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Challenges of Gel Treatment Application for Conformance Control

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Abstract

This article explores challenges in gel treatments, including polymer dissolution, gelation time, gel strength, stability, resistance to brine flow, and permeability damage in oil reservoirs. It highlights strategies to enhance gel performance, such as optimizing the polymer/crosslinker ratio to reduce syneresis due to limited mixing time and using chromium chloride for rapid gelation at low temperatures. As for large gel treatments, 1-day gelation can be achieved with high molecular weight gels (~20-30 million Da) using 0.05 wt.% chromium acetate at 25 °C, although they exhibit reduced stability at higher temperatures. For high-temperature conditions, 500,000 Da gels are recommended at a minimum concentration of 2.5 wt.%, with cold water pre-flush as an effective gelation delay method. Core flooding tests demonstrate that 500,000 Da gels can maintain efficacy under challenging conditions, achieving post-flush pressure gradients comparable to those provided by 0.5 wt.% high molecular weight gels in more favorable scenarios. Permeability damage remains a significant issue, as evidenced by 0.5 wt.% polymer gel reducing sand pack oil permeability by 340 times, with no significant recovery through NaOH treatment. Developing effective formulations to restore oil permeability after unsuccessful treatments could enhance confidence in using gels, potentially reducing water cut and minimizing oil production losses.

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1. Introduction

Currently, many oil fields are facing a phase with elevated water cut, resulting in a considerable amount of crude oil being left within the reservoir.[1–3] Factors such as the high mobility ratio between water and oil and the heterogeneity of the reservoir play significant roles in worsening this challenge.[4–6] Water produced alongside hydrocarbons causes significant technical problems such as equipment corrosion,[7,8] increased oil lifting costs,[9] investments needed to treat and dispose the produced water,[10,11] as well as environmental issues.[12]

One method to decrease water production involves selectively reducing the permeability of water-producing zones using polymer gels.[13,14] For instance, in the Huabei field (China), an injection well situated in fractured tight sandstone, experiencing poor sweep efficiency after water flooding, underwent gel treatment with an estimated gel volume of 1,500 m³. Three months post-treatment, the average oil production rose from 0.36 t/d to 0.9 t/d, while the water cut decreased from 95.77% to 88.7%. Even after a year, the positive effects on oil production persisted due to this gel treatment.[15] Review of field projects indicates that, on average, gel treatments in injection wells result in a 32% increase in oil production rate and can incrementally provide 22 STBO per kg of dry polymer powder. Furthermore, the practice demonstrates that gel treatments are more effective in naturally fractured and carbonate reservoirs compared to others.[16]

The application of gel treatments for water shut-off in production wells is also crucial for extending the lifespan of the well and revitalizing oil production from uneconomic producers. For example, the injection of approximately 770 m³ of HPAM gel into a horizontal well suffering from severe water coning allowed for an increase in oil flow rate from 0 to 16.3 t/d and a decrease in water cut by approximately 6%.[17] In another instance, the treatment of a horizontal well with 500 m³ of HPAM gel resulted in an increase in oil production from

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9.2 to 20 m^3/d and a decrease in water cut from 90% to 8%, although it rebounded to 70% over the following 14 months.[18] However, the application of polymer gel treatments is associated with several challenges, including:

1-Long dissolution time for high molecular weight polymers;[19]
2-Gel syneresis;[20, 21]
3-Extended gelation time at low reservoir temperatures;[22]
4-Rapid gelation at high reservoir temperatures;[23]
5-Insufficient resistance to brine flow in wide fractures;[24]
6-Risk of oil permeability damage.[25,26]

It is important to highlight that the risk of oil permeability damage has been extensively investigated.[27] Depending on the experimental conditions, oil permeability can decrease by factors ranging from 1.3 to 1,279 times.[28,29] Factors such as gellant composition and oil viscosity have been shown to significantly influence oil permeability after gel treatment.[30] To minimize the impact of polymer gels on oil-saturated matrices, brine-initiated gels have been proposed, which gel upon the contact with saline water. For instance, a hydroxypropyl cellulose/sodium dodecyl sulfate solution reduced brine permeability by 95% upon contact with saline water but did not gel when injected into core samples with oil saturation levels between 0.75 and 0.8. This indicates that oil permeability damage is unlikely, as the gellant infiltrates oil-producing zones.[31] Although these formulations demonstrate promising results, their application is limited by the high cost of materials. Consequently, hydrolyzed polyacrylamide remains the most widely used polymer for water shut-off treatments.

This paper describes a series of bottle tests and core flooding experiments aimed at elaborating on the challenges associated with polymer gel treatments. The goal is to provide reservoir engineers with additional information and details necessary for making informed decisions. The information obtained from bottle tests can be particularly helpful in selecting suitable polymers, crosslinkers, and their concentrations under given conditions. Additionally, the results from fractured core flooding tests demonstrate the potential of using regular HPAM/Cr(III) gels for plugging relatively wide fractures (2-mm-wide) in high-temperature (95 °C) fractured carbonate rocks. Furthermore, the results of sand pack flooding tests highlight the detrimental effect that HPAM/Cr(III) gel can have on oil permeability in unconsolidated sandstones. These findings underscore the importance of carefully considering the selection and application of gel treatments in diverse reservoir conditions.

2. Materials and methods

2.1 Brines

The chemical composition of the brines used in this study was determined by a professional laboratory following established analytical chemistry procedures, as commissioned by the research team. The chemical composition of brines used in this study are shown in Table 1.

2.2 Crude oil

In this study, crude oil with a density of 0.937 g/cm^3 and a dynamic viscosity of 796 cP at 25 °C, measured using a Stabinger SVM 3000 viscometer, was used.

2.3 Polymers

Hydrolyzed polyacrylamide (HPAM) samples provided by a commercial manufacturer were used in this study. Table 2 lists the molecular weight and hydrolysis degree of the polymer samples.

The chemical identity of the dry polymer samples was checked by using an FT/IR-6X Fourier Transform Infrared Spectrometer within the spectral range of 4000-400 cm^{-1}. The samples were prepared as tablets by pressing 2 mg of the polymer with 100 mg of KBr. KBr spectrum was used as the reference. Spectral analysis was conducted with Spectra Manager Ver.2.5 software, utilizing infrared spectral databases available in the KnowItAll software. The results of are shown in Fig. 1.

Table 1. Chemical composition of brines used in this study.

Parameter	Field C low salinity brine	Caspian Sea brine	Field K brine	Uzen reservoir brine	High salinity synthetic brine	Ayrankol synthetic brine	Ayrankol XIV brine
TDS, g/L	0.818	16.3	26.6	61.144	217	222	239.5
K^+, mg/L	2.67	4,473 (K^+ and Na^+)	70.94	18,816 (K^+ and Na^+)	-	660.8	663.2
Ca^{2+}, mg/L	108.1	400	2	3,206	12,250	3,400	3,403
Mg^{2+}, mg/L	41.3	730	12.2	1,094	2,400	2,297	2,310.4
Na^+, mg/L	191.8	4,473 (K^+ and Na^+)		18,816 (K^+ and Na^+)	68,570	79,740	79,887.6
SO_4^{2-}, mg/L	212	3,153	12,532	80.7	-	-	-
Cl^-, mg/L	186	7,232	17,373.9	37,605	134,510	136,299	157,784

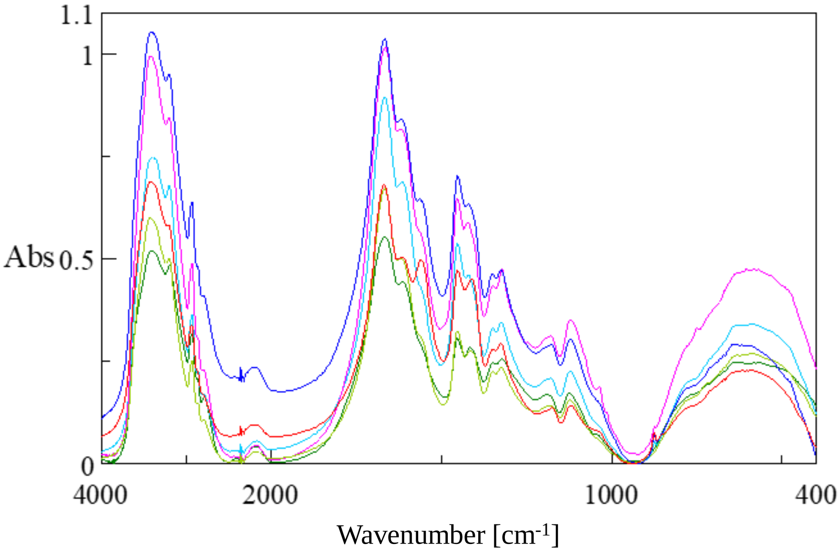


Fig. 1 v transform infrared spectrometry. Polymer sample # 1 - green; # 2- sky blue; # 3 - pink; # 4 - lime; # 5 - blue; # 6 - red.

Table 2. Polymers used in this study.

#	Molecular weight, Da	Hydrolysis degree, %
1	500,000	5
2	~5,000,000	5
3	6-7,000,000	5
4	~20,000,000	5
5	~20,000,000	10
6	~20-30,000,000	30

The IR spectra were analyzed, focusing on the following key regions:

3400-3200 cm⁻¹: This region corresponds to the stretching vibrations of -OH groups, which may indicate the presence of hydroxyl groups or bound water. Amide N-H stretching vibrations, characteristic of polyacrylamide, may also be observed here.

2920-2850 cm⁻¹: These peaks represent the stretching vibrations of C-H bonds, characteristic of methyl (CH_3) and methylene (CH_2) groups within the polyacrylamide main chain.

1665-1600 cm⁻¹: This region features the amide I band, which is characteristic of the C=O stretching vibration in polyacrylamides.

1450-1370 cm⁻¹: This region is associated with C-H bending vibrations in methyl and methylene groups, as well as N-H bending vibrations (amide II) in the polyacrylamide structure.

1050-1000 cm⁻¹: The C-N or C-O stretching vibrations, potentially related to the polyamide chain, are observed in this region.

700-600 cm⁻¹: C-H bending vibrations in methylene groups, or other specific molecular features, may be present in this region.

The FTIR spectra of the HPAM samples show consistent peak positions and overall spectral shapes, indicating a similar chemical structure across the samples. However, variations in peak intensities suggest differences in molecular weight, hydrolysis degree, and potentially other factors such as water content and polymer microstructure. These variations influence the absorption characteristics, leading to the observed differences in the spectra.

Either a Brookfield LVT (AMETEK Brookfield, USA) or a Model 900 rotational viscometers (Ofite, USA) were used to measure the viscosity values of polymer solutions.

2.4 Bottle tests

For the bottle tests, the polymers listed in Table 2 were utilized. All polymers were supplied in dry form by a manufacturer and were added to brine while stirring, employing either a magnet bar or overhead impeller stirrers. Chromium acetate and chromium chloride were employed as crosslinkers and added to the polymer solutions to induce gel formation. The concentrations of polymer and crosslinker for each gel sample is specified in the text. The gels were visually evaluated using bottle testing, following the method outlined by Sydansk (1990). The viscosity of the gels was measured using BDV-2C viscometer with R#2 spindle at 10 rpm.

