

## SCREENING OF EFFECTIVE FACTORS ON GELATION TIME OF HYDROGELS

Asefe Mousavi Moghadam<sup>a</sup>, Mahsa Baghban Salehi<sup>a</sup>, Mohsen Vafaie Sefti<sup>a</sup>, Ahmad Dadvand Koochi<sup>b</sup>,  
Hasan Naderi<sup>c</sup>

<sup>a</sup>Chemical Engineering Department, Tarbiat Modares University, Jalal Ale Ahmad Highway, P.O. Box: 14155-143, Tehran, Iran.

<sup>b</sup>Chemical Engineering Department, Guilan University, P.O. Box 1841, Rasht, Iran.

<sup>c</sup>Center for Exploration and Production Studies and Research Division, Research Institute of Petroleum Industry (RIPI), P.O. Box: 18745-4163, Tehran, Iran.

\*Correspondence author: Fax: +98 21 82884931 E-mail: vafaiesm@modares.ac.ir

### ABSTRACT

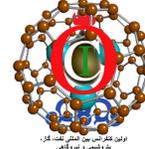
In this work, a hydrogel was prepared by crosslinking of aqueous solutions of sulfonated polyacrylamide/chromium triacetate in purpose of water shut off treatment. In order to screen the effective factors on the gelation time and investigate their interactions, fractional factorial design method was used in experimentation with eight factors (pH, CaCl<sub>2</sub> concentration, crosslinker/co-polymer ratio, NaCl concentration, co-polymer concentration, sodium lactate, nanoclay and thiouria) and one response (gelation time). Furthermore, the analysis of variance (ANOVA) was implemented as a significant tool to evaluate the quality of the quadratic model which was shown the 99% confidence limit of significant. Among the eight factors, sodium lactate, co-polymer concentration, nanoclay, thiouria and crosslinker/co-polymer ratio were the main effects respectively and the interactions between sodium lactate and crosslinker/co-polymer ratio, pH and crosslinker/co-polymer ratio were highly effective.

### INTRODUCTION

Water production in oil-producing wells becomes a more serious problem as the wells mature. Remediation techniques for controlling water production, generally referred to as conformance control, are selected on the basis of the water source and the method of entry into the wellbore [1]. Water shut-off methods can be classified in two different types: mechanical and chemical methods [2-4]. The chemical methods, extensively used in the last decade, consist namely on chemical products that are pumped into producer or injector wells. Most of these systems are based on polymer solutions that after a given time turn from low viscosity liquids to strong

or weak gels 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. Several authors have reported the characteristics of gel polymers (hydrogels) utilized for other purposes in detail [5-7]. Selection of a polymer gel 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 polymer gel system include salinity of the formation water, permeability of the target zone, and the lithology of the formation [8-11]. Al-Muntasheri et al. [12] studied the effect of different parameters (polymer concentration, crosslinker concentration, salinity and pH) on the gelation time of polyacrylamide and polyethyleneimine (PEI). They found that initial pH value had a strong influence on gel viscosity. Higher viscosities were obtained at higher initial pH values. Under acidic conditions, the gelation time was short and the gel did not last for a long time.. Zolfaghari et al. [13] used nanocomposite type of hydrogels (NC gels) by crosslinking the polyacrylamide/montmorillonite (Na-MMT) nanoclay aqueous solutions with chromium (III). They showed that the rate of gelation is retarded specially for the gelant solution composed of 2% (volume percent) of the sodium lactate as retarder. They observed that by adding retarder to the gelant; the more the retarder content increased, the less the syneresis became.

There were several factors that may affect the gelation time investigated by several authors but the availability of interactions between the factors and their effects on the gelation time have not been



studied yet. In this work, the effect of eight factors (pH,  $\text{CaCl}_2$  concentration, crosslinker/co-polymer ratio, NaCl concentration, co-polymer concentration, sodium lactate, nanoclay and thiouria) was investigated on the gelation time and their main effects were identified and at last their interactions were determined by two-level factorial designs. Consequently, the main purpose of this research was to establish a functional relationship of the gelation time where the parameters influencing the gelation time were investigated with the central idea of conducting the least number of experiments, and finally a quadratic model predicting the gelation time was presented. Besides, by using two-level factorial designs not only the important parameters were screened but also the interactions between parameters were determined.

