Performance of Sulfonated Polyacrylamide/ Chromium Triacetate Hydrogels for Water Shut-off Treatment

A. Dadvand Koohi[1], M. Vafaie Sefti[2*], A. Zarrin Ghalam [2], A. Mousavi Moghadam[3] & H. Naderi[4]
1- Chemical Engineering Department, Guilan University, Rasht, Iran
2- Chemical Engineering Department, Tarbiat Modares University, Jalal Al Ahmad Highway, Tehran, Iran
3- Oil, Gas and Thermodynamic TMU Research Group, Tehran, Iran
4- Center for Exploration and Production Studies and Research Division, Research Institute of Petroleum Industry (RIPI)
Vafaiesm@modares.ac.ir

Abstract
A hydrogel was prepared by crosslinking aqueous solutions of sulfonated polyacrylamide/chromium triacetate for use in water shut off operations. Gel swelling and the effects of salinity, injection time and flow rate on residual resistance factor (F_{rr}) were investigated. In the presence of electrolytes, gel swelling decreased by about 80%. Results showed that oil permeability increased as injection time increased. The results also indicated that the effect of gel treatment increased with decreasing injection rate. However, when sand packed was water flooded by formation water, Frrw decreased by about 26%. According to the results, Disproportionate Permeability Reduction (DPR) was the result of gel swelling by water injection and dehydration by oil injection.

Key words: Polymer Gel, Sulfonated Polyacrylamide, Swelling, Dehydration, Residual Resistance Factor.

Introduction
As more oil reservoirs become mature, unwanted water production in association with crude oil becomes a big production concern in petroleum industry. This phenomenon often decreases the economic life of a well. Therefore, there is a dire need to reduce excessive water production from the reservoirs [1-6]. Hydrogels have many applications in drug delivery [7], agriculture [8], membrane technology [9] and enhanced oil recovery [10]. Polymer gels used for improved oil recovery (IOR) are typically composed of a polymer or co-polymer and a crosslinker (soluble in water), which are injected into the target zones [11]. Most of these systems are based on polymer solutions that turn from low viscosity liquids to strong or weak gels after a given time depending on their formulations. These gels, which are the basis of most water shut-off treatments, can partially or completely block the channels through which water is being produced. Chromium acetate/partially hydrolyzed polyacrylamide [12, 13] or polyethyleneimine/co-polymer of acrylamide and t-butyl acrylate [14] are two well known examples of these hydrogels. The former is an ionic crosslinked gel and the latter is a covalent gel.

Zolfaghari *et al* [15] used nanocomposite type of hydrogels (NC gels) by crosslinking the polyacrylamide/ Na-montmorillonite nano clay aqueous solutions with chromium (III). They showed that the rate of gelation is retarded; especially for the gelant solution composed of 2% (volume percent) of sodium lactate as retarder. They observed that in adding retarder to the gelants, the more the retarder content increased, the less synergism happened. Aalaie *et al* [16] analyzed the gelation process and effects of clay (montmorillonite) content and ionic strength on the swelling behavior of PAMPS/ Cr(III)-acetate using dynamic rheometry. They showed that the swelling ratio of nanocomposite gels in tap water decreased as the concentration of the clay increased. It was also found that with increasing the clay content, the viscous energy dissipation properties of the nanocomposite gels increased. Lee *et al* [17]. showed that double network hydrogels (DN-gels) prepared from the combination of a moderately crosslinked copolymer of 2-acrylamido-2 methyl propanesulfonic acid sodium salt and acrylamide (PAMPS) and an uncrosslinked linear polymer (polyacrylamide, PAAm) solution showed strong mechanical properties far superior to those of their individual constituents. Jia et al. [18] determined the effects of some parameters such as molecular weight of polymer, concentration of polymer and crosslinker, which lead to decrease in gelation time, and concentra tion of inorganic salts, which lead to increase in gelation time. Some researchers studied the performance of polymer gel systems in the near well bore area to decrease water permeability much more than oil permeability, the so-called disproportionate permeability reduction (DPR) effect [19, 20]. Another application of gelling system is to seal fractures, which are responsible for increasing abruptly the unwanted produced water [21]. Willhite et al. [13] demonstrated that disproportionate permeability was thought to occur because the residual oil was trapped in the new pore structure when the oil was displaced by water. Disproportionate permeability reduction has been found to be a function of the pressure gradient initially applied to dehydrate the gel and the flow rate (or pressure gradient) in the new pore structure created by dehydration. Liang et al. [22] found that permeability to brine significantly increased with the flow rate, but they reported insignificant changes in permeability to oil with the flow rate.