2.5 Fractured core flooding experiments

Carbonate core sample from depth interval 4,285.7 – 4,286 m was used in this study. The mineral composition of the rock was determined using Panalytical X'Pert Pro X-ray diffraction (XRD) and analyzed with the PDF-2 database. The analysis revealed the following chemical composition of the sample: dolomite (calcium magnesium carbonate, $CaMg(CO_3)_2$) with a hexagonal crystal system, PDF reference 00-011-0078, and a calculated density of 2.86 g/cm³. As Fig. 2 shows, the core was severely fractured with natural fractures which complicated the drilling process.

In order to construct a fractured core for flooding cylindric core was drilled out and cut along the axis (Fig. 3), metal spacers were inserted in between the two rock pieces, and then the core was fixed with clamps. Subsequently, the core was coated with 5 min epoxy and put into a metal sleeve. The annular space between the core and the metal sleeve was filled with 24 h epoxy mixed with clay. Once the casting material cured, the core was faced off in a machine shop using a standard lathe, and pressure taps were drilled out through holes in the metal sleeve. The distance between the two internal pressure taps was equal to 4 cm (0.131 ft). Endcaps made of stainless steel, nuts, washers and bolts were used to assemble the core holder.

Fig. 2 Photos of the dolomite core from depth 4285.7 – 4286 m.

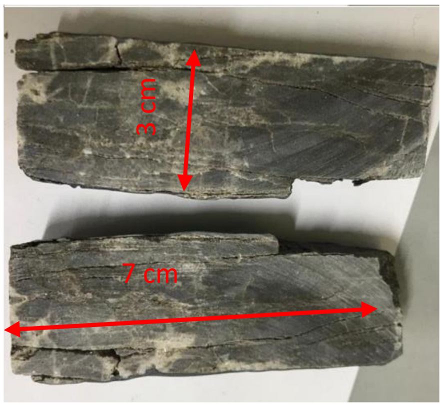


Fig. 3 Cut dolomite core sample from depth interval 4,285.7 – 4286 m.

Figure 4 shows the core flooding set up used in this study. The set up consisted of high-pressure piston pump, piston accumulator, valves, and injection lines with inner diameter of 4 mm, core holder, as well as pressure measuring devices (P_1-P_3).

The core flooding experiments were conducted according to the following procedure:

1-Vacuuming the core at 95 °C.

2-Saturating the core with 217 g/L synthetic brine (Table 1) at 95 °C.

3-Gel was put inside of the accumulator and aged for 1 day at 60 °C. This step was executed to mimic the temperature conditions experienced by the gel in the wellbore and in the vicinity of the well, as the temperature in this area is expected to be around 60 °C during most of the treatment process.

4-[2.5 wt.% 500,000 Da and 5% HD polymer / 0.2 wt.% chromium acetate] gel was injected into the fracture at 0.43 cm^3/min and 95 °C. Gel was prepared in Field C low salinity brine (Table 1).

5-Post-flush by using 217 g/L synthetic brine (Table 1) from the opposite direction to mimic the inflow of brine from the formation into the wellbore.

6-[5 wt.% 500,000 Da and 5% HD polymer / 0.5 wt.% chromium acetate] gel was injected into the fracture at 0.43 cm^3/min and 95 °C. Gel was prepared by using the low salinity brine and aged at 60 °C for 24 h before the injection.

7-Post-flush by using 217 g/L synthetic brine (Table 1) from the opposite direction to mimic the inflow of brine from the formation into the wellbore.

The fractured core length and diameter were equal to 6.5 and 3 cm, respectively. The width of the fracture and the inner diameter of the injection lines were equal to 2 and 4 mm, respectively.

2.5 Sand pack flooding

The sand pack flooding was conducted by using 8.6-cm-length and 4.3-cm-diamter sand pack filled with reservoir

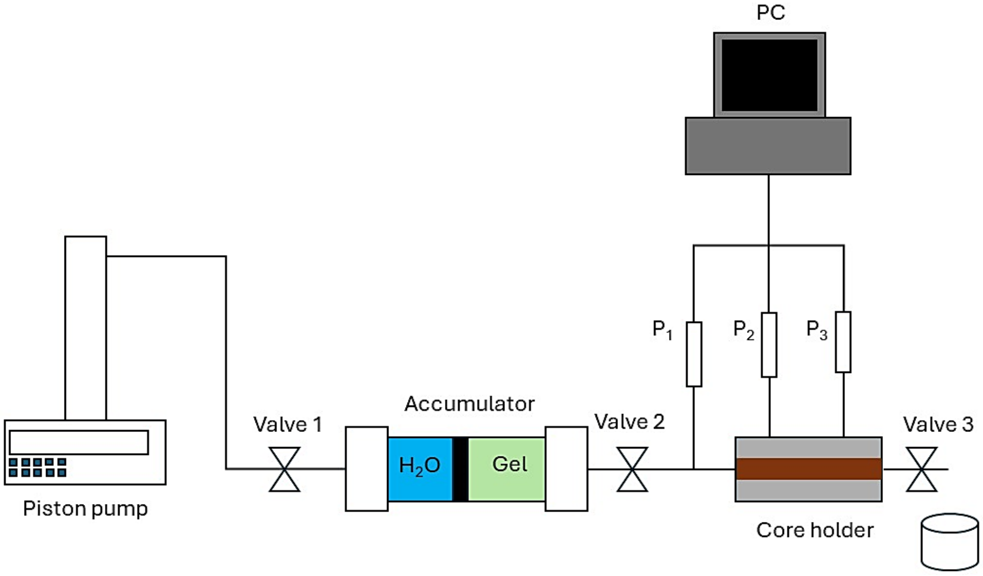


Fig. 4 Schematic of core flooding setup.

sand from a shallow viscous oil reservoir. The sand was used as received (i.e., with resident oil and water in place).

The core flooding experiments were conducted according to the following procedure:

1-Saturating the model with 26.6 g/L Filed K brine (Table 1) to measure porosity and permeability.

2-Saturating the model with oil at 1 cm^3/min.

3-Oil displacement by 26.6 g/L brine at 1 cm^3/min.

4-The injection of freshly prepared [0.5 wt.% 6-7 mln Da and 5 % HD HPAM / 0.5 wt.% chromium acetate] gelling solution at 1 cm^3/min.

5-Aging the model during 3 days for gelation to take place.

6-The injection of water in the direction opposite to the injection of gelling solution to model the inflow of brine from the reservoir towards the wellbore (production direction).

7-The injection of oil in the production direction at 1 cm^3/min and 0.25 cm^3/min.

8-In order to restore permeability to oil the model was flooded with 1 PV of 2% sodium pyrophosphate solution prepared in fresh water.

9-Aging the model for one day.

10-Brine post-flush at 1 cm^3/min in production direction.

11-The injection of oil at 0.25 cm^3/min in production direction.

12-The injection of 1 PV of 4% NaOH in order to restore permeability to oil.

13-Aging the model for one day.

14-The injection of oil at 0.25 cm^3/min in production direction.

All the steps were conducted at 25 ºC. The gellant, sodium pyrophosphate and NaOH solutions were injected in the injection direction, whereas brine and crude oil were injected in the production direction.

3. Results and discussion

3.1 Polymer dissolution rate

Ensuring the complete dissolution of the polymer is imperative to meet the technological standards of field treatments. In fact, hydrolyzed polyacrylamide (HPAM) may take from 2 to more than 24 hours to completely dissolve in brine. The dissolution time of HPAM strongly depends on the brine salinity and polymer molecular weight. For example, Fig. 5 shows the percentage of the maximum viscosity that can be possible achieved by a polymer solution versus the mixing time in 26.6 g/L field brine. As evident from the data, a polymer solution with a molecular weight of 6-7 million Da achieves 60 % of its maximum viscosity within 30 minutes of stirring, while higher molecular weight polymers require around 90 minutes of stirring to reach the same result.

A higher degree of hydrolysis was anticipated to enhance solubility by increasing the number of carboxylate groups, thereby making the polymer more hydrophilic. However, for HPAM with a molecular weight of 20-30 million Da, even a 30 % degree of hydrolysis does not significantly facilitate the polymer dissolution to the level seen with a polymer of 6-7 million Da (Fig. 5).

These observations are crucial for choosing the appropriate polymer type for a field project. Typically, gel treatments are conducted using a single 2 m^3 mixing tank with a 5 m^3/h injection rate, equating to less than a 25-minute dissolution time for the polymer within the tank. However, as depicted in Fig. 5, this duration is often insufficient for complete polymer dissolution in brine, especially if high molecular weight polymers are used.

Nevertheless, incomplete polymer dissolution in surface facilities has frequently been disregarded in field operations,

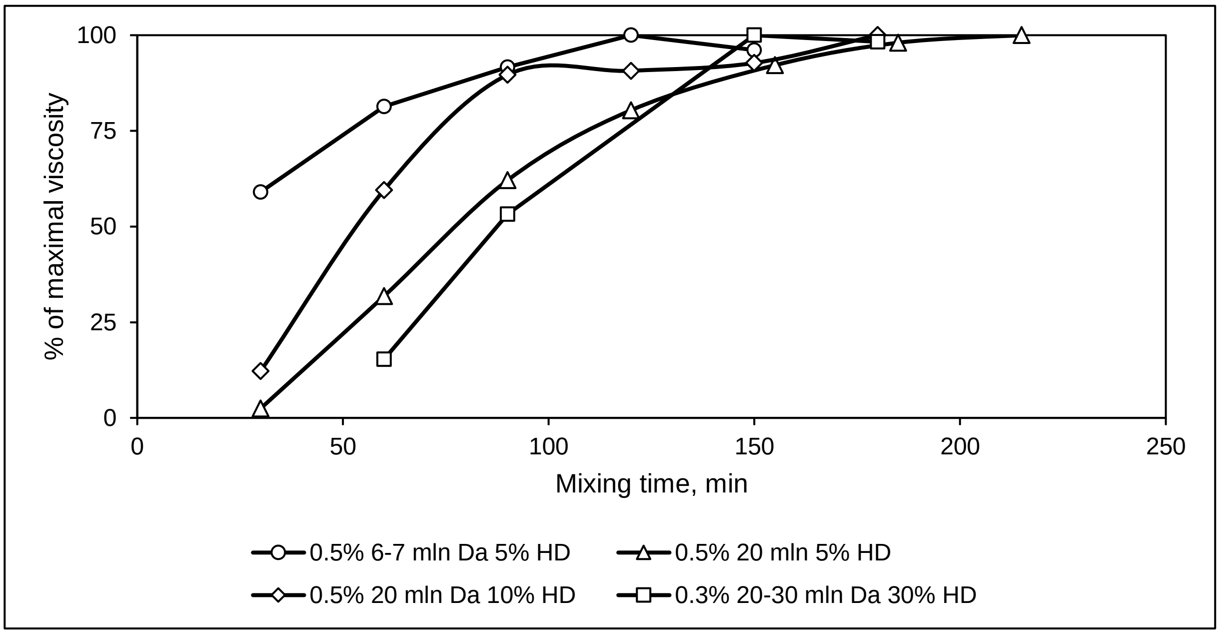
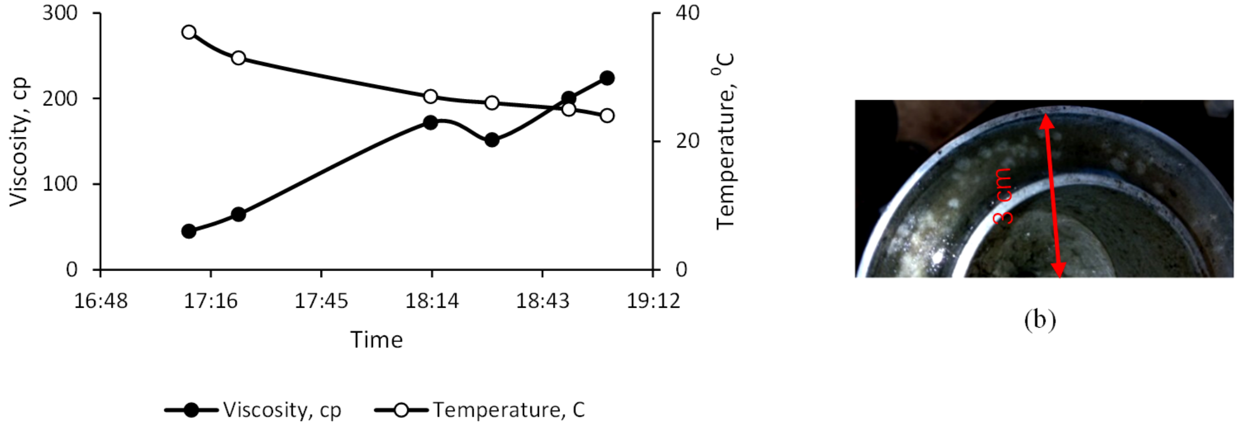


