088,835, respectively.

Of course, the delay in polymer propagation also delays oil recovery. Fig. 2 illustrates this point using fractional flow calculations (from <http://baervan.nmt.edu/andy/>). For these calculations, we assumed oil viscosity of 1,000 cp, water viscosity of 1 cp, and the reservoir was initially at connate water saturation ($S_{wr}=0.3$). The reservoir was then flooded with one PV of

water (before continuous polymer flooding with 100-cp polymer), one homogeneous layer was present, flow was linear, and the following relative permeability curves (Eq. 2 and 3) were used.

$$k_{rw} = 0.1 \times [(S_w - 0.3) / (1 - 0.3 - 0.3)]^2 \dots\dots\dots(2)$$

$$k_{ro} = 1 \times [(1 - 0.3 - S_w) / (1 - 0.3 - 0.3)]^2 \dots\dots\dots(3)$$

In Fig. 2, the term, *IAPV*, refers to inaccessible PV, which is defined as the fraction of the pore space being inaccessible to the large polymer molecules but accessible to the small solvent and salt molecules and ions. *IAPV* accelerates polymer propagation, whereas polymer retention (*PV_{ret}*) retards it. Three different levels were considered in Fig. 2—where retention plus *IAPV* (1) were perfectly balanced to cause no delay in polymer propagation (i.e., *PV_{ret}* + *IAPV*=0), (2) caused a one PV delay (i.e., *PV_{ret}* + *IAPV*= -1), and (3) caused a 2.5 PV delay (i.e., *PV_{ret}* + *IAPV*= -2.5). Fig. 2 illustrates that the delay in the arrival of the oil bank is directly proportional to the delay in polymer propagation. Consequently, high polymer retention is economically detrimental because of increased cost for polymer and delayed oil recovery.

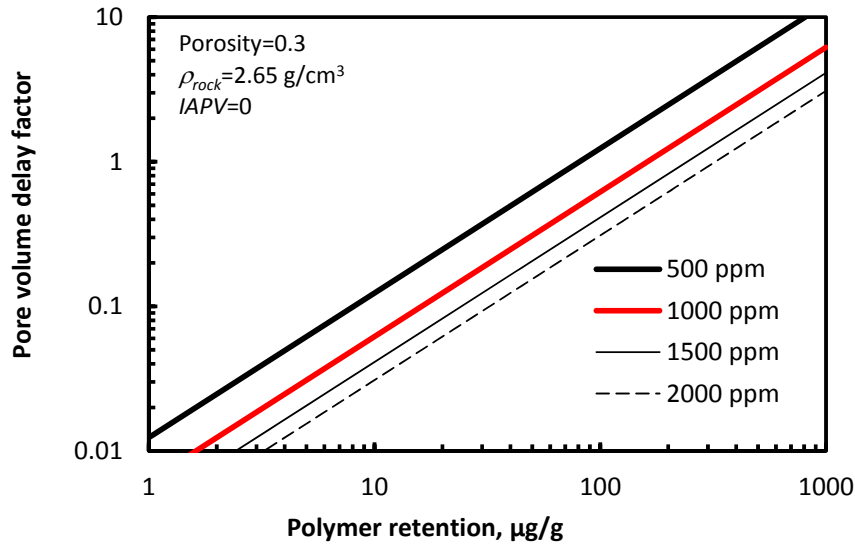


Fig. 1—Polymer bank delay factors associated with polymer retention.

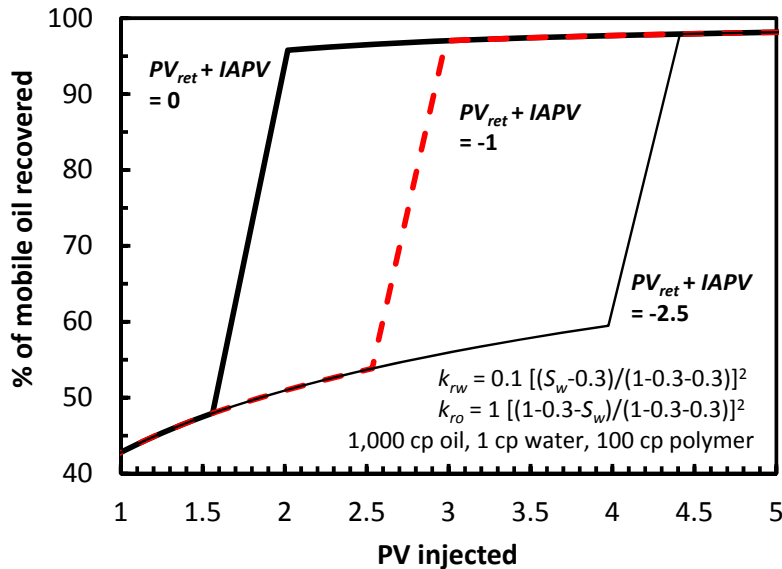


Fig. 2—Delay in oil recovery caused by retention.

