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Advanced Investigation of Hydrolyzed Polyacrylamide Gels with High-Purity Silica Nanoparticles under Fractured Reservoir Conditions

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Abstract

Silica nanoparticles have shown strong potential to enhance gels for water shut-off applications. However, previous studies have largely relied on static tests with hydrophilic silica nanoparticles, and their behavior under dynamic fractured-reservoir conditions remains poorly understood. In this study, hydrolyzed polyacrylamide (HPAM) gels reinforced with high-purity silica nanoparticles (HPSN) were systematically evaluated under static and core-scale dynamic conditions representative of fractured reservoirs. Static tests examined gelation, rheological properties, thermal stability, nanoparticle dispersion, and swelling, while fractured-core flooding experiments assessed dynamic performance. Results showed that increasing HPSN concentration shortened gelation time. Rheological analysis indicated that at a low HPSN concentration of 0.2 wt.%, where dispersion was more uniform, the storage modulus decreased while viscoelasticity increased; at a higher concentration of 0.4 wt.%, the opposite trend was observed. Increasing HPSN concentration also reduced gelant viscosity, with dispersion quality significantly affecting this behavior. Long-term thermal stability tests at 90°C and DSC tests showed that high HPSN concentrations increased bound water content and initially improved thermal syneresis, but eventually caused over-crosslinking due to progressive HPAM hydrolysis. By comparison, low HPSN concentrations provided slightly better thermal stability despite lower bound water content. Swelling behavior and dynamic flooding tests were done for the 0.2 wt.% HPSN gel. In swelling tests conducted in formation water and crude oil at 90°C, 0.2 wt.% HPSN initially improved syneresis resistance in formation water but had no significant effect on the final equilibrium swelling ratio in either fluid. Dynamic flooding tests confirmed differences between static and dynamic gelation, with residual resistance factors of 58.2 in formation water and 28.2 in crude oil after 72 hours.

Unintended water production, along with crude oil, presents a significant challenge in the oil industry, particularly affecting mature reservoirs. Managing and separating substantial amounts of water incurs high costs and poses environmental concerns. High volumes of produced water from permeable layers, coupled with a tendency for emulsification, exacerbate the problem, leading to a reduced reservoir lifespan¹⁻³. Various mechanical and chemical techniques exist to mitigate water production, but recent advancements include using gels to reduce water production⁴⁻⁶. This method offers a cost-effective and safe operational approach⁷⁻⁹. Gel technology is a versatile Enhanced Oil Recovery (EOR) method applicable in injection and production wells. In injection wells, gels block high-permeability layers or fractures, redirecting water to inswept zones. In production wells, injecting gels into fractures or high-permeability zones reduces excessive water production, drilling fluid loss, enhances pressure drawdown, controls sand production, and lowers water management costs^{6,10-16}.

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Among gels, those based on Hydrolyzed Polyacrylamide (HPAM) with organic crosslinkers are frequently used for water shut-off due to their favorable performance under complex reservoir conditions. These gels are particularly well-suited for high-temperature applications, owing to the covalent bonds formed between their components^{17–19}. Additionally, the gelation time can be adjusted by modifying the polymer-to-cross-linker ratio and the concentration of additives^{18,20}. Researchers^{21,22} have noted a significant delay in gelation and stability within the temperature range of 70 to 140°C. These gels show improved rheological properties, including a higher storage modulus, compared to gels relying on ionic bonding²³. However, HPAM is susceptible to hydrolysis at temperatures above 60°C, which converts its acrylamide groups into carboxylic groups²⁴, which can cause the over-crosslinking of the gel over time. Particularly in saline environments, this chemical transformation triggers syneresis—a process where excessive attractive forces expel water from the gel’s three-dimensional polymer network²⁵.

To further enhance HPAM gels, recent research shows that incorporating nanoparticles can significantly improve their properties under harsh reservoir conditions^{26–33}. Among these, silica nanoparticles are commonly employed, with the majority of studies focusing on hydrophilic silica. However, the distinct surface properties and functional behaviors of hydrophilic and high-purity silica nanoparticles (HPSN) lead to different effects on HPAM gel systems, as summarized in Table 1. While considerable attention has been given to hydrophilic silica, the role of HPSN in HPAM-based gels remains notably underexplored—particularly under the high temperature and salinity conditions of fractured reservoirs. Furthermore, most existing investigations are limited to static experiments, which fail to replicate the dynamic flow conditions in fractured reservoirs. This study addresses an existing research gap by comprehensively investigating the influence of HPSN on gelation behavior, rheological properties, thermal stability, nanoparticle dispersion, swelling behavior, and core flooding performance of fractured cores. These experiments provide additional insight into the role of HPSN in enhancing HPAM gel behavior, thereby enabling the rational and field-relevant design of advanced gel systems for effective water shut-off in fractured reservoirs under harsh conditions.

References	Polymer/ Crosslinker	Nanoparticle Type	Nanoparticle Concentration (wt.%)	Result
Yang Q et al. ³⁷	HPAM/Organic zirconium	High purity silica	0-0.1	Silica nanoparticles increase network density and adsorb onto the polymer, enhancing chain strength.
Pérez-Robles S et al. ³⁸	HPAM/Resorcinol, Formaldehyde	High purity silica	0-0.01	Silica nanoparticles negatively impact thermal syneresis but have no significant effect on stiffness.
Asadizadeh S et al. ³⁹	HPAM/Cr ³⁺	High purity silica	0-0.2	Silica nanoparticles increase the intensity of the gel network, enhancing thermal stability.
Guo H et al. ³⁴	HPAM/Water-soluble phenolic resin	Hydrophilic silica	0-1.5	Silica improved gel performance by preventing dehydration and oxidation, ensuring uniform polymer dispersion, and boosting strength up to an optimal concentration threshold.
Wu Q et al. ³⁵	HPAM/Water-soluble phenolic resin	Hydrophilic silica	0.1	Silica preserved gel integrity by preventing syneresis and improved thermal stability and increased the storage modulus, all without having any noticeable effect on gelation time.
Wu Q et al. ³⁶	HPAM/Water-soluble phenolic resin	Hydrophilic silica	0-0.4	Silica increased the gel’s strength and thermal stability while expanding the polymer solution’s hydrodynamic radius and inhibiting polymer degradation, all without impacting the gelation time.

Table 1. Review of research on the effects of high-purity and hydrophilic silica nanoparticles on HPAM gel.

Materials and methods

Materials

In this work, an HPAM polymer with an average molecular weight of 12000–15000 kg/kmol and a negative charge was supplied by Isfahan Copolymer Company. Hexamethylenetetramine (HMTA) was used as the crosslinking agent, and hydrochloric acid (HCl, 37%) was employed as the activator, both purchased from Sigma-Aldrich. Additionally, HPSN with an average diameter of

8 nm was provided by the US Research Nanomaterial Company. The crude oil utilized in this study was sampled from an oil reservoir located in southwestern Iran. The formulations for the synthetic base water used to prepare the gelant and the formation water are listed in Table 2 and Table 3, respectively. Furthermore, as the literature indicates that the presence of thiourea can significantly influence gel performance⁴⁰, it was added at 1 wt.% to act as an antioxidant and reduce gel syneresis at elevated temperatures in all gel samples⁴¹.

Ions	Concentration (mg/L)
Sodium	4750
Calcium	900
Chloride	9480
TDS	15130

Table 2. Composition of synthetic base water.

Ions	Concentration (mg/L)
Sodium	52375
Calcium	8800
Chloride	105615
Magnesium	1700
Sulfate	1440
Carbonate	0
Bicarbonate	390
Hydroxide	0
TDS	170320

Table 3. Composition of formation water.

Solution preparation

The gelants were prepared in three sequential steps, which involve dispersing HPSN in synthetic base water using a magnetic stirrer (IKA CMAG MS4) capable of mixing the solution at 400 rpm. This stirring process typically lasts 2 hours to ensure that HPSN is evenly distributed. Next, a polymer solution consisting of HPAM, HMTA, HCl, and thiourea in synthetic water is prepared at 400 rpm for 2 hours. Finally, the polymer solution and HPSN fluid are mixed and stirred using a mechanical stirrer at 100 rpm for another 2 hours.

Experimental approaches

Gelation process tests

Firstly, Fourier transform infrared spectroscopy (FTIR; PerkinElmer Spectrum) of dry HPSN and two gels with and without HPSN was used to analyze the reaction of HPSN with gel. Subsequently, gelation time, which is essential in determining the quality and performance of the gel, especially for downhole applications, was measured. The gelation time refers to the time at which the gel has completely formed and reached its final strength. In another definition, gelation time is when the gel's viscosity increases significantly as temperature increases⁴². In this study, the bottle test was used to determine the gelation time and strength of gels. This test is performed qualitatively, based on the Sydansk coding system⁴³. In this system, the gel state is classified into nine categories, from code A to code I, based on the gel sample's flowing conditions and suspension height, as shown in Fig. 1.

