Experimental Investigation of a Novel Nanocomposite Particle Gel for Water Shutoff Treatment in Mature Oilfields

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ABSTRACT: Conventional preformed particle gels suffer from insufficient salt tolerance and weak mechanical properties after water absorption, which reduce the water shutoff effect in mature oilfields. In this paper, a nanocomposite particle gel (NCPG) is synthesized by copolymerization of acrylamide (AM) and 2-acrylamido-2-methylpropane sulfonic acid (AMPS) using laponite RD (LPT) as a physical cross-linker and N,N-methylene-bisacrylamide (MBA) as a chemical cross-linker via in situ free radical polymerization. Compared with the NCPG without LPT, both the swelling rate and mechanical properties of NCPG added with LPT are found to be improved. In addition, the pore sizes of the network of the swollen NCPG are smaller than those of the sample without LPT, and the thermal stability is also slightly enhanced. The swelling rate of NCPG increases with increasing AMPS concentration. The water absorbency of NCPG first increases and then decreases with increasing MBA and APS concentrations. The NCPG is sensitive to alkaline medium due to the presence of sulfonic acid groups on the molecular chains of the NCPG. The synthesized NCPG exhibits good salt tolerance at 80 °C in formation water. The plugging rate of the NCPG to a sand-pack is above 90%, and the residual resistance factor reaches 19.2 under reservoir conditions. These results indicate that the NCPG may have potential application for water shutoff treatment in mature oilfields.

1. INTRODUCTION

Many oilfields have entered the stage of high water cut with the further development of oilfields. Chemical water shutoff, as a cost-effective technology, has become the main method for enhancing oil recovery in mature oilfields. As the water shutoff technologies developed, petroleum workers have developed different types of plugging agents, such as in situ polymer gels, preformed particle gels (PPGs), and so forth. In situ polymer gels were cross-linked polymer gels (CPGs) composed of polymers and cross-linkers. These kinds of polymer gels were injected as a solution and formed gels in the formation to plug the fixed water-flowing channels of reservoirs. However, CPGs had some drawbacks, such as chromatography, shear degradation, and dilution by formation water, which would reduce the plugging capacity of CPGs.

PPGs were synthesized at surface facilities and overcame the drawbacks that existed in CPGs. After injected into the formation, PPGs could go deep into the formation by elastic deformation and block the high permeability area of the reservoir after absorbing water and swelling. So far, some types of PPGs have been developed. For example, Zhou et al. synthesized an acid-resistant PPG system by the radical polymerization of acrylamide (AM), N,N′-methylene bisacrylamide (MBA), and dimethyldiallylammonium chloride, and this PPG system exhibited good water absorbency and viscoelastic properties under acidic conditions for conformance control. Heidari et al. synthesized a PPG from sulfonated polyacrylamide and Cr(OAc)₃. The equilibrium absorbency of this PPG system was 470.49 in pure water and 12.61 in formation water of 15,000 mg/L TDS at 80 °C. However, the swelling ratio of the conventional PPG was too fast, which made it difficult to migrate into the depth of the reservoir. The swollen conventional PPG was fragile. It resulted in a decrease in the plugging efficiency of PPG. In addition, conventional PPGs were more sensitive to the harsh conditions of reservoirs, such as high salinity. It could cause a dramatic decrease in the water absorbency of PPGs.
In recent years, new types of PPGs such as nanocomposite hydrogels were developed for water shutoff due to their good thermal stability and deformability.\(^{17,18}\) Furthermore, nanocomposite hydrogels exhibited better mechanical properties than conventional hydrogels.\(^{17,19}\) However, these investigations were conducted at room temperature, but no systematic study on pH-sensitive PPGs was reported for water shutoff treatment in a mature reservoir, considering the reservoir conditions such as the temperature, salinity, and pH values of formation water. In this paper, a pH-sensitive nanocomposite particle gel (NCPG) was developed using AM and 2-acrylamido-2-methylpropane sulfonic acid (AMPS) as monomers by in situ copolymerization. Laponite RD (LPT) nanoparticles were added to the polymerization system as a physical cross-linker, and N,N-methylene-bisacrylamide (MBA) was used as a chemical cross-linker. Considering different parameters, such as the AM/AMPS ratio, LPT concentration, cross-linker, and initiator concentration, the swelling rate of NCPG was investigated at different temperatures, and the pH sensitivity and salt tolerance of NCPG were also evaluated. The thermal stability and microstructure of NCPG were characterized using Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), and thermogravimetry–differential scanning calorimetry (TG–DSC). The sand-pack flowing experiments were conducted to evaluate the plugging capacity of NCPG for water shutoff operation.

