

## Effects of reservoir temperature and water salinity on the swelling ratio performance of enhanced preformed particle gels

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**Abstract**—Preformed particle gel (PPG) treatment is now one of the most effective remediation techniques for conformance controlling and selective plugging of high-water flow conduits in mature water-flooded oil reservoirs. Recognizing the intrinsic properties of PPGs, e.g., the swelling ratio, in reservoir condition is of prime importance to the optimization of their performance as plugging agents. In this study, the classical and three-level full factorial experimental design methods are joined with laboratory measurements to investigate the swelling ratio dependency of a new class of enhanced PPGs at different brine salinities and reservoir temperatures. To cover severe reservoir conditions, the reservoir temperature from 298 to 418 K and brine salinity from 0 to 225,000 ppm were considered during the laboratory measurements. The results show that the swelling ratio decreases by rising water salinity. Moreover, the swelling ratio rises by increasing reservoir temperature up to 380 K and then starts to decrease. The factor screening illustrates that the swelling ratio is more dependent on salinity than the reservoir temperature in low salinity solutions, while is less dependent on salinity in high salinity solutions. In addition, a precise mathematical model was developed to predict the swelling ratio of PPGs in a wide range of salinities and temperatures. The results of this study present a practical insight into the swelling-related behavior of the PPGs at reservoir conditions.

Keywords: Preformed Particle Gels, Swelling Ratio, Experimental Design, Reservoir Temperature, Water Salinity

### INTRODUCTION

Heterogeneity of oil reservoirs causes the profile of injection water to be not uniform. The consequences of this condition may result in very poor sweep efficiency and, consequently, a high water production ratio in production wells. For that reason, water production control, also called conformance control, has been the subject of many studies during the past years [1,2].

Polymeric hydrogel treatment is one of the most efficient and cost-effective methods for conformance control in oil reservoirs [3-8]. Hydrogels are cross-linked materials that can absorb large quantities of water without dissolving into water phase. Softness and capacity to store water make hydrogels unique [9]. These gels that exhibit swelling variation in response to environmental changes such as temperature, salt concentration, type of surfactants, pH, etc., are promising as intelligent materials [10].

Among the different methods of gel treatments, the newest method, preformed particle gel (PPG), is preferred because of its capability of overcoming some distinct drawbacks inherent to in-situ gelation systems [5,7,21]. The PPGs are environmentally friendly, strength-, and size-controlled particles that have adjustable mechanical properties and controllable swelling ratio. PPGs have been applied in many case studies to reduce the fluid channels in water or

polymer flooding projects [11,12].

Swelling can substantially change the volume and properties of a gel placed in a formation, and therefore, influence the effectiveness of a polymer treatment project [14]. The swelling ratio of PPGs has a great impact on the injectivity index of a well and extremely affects blocking in porous media [15]. The swelling ratio of PPGs is reported to be affected by only water salinity in many studies and higher water salinity results in a lower swelling ratio [2,4,7,15-17]. In addition, these particles are stable in a wide range of formation salinities [18]. Mousavi Moghadam et al. investigated the effects of both water salinity from 0 to 30,000 ppm and pH from 3 to 11 on the swelling ratio of PPGs [19]. Their results show that the swelling ratio depends more on the water salinity than the pH of solution. The swelling ratio of polymer gels can be changed due to temperature variations. Molloy et al. inspected the effect of water salinity in the range of 5,000 to 35,000 ppm on the swelling ratio of polyacrylamide gels. For each salinity, the effect of temperature from 5 °C to 40 °C was investigated. It is found that polyacrylamide gels shrink in all solutions and this effect is most evident at higher salinities. The effect of temperature on all solutions is minimal, promoting a decreasing volume change as temperature rises [20].