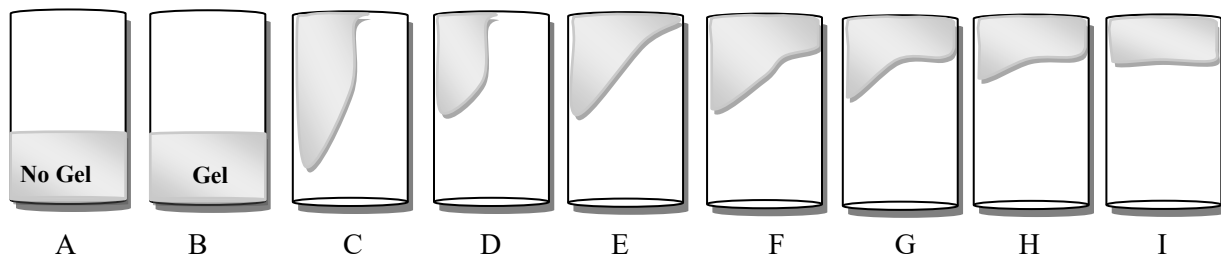


Fig. 1. Sydansk gel-strength code classification: Code A (no gel), B (highly flowing), C (flowing), D (moderately flowing), E (barely flowing), F (highly deformable nonflowing), G (moderately deformable nonflowing), H (slightly deformable nonflowing), and I (rigid gel).

Rheological properties tests

The gelant viscosity was measured at various shear rates at 25°C using the Brookfield viscometer (RVDV-II + Pro) with a coaxial cylinder setup, where the sample cup has an inner diameter of 42 mm, and the rotor had a diameter of 39 mm.

The rheological characteristics of the gel samples were assessed using a rheometer (MCR 300, Anton Paar). The tests were conducted at 25°C using a parallel-plate system with a 20 mm diameter and a 1 mm gap. The gel's strength was assessed by measuring its storage modulus (G') across a strain range from 1.31% to 1480% at a frequency of 1 Hz.

Thermal stability tests

The gel thermal syneresis over time at a temperature of 90°C was assessed through weight measurement using the following equation (Eq. 1)⁴⁴:

$$\%Syneresis = \left(\frac{m_1 - m_2}{m_i} \right) \times 100 \quad (1)$$

Here, m_1 represents the weight of the gel placed inside the bottle, m_2 is the weight of the bottle after removing the water, and m_i is the weight of the gel itself, excluding the weight of the bottle. It is worth noting that, after removing each gel bottle sample from the oven, water in the bottle was extracted using a syringe, and the weight of the gel sample was measured. Subsequently, the samples were sealed and returned to the air-forced oven setup at 90°C.

The DSC measurements of samples were characterized using a Differential Scanning Calorimeter (DSC; STA 449 F3, Netzsch). Before testing, Nitrogen was filled in the test environment, 29.5 mg of the samples were placed in a hermetic pan, and the temperature was raised from -70 to 180°C stepwise at 10°C/min. The amount of free/bound water obtained from the DSC test by the area under the exothermic peak in the temperature range of 0°C by the following formula (Eq. 2)^{41,45}:

$$W_f = \frac{\Delta H_f}{\Delta H_0} \times 100 \quad (2)$$

ΔH_f : Melting enthalpy of free water in gel structure

ΔH_0 : Melting enthalpy of bulk water (333.5 J/g)

The amount of bound water can also be obtained after obtaining the percentage of free water using the formula below.

$$W_B = 100 - W_f \quad (3)$$

Dispersion tests

The dispersion of the HPSN in the gelant samples was assessed using Dynamic Light Scattering (DLS; VASCO, Cordouan Technologies). Before testing, 1 gram of gelant was mixed with 15 mg of deionized water and stirred at 400 rpm for 30 minutes. Subsequently, the samples were tested at 25°C.

Micro-morphology measurements of gel samples were carried out using a Scanning Electron Microscopy (SEM; AIS2100, Seron Technologies) device. Initially, the gel samples were frozen at a negative temperature and then dried using a vacuum device. Then, the cross-sectional surface of the sample was photographed.

Swelling tests

Swelling of the gel was investigated in different solutions: formation water and crude oil (all at 90°C), where approximately 5 grams of gel were placed in 100 mL of the selected solution. Before weighting, the surfaces of the gel samples were dried using soft paper^{46,47}. The gel Equilibrium Weight-Swelling Ratio (EWSR) was calculated with the following equation (Eq. 4).

$$\%EWSR = \left(\frac{W_s(t) - W_g}{W_g} \right) \times 100 \quad (4)$$

Where, $W_s(t)$ represents the swollen weight of the same sample at time (t) and W_g is the weight of the sample before swelling⁴⁸.

Fractured porous media core flooding tests

A fractured porous medium, whose properties are provided in Table 4, was engineered by cutting a core and creating a controlled fracture using thin, narrow aluminum sheets placed along its length. This engineered core enabled a dynamic laboratory evaluation of gel performance under conditions that closely simulate fractured reservoir conditions.

Core characteristic	Value
Diameter (cm)	3.81
Length (cm)	7.6

Fracture opening (mm)	0.1
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Table 4. The characteristics of the fractured core.

A schematic diagram of the laboratory setup is depicted in Figure 2, and the procedure for the core flooding experiments, conducted at a temperature of 90°C, is detailed below.

- (1) The fractured core sample is initially fully saturated with formation water at a 0.1 cc/min flow rate.
- (2) Before the optimized HPSN gel treatment, crude oil and formation water were injected into the core at a 0.1 cc/min flow rate, respectively. Then, the effective permeability was measured (K_{eo1} for crude oil and K_{ew1} for formation water).
- (3) The optimum HPSN gelant was injected into the fractured core, followed by a 72-hour aging period. To monitor the gel's behavior during curing, two samples: one from the core effluent optimum HPSN and the other from an in situ prepared one, were placed in an oven at 90°C for visual observation.
- (4) After the optimized HPSN gel treatment, crude oil and formation water were injected at the same 0.1 cc/min rate, respectively. Then, the effective permeability (K_{eo2} for crude oil and K_{ew2} for formation water after the optimized HPSN gel treatment) was measured to evaluate the performance of the gel.

Finally, RRF is defined as the ratio of the mobility of formation water (or crude oil) before gel treatment to the same parameter after gel treatment was measured⁴⁹. The following equations present the RRF formula for the formation water and crude oil phases:

$$RRF_w = \frac{K_{ew1}}{K_{ew2}} \quad (5)$$

$$RRF_o = \frac{K_{eo1}}{K_{eo2}} \quad (6)$$

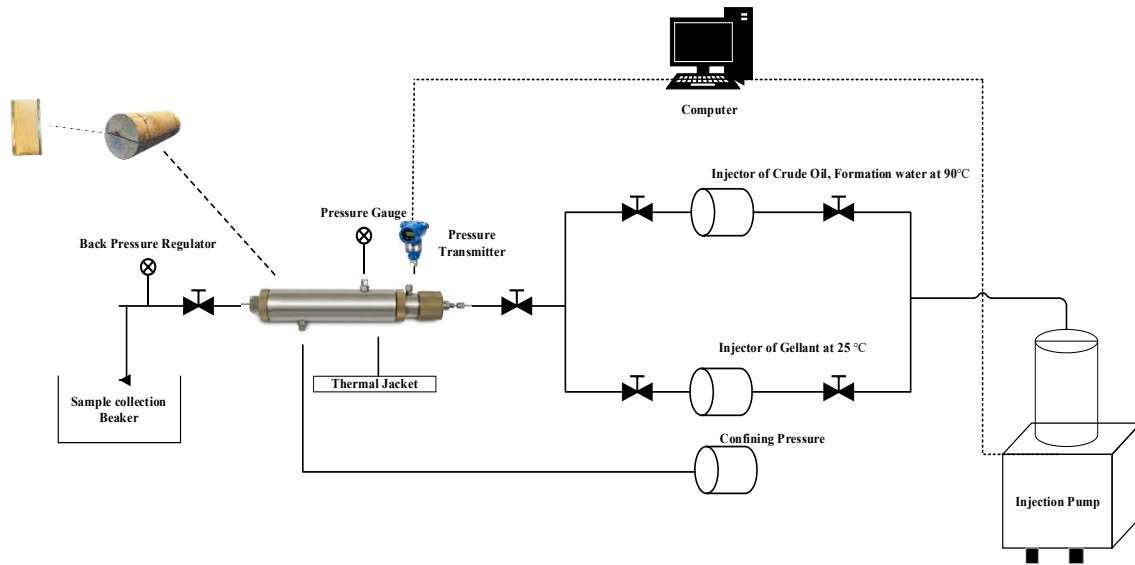


Fig. 2. Schematic of the laboratory core flooding setup.

Results and discussion

Gelation process

The lone pair of electrons on the nitrogen atom in HMTA can be donated to the empty orbital of the proton from HCl, forming a coordinate covalent bond. This interaction generates a positive charge on nitrogen, which is stabilized by the withdrawal of the bonding electrons in the adjacent C – N bond toward the more electronegative nitrogen atom, leading to the formation of an iminium ion intermediate (Fig. 3(a)). In an acidic medium, this iminium ion can react with nucleophilic groups, such as negatively charged sites produced during polymer hydrolysis, which helps explain the gelling behavior of HPAM with HMTA. For effective gel-network formation, the iminium ion must be regenerated on the same HMTA molecule. As shown in step a of Fig. 3, the iminium ion is re-formed. Upon reaction with other nucleophilic groups on neighboring polymer chains, crosslinking occurs as illustrated in step b⁵⁰, resulting in a three-dimensional gel network. In addition, if the synthetic water or the water

contacting the gel contains mono- or divalent cations, these ions may also interact with the carboxylate groups on the polymer chains through ionic association, further influencing the gel structure.