## EXPERIMENTAL MEASUREMENT

**Materials:** In this section, a general description of experimental measurements and calculations are presented. The tests were conducted by the use of a co-polymer of 2-acrylamido-2-methylpropanesulfonic-acid sodium salt (AMPS) and acrylamide (AcA), with an average molecular weight of 8,000,000, sulfonation degree of 25% and water content of less than 10 wt%, provided by SNF Co. (France). It is also called sulfonated polyacrylamide (PAMPS), under the trade name of AN125, in powder form. Furthermore, chromium triacetate, as a metallic crosslinker, purchased from Carlo Erba Co. (Italy), was used in powder (pure) form. NaCl and  $\text{CaCl}_2$  of analytical grade, HCl (0.01 M) and NaOH (0.01 M) were used for preparation of acidic or basic water, respectively. Nanoclay used in this study was Nanomtorillonite with  $d_{001}$  interplanar spacing of 12 supplied from Advanced Technology (China). Sodium lactate was also used as retarder, and was provided by Merck Co. (Germany) in the form of a colorless liquid. Thiouria from Merck was used as a stabilizer against oxygen-induced polymer degradation.

### Samples Preparation and Characterization:

The polymer gels were prepared according to the following steps: A) PAMPS solutions, at the concentration of 2%, were obtained by mixing the co-polymer powder and distilled water for the period of 2 hrs. The mixture was then held, without stirring, for 2 days to obtain a homogeneous solution. Shortly before the commencement of the experiment, the PAMPS solutions were diluted to the required concentrations and the mixtures were stirred for 5

min. B) Cr (III)-acetate (as crosslinker) was mixed with required amount of NaCl,  $\text{CaCl}_2$ , sodium lactate, thiouria, nanoclay and acidic or basic water according to the experimental plan of the tests at room temperature, using a heater magnetic stirrer (Stuart CB162) for 5 min, as a "second solution". It must be mentioned that a 2%wt of nanoclay solution was mixed for 24 hrs before this step in order to be used in each sample preparation. C) The PAMPS and second solutions were mixed for 5 min to obtain a gelant solution.

Since most of the south Iranian reservoirs have a high temperature, around  $90^\circ\text{C}$ , this temperature was selected for experiments in the present work and PAMPS was selected for the experiments, because it has higher thermal stability and salt resistance than standard hydrolyzed polyacrylamides. Moreover, because of the high amount of sodium and calcium ions in formation water, respectively, in contrast with other ions, they were selected as monovalent and divalent ions to examine their effects on the gelation time.

**Bottle Test:** In this work the bottle test method was used to study the effect of various parameters on the gelation time of polymer gel. 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 faster and inexpensive method to study gelation kinetic. In this method, which was defined by Sydansk [10], gel strength during development of gelation kinetic was expressed as an alphabetic code of A through I which is shown in Table 1.

Table 1  
Gel strength code [10]

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

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



[8]. Therefore, the gelation time was considered in this work as the period of time which 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 90°C. 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 deformations 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 [14].

**Fractional Factorial Design:** In order to present the general result, two-level factorial design, the most popular method among factorial designs, which are widely used in industrial experiments, was used. Two-level factorial designs, is  $2^{k-p}$  fractional factorial design – a fraction of the  $2^k$  full factorial design, generated by p generators. Each factor of this method varies in two levels of maximum and minimum. Two-level fractional factorial ( $2^{k-p}$ ) design allows us to study many factors with relatively small run size and commonly used for finding important effects, especially when runs are expensive. When designing the basic experiment, the unknown response function is, in principle, approximated by a polynomial of the corresponding degree where regression coefficients are estimated on the basis of experimental results. A quadratic (polynomial equation) mathematical model is considered in the first phase of a research. Defining the two order regression model is the first phase of a study objective at obtaining the interpolation model or function, the knowledge of which facilitates estimating response values in different points of the studied factorial space. A quadratic model is, additionally, also used when moving to the optimum region, the same as when we use the steepest ascent method as an optimization technique. Later, if necessary, the polynomial degree is increased. Lack of fit of polynomial models is checked by methods of statistical analysis. In defining a quadratic model, coefficient  $b_0$  and all coefficients are calculated.