It is widely accepted that polymer retention comprises polymer adsorption on the rock surface and mechanical entrapment in small throats and pores (Gogarty 1967). A third type of retention is related to the hydrodynamic force acting on polymer molecules, and this flow-induced retention is called hydrodynamic retention (Maerker 1973; Dominguez and Willhite 1976; Aubert and Tirrell 1980; Zaitoun and Kohler 1987; Huh *et al.* 1990).

Petroleum literature review shows that among the factors influencing polymer retention, polymer concentration is controversial. Some researchers suggested that polymer retention in the porous media is concentration-dependent or fits the Langmuir isotherm (Mungan 1969; Dawson and Lantz 1972; Szabo and Corp 1975, 1979; Deng *et al.* 2006). Close examination of these reports reveals that most measurements were performed using a static method. The Langmuir adsorption model is currently used in most chemical flooding simulators to describe polymer retention in porous media (Satter *et al.* 1980; Vossoughi *et al.* 1984; Camilleri *et al.* 1987; Yuan *et al.* 2010; Dang *et al.* 2011). In contrast, results from other researchers show polymer concentration is a minor factor (Shah *et al.* 1978, Green and Willhite 1998, Zheng *et al.* 1998). Generally, these studies used dynamic measurements, where the same cores were used repeatedly in the tests. Specifically, a given core was flooded first with a low polymer concentration; then retention was assessed; then a higher polymer concentration was injected into the same core to assess whether any further retention occurred. Although Zheng *et al.* claimed their data fit the Langmuir isotherm, their retention values increased from 40 $\mu\text{g/g}$ at 250 ppm to only 58 $\mu\text{g/g}$ at 1,500 ppm. Only a few studies employed new cores or sandpacks for each concentration; these studies demonstrated moderate concentration dependence for retention (Szabo and Corp 1975, Huang and Sobie 1993).

Results from different literature studies are comparable only if measurements were conducted under similar conditions. This is especially true when comparing results from static measurements with those from dynamic measurements. In this paper, a series of tests were designed to clarify literature discrepancies concerning how polymer concentration affects retention in porous media. Several types of experiments were performed, including static measurements of polymer retention on fresh sand for each concentration case; and dynamic measurements of polymer retention in new sandpacks with similar permeability and porosity for different HPAM concentrations. We also examined polymer retention measurements where a single sand, sandpack, or sandstone core was exposed to successive solutions with increasing polymer concentration. HPAM polymer solutions with a broad concentration range (from 10 to several thousand parts per million) were utilized.

Materials and Methods

Polymer and Brine. SNF Flopaam 3230S, a partially hydrolyzed polyacrylamide (HPAM) polymer, was used in our tests. It was provided by the manufacturer as a white granular powder with an estimated molecular weight of 6-8 million Daltons and degree of hydrolysis of approximately 30%. After the polymer was dissolved using the magnetic stirrer vortex method, the solution was filtered through a 10- μm filter to remove any microgels and debris. Two brines were used: one was 2% NaCl for the static measurements and coreflooding using sandpacks; the other brine contained 2.52% TDS (2.3% NaCl and 0.22% NaHCO_3) when consolidated sandstone cores were used. Both brines were filtered through 0.45- μm filters before application.

Sand Preparation. Sand grains with particle sizes between 106–180 μm were prepared as the adsorbent by crushing and sieving Berea sandstone. To reduce the presence of very fine particles, special processes were undertaken for the treatment of these disaggregated sands. First, the sand was put into a bottle with brine and rotated at 300 rpm for 8 hours. Then, the muddy liquid phase was separated from the sand. Next, the sand was washed with distilled water to remove newly-generated fine particles and residual salt until the upper water phase was clear. Polymer concentration was determined using a total organic carbon analyzer. Finally, the sand was dried at 110 $^{\circ}\text{C}$.

Porous Media. Disaggregated sands prepared as described above were used for static adsorption measurements. To determine dynamic polymer retention in porous media, both sandstone cores and sandpacks were used. The Dundee sandstone core had a permeability of 347 mD and porosity of 0.23. This rectangular core (with 15-cm length and a 14.5-cm² cross section) was cast in epoxy. Sandpacks with relatively similar permeability and porosity were prepared from the same sands used for static measurements. Sandpacks were 6.35-cm long and 14.5 cm² in cross section.

Polymer Concentration Determination. For static measurements, the concentration of the initial and equilibrium polymer solutions were determined using a total organic carbon (TOC) analyzer (Shimaduz Model TOC—V_{CSH}). If a polymer solution was too viscous, dilutions were made before measurement. When dynamic measurements were performed, polymer concentration in the effluent was determined based on fluid viscosity. When flowing through a 10- μm filter combination (see Seright *et al.* 2009) connected to the outlet of the core, reliable pressure drops were detected even for HPAM concentrations as low as 25 ppm (Seright *et al.* 2011). Using this method, the effluent polymer concentration could be monitored continuously.