Gel swelling ratio and its effect on fluid flow have been less studied [16]. In previous studies, the test conditions did not meet severe reservoir conditions regarding salinity and temperature. Therefore, the main idea of this study is the recognition of PPG swelling behavior at reservoir conditions. Here, the effects of both water salinity

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and reservoir temperature on the swelling ratio of an enhanced PPG were studied. The implemented PPGs in this study were prepared for conformance control in severe reservoir conditions up to a water dissolved solid of 225,000 ppm and a temperature of 418 K [13]. To study the swelling behavior of PPG, this paper is organized as follows: in the next section, the statistical and experimental methods used in this study are described. Then a detailed discussion of the analysis is presented. This is followed by our conclusion summarizing the key findings of this study.

## MATERIALS AND METHODS

### 1. Synthesis of PPGs

The PPGs were produced with 30 wt% of a 2:1:2:1 molar ratio of Acrylamide (AM): N,N-dimethyl acrylamide (DA): 2-acrylamido-2-methylpropane sulfonic sodium salt (AMPSNa): N-vinylpyrrolidone (NVP) monomers through free-radical crosslinking polymerization at room temperature in distilled water. N,N-methylenebis (acrylamide) (MBA) with a weight percent of 0.55% was used as a cross-linker. The mechanical properties of these new PPGs were enhanced by adding a 2.5 wt% nano-clay montmorillonite Na<sup>+</sup>. A temperature stability agent was also used to make these special PPGs compatible with higher temperature and salinity at reservoir conditions. The chemicals used to formulate the PPGs are itemized in Table 1.

The weighed powder of nano-clay montmorillonite Na<sup>+</sup> was poured into the proper amount of distilled water in a beaker. The beaker was dispersed for several hours by an ultrasonic disperser. Subsequently, a specific amount of AM, NVP, DA, AMPSNa monomers, and the temperature-stability agent were added to the dispersed solution every 5 minutes. The mixture was stirred until all monomers were completely spread. Afterward, the system was purged by nitrogen and the MBA was added to the mixture. The nitrogen purging and solution stirring were continued for 40 minutes,

and then a specific amount of N,N,N',N'-tetramethylethylenediamine catalyst and the sodium persulfate initiator were added to the beaker. The polymerization reaction was initiated immediately, and gel bulk was observed within 20 minutes. The gel was immersed in distilled water for 24 hours and then cut into small parts and dried in a vacuum oven at 328 K for 24 hours [13]. It was then crushed and sieved to get the proper-sized PPGs.

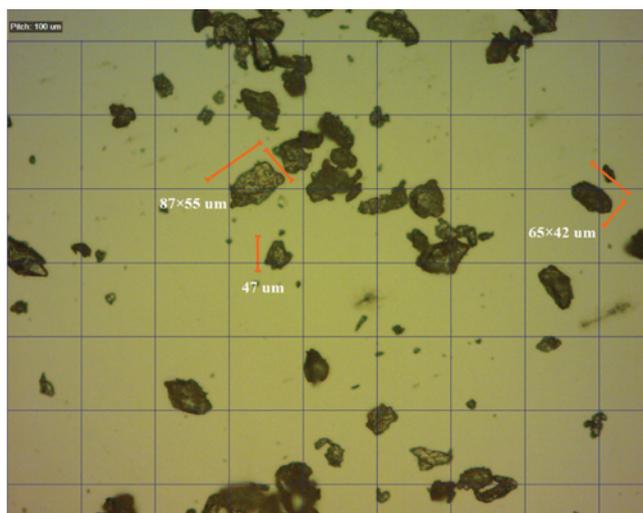
### 2. Experimental Approach

The swelling ratio of PPGs can be measured either by comparing the volume (diameter) of particles before and after the swelling, i.e., volume-fraction method [5,22,23] or by comparing the mass of particles before and after swelling, i.e., mass-fraction method [17-19,24-26]. Fig. 1 depicts two microscopic photos of PPGs before and after swelling. The dried particles before swelling were sieved between 32 and 40 μm. As indicated in Fig. 1(a), the shape of these particles is quite irregular and this irregularity is increased after swelling (Fig. 1(b)). Diameter measurement before and after swelling under a microscope can provide us a true picture of swelling process at the particle scale; however, this measurement cannot provide a unique value of PPGs diameter before and after swelling in our system. Alternatively, a mass ratio of swelled-to-dried PPG, measured by a precise laboratory balance, was implemented as an indicator for swelling ratio.