$$y = b_0 + \sum_{i=1}^k b_i x_i + \sum_{i \geq j=1}^k b_{ij} x_i x_j \quad (1)$$

where y is response value;  $b_i$  are coefficients;  $b_{ij}$  are regression coefficients of double factor interactions. Accuracy and confidence of the obtained estimates

for regression coefficients depend on the used design of experiments [15, 16]. As it is mentioned above, each factor varies in two levels of maximum and minimum. In this experimental design method code (+1) was belonged to the maximum level and code (-1) to the minimum level (Table 2). A model was also presented to pretend the response. In order to study the random error, central points were used during the experimental design where each factor of these points was applied in the average amount of its maximum and minimum value to be an evidence for the presented quadratic model.

## RESULTS AND DISCUSSION

The selected effective factors (pH,  $\text{CaCl}_2$  concentration, crosslinker/co-polymer ratio, NaCl concentration, co-polymer concentration, sodium lactate, nanoclay and thiouria) on the gelation time are illustrated in Table 2.

Table 2  
The selected levels of variables

Factor	Min (-1)	Max (+1)
A: pH	3	9
B: $\text{CaCl}_2$ concentration (ppm)	0	15000
C: Crosslinker/co-polymer ratio	1/10	1/2
D: NaCl concentration (ppm)	0	15000
E: Co-polymer concentration (ppm)	5500	9500
F: Sodium lactate (wt%)	0	1
G: Nanoclay (wt%)	0	1
H: Thiouria (ppm)	0	5000

The experimental plan of the tests and its results according to two level fractional factorial design method is illustrated in Table 3.

Table 3  
The experimental plan of the tests and its results

Run	A	B	C	D	E	F	G	H	Gelation Time (hr)
1	1	-1	-1	1	1	-1	-1	1	4
2	1	1	-1	-1	-1	1	1	-1	280
3	-1	-1	-1	-1	-1	-1	-1	1	60
4	1	1	-1	-1	-1	-1	-1	1	50
5	-1	-1	1	1	-1	1	-1	-1	190
6	-1	-1	-1	-1	1	1	-1	1	120
7	-1	1	-1	1	-1	1	-1	1	240
8	1	1	1	1	1	1	1	1	144



9	-1	1	1	-1	1	1	-1	-1	50
10	-1	-1	-1	-1	1	-1	1	-1	168
11	-1	-1	-1	-1	-1	1	1	-1	350
12	1	1	1	1	-1	1	-1	-1	140
13	-1	1	-1	1	1	1	1	-1	220
14	-1	1	-1	1	1	-1	-1	1	40
15	1	-1	-1	1	1	1	1	-1	160
16	1	-1	1	-1	1	1	-1	-1	144
17	-1	-1	1	1	1	-1	-1	-1	48
18	1	1	-1	-1	1	-1	1	-1	5
19	1	1	1	1	-1	-1	1	1	22
20	-1	-1	1	1	1	1	1	1	160
21	-1	1	1	-1	-1	-1	-1	-1	80
22	1	-1	-1	1	-1	-1	1	-1	34
23	1	-1	1	-1	-1	1	1	1	120
24	-1	1	1	-1	1	-1	1	1	60
25	1	1	1	1	1	-1	-1	-1	7
26	1	-1	1	-1	-1	-1	-1	-1	168
27	-1	1	1	-1	-1	1	1	1	120
28	1	-1	1	-1	1	-1	1	1	168
29	1	1	-1	-1	1	1	-1	1	130
30	-1	-1	1	1	-1	-1	1	1	48
31	-1	1	-1	1	-1	-1	1	-1	130
32	1	-1	-1	1	-1	1	-1	1	168

In order to reduce the random error, the samples were prepared by following the first column of Table 3 which was ordered coincidentally. All eight factors were coincided and varied in two levels of maximum and minimum to denote not only the main effects, but also the interactions between the factors.