Static Adsorption Measurements. Bottles containing sand and polymer solution (of known mass) were fastened to a roller, which rotated at a speed of 6 rpm for 1 hour. One hour contact was considered sufficient because the polymer adsorption on the rock surface is believed to be instantaneous (adsorption kinetics will be discussed later). After rotation, the upper polymer solution was transferred to a plastic tube to be centrifuged at 3,000 rpm to settle any sand particles. Equilibrium polymer concentration was determined by a total organic carbon analyzer. Polymer adsorption for each concentration was calculated

using Eq. 4 (based on a mass balance):

$$R_{pret} = (C_0 - C_{eq}) \times V_p / W_{sg} \dots\dots\dots(4)$$

where R_{pret} is polymer adsorption, $\mu\text{g/g}$ sand, C_0 and C_{eq} are initial and equilibrium polymer concentrations, ppm. V_p is polymer volume, cm^3 , and W_{sg} is sand weight, g. Both polymer and brine density were assumed to be 1 g/cm^3 .

Dynamic Retention Measurements. Several methods were proposed to measure polymer retention in porous media (API RP63 1990, Dawson and Lantz 1972, Szabo 1975, 1979, Dominguez and Willhite 1977, Gupta and Trushenski 1978, Castagno *et al.* 1987, Huh *et al.* 1990, Mezzomo *et al.* 2002). Several of them advocate injection of a slug of polymer solution, followed by brine, and performing a mass balance on the polymer (i.e., retention = polymer injected minus polymer produced). Key problems with this type of method are (1) recovery of the polymer may require an extended period of brine injection because of the unfavorable displacement and (2) cumulative errors associated with measurements of low polymer concentrations in the produced fluid can introduce considerable uncertainty to the mass balance.

We prefer the method used by Lotsch *et al.* (1985), Hughes *et al.* (1990), and Osterloh and Law (1998). In this method, two banks of polymer solution are injected which are separated by a brine slug. Polymer retention can be determined by the plot of the two effluent polymer concentration profiles versus pore volume injected. Fig. 3 shows the schematic diagram of our experimental unit to determine polymer retention in porous media.

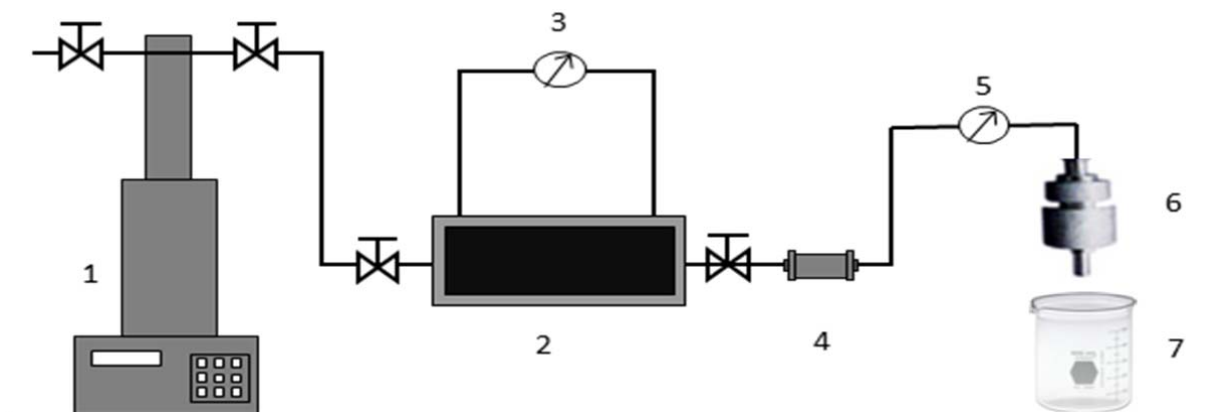


Fig. 3—Schematic diagram of polymer retention determination system.

Note: 1. ISCO syringe pump (Model 500D); 2. Sandstone core or sandpack; 3. Pressure transducer #1; 4. $7 \mu\text{m}$ Swagelok filter; 5. Pressure transducer #2; 6. $10 \mu\text{m}$ Millipore filter combination, 7. Beaker.

Results and Discussions

Adsorption Kinetics. The kinetics of polymer adsorption was first analyzed by mixing 100-ppm polymer solution with sand. Samples of the polymer solution were taken periodically for polymer concentration determination. The results of adsorption versus time are plotted in Fig. 4. Adsorption reached the maximum (about $40 \mu\text{g/g}$ sand) within about three minutes and then leveled off. This indicates polymer adsorption on the sand surface can be considered instantaneous. In our tests, the contact of polymer solution and sand lasted 1 hr to ensure adsorption equilibrium.

Desorption Test. After adsorption, desorption tests were carried out to estimate the amount of polymer that can be removed. In this test, excess polymer solution was decanted from the top of the sand. Then fresh brine was added and again the bottle containing both sand and brine was rotated at 6 rpm for 1 hr. When the sands settled, the upper phase solution was sampled for polymer concentration determination. The residual polymer adsorption was calculated using mass balance. This procedure was repeated until no more desorbed polymer was detected. Figure 5 shows the results for 100-, 500-, and 1,000-ppm HPAM. Calculations show that the percentage of the reversible adsorption for these three cases was 6.6%, 2.4%, and 2.9%, respectively. This result was similar to that from Deng *et al.* (2006). Because EOR polymers have high molecular weights and extended chains, many polar groups along the polymer chain will attach to many different polar points on the rock surface. It is statistically very unlikely that a polymer molecule would release all points of attachment at the same time. Therefore, polymer adsorption on the sand surface can be treated as almost irreversible.