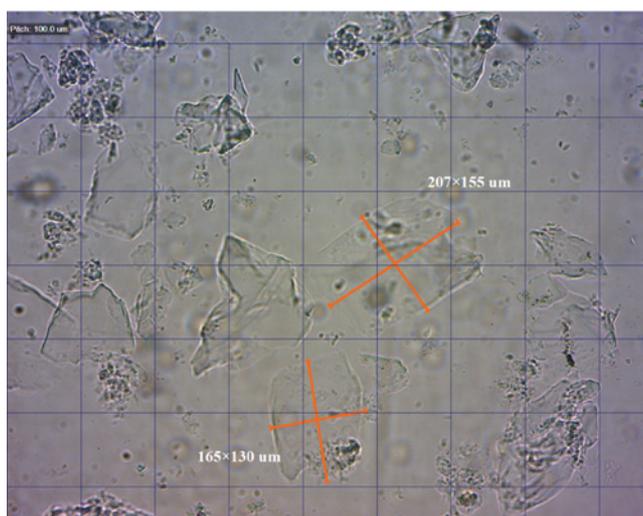
To measure the swelling ratio, one gram of dried PPGs ( $W_d$ ) was slightly soaked into a test tube that was filled with water of a desired salinity. Then, the test tube was placed into an oven at a preset temperature. The interface of PPGs and pure brine was monitored up to a point wherein no more swelling occurred. At that point, the PPG solution was poured into a wet paper strainer of pre-known mass to eliminate the excess water. Consequently, the final mass of the swelled PPGs ( $W_s$ ) was measured by laboratory balance. All experiments were accomplished several times until two successive masses are equal within the limits of the experimental error of 1%. The swelling ratio was obtained from Eq. (1):

**Table 1. List of chemicals used to prepare the PPGs**

Name	Functionality	Purity (%)	PPG Ingredients		Made by
			Weight percent	Molar mass ratio	
Acrylamide	Monomer	98.5	30	2	Beijing Chemical (China)
N,N-Dimethyl Acrylamide	Monomer	98.5		1	
2-Acrylamido-2-Methylpropane Sulfonic Sodium Salt	Monomer	99.0		2	
N-Vinylpyrrolidone	Monomer	98.0		1	
N,N'-Methylenebis (Acrylamide)	Crosslinking Agent	99.0	0.55		
N,N',N,N' Tetramethylethylenediamine	Catalyst	99.0	0.05		
Sodium Persulfate	Initiator	99.0	0.1		
Nano Clay Montmorillonite Na <sup>+</sup>	Mechanical Property Modifier	99.0	2.5	-	Aldrich (St Louis, MO, USA)
Nitrogen Gas	-	99.995	-	-	Delvar Afzar Gas Industrial group (Iran)



(a)



(b)

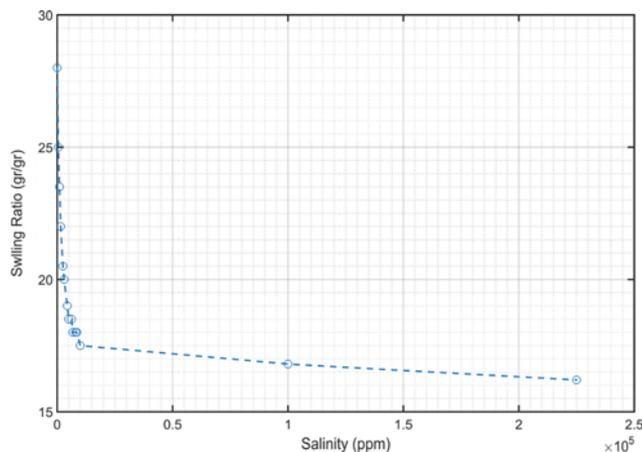
**Fig. 1.** The microscopic photos of (a) dry PPGs and (b) swelled PPGs after 24 hours. The swelling was done in distilled water at room temperature (298 K). The approximate diameters of swelled gel particles were increased around three times. The gridline spacing is 100  $\mu\text{m}$ .

