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Application of a Salt-Resistant In Situ Cross-Linked Terpolymer Gel for the Daqing Oilfield

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ABSTRACT: Pubei Block, which is a subdivision of Daqing Oilfield, presents a challenging environment for conformance control due to its high temperature (average 80 °C) and salinity (13,451 mg/L), making it difficult for the polyacrylamide-based gel to maintain the necessary gel strength. To address this issue, this study aims to evaluate the feasibility of using a terpolymer in situ gel system that provides greater temperature and salinity resistance and pore adaptation. The terpolymer used here consists of acrylamide, acrylamido-2-methylpropane sulfonic acid, and N,N'-dimethylacrylamide. We determined that a formula with a hydrolysis degree of 15.15%, a polymer concentration of 600 mg/L, and a polymer−cross-linker ratio of 2.8 yields the greatest gel strength. The hydrodynamic radius of the gel was found to be 0.39 μm, indicating no conflict with the pore and pore-throat sizes determined by the CT scan. In the core-scale evaluation, the gel treatment improved oil recovery by 19.88%, of which 9.23% was through gelant injection and 10.65% through post water injection. A pilot test began in 2019 and has continued for 36 months until now. Within this period, the oil recovery factor increased by 9.82%. The number is likely to keep going up until the water cut (so far 87.4%) reaches the economic limit.
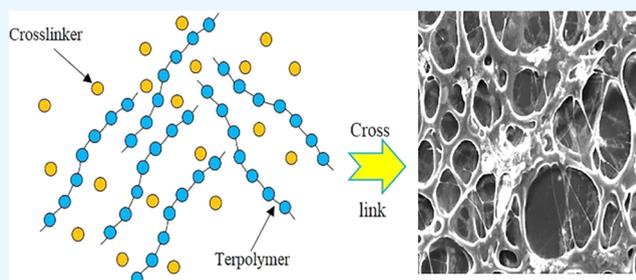
1. INTRODUCTION

Water flooding has been widely implemented to improve oil recovery after the primary recovery stage. The injected water replenishes the reservoir energy and displaces some amount of remaining oil, depending strongly on the sweep efficiency. For severely heterogeneous reservoirs, water typically seeks high-permeability flow paths such as natural or artificial fractures and only covers a small portion of the reservoir. In such circumstances, the amount of oil withdrawal is very limited and more importantly, water treatment becomes a headache. Liu et al. reported that the yearly cost of handling excessive water production across the globe is roughly 50 billion dollars. In consequence, there has been a growing interest in conformance control techniques in recent decades to address this issue.[1]

Over the years, the polymer gel has become one of the most promising conformance control agents as it is environmentally friendly, efficient, and cost-effective.[2] It typically can be classified into two major categories: in situ gel and preformed gel. In situ gel treatment refers to the injection of solutions of monomer/polymers and cross-linkers into the reservoir and the cross-linking process takes place in the desired areas with properly designed gelation time.[3] In contrast, preformed gel (PG) treatment uses gel particles directly for injections. They both have pros and cons and target different reservoir conditions. The in situ gel can transport deep into the reservoir and thus perform indepth treatments. However, preformed gel, considering the gel particle size and injectivity,

usually aims at plugging extra-large permeability channels such as fractures.[4] It tends to be more stable compared to the in situ gel since its performance is independent of the gelation time and the composition of the gelant, which may vary during transport.[5] Even though the development of microgels and nanogels broke the boundary a little, in situ gel is still very commonly used now.