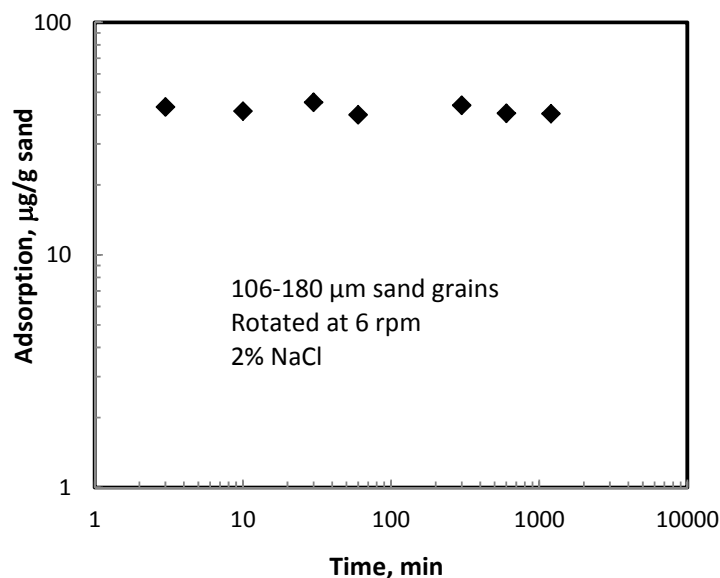


Fig. 4—Kinetics of polymer adsorption on sand.

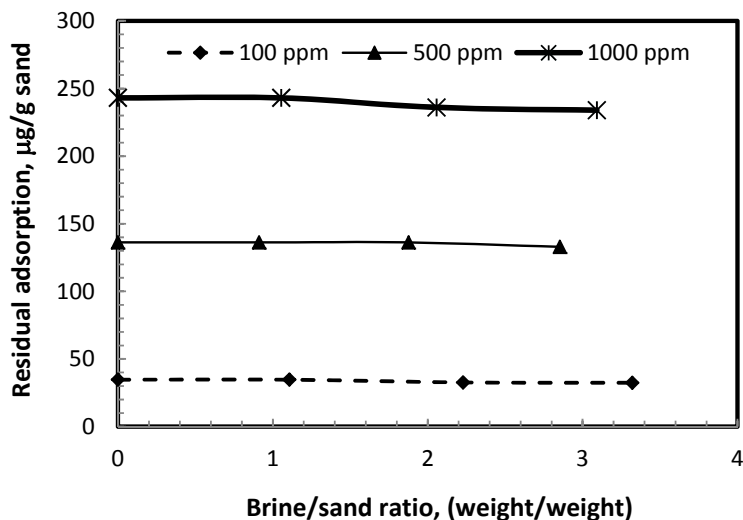


Fig. 5—Desorption tests for 100-, 500-, and 1,000-ppm HPAM.

Effect of Polymer Concentration. To investigate the effect of polymer concentration on retention, polymer solutions were examined with concentrations from 10 ppm to 6,000 ppm. The results are illustrated in Fig. 6, which suggests three distinct concentration-related retention behaviors. First, in the very low concentration region (from 10 ppm to about 100 ppm), polymer retention stabilized approximately at a value of 20 µg/g. In the intermediate-concentration region (from 100 ppm to about 4,000 ppm), polymer retention increased from 35 to 420 µg/g, increasing almost linearly with polymer concentration. In the very high concentration region (above 4,000 ppm), nearly constant retention (~ 420 µg/g) was achieved.

These results (especially the concentration-dependent observation) agree with the previous findings where most of the measurements were made in the intermediate concentration region (Mungan 1969; Espinasse and Siffert 1979). Our findings indicate that polymer retention does not fit the Langmuir isotherm, which is commonly used to describe the reversible adsorption of small molecules such as surfactants and gas. For EOR polymers with high molecular weights and extended chains, their adsorption on rock shows little reversibility (see Fig. 5). It is postulated that at very low concentration, polymer molecules continue to be adsorbed until the maximum coverage is reached. During this process, few adsorbed polymer molecules are likely to detach from the surface. Therefore, unlike the adsorption described by the Langmuir isotherm, polymer adsorption at very low concentration approaches a constant non-zero value.

