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Experimental and Theoretical Investigation of Gelation Time of Nanostructured Polymer Gels by Central Composite Approach

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Abstract

Currently available polymers as a component of in-situ gels are unsuitable for treating high-temperature/high-salinity reservoirs due to their chemical and thermal degradation. In this study, a new copolymer-based gel system including high molecular weight nanostructured polymers (NSPs) was developed to address the excessive water production problem in reservoirs under harsh conditions. The stability of conventional polymer systems and NSPs was investigated under conditions of 40 days aging at 87000 ppm salinity and 90 °C. Then, gelation time optimization of gel systems composed of NSPs and chromium (III) acetate was performed with regards to the effect of copolymer concentration and copolymer/cross-linker ratio and their interactions during the gelation time. The central composite approach was used to design experiments and build a mathematical model. The analysis of variance (ANOVA) was used to estimate the deviation of the model predictions from the data. The results of stability analysis demonstrated the advantages of NSPs over conventional polymers by a viscosity reduction of 69, 36, and 18% for Flopaam3310, AN105, and NSPs respectively. The model developed for the prediction of gelation time of NSPs gel was significant at a confidence level of 98.6% against the test data. Moreover, it was found that gelation time became longer with a decrease in copolymer concentrations and/or increase in copolymer/cross-linker ratio.

Keywords: Central Composite Approach, Gelation Time, Polymer Stability, Nanostructured Polymers, Water Shutoff

1. Introduction

One of the major problems in mature oil fields is excessive water production that leads to reduced oil production, corrosion, and sand production, while extra facilities are also required to remove the excess produced water (Al-Muntasheri et al., 2008; Jayakumar et al., 2013; Zhao et al., 2015; Alhashim et al., 2018). A variety of chemical techniques are commonly used as water shutoff methods to reduce the excessive water production (Pham and Hatzignatiou, 2016). Polymer gel treatment is currently the most important chemical water shutoff method (Sengupta et al., 2012). The polymer gel system is based on a

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mixture of polymer and a cross-linker mixed to form a gellant that is injected into the reservoir, where it forms a three-dimensional structure by performing a crosslinking reaction to act as a barrier against the water flow (ElKarsani et al., 2015). Synthetic polymers such as polyacrylamides have become the most commonly used ones for conformance control (Prud'homme et al., 1983). Different cross-linking agents have been used for in-situ gel applications that are generally classified into inorganic and organic types. The most widely used inorganic cross-linker is chromium (III) acetate (Hardy et al., 1999).

Gelation characteristics such as gelation time and gel stability are important parameters involved in water shutoff operations (Bia et al., 2015). An extremely important factor in in-situ gels is the time it takes for polymer-containing formulation to form gel (Al-Anazi et al., 2011). One of the disadvantages of in-situ gel is that its application is limited to near wellbore areas due to its short gelation time. In high-temperature reservoirs, the rapid formation of the gel may prevent the uniform distribution of the gelling fluid prior to gelation. It is even critical to prevent gelation inside the wellbore. A successful polymer gel process typically requires the gellant to be placed deeply into the reservoir in order to divert the water path and to block high permeable zones in desirable areas. Accordingly, there is a need to test the gelation time of the gelling fluid in order to form a gel in a satisfactory time frame. Another problem with in-situ polymer gels is instability of gel bulk due to thermal and chemical degradation of polymers especially in high-temperature/high-salinity reservoirs. By using highly thermal-resistant and highly ion-tolerant polymers in the gel structure, the stability of in-situ gel bulk is preserved in reservoirs under harsh conditions. Presently, the NSPs are being used for enhanced oil recovery (EOR) applications because of many merits such as being size-controlled, highly swelling, tolerant to temperature and salinity, and environmentally friendly (Feng et al., 2003; James et al., 2003; Frampton et al., 2004; Cozic et al., 2008 Yao et al., 2013; Li et al., 2014). Based on the properties of NSPs, it seems that they are able to resolve the common problem of in-situ gels. However, the applicability of NSPs in the water shutoff process has not been clearly investigated, and more research is crucial for that aim. The response surface methodology based on central composite design is one of the strongest mathematical and statistical methods which can be used to design experiments, build empirical models, evaluate the effects of independent variables, and predict targeted responses (Badday et al., 2013; Danmaliki et al., 2017; Hosseini et al., 2017). In this research, a central composite design was employed to investigate the effects of copolymer concentration and copolymer/cross-linker ratio on the gelation time of NSPs gel. Briefly, the main objectives of this study are to first investigate the stability of NSPs compared to that of conventional polymers under harsh conditions and then determine the gelation time of NSPs gel as a function of copolymer concentration and copolymer/cross-linker ratio.