**Modeling Process Remarks:** Thirty two bottle testing experiments are designed applying two level fractional factorial design. The results were inserted in “Design Expert (DX)” software, so a quadratic relation was obtained which can predict the gelation time in the range beyond the selected range of experiments.

$$\begin{aligned}
 \text{GelationTime} = & 119.63 - 10.63A - 12.25B - 15.31C \\
 & - 9.94D - 17.88E + 51.38F + 17.19G - 16.25H \\
 & + 0.5AB + 20.44AC - 14.19AD + 4.13AE + 0.37AF \quad (2) \\
 & - 9.56AG + 8AH - 7.5BE + 6.75BF - 1.94BG \\
 & + 9.63BH + 11.19CE - 22.19CF + 6DE + 16.69DF \\
 & - 12.13DG + 9.8DH + 17.75EH - 4.5FH
 \end{aligned}$$

In this model, all variables are indicated through in coded values that are illustrated in the first column of Table 2. As can be seen in Eq. (2), the interactions between variables have significant effects on responses; so here the results are preferably presented and discussed in terms of interactions. The statistical significance of Eq. (2) is shown in Table 4, where the mean square, sum of squares and model degree of

freedom (DOF) terms are, respectively, defined as the estimation of the model variance, total of the sum of squares for the terms in the model and the number of model terms.

Table 4  
The ANOVA results of the developed model

		Sum of square	DOF	Mean square	P-value
Gelation time	Model	2.117 E+005	27	7841	0.0085
	Residual	2073.5	4	518.38	-

DOF shows the total number of model terms, including intercept minus one. It is evident that the model is highly significant, as suggested by the model F value and a low probability value (P-value= 0.0085). The P-value is a quantitative measurement for reporting the result of a test hypothesis. It is the probability of the test statistics being at least as extreme as the one observed given that the null hypothesis is true. The analysis of variance (F-test) shows that the second order model (quadratic polynomial) well fits the experimental data. Figure 1 represents the predicted values versus actual values of the gelation time. The high value of R-square ( $R^2 = 0.9903$ ) indicated that the equation was capable of representing the system under the given experimental domain.

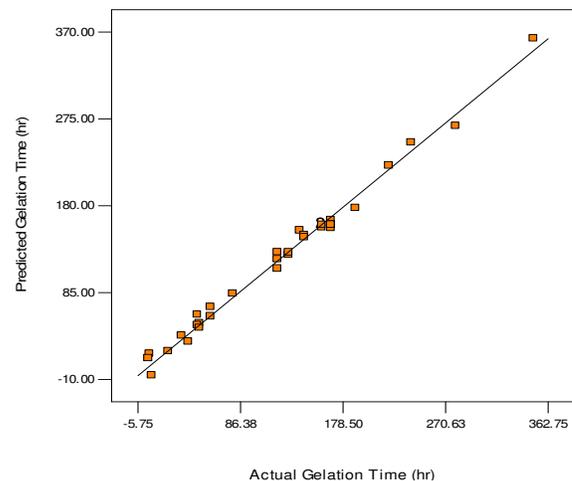


Figure 1  
Predicted based on the model in Eq.6 vs. actual values of the gelation time (Label 2 shows the number of replication of one point.)

As in Eq. (2) is also specified, factors F, E and G which are sodium Lactate, co-polymer concentration



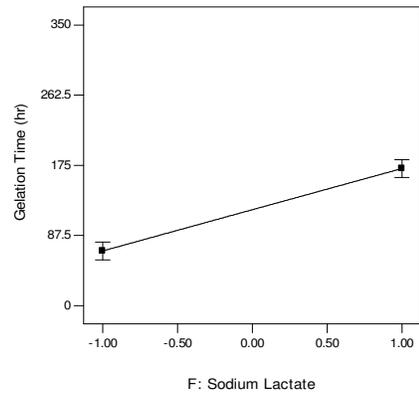
and nanoclay are the main effects between factors respectively because of higher coefficients, and all binary letters (AB,AC,...) are the main interactions between two factors. Positive sign represent the increasing effect on the respond i.e. increase of the factor causes an increase on the gelation time, and the negative sign represent the decreasing effect on the respond i.e. increase of the factor causes a decrease on the gelation time.

