

Polyacrylamide Gel Polymer as Water Shut-off System: Preparation and Investigation of Physical and Chemical Properties in One of the Iranian Oil Reservoirs Conditions

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ABSTRACT: *In this paper, polyacrylamide/chromium acetate gel polymer system was prepared as water shut-off system to control water production in one of the Iranian oil reservoirs condition. Effect of various parameters such as temperature (90 °C) and water formation from view point of salinity and divalent cations, was evaluated on the gelation kinetic and gel strength using bottle testing method. The results showed that the increase of temperature caused a decrease in the gelation time. Also, presence of divalent cations in formation water, decreased gelation time and by precipitation could cause instability in gel structure.*

KEY WORDS: *Water shut-off, Gel polymer, Bottle testing method, Reservoir condition.*

INTRODUCTION

Unwanted water production in association with crude oil is one of the major production difficulties for the petroleum industry, as more reservoirs become mature. Costs of lifting, handling, separation and disposal of large amounts of produced water; environmental concerns about this water; coning due to bottom water drive; large amounts of produced water from high permeability layers; increased corrosion rates; higher tendency for

emulsion; and, scale formation are among the main problems due to water production which often decrease the economic life of a well. Therefore, there is a need to reduce excessive water production [1,2].

Several mechanical and chemical methods are available for water shut-off treatments in the hydrocarbon wells. Mechanical methods, which include drilling horizontal, multi-lateral wells, placing a linear to block

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water production and down hole separation equipment such as hydro cyclones, usually require a work-over rig. These methods are often expensive. Various chemicals have been used as blocking and diverting agents to treat injection and production wells for more than six decades [2-4]. Several factors determine the success of a chemical treatment in the field, including: candidate selection, identification of the source water, proper choice of the chemical system, and placement of the chemicals into the target zone [2,3].

Today, gel polymer treatment is one of the most useful chemical methods to reduce water production. Gel polymer systems have a penetration property greater than the mechanical methods and cement to provide a deeper barrier against the excess water. Also, plugging due to gel polymers can be removed unlike physical cement plugging which leads to a permanent plug in the porous media so that the residual oil can not be produced from the treated region by cementing method after decreasing of excess water production [5].

Use of gelling systems for treatment of injection and production wells is widely practiced to improve production of oil and gas. Various gel polymer treatments were developed in the Arbuckle reservoir in central Kansas by *Willhite et al.* [6]. *Sydansk et al.* [7] have reported several field tests of gel treatment for controlling of water production in Wyoming's Big Horn Basin. Recently, several gel polymer water (and gas) shut-off treatments have been experimented in the Middle East region [8,9]. Very few attempts have been made to prepare gel polymer water shut-off systems for the Iranian oil (and gas) reservoir [10-12].

Gel polymer treatment can be used as an improved oil recovery (IOR) method in injection and production wells. In the injection well treatment, the gelant is placed into high permeability fractures. This application, called profile modification or conformance control, diverts injected water or gas to un-swept zones and improves the distribution of injected fluids into a heterogeneous reservoir. The production well treatment involves injection of the gelant into the fractures or high permeability zones that produces a lot of water, thereby reducing oil production. This application not only improves drawdown from the productive zones; but, it also reduces or eliminates the costs associated with the produced water [13-15].

Gel polymer systems are typically composed of a water soluble polymer and a crosslinking agent which are dissolved in water. This solution is considered as gelant solution. After allowing sufficient time, the gelant sets into a semisolid mass, and behaves as flow diverting or blocking agent. Selection of a gel polymer system for a given well treatment strongly depends on reservoir conditions such as temperature, salinity, hardness and the pH of the water used for preparation of the gelant. Other parameters to be considered for the proper selection of a given gel polymer system include salinity of the formation water, permeability of the target zone, and the lithology of the formation [16-19].

Several gelled polymer systems are available for water shut-off treatment. Most polymers are polyacrylamide with different degrees of hydrolysis (partially hydrolysed polyacrylamide) and polysaccharide such as xanthan biopolymer. These polymers can be cross linked with metallic and organic cross linkers to produce a three dimensional polymer structure of the gel [6, 7, 13, 14, 17, 20, 21].