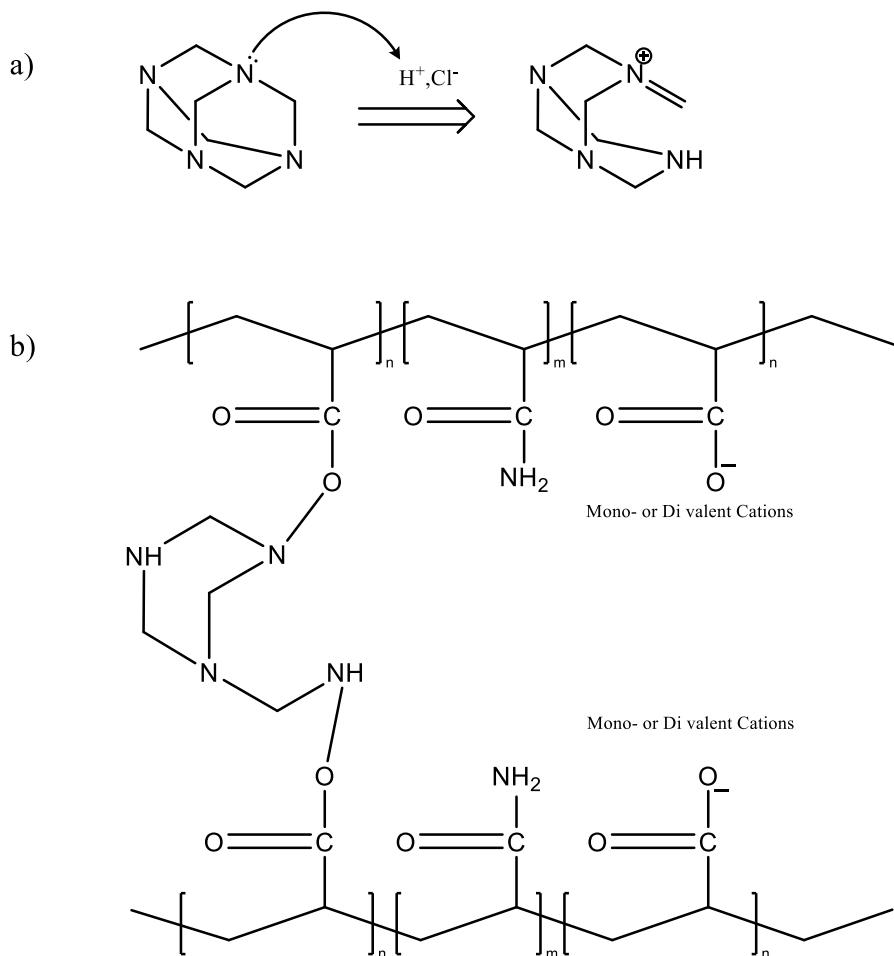


Fig. 3. Proposed reaction mechanism of hydrolyzed polyacrylamide with hexamethylenetetramine, hydrochloric acid, and mono- and divalent cations.

To investigate the interaction of HPSN with this HPAM gel, two types of dried gels and a sample of HPSN were subjected to FTIR. Fig. 4 shows the FTIR tests for nanoparticle-free gel (Nanoparticle=0), a gel with 0.2 wt.% nanoparticles (Nanoparticle=0.2), and a sample of High Purity Silica (HPS) nanoparticles. As the HPS curve in Fig. 4 shows, peaks for three groups near 1100 cm^{-1} , 800 cm^{-1} , and 480 cm^{-1} are observed, which indicate stretching and bending vibrations for normal Si – O bonds observed in all silica polymorphs. However, the nanoparticle-free gel shows no peak points in this range. Also, the 1632 cm^{-1} region on the HPS curve corresponds to the stretch of C = O and the bending of N – H, indicating the presence of alkyl and amide groups in the nanoparticle structure. Moreover, another peak for the nanoparticle is observed at 3400 cm^{-1} , indicating the presence of hydroxyl groups in the nanoparticle structure. Although this peak is not considerable, hydroxyl groups may lead to bonding with carboxylate or amide groups of hydrolyzed polymers through hydrogen binding^{51,52}. The decrements of peak near 3400 cm^{-1} for the gel containing 0.2 wt.% HPSN suggests stronger interactions between HPSN and the HPAM chains, which may reduce amide-group hydrolysis and enhance gel stability. The peak at 2923 cm^{-1} is attributed to C–H stretching vibrations of alkyl groups on the nanoparticle surface, indicating surface functionality that may interact with the polymer matrix.

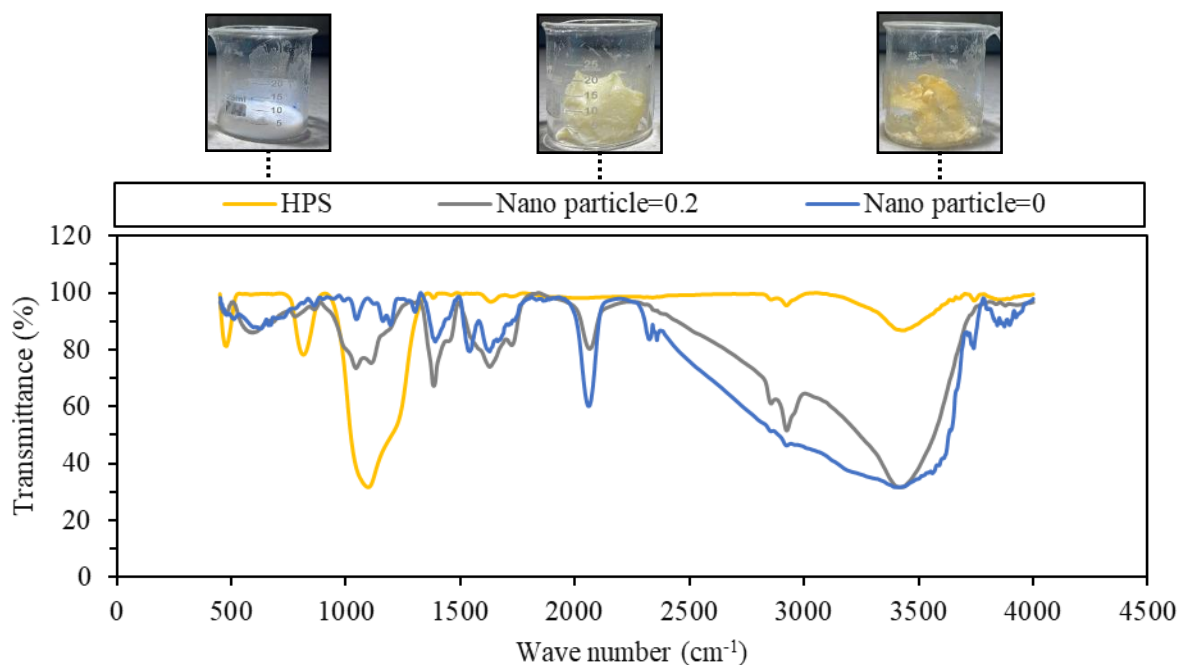


Fig. 4. FTIR spectrum analysis of High Purity Silica (HPS) nanoparticles, gel without nanoparticles (Nanoparticle=0), and gel with 0.2 wt.% nanoparticles (Nanoparticle=0.2).

To further investigate the gelation process, the bottle test to investigate the HPSN on the gelation time was performed. The impact of nanoparticles on the gelation time of the gel varies based on factors like the type and concentration of nanoparticles^{34–39}. Investigations into the impact of HPSN concentration (ranging from 0 to 0.4 wt.%) on gelation time revealed a consistent reduction in gelation time with increasing nanoparticle concentration. The gel containing 0.2 wt.% and 0.4 wt.% of nanoparticles achieved Code I strength within 24 and 15 hours of gelation, respectively, as shown in Table 5. In contrast, the gel with 0 wt.% only reached Code H after 48 hours, as illustrated in Fig. 5.

Nanoparticle (wt.%)	0	0.2	0.4
Gelation time (hr)	48	24	15
Gel strength (Sydansk Code)	H	I	I

Table 5. Effect of high purity silica nanoparticle concentration on HPAM gel gelation time at 90°C.