As it is mentioned above sodium lactate (with positive sign) is the main effect. When it is mixed with Cr (III) acetate, the acetate ligand is exchanged with lactate, which is a weaker ligand [17]. Consequently, it takes longer time for chromium to separate from its ligand (lactate) and react with the polymeric chains.

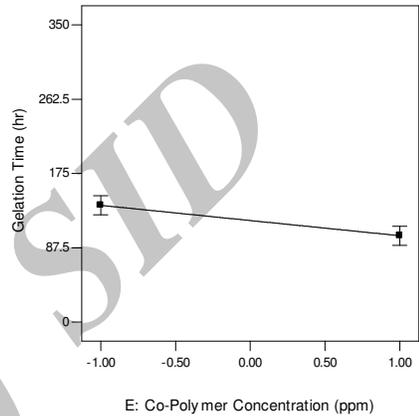
The other main effect is co-polymer concentration which its increase causes a decrease on the gelation time. This variation in the gelation time with the changes in co-polymer may be explained as follows; In general, gelling network structures are highly heterogeneous [18]. The heterogeneity of gels has a lattice-like structure consisting of two domains: the dilute domain and the dense domain. An increase in co-polymer concentration causes an increase in the dense domain, which in turn results in the reduction of the gelation time.

After sodium lactate and co-polymer concentration, nanoclay has the main effect. As it shown in Eq. (2) increase of nanoclay causes an increase on the gelation time which corroborated the previous result [19]. This result can be explained as follows; According to the mechanism suggested by Haraguchi [20], the polyacrylamides chains are bound to the surface of the nanoclay particles due to hydrogen bonds between the oxygen atoms of nanoclay and the amide protons of the acrylamide as well as due to complex formation between the metal ions on the nanoclay surface and the carbonyl oxygen of the acrylamide. The bottle tests results also corroborated that the initial viscosity is reduced by increase of nanoclay concentration. It seems that the presence of nanoclay in network caused a decrease in reaction between co-polymer and crosslinker. Consequently, potential cross-linking sites on the co-polymer chain are not as accessible to the cross-linker molecules. The gelation induction period will be longer in this case and hence the gel will take a longer period of time to form. Figure 2 shows the main effects of variables on the gelation time and interactions of variables on the gelation time is shown in Fig. 3.