Since most of the Iranian reservoirs have high temperatures (about 90°C) and salinities, in this work, PAMPS was selected for the experiments due to its higher thermal stability and salt resistance compared with standard hydrolyzed polyacrylamides. In addition, it is used in enhanced oil recovery applications up to 120 °C. In general, the rheological properties of Cr(III)-acetate/PAMPS hydrogels and the effect of different parameters on the gel strength have been well studied.

Generally, most reported works concerning polymer gel have been performed on hydrolyzed polyacrylamides in comparison with sulfonated polyacrylamides. In addition, most of the studies on sulfonated polyacrylamide were carried out on the properties of gel swelling and strength in comparison with the effect of gel in porous media. Therefore, determination of the effect of such parameters as swelling, salinity and flow rate on the performance of polymer gels is highly significant. The present work is an attempt to study the effect of flow rate and salinity on the permeability reduction of oil and brine. The experiments were planned to gain further information on the role of gel rehydration and salinity in explaining the DPR mechanism. In this work, at first the gel swelling treatment of sulfonated polyacrylamide/chromium triacetate was investigated in the presence of formation water and distilled water. Then, a series of coreflood experiments were conducted using tap water and formation water (with the total dissolved solid of 84.74 wt %) at 90°C. The core flooding results were analyzed based on the variation of Frr (residual resistance factor) and pressure drop evolution. Finally, the comparison between the results obtained using polymer gel and that of other polymer gels was carried out.

Experimental

Materials

Sulfonated polyacrylamide (PAMPS) with an average molecular weight of 8,000,000, sulfonation degree of 25% and water content of less than 10 wt%, was provided by SNF Co. (France). It is sold under the trade name of AN125 in powder form. The molecular structure of this co-polymer is shown in Figure 1. Chromium triacetate, as a metallic crosslinker, was purchased from Carlo Erba Co. (Italy) and used in powder (pure) form. Distilled water was prepared in situ and used as a solvent to prepare gelant solutions. To evaluate gel performance during core flooding, tap and formation waters were used as injection fluids. Table 1 presents the properties of formation water.

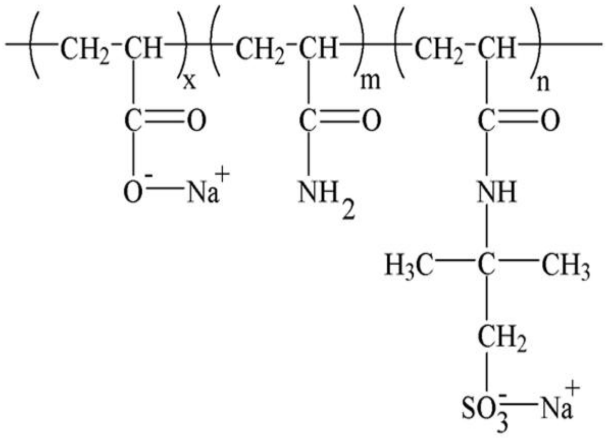

Figure 1. Sulfonated polyacrylamide structure

Table 1. Composition and properties of formation water (all concentrations were expressed in weight percent (wt %), Density=1.145 g/cm^3)

Parameter	Concentration (wt %)
Sodium	29.66
Calcium	2.33
Magnesium	0.69
Potassium	0.54
Total Iron	0.07
Chloride	44.71
Carbonate	6.6
Bicarbonate	390
Total Inorganic Dissolved Solids	84.74
pH	7.3

Sample Preparation and Characterization

The polymer gels were prepared in the following three steps: A) PAMPS solutions at the concentration of 2 % (w/w) were prepared by adding co-polymer powder into distilled water and stirring for 2 hours. Then, stirring was stopped for 2 days until a homogeneous solution was obtained. Shortly before the commencement of the experiment, the PAMPS solutions were diluted to the required concentrations. B) Cr(III)-acetate (as crosslinker) and the other additives were mixed with distilled water at room temperature using a magnetic heating stirrer (Stuart CB162) for 10 min to obtain the «second solution». C) The PAMPS and second solutions were mixed to obtain a gelant solution.