Different methods are presented in the literature for the determination of gel polymer properties such as bottle testing method, sealed tube method, dynamic shear method (rheometer) and static shear method (viscometer) [18, 21, 22]. In this work, the bottle testing method was used to study the effect of various parameters on the physical and chemical properties of gel polymer.

Generally, the bottle testing method, as an experimental technique, provides a semi-quantitative measurement of gelation rate and gel strength. Also, it can be considered as a faster and inexpensive method to study gelation kinetic. In this method, which was defined by *Sydansk* [18], gel strength during development of gelation kinetic was expressed as an alphabetic code of A through I which is shown in table 1. According to this method, the gel strength codes were ranged from high flowing gels with barely any gel structure visibly detectable to rigid rubbery gels [16]. Therefore, the gelation time was considered in this work as the period of time when change is no longer observed in the gel strength code.

In the present work for each test, gelant solution was formulated and placed in the bottle at a specific temperature. The bottle was inverted during each reading time at different intervals and the gel property was recorded under the influence of gravity.

Finally, a gel strength code was allocated as defined in table 1. For example, code A was assigned to a sample, its final viscosity was the same as the original gelant viscosity. Likewise, a code of I indicated that there was no deformation on the gel surface upon inversion, and fluidity of the gel network was very low, while the final viscosity was much higher than the original gelant viscosity.

The present work aimed at experimentally testing a preparation of polyacrylamide gel polymer system which included partially hydrolyzed polyacrylamide as a water soluble polymer and chromium acetate as a metallic crosslinking agent. Each of the effects of various parameters such as polymer concentration, crosslinking agent concentration, salinity percent of water, pH of solution and different additives, were investigated independently on the physical and chemical properties of polyacrylamide gel polymer using bottle testing method. Finally, the performance of the polyacrylamide gel polymer as water shut-off system was investigated in one of the Iranian oil reservoirs condition considering high temperature and high salinity.

EXPERIMENTAL STUDIES

Materials

Partially hydrolyzed polyacrylamide was used as completely water soluble polymer which was synthesized by Iran Polymer and Petrochemical Institute (IPPI). Physical properties of the polymer are depicted in table 2

Chromium acetate was used as the crosslinking agent which was provided by Aldrich company. Sodium chloride, potassium chloride, calcium chloride and magnesium chloride were used as different additives which were provided by Merck. The chemicals which were used in the experiments are demonstrated in table 3. Water formation which was received from one of the Iranian south oil reservoirs was used as in situ fluid to evaluate gel polymer performance. Also, wash water was used as a solvent to prepare gelant solution which was provided from southern region of Iran. The properties of wash water are presented in table 4.

PREPARATION OF GELANT SOLUTION

Gelant solution was prepared by mixing of predetermined amounts of polyacrylamide solution and crosslinking agent at room temperature. The polymer

Table 1: Gel strength code.

Gel Strength Code	Gel Description
A	No detectable gel formed
B	Highly flowing gel
C	Flowing gel
D	Moderately flowing gel
E	Barely flowing gel
F	Highly deformable non flowing gel
G	Moderately deformable non flowing gel
H	Slightly deformable non flowing gel
I	Rigid gel

Table 2: polyacrylamide properties.

Molecular weight, (g/mol)	13×10^6
pH	8.3
Degree of hydrolysis	30%

Table 3: wash water properties.

Chloride content (in ppm)	3
pH	7.8
Turbidity (in NTU)	5

Table 4: Chemicals used in experiments.

Designation	Description	Source
PHPA	Partially Hydrolyzed Polyacrylamide	Iran Polymer and Petrochemical Institute
Cr(III)	Chromium Acetate	Aldrich
NaCl & KCl	As monovalent cation	Merck
CaCl ₂ & MgCl ₂	As divalent cation	Merck
Water formation	As in situ fluid	One of the Iranian oil reservoirs
Wash water	As solvent	South of Iran

solution was first diluted to the required concentration using wash water. The solution was continuously stirred on a magnetic stirrer until uniform viscous solution was obtained. Then, chromium acetate solution was prepared by adding a pre-weighted amount of chromium acetate powder into wash water. Afterward, polymer and crosslinking solution were mixed at a specific ratio. Then, the solution was stirred to form a homogeneous gelant

solution. In this work, the crosslinking reaction is initiated by heating the gelant solution up to 90 °C (reservoir temperature) in the air bath. Fig. 1 depicts a schematic representation of gel polymer network formation [23].