Daqing oilfield is located in northeast China, which falls into the category of continental sedimentary basin. Most of the reservoirs show significant contrast in permeability, in both horizontal and vertical directions. Long-term water flooding (some are applied from the very early stage) has caused a few problems that worsen the heterogeneity, such as fine migration and sand production.[1] In such cases, gel treatment has been often used to control conformance. The application of gel treatment in Daqing oilfield can be traced back to the early 21st century (applications of clay gels in the 1990s are ignored).[6−8] For the in situ gel system, polyacrylamides (PAM) and hydrolyzed polyacrylamides (HPAM) are
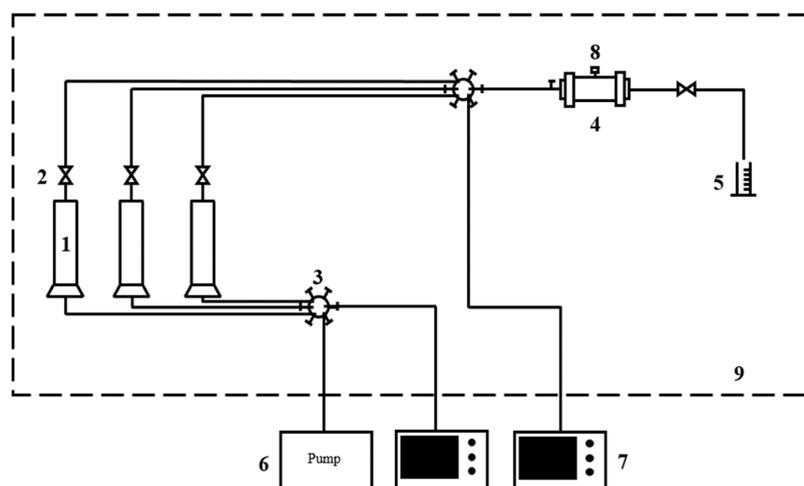
Figure 1. Experimental setup of the injection test and core-scale evaluation. Numbered parts are as follows: 1——stainless steel piston container, 2——switch, 3——six-way valve, 4——core holder, 5——graduated cylinder, 6——syringe pump, 7——pressure meter, 8——core holder, and 9——oven.

common choices of synthetic polymers. PAM- or HPAM-based in situ gels are profitable with a price of approximately 2−4 dollars per kilogram.[9] The formed bulk gel has a favorable gel strength, and the gelation time is adjustable by varying the polymer concentration and type of cross-linker. Owing to these advantages, its implementation in Daqing oilfield has been successful.[10−12] These gel systems also have notable disadvantages in terms of thermal and salt resistances. At high temperatures and salinities, their performances could be greatly compromised. Here, the high temperature and salinity do not have a uniform range, depending on the gel properties and additives involved in the gelant, but in general, refer to above 70 °C and 10,000 mg/L, respectively.[9,13]

Under these conditions, the molecular chains are more likely to be coiled, in which case, the gel tends to degrade more easily, and the gelation time becomes uncontrollable. To address such problems, researchers proposed introducing hydrophobic monomers into the polymer system, such as N,N'-dimethylacrylamide (DMAM), which restrain the hydrolysis of amide functional groups.[14−16] However, the presence of hydrophobic groups could cause low solubility of PAM or HPAM in brine, narrowing down the available concentration range. This problem can be resolved, to a certain extent, by bringing in an alkane solution to the gelant. Another way of countering high temperature and salinity is to use different types of copolymers. Bipolymers are often incorporations of acrylamide (AM) tert-butyl acrylate (tBA) or AM and acrylamido-2-methylpropane sulfonic acid (AMPS).[17−20] Terpolymers include AM-AMPS-N,N'-dimethylacrylamide (DMAM) and AM-AMPS-N-vinyl pyrrolidone (NVP).[21,22]

Pubei Oilfield is a subdivision of Daqing oilfield, of which many producing reservoirs have a temperature greater than 70 °C and a salinity of 12,000 mg/L on average. Recently, we have noticed that HPAM-based gel showed inefficiency while treating some of the reservoirs. Taking P-84 block as an example, it has a salinity of 14,712 mg/L and a temperature of 74 °C. It might be an extreme case due to excessive salinity but a good sample to state the problem. The operators considered solely permeability and porosity for the gel system design and ended up using HPAM and aluminum citrate (cross-linker). It turned out that the water cut was reduced by only 2.5% and the additional oil recovery was only about 3.52%. Another

block with less salinity (6517 mg/L) achieved 10.73 and 14.27% in those parameters. From laboratory measurement, the gel viscosities under these two conditions were 317 and 2684 mPa s, respectively. Here, we can tell that a salt-resistant gel system is needed for treating those reservoirs. In this work, we selected AM-AMPS-DMAM terpolymer and N,N'-methylene bisacrylamide (MBA) as the cross-linker to be our study object. We evaluated the strength of the gel synthesized with different formulas at a salinity of 13,451 mg/L and temperature of 80 °C to screen out the desired formula. We tested the size adaptability by comparing the gel hydrodynamic radius to the pore and pore-throat sizes. After that, we performed a core-scale test using a three-layered heterogeneous sandstone score and a pilot test in the P-16 block to further confirm its applicability. We believe this study can provide some references for gel treatment in not only Pubei oilfield but also others with similar reservoir properties.