2. MATERIALS AND METHODS

2.1. Materials. AM (99%) was purchased from Jining Hongwei Chemical Co., Ltd. (Hong Kong, China). 2-AM-2-methylpropylsulfonic acid (AMPS, 99%) was purchased from Jinan Beate Chemical Co., Ltd. (Shandong, China). LPT nanoparticles (99%, average size: 28 nm) were provided by Nanjing Baiyike New Material Technology Co., LTD (Jiangsu, China). The reagents including ammonium persulfate (APS, 99%), MBA (99%), sodium hydroxide (NaOH, 97%), and hydrochloric acid (HCl, 36.5%) were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China), which were of AR grade. All chemicals and reagents were used without further purification. Freshwater and formation water were used in these experiments. The formation water was provided by the Ansai Oilfield (Shanxi, China), and its ionic content is given in Table 1.

2.2. Methods. 2.2.1. Preparation of NCPG. LPT (0.75 g) was dissolved in fresh water (100 mL) with magnetic stirring, then AM (18 g) and AMPS (2 g) were added into the solutions and stirred for 2 h until a uniform solution was formed. The chemical cross-linker (0.05 g) and initiator (0.05 g) were added to the solution and stirred until completely dissolved. The prepared solution was set in a 60 °C thermostat water bath for 6 h. The formed bulk gel was cut into 1−2 cm pieces and placed into the fume hood to dry spontaneously. The schematic diagram of synthesis of NCPG is shown in Figure 1.

2.2.2. Water Absorbency. The weighted NCPGs were soaked in water, and their quality was measured regularly after draining the water on the surface of the NCPG. The swelling rate of NCPG was calculated using the following formula

\[
Q = \frac{M_t - M_0}{M_0} 
\]

where \(Q\) is the swelling rate of NCPG in g/g; \(M_0\) is the initial weight of NCPG in g; and \(M_t\) is the weight of NCPG after soaking in water for \(t\) hours in g.

2.2.3. Mechanical Properties. The mechanical properties, such as the stretched length and breakdown pressure, were measured based on the bulk gel using a universal material tester and a gel strength tester. A gel sample with a length of 3 cm and a diameter of 1 cm was prepared. The uniaxial tensile test was carried out at a rate of 10 mm/min until the NCPG sample broke and recorded the maximum tensile length. The bulk gel with a diameter of 3 cm and a height of 1 cm was prepared. The breakdown pressure of the bulk gel was measured using a gel strength tester after soaking in water for 48 h.

2.2.4. Thermogravimetry–Differential Scanning Calorimetry. The thermal stability of NCPG was evaluated by TG–DSC. The NCPG was set in a hermetic pan after absorbing formation water for 48 h, and the measurement was conducted...
in the temperature range of 30–300 °C at a scanning rate of 5 °C/min in a nitrogen atmosphere.

2.2.5. Scanning Electron Microscopy. The particle gels were frozen and dried in vacuum after swelling for 48 h in water. The dried particle gels were sliced and sprayed with gold, and the microstructure was observed using a scanning electron microscope.

2.2.6. Fourier Transform Infrared Spectroscopy. The FTIR samples were prepared using the KBr pellet method, and the spectra were recorded on a Nicolet iS 10 FTIR spectrometer.

2.2.7. Sand-Pack Flowing Experiment. The sand-pack flowing experiments were conducted to evaluate the plugging capacity of NCPG in this study, and the schematic of these experiments is shown in Figure 2. The experimental steps are as follows:

(1) The sand-pack was filled with the quartz sand of 80–100 meshes. The water flooding was carried out, and the initial permeability of the sand-pack was calculated using Darcy’s formula.

\[ k = \frac{q \mu L}{A (P_1 - P_2)} \]  

where \( k \) is the permeability in mD; \( q \) is the flow rate in mL/min; \( \mu \) is the viscosity of flooding water in mPa·s; \( L \) is the length of sand-pack in cm; \( A \) is the cross-sectional area in cm²; and \( P_1 \) and \( P_2 \) are the pressures of the sand-pack inlet and outlet in MPa, respectively.

(2) A predetermined concentration of NCPG solution was prepared. 0.5 PV NCPG solution was injected into the sand-pack. After that, 0.5 PV water was injected to replace the NCPG solution. The sand-pack was sealed and set at 80 °C for 48 h.