$$\text{Swelling Ratio} = \frac{W_s - W_d}{W_d} \quad (1)$$

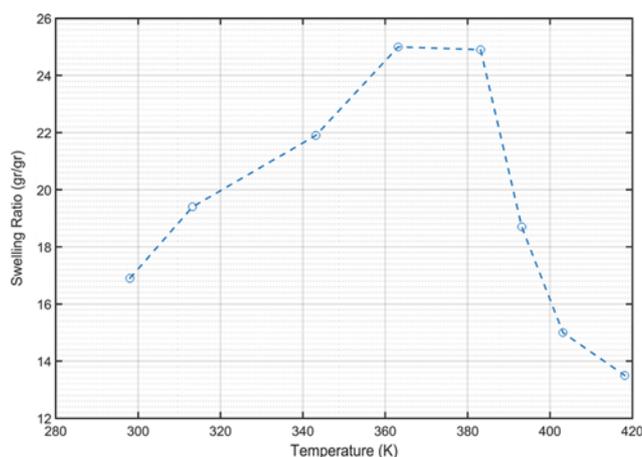
### 3. Design of Experiments

To investigate the swelling ratio response of the new enhanced PPGs in a wide range of temperatures and water salinities, we implemented the design of experiment (DOE) method. This statistical cost-effective method can be used to make a purposeful point selection and reduce the number of experiments [27]. The response of DOE was based on two key reservoir factors, temperature (T) and water salinity (S), as well as their interactions ( $\text{SR} = f(T, S)$ ). In addition, the results can be used to screen the effective factor (temperature or salinity) and reveal how they affect the response.

The two-level and three-level experimental designs are common methods in effect screening and the response surface model-



**Fig. 2.** The effect of NaCl concentration on swelling ratio of PPGs at room temperature (298 K).



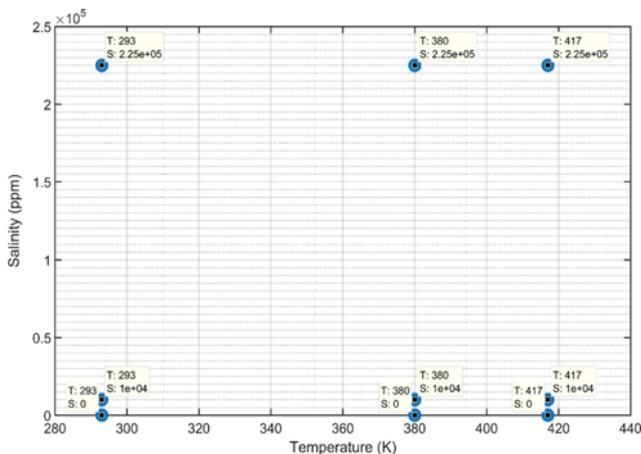
**Fig. 3.** The effect of temperature on swelling ratio of PPGs in the NaCl water salinity of 40,000 ppm.

ing of laboratory measurements [27,28]. However, to select an appropriate DOE method, to suggest the distribution of the test input points in 2-D space of T and S, and finally to specify the type of proposed proxy model, primary classical design should be used, in which one variable is changed at a time. The classical method is necessary if one seeks to understand the fundamental relationship between input data (in our problem: T, S) and output (in our problem: SR). In this study, the variations of temperature and water salinity (i.e., NaCl based salinity) were considered in the range of 298–418 K and 0–225,000 ppm, respectively.