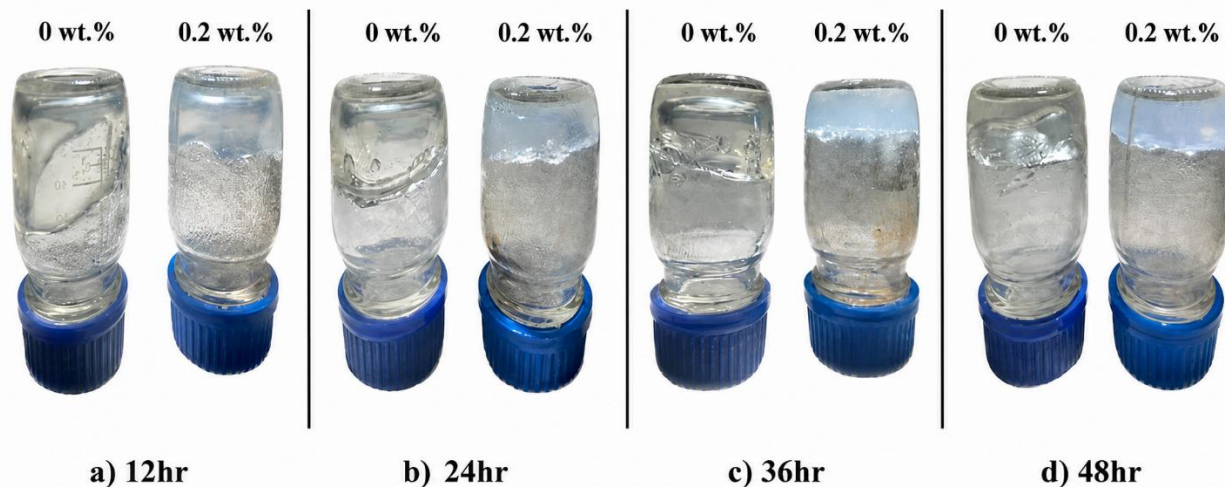


Fig. 5. The gelation time for HPAM gel with 0 wt.% (left) and 0.2 wt.% (right) high purity silica nanoparticles over different time intervals at 90°C.

Rheological properties

Since a low-viscosity gelant is essential for successful injection into porous rock formations⁵³, the effect of HPSN on the viscosity of HPAM-based gelants was investigated. Viscosity measurements were carried out at different nanoparticle concentrations (ranging from 0 to 0.4 wt.%) under shear rates of 1.32–44 s⁻¹ at 25°C. As shown in Fig. 6, increasing the nanoparticle concentration to 0.2 wt.% led to a pronounced reduction in gelant viscosity, decreasing from 15837 to 10558 cP at a shear rate of 1.32 s⁻¹. In contrast, further increasing the nanoparticle concentration from 0.2 to 0.4 wt.% caused the viscosity to rise, reaching 12317 cP at the same shear rate. To further investigate this behavior, logarithmic shear stress was plotted against logarithmic shear rate (Fig. 7). The results were consistent with shear-thinning behavior, which is typically described by the power-law model^{39,54}:

$$\tau = K \cdot \gamma^n \quad (7)$$

Taking the logarithm of equation (7) gives:

$$\log \tau = \log K + n \cdot \log \gamma \quad (8)$$

Where τ is the shear stress, K is the consistency coefficient, γ is the shear rate, and n is the power-law exponent. This model is widely applied to classify flow behavior: for $n = 1$, the fluid is Newtonian; for $n < 1$, it exhibits pseudoplastic (shear-thinning) behavior; and for $n > 1$, it exhibits dilatant (shear-thickening) behavior. As shown in Fig. 7, as the percentage of nanoparticles increases, the value of n also grows. In fact, with an increase in the nanoparticle content to 0.4 wt.%, the gel state shifts from regular to less homogeneous. An important point to note is that nanoparticles with 0.2 wt.% have improved the shear-thinning property, even better than that of the gel without nanoparticles.

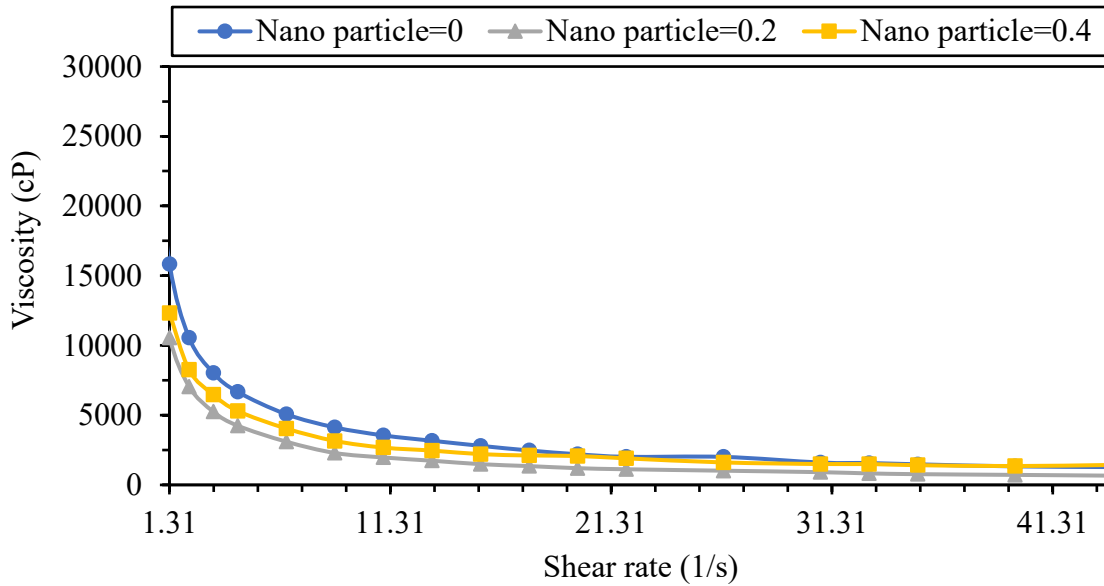


Fig. 6. The effects of *high purity silica* nanoparticle concentrations of 0, 0.2, and 0.4 wt.% on the viscosity of the HPAM gelant at different shear rates at 25°C.

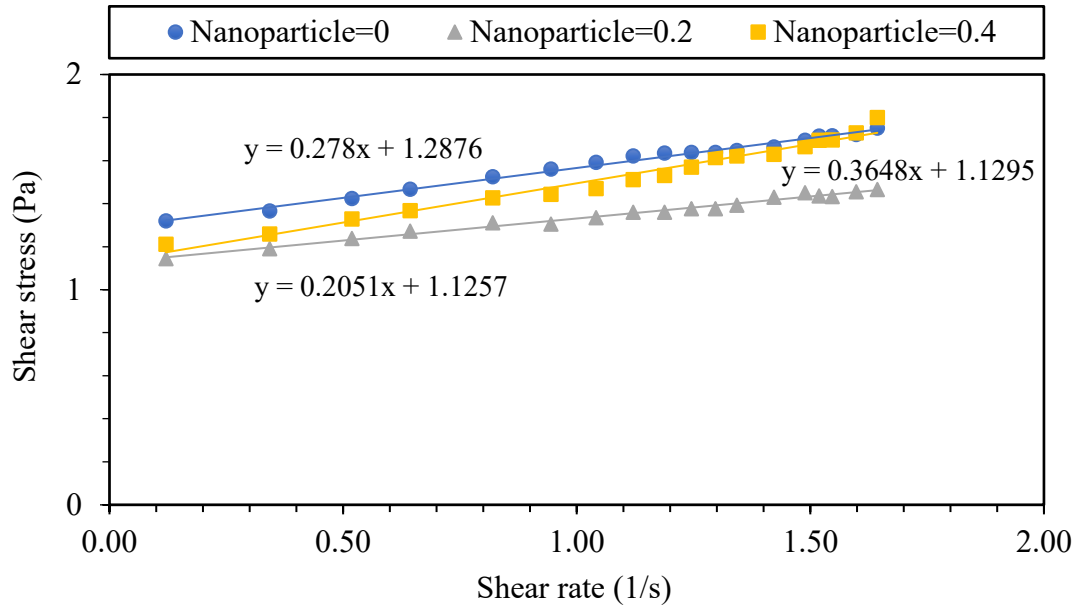


Fig. 7. The relationship between shear stress and shear rate of 0, 0.2, and 0.4 wt.% of high purity silica nanoparticle concentration on HPAM gelant at 25°C

The rheological properties of the gel were investigated using the strain sweep tests of HPAM gels containing HPSN at 25°C, with HPSN concentrations of 0, 0.2, and 0.4 wt.% and strain levels ranging from 1.31% to 1480%. Strain sweep tests were conducted to evaluate the storage modulus, which reflects the elastic properties of the gel and its ability to store energy under applied deformation. In the strain sweep test plots, there is a viscoelastic region in which the storage modulus remains constant⁵⁵. Within this region, there is a linear relationship between the applied force and the resultant deformation, signifying that there is no slippage between gel layers and that the gel's response is independent of its size or the degree of deformation. Beyond the viscoelastic region, the gel begins to deform and eventually fractures. Gels with heterogeneous networks often demonstrate suboptimal performance, even when exhibiting a high storage modulus. This indicates that a high storage modulus alone is insufficient for defining a robust gel³⁵. In fractured reservoirs, the performance of gels is significantly influenced not only by their initial strength but also by their response to applied strain⁵⁰. Before strain sweep tests, to ensure consistency, all tested gels were allowed to stabilize for 7 days. As shown in Fig. 8, the results revealed that increasing the HPSN concentration to 0.2 wt.% reduced the storage modulus from 35.9 to 22.5 Pa, while the viscoelastic region expanded significantly up to 235% (compared to 69.3% for the gel without nanoparticles). In contrast, at 0.4 wt.%, the storage modulus increased from 35.9 to 72.2 Pa, and the viscoelastic region decreased to 20.8%.