(3) The permeability of the sand-pack was tested by water flooding again. The plugging rate (\( \phi \)) and the residual resistance factor (RRF) of the NCPG were calculated using the following equations.

\[ \phi = \frac{k_0 - k_1}{k_0} \times 100\% \]  

\[ \text{RRF} = \frac{k_0}{k_1} \]  

where \( k_0 \) and \( k_1 \) are the permeability of sand-pack before and after injecting the NCPG solution, respectively.

3. RESULTS AND DISCUSSION

3.1. Fourier Transform Infrared Spectroscopy. The FTIR spectra of NCPG are shown in Figure 3. The FTIR spectra of LPT, characteristic absorption peaks of Si—O stretching and bending bands appeared at 660 cm⁻¹, which are shifted to 625 cm⁻¹ in the spectra of NCPG. This effect is probably due to the interactions of silica groups of LPT with sulfonic groups and carboxylic groups (formed by the hydrolysis of amide groups) that generate electrostatic attraction, as shown in Figure 4, thereby changing the IR absorption of Si—O groups. NH— stretching, C—N stretching, and C==O stretching peaks are observed at around 3419, 1150, and 1630 cm⁻¹, respectively. The absorption peak at 2938 cm⁻¹ is due to the stretching vibration of the C—H bond in saturated —CH₃ and the absorption peak at 1401 cm⁻¹ is the vibration absorption peak of sulfonate —SO₃. These revealed that chemical polymerization occurs between monomers, resulting in the formation of nanocomposites.

3.2. Microstructure of NCPG. After swelling for 48 h in freshwater, the microstructures of the particle gels with or without LPT nanomaterials evaluated by SEM are shown in Figure 5a,b, respectively. It can be observed that the uniform network structures are formed in both particle gels. The network structure after expansion by water absorption is regular shaped and smooth in the gel without LPT, whereas the network structure is random-shaped and corrugated in the NCPG. A similar phenomenon was also observed by Tongwa et al. In addition, the pore size of NCPG after swelling for 48 h in fresh water is distributed in the range of 10—15 μm, which is smaller than that of the gel without LPT (approximately 15—25 μm). This phenomenon facilitates LPT platelets to act as multifunctional cross-linkers with the polymer chains linked on them in the NCPG.

3.3. Thermal Stability of NCPG. TG–DSC analysis of the NCPG after swelling in formation water at 80 °C was conducted to investigate the thermal stability within the reservoir temperature (30–300 °C). The result is shown in Figure 6. From the TG–DSC curves, it can be seen that the TG and DSC curves of NCPG move to the right compared to the two curves of the NCPG without LPT. Only one endothermic peak is observed in the DSC curve of the NCPG and the sample without LPT. The weight loss of the NCPG and the NCPG without LPT is 40 and 54%, respectively, as the temperature increases from 30 to 100 °C. This may be due to the evaporation of free water inside the hydrogel. The mass loss, in the range from 100 to 125 °C, is
attributed to the evaporation of bond water (bonded to the polar hydrophilic groups in the polymer chains) inside the hydrogel. Up to 125 °C, the TG curves of the NCPG and the NCPG without LPT appear to be constant with the total mass loss of 93 and 97%, respectively. It may be concluded that the presence of LPT nanomaterials slightly enhances the thermal stability of NCPG.

3.4. Factors Influencing the Water Absorbency of NCPG. 3.4.1. Effect of the AM to AMPS Ratio on Water Absorbency. Keeping the total amount of monomers constant, NCPG was prepared by varying the ratio of AM and AMPS, and the water absorbency was measured at room temperature (25 °C), 50, and 80 °C, respectively. The experimental results are shown in Figure 7. It can be observed that the water absorbency of the NCPG with the addition of AMPS is significantly higher than that without AMPS. Increasing the AMPS concentration results in a high swelling rate. It is well known that the water absorbency of a hydrogel depends on the electrostatic repulsion of the ionic charges of its network.24,25 The anionic groups (−SO3−) on the NCPG molecular chains increase with the increase of the AMPS concentration. Consequently, an increasing swelling rate is obtained. Furthermore, the swelling rate of the NCPG increases with the increase of the temperature. It may be due to the fact that more amide groups (−CONH2) on the NCPG molecular chains hydrolyze into carboxyl groups (−COO−), and sulfonic acid groups (−SO3H) ionize with increasing temperature, which improves the ionic charges inside the NCPG. Besides, when the ratio of AM and AMPS is 16:4, the NCPG reaches the equilibrium water absorbency at 10 h. As the ratio of AM and AMPS is over 18:2, the NCPG still absorbs water after swelling for 48 h at 80 °C. It allows NCPG to migrate farther in the reservoir with the temperature above 80 °C.