Materials and methods

1.1. Materials

a. Brine

Sodium chloride, calcium chloride, magnesium chloride, sodium bicarbonate, and sodium sulfate used as additives for brine preparations were supplied by Merck Co. and were used as received without further purification. The chemical composition of synthetic brine is listed in Table 1.

Table 1

The composition of the synthetic brine as the gellant solvent.

Additive	NaCl	$MgCl_2$	$CaCl_2$	$NaHCO_3$	Na_2SO_4	Total dissolves solid
Concentration (ppm)	69900	3325	12460	270	1065	87000

b. Polymers

In order to perform the stability analysis, three different high molecular weight polymers were used. Partially hydrolyzed polymers used were Flopaam 3330S and AN105 supplied by SNF Floerger, and polymeric nanoparticles, including copolymer of acrylamide (AAm) and 2-acrylamido-2-methyl-1-propane sulfonic acid (AMPS) (chemical formula: $C_{10}H_{18}N_2SO_5$), were synthesized in our laboratory by inverse emulsion polymerization and used as high molecular weight NSPs. Properties of the used polymer are presented in Table 2.

Table 2

Properties of polymer used in this research.

Polymer	Physical form	Monomer in the structure	Molecular weight (mDa)	Particle size	Morphology
NSPs	White powder	AAM, AMPS	5.75	43.5–81.5 nm	Spherical
AN105	White powder	AAM, AMPS	6	0.5–2 mm	Irregular shape
Flopaam3330	White powder	AAm	8	0.5–2 mm	Irregular shape

c. Cross-linker

Green crystalline powder of chromium (III) acetate supplied by Carlo Erba Co. was used as a metallic cross-linker for gel preparation.

1.2. Stability analysis

Polymer stability is one of the important parameters in water shutoff projects by in-situ gel treatment, and it is defined as the ability of polymer to maintain its viscosity with the passage of time. In order to investigate the stability of different polymers under simulated reservoir conditions, i.e. at 90 °C and salinity of 87000 ppm, prepared polymer solutions composed of 6000 ppm of each polymer dissolved at the synthetic brine were distributed into glass bottles and then sealed; they were then placed in an oven and aged at 90 °C. At consecutive time intervals, the solutions were taken out for gel viscosity monitoring. The viscosity of the gellant solutions were measured using a DV-II Pro Brookfield viscometer (Brookfield Engineering Laboratories, Inc., Middleboro, MA) at 90 °C and a constant shear rate of 7.3 s^{-1}.

1.3. Gellant preparations

In order for gellant preparations to be completed, specified amounts of polymeric materials were dissolved in the synthetic brine at room temperature and continuously stirred with a magnetic stirrer until a uniform viscous solution was obtained. At the next step, chromium (III) acetate was mixed with the synthetic brine and then added to the polymer solutions. The solutions were allowed to stir until a homogeneous gellant solution was formed. Gellant solutions with different formulations were prepared for gelation studies.

1.4. Bottle test

In this research, the bottle test method was used to evaluate gelation time over an extensive range of copolymer concentration and copolymer/cross-linker ratio. Generally, the bottle test method as an experimental technique provides a semi-quantitative measurement of gelation rate and gel strength.

Also, it can be considered as a fast and inexpensive method to study gelation kinetics (Al-Anazi et al., 2011). Bottle test was conducted at a temperature of 90 °C using laboratory bottles. The bottles were filled with gellant solutions, and the cross-linking process was monitored as a function of time, starting at the point when the gellant is put into the bottle. The bottles were next placed in an oven at a temperature of 90 °C and were inverted periodically in specific intervals to check the sample flow behavior under the influence of gravity, and each bottle was assigned a strength code as defined in Table 3. A schematic diagram of different gel strength codes is shown in Figure 1. In some cases, to ensure the validity of the results, gel preparations were repeated, and the bottle tests were performed again.