2. MATERIALS AND GEL SAMPLE PREPARATION

2.1. Terpolymer Synthesization. We put AM, AMPS, and DMAM with a mass ratio of 4.27 g:0.44 g:0.81 g into a three-necked flask, as well as a small amount of initiator, ammonium persulfate (AS-1). We used NaOH solution to adjust the pH to 7 and injected nitrogen to remove the oxygen in the flask. After that, the reaction started and continued for 6 h at a constant temperature of 55 °C using a water bath. We then used a rotatory evaporator to dry out the produced colloidal substance and ground it to obtain the terpolymer for further operations.

2.2. Gel Preparation. Here, we prepared a series of gel systems by dispersing the prepared terpolymer, MBA (cross-linker), and BS-99 (Stabilizer) into brine with a salinity of 13,451 mg/L. BS-99 is a compound of thiourea and sodium sulfite. For convenience, we name this gel system RST in the following content. The degree of hydrolysis considered in this study varied from 5 to 25% by adjusting the concentration of alkene. The concentrations of AAD and MBA were 300−1800 and 60−3600 mg/L, respectively. The purpose of manipulating these parameters is to determine the desired formula, which provides the greatest gel strength in the reservoir condition. We used a rotary agitator to stir these solutions at 200−300 r/

min for 2.5 h and then placed them into an oven at 80 °C on standby.

3. METHOD AND PROCEDURE

3.1. Gel Strength Evaluation. We used Brookfield DV-II viscometer to continuously measure the viscosity of the gelant and we called gelation finished at the point where the viscosity stopped increasing. After that, we evaluated the gel strength through the viscosity, elastic modulus (G'), and loss modulus (G'') (measured using a viscometer). This allowed us to analyze the effects of each parameter in Section 2.2 on the gel strength and to screen out the formula that yields the greatest gel strength. We also took pictures of the gel structure using a scanning electron microscope (SEM), which could provide some understanding of the results from a microscopic viewpoint.

3.2. Pore-Throat Size Determination and Intrusion Test. In this part, we first determined the pore and pore-throat size distribution using computerized tomography (CT) scan techniques with cores collected from the target area. The next step is to compare the sizes with the hydrodynamic radii of RST obtained from the BI-200SM laser light scattering instrument. After that, we performed a core-flooding test with a core length of 30 cm × 30 cm × 4.5 cm and a permeability of 300 mD to examine the injectivity, resistance factor (RF), and residual resistance factor (RRF) of RST. The test involved initial water injection, gelant injection, and post water injection. The confining pressure used in the test was 5 MPa, and the injection rate was 0.1 mL/min for all injections. Figure 1 gives the experimental setup for this intrusion test and the following core-scale evaluation. It consists of three stainless steel piston containers (here, we only use two to fill with gelant and water), several valves, two six-way valves, a core holder, a graduated cylinder to collect the effluent, a syringe pump, two pressure meters set at the inlet and outlet, an annular pressure valve, and an oven, following the order in the chart.

3.3. Core-Scale Evaluation. Here, we used a three-layer artificial core to perform the oil displacement experiment. The permeabilities are approximately 100, 300, and 450 mD from top to bottom, with an average permeability of 300 mD and porosity of 22.7%. We used the same experimental setup but filled the other accumulator with oil. The detailed operations include the following:

(1) saturate the core with brine and measure the porosity and permeability of the core;

(2) inject oil into the core at 0.1 mL/min until no water is produced from the outlet and calculate the oil saturation based on the injected volume and pore volume;

(3) inject water at the same rate until the water cut reaches 98% from the outlet, which is determined by the proportion of water in the effluent for a certain period;

(4) inject 0.264 PV of RST solution into the core and turn off the inlet for 48 h to allow gelation; and

(5) reinject water (same as step 3).

We recorded the water cut, injection pressure, and oil recovery ratio throughout the experiment to evaluate the performance of RST.

3.4. Pilot Test. We picked a P-16 block as our test object because its salinity and temperature are close to the averaged values. It has an oil-bearing area of 0.72 km² and original oil in place (OOIP) of 61.77 × 10⁴ t. The permeability varies between 37.8 and 727.1 mD, with an average of 311.5 mD and

a range of 19.24. It currently contains three injectors and six producers. Before implementing gel treatment, the water cut was around 97%. To date, the gel treatment has lasted for 36 months. Similarly, we collected water cut, injection pressure, and oil recovery ratio to assess its performance.