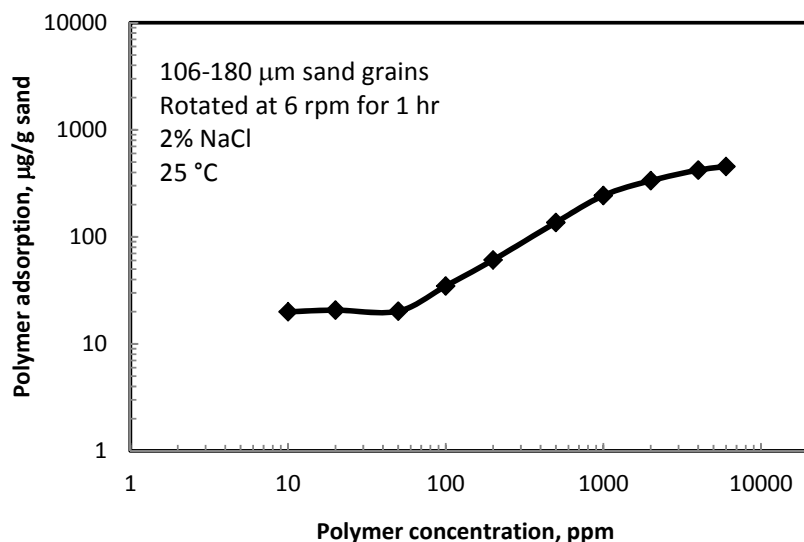


Fig. 6—Adsorption isotherm of HPAM using the static method.

Re-Adsorption Test. Fresh sands were used for each case to generate the adsorption isotherm shown in Fig. 6, which illustrates the concentration-related adsorption behaviors. After the desorption tests in Fig. 5, 1,000-ppm polymer solution was added to the sands previously contacted with 100-ppm and 500-ppm polymer solution to check if polymer re-adsorption occurred. Little additional polymer was adsorbed onto the used sands. For instance, for the 100-ppm concentration case, the retention increased from 32.4 to 35.8 µg/g, increasing by 10.3%. For the 500-ppm case, retention rose from 132.9 to 141.1 µg/g—merely a 6.1% increase. Compared to the adsorption at a concentration of 1,000 ppm (243 µg/g, as shown in Fig. 7), a large retention difference existed between the fresh sand and sand whose retention was satisfied by low-concentration polymer. Apparently, even though polymer adsorption was relatively small at low concentration, the surface was already fully covered by adsorbed polymer molecules, and no vacant sites were available for further attachment.

Dynamic Method

Retention in Sandpacks. Dynamic measurements were performed in sandpacks made from the same sand source that was used for static measurements. For each concentration, a new sandpack was used. As shown in Table 1, these sandpacks had very similar properties. Permeability ranged from 4.69 to 5.51 Darcy, and porosity ranged from 0.432 to 0.441. Because of the high permeability, we suggest that polymer adsorption dominated the retention (Szabo and Corp 1975, Huh *et al.* 1990). Polymer solutions with concentrations of 20, 50, 100, 500, 1,000 and 2,000 ppm were investigated using an injection rate of 120 cm³/hr (6.6 ft/day flux). The retention is shown by both Table 1 and Fig. 8. Retention was around 5 µg/g at low concentrations from 20 ppm to 100 ppm. With the increase of concentration from 100 ppm to 2,000 ppm, retention increased from 5.71 to 27.8 µg/g—increasing by a factor of nearly 5.

After completion of measurements for 100- and 500-ppm cases (Sandpacks #4 and #3 in Table 1), 1,000-ppm solution was injected. Retention increases of 5.6% and 7.3% were detected in these two used sandpacks, respectively. This result agrees with the static measurement on used sands, which also confirms that the adsorbed molecules occupy almost all the vacant sites on the sand surface and prevent further attachment.

Retention in Dundee Sandstone Cores. In addition to the disaggregated sands and highly permeable sandpacks, retention was also measured in two consolidated Dundee sandstone cores. As shown in Table 2, these two cores had similar properties of permeability and porosity. Two distinct strategies were employed. For Core 1, polymer solutions with concentrations from 20 through 1,000 ppm were injected sequentially to determine retention for each concentration. In contrast, for Core 2, only 1,000-ppm solutions were injected. The result from Core 1 is shown in Fig. 9. Retention was approximately 15-16 µg/g. When polymer concentration was greater than 100 ppm, the retention reached a plateau, with the maximum retention of approximately 16 µg/g. In contrast, the retention for Core No. 2 was as high as 56.5 µg/g rock—3.5 times higher than that for the first core (see Table 2). These results agree with our findings mentioned previously. The result from Core 1 was also consistent with other studies where the same core was repeatedly used (Shah *et al.* 1978, Zheng *et al.* 1998).