Table 3

Bottle test gel strength codes (Sydansk and Perry, 1987).

Gel strength code	Gel description
A	No detectable gel formed: The gel appears to have the same viscosity as the original polymer solution, and no gel is visually detectable.
B	Highly flowing gel: The gel appears to be only slightly more viscous than the initial polymer solution.
C	Flowing gel: Most of the obviously detectable gel flows to the bottle cap upon inversion.
D	Moderately flowing gel: A small portion (about 5 to 15%) of the gel does not readily flow to the bottle cap upon inversion.
E	Barely flowing gel: The gel slowly flows to the bottle cap and/or a significant portion (> 15%) of the gel does not flow upon inversion.
F	Highly deformable nonflowing gel: The gel does not flow to the bottle cap upon inversion, and gel does not flow enough to reach the bottle cap.
G	Moderately deformable nonflowing gel: The gel flows about halfway down the bottle upon inversion.
H	Slightly deformable nonflowing gel: Only the gel surface deforms slightly upon inversion.
I	Rigid gel: There is no gel-surface deformation upon inversion.

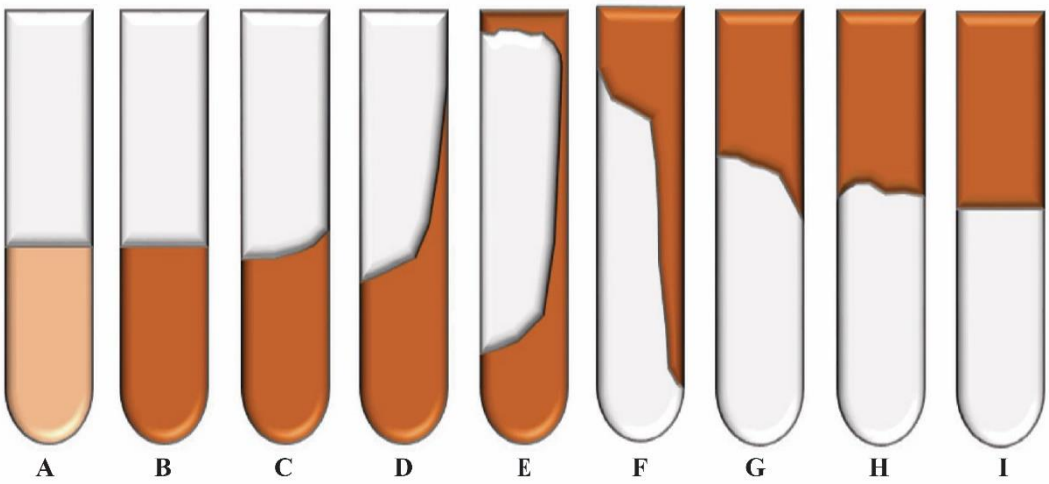

Figure 1

A schematic of different gel strength codes by bottle test method.

1.5. Central composite design

The central composite design is one of the most common designs used in the response surface measurement. In this work, the central composite design was used to evaluate the effects of copolymer

concentration and copolymer/cross-linker ratio on the gelation time of NSPs gel. Generally, in the central composite design method, the response surface is presented as quadratic equation as follows:

$$Y = \beta_0 + \sum_{i=1}^{4} \beta_i X_i + \sum_{i=1}^{4} \beta_{ii} X_i^2 + \sum_{i=1}^{3} \sum_{j=i-1}^{4} \beta_{ij} X_i X_j \quad (1)$$

where Y is the predicted response by the model (gelation time in hour), and X_i and X_j are the independent variables; β_0, β_i, β_{ii}, and β_{ij} represents the regression coefficients of the fitted model. In this research, the validity of the predicted model was verified by ANOVA, the quality of the second-order model was assessed by the determination coefficient (R^2); the analysis was also carried out with a Fisher's F-test, and the probability value was constructed around the mean or regression coefficients with 95% confidence limits or intervals. The whole experimental design, data analysis, and quadratic model building were accomplished using Design Expert software (Version 11.0.3, Stat-Ease Inc., Minneapolis, USA).