4. RESULTS AND DISCUSSION

4.1. Gel Strength Evaluation. *4.1.1. Effect of Hydrolysis Degree.* In general, the hydrolysis degree of the terpolymer greatly impacts the gel's properties. If it is either too low or high, the terpolymer tends to have insufficient strength.[7] Thereby, we controlled the hydrolysis degree by adjusting alkane concentration and measured the corresponding gel viscosity. The polymer concentration and polymer–cross-linker ratio (PCR) were 600 mg/L and 3, respectively. As shown in Figure 2, the gel viscosity increases from 668 to 2174
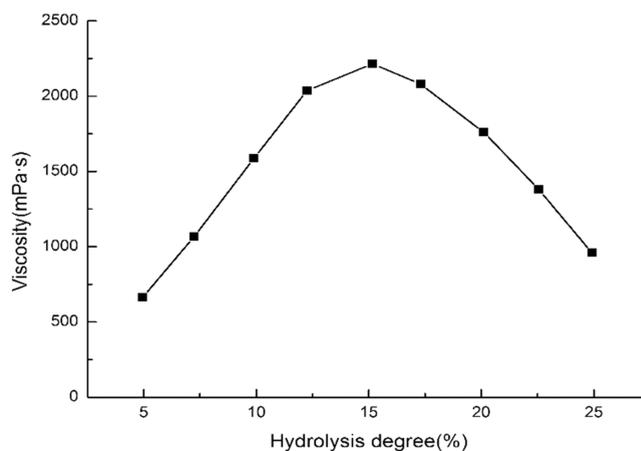
Figure 2. Bulk gel viscosity variation with the hydrolysis degree of the terpolymer.

mPa s when the hydrolysis degree changes from 5 to 15.15%. As the hydrolysis degree increases, the molecular chains hold more negative charges, which provide greater repulsive forces to stretch the molecular chains.[23] Once the hydrolysis degree gets above a certain value, the presented hydrogen bonds overshadow the repulsive force, and the polymer chain becomes less stretched. This explains the following decreasing trend, where the viscosity drops all of the way to 972 mPa s. The peak presents at 15.15% and thus, in the following content, if not specified, gels were prepared under this hydrolysis degree.

4.1.2. Effect of Terpolymer and Cross-Linker Concentration. In this part of the study, we considered polymer concentration of 300–1800 mg/L and PCR of 0.6–5.0. As exhibited in Figure 3, gel viscosity generally increases with the polymer concentration. Note there are some exceptions at higher PCR. But these points lay on the downtrend, deviating from the highest gel strength, and thus we will not discuss them systematically here. The increase is very significant when polymer concentration jumps from 300 to 600 mg/L and becomes less notable at higher values. The correlation between gel strength and PCR is very consistent, which shows an arch shape for all samples. Next, we used SEM to observe the formation of polymer network during gelation. We can divide such a process into the following two stages. The first stage denotes the tenuous reticulate structure caused by the electrostatic interaction and molecular force during dissolution
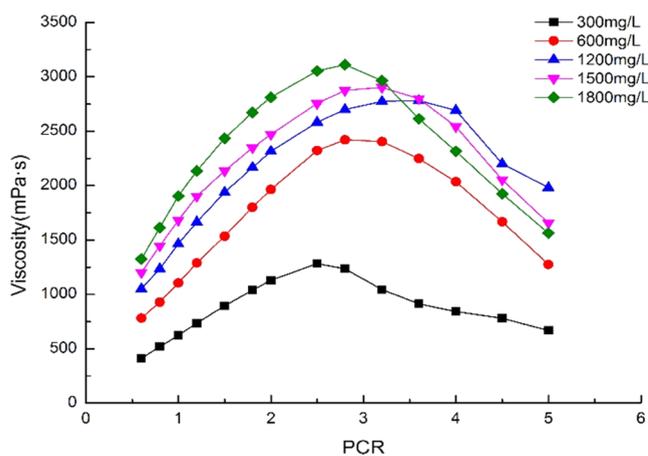
Figure 3. Bulk gel viscosity variation with polymer and PCR.

Figure 5. Log−log plot of gel storage and loss modulus at various polymer concentrations.

Figure 6. Gel storage and loss modulus at the polymer−cross-linker ratio of 0.6, 2.8, and 5.0.

and swelling processes. The second refers to the network structure formed by intramolecular and mainly intermolecular cross-linking.[7]

The gel strength depends on the structure of the network and the internal friction between the networks.[6] Both under-cross-linking and over-cross-linking could lead to inadequate gel strength. Figure 4 gives the gel network structures at elevated PCR. At PCR = 0.6 (a), the gel is over-cross-linked, giving an irregular, scrambled, and weak network structure. However, at PCR = 2.8 (b), the network becomes dense and firm, the condition under which it has great structural strength and inner frictions. This explains the upward trend in Figure 3. As PCR keeps increasing, such as (c), the gel tends to be under-cross-linked. That means the insufficient amount of cross-linker is unable to connect all of the polymer chains, which is the reason for the right part of the arch.