MEASUREMENT OF GEL POLYMER NETWORK PROPERTIES

In this study, increment of gelant solution viscosity and gel strength value during gelling reaction was considered as an indication of development of gelation kinetic. Initially, viscosity of gelant solution was very low and gel strength was the same as that of polymer solution strength; so that, any three-dimensional gel polymer network structure, was not detected. However, through aging at a specific temperature, the rate of gelling reaction can be increased. Therefore, when the crosslinking reaction took place in the gelant solution, it finally led to a three-dimensional gel polymer network structure. During this process, bottle testing method was used to study the effect of various parameters on the physical and chemical properties of PHPA/Cr(III) gel polymer.

RESULTS AND DISCUSSION

Effect of Polymer Concentration

The polymer concentration is critical for the structure and properties of the gel polymer network. Any type of gel system needs a minimum concentration of polymer which is called critical overlap concentration (COC). As indicated by the bottle testing results in table 5, the COC value is about 5000 ppm for the synthetic polymer. The gel strength code that is an indication of gel network formation was A after 150 hours aging at 90 °C, meaning that no detectable gel formed at 5000 ppm. The gel had the same viscosity (fluidity) as the original polymer solution and no gel was visually detectable. Therefore, no gel was formed below COC.

The results in table 5 show that gel polymer is formed over a broad range of polymer concentrations above COC. As can be seen from bottle testing results, gel strength increased by increment in polymer concentration; so that, the final gel strength range varied from E to I at the constant polymer to crosslinking agent ratio after 150 hours aging at 90 °C. In other words, the gel polymer bulk, upon inversion changes from a state of

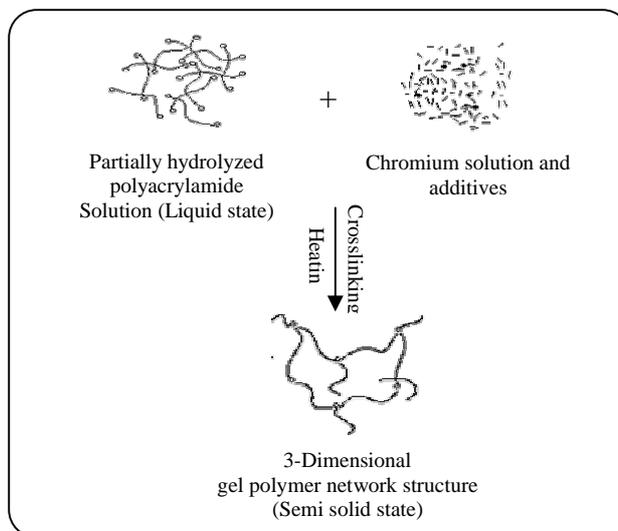


Fig. 1: A schematic representation of gel polymer network formation.

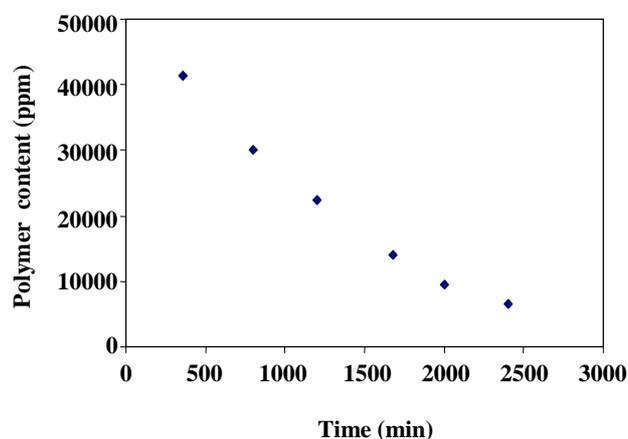


Fig. 2: Effect of polymer concentration on gelation time (Experimental condition: concentration of chromium content is 16.5 g/L, solvent is tap water, T= 30 °C).

barely flowing gel to one of rigid gel. Also, polymer concentration has a significant effect on the gelation time. Fig. 2 demonstrates the effect of polymer concentration on the gelation time. As can be observed, the required time to obtain a non-flowing gel polymer with a tolerable strength decreased when the polymer dosage was increased.