Fig. 5 % of maximal viscosity achieved after stirring of the polymer solutions at 300 rpm and ~23 ºC with impeller overhead stirrer. Maximal viscosity values are: 54.2 cp for 0.5 wt.% 6-7 mln Da 5 % HD; 153 cp for 0.5 wt.% ~20 mln Da 5 % HD; 132 cp for 0.5 wt.% ~20 mln Da 10 % HD; and 58 cp for 0.3 wt.% ~20-30 mln Da 30 % HD polymer solutions. Viscosity was measured at 6 rpm by using Ofite 900 model viscometer.



(a)

(b)

Fig. 6 a) the viscosity of gellant measured by Brookfield LVT viscometer at 7.34 sec^{-1} versus time; b) photo of the gelling solution. Brine: reservoir brine Uzen. Polymer: 0.5 wt.% of 6-7 mln Da HPAM. Crosslinker: 0.05 wt.% chromium acetate.

assuming that turbulent conditions within the wellbore assist in completing the dissolution of the polymer during the gellant transport from the surface to the bottom hole zone. To the best of our knowledge, this assumption has not yet been tested experimentally. However, field surveys show that viscosity of the gellants sampled from mixing tanks in surface facilities is relatively high (~45 cp) and increases right after the addition of chromium acetate in 3-4 times in 1 h, even if aged at room temperature (Fig. 6).

Basic calculations of the Reynolds number show that the high viscosity of polymer gellants makes turbulent flow inside the wellbore highly improbable. For example, Fig. 7 demonstrates the viscosity of a 0.3 wt.% polymer solution and the corresponding Reynolds numbers versus injection rate. The viscosity values of the polymer solution were derived from the empirical formula (1) obtained for this polymer solution in laboratory, whereas the shear rate values were calculated for 62-mm-diameter pipe by using formula (2)[32]:

$$\mu = -8.888 \cdot \ln(\gamma) + 69.56 \quad (1)$$

where μ – dynamic viscosity, cp; γ – shear rate, 1/sec.

$$\gamma = \frac{4 \cdot Q}{\pi r^3} \quad (2)$$

where γ – shear rate, 1/sec; Q – flow rate, m^3/sec; r – radius of the pipe, m.

As can be seen, for 0.3 wt.% HPAM solution in 26.6 g/L brine at 25 °C, the injection flow rate inside the 62-mm-diameter pipe should be at least 11 m^3/h for turbulent flow to occur. However, in field practice the injection flow rarely exceeds 5 m^3/h. Besides that, the increase of injection rate to 11 m^3/h and higher will result in the reduction of surface polymer mixing time at least in 2 times.

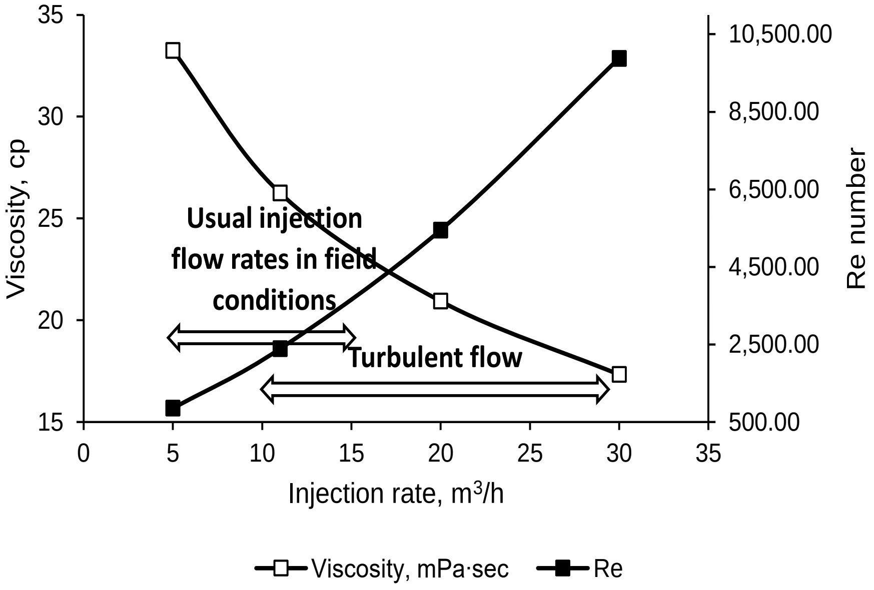


Fig. 7 Viscosity of 0.3 wt.% very 20-30 mln Da HPAM with 30 % HD in 26.6 g/L brine versus injection rate and corresponding Re number. Shear rate calculated for 62-mm-inner diameter injection pipe.

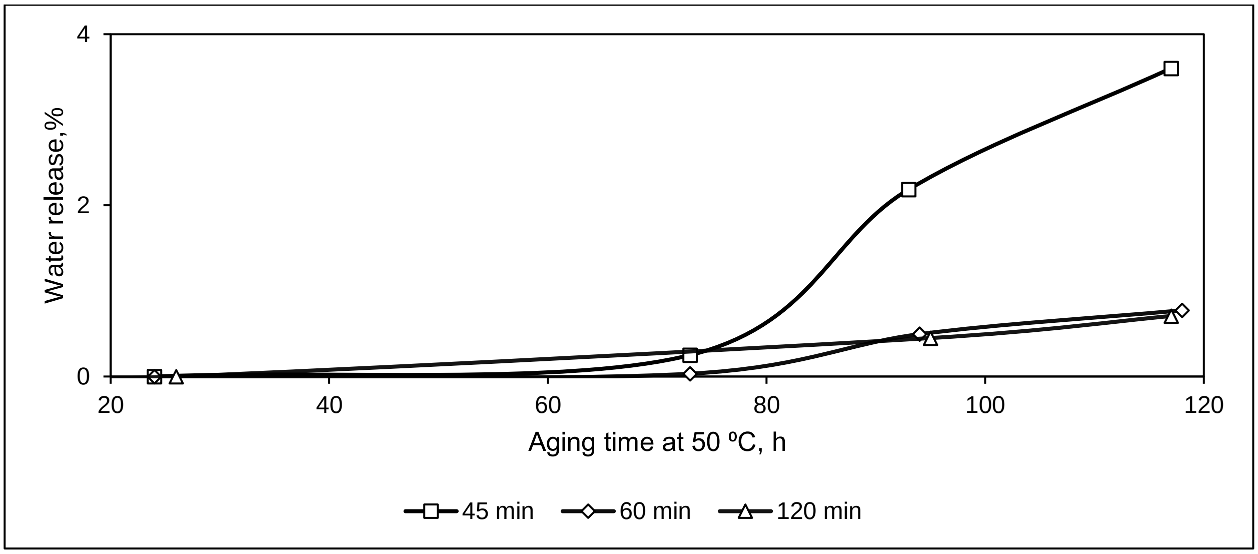


Fig. 8 The effect of 6-7 mln Da and 5 % HD polymer dissolution time in sea brine on [0.5 % polymer / 0.05 % chromium acetate] gel water release over time.

The observations detailed in this section suggest that if the available surface dissolution time is less than 30 minutes, as is typically the case, lower molecular weight HPAMs (~6 million Da) may be more suitable. This is because HPAMs with a molecular weight of approximately 20 million Da require at least 90 minutes of stirring to achieve 60 % of their maximum viscosity in brine with a total dissolved solids (TDS) concentration of 25-30 g/L, which is relatively low salinity for Kazakhstani oil fields. If the brine salinity is high (above 200 g/L), the use of high molecular weight polymers becomes more problematic, as even 3 hours of constant stirring is insufficient to completely dissolve the polymer. Assuming that turbulence will facilitate the dissolution of HPAM is reasonable only at high injection rates (higher than 10-11 m^3/h) which in turn reduces the surface mixing time.

3.2 The effect of polymer mixing time

In some instances, incomplete dissolution of the polymer has been demonstrated to induce syneresis in gels. For instance, Fig. 8 illustrates that at 50 °C, the water release from the gel decreases from 3.6 % to 0.7 % as the polymer mixing time increases from 45 to 60-120 minutes. However, as mentioned earlier, in field conditions, dissolution times may often be less than 30 minutes.

Figure 9 illustrates the water release from polymer gels over aging time at 50 °C. In these experiments, the polymer was mixed for 30 minutes by impeller stirring at approximately 330 rpm in seawater, and for an additional 5 minutes after the addition of chromium acetate. As evident, water release increases with chromium acetate concentration. Fig. 10 demonstrates that at the bottom of the test bottles, non-flowing gels were formed from partially dissolved polymer particles segregated due to gravity during the static aging process, whereas the remaining portion of the samples is represented by less strong gels. However, even at low concentrations of the crosslinker (0.0125-0.025 %), the gels appear to fall within the category of tonguing gel (gel D) according to the Sydansk gel strength code.