The effect of water salinity changes at room temperature and the effect of temperature changes at a water salinity of 40,000 ppm are illustrated in Fig. 2 and Fig. 3. The response of swelling ratio to salinity has a logarithmic behavior (Fig. 2), while the response of swelling ratio to temperature is quadratic (Fig. 3). This classical design method helped us to select the important levels of factors in their domains (i.e., level 1 to 3 in Table 2). These levels influenced the response trend of the swelling ratio in Fig. 2 and Fig. 3; consequently, they should be examined in the modeling of it. These results also showed that to develop a proxy model that can predict

**Table 2. Parameters constraints for DOE**

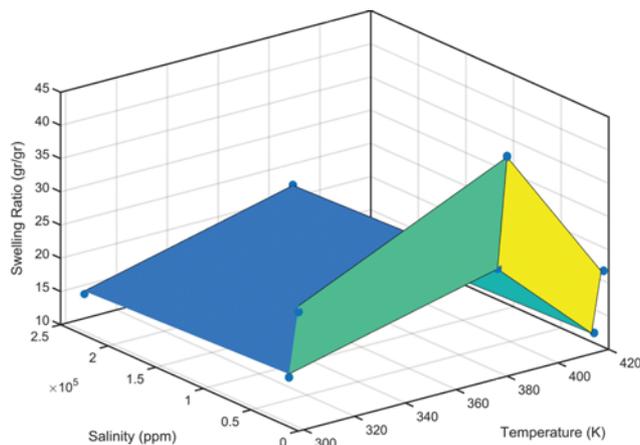
Factors	Unit	Level of factors		
		Min. (1)	Arbitrary (2)	Max. (3)
Water salinity (NaCl)	ppm	0	10000	225000
Temperature	K	298	380	418



**Fig. 4. Experimental conditions proposed by the three-level full factorial design method. The main experiments and their replications were done at nine different salinities and temperatures.**

the swelling ratio of PPGs, we should go beyond the two-level experimental designs. Subsequently, a three-level full factorial design, which is one of the most efficient design approaches, was used to present all possible combinations of this set of factors [29]. Table 2 shows low (1), arbitrary (2) and high (3) levels of T and S.

This design acts the same as a central composite design, except its central levels are set at arbitrary positions. To select the central levels in our experiments, the measurement results of Fig. 2 and Fig. 3 were considered. If the central level for salinity was routinely selected (as in the three-level central composite design) at the mean level (112,500 ppm) between minimum and maximum level of salinity, then the quick drop of swelling ratio between salinity points of 0-10,000 ppm could not be detected. The designed points for further analyses are shown in Fig. 4. Nine laboratory tests and



**Fig. 5. The results of swelling ratio measurements for different salinities at different temperature conditions.**

their replication ( $2 \times 3^2$  analyses) were conducted under a three-level full factorial design to fit a swelling ratio surface to these continuous factors.

**RESULTS AND DISCUSSION**

The swelling ratio of all experimental patterns of T and S (Fig. 4) was measured. The associated results are shown in Table 3 and Fig. 5. The least squares method was used to fit the response surface of swelling ratio as a model with water salinity, temperature, and their cross-interaction terms (Fig. 6). The results show a model with an  $R^2$  value of 0.97 and an adjusted  $R^2$  of 0.96, as Eq. (2):

$$SR = -4.574 \times 10^{-3} \times (T - 365)^2 - 5.243 \times 10^{-4} \times (T - 365) \times \ln(S) - 0.111 \times T - 0.742 \times \ln(S) + 77.648$$

$$0 < S \leq 225000 \text{ ppm}$$

$$298 \leq T \leq 418 \text{ K}$$

To show the accuracy of the proposed model, a plot of the actual swelling ratios versus predicted ones is shown in Fig. 7. The analysis of variance (ANOVA) was also implemented to state the significance of the model factors (Table 4). The large value of model sum of square, relative to small value of error sum of square, resulted in large F-ratio. Considering the F-ratio and the low proba-

**Table 3. Matrix of experiments for the full factorial design method**

Run	Pattern	Temperature (K)	Water salinity (ppm)	Swelling ratio (gr/gr)	
				1 <sup>st</sup> Replication test	2 <sup>nd</sup> Replication test
1	1,1	298	0	28.0	27.9
2	1,2	298	10000	17.5	17.4
3	1,3	298	225000	16.2	16.2
4	2,1	380	0	43.0	43.2
5	2,2	380	10000	25.5	25.7
6	2,3	380	225000	24.3	24.4
7	3,1	418	0	22.1	22.0
8	3,2	418	10000	12.1	12
9	3,3	418	225000	11.0	10.9