2. Results and discussion

2.1. Stability analysis

As mentioned above, the remaining viscosity at a high temperature represents a primary criterion for any chemicals to be used in reservoirs under harsh conditions. In this study, prepared polymer solutions composed of 6000 ppm of each polymer dissolved in 87000 ppm of the synthetic brine were aged at 90 °C, and viscosity measurement was performed in different time intervals. Figure 2 depicts the variation of polymer solution viscosity as a function of the aging time, and Figure 3 displays the final viscosity reduction percentage for different polymer solutions after a 40-day ageing time.

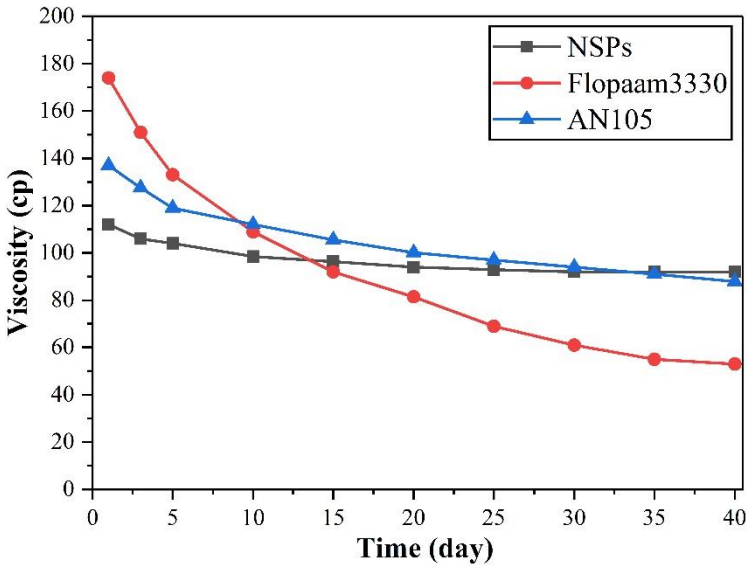

Figure 2

Viscosity evaluation with times for different polymers aged at 90 °C and 87000 ppm salinity.

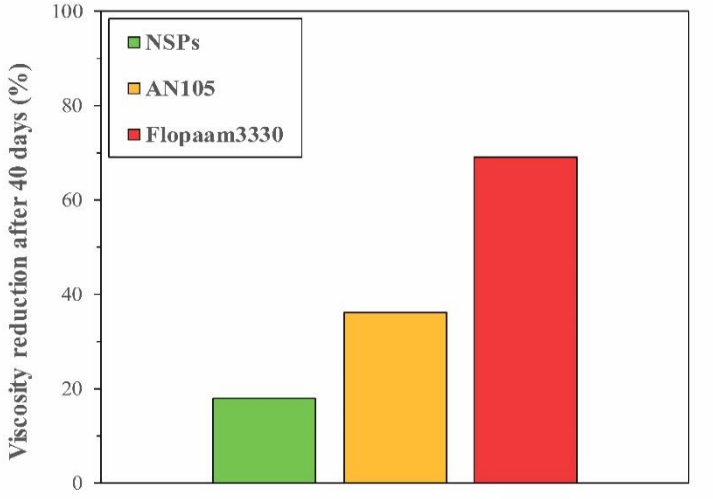

Figure 3

Viscosity reduction percentage for different polymer solutions after 40 days of ageing at 90 °C and at 87000 ppm salinity.

Based on the results, the levels of viscosity reduction of Flopaam3330, AN105, and NSPs after 40 days of aging under harsh conditions were about 69, 36, and 18% respectively. The considerable chemical degradation of polyacrylamide molecules in the presence of salt ions and also their thermal degradation are the reasons for a high viscosity reduction of Flopaam3330. The NSPs exhibited improved stability, and the existence of the sulfonate group in NSPs molecules hindered further viscosity reduction. Therefore, based on stability results, NSPs are applicable to reservoirs under harsh conditions for water shutoff application and were selected as an optimum case for further in-situ gel investigations.

