Original Paper

Effect of nano TiO_2 and SiO_2 on gelation performance of HPAM/PEI gels for high-temperature reservoir conformance improvement

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A R T I C L E   I N F O

A B S T R A C T

Nanoparticles have been widely used in polymer gel systems in recent years to improve gelation performance under high-temperature reservoir conditions. However, different types of nanoparticles have different effects on their gelation performance, which has been little researched. In this study, the high-temperature gelation performance, chemical structure, and microstructure of polymer gels prepared from two nanomaterials (i.e., nano-SiO_2 and nano-TiO_2) were measured. The conventional HPAM/PEI polymer gel system was employed as the control sample. Results showed that the addition of nano-TiO_2 could significantly enhance the gel strength of HPAM/PEI gel at 80 °C. The gel strength of the enhanced HPAM/PEI gel with 0.1 wt% nano-TiO_2 could reach grade I. The system also had excellent high-temperature stability at 150 °C. The enhanced HPAM/PEI gel with 0.02 wt% nano-TiO_2 reached the maximum gel strength at 150 °C with a storage modulus (G') of 15 Pa, which can meet the need for efficient plugging. However, the nano-SiO_2 enhanced HPAM/PEI polymer gel system showed weaker gel strength than that with nano-TiO_2 at both 80 and 150 °C with G' lower than 5 Pa. Microstructures showed that the nano-TiO_2 enhanced HPAM/PEI gel had denser three-dimensional (3D) mesh structures, which makes the nano-TiO_2 enhanced HPAM/PEI gel more firmly bound to water. The FT-IR results also confirmed that the chemical structure of the nano-TiO_2 enhanced HPAM/PEI gel was more thermally stable than nano-SiO_2 since there was a large amount of $-OH$ groups on the structure surface. Therefore, nano-TiO_2 was more suitable as the reinforcing material for HPAM/PEI gels for high-temperature petroleum reservoir conformance improvement.

1. Introduction

With the rapid development of the oil industry, the exploration and development of high-temperature reservoirs and ultra-high-temperature fields continue to expand (Song et al., 2023; Zhu, 2020). Almost all reservoirs remaining today are non-homogeneous, which allows injection water to escape along high-permeability layers during long-term water-flooding development (Aldhaheri et al., 2022; Seright and Brattekas, 2021; Zhu et al., 2017a). Excessive water production continuously exacerbates the problem of production of excessive water from producing wells, leading to higher-cost wastewater treatment and more serious environmental pollution (Schuman et al., 2022; Zhu et al., 2017c). It

also leaves a large amount of remaining oil in the reservoir (Zhao et al., 2022; Zhu et al., 2019b). Therefore, adjusting the injection profile and increasing sweep efficiency is always vital in improving oil recovery in mature oil fields (Soka and Sidiq, 2022; Zhu et al., 2019a). In recent years, among different reservoir conformance improvement techniques, crosslinked polymer gel systems are considered more widely used to control excess water production and improve wave efficiency (Sun et al., 2016, 2021; Zhu et al., 2017b).

In recent years, HPAM/PEI has been widely used as a water control and plugging agent (Zhu et al., 2019a). It is because HPAM has good water solubility when used as a polymer, and PEI has non-toxicity and excellent environmental characteristics when used as a crosslinker. However, the gel strength and high-temperature stability of PEI crosslinked polymer gel are much weaker than phenolic crosslinked polymer, especially when the reservoir

temperature is higher than 130 °C (Zhu et al., 2017a, 2022). Through extensive experiments, researchers have found that the high-temperature stability of PEI crosslinked polymer gel can be improved by changing the chemical structure or polymer concentration. Deolarte et al. (2009) used a PAtBA/PEI polymer gel system with a copolymer PAtBA concentration of 7 wt%, containing 4.7 mol % of tBA groups and relatively low molecular weight (2.5–5 million). In indoor experiments, the gel system can provide sufficient polymer gel strength to withstand pressure differences above 3600 psi and maintain excellent thermal stability at temperatures up to 191 °C. Vasquez et al. (2006), Eoff et al. (2008), and Al-Muntasheri et al. (2014) found that the gel system has a suitable gelation time of 129 °C. However, the gelation time is still too short for the gelant to transport deep in the porous medium at high temperatures. In addition, the polymer PAtBA has been used at a very high concentration (7 wt%), making its application very costly.