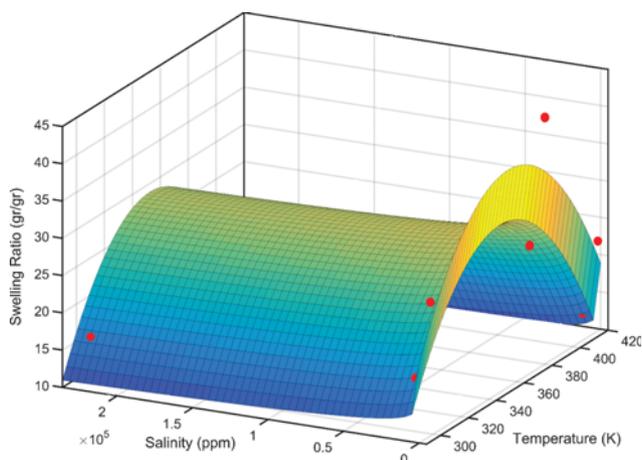


Fig. 6. Response surface of the proposed model of swelling ratio as a function of salinity and temperature. The red points in the figure show the measured results of the experiments.

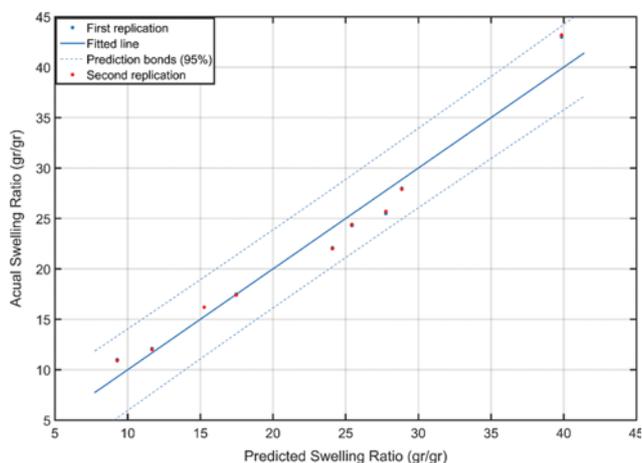


Fig. 7. Actual swelling ratio versus predicted swelling ratio. The  $R^2$  and adjusted  $R^2$  indicate that the regression line significantly fits the measured data ( $R^2=0.97$ , Adjusted  $R^2=0.96$ ).

Table 4. Analysis of variance of SR model

Model	Source	DF	SS	MS	F ratio	Prob> F
SR	Model	4	1498.1	374.5		
	Error	13	50.4	3.9	96.6	<0.0001
	C. Total	17	1548.5			

bility value of the fit, the model is highly robust.

Fig. 5 shows that by increasing salinity the swelling ratio decreases. It is proved that the hydrogel ability to absorb water is based on repulsive interactions between negatively charged groups in polymeric chains and hydrophilic groups connected to the polymer backbone; whereas, the resistance to dissolution arises from cross-links between polymeric chains [9,30,31]. By increasing the salt concentration, the interaction between the negatively charged polymeric chains with cationic metals results in an imperfect anion-anion electrostatic repulsion, directed to a decreased difference in osmotic pressure between the hydrogel network and the external

solution [32,33]. Thus, the volume of PPGs is reduced at higher salinities. Furthermore, the gel strength of PPGs swelled in a high-salinity solution is higher than that in a low-salinity solution. Higher gel strength results in a smaller swelling ratio [4,7,34]. Our results shown in Fig. 2 and Fig. 5 agree with these arguments.

The swelling ratio shows a quadratic behavior at different temperatures (Fig. 3). The swelling ratio of gels is controlled by the free energy of elasticity at constant salinity [10]. The negative dependence of the storage modulus to temperature is also observed in cross-linked hydrogels [35]. Therefore, in our case, up to 380 K, the swelling ratio increased by rising temperature.