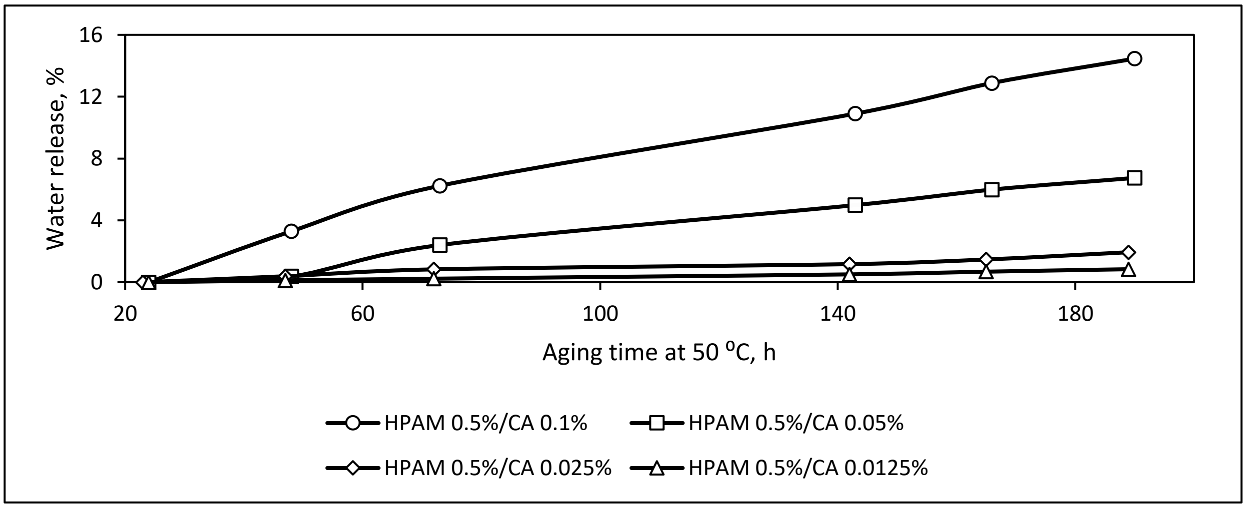


Fig. 9 The effect of crosslinker concentration on the water release (syneresis) of 6-7 mln Da 5 % HD HPAM gel in sea brine. Polymer mixing time – 30 min.

Fig. 10 0.5 % 6-7 mln Da 5 % HD HPAM / 0.1-0.0125 % (decreasing from left to right) chromium acetate in sea brine. Chromium acetate concentration decreases from left to right. Red rectangle – strong gel. Yellow rectangle – tonguing gel.

An important observation from Figs. 9 and 10 is that limited mixing time can result in the formation of a non-flowing gel (indicated by the red rectangle) and/or tonguing gels (indicated by the yellow rectangle). The non-flowing gel comprises swollen polymer particles, a low-concentration polymer solution, and chromium acetate. In contrast, the tonguing gel consists only of a low-concentration polymer solution and chromium acetate. A significant limitation of the non-flowing gel component is that the swollen polymer particles are unlikely to infiltrate the rock matrix but can be extruded through fractures. Conversely, the tonguing gel can penetrate the matrix before gelation occurs but is unlikely to provide high residual resistance factors in fractures due to its relatively low strength.

3.3 The effect of reservoir temperature

In practice, in large volume applications of HPAM gels, the fluid must be formulated to function as a gellant at the surface. Subsequently, gelation should occur around the time of its departure from the wellbore and entry into the fracture. This approach confers the advantage of utilizing a gellant during pumping, with a preformed gel extruding through the fracture for much of the placement duration.

It is worth noting that under low reservoir temperature conditions (25-30 °C), gelation may occur more slowly than typically anticipated. For instance, Fig. 11 illustrates that at 26 °C, a formulation consisting of 0.5 wt.% polymer and 0.05 wt.% chromium acetate did not gel within one day. In fact, it took four days to achieve a week flowing gel from this formulation (Fig. 11c).

Several methods can accelerate gelation at low temperatures, one of which involves increasing the concentration of chromium acetate, a commonly used crosslinker. However, as shown in Fig. 12, a tenfold increase in chromium acetate concentration (from 0.05 to 0.5 wt.%) results in only a slight increase in gel viscosity. In contrast, substituting 0.05 wt.% of chromium acetate with an equivalent amount of chromium chloride leads to a much more significant increase in viscosity. The measurement was terminated because the polymer gel began climbing up the spindle of the viscometer.

However, substituting chromium chloride for chromium acetate results in the formation of a barely flowing gel within only 96 minutes. That is why to prevent the formation of gel inside of the mixing tank chromium chloride should be added at some point after the high-pressure injection pump. Fast gelation ensures the formulation gels at the bottomhole, efficiently filling significant cavities and preventing absorption of cement slurries. This method involves injecting small volumes (10-20 m^3) before cement slurries, thereby improving the efficacy of cementing operations to isolate depleted formations.

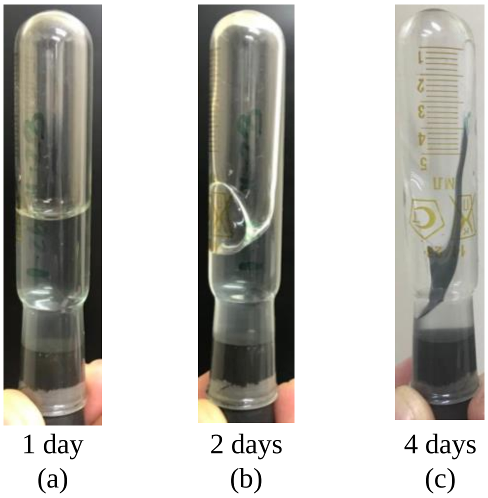


Fig. 11 Gel formulation [0.5 wt.% 6-7 mln Da and 5 wt.% HD polymer / 0.05 wt.% chromium acetate]. Brine –26.6 g/L. Temperature – 26 ºC.

In larger volume injection well gel treatments (50-200 m^3), the optimal gelation time may be within 1-2 days. To achieve this, polymers with different molecular weights were tested. For example, 20-30 million Da HPAM allows for the formation of a flowing gel (designated as gel C) within one day at 25°C, even in highly saline brine conditions (Ayrankol XIV brine). Subsequent aging at lower temperatures enhances the gel, leading to a moderately deformable non-flowing gel state (gel G) after 44 days (see Fig. 13). Polymers with lower molecular weights gel significantly more slowly, forming only weak, highly flowing gels (gel B) after 24 hours of aging.

The viscosities of the 20-30 million Da and 20 million Da polymer gels are presented in Fig. 14. The viscosity of the one-day-old gel consistently increased during the measurement interval, reaching up to 5,034 cp. In contrast, the viscosity of the 20 million Da HPAM stabilized at around 3,000–3,200 cp. The viscosity of the ~5 million Da polymer gel was too low to be detected by the viscometer used. A comparison of Figs. 12 and 14 clearly shows that polymers with higher molecular weights exhibited significantly higher viscosity values, even when the salinity of the brine was increased by as much as eight times.

Even though higher molecular weight gels demonstrated better performance under low-temperature conditions, aging

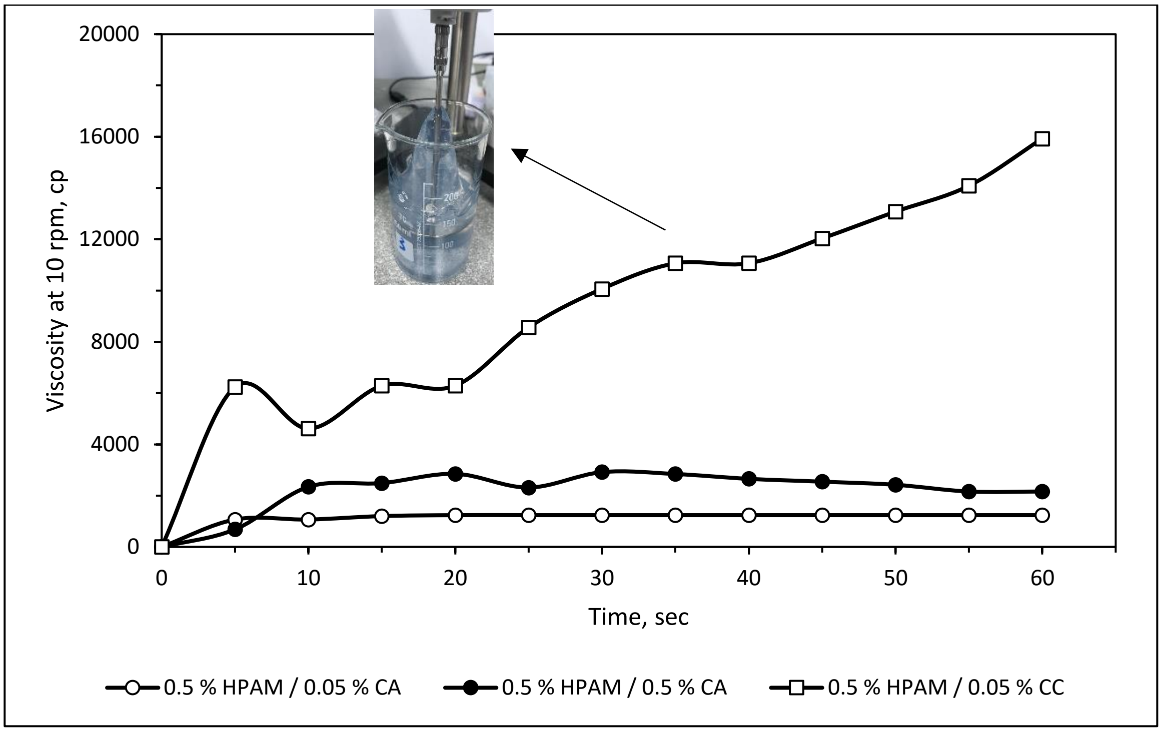


Fig. 12 Viscosity of 6-7 mln Da and 5% HD HPAM gels prepared in 26.6 g/L brine and aged at 27 °C for 24 h. Brine –26.6 g/L. Temperature – 26 °C. BDV-2C viscometer with R#2 spindle at 10 rpm.

Table 3. Gel behavior in high brine salinity (Ayrankol XIV) and elevated temperature (70 ºC).

Polymer	Time, days							
	1	2	3	4	20	30	40	60
20-30 mln Da 30% HD	F	F	F	Degraded gel				
~20 mln Da 5% HD	F	F	F	F	F	E	C	Degraded gel
~5 mln Da, 5 % HD	F	F	F	F	F	F-E	E	E

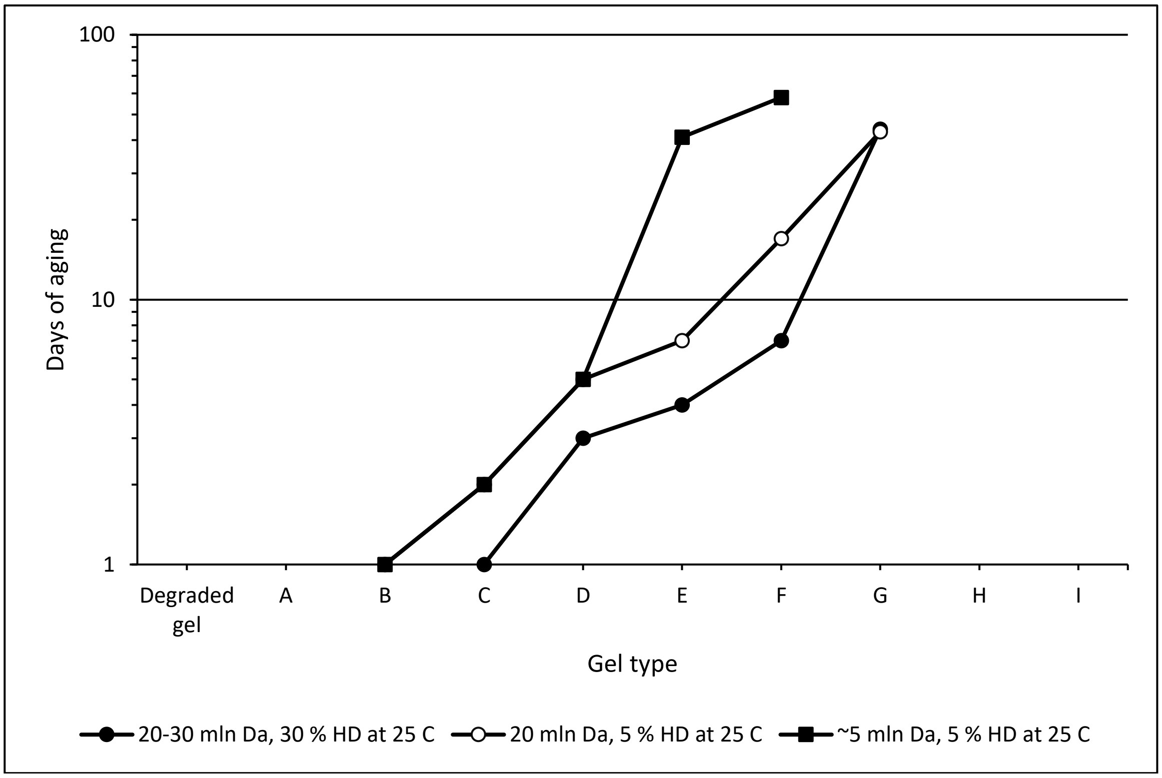


Fig. 13 The effect of aging on the gelation of [0.5 wt.% polymer / 0.05 wt.% chromium acetate] gels at low temperature (25 °C). Brine - high salinity Ayrankol XIV.