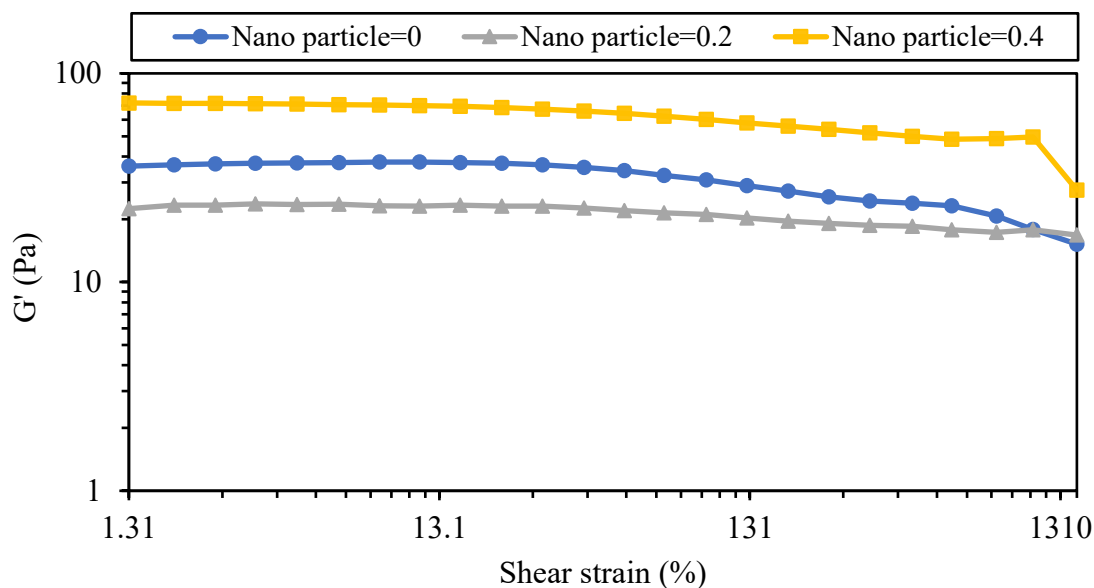


Fig. 8. The effects of 0, 0.2, and 0.4 wt.% high purity silica nanoparticle concentration on the storage modulus of the HPAM gel at 25°C.

Thermal stability

Syneresis is a process in which a gel network shrinks due to strong chemical attractions between the polymer and the crosslinker, causing water to be expelled from the gel. This phenomenon occurs when there is an imbalance between the amount of crosslinker and polymer. Adding too much crosslinker leads to excessive bonding, while adding too little results in weak gel strength. The excess crosslinking sites can be generated by intentionally adding a chemical to the gel or by hydrolysis of the polymer⁵⁶. As shown in Fig. 9, the effect of nanoparticle concentration on syneresis over time was investigated at concentrations of 0, 0.2, and 0.4 wt.%. The results indicated that increasing the nanoparticle concentration to 0.4 wt.% leads to a significant initial reduction in syneresis. This reduction is attributed to the nanoparticle-induced enhancement of the gel's hydrophilicity, which effectively suppresses syneresis. However, over time, due to the presence of a high percentage of crosslinker and progressive hydrolysis of HPAM, syneresis is promoted. The critical point is that nanoparticles at 0.2 wt.% appear to interact with the polar functional groups of the HPAM polymer, reducing the polymer's polarity and hydrophilicity. This interaction likely decreases the polymer percentage and initially contributes to increased syneresis at 0.2 wt.% of nanoparticles. However, due to the absence of excessive crosslinking, the syneresis remains constant even after 27 days.

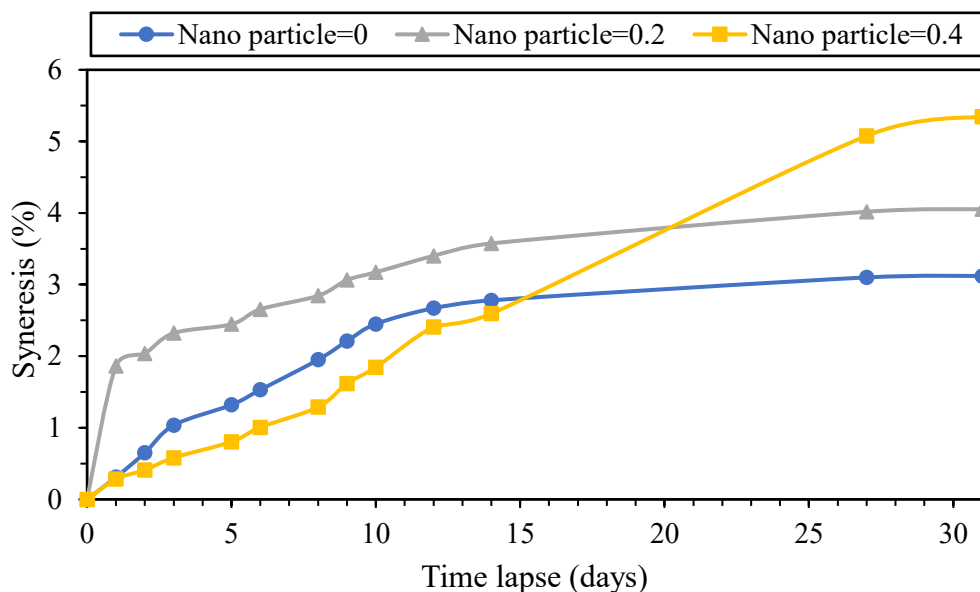


Fig. 9. Syneresis development in HPAM gel with high purity silica nanoparticle concentrations of 0, 0.2, and 0.4 wt.% at 90°C.

To further study the gel's temperature resistance with different HPSN concentrations and validate the results of Fig. 9, a DSC test was conducted within the range of -70 to 180°C (Fig. 10). The results revealed that the peak inflection points for nanoparticle concentrations of 0, 0.2, and 0.4 wt.% are 104.8, 108.3, and 108.0°C, respectively. For temperature ranges beyond the inflection point of this peak, gel structures experience a rapid decrease in heat flux due to the evaporation of all water molecules present in the gel. This implies an increased absorption of energy from chemical bonding threshold energies, which can lead to the breakage of chemical bonds, the initiation of heat dissipation, and the destruction of the gel structure. As is obvious from the use of HMTA as an organic crosslinking agent, the gel exhibited high-temperature capabilities. This is attributed to covalent bonds in the structure of these crosslinking agents, which have strong bonds⁵⁷. Moreover, the results revealed that combining HPAM gel and HPSN enhanced thermal stability in the gel system by improving lateral interactions, heat distribution, and preventing gel matrix breakdown. The slightly lower thermal tolerance at 0.4 wt.% compared to 0.2 wt.% might be due to uneven nanoparticle distribution. Also, the free and bonded water content was calculated from the DSC results. The results, as provided in Table 6, revealed that bonded water increased from 43.8% to 56.8% at 0.4 wt.%, compared to a decrease from 43.8% to 40.5% at 0.2 wt.% relative to the gel without nanoparticles. These findings were consistent with the results of Fig. 9.

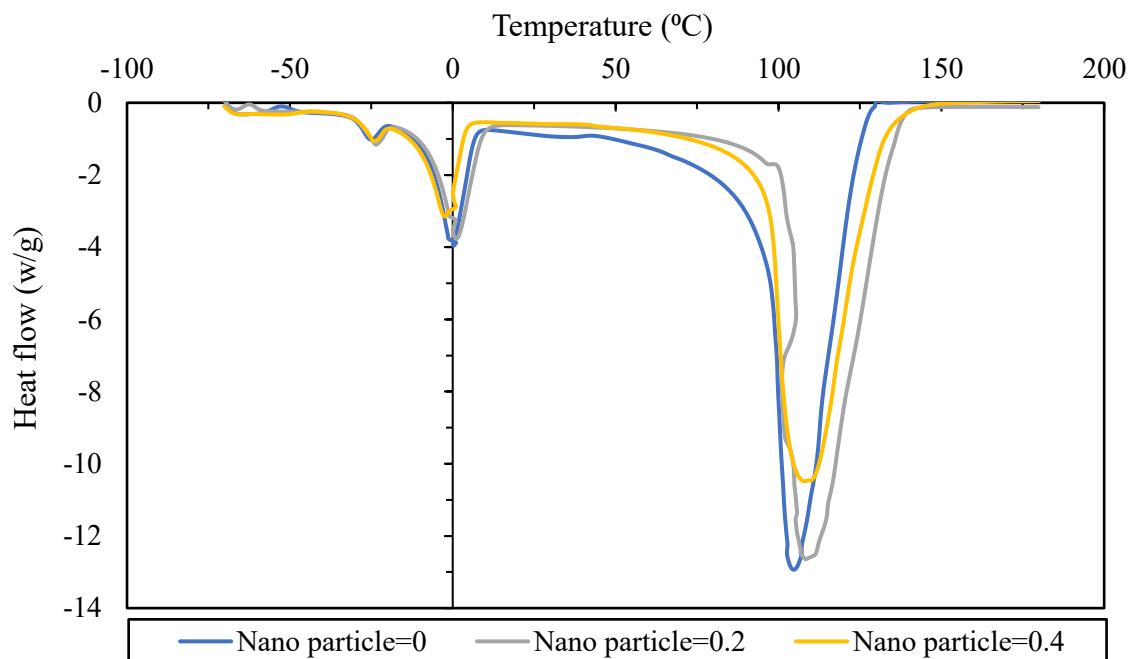


Fig. 10. DSC curves for HPAM gel with high purity silica nanoparticle concentrations of 0, 0.2, and 0.4 wt.% within a temperature range of -70 to 180°C.