However, above 380 K, the trend is reversed and the swelling ratio declines with temperature rising. Different explanations can describe such a behavior such as disentanglement of the remaining unreacted components (e.g., polymer chains) from synthesis, ionic cross-linking with divalent metal cations, weakening hydrogen bond, and thermal destruction.

To explain such behavior, we start with the explanation of fundamental junctions of hydrogel, i.e., chemical or physical junctions. Chemically cross-linked networks have permanent junctions, while physical networks have temporary junctions constructed from polymer chain entanglements or physical interactions such as ionic interactions, hydrogen bonds, or hydrophilic-hydrophobic interactions [30,35].

The hydrogel volume made from temporary junctions can be affected by unreacted components entanglements remaining from synthesis. The post-washing process of synthesized hydrogels with distilled water can remove a major amount of these unreacted components. However, if they remain in the system, then volume increasing of hydrogels due to temperature rise as well as the stress generated by the swelling may result in disentanglement of these components. This occurrence causes the trapped water between these chains to expel out from the gel network and reduce the hydrogel volume [30,36,37].

Ionic interaction is also one of the factors that can influence the swelling ratio of a hydrogel. The amide groups ( $-\text{CONH}_2$ ) of the PPGs hydrolyze to acidic carboxylic groups ( $-\text{COOH}$ ) at high temperatures, which enhances the posterior ionic crosslinking with divalent metal cations present in water and reduce the volume of the particle gel due to gel syneresis [3,37].

The volume change behavior of hydrogels in water can be influenced significantly by hydrogen bonding. By temperature rising, the entrapped water molecules in the network are exposed to free as the hydrogen bond interactions become weakened or destroyed and the interactions of hydrophobic carbonaceous groups become fully dominant [35,38,39].

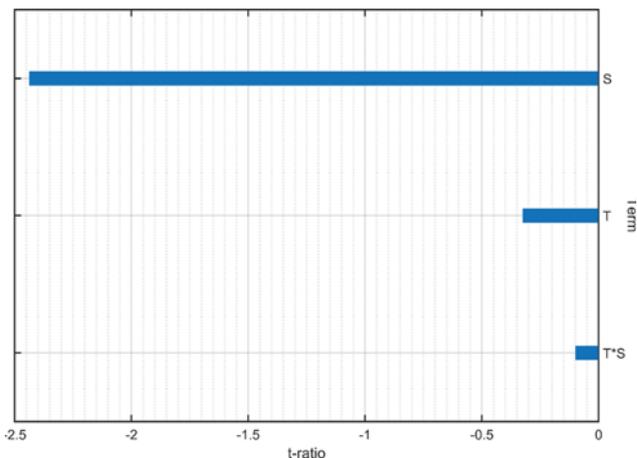
Furthermore, when a gel of Acrylamide monomers and  $N,N'$ -methylenebis (acrylamide) cross-linker is subjected to high thermal conditions, the release of acrylamide monomers and gel network destruction is started at 95 °C (368 K) [36]. The swelling behavior of PPGs at a temperature of 418 K is visualized in Fig. 8. The blue arrow in Fig. 8 shows the volume of PPGs at room temperature (298 K). The swelling process of PPGs at high temperature causes the PPGs to lose water (green arrow).

By analyzing the data, we selected the most effective factor influencing the swelling performance of PPGs between salinity and tem-