To measure the equilibrium swelling ratios, tea bags (i.e., a 100 mesh nylon screen) containing pre-weighed dry gel samples were entirely immersed in distilled or formation water and allowed to soak for 7 days to reach equilibrium. The equilibrated swollen gels were allowed to drain by removing water from the tea bags and the excess surface water was removed by filter paper. The equilibrium swelling ratio (ESR) was measured using the following equation [16]:

$$ESR = \frac{W_s - W_0}{W_0} \tag{1}$$

where W_0 and Ws are the weights of dry and the swollen gel, respectively. The salt sensitivity factor (f) for each gel in the formation water and distilled water was calculated from the following equation:

$$f = \frac{ESR - ESR_e}{ESR} \tag{2}$$

where ESR and ESR_e are the equilibrium swelling ratios of each gel in distilled and formation water, respectively.

Coreflood experiments were conducted to evaluate the effectiveness of Cr(III)-acetate/PAMPS co-polymer gel system to reduce its water permeability. Figure 2 depicts the experimental set up used for the dynamic coreflood tests. The procedure of the experiments was as follows:

1- Sand packed (containing SiC sands) was placed in the core holder (internal diameter: 6.1 cm and length: 30 cm). Then, the sand packed was saturated with tap water or formation water to measure sand packed pore volume and porosity.

2- Tap water or formation water was injected into the sand packed at 90°C until a stable pressure difference was recorded. Then, the absolute permeability of sand packed was calculated by using Darcy's flow equation.

3- Oil (prepared from the south of Iran) was subsequently injected into the sand packed at 90°C to calculate $k_{ro}@S_{cw}$.

4- In order to reach residual oil saturation, tap water or formation water was injected again into the sand packed. Thus, the effective water permeability at the residual oil saturation (k_{rw}@Sor) was calculated.

5- Gelant solution (450 cc), containing 0.95 % (w/w) of co-polymer and 1:5 of crosslinker/co-polymer weight ratio, was injected into the sand packed [23].

6- All connecting lines of the core holder were flushed by using fresh water to remove the gel solution from the connecting lines.

7- At the end of the injection of gelant, the apparatus was shut in order to evaluate the gel treatment performance.

8- After the shut-in period, tap or formation water and oil were alternatively injected into the treated sand packed to evaluate the effectiveness of gel in reducing water or oil permeability.

Results and Discussions

Swelling of the Gel

The characteristics of the used samples are presented in the second column of Table 2. The amount of the gel swelling is given in Figures 3 and 4. The results showed that swelling of the gels decreased with increasing the crosslinker concentration because of an increase in the crosslinking density. An increase in co-polymer concentration caused increased availability of carboxylate and amide groups in the system. Thus, the osmotic pressure difference increased between the gel network and the solvent and thus gel swelling increased