2.2. Effects of copolymer concentration and copolymer/cross-linker ratio on gelation time

As mentioned above, based on the results of the stability analysis, NSPs were selected for gelation studies. A copolymer concentration in the range of 6000–12000 ppm and a reasonable range of cross-linker concentration was selected to study their effects on gelation time. The low level and high level of independent parameters were selected based on the results of the primary tests to ensure gel formation. The control variables used in the central composite design method were copolymer concentration (X_1) and copolymer/cross-linker ratio (X_2) while each factor was varied at five levels as presented in Table 4.

Table 4

The level of variables in the central composite design.

Variable	Low axial – α = 1.168	Low factorial (–1)	Center (0)	High factorial (+1)	High axial + α = 1.168
X_1: Copolymer concentration (ppm)	5500	6000	9000	12000	12500
X_2: Copolymer/cross-linker ratio	9.16	10	15	20	20.8

A set of nine gel systems was designed and prepared to determine their gelation times and to distinguish the interaction effects of copolymer concentration and copolymer/cross-linker ratio on gelation time utilizing the central composite design method. The gel experimental plans and the gelation times as their responses are presented in Table 5. Every gel system was aged at a temperature of 90 °C, and in different time intervals, their gelation state was visually observed. It is notable that in this research the time to

reach the G code corresponding to moderately deformable nonflowing gel was considered to be the gelation time.

Table 5

The design of experiments and results of the measured and predicted gelation time for different gel systems.

Gel system	X_1 (ppm)	X_2	Gelation time (hour)	
			Measured	Predicted
1	5500	15	81	86.2
2	6000	20	93	88.7
3	12000	10	5	10.99
4	9000	20.8	23	27.9
5	9000	9.16	12	8.9
6	12500	15	18	14.6
7	12000	20	11	12.9
8	6000	10	58	57.9
9	9000	15	16	16.9

The results of the bottle tests for different gel systems based on Sydansk's gel code is listed in Table 6. It should be noted that the stability of gels numbered 1, 2, 4, 6, 7, 8, and 9 were maintained during the whole time of the experiments without any degradation. However, in gels marked with numbers 3 and 5, some instability in the gel structure was observed, which may be due to the high degree of crosslinking reaction resulted in gel collapse. These results demonstrated the existence of some limitation in the amount of copolymer and cross-linker to form stable gels.

Table 6

Gel strength based on Sydansk's gel code for different NSPs gel systems at 90 °C and 87000 ppm salinity.

Time (hour)	Gel 1	Gel 2	Gel 3	Gel 4	Gel 5	Gel 6	Gel 7	Gel 8	Gel 9
1	A	A	C	B	B	B	B	A	B
2	B	A	D	D	D	C	D	B	C
4	B	B	F	E	F	E	F	C	D
16	C	B	G	F	G	F	G	D	G
24	D	C	G	G	G	G	G	E	G
36	E	D	H	G	H	G	G	F	G
48	F	E	H	H	H	H	H	F	H
72	F	F	H	H	I	H	H	G	H
89	G	F	I	H	I	H	I	G	I
96	H	G	I	I	Gel instability	I	I	H	I
120	I	H	Gel instability	I	–	I	I	I	I

In order to validate the results obtained through the bottle test method, gel viscosity measurement was performed with gel #2 using DV-II Pro Brookfield viscometer at a shear rate of 7.3 s^{-1} (Figure 4). The gelation time was considered as the time needed to reach the inflection point on the viscosity versus time curve. The gel viscosity measurement confirmed the gelation times observed by using the bottle

test method, and the difference in gelation times that were observed via the two methods was approximately 3.7%.

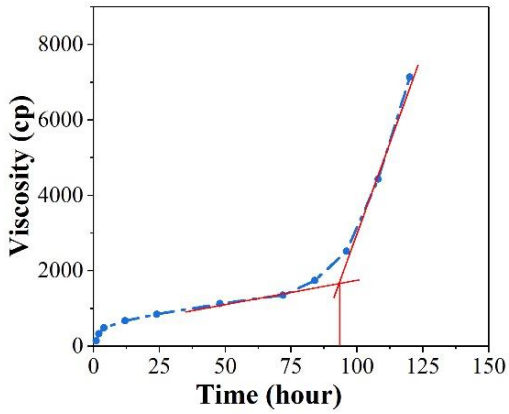

Figure 4

Variation in gel viscosity as a function of time (gel #2).