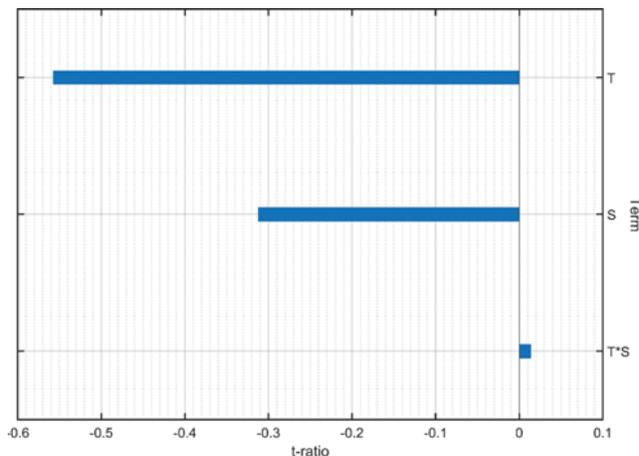


**Fig. 8.** Swelling ratio measurement of PPGs in NaCl-salinity of 225,000 ppm and different temperatures. (a) 298 K, (b) 418 K. The solid arrow shows the amount of swelled PPGs at room temperature (298 K). The rise of temperature causes the gel to undergo water expulsion and volume reduction (dotted arrow).

perature. The response of the swelling ratio to salinity consisted of two linear sections (Fig. 2). For that reason, the data space was divided into two smooth regions: one with  $S < 10,000$  ppm and the other with  $S > 10,000$  ppm. In each region, a simple polynomial model was fitted to salinity, temperature, and their interaction. The coefficients of the model show the statistical importance of the



**Fig. 9.** Pareto plot of parameter effects for  $S < 10,000$  ppm shows that swelling ratio is more depended on water salinity in low water salinity region.



**Fig. 10.** Pareto plot of parameter effects for  $10,000 < S < 225,000$  ppm shows that swelling ratio is more depended on the temperature in high water salinity region.

model terms. The coefficients should be made orthogonal to be uncorrelated and have equal variances [27]. Afterward, the mean and standard deviation of each coefficient were used to calculate a t-test. Finally, the factors were sorted by the absolute value of the t-ratio, placing the most significant on the top. The results are presented in Fig. 9 and Fig. 10.

The results of this analysis show that water salinity is the most effective factor in low water salinity conditions and higher water salinity results in a smaller swelling ratio. By increasing salinity, a quick reduction of the swelling ratio is observed in the low concentration region. This behavior can be described by the mitigation of large repulsion forces between negatively charged groups in polymeric chains and their low attraction toward water in the presence of positive ions [3]. On the other hand, raising the salt concentration beyond a certain value (10,000 ppm) has no remarkable effect on the swelling ratio of PPGs (Fig. 2). The reason is that the anionic fragments in the polymeric network are the limiting reagents for the ionic crosslinking process. When all negatively charged groups of the network participate in the ionic interactions, the swelling ratio does not decrease while the concentration of metallic ions increases. These arguments agree with the logarithmic term of salinity in Eq. (2). In this case, temperature becomes the most important factor, because of its effects on the elastic modulus of gel and polymer chain junctions [3,32,35].

**SUMMARY AND CONCLUSIONS**

A laboratory analysis was conducted to evaluate the swelling ratio performance of new enhanced PPGs in a wide range of salinity and temperature conditions. This analysis was organized through performing the classical and the flexible full factorial experimental design approaches to cover the entire feasible experimental conditions and select the representative states. The swelling ratio of the PPGs was accurately predicted by a precise mathematical model. The more sophisticated and statistical results reveal that the swelling ratio of the PPGs is dependent on its surrounding environmental conditions. The swelling ratio decreases by increasing water

salinity. Moreover, the swelling ratio increases by raising reservoir temperature up to 380 K and then starts to decrease. The results illustrate that swelling ratio is more dependent on water salinity in the low water salinity region and more dependent on temperature in the high water salinity region.

## NOMENCLATURE

AM	: acrylamide
AMPSNa	: 2-acrylamido-2-methylpropane sulfonic sodium salt
DA	: N,N-dimethyl acrylamide
DF	: degree of freedom
MBA	: N,N'-methylenebis (acrylamide)
MS	: mean square
NVP	: N-vinylpyrrolidone
S	: water salinity
SR	: swelling ratio
SS	: sum of squares
T	: temperature
$W_d$	: mass of dried PPGs
$W_s$	: mass of swelled PPGs

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