Nanoparticle (wt.%)	0	0.2	0.4
ΔH (J/g)	-187.3	-198.1	-143.8
Free water content (%)	56.1	59.4	43.1
Bound water content (%)	43.8	40.5	56.8

Table 6. Mass fraction of free and bound water in HPAM gel with high purity silica nanoparticle concentrations of 0, 0.2, and 0.4 wt.%.

Dispersion

Dynamic light scattering (DLS) was employed to characterize the hydrodynamic size distribution of scattering species within the HPAM–HPSN gelant systems. To minimize multiple scattering and suppress polymer-chain entanglement, each sample was diluted by adding 15 cm³ of deionized water to 1 g of gelant. As illustrated in Fig. 11, the hydrodynamic radius (R_h) shifted toward larger values as the HPSN concentration increased from 0 to 0.4 wt.%. To evaluate the uniformity of these scattering entities, which may include solvated HPAM coils, silica aggregates, and HPAM–HPSN complexes, the Polydispersity Index (PDI) was analyzed. A lower PDI indicates a narrower and more homogeneous size distribution, whereas a higher PDI indicates a broader and more heterogeneous distribution. In this complex system, the PDI represents the distribution of the overall hydrodynamic size of the complexes rather than the molecular-weight distribution of the HPAM chains. Table 7 summarizes the PDI values for different HPSN concentrations. The results revealed that the addition of 0.2 wt.% HPSN resulted in the lowest PDI (0.241), suggesting the formation of relatively uniform HPAM–HPSN-associated structures. In contrast, increasing the concentration to 0.4 wt.% led to a substantial increase in PDI (0.612), indicating structural heterogeneity. Based on the findings up to this section, the 0.2 wt.% HPSN formulation was identified as the optimal concentration and selected for subsequent investigations.

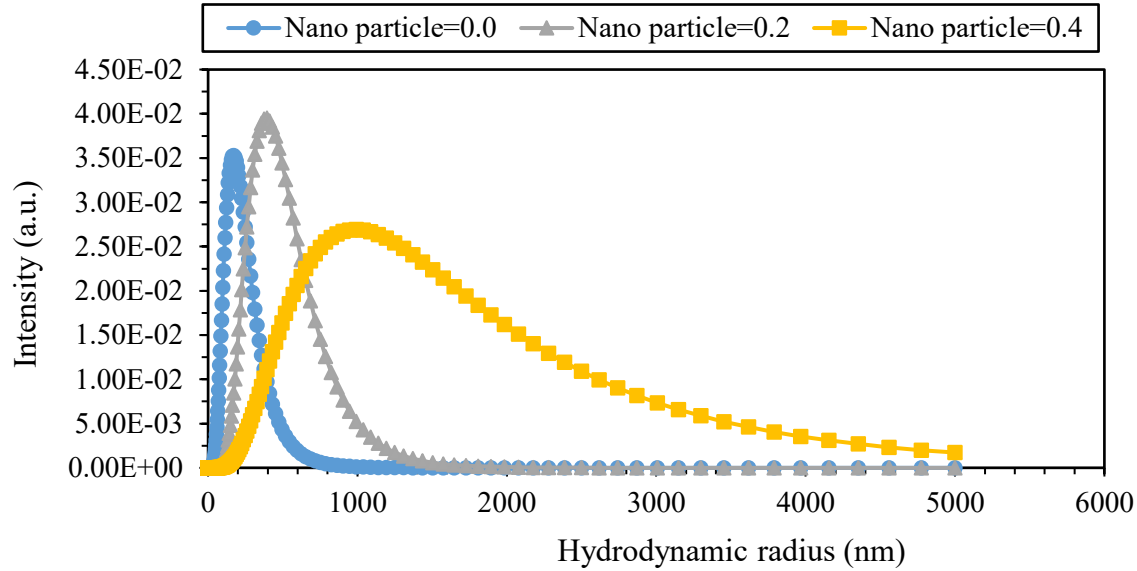


Fig. 11. Effect of high purity silica nanoparticles at concentrations of 0, 0.2, and 0.4 wt.% on the hydrodynamic radius of HPAM gelant at 25°C.

Nanoparticle (wt.%)	0	0.2	0.4
PDI	0.321	0.241	0.612

Table 7. Polydispersity index (PDI) of HPAM gelant with high purity silica nanoparticles at concentrations of 0, 0.2, and 0.4 wt.% at 25°C.

Additionally, to gain a deeper understanding of how 0.2 wt.% nanoparticles reinforce the HPAM gel, SEM analysis was performed. As depicted in Fig. 12, the microstructures of two gels at the same level of magnification with and without nanoparticles exhibit distinct differences. The microstructure of the gel without nanoparticles (Fig. 12(a)) shows a network with large pore spaces. Increasing the nanoparticle concentration to 0.2 wt.% (Fig. 12(b)) results in a denser three-dimensional network structure. This increase in density is accompanied by enhanced uniformity, achieved by reducing polymer entanglement.

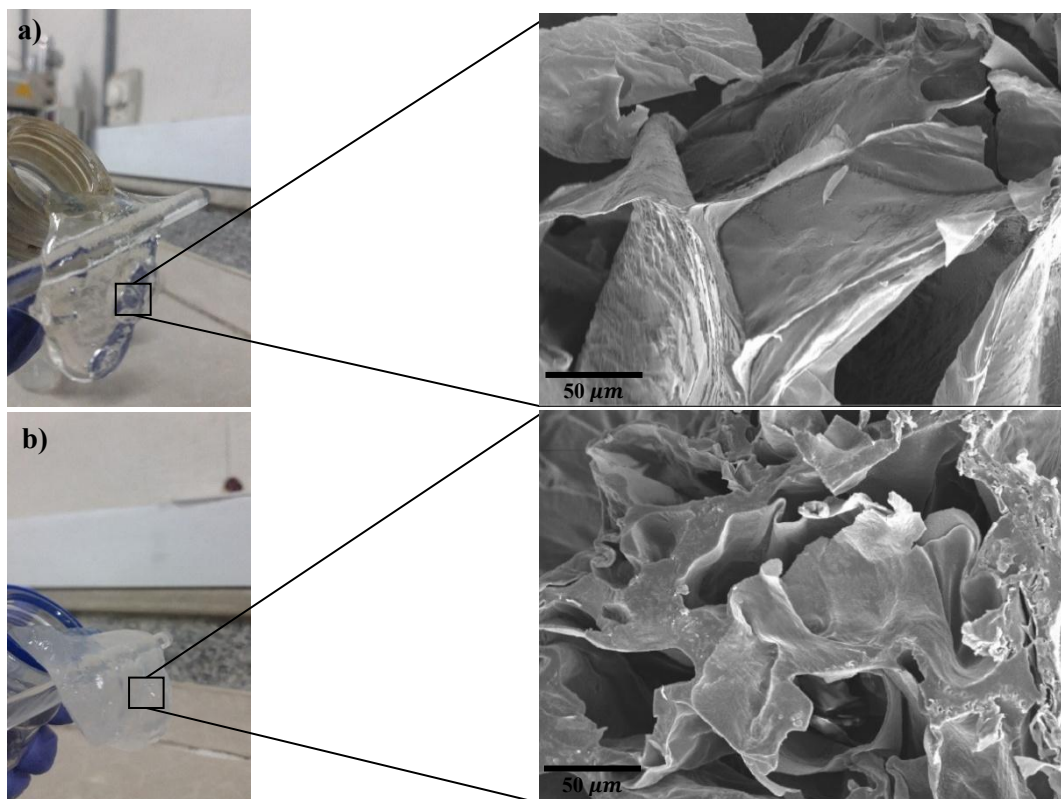


Fig. 1. SEM micrograph of frozen dried HPAM gel samples prepared with 0 wt.% (a) and 0.2 wt.% high purity silica nanoparticles (b).