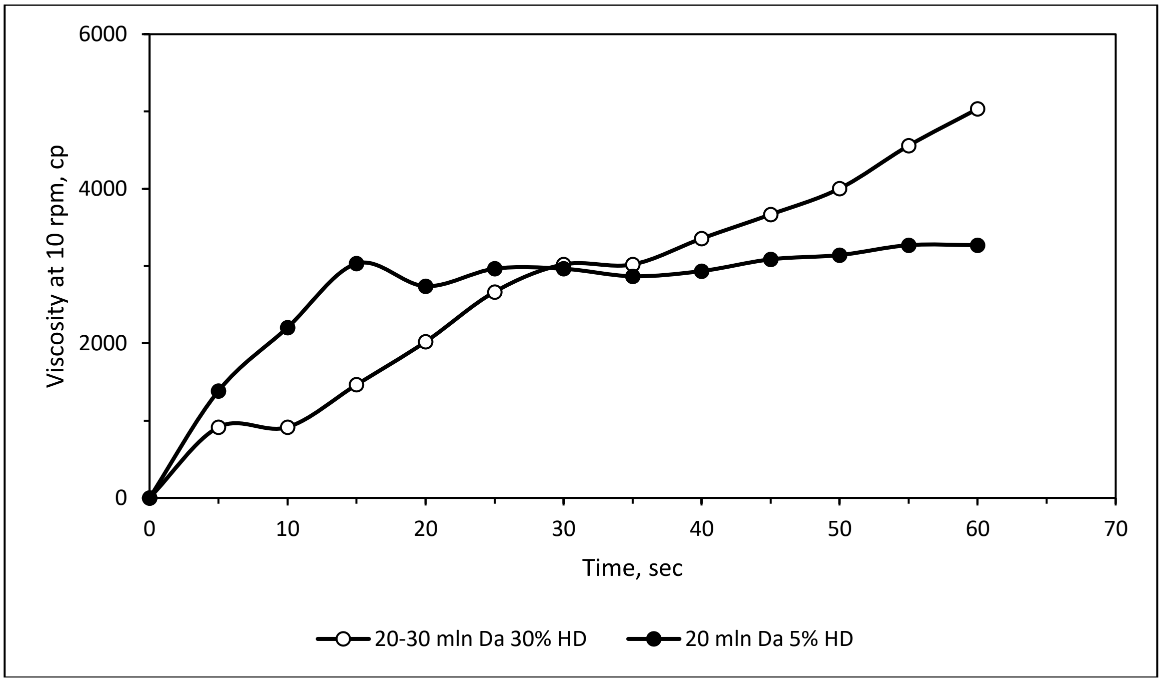


Fig. 14 Viscosity of 20-30 mln Da 30% HD and 20 mln Da 5% HD HPAM gels prepared in 222 g/L synthetic brine and aged at 27 °C for 24 h. Brine –222 g/L. Temperature – 26 °C. BDV-2C viscometer with R#2 spindle at 10 rpm.

at higher temperatures showed that gels with lower molecular weights are much more stable than those with higher molecular weights. For example, ~5 million Da HPAM gels show a reduction in strength after 30 days of aging at 70 °C but remain stable for two months. In contrast, 20 million Da and 20-30 million Da gels degrade after 40 and 3 days, respectively (Table 3). The same concept is demonstrated by Fig. 15 which shows the photos of the gels.

1 day 4 days 1 day 40 days 1 day 60 days

20-30 MW 30 % HD HPAM ~20 mln Da 5% HD HPAM ~5 mln Da, 5 % HD HPAM

Fig. 15 [0.5 wt.% HPAM / 0.05 wt.% chromium acetate] aged at 70 °C. Brine: high salinity Ayrankol XIV brine.

Given that many reservoirs operate at temperatures exceeding 70 °C, it is essential to study the behavior of gels at these higher temperatures (up to 95 °C) to ensure their effectiveness and stability under such extreme conditions. While stronger gels can be achieved by increasing polymer concentration, using high concentrations of high molecular weight polymers may present challenges due to the long dissolution time and elevated viscosity of the gellant, which can cause injectivity issues. To address this challenge for elevated temperature conditions (above 90 °C), the use of high concentrations of low molecular weight polymers in gels has been tested by various authors to treat conformance problems located near the wellbore,[33,34] as they allow for the formation of strong gels without significantly increasing the viscosity of the gellant. At the same time, low molecular weight polymers may be preferred over high molecular weight polymers, as the former dissolves much faster in brine. For example, lab observations show that a low molecular weight (~500,000 Da, 5 % HD) polymer dissolves in low salinity brine within 15 minutes at room temperature (20 °C). The fact that this polymer readily dissolves in fresh water or brine has also been noted in.[35]

Table 4 shows the results of the bottle tests conducted by using ~500,000 Da and 5% HD HPAM dissolved in Field C low salinity brine and crosslinked with chromium acetate. The gels were aged at 95 °C. As shown in Table 4, the [1 wt.% HPAM / 0.1 wt.% chromium acetate] formulation does not form a strong gel even after 3 days of aging at 95°C and was therefore disqualified from further testing. Increasing the polymer concentration to 1.27 and 1.5 wt.% allows for the formation of a type H gel; however, a notable reduction in gel strength is observed with further aging over 10-20 days. Increasing the polymer concentration to 2.5 or 5 wt.% ensures high gel strength and stability, provided that the concentration of the crosslinker is also relatively high, as the formulation with 0.05 wt.% chromium acetate did not gel even after three days of aging (Table 4).

Considering the described above observations, the gel with 2.5 wt.% polymer and 0.2 wt.% crosslinker was chosen as the optimum variant due to its moderate concentration of chemicals and excellent stability over time. However, when using much higher brine salinity (Ayrankol XIV brine) the same formulation could not maintain its gel strength after 20-

Table 4. The results of the bottle tests at 95 ºC with ~500,000 Da and 5 % HD HPAM.

Polymer concentration, wt.%	Chromium acetate, wt.%	Aging time					
		A	B	C	F	H	I
1	0.1	-	1	2-3 days	-	-	-
1.27	0.1	-	-	-	1 day 20 days	2 days	-
1.5	0.1	-	10-12 days	-	-	1 day 4 days	-
2.5	0.2	-	-	-	-	-	1 day 25 days
2.5	0.05	3 days	-	-	-	-	-
5	0.675	-	-	-	-	-	1 day 25 days

25 days of aging (Fig. 16). This can be attributed to the fact that under high salinity conditions, metal ions cause electrostatic screening of the negatively charged functional groups on the polymer chain, leading to the shrinking of polymer molecules. As a result, the gel formed is less strong and stable.

In low salinity brine (Field C)

In high salinity brine (Ayrankol XIV)

Fig. 16 [2.5 wt.% polymer / 0.2 wt.% chromium acetate] gels aged at 95 °C for 25 days. Polymer – 500,000 Da, 5 % HD HPAM. Brine – high salinity Ayrankol XIV brine.

Since the chosen gel formulation (2.5 wt.% polymer / 0.2 wt.% crosslinker) gels within less than 20 minutes and 3-4 hrs at 95 °C and 60 °C, respectively, it is recommended to cool the bottom hole zone of the well to at least 60 °C using a cold-water pre-flush before injecting the gel to ensure it does not become stuck inside of the wellbore.

3.4 The challenge of reducing permeability in fractures

The previous sections discussed the behavior of polymer gels in bulk volume. However, the final goal and one of the main challenges of conformance control and water shut off treatment is permeability reduction to water inside of highly conductive channels and wide fractures. Based on the results of the bottle tests shown in the previous section in this study 500,000 Da polymer with 5% hydrolysis degree was chosen for the permeability reduction inside of a fractured carbonate core.

The core flooding was done according to the procedure described above. Before the injection all the gels were matured at 60 °C to simulate the temperature regime that the gel will experience at the well bore and in the vicinity of the well. All the injections were done at 95 °C to simulate the temperature regime experienced by the gel and brine deep inside of the reservoir.

Figure 17 displays the pressure values recorded at the inlet (measured across the injection tube) and at two locations inside the fracture during the gel injection. As observed, all pressure values stabilized after approximately 1.5 fracture volumes (FVs) were injected into the model. In total, ~ 3.5 FVs of gel were injected into the model. The pressure gradient required to extrude the gel through the fracture is illustrated in Fig. 18 and is approximately 0.4 MPa/m. The stabilization of pressure values, coupled with the collection of gel effluent at the core outlet (Fig. 18), indicates the absence of inlet plugging. In other words, the fracture was filled with gel.

As the next step, brine post-flush was initiated at 95 °C. As Fig. 19 shows, the maximal pressure gradient required for the brine to rupture the gel was equal to 0.2-0.325 MPa/m.

In order to achieve a higher degree of plugging, [5 wt.% polymer / 0.5 wt.% crosslinker] gel was injected after the first post-flush. Figs. 20 and 21 show that notably higher pressure values were requried to extrude this gel through the fracture, as compared with the previous recipe (compare Fig. 17 and 18 with Fig. 20 and 21). However, the absence of continious pressure rise shows that the [5 wt.% polymer / 0.5 wt.% crosslinker] gel also propogated through the fracture and filled it.

The post-flush from the opposite direction (to mimic the inflow of brine from the formation into the wellbore) required 0.4 MPa/m to rupture the gel (Fig. 22). This is notably higher than that registered during the first post-flush (compare Figs. 19 and 22) and is explained by the higher concentration of the gel.

After the experiment the core was inspected for the presence of gel inside of the fracture. As Fig. 23 shows, the fracture was filled with concentrated gel which reduced the permeability to brine. The Table 5 below summarizes the results of the core flooding experiments. As can be seen, treating the 2-mm-wide fracture with the gels containing [2.5 wt.% polymer / 0.2 wt.% crosslinker] and [5 wt.% polymer / 0.5 wt.% crosslinker] respectively allowed for the attainment of a post-flush pressure gradient of 0.4 MPa/m (17.7 psi/ft).