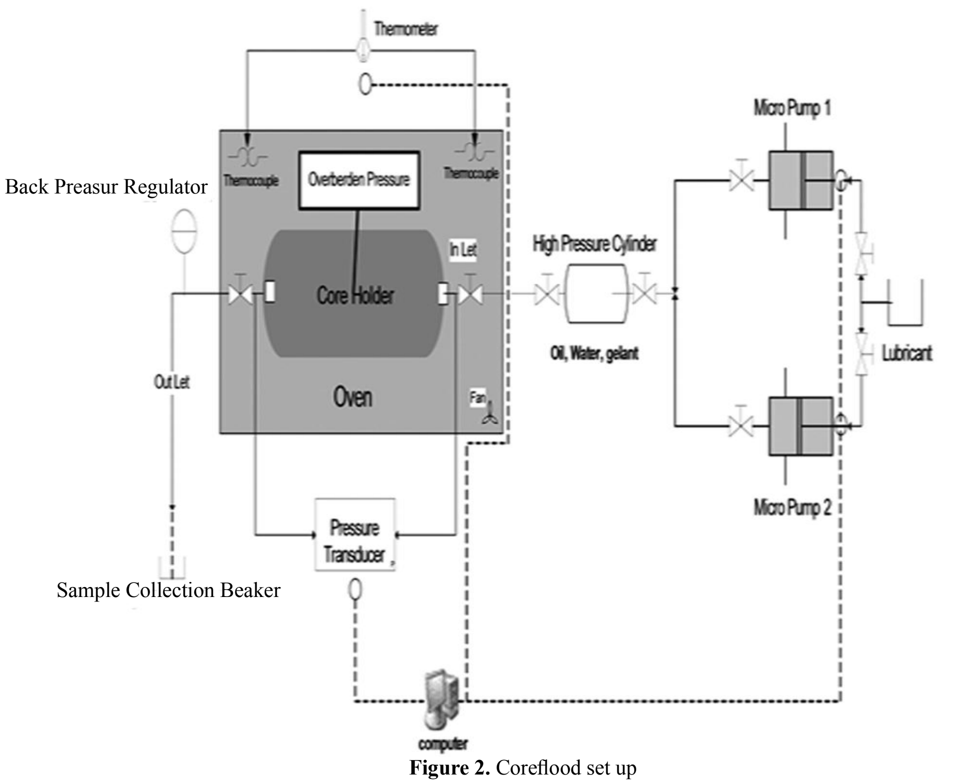

Figure 2. Coreflood set up

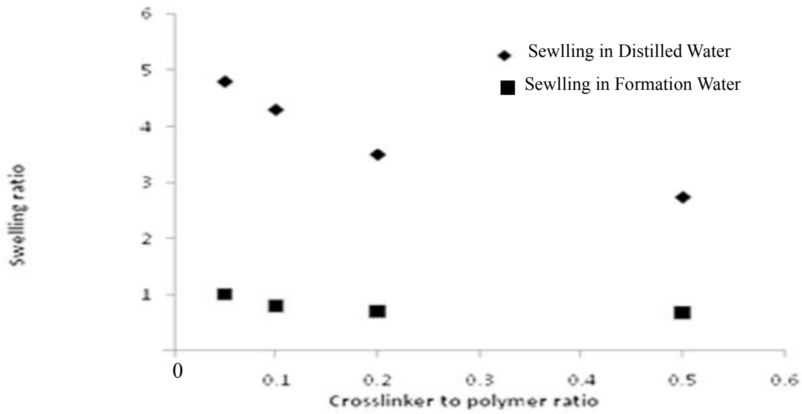

Figure 3. The effect of ratio of crosslinker/co-polymer on the swelling (9500 ppm of polymer concentration)

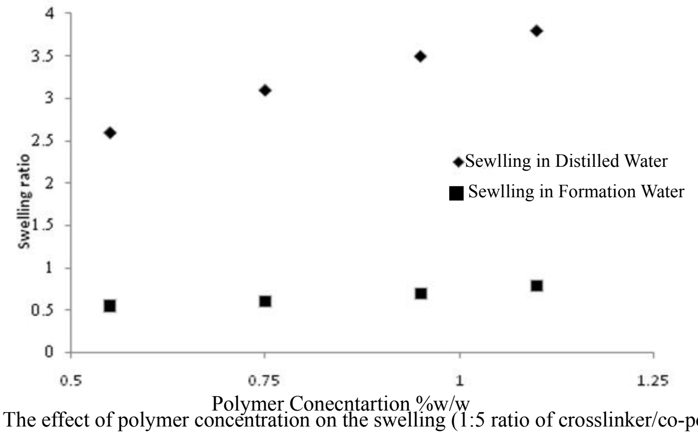

Figure 4. The effect of polymer concentration on the swelling (1:5 ratio of crosslinker/co-polymer).