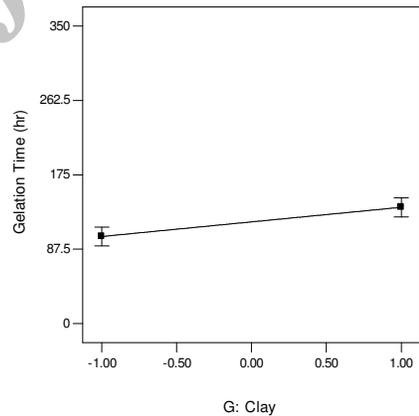
**a**



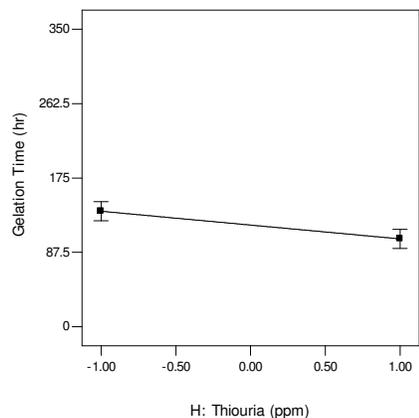
**b**



**c**



**d**



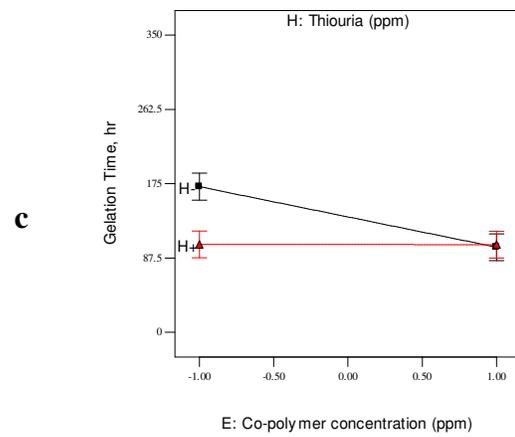
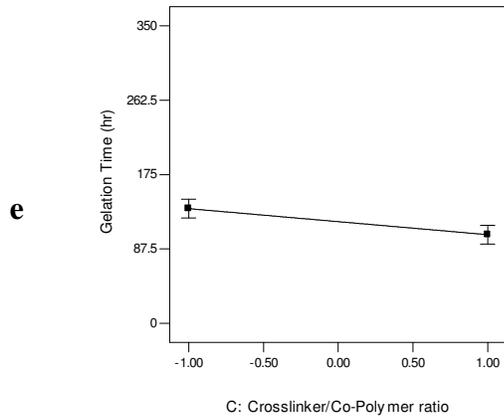


Figure 2

Main effects of variables on response-gelation time

Plots make it easy to interpret two factor interactions. They will appear with two non-parallel lines, indicating that the effect of one factor depends on the level of the other.

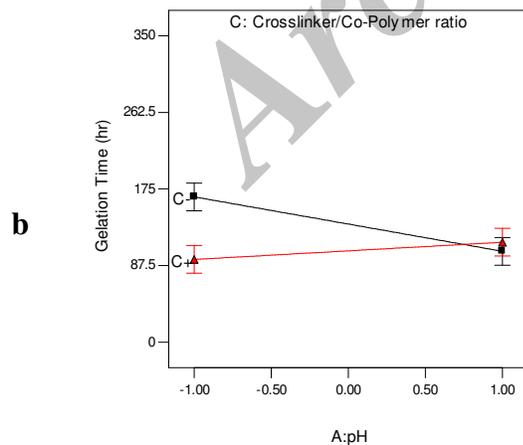
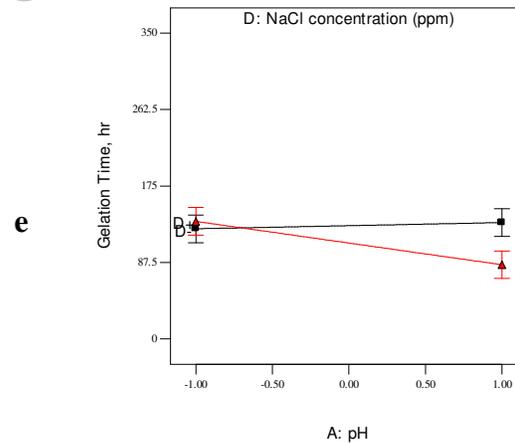
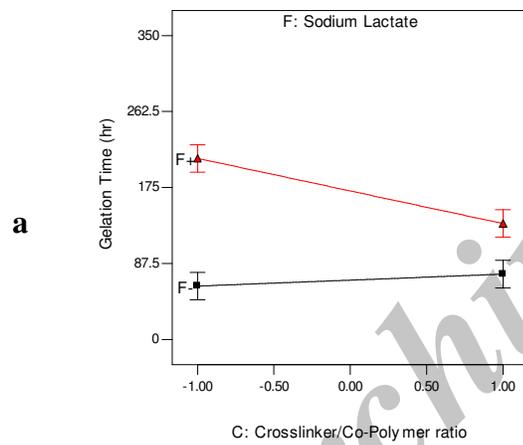
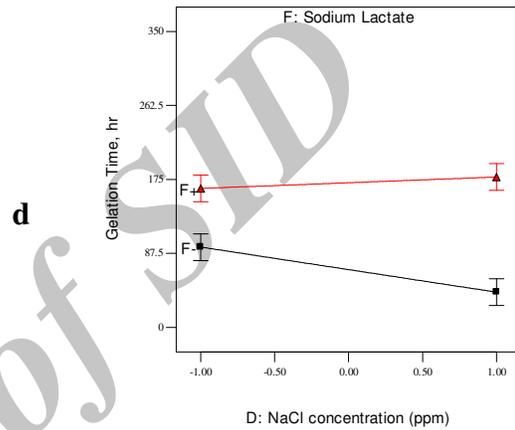