Effect of Crosslinking Concentration

Brosslinking of polyacrylamide solution by a crosslinking agent gives better physical properties (viscosity and gel strength) than the polymer solution that

Table 5: Experimental condition: weight ratio of PHPA: Cr(III) is 40:1, solvent is wash water, T= 90 °C).

Polymer Concentration (ppm)	5000	7500	10000	15000
Time(hr)	Gel code			
1	A	B	C	D
6	A	C	D	E
24	A	D	E	G
150	A	E	G	I

Table 6: Experimental condition: PHPA is 15000ppm, solvent is wash water, T= 90 °C.

Weight ratio of PHPA: Cr(III)	20:1	40:1	60:1	80:1
Time(hr)	Gel code			
5	F	E	C	B
45	-	G	E	C
70	-	H	F	D
100	-	H	G	E

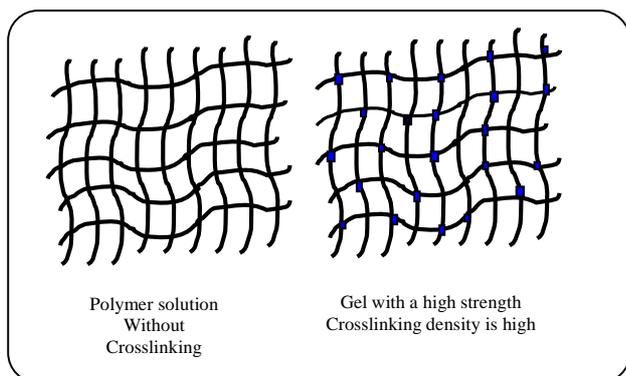


Fig. 3: Effect of crosslinking agent on gel strength.

facilitates physical plugging of porous media. Crosslinking agent is a multi-functional group which can build a complex network with carboxylate groups of polymer and form a 3-dimensional gel network structure.

In the polyacrylamide structure, amide group (-CONH₂) is converted to hydrophilic carboxylate group (-COOH) by hydrolysis reaction.

Crosslinking agent concentration has a significant effect on the gel strength. Fig. 3 depicts a schematic representation crosslinking agent effect.

After determination of critical overlap concentration of polymer, several samples were prepared to investigate the effect of crosslinking agent on the network strength. Bottle testing results shown in table 6 indicate that when the ratio of polymer to crosslinking agent was decreased from 80:1 to 40:1, gelation rate increased; so that, gel strength was promoted to G after 45 hours aging at 90 °C. In other words, when crosslinking agent concentration was increased, the gel polymer bulk changed from a state of flowing gel to one of deformable non-flowing gel.

Increase of crosslinking concentration only up to a point promotes gel strength because when level of crosslinking agent increased from 40:1 to 20:1, final gel strength dramatically decreased during gel formation at 90 °C. In spite of the fact that the gel polymer network with the ratio 1:20 had a significant strength and the gelation rate was faster than other samples, it was subsequently destroyed after 5 hours during gelation reaction. In explaining this phenomenon, it can be said that it is related to phase stability, which refers to the ability of a gel network to resist syneresis. Generally, syneresis is a collapse of the gel network which is characterized by a loss of inherent adhesion of gel system, reduction of gel volume and expulsion of water from gel structure [13,18].