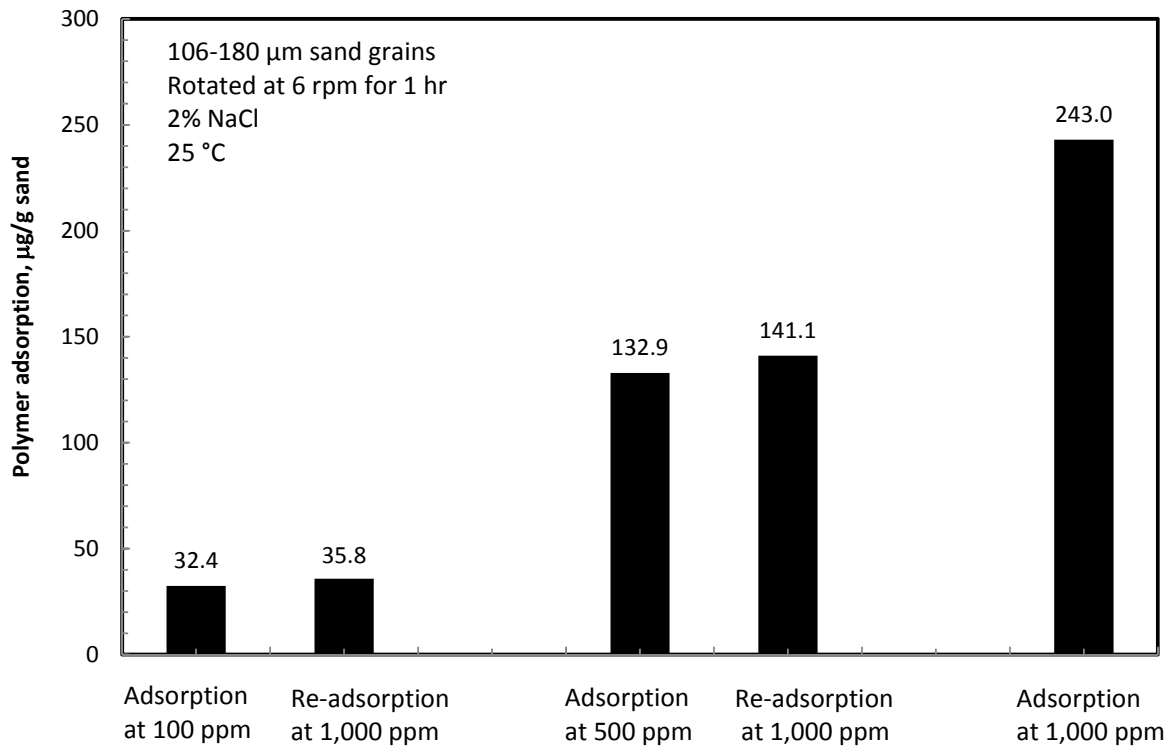


Fig. 7—Comparison of retention on fresh sands and used sands.

Table 1—Dynamic retention in sandpacks.

SP No.	Length, cm	Area, cm ²	Weight of sand, g	Pore volume, cm ³	Porosity	Permeability, mD	Polymer conc, ppm	Retention, µg/g sand
1	6.25	14.19	140.4	39.1	0.441	5.51	2,000	27.8
2			139.7	39.0	0.440	5.04	1,000	14.3
3			140.2	38.7	0.436	4.88	500	10.2
4			141.4	38.3	0.432	4.69	100	5.71
5			141.0	38.7	0.436	5.03	50	4.85
6			140.6	39.1	0.441	5.37	20	4.63

Table 2—Dynamic retention in Dundee sandstone cores.

Core No.	Length, cm	Section area, cm ²	PV, cm ³	Porosity, %	Permeability, mD	Retention, µg/g
1	15	14.5	49	22	347	16.1
2			51	23	449	56.5

Injection rate: 60 cm³/hr or 3.3 ft/day.

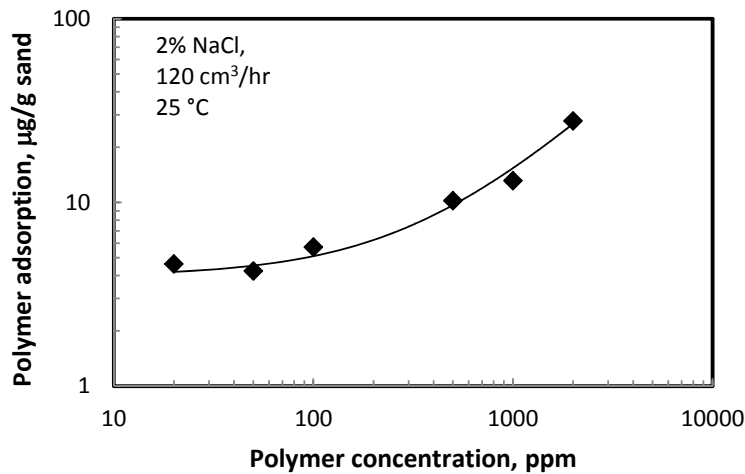


Fig. 8—Adsorption isotherm using the dynamic method (new sandpacks were used for each concentration).