In order to develop a mathematical model that fits the results well, the results were fed into Design Expert software. Based on the analysis of the results, a second-order polynomial equation was obtained, representing the effect of two independent variables on the gelation time.

$$Y = 251.5874 - 0.0523X_1 + 4.5960X_2 - 0.00048X_1X_2 + 2.737 \times 10^{-6}X_1^2 + 0.0452X_2^2 \qquad (2)$$

Figure 5 presents the predicted values of the obtained model versus the actual values of the experimental results of the gelation time. The predicted gelation time values were obtained by using Equation 2. The accuracy of a fitted-response surface model was statistically evaluated based on the coefficient of determination (R^2) and the ANOVA analysis.

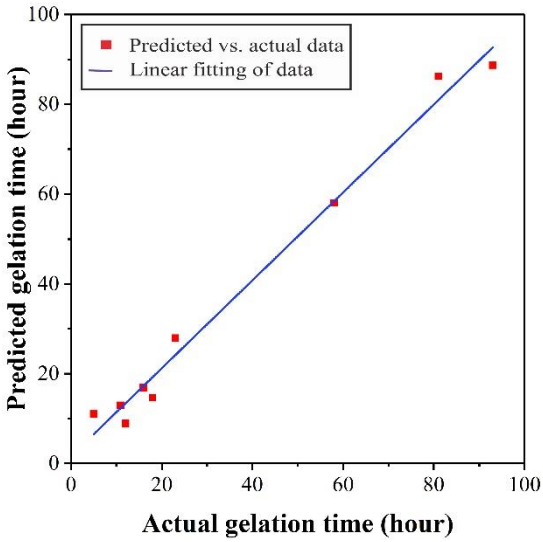

Figure 5

Values predicted using the developed model versus the actual values of the gelation time.

The results of the statistical evaluation of the developed model are shown in Table 7, where the sum of squares, mean square, and the model's degree of freedom (DOF) are respectively defined as the total of the sum of squares for the terms in the model, the estimation of the model variance, and the number of model terms. The F-test is a way for the analysis of the model capability to fit the experimental data,

and the P-value is a quantitative measurement for reporting the result of a test hypothesis. It is probable that the test statistics are at least as extreme as the one observed given that the null hypothesis is true. The model F-value of 42.15 was greater than 0.001, and the model P-value was less than 0.005, which means that the model was highly significant, and the fitted model is noteworthy. The R^2 value was 0.9860, indicating that the prediction of the gelation time on an experimental scale with the obtained model can be performed efficiently, and that the quadratic polynomial was capable of representing the systems over the given experimental domain.

Table 7

ANOVA results and the coefficient of determination (R^2) of the models developed for gelation time.

Source	Sum of squares	DOF	Mean square	F-value	P-value	R^2
Model	8683.95	5	1736.79	42.15	0.0046	0.9860
Residual	123.61	3	41.20	–	–	

Figures 6 and 7 represent the contour plot and response surface plot for the combined effects of simultaneous changes in the copolymer concentration and copolymer/cross-linker ratio on the gelation time respectively. Based on the results, the variations in the two parameters led to varying gelation times in the range of 11 to 93 hours. The maximum gelation time for gel systems was obtained at the minimum copolymer concentration and the maximum copolymer/cross-linker ratio. In other words, as a result of a decrease in the copolymer concentration and an increase in the copolymer/cross-linker ratio, higher gelation times were achieved for the NSPs gel systems. Decreasing copolymer concentration made lower sites available for the formation of complexes with a cross-linker. Moreover, higher copolymer/cross-linker ratio caused a decrease in the crosslinking reaction rate, which led to a deaccelerated gelation process, thereby resulting in longer gelation times. Therefore, the gelation time depends on the gel composition and the environmental conditions and can be controlled from a few hours to several days. In other words, by properly designing gel components, including copolymer and cross-linker, the desirable gelation time under reservoir conditions can be achieved. It is notable that before any polymer gel experiments, the stability of polymer as a criterion for maintaining gel stability should be investigated. Unstable polymer under the intended conditions does not lead to the formation of a stable gel under the same conditions.