These states are usually reported as instability parameters of gel polymer network. At this point it can be said that the main cause of instability obtained, was related to the significant increment of crosslinking density and decrease of molecular weight between two polymer segments in the gel network. Therefore, due to reduced space between two polymer connections in the network the capacity of water keeping decreased, causing water to be removed from the structure leading to gel shrinkage. Fig. 4 depicts the instability of gel polymer network due to excess crosslinking agent content in the gel network which leads to decline in the final gel strength.

Effect of Ionic Strength

In this section of the experiment, several samples were prepared with a constant content of polymer and crosslinking agent. The samples properties are shown in table 7.

The results from bottle testing experiments showed

Table 7: Experimental condition (Solvent is wash water, T= 90 °C).

PHPA (ppm)	PHPA: Cr(III) (wt%)	Additive
12500	40:1	Reference sample
12500	40:1	NaCl (25 g/l)
12500	40:1	KCl (25 g/l)
12500	40:1	CaCl ₂ (25 g/l)
12500	40:1	MgCl ₂ (25 g/l)
12500	40:1	HCl (pH= 4)
12500	40:1	Toluene (125 g/l)

that gelation time can be decreased somewhat by increasing ionic strength of Na⁺ and K⁺. Table 8 shows the influence of salinity on the property of gel polymer network using strength code. The gel strength code is about F (highly deformable, non-flowing gel) while reference sample strength code is about E (barely flowing gel) after 2.5 hours aging at 90 °C. But, when gel was matured by developing of crosslinking reaction, the strength value of the samples was same for both conditions in comparison to the reference sample. The strength of samples was about H (slightly deformable, non flowing gel) after 120 hours aging at 90 °C.

Table 8 describes the effects of Ca²⁺ and Mg²⁺ on the gelation rate and gel strength. It was observed that percent hydrolysis of polyacrylamide can be considered as a key factor for dissolving of polymer in the solvent which contains divalent cations. For the polymer which was used in the experiment, when the solvent used for diluting the polymer contained Ca²⁺, the polymer could not be properly dissolved; so that, it precipitated after addition of the solvent to it. Also, when crosslinking solution was added to the polymer solution, which contains Mg²⁺, gelling reaction instantaneously took place at room temperature. In addition, the network strength dramatically decreased after a short aging at 90 °C; so that, the gel structure was destroyed after 2.5 hours aging at 90 °C.

Also, divalent cations have a negative effect on gel properties especially when reservoir temperature is high. It seems that in high temperature condition acrylamide groups from the polymer structure can be thermally hydrolyzed into carboxylate groups, and this conversion



Fig. 4: Effect of excess crosslinking concentration on gel polymer network strength, leading to phase stability (Vertical sample) and phase instability (Horizontal sample).

produces additional crosslinking sites that interact with divalent cations that are typically present in water formation. Therefore, excessive crosslinking can occur between additionally formed negative sites with the divalent cations.

The process of excessive crosslinking results in expulsion of water from the gel structure and significant shrinkage of gel volume. This phenomenon is also referred to as gel syneresis which is considered as instability of gel network. From the molecular point of view, the main cause of syneresis could be related to noticeable increase in the crosslinking density and decrease in the space between two crosslinked segments for water keeping in the gel network.

Fig. 5 depicts syneresis effect due to divalent cations in a gel polymer network with polymer content of 12500 ppm and polymer to crosslinking agent ratio of 40:1. The sample in the right side includes a solvent which contains divalent cations and the sample to the left does not have any divalent cations. In both samples, gelant solution was aged at 90 °C.

Effect of pH and Organic Material

Result from bottle testing demonstrated that the PHPA/Cr(III) gel polymer system used in the present study can be formed in acidic pH which is in contrast to conventional gel polymer systems such as chrome/ redox/ PHPA gel. As indicated in table 8, gel strength was relatively reduced in the acidic pH after 120 hours aging at 90 °C. However, it is preferable the PHPA/Cr(III) gelation reaction was performed in alkaline pH [18].

Table 8: Experimental condition: PHPA 12500 ppm, (weight ratio of PHPA to Cr(III) is 40:1, solvent is wash water, T= 90 °C).