Figure 3

Interactions of variables on response-gelation time

As it shown in Fig. 3 each curve has two lines for two steps which in that the gelation time is curved once in maximum value of the factor (black line) and once in minimum value of the factor (red line) via the other factor. Among the all factors interactions, the interactions between sodium lactate and crosslinker/co-polymer ratio, pH and crosslinker/co-polymer ratio have the higher effects on the gelation time, respectively. The strongest interaction is between the factors C and F (sodium



lactate and crosslinker/co-polymer ratio) because of the highest coefficient in Eq. (2) which means its effect is highly significant. As it shown in Fig. 3 in minimum value of sodium lactate, increase of crosslinker/co-polymer ratio causes an increase on the gelation time where in maximum value of sodium lactate, increase of crosslinker/co-polymer ratio causes a decrease on the gelation time. The high difference of the gelation time between the maximum and minimum value of -1 and the maximum and minimum value of +1 levels of crosslinker/co-polymer ratio showed the high value of interaction between these two factors. The interaction between the factors A and C (pH and crosslinker/co-polymer ratio) has the most significant effect after the interaction between CF. According to Fig. 3, in minimum value of crosslinker/co-polymer ratio, increase of pH causes a decrease on the gelation time where in maximum value of crosslinker/co-polymer ratio, increase of pH causes an increase on the gelation time. The two non-parallel lines of these curves shows the interaction incidence of the two factors during the selected range of the experiments.

## CONCLUSIONS

Screening of eight effective factors (pH,  $\text{CaCl}_2$  concentration, crosslinker/co-polymer ratio, NaCl concentration, co-polymer concentration, sodium lactate, nanoclay and thiouria) was accomplished on the gelation time of the PAMPS/Cr (III)-acetate hydrogels by applying two level fractional factorial design method which cause to thirty two bottle testing experiments. A quadratic relation was obtained which can predict the gelation time by inserting the results of experiments in "Design Expert (DX)" software. The results showed that sodium lactate, co-polymer concentration, nanoclay, thiouria and crosslinker/co-polymer ratio were the main effects, respectively. Increase of sodium Lactate and nanoclay caused an increase on the gelation time, where increase of co-polymer concentration caused a decrease on the gelation time. Moreover, among the all interactions between the effective factors on the gelation time, the interactions between sodium lactate and crosslinker/co-polymer ratio, pH and crosslinker/co-polymer ratio had the higher effects on the gelation time, respectively.

## KEYWORDS

Polymer Gel, Gelation Time, Two Factorial Design, Fractional Factorial Design, Interaction