Table 2. Salt sensitivity factor in distilled and formation water

Sample No.	Polymer Concentration and Crosslinker to Polymer Weight Ratio	f (Sensitivity Factor)
1	0.95 % (w/w), 1:2	0.755474
2	0.95 % (w/w), 1:5	0.8
3	0.95 % (w/w), 1:10	0.813953
4	0.95 % (w/w), 1:20	0.791667
5	0..55 % (w/w), 1:5	0.788462
6	0.75 % (w/w), 1:5	0.806452
7	1.1 % (w/w), 1:5	0.8

The salt sensitivity factor (f) showed high sensitivity (about 80%) of gels in the presence of electrolytes (Table 2). The results confirmed the results of the previous workers showing the reduction of gel swelling in electrolyte solution [16, 24].

The ratio of gel swelling was considerably reduced in formation water since the ionic osmotic pressure would be reduced between electrolyte solvent and the gel network [16]. Generally, the swelling ratio of polyelectrolyte gels depends on the association state of the ionic groups within the polymer and the affinity of the hydrogel for water [25].

Increase of NaCl and $CaCl_2$ concentration caused increase of osmotic pressure of external solution, so that the ionic osmotic pressure between gel and the surrounded solution decreased. Furthermore, this result is also due to the complexing ability of the anionic sites of the chains and the divalent cation (Ca^{2+}), leading to decreased osmotic pressure of the ionic network and an increased degree of ionic crosslinking, which results in loss of swelling [16, 26].

Dynamic Coreflood Experiments

Test 1

The purpose of this test carried out by consecutive injection of oil and water is to study the different behavior of gel with respect to oil and water. Results of test 1 are shown in Tables 3.The results of bottle test showed that the gelation time of this system (a gel with the Sydansk's gel strength code about H code) is about 1.5 days [23, 27]. However, coreflood was kept at shut-in state for 6 days to assure complete gelation.

Table 3. Conditions and results of the first coreflood test

Sand Packed Characterization	$K_{absolute}$=253 mD	$K_{rw@Sor}$=0.51	$K_{ro@Scw}$=0.94	Porosity=34%
First water injection after gel treatment, Q=250 cc/hr				
Results	ΔP=16 bar K_{rw} after 6 days=0.005 Time=2 hr	ΔP=15.7 bar K_{rw} after 9 days=0.005 Time=2 hr		F_{rrw} after 9 days=93
First oil injection after gel treatment, Q=250 cc/hr				
Results	ΔP=3.1 bar	K_{ro} after 9 days=0.12	Time=1.5 hr	F_{rro}=7.8
Second water injection after gel treatment, Q=250 cc/hr				
Results	ΔP=11.2 bar	K_{rw} after 9 days=0.0078	Time=2 hr	F_{rrw}=65.38
Second oil injection after gel treatment, Q=250 cc/hr				
Results	ΔP=1.8 bar	K_{ro} after 9 days=0.21	Time=2 hr	F_{rro}=4.5
Third water injection after gel treatment, Q=250 cc/hr				
Results	ΔP=12 bar	K_{rw} after 9 days=0.0073	Time=3 hr	F_{rrw}=70

As indicated in Table 3, after 6 days of gel placement in the sand packed, tap water was injected into the sand packed to increase pressure difference up to 16 bar, indicating that the sealant gel acted like a physical barrier and prevented the water entry into the porous media. In fact, an additional resistance was formed against the water flow due to the plugging of pore spaces, which caused reducing of water relative permeability to 0.005. The sand packed was kept at 90°C in an oven for 3 days. After 9 days, water relative permeability was about 0.005, like previous step. Up to this step, water residual resistance factor (F_{rrw}) was about 93. This shows that the final strength of the gel was achieved during 6 days, which confirms the rheological results [23].

Subsequently, oil was injected into the sand packed and the pressure difference was measured. The pressure difference and F_{rro} were about 3.1 and 7.8, respectively. In order to measure the pressure difference and the residual resistance factor, water, oil and again water were consecutively injected. As shown in Table 3, the gel has shown different kinds of behavior towards oil and water [28]. The procedure can be explained as follows: The gel was swollen in face of water. However, the swollen gel was dehydrated when oil was injected. The results also showed that oil permeability increased as the injection time increased, which is similar to the results presented by Seright [19]. Thus, by the second oil injection, the relative permeability of Frro and oil were about 4.5 and 0.21, respectively. It seems that by passing water through porous media, gel can find its way. Therefore, when oil passes through the porous media, it flows through the paths opened by the water.