Viscosity itself cannot adequately represent the gel strength because it only reflects the shear resistance of the gel.[24] Thus, we further analyzed its viscoelastic properties through G' and G''. From Figure 5, one can tell that both G' and G'' increase with the polymer concentration (PCR = 2.8). A big increase occurs when changing the polymer concentration from 300 to 600 mg/L, which is consistent with what has been found above. The other changes are relatively small, especially for G', at which those curves nearly overlap each other. Considering these findings and taking economic benefits into account, we call 600 mg/L the proper polymer concentration in this study.

Next, we measured G' and G'' of gels with a polymer concentration of 600 mg/L and different PCRs of 0.6, 2.8, and 5 (Figure 6). Unfortunately, the differences between curves are very subtle for us to make a judgment. The measured
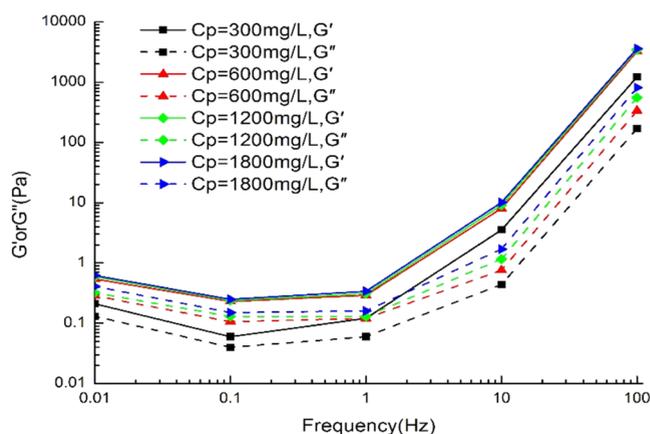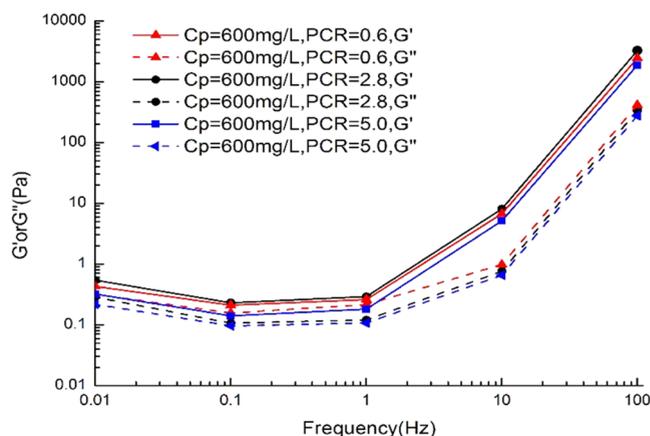
hydrodynamic radius (Figure 7), to a degree, proves that PCR of 2.8 yields the highest gel strength. Now we obtained the desired formula, which includes a hydrolysis degree of 15.15%, polymer concentration of 600 mg/L, and PCR of 2.8. We used this gel formula in the following studies.