Additives	Reference	NaCl	KCl	CaCl ₂	MgCl ₂	HCl	Toluene
Time(hr)	Gel code						
2.5	E	F	F	-	F	E	D
45	F	G	G	-	D	F	E
120	H	H	H	-	-	D	F
150	H	H	H	-	-	-	E

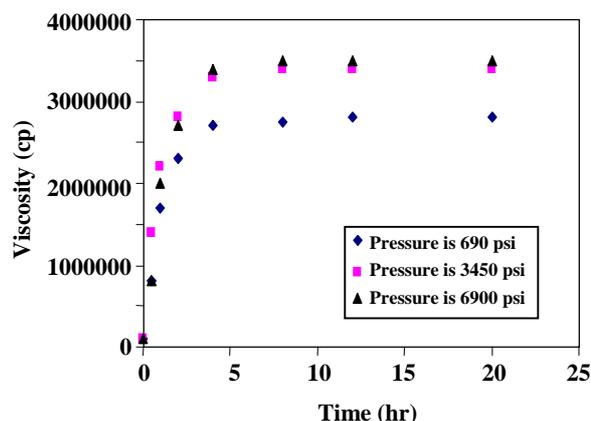
**Fig. 5: Effect of divalent cations on gel property, Leading to high degree of syneresis (right) and no syneresis (left).**

With respect to the effect of organic material on the final gel polymer strength, it seems that organic material does not have significant effect on the gel strength; so that, after 150 hours aging at 90 °C, the sample has the same strength as the reference sample.

Effect of Pressure

Generally, pressure does not have a significant effect on the gelation kinetic. Sydansk [18] has reported that gelation rate is a weak function of pressure and increases slightly with pressure. Fig. 6 depicts the effect of pressure on the gelation kinetic. As can be observed, gelation time is approximately 5 hours for all samples. Note that in Fig. 6 the final viscosity is fairly insensitive to pressure. When pressure was increased by a factor of 10, final viscosity was increased by a factor of only 1.25.

In the present study, upper limit of pressure was about 4000psi. Therefore, pressure effect is negligible in comparison to the effect of other parameters such as temperature, gelant composition.

**Fig. 6: Effect of pressure on gelation kinetic, Experimental condition: hydrolysis percent <1%, polymer molecular weight 11000000, polymer concentration 20000 ppm, polymer to crosslinking agent ratio 20:1, T= 60 °C, solvent is tap water.**

INVESTIGATION OF GEL POLYMER BEHAVIOR IN THE RESERVOIR CONDITION

In this section of the experiments, behavior of gel polymer was investigated in one of the Iranian oil reservoirs condition considering reservoir temperature and water formation. Various samples with polymer content of 12500 ppm, and ratio of polymer to crosslinking agent of 40:1, were prepared in the wash water. Next, water formation as in situ fluid was added to it. Composition and properties of water formation is presented in table 9.

Gelant solution with a low viscosity was aged at reservoir temperature of about 91 °C. Also, some pictures were taken at different periods of time to provide a good analysis about gel polymer behavior in the reservoir condition during the gelling reaction process. Fig. 7 shows the samples which were initially aged at the ambient temperature for 15 hours. The First sample, from left to right, was considered as a reference sample and the other samples were placed in water formation media as in situ fluid.

Table 9: Composition and properties of water formation (All concentrations were expressed in mg/L. Density=1.145 g/cm³ at 30 °C).

Parameter	Concentration
Sodium	54200
Calcium	8800
Magnesium	1700
Sulfate	1440
Chloride	105615
Carbonate	0
Bicarbonate	390
Hydroxide	0
Total dissolved solids	170500
pH	6.8

As can be observed, the gel strength code for all gelant samples is about G in comparison to the reference sample, in which the gel strength code is about D, after 15 hours aging at ambient temperature. Strength differences among samples were also noticeable based on differences in bulk viscosity between the reference sample and the other samples. Therefore, the effect of water formation, as an in situ reservoir fluid could be easily recognized on gel characteristics during the aging process at low temperature condition which is related to the quality of water formation. In addition to crosslinking reactions that occur between polymer itself and chromium acetate, divalent cations such as Ca⁺² and Mg⁺² usually found in the water formation are crosslinked with negative sites which led to excessive crosslinking. Therefore the strength of gel polymer network was dramatically decreased.