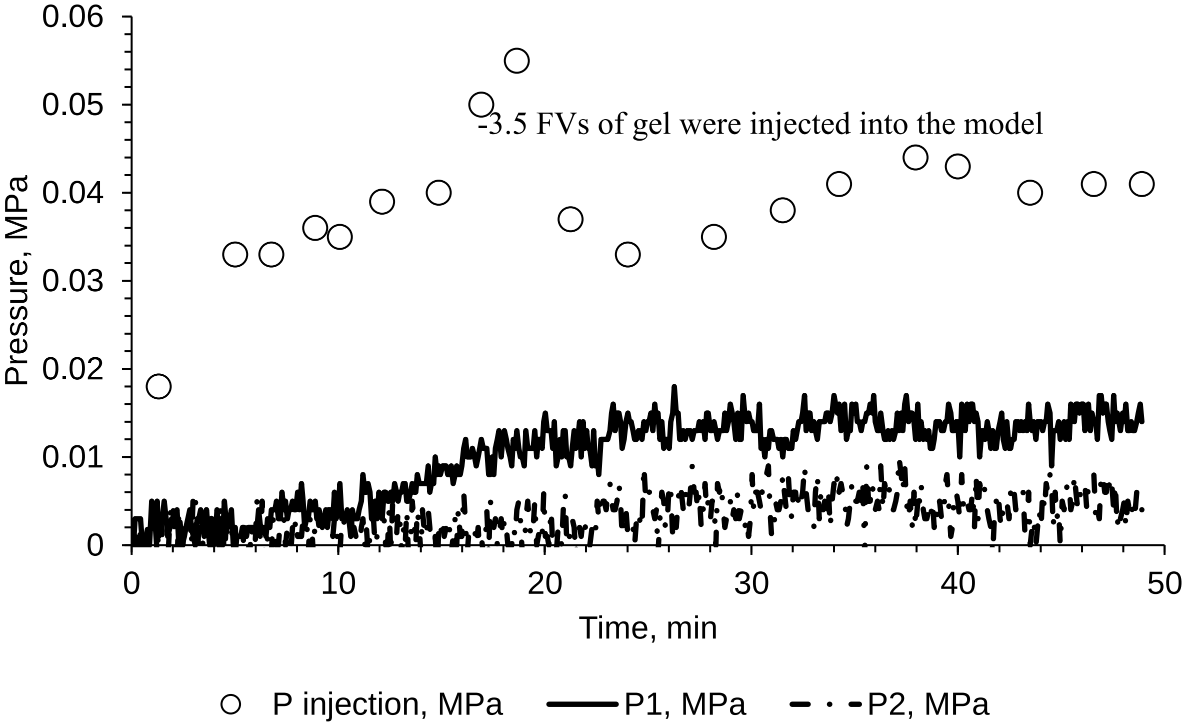


Fig. 17 The injection of [2.5 wt.% polymer / 0.2 wt.% crosslinker] gel at 0.43 cm^3/min and 95 °C into the 2-mm-wide fracture. Pressure values measured at three different locations: P injection – at the inlet; P1 – at the distance of 1.25 cm from the inlet; P2 – at the distance of 5.25 cm from the inlet.

Table 5. Results of the core flooding experiments.

Step	Maximal pressure gradient, MPa/m
[2.5 wt.% polymer / 0.2 wt.% crosslinker] gel injection	0.4
Brine post-flush from the opposite direction	0.2-0.325
[5 wt.% polymer / 0.5 wt.% crosslinker] gel injection	0.6
Brine post-flush from the opposite direction	0.35-0.4

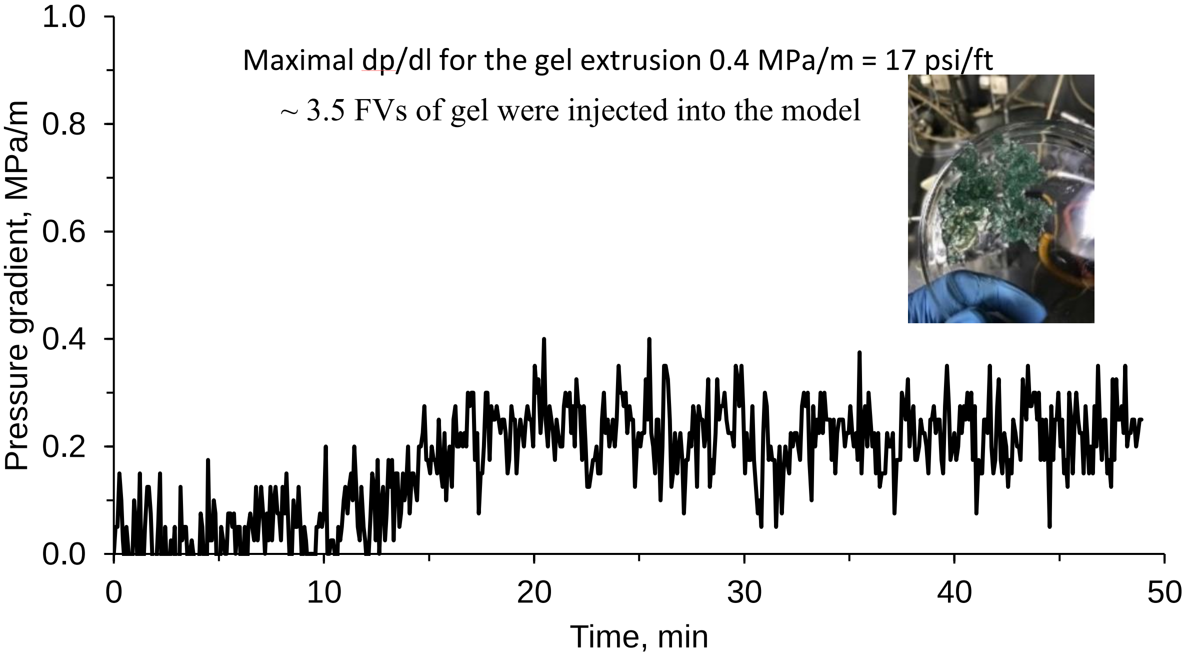


Fig. 18 The injection of [2.5 polymer wt.% / 0.2 wt.% crosslinker] gel at 0.43 cm^3/min and 95 °C into the 2-mm-wide fracture. Pressure gradient measured inside of the fracture.

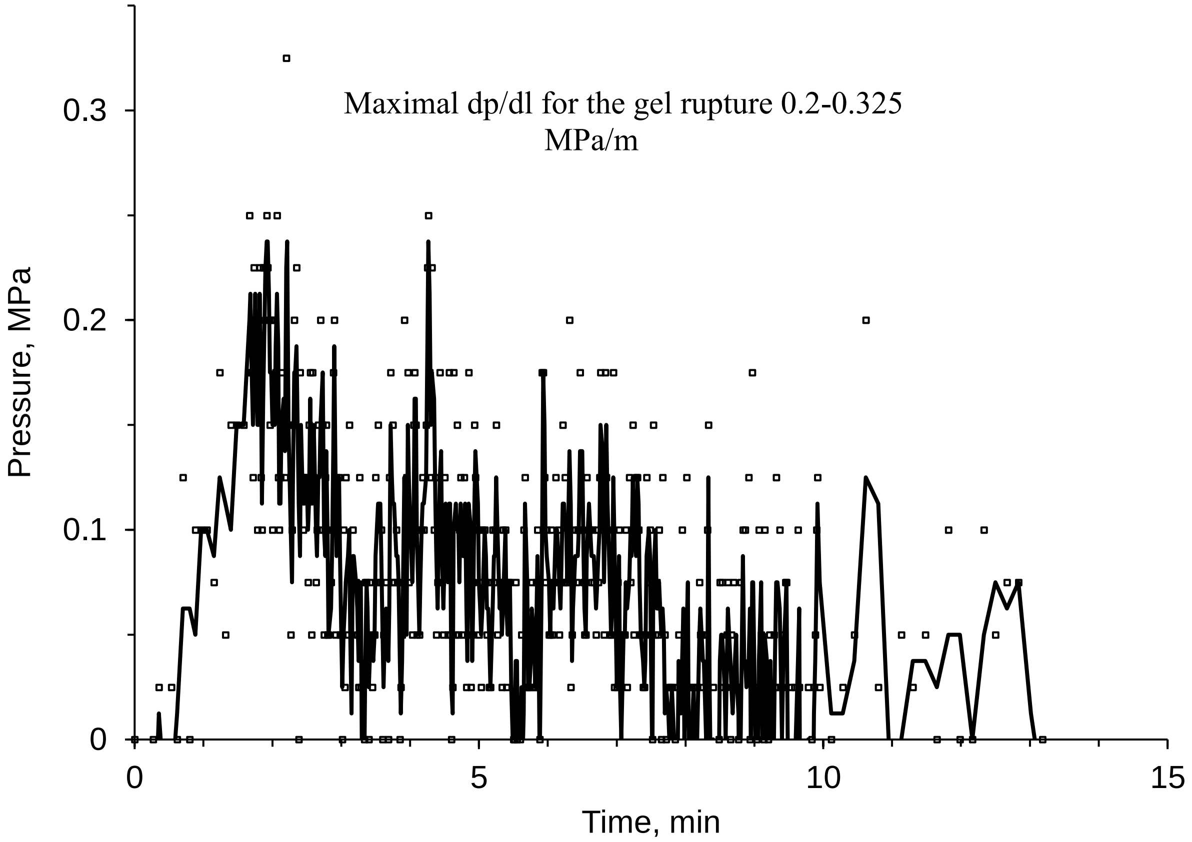


Fig. 19 Brine post-flush from the "production direction".

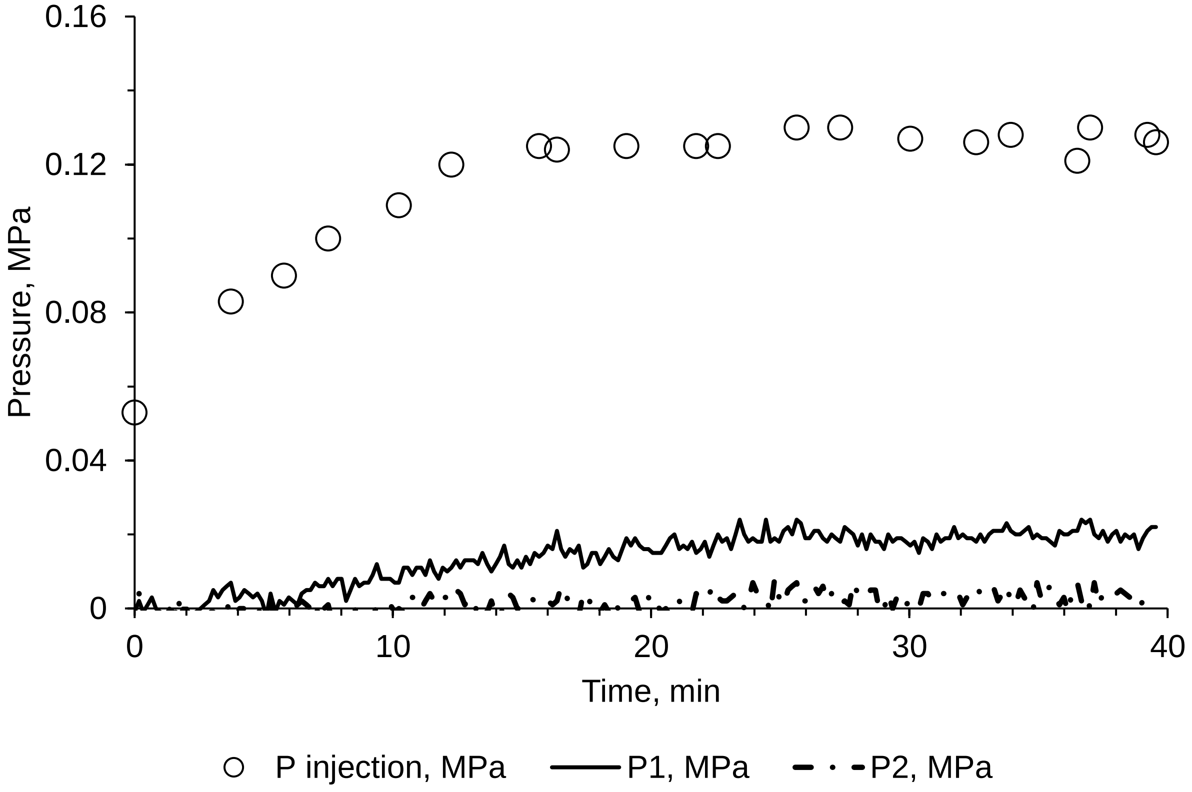


Fig. 20 The injection of [5 wt.% polymer / 0.5 wt.% crosslinker] gel at 0.43 cm^3/min and 95 °C into the 2-mm-wide fracture. Pressure values measured at three different locations: P injection – at the inlet; P_1 – at the distance of 1.25 cm from the inlet; P_2 – at the distance of 5.25 cm from the inlet.