To overcome these problems, researchers have found that the high-temperature stability of PEI crosslinked polymer gel can also be improved by adding inorganic particles, such as powders like cement and SiO_2 (Aqcheli et al., 2020; Lei et al., 2022). Lashari et al. (2018) found that the thermal stability of the system at 60 °C was significantly enhanced after adding nano-SiO_2 to the HPAM/uromodulin/resorcinol gel system. Moreover, no obvious dehydration was observed after aging for 90 d. On the contrary, Pérez-Robles et al. (2019) discovered that the storage modulus G' was essentially unchanged after adding 25–100 mg/L nano-SiO_2 to the HPAM/phenolic resin gel system. Moreover, its thermal stability at 70 °C became significantly worse. Using auxiliary materials, such as inorganic particles, can enhance the gel strength at a lower cost. Jia et al. (2020) found that nano-SiO_2 formed hydrogen bonds with polymers and water molecules, thereby enhancing the strength of free radical polymerization gels. In addition, nano-SiO_2 was also embedded in the microscopic grid of the gel to promote the gelation rate (Li et al., 2022). However, the properties of its high-temperature stability are unclear (Zhu et al., 2017a). Therefore, it is necessary to systematically study the performance of different nanomaterials enhanced PEI polymer gels and analyze their regulation mechanism by molecular structure and microscopic characteristics. By doing so, we can provide a theoretical basis and research guidance for the practical design of its effective action parameters in the pilot test.

In this study, we proposed to develop high-temperature HPAM/PEI crosslinked polymer gels using nano-SiO_2 and nano-TiO_2 and analyzed their performance differences. The differences between nano-SiO_2 and nano-TiO_2 in changing the viscoelastic modulus, viscosity, and gelation performance of HPAM/PEI crosslinked polymer gels at high temperatures (i.e., 80 and 150 °C) were compared using the Sydansk bottle method. Therefore, the gelation performance and the rheological properties of polymer gels could be evaluated. The effects of two nanomaterials on the chemical structures of HPAM/PEI crosslinked polymer gel was compared using infrared spectroscopic analysis. Finally, the influence of different nanoparticles on the polymer gel microstructures was analyzed by scanning electron microscopy. The research results of this study could guide the development of HPAM/PEI crosslinked polymer gel to improve the gelation performance at high temperatures.

2. Experimental materials and methods

2.1. Experimental materials

Na_2CO_3 (analytically pure, ≥98 wt%) was purchased from Shanghai Macklin Biochemical Technology. Polymers (ZP-4), PEI-4 (750,000 daltons, 30% hydrolysis), high-temperature stabilizer

(WDJ-1), and retarder (HNJ-2) were made by Beijing Yuanyang Huangyu Technology Company. Deionized water was provided by the China University of Petroleum (Beijing). Thiourea (≥99 wt%) was purchased from Shanghai Macklin Biochemical Technology. Nano-TiO_2 (XFI02, 20–40 nm diameter, 99 wt% purity) and nano-SiO_2 (XFI03, 20 nm diameter, ≥99 wt% purity) were purchased from Jiangsu Xianfeng Nanotechnology.

2.2. Preparation of nanomaterials enhanced HPAM/PEI gel

First, 0.25 g of high-temperature stabilizer WDJ-1 and 0.05 g of retarder HNJ-2 were dissolved in 500 mL deionized water. Then, different amounts of nano-TiO_2 or nano-SiO_2 particles were weighed and added to the above solutions. The polymer ZP-4 powder was added uniformly at a speed of 400 r/min, stirred for 4 h, and then aged for 24 h. Finally, a certain amount of crosslinker (PEI) was added subsequently and the gelant of nanomaterials-enhanced HPAM/PEI gels were generated after stirring for 10 min using a glass rod. The above polymer gelants were prepared for evaluations of gelation performance, as shown in Fig. 1.

2.3. Determination of the gelation properties of nanomaterials enhanced HPAM/PEI gel

The above polymer gelants were placed in ovens at 80 and 150 °C, and the gel strength and the amount of water dehydrated from the polymer gel system were recorded at regular intervals. The Sydansk bottle test method was used to determine the gel strength of polymer gel in ampoules based on the changes in viscosity, flow states, and surface deformation with aging time (Sydansk, 1988). In each evaluation experiment, we used a freshly prepared sample of the polymer gelant, also called gelant. Table 1 shows the description of different polymer gel strength codes, which classify different gel strengths into nine classes.