Fig. 8 depicts the samples that were initially placed for 15 hours at ambient temperature and then aged at 90 °C for 6 hours. As can be seen, gel strength of the reference sample (sample at extreme right) increased significantly after aging at reservoir temperature. The range of strength code of the samples was similar.

Therefore, temperature and the quality of water formation, as effective environmental parameters, can have a significant influence on gel polymer properties, especially on the gelation rate, gel strength and degree of syneresis.

It seems that if the harsh environmental conditions are

moderated as much as possible, gel polymer properties improved noticeably. For example the salinity and temperature of the treatment zone could be reduced by several wash water preflushes. This idea was investigated through the bottle testing method. Thus, gelant solution was placed in various media with different salinity concentrations. Then, gel stability was investigated during the course of the experiment. This is shown in Fig. 9. In the assessment of media salinity on gel stability, experimental condition was determined as follows: polymer concentration was 125000 ppm, polymer to crosslinking agent ratio was 40:1, T= 90 °C and wash water was used as a solvent. After preparation of gelant, water as a media was added to the solution with different salinity concentration.

As revealed from Figure 9, gelation rate of sample "a", which only includes water formation, was faster than that of the others. On the contrary, gel stability for this sample was lower than that of the others. But, if salinity was moderated by dilution of water formation by wash water, as was depicted of samples "b" and "c", gel stability could be improved more than sample "a". Therefore, gelation rate can be properly controlled during gel injection process in order to prevent unexpected plugging by adjustment of environmental parameters. Consequently, it is preferable the preflush is done in the treatment zone before any gel polymer treatment.

CONCLUSIONS

In this work, polyacrylamide/chromium(III) gel polymer as water shut-off system was prepared using bottle testing method for one of the Iranian oil reservoirs conditions considering reservoir temperature and water formation. Also, the effects of various additives were investigated on gel polymer properties. A summary of the results is presented as follows:

- 1) Gelation time is a controllable parameter from a few minutes to several days which depends on the gel composition and environmental condition.
- 2) The minimum concentration of polymer needed to form a three-dimensional gel network was about 5000 ppm for the synthetic polyacrylamide, with molecular weight 13,000,000 and degree of hydrolysis 30%.
- 3) As weight percent of polymer increased in the gelant solution, the gel strength increased and gelation time decreased.
- 4) Increase of crosslinking concentration only up to a



Fig. 7: Effect of water formation as an in situ reservoir fluid on gel polymer properties after 15 hours aging at ambient temperature.



Fig. 8: Effect of water formation on gel polymer properties after 6 hours aging at the reservoir temperature.



Fig. 9: Effect of media salinity on gel stability From left to right, sample (a): water formation (100 wt %), sample (b): water formation and wash water (50:50 wt %), sample (c): water formation and wash water (30:70 wt %), sample (d): wash water (100 wt %), sample (e): reference sample.

point promotes gel strength; so that, when the ratio of crosslinking agent to polymer was increased from 1:40 to 1:20, final gel strength was dramatically decreased which was considered as excessive crosslinking.

5) Gelation time is decreased somewhat due to increases in the ionic strength by sodium and potassium salts.

6) Divalent cations such Ca^{+2} and Mg^{+2} have a significantly negative effect on gel strength, especially when reservoir temperature is high.

7) Polyacrylamide gel polymer formed in the acidic condition. However, it is preferable to do the PHPA/Cr(III) gelation reaction in the alkaline pH

8) It seems that organic materials somewhat retard gelation kinetic but gel strength remained at a considerable level in comparison to the reference sample after aging at testing temperature.

9) Temperature and quality of water formation as effective environmental parameters can have significant influence on the gel polymer properties, especially on the gelation rate, gel strength and degree of syneresis.

10) It seems that if the harsh environmental condition was moderated as much as possible, gel polymer properties could improve noticeably.

11) In order to prevent unexpected plugging, gelation rate can be properly controlled during gel injection process by adjustment of environmental parameters.

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