4.2. Size Analysis and Injection Test. While the gelant flows through the porous media, it is subjected to internal friction between the molecules and the viscous and elastic forces between the gelant molecules and the pores walls. If the size of the gelant molecules is inadequate, they may encounter great resistance during the injection, leading to injectivity problems. Or even worse, it could plug the flow channel and
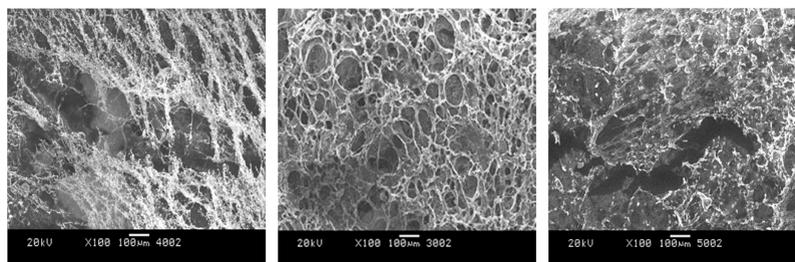
（a）C_p=600mg/L,PCR=0.6   （b）C_p=600mg/L,PCR=2.8   （c）C_p=600mg/L, PCR=5.0
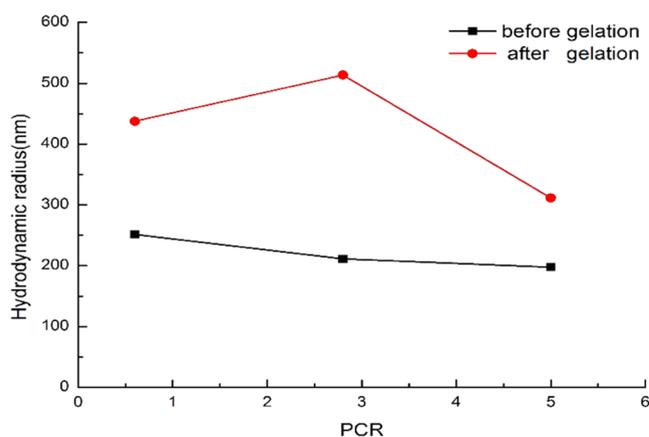
Figure 4. Gel structures revealed under SEM.

Figure 7. Gel hydrodynamic radius before and after gelation, at the polymer−cross-linker ratio of 0.6, 2.8, and 5.0.

damage the low permeability, oil-rich zones. Thus, we performed a size analysis with a CT scan, examining whether the size of the gel is proper, with respect to the pore and pore-throat size. From Figure 8, the pore radius distributes in a range of 1.7−5.3 μm, with an average radius of 2.4 μm. The pore-throat radius is between 3.5 and 11.3 μm, with an average of 8.7 μm. The molecules involved in the gelant have an average radius of 0.39 μm, which is less than half of the pore radius and one-eighth of the pore-throat radius. This

comparison could partly reveal the adaptability of this gel system, and we need to test its injectivity to provide more evidence. As seen in the injection pressure recording (Figure 9), there is no sign of plugging or extreme difficulty in
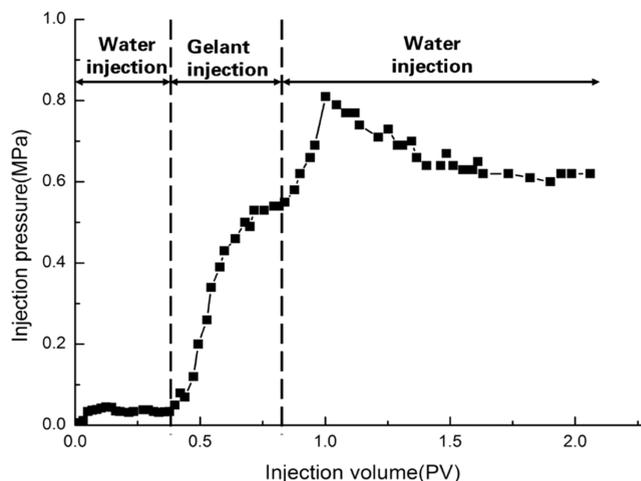

Figure 9. Injection pressure versus injection volume during the injectivity test.