Swelling behavior

It has been mentioned that a high concentration of Cl^- ions in solutions can reduce osmotic pressure. As a result, the swelling ratio of the gel may decrease or even result in syneresis within the gel^{60,61}. Furthermore, the existence of divalent ions and high temperatures can induce the occurrence of syneresis. At elevated temperatures, the amide functional group ($-\text{CONH}_2$) transforms into the carboxylate functional group ($-\text{COOH}$), resulting in numerous sites for interaction with divalent ions within the gel structure. Consequently, these bond formations make the gel structure more compact, leading to water being expelled from the structure⁶². This chemical reaction is considered the most significant factor leading to the syneresis of gel. According to gel network theory, other forces also influence the syneresis and swelling of gel. These forces include mixing potential and elastic potential^{63,64}. As a result, the mixing potential encourages gel swelling, while the elastic potential hinders chain deformation. Consequently, the mechanism behind the syneresis of poly-electrolyte gel in saline solutions is a highly intricate phenomenon⁴¹.

To investigate the effect of formation water (composition provided in Table 3) and crude oil on the gel's swelling behavior, the gel with 0 and 0.2 wt.% HPSN was prepared and immersed in the formation water and crude oil. Then, the EWSR of the gels were evaluated over different times at the temperature of 90°C (using Eq. 4). As provided in Fig. 13(a), when the gel without HPSN was immersed in the formation water, the swelling ratio began to decrease due to osmotic pressure differences, hydrolysis of the polymer, and its interaction with divalent ions, eventually reaching equilibrium in the formation water. In contrast, the swelling ratio of the gel containing 0.2 wt.% HPSN initially increased and after a while started to decline. This temporary increase indicates that the presence of nanoparticles probably delayed the hydrolysis of amide groups, as confirmed by the FTIR analysis. The difference in the ultimate equilibrium swelling ratio was approximately 10%, demonstrating that while HPSN provided a short-term stabilizing effect, it had no permanent influence on the swelling behavior of the gel in the formation water. The gel was also placed within the crude oil. As shown in Fig. 13(b), it was observed that both the gel with and without HPSN underwent shrinkage in crude oil, resulting in diffusion factors and the loss of the hydrophilic property of the gel. This, in turn, causes synthetic water to exit from within the gel into the solution. However, same as the results of formation water, the HPAM gel with 0.2 wt.% HPSN exhibited better resistance to syneresis in crude oil over time.

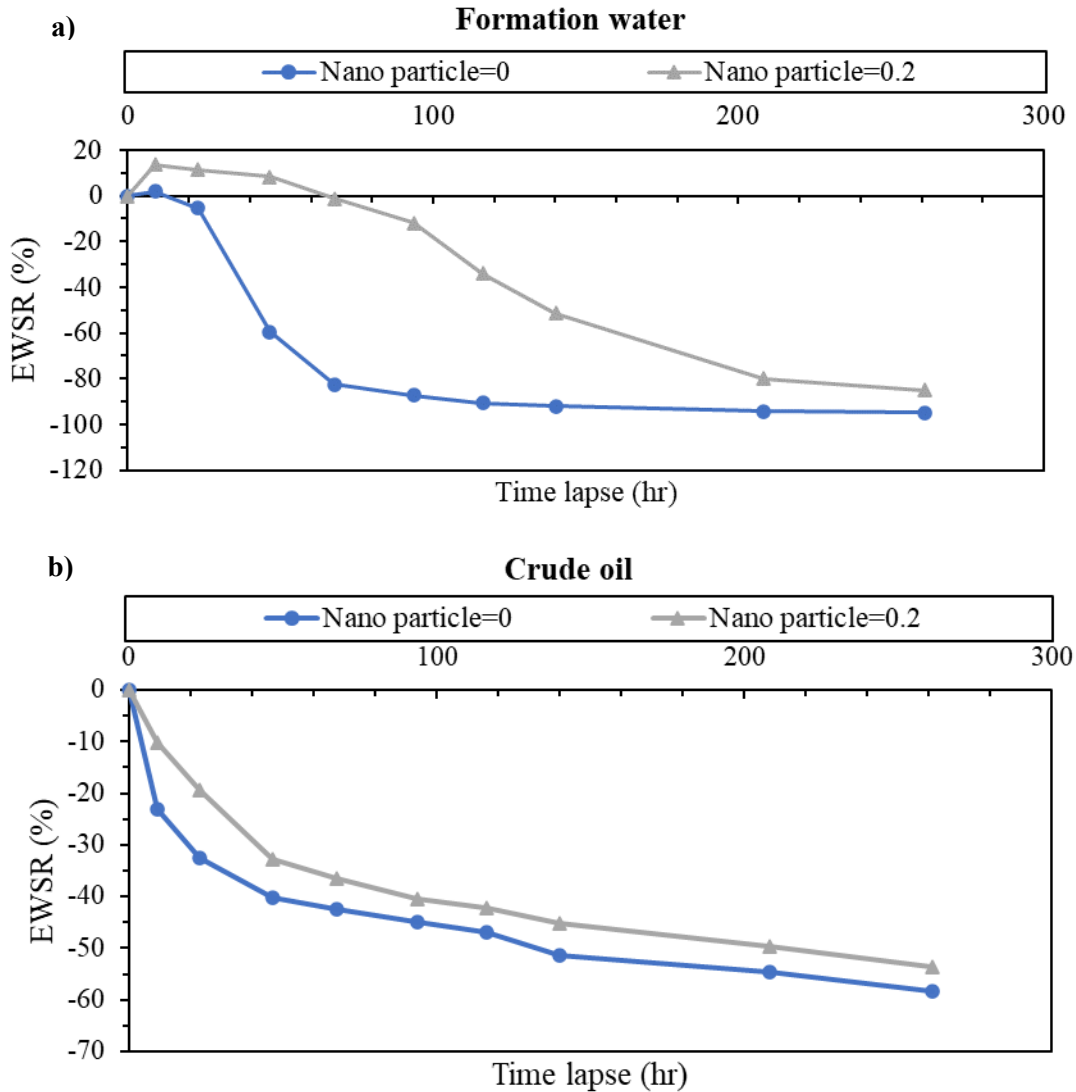


Fig. 13. Variation in swelling ratio over time at 90°C for HPAM gel samples immersed in **a)** formation water and **b)** crude oil, with 0 and 0.2 wt.% high purity silica nanoparticles.

Fractured porous media core flooding tests

A core flooding test was performed to evaluate the performance of the HPAM gel containing 0.2 wt.% HPSN in fractured porous media. Before injection, the viscosities of formation water and crude oil at 90°C were measured to be 1.2 and 9 cP, respectively. The fractured core was first saturated with formation water. Then, approximately three pore volumes of crude oil and formation water were injected until a stable pressure drop of approximately 313 mbar and 300 mbar, respectively, was observed, as shown in Fig. 15(a). The corresponding effective permeabilities for each phase were calculated before gel treatment, as shown in Table 8. The HPSN gelant was then injected into the fractured core. Simultaneously with the core aging process, two gel samples were cured in the oven: one collected as the effluent from the core flooding experiment and the other prepared in situ. This allowed observation of the gel state and the effect of the porous medium on gelation time. As provided in Fig. 14, the effluent HPSN gelant sample collected at the core outlet exhibited delayed gelation after contact with the fractured core. This delay was likely caused by shear forces, chromatographic effects, and especially dilution effects^{18,65-68}. After 7 days, once the gel reached code G within the core (as shown in Fig. 14), crude oil and formation water were reinjected following the same procedure as in the first cycle. The pressure stabilized at approximately 8825 mbar for crude oil and 19029 mbar for formation water, as shown in Fig. 15(b).

This figure shows that the pressure remained high during formation water injection, and the formation water encountered significantly higher flow resistance than crude oil following the gel treatment. Finally, to better investigate the effect of gel treatment, the RRF, a key indicator of gel performance, should be evaluated. To do this, the effective permeability of each fluid after gel treatment was calculated, with the results presented in Table 8. Then, the calculated RRF for each fluid was calculated to be 58.2 for formation water and 28.2 for crude oil. This demonstrates that the gel imposed nearly twice as much resistance to water as it did to oil, highlighting its water-selective permeability reduction. This selective behavior is attributed to the hydrophilic nature of the HPAM-based gel system.

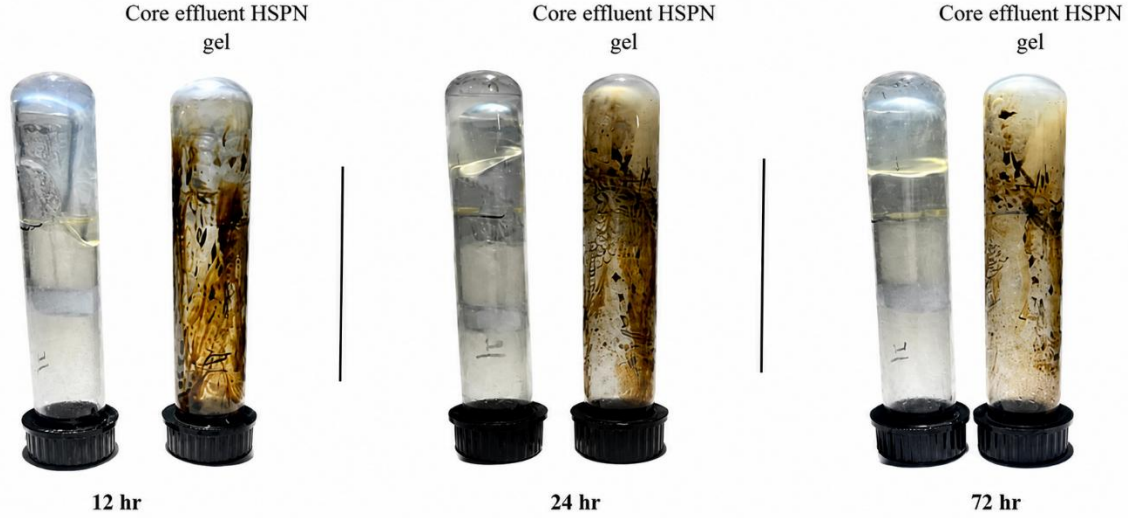


Fig. 14. Gelation time of HPAM gel with 0.2 wt.% high purity silica nanoparticles from core effluent and in-situ samples at 90°C over various aging times.