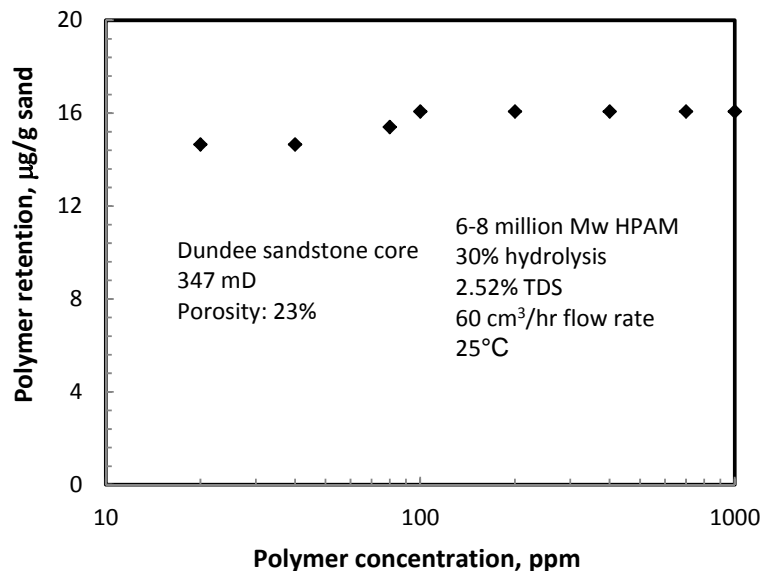


Fig. 9-Adsorption isotherm on 347-mD Dundee sandstone core (the same core was used for all concentrations).

Proposed Adsorption Model

Based on the experimental results, a polymer concentration-related retention model is proposed which accounts for the observed retention behavior. It is well known that polymer molecules may interact with each other in solution and the degree of interaction depends greatly on polymer concentration. Three concentration regimes were proposed (de Gennes 1979; Ying and Chu 1987) as dilute ($c < c^*$), semidilute ($c^* < c < c^{**}$), and concentrated ($c^{**} < c$), where c^* is the overlap concentration crossover from dilute to semidilute regimes and c^{**} is the overlap concentration crossover from semidilute to concentrated regimes. More specifically, as shown in Fig. 10, in the dilute regime, polymer molecules exist in solution as free coils where little interaction occurs. In the semidilute regime where polymer concentration is greater than the overlap concentration, c^* , macromolecules start to contact each other and intermolecular interactions occur. With further increase in concentration (especially when the concentration is above c^{**}), the intermolecular entanglements dominate the interaction, resulting in the formation of a network structure (Ferry 1948). When dealing with polymer retention on sand surfaces, this concentration-based interaction among polymer molecules in solution may be used to explain the adsorption mechanism.

In the dilute regime, polymer molecules exist in solution as free coils but tend to take a flat orientation when they adsorb onto the rock surface. In this configuration, most, if not all of the molecular segments are in contact with the surface. It was called two-dimensional adsorption (Peterson and Kwei 1961). In this regime, two-dimensional adsorption dominates retention, and polymer molecules continue to be adsorbed until the maximum coverage is reached. As shown by Region A in Fig. 11,

adsorption is independent of polymer concentration.

In the semidilute regime, the intermolecular interaction in solution will result in a mixed adsorption, i.e., some molecules will be adsorbed with all the segments in contact with the surface, while others will be adsorbed with only partial segments in contact with the surface. The latter orientation will be labeled as three-dimensional adsorption. Increasing the polymer concentration will increase the three-dimensional adsorption as well as the total adsorption, as shown by Region B in Fig. 11. Polymer retention is concentration-dependent in the semidilute regime.

In the concentrated regime, the molecular entanglement in solution causes the three-dimensional adsorption to dominate. In this case, almost no additional polymer molecules can be adsorbed with increasing concentration because all sites are taken. As shown by Region C in Fig. 11, the adsorption is concentration-independent.

This model also explains why no significant adsorption occurred during exposure to a high-concentration solution after sand was first contacted with dilute polymer solution. After the surface maximum coverage is achieved, no more vacant sites are available.

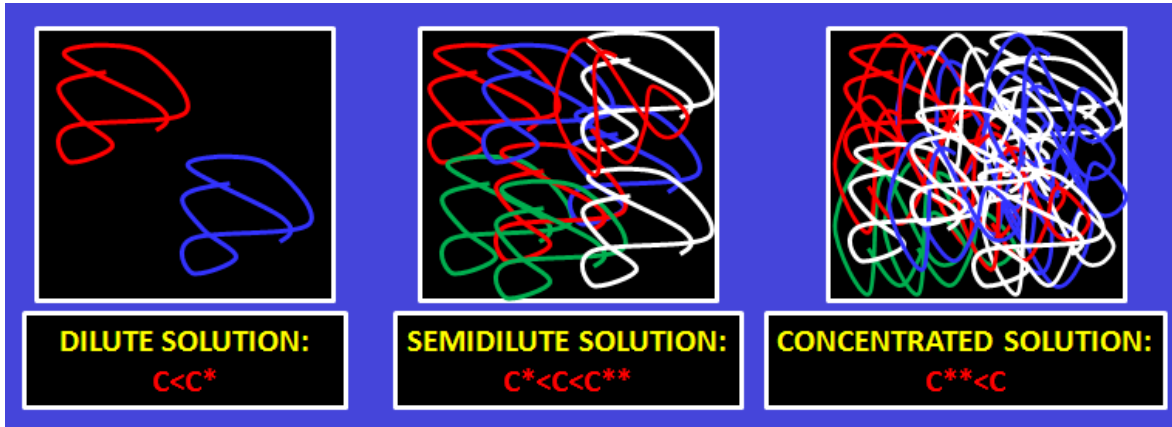


Fig. 10—Polymer molecule interaction at different concentrations.