penetrating during gelant injection. The injection pressures did increase because the gelant is thicker than brine, but the curve
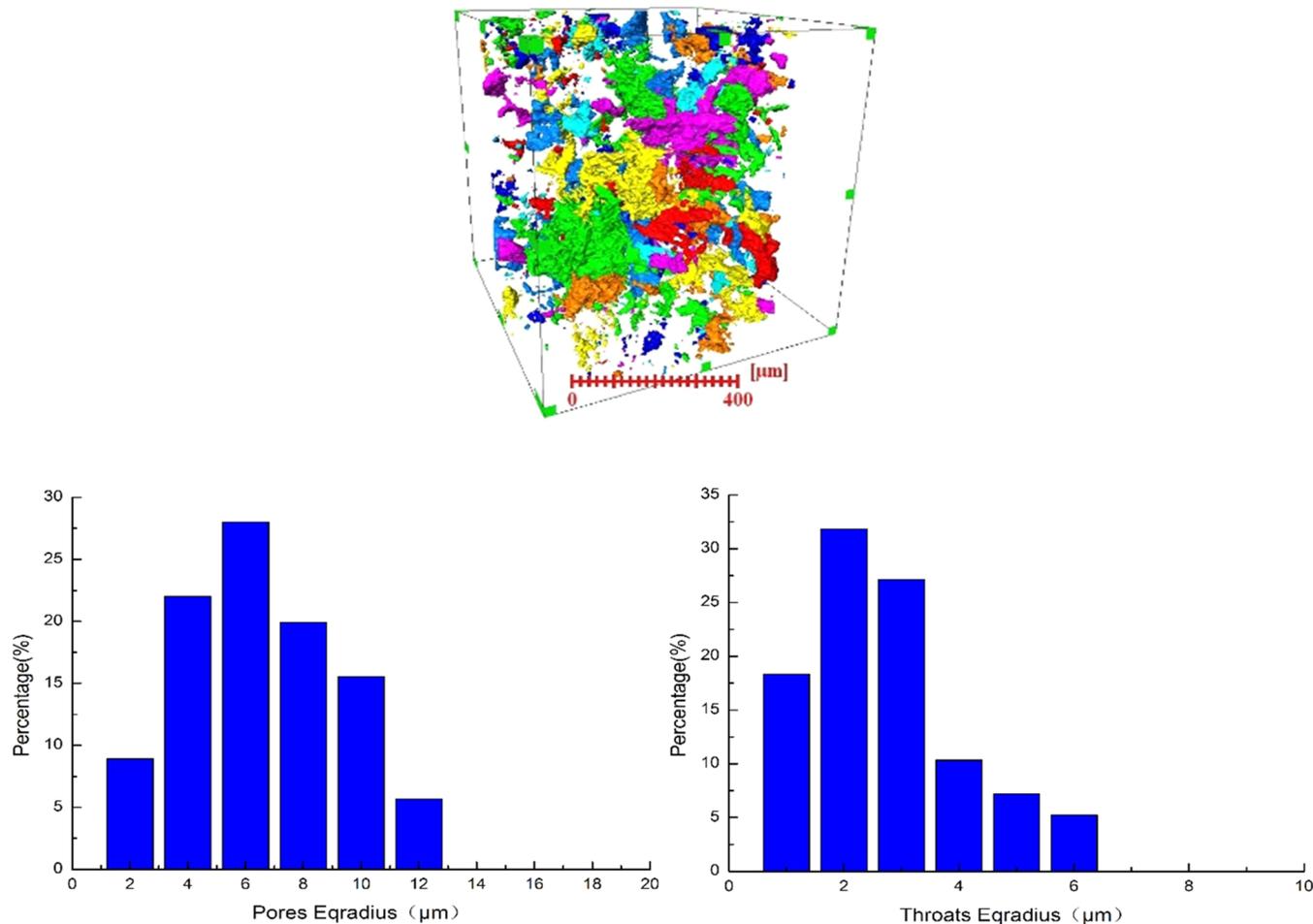

Figure 8. Pore and pore-throat size distribution given by CT scan.

turned flat at the very end of this stage, which demonstrates that the flow is sort of a "steady state". Here, RF and RRF are roughly 14.0 and 17.5, respectively, indicating the gel could bring some resistance in the high-permeability zones, either as a mobility control (before gelation) or as a plugging agent.

4.3. Core-Scale Evaluation. Figure 10 gives the injection pressure, water cut, and oil recovery ratio recorded during the
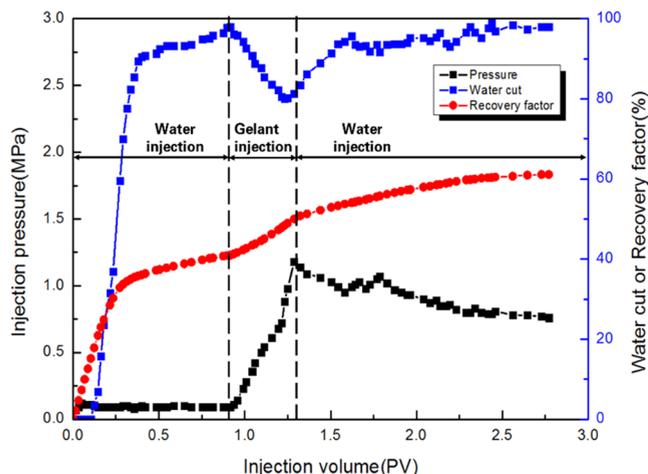

Figure 10. Injection pressure, water cut, and oil recovery factor recorded during the core-flooding test.

test. The first round of water injection produced 41.47% of the OOIP and then stopped when reaching the dummy economic limit, a water cut of 98%. The gelant injection started at this point, which functioned very similarly to polymer flooding, increasing the viscosity of the displacing fluid to improve the sweep efficiency. With no presence of fractures or large flow channels, the improvement was very significant. It increased the recovery factor by 9.23% and reduced the water cut significantly, from 98 to 80%. In the second round of water injection, RST hindered the flow in high-permeability zones and diverted water into oil-bearing, low permeability zones.