Maximal dp/dl for the gel extrusion 0.6

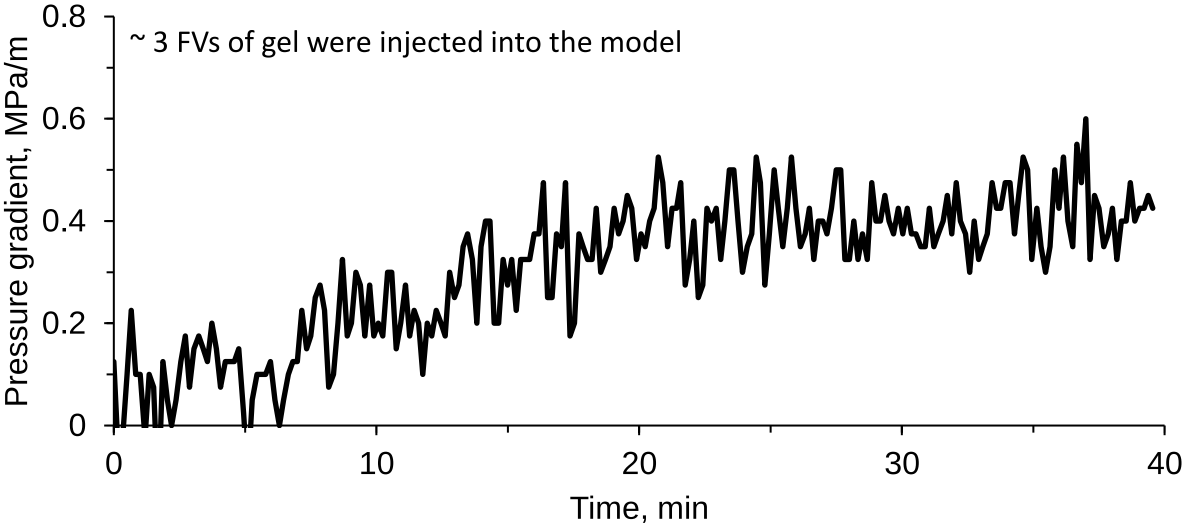


Fig. 21 The injection of [5 wt.% polymer / 0.5 wt.% crosslinker] gel at 0.43 cm^3/min and 95 °C into the 2-mm-wide fracture. Pressure gradient measured inside of the fracture.

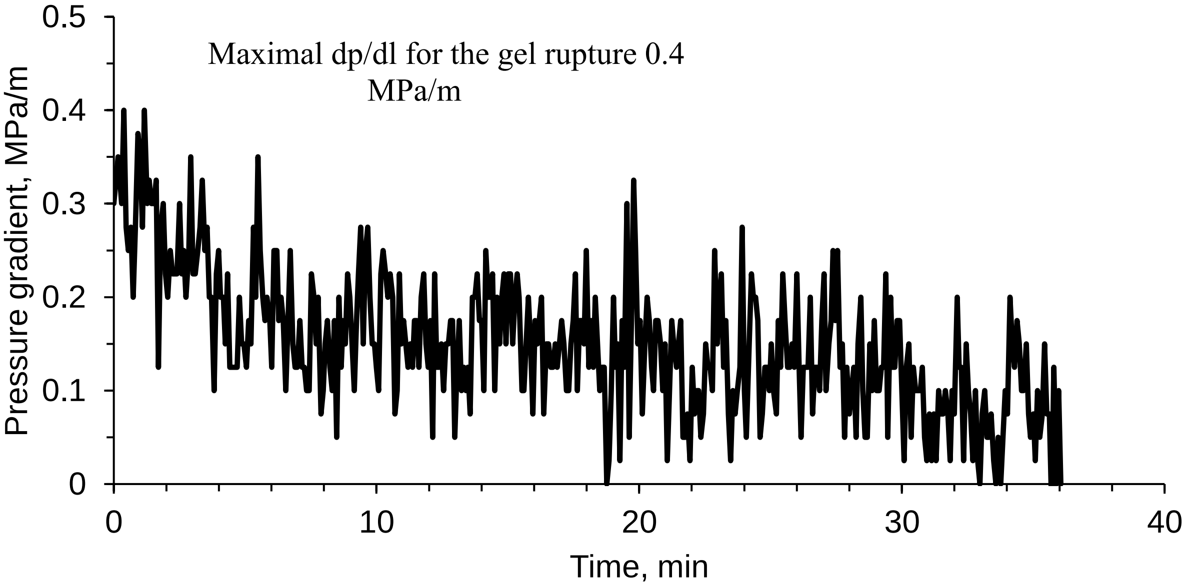


Fig. 22 Brine post-flush from "production direction".

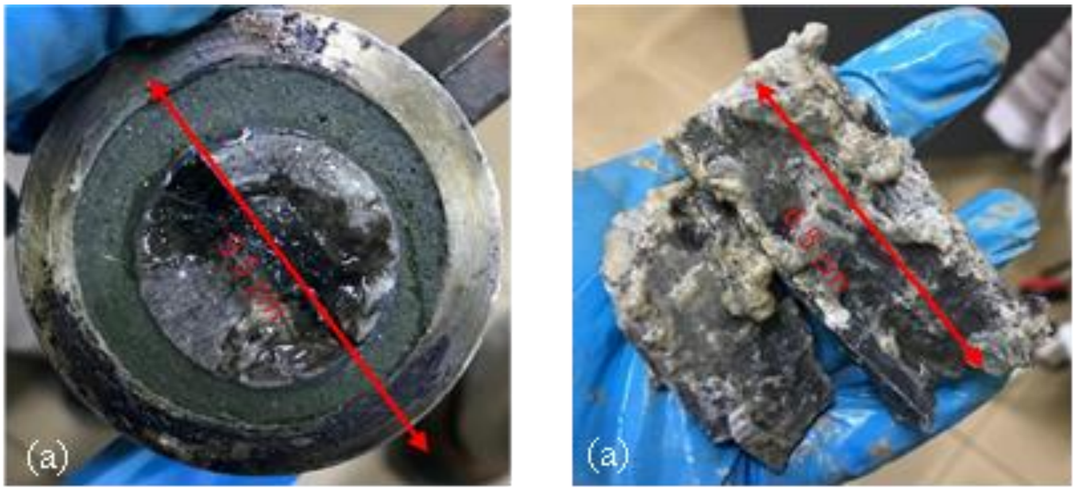


Fig. 23 (a) Core face after the treatment with polymer gel; (b) the concentrated polymer gel on the wall of the fracture.

This achievement is comparable to the post-flush pressure gradients obtained in low-temperature conditions by using low concentrations (0.5 wt.%) of high molecular weight polymer in gels and 1-mm-wide fractures at 40 °C.[36] This result demonstrates that high concentrations of low molecular weight polymer gels can achieve outcomes under unfavorable conditions — wide fracture, low matrix permeability, high post-flush brine salinity, and reverse injection directions — similar to those previously obtained in favorable conditions.

3.5 Risk of damaging oil permeability

Another challenge in conformance control and water shut-off operations is achieving selective permeability reduction to water without significantly damaging oil permeability. Bullhead injections, which are performed without zonal isolations, are often considered a fast and cost-effective option. However, properly evaluating the permeability damage to oil caused by polymer gel invasion into the oil-producing matrix is essential for making informed decisions on the design of the gel treatment. The invasion of polymer gel into the oil-producing matrix is more likely when the temperature is not high enough to gel the formulation upon exiting the wellbore, and the permeability of the matrix is relatively high. This situation often occurs in shallow, unconsolidated sandstone reservoirs. Therefore, this study examined the effect of polymer gel on oil permeability damage using a sand pack model.

The sand pack flooding experiment was conducted as described above. After injecting oil, the water and oil saturation values were established at 10.3% and 89.7%, respectively. The oil injection pressure stabilized at 0.07 MPa, corresponding to an oil phase permeability at connate water saturation of 11.2 Darcy. Subsequent injection of approximately 3 PVs of water resulted in pressure stabilization at 0.004 MPa, displacing 19.53 cm³ of oil from the model. The oil displacement coefficient was 41.4 %, resulting in an oil saturation of 52.5 % before injecting the gelling composition. Next, approximately 3 PVs of a [0.5 wt.% 6-7 million Da and 5% HD HPAM / 0.5 wt.% chromium acetate] gelling solution were injected at 1 cm³/min and 25 °C. During the injection of the gelling solution, the pressure increased from 0.03 MPa to 0.085 MPa, indicating a reduction in permeability due to gelation. The model was then shut in for three days to allow gelation to occur.

Three days after adding the cross-linker to the polymer solution, the model underwent water injection in the direction opposite to that of the gel-polymer composition injection. As a result, water permeability was found to be reduced by 190-200 times. Since the main purpose of the test was to evaluate oil permeability damage, only oil injection curves are shown below.

Next, oil was injected into the model in the opposite direction to the injection of the gelling formulation into the sand pack. The rapid increase in pressure during oil injection at a rate of 1 cm³/min after gel treatment indicates a significant decrease in oil permeability (Fig. 24). For comparison, the pressure during oil injection before gel treatment was only 0.07 MPa. Therefore, it can be concluded that oil permeability decreased by at least 200 times (14.056 / 0.07).