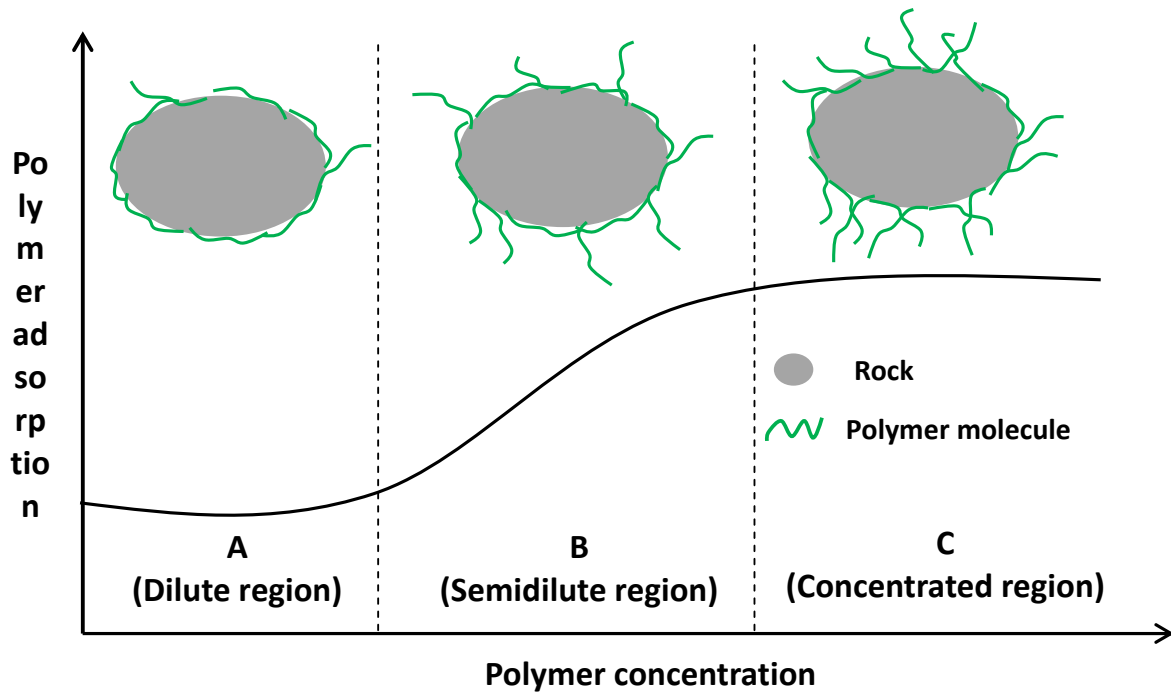


Fig. 11—Proposed polymer adsorption mechanism on the rock surface.

Conclusions

1. HPAM polymer adsorption on a rock surface can be considered instantaneous and irreversible.
2. Different polymer retention behaviors are observed in dilute, semidilute and concentrated regions. In both dilute and concentrated regions, polymer retention is basically concentration-independent. In contrast, in the semidilute region, polymer retention is concentration-dependent.
3. If a porous medium is first contacted with dilute HPAM solution to satisfy the retention, no significant additional retention occurs when exposed to higher concentrations. In field applications of polymer and chemical floods, reduced polymer retention may be achieved by first injecting a low-concentration polymer bank
4. Based on the experimental results, a concentration-related retention mechanism is proposed that considers the orientation of the adsorbed polymer molecules and the interaction between molecular coils in solution.

Acknowledgement

We thank Statoil for financially supporting this work.

Nomenclature

c^*	= polymer overlap concentration crossover from dilute to semidilute regime
c^{**}	= polymer overlap concentration crossover from semidilute to concentrated regimes
C_0	= initial polymer concentration, ppm
C_{eq}	= equilibrium polymer concentration, ppm
C_{poly}	= polymer concentration, ppm
k_{ro}	= relative permeability to oil
k_{rw}	= relative permeability to water
PV_{ret}	= pore volume delay per pore volume injected
R_{pret}	= polymer adsorption, $\mu\text{g/g}$ sand
S_{wr}	= residual water saturation
V_p	= volume of polymer solution, cm^3
W_{sg}	= weight of sand, g
ϕ	= porosity
ρ_{rock}	= rock density, g/cm^3
<i>EOR</i>	= enhanced oil recovery
<i>HPAM</i>	= partially hydrolyzed polyacrylamide
<i>IAPV</i>	= inaccessible pore volume
<i>PV</i>	= pore volume
<i>rpm</i>	= revolution per minute
<i>TOC</i>	= total organic carbon

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SI Metric Conversion Factors

ft × 3.048*	E-01 = m
in. × 2.54*	E+00 = cm
lb × 4.535 9237	E-01 = kg
md × 9.869 233	E-01 = μm ²

*Conversion factor is exact.