## REFERENCES

1. Reddy, B. R.; Larry Eoff; Dwyann Dalrymple, E.; Black, K.; Brown, D.; Rietjens, M. A Natural Polymer-Based Crosslinker System for Conformance Gel Systems. Presented in the SPE/DOE Improved Oil Recovery Symposium. Tulsa. Oklahoma USA. **2002**; Paper 75163.
2. Vega, I.; Morris, W.; Robles, J.; Peacock, H.; Marin, A. Water Shut-off Polymer Systems: Design and Efficiency Evaluation based on Experimental Studies. Presented in the SPE Improved Oil Recovery Symposium. Tulsa. Oklahoma. USA. **2010**; Paper 129940.
3. Sydansk, R. D.; Southwell, G. P. More Than 12 Years Experience with A Successful Conformance Control Polymer Gel Technology. **2000**; SPEPF, 15(4), 270-278.
4. Seright, R.S.; Lane, R. H.; Sydansk, R. D. A Strategy for Attacking Excess Water Production. **2003**; SPEPF, 70067, 158-169.
5. Ganji, F.; Vasheghani-Farahani, E. Hydrogels in Controlled Drug Delivery Systems. Iran. Polym. J. **2009**; 18, 63-68.
6. Rudzinski, W.E.; Dave, A.M.; Vaishnav, U.H.; Kumbar, S.G.; Kulkarni, A.R.; Aminabhavi, T.M. Hydrogels as Controlled Release Devices in Agriculture. Design. Mon. Polym. **2002**; 5, 39-65.
7. Huang, J.; Wang, X.I.; Yu, X.H. Solute Permeation Through the Polyurethane-NIPAAm Hydrogel Membranes with Various Crosslinking Densities. Desalination. **2006**; 192, 125-131.
8. Sydansk, R. D. A New Conformance Improvement Treatment Chromium (III) Gel Technology. Presented in the SPE Enhanced Oil Recovery Symposium. Tulsa. Oklahoma. **1988**; Paper 17329.
9. Vossoughi, S. Profile Modification Using in Situ Gelation Technology-A Review. Journal of Petroleum Science and Engineering. **2000**; 26, 199-209.
10. Sydansk, R. Conformance Improvement in a Subterranean Hydrocarbon-Bearing Formation Using a Polymer Gel. US Patent. No.4683949. **1987**.
11. Seright, R.S.; Martin, F.D. Impact of Gelation pH, Rock Permeability, and Lithology on the Performance of a Monomer-Based Gel. SPE Reservoir Engineering. **1993**; 8(1), 43-50.
12. Al-Muntasheri, G.A.; Nasr-El-Din, H.A.; Ibelwaleed A.; Hussein A. Rheological Investigation of A High Temperature Organic Gel



- Used for Water Shut-off Treatments. *J. Pet. Sci. Eng.* **2007**; 59(1-2), 73-83.
13. Zolfaghari, A.; Katbab, A. A.; Nabavizadeh, J.; Yousefzadeh Tabasi, R.; Hossein Nejad, M. Preparation and Characterization of Nanocomposite Hydrogels Based on Polyacrylamide for Enhanced Oil Recovery Applications. *Journal of Applied Polymer Science.* **2006**; 100, 2096–2103.
14. Simjoo, M.; Vafaie Sefti, M.; Dadvand Koochi, A.; Hasheminasab, R. Polyacrylamide Gel Polymer as Water Shut-off System: Preparation and Investigation of Physical and Chemical Properties in One of the Iranian Oil Reservoirs Conditions. *Iran. J. Chem. & Chem. Eng.* **2007**; 26.
15. Engineering Statistics Handbook, National Institute of Standards and Technology and the International SEMATECH, **2003**.
16. Živorad R. Lazic. Design of Experiments in Chemical Engineering. **2004**.
17. Lockhart, T.P.; Burrafato, G. US Patent 5131469, **1992**.
18. Grattoni, C.A.; Al-Sharji, H.H.; Yang, C.; Muggeridge, A.H.; Zimmerman, R.W. Rheology and Permeability of Crosslinked Polyacrylamide Gel. *J. Colloid and Interface.* **2001**; 240, 601-607.
19. Aalaie, J.; Vasheghani-Farahani, E.; Rahmatpour, A.; Semsarzadeh, M.A. Effect of Montmorillonite on Gelation and Swelling Behavior of Sulfonated Polyacrylamide Nanocomposite Hydrogels in Electrolyte Solutions. *European Polymer Journal.* **2008**; 44, 2024–2031.
20. Haraguchi, K.; Li, H.; Matsuda, K.; Takehisa, T.; Elliott, E. Mechanism of Forming Organic/Inorganic Network Structures During In-situ Free-radical Polymerization in PNIPA-Clay Nanocomposite Hydrogels. *Macromolecules.* **2005**; 38, 3482–90.

Archive of SID