Test 2

The purpose of this test is to study the effect of oil and water rate on the permeability of sand (gel efficiency). Table 4 shows the results of the second test. The injected gel sand packed was kept at 90°C for 6 days. During the first step, tap water was injected at 250 cc/hr flow rate after 6 days. Therefore, the relative permeability of water and F_{rrw} were measured to be about 0.0095 and 101, respectively. Then tap water was injected at different rates into the sand packed. As shown in Table 4, F_{rrw} increased with the decrease of water injection rate. It seemed that by increasing the injection rate, water can pass the gel through more paths [22]. In the next step, the system was kept at 90°C and atmospheric pressure for one day in order to let the available water in the sand packed evaporate. The results showed this had negligible effect on Frrw so that F_{rrw} was about 96 for second water injection. Then oil was injected into the sand packed in different rates. At first, at an injection rate of 250 cc/hr, the pressure drop increased up to 7.2 bar. Then, the pressure difference of 4.4 bar was measured with decreased injection rate to 150 cc/hr. During the next step, the rate of injection was increased again to 250 cc/hr so that the pressure difference increased to 5.5 bar. The explanation is that in the first step of oil injection, there was still some water remaining in the system so that the pressure drop was more than that in the second step. The results also indicated the pressure drop remained approximately constant by increased injection rate, which can be explained by gel dehydration during the injection [22]. After dehydration, the gel was not swollen against the oil. Consequently, as shown in Table 4, the pressure difference was about 5.5 bar at the second oil injection (250 cc/hr). Then, water was injected in the third step. The results indicated that oil presence in the sand packed had no effect on the efficiency of co-polymer gel similar to the first test, which can be ignored, leading to the reduction of F_{rrw} from 96 to 86.5, due to decreasing gel swelling.

Table 4. Conditions and results of the second coreflood test

Sand Packed Characterization	$K_{absolute}$=111 mD	$K_{rw@Sor}$=0.96	$K_{ro@Scw}$=0.83	Porosity=40%
First water injection after gel treatment				
Q, cc/hr	ΔP, bar	K_{rw}	F_{rrw}=101	
250	20.9	0.0095		
150	17.8	0.0067		
250	19.5	0.0102		
350	20.5	0.0136		
450	20.4	0.0176		
250	20.9	0.0095		
Second water injection after one day at Q= 250 cc/hr				
250	20	K_{rw}= 0.01	F_{rrw}=96	
First oil injection after second water injection				
Q, cc/hr	ΔP, bar	K_{ro}	F_{rro}=5.3	
250	7.2	13.19		
150	4.4	12.95		
250	5.5	17.26		
350	5.9	22.53		
450	5.9	28.96		
500	5.7	33.31		
Third water injection after first oil injection at Q= 250 cc/hr				
ΔP= 18 bar	K_{rw}= 0.0111		F_{rrw}=86.5	

Test 3

The purpose of this test is to study the effect of gel swelling on DPR. In this test, instead of tap water, formation water was injected into the sand packed after 6 days at different rates and the pressure drop was recorded. The results are presented in Table 5. Comparison of the results of the second and third tests showed that Frrw decreased by about 67% in the third test, which can be attributed to the lower gel swelling in the presence of formation water. The system was then kept at 90°C for one day in order to let the available water in the sand packed to evaporate. The results showed that the pressure drop was lower than the first step of formation water injection because of lower gel swelling in face of formation water. As shown in the Table 5, F_{rrw} was decreased by about 26%, which was due to the low gel swelling in the presence of formation water.

During the next step, oil was injected into the sand packed at different rates. It seems that when the system is at higher temperatures (e.g. 90°C) the water evaporates from the sand packed and gel, leaving salty precipitation.