There was a continuous but slight drop in the injection pressure, indicating the gel has a sustained effect during water injection. This stage increased the recovery factor by another 10.65%, and the overall improvement was 19.88%. This number by no means represents the improvement it could bring to the field operations but shows that gel treatment with RST has great potential to succeed in Pubei block.

4.4. Pilot Test. The pilot test started with injecting 0.45 PV gelant into the reservoir, which took around 12 months. Following that was a short period of shut-in to enable gelation and the post water flooding. As shown in Figure 11, the injection pressure changes from 11.01 to 14.75 MPa during gelant injection and remains quite stable for the next 14 months. It then dropped 1.7 MPa in about 11 months. The treatment improves the daily oil production considerably, from an average of 5.7 to 26.7 m^3/d. A sustained decrease is inevitable as time goes on, but at a relatively low rate. For the entire treatment, it improved the oil recovery efficiency by 9.82%. In addition, it showed the ability to reduce water production by reducing the water cut by a dozen percent in the early stage and a few percent later. Up to the end of the trial, the water cut was still 10.6% less than the economic limit, which means the oil recovery can be further improved.

5. CONCLUSIONS

This study aimed to evaluate the effectiveness of RST gel treatment in the Pubei block of Daqing oilfield in a systematic manner. Initially, we conducted a sensitivity analysis to examine the impact of various parameters such as the hydrolysis degree, polymer concentration, and cross-linker concentration on the gel strength. Based on the results, we identified the optimal formula for gel formation that includes a hydrolysis degree of 15.15%, a polymer concentration of 600 mg/L, and a PCR of 2.8. Additionally, we verified that the molecular size of the RST gel is suitable for the pore and pore-throat size of the reservoir.

The results of this study indicate that the RST gel treatment was effective in improving oil recovery in the Pubei block of
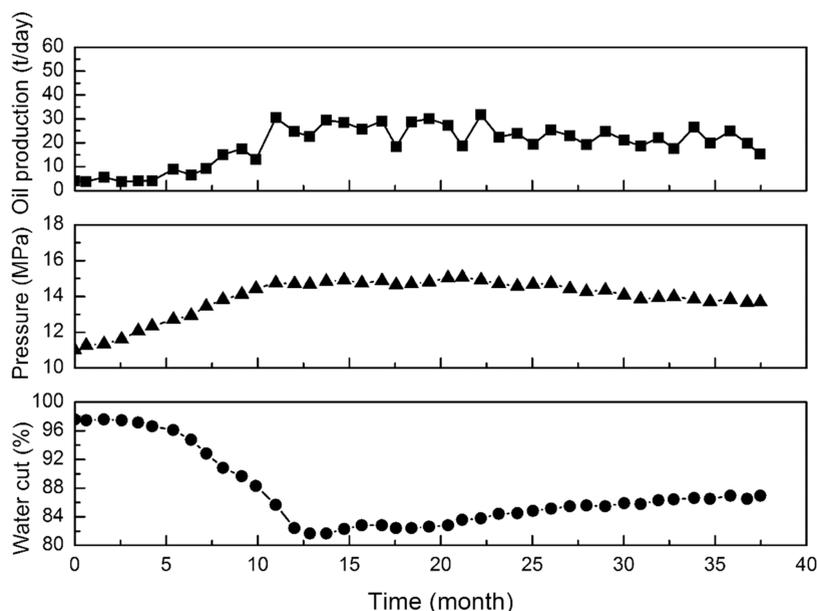

Figure 11. Oil production, pressure, and water cut recorded during the gel treatment in the P-16 block.

Daqing Oilfield, where the high temperature and salinity posed challenges to conventional treatments. The core-scale evaluation showed that gelant injection and post water flooding improved oil recovery by 9.23 and 10.65%, respectively. The pilot test over a 3-year period resulted in a 9.82% increase in oil recovery, and there is potential for further production as the water cut remained below the economic limit by the end of the test. The molecular size of the RST gel was found to fit well with the reservoir pore and pore-throat sizes. These findings suggest that RST gel treatment could be a viable option for other reservoirs with similar properties, and this study may serve as a reference for future gel treatments.

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Notes

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