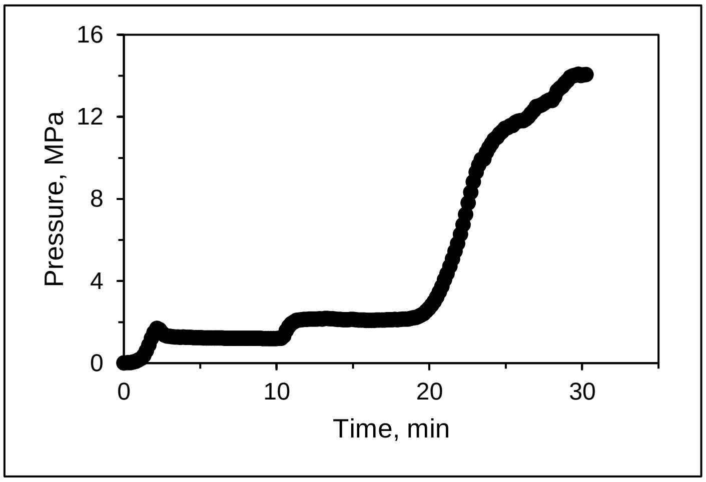


Fig. 24 The injection of oil at 1 cm³/min after gel treatment.

There is a likelihood that continuing the experiment would have led to a further increase in pressure (beyond 14.056 MPa), but to prevent model breakthrough, the flow rate was reduced to 0.25 cm³/min. Fig. 25 shows the results of oil injection into the model at a flow rate of 0.25 cm³/min. As can be seen, the injection pressure stabilized at 5.9 MPa.

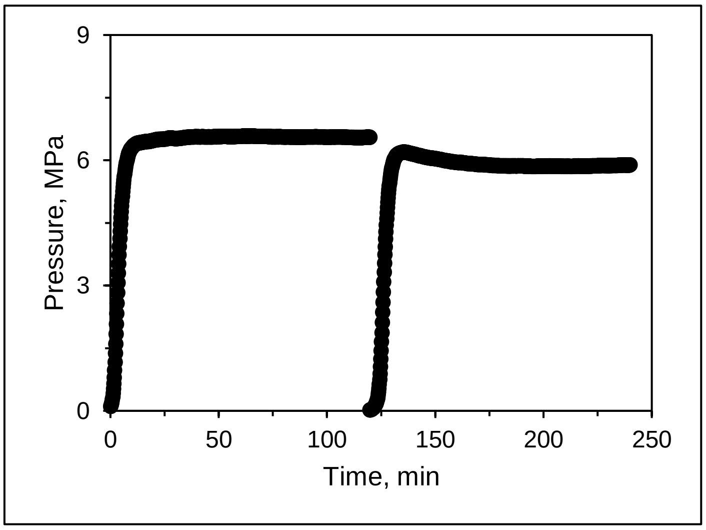


Fig. 25 The injection of oil at 0.25 cm³/min after gel treatment.

Injecting oil at a flow rate of 0.25 cm³/min until the pressure reached a steady state allowed the determination that the oil permeability had decreased from 11200 to 33 mD (by a factor of 340). The results of the calculations of oil permeability before and after gel treatment are shown in Table 6.

As shown in Table 6, the polymer gel used in this study [0.5 wt.% 6-7 million Da HPAM and 5% HD HPAM/0.5 wt.% chromium acetate] reduced the oil permeability of the sand pack by approximately 340 times. This result falls within the range reported by other researchers. For instance, the

Table 6. Permeability of the sand pack to oil before and after gel treatment.

Before gel treatment			After gel treatment			
Flow rate, cm^3/min	Pressure drop, MPa	Permeability to oil, mD	Flow rate, cm^3/min	Pressure drop, MPa	Permeability to oil, mD	RRF$_o$
1	0.07	11200	0.25	5.9	33	340

authors of[29] found that a polymer gel with a concentration of [0.4 wt.% 6-7 million Da HPAM / 0.01 wt.% chromium] reduced the oil permeability of a sand pack by 69 times. The difference in results can be attributed to the lower concentrations of polymer and crosslinker used in their study. Conversely, in the study[28] the oil permeability was reduced by 1,279 times following the treatment of a crushed carbonate sand pack with a gel composed of [0.75 wt.% 7 million Da HPAM / 0.03 wt.% chromium acetate]. The significantly higher permeability reduction to oil in this case is explained by the higher polymer concentration used.

To recover permeability to oil, 1 PV of 2 wt.% sodium pyrophosphate was injected into the sand pack and aged for 36 hours. The subsequent injection of oil resulted in an increase in pressure up to the level observed right after the gel treatment, indicating that the 2 wt.% sodium pyrophosphate solution could not restore permeability to oil (Fig. 26).

Since the 2 wt.% solution of sodium pyrophosphate proved ineffective for restoring oil permeability, and its solubility does not exceed 3 wt.% in cold water, it was decided to inject a 4 wt.% solution of sodium hydroxide into the model. After injecting around 1 PV, the injection pressure stabilized at 0.296 MPa. Oil was injected 28 hours after the NaOH injection. As seen in Fig. 26, the injection of sodium hydroxide proved more effective in restoring oil permeability, as the oil injection pressure decreased from 5.9 MPa to 2.085 MPa. However, complete restoration of oil permeability requires the pressure to decrease to 0.07 MPa, which was not achieved (Table 7).

Based on the results above, it can be concluded that polymer gellants can drastically reduce oil permeability, potentially by hundreds of times. Therefore, it is crucial to carefully manage the treatment of production wells to prevent polymeric gellants from infiltrating the oil-producing matrix. The use of sodium pyrophosphate and sodium hydroxide solutions proved ineffective in restoring oil permeability after gel treatment. Consequently, there is significant interest in identifying and testing alternative formulations for breaking the gel and restoring permeability, which can be investigated through core and/or sand pack flooding experiments. For instance, chemicals like sodium hypochlorite[37] and ammonium persulfate[38] can be used for breaking gels in bulk volumes. However, their effectiveness in restoring oil

Table 7. Permeability of sand pack to oil before and after the gel treatment and sodium hydroxide injection.

Before gel			After gel			After 4% NaOH		
Flow rate, cm^3/min	Injection pressure, MPa	Permeability, mD	Flow rate, cm^3/min	Injection pressure, MPa	Permeability, mD	Flow rate, cm^3/min	Injection pressure, MPa	Permeability, mD
1	0.07	11,200	0.25	5.888	33	0.25	2.085	94

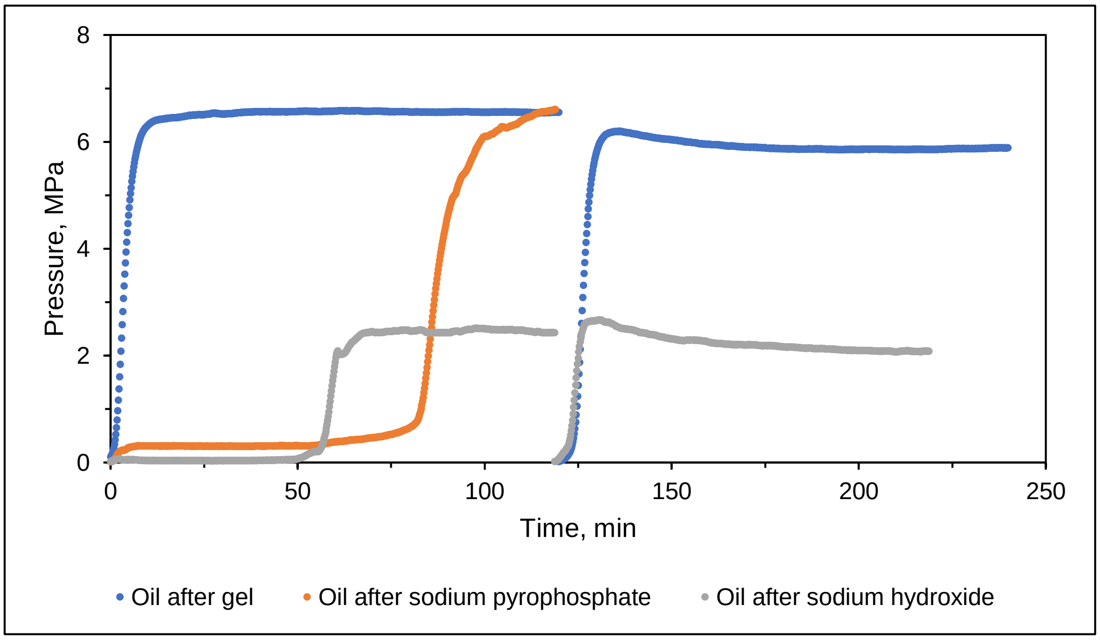


Fig. 26 Injection pressure observed during the oil injection after the gel treatment and following the injection of sodium pyrophosphate and sodium hydroxide solutions.

permeability still needs to be investigated through core and sand pack flooding tests.

The subject matter discussed in this article is highly relevant to the advancement of oil field development in Kazakhstan, which is home to approximately 250 oil deposits with total reserves exceeding 4.6 billion tons.[39] In recent years, there has been a growing trend among Kazakhstani researchers to explore various chemical enhanced oil recovery (EOR) methods, particularly the use of polymer gels.[40-42] This increasing focus underscores the critical role of these technologies in the development of mature oil fields.

4.Conclusion

This study has examined the multifaceted challenges in gel treatments for oil reservoirs, focusing on polymer dissolution, gelation time, gel strength, stability, resistance to brine flow, and oil permeability damage.

1-When polymer surface mixing time is limited to 30 minutes, lower molecular weight polymers (~7 million Da) should be preferred over high molecular weight polymers (~20 million Da). The latter cannot achieve even 25% of their maximum viscosity in 26.6 g/L brine, which is relatively low salinity for Kazakhstani oilfields.

2-Insufficient polymer mixing time can lead to severe water release from gels, which can be mitigated by increasing the polymer/crosslinker ratio.

3-Under low-temperature conditions, polymer gelation can be accelerated to within 20 minutes using chromium chloride. This formulation can be injected before cement slurries to prevent cement loss in large cavities.

4-The gelation time at low temperatures for 6-7 million Da gels, which can take over four days, can be reduced to 1-2 days by using very high molecular weight (~20-30 million Da) polymer gels with a standard formulation of 0.5 wt.% polymer and 0.05 wt.% chromium acetate, even in high salinity brine. However, these gels are not stable at 70°C.

5-At 95°C, sufficient gel strength and stability can be achieved using 500,000 Da HPAM with low salinity brine. Gelation time can be extended to several hours by pre-flushing with cold water. In exploratory wells with unproven reservoirs where cementing is not economically justified, this formulation can effectively temporarily isolate water-producing zones to flow test other intervals for hydrocarbon presence.

6-Core flooding has shown that treating a 2-mm-wide fracture under unfavorable conditions (high temperature, low matrix permeability, high post-flush brine salinity, and reverse flow conditions) with 2.5 wt.% and 5 wt.% 500,000 Da HPAM gels can provide a post-flush pressure gradient of 0.4 MPa/m. This is comparable to what 0.5 wt.% high molecular weight polymer gels achieve in a 1-mm-wide fracture under more favorable conditions.

7-Permeability damage remains a significant issue in gel treatments for production wells. 2 wt.% sodium pyrophosphate and 4 wt.% NaOH solutions were ineffective in restoring the permeability of the sand pack to oil after gel placement. As the potential of gel treatments to reduce unwanted water production comes with a high risk of losing oil production due to gel-induced permeability damage, developing more effective oil permeability restoring fluid formulations will stimulate interest among oil producers in gel treatments.

8-Future research should explore the use of sodium hypochlorite and ammonium persulfate to restore oil permeability after gel-induced damage in rocks with varying permeability.

9-It is recommended to investigate the use of fibers as a means to reinforce polymer gels, improving their strength and stability under high temperature and salinity conditions.

Acknowledgement

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not applicable.

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