	Formation water		Crude oil	
	Rate (cm ³ /min)	Pressure drops (mbar)	Rate (cm ³ /min)	Pressure drops (mbar)
Before gel treatment	0.1	300	0.1	313
After gel treatment	0.1	19029	0.1	8825
	$k_{ew1} = 1.3 \text{ d}$ $k_{ew2} = 0.023 \text{ d}$		$k_{eo1} = 9.6 \text{ d}$ $k_{eo2} = 0.34 \text{ d}$	
	$RRF_w = \frac{k_{ew1}}{k_{ew2}} = \frac{1.3}{0.023} = 58.2$		$RRF_o = \frac{k_{eo1}}{k_{eo2}} = \frac{9.6}{0.34} = 28.2$	

Table 8. Results of fractured core flooding experiments using HPAM gel with 0.2 wt.% high purity silica nanoparticles at 90°C.

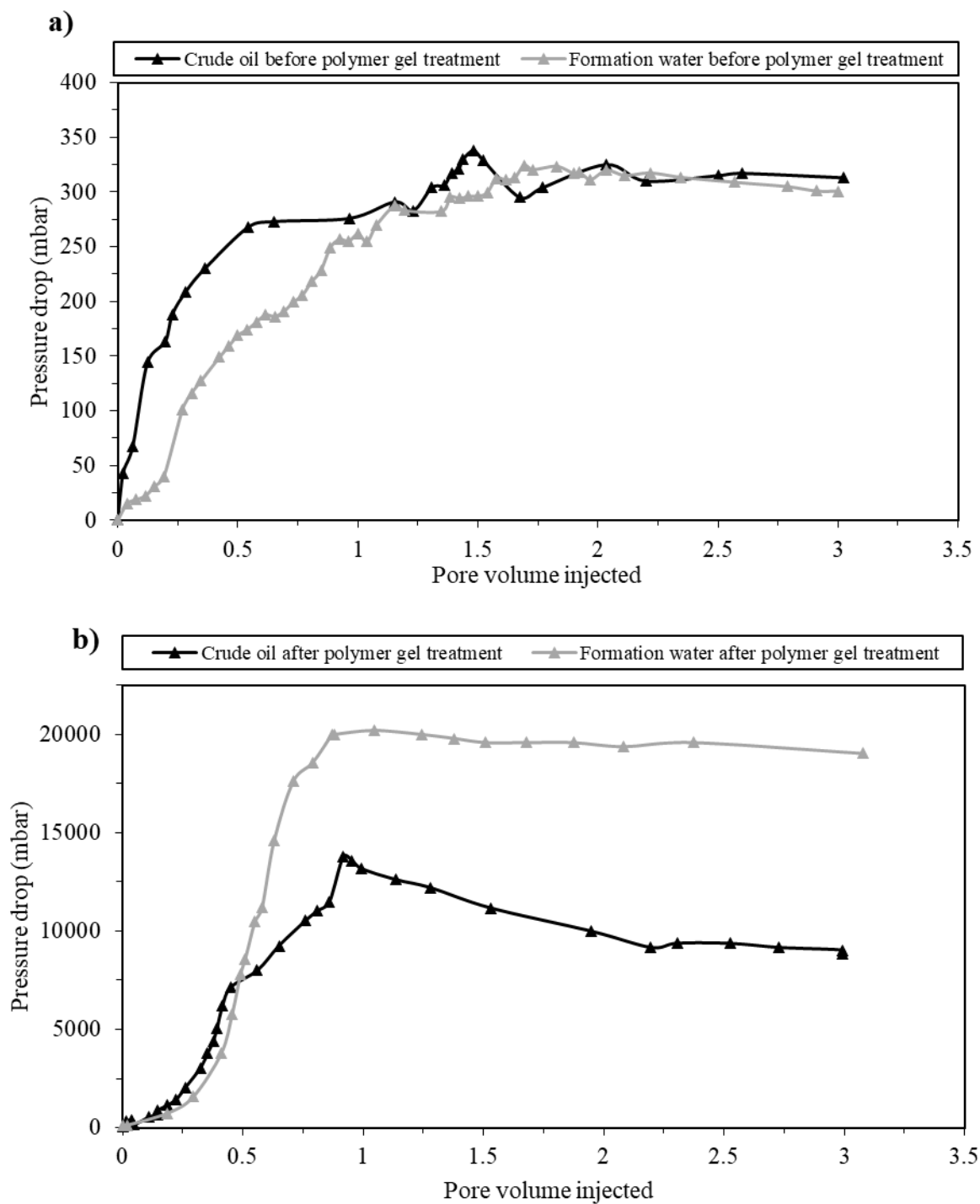


Fig. 15. The pressure drops for formation water and crude oil before (a) and after (b) 72 hours of HPAM gel treatment with 0.2 wt.% high purity silica nanoparticles at 90°C.

Conclusion

High-purity silica nanoparticles (HPSN) were incorporated into an HPAM/HMTA, with HCl as activator, gel system to enhance its performance for water shutoff applications in fractured reservoirs. The effects of nanoparticle concentration on the gelation process, rheological properties, thermal stability, nanoparticle dispersion, swelling behavior, and fractured core performance were systematically investigated. The major findings are summarized as follows:

- The gelation process was investigated using FTIR analysis and bottle tests. FTIR results suggest possible hydrogen-bond interactions between hydroxyl groups on HPSN and the amide/carboxylate groups of HPAM, which could strengthen the polymer network and may suppress polymer hydrolysis. Consistent with these findings, bottle tests showed that incorporating HPSN accelerated gel formation and reduced gelation time.
- The rheological properties of the gelant were strongly affected by nanoparticle concentration. Increasing the HPSN concentration to 0.2 wt.% reduced the gelant viscosity from 15837 to 10558 cP at a shear rate of 1.32 s^{-1} , improving injectivity while enhancing shear-thinning behavior. In contrast, increasing the concentration to 0.4 wt.% increased the viscosity, compared to 0.2 wt.%, because of poor nanoparticle dispersion. Strain sweep tests showed that 0.2 wt.% HPSN expanded the linear viscoelastic region from 69.3% to 235%, despite reducing the storage modulus from 35.9 to 22.5 Pa, indicating a more deformable and mechanically resilient gel. Conversely, 0.4 wt.% HPSN produced a stronger but more brittle gel with a substantially narrower linear viscoelastic region, making it less suitable for fractured reservoir applications.
- Thermal stability tests demonstrated that HPSN significantly influenced the long-term stability of the gel at 90°C. Although 0.4 wt.% HPSN initially reduced syneresis by increasing gel hydrophilicity, prolonged aging promoted excessive crosslinking and ultimately accelerated water expulsion. In contrast, the gel containing 0.2 wt.% HPSN exhibited nearly constant syneresis throughout the aging period, indicating superior long-term stability. Differential scanning calorimetry further confirmed that 0.2 wt.% HPSN provided the highest thermal stability, and the measured free and bound water contents were consistent with the syneresis results.
- The DLS tests showed that nanoparticle concentration strongly affected nanoparticle distribution and gel microstructure. The formulation containing 0.2 wt.% HPSN exhibited the lowest polydispersity index (0.241), indicating the most uniform nanoparticle dispersion, whereas increasing the nanoparticle concentration promoted poor nanoparticle dispersion. SEM images further revealed that 0.2 wt.% HPSN produced a denser and more homogeneous three-dimensional polymer network with reduced polymer entanglement compared with the nanoparticle-free gel.
- Swelling experiments conducted in both formation water and crude oil at 90°C showed that incorporating 0.2 wt.% HPSN improved resistance to syneresis, initially for the formation water, but the equilibrium syneresis was not significantly different in both fluids for the 0 and 0.2 wt.% HPSN gels.
- Fractured core flooding experiments confirmed the effectiveness of the optimized gel formulation containing 0.2 wt.% HPSN for water shutoff applications. After gel treatment, the residual resistance factors reached 58.2 for formation water and 28.2 for crude oil, demonstrating that the gel provided approximately twice the flow resistance to formation water as to crude oil. Furthermore, delayed gelation of the core effluent compared with the in-situ gel indicated that shear, chromatographic separation, and dilution occurred during propagation through the fractured porous medium.
- Future studies should further validate the developed gel through long-term core flooding experiments under diverse fractured reservoir conditions to confirm its field applicability.

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