This phenomenon leads to blockage of the oil paths, which causes the increase of pressure difference. Therefore, as shown in Table 5, Frro was higher in this test than the second test. Then, the sand packed was kept for 24 hours below 90°C and the atmospheric pressure, followed by oil injection into the sand packed. As shown in Table 5, Frro increased due to evaporation of the available water in the system, leaving salty sediments in the system, which obstructed the paths of these sediments. As a result, oil permeability was decreased.

Table 6 gives the literature values reported for the permeability reduction of various acrylamides crosslinked with various crosslinkers [29-33]. As shown, prepared polymer gels show not only different natures in face of oil and water, but they also show the capacity to be suitable in the oil reservoirs for water shut-off treatment.

Mechanism for DPR

When water was injected into the sand packed, oil was trapped in some pores and the injected water was absorbed by the gel. The gel then swelled. The sand permeability toward water decreased due to the low inherent permeability of polymer gel toward water, while oil formed a relatively small flow channel or “wormhole” through the gel during the oil injection so that oil permeability increased. Even low pressure gradients, oil forced the pathways by destroying or dehydrating the gel. Gel dehydration is a process of removing water from the gel by imposing a pressure gradient on the gel [34]. During subsequent water injection, the pathways could partially close when the gel re-hydrates, but not as much as the previous injection. Therefore, water permeability reduction is less than the previous injection.

Conclusions

A series of swelling and coreflood experiments were performed as a function of various key parameters. The following conclusions were made:

1. Increase of co-polymer concentration caused an increase in gel swelling, but an increase in crosslinker concentration caused a decrease in gel swelling.
2. Salt sensitivity factor (f) of the gels was higher (about 80%) for the electrolytic media.

Table 5. Conditions and results of the third coreflood test

Sand Packed Characterizations	$K_{absolute}$=250 mD	$K_{rw@Sor}$=0.5	$K_{ro@Scw}$=0.92	Porosity=35%
First water injection after gel treatment				
Q, cc/hr	ΔP, bar	krw	F_{rrw}=51.5	
250	4.8	0.018		
150	3.6	0.014		
250	4.4	0.02		
350	5.6	0.022		
450	6.2	0.025		
Second water injection after one day				
Q, cc/hr	ΔP, bar	krw	F_{rrw}=20	
250	3.6	0.024		
150	2.8	0.019		
250	3.6	0.025		
350	4.2	0.029		
450	5.1	0.031		
First oil injection				
Q, cc/hr	ΔP, bar	kro	F_{rro}=9.2	
150	2.6	0.087		
250	3.9	0.097		
350	4.8	0.11		
450	5.6	0.122		
Second oil injection after one day				
Q, cc/hr	ΔP, bar	kro	F_{rro}=13.14	
150	3.2	0.071		
250	4.8	0.079		
350	5.9	0.09		
450	6.2	0.11		

Table 6. Permeability reduction of acrylamide based polymers reacted with different crosslinkers

Gelling System	F_{rrw}	F_{rro}
HPAM + Organic Crosslinker[25]	200	20
PDVSA gel system[26]	118-329	7-135
HPAM + Chromium acetate[27]	706-17600	4.8-17.9
Colloidal dispersion gels (CDG)[28]	8.1	1.6
PAtBA+PEI[29]	18-20	----
Present Work, Sulfonated polyacrylamide + Chromium acetate	50-100	4-10

3. The effect of the gel treatment increased with decreasing the water injection rate.
4. During the oil injection, polymer gel was dehydrated and the oil relative permeability increased.
5. Formation water decreased the water residual resistance factor by about 67%.
6. According to the results, Disproportionate Permeability Reduction (DPR) was the phenomena due to the gel swelling versus injected water and dehydration versus injected oil.

Nomenclature

DPR= Disproportionate Permeability Reduction
PAMPS= Sulfonated Polyacrylamide
ESR= Equilibrium Swelling Ratio
f= Sensitivity Factor
F_{rr}= Residual Resistance Factor
S_{cw}= Connate Water Saturation
S_{or}= Residual Oil Saturation
K_{rw}= Water Relative Permeability
K_{ro}= Oil relative Permeability

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