3.4.2. LPT Nanoparticle Effect on the NCPG. LPT nanoparticles were introduced into the polymerization system to prepare the NCPG for improving the strength of PPGs after water absorption. The water absorbency of the NCPG was tested at room temperature (25 °C), 50, and 80 °C. As shown in Figure 8, the swelling rate of NCPG increases with the increase of the LPT concentration and then decreases. The maximum swelling rate is obtained when the LPT concentration is 0.75%. Compared with the NCPG without LPT, the water absorbency of this NCPG after swelling for 48 h is increased by 48% at 80 °C. A nanocomposite-preformed particle gel reinforced by fly ash also exhibited a similar result.26 Based on the bulk gel, the breakdown pressure and stretched length of NCPG after swelling for 48 h were measured to evaluate the effect of LPT on the mechanical properties of the NCPG, respectively. Figure 9 shows the stretched length of the NCPG by adding different concentrations of LPT and their breakdown pressure after absorbing water for 48 h. It can be observed that both the breakdown pressure and stretched length increase with increasing LPT concentration. Compared with the sample that is LPT free, the breakdown pressure of the NCPG added with 0.75% LPT is increased by 146%. In addition, the stretched length is also observed to increase by approximately 120%. The experimental results indicate adding a suitable amount of LPT not only enhances the water absorbency of the NCPG but also improves its mechanical properties. This observation is consistent with the results reported by Haraguchi and Kumar et al.27,28 That is because LPT nanoparticles play a physical cross-linking role in the polymerization system due to the nature of LPT. It is conducive to improving the plugging strength of the NCPG for water shutoff.

3.4.3. MBA Cross-Linker Effect on Water Absorbency. The AM/AMPS ratio and LPT concentration were kept at 18:2 and 0.75%, respectively. The swelling rate of the prepared NCPG was measured at different temperatures. The result is shown in Figure 10. The swelling rate of NCPG first increases and then decreases with increasing MBA concentration. The highest swelling rate is obtained while the MBA concentration is
0.05%. This observation is because a low MBA concentration leads to a low effective cross-linking degree of the NCPG network. Part of the network dissolves due to water absorption. Consequently, the obtained swelling rate is low. Moreover, excessive MBA concentration results in a dense cross-linking network, which reduces the water absorption capacity of the network structure. Therefore, a decrease in the water absorbency of NCPG is observed.

### 3.4.4. APS Initiator Effect on Water Absorbency

Figure 11 shows the swelling rate of the NCPG with different APS concentrations. According to the experimental results, the effect of APS on the swelling rate is similar to that of MBA. When the APS concentration is low, fewer free radicals are produced during polymerization. Some monomers do not participate in the polymerization, which reduces the hydrophilic groups on the molecular chain of the NCPG. Consequently, a low swelling rate is obtained. The swelling rate of the NCPG reduces as the APS concentration exceeds 0.05%. It is because a high APS concentration produces more free radicals, which make the cross-linking reaction easier to occur. Consequently, a higher cross-linking density is obtained, and the swelling rate of NCPG decreases. This result is different from that reported by Kang et al.\(^{29}\) This may be due to the different concentration ranges of the initiator selected in the experiment.
3.5. pH Sensitivity of NCPG. The NCPG was prepared with the formula: 18% AM, 2% AMPS, 0.75% LPT, 0.05% MBA, and 0.05% APS, and the water absorbency of the NCPG was measured at different pH values at different temperatures. As shown in Figure 12, the swelling rate of NCPG in alkaline medium is much higher than that in acidic and neutral media. The swelling rate of NCPG reaches 47.1 times at a pH value of 9 at 80 °C. Compared with the swelling rates of the NCPG at pH values of 5 and 7 at 80 °C, it is increased by 3.99 and 2.51 times, respectively. This observation indicates that NCPG is sensitive to alkalinity and exhibits a higher water absorption capacity in alkaline medium. This phenomenon is because more sulfonic acid groups (SO₃H) in the molecular chains of NCPG are transformed into sulfonate ions (SO₃⁻) under alkaline conditions, which expand the molecular chains of NCPG due to the charge repulsion. Consequently, the water absorbency is improved. In addition, the surface of the LPT lamellar structure is negatively charged due to the presence of silanol groups (Si–O⁻). It creates an effective anionic repulsion, enhancing the water uptake capacity of NCPG. This may be another reason why NCPG is pH sensitive.