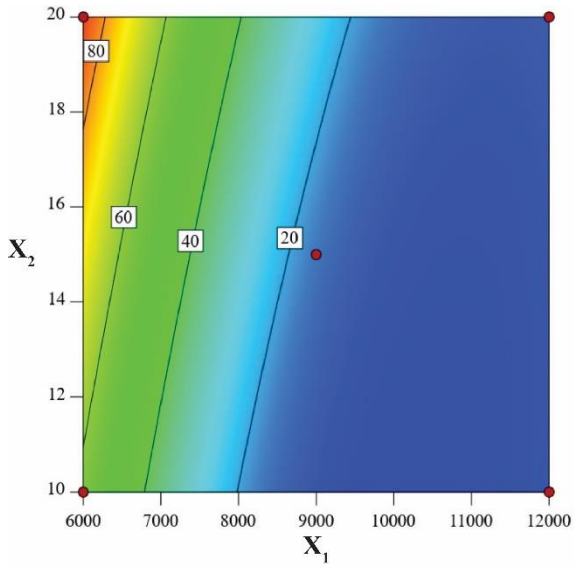

Figure 6

Contour plot of the combined effect of copolymer and copolymer/cross-linker ratio on gelation time.

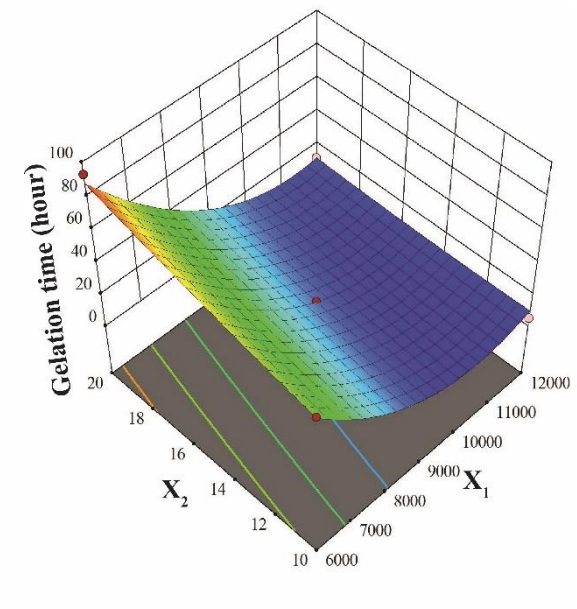

Figure 7

Response surface plot illustrating the mutual effects of copolymer and copolymer/cross-linker ratio on the gelation time.

3. Conclusions

In this study, three polymers were compared in terms of their stability at a temperature of 90 °C and salinity of 87000 ppm. Then, the NSPs as an optimum case were selected for gelation studies, and nine gel systems composed of NSPs and chromium (III) acetate were prepared by using the response surface methodology based on the central composite design. The gelation time of the prepared gel systems at a temperature of 90 °C and salinity of 87000 ppm was investigated by using the bottle test technique. The following conclusions can be drawn from this research:

- Flopaam3330 and AN105 as conventional polymers exhibited a viscosity reduction of 69 and 36% after 40 days respectively, which demonstrated their disadvantage in reservoirs under harsh conditions.
- The stability analysis demonstrated the effectiveness of the NSPs as stable polymers for future EOR demands in high-temperature/high-salinity reservoirs.
- According to the central composite design results, the NSPs gel system had an optimum gelation time at a copolymer concentration and a copolymer/cross-linker ratio of 6000 ppm and 20 respectively.
- Decreasing copolymer concentrations and an increase in copolymer/cross-linker ratio led to the prolonged gelation time in the NSPs gel systems.
- A quadratic polynomial model was presented for the NSPs gel system to predict its gelation time.
- Large values of the predicted R^2 validated that the developed model can well fit the experimental data of gelatin time.

Nomenclature

AAm	Acrylamide
AMPS	2-acrylamido-2-methyl-1-propane sulfonic acid
ANOVA	Analysis of variance

DOF	Degree of freedom
EOR	Enhanced oil recovery
NSPs	Nano-structured polymers

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