3.6. Compatibility with Formation Water. The swelling rate of NCPG was tested in fresh water and formation water at different temperatures, respectively. As shown in Figure 13, the swelling rate of NCPG in formation water is lower than that in fresh water. This is due to the fact that many metal salt ions (such as Na⁺, K⁺, Ca²⁺, etc.) that are present in the formation water penetrate into the network structure of the NCPG and screen the carboxylate and sulfonate groups in the molecular chains of the NCPG. Therefore, the water absorption of the NCPG is impaired. Nevertheless, it can still expand 12.4 times at 80 °C after soaking for 48 h in 81521.2 mg/L formation water.

Figure 14 shows the water absorbency of NCPG in fresh water and formation water for 30 days at 80 °C. The equilibrium absorbency of NCPG is reached after absorbing formation water for 7 days, while it is observed in fresh water after more than 10 days. After that, the swelling rate of NCPG remains constant after placing in fresh water and formation water for 30 days at 80 °C, which are 26.8 and 18.9, respectively. This indicates that NCPG has good salt tolerance and compatibility with the highly saline formation water under reservoir conditions.

3.7. Plugging Capacity of NCPG. The sand-pack, which was filled with quartz sand, was used to simulate the high-permeability zone of the formation. The sand-pack flowing experiment was conducted to evaluate the plugging capacity of NCPG to the high permeability zone in mature reservoirs. As shown in Table 2, the higher the NCPG concentration, the greater the plugging rate. The plugging rate can reach above 90%, while the RRF is 19.2 as 0.3% NCPG solution was injected into the sand-pack. Moreover, the RRF increases with the increase of the NCPG concentration. It suggests that the difficulty of water flow in the sand-pack enhances as the amount of NCPG injected is increased. The experimental results indicate that NCPG has good plugging capacity to the high-permeability zone of the formation.
4. CONCLUSIONS

The NCPG was synthesized from AM, AMPS, and LPT by free radical polymerization and characterized by FTIR spectroscopy, SEM, and TG–DSC. Here, several factors influencing the swelling rate of the NCPG were studied at different temperatures. In addition, the pH sensitivity, compatibility, and plugging capacity were evaluated for water shut off treatment. Based on the experimental results, we can say that LPT nanoparticles play the role of a physical cross-linker in the polymer network. Compared with the swollen NCPG without LPT, the pore sizes of the NCPG were smaller and slightly enhanced the thermal stability. Adding the right amount of LPT nanoparticles into the polymerization system can significantly improve the swelling rate and mechanical properties of the NCPG. The water absorption equilibrium time of the NCPG was 10 h at temperatures below 50 °C, while it still swelled continuously after soaking in water for 48 h at 80 °C. In addition, NCPG exhibited sensitivity to temperatures. In addition, the pH sensitivity, compatibility, and plugging capacity were evaluated for water shut off treatment. Based on the experimental results, we can say that LPT nanoparticles play the role of a physical cross-linker in the polymer network. Compared with the swollen NCPG without LPT, the pore sizes of the NCPG were smaller and slightly enhanced the thermal stability. Adding the right amount of LPT nanoparticles into the polymerization system can significantly improve the swelling rate and mechanical properties of the NCPG. The water absorption equilibrium time of the NCPG was 10 h at temperatures below 50 °C, while it still swelled continuously after soaking in water for 48 h at 80 °C. In addition, NCPG exhibited sensitivity to

![Figure 11. Effect of APS on the swelling rate of NCPG.](image)

![Figure 12. Swelling rate of NCPG at different pH values of water.](image)

![Figure 13. Swelling rate of NCPG in freshwater and formation water at different temperatures.](image)

![Figure 14. Long-term stability of NCPG in fresh water and formation water at 80 °C.](image)

| Table 2. Plugging Efficiency of the NCPG Solution |
|-----------------------------------------------|
| NCPG concentration (%) | permeability (mD) | before injecting NCPG | after injecting NCPG | plugging rate (%) | RRF |
| sand-pack 1 | 0.3 | 1816.5 | 94.6 | 94.8 | 19.2 |
| sand-pack 2 | 0.5 | 2079.1 | 88.3 | 95.7 | 23.5 |
alkalinity. The swelling rate of NCPG reached 47.1 times at a pH value of 9 at 80 °C. The NCPG showed good salt tolerance, and the plugging rate and RRF of 0.3% NCPG solution were 94.8% and 19.2%, respectively. These results indicate that NCPG may be suitable for plugging the high permeability of mature reservoirs.

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Notes

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