2.4. Determination of rheological properties of nanomaterials enhanced HPAM/PEI gel

A rheometer (HAKE MARS III, Thermo, Germany) PP20 plate test system was used to test the rheological properties of the polymer gel system. The experimental device was composed of upper and lower discs with the same axis and the same diameter. During the test, the sample of the polymer gel system was placed between two plates. The base plate was fixed, only the upper plate rotated, and the torque and angular velocity of the rotor were recorded. Since the moisture in the polymer gel evaporated at high temperatures, the test temperature of the measurement system was kept constant at 25 °C (Zhu et al., 2022). All rheological experiments were performed at constant shear (CR) conditions of 7.1 s^{-1}, 0.1 Hz, and 1 Pa.
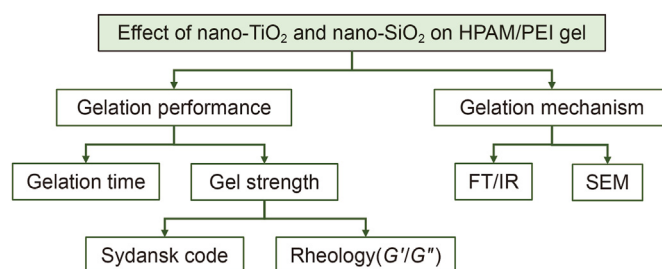

Fig. 1. Flow of the experimental works in this study.

Table 1
Different gel strength code.

Gel strength code	Gel strength description
A	Viscosity is basically the same as the initial configuration solution
B	Slightly more viscous than the initial configuration solution
C	High viscosity, no wall hanging, and almost all polymer gel flows to the mouth of the bottle
D	Weak mobility, only a tiny portion of the polymer gel does not flow down to the mouth of the bottle due to gravity
E	The flowability is weak. Most of the polymer gel hangs on the top of the test tube and the test tube wall, and only a tiny amount of polymer gel flows to the mouth of the bottle
F	High deformation, but polymer gel does not flow to the mouth of the bottle when the tube is flipped
G	Medium deformation does not flow, but the gel can only flow to the middle of the bottle when flipping the ampoule bottle
H	Microdeformation does not flow, but part of the polymer gel surface deforms when the tube is flipped
I	Completely immobile, with no deformation of the polymer gel when the tube is turned

2.5. Infrared spectroscopy analysis of nanomaterial-enhanced HPAM/PEI gels

The high-temperature polymer gel was freeze-vacuum dried in liquid nitrogen. Then, the dried polymer gel samples were ground with potassium bromide powder and pressed into tablets for sample preparation. The infrared spectra of nano-TiO$_2$ and nano-SiO$_2$ enhanced PEI crosslinked polymer gel were tested separately using the infrared spectrometer (Nicolet iS20, Thermo Fisher Scientific Company). The wavenumber ranged from 400 to 4000 cm^{-1}, and the test temperature was room temperature. Through infrared spectroscopy, we tried to analyze whether incorporating nanomaterials reacted with the polymer to produce new functional groups. In this way, we can further analyze the mechanism of the effect of nanomaterials on strengthening the PEI crosslinked polymer gel.

2.6. Microstructural analysis of nanomaterials enhanced HPAM/PEI gel

The high-temperature polymer gel was freeze-vacuum dried in liquid nitrogen after gelation. Then, the samples were fixed on the test bench with conductive adhesive. After evacuation and gold spraying, the microstructure was observed using a scanning electron microscope (Quanta 200 F).

3. Results and discussion

3.1. Gelation of nanomaterials enhanced HPAM/PEI gels at high temperature

3.1.1. The gelation performance of nano-TiO$_2$ enhanced HPAM/PEI gel at 80 °C

To investigate the effect of nano-TiO$_2$ concentration on the gelation performance and high-temperature stability of the enhanced HPAM/PEI gel system, we set the nano-TiO$_2$ concentration to be 0.002 to 0.1 wt%. The polymer concentration of the system was fixed at 0.8 wt%, the crosslinker concentration at 0.3 wt%, the retarder at 0.01 wt%, and the thiourea at 0.05 wt%. The gelation time and the gel strength of the nano-TiO$_2$ enhanced HPAM/PEI was measured at 80 and 150 °C, respectively. Table 2 shows the gel strength variations of the enhanced PEI crosslinked polymer gel system at 80 °C with different nano-TiO$_2$ concentrations.

As can be seen from Table 2, the maximum gel strength of the PEI crosslinked polymer gel system at 80 °C without the addition of nano-TiO$_2$ was level D in 12 d, while the addition of nano-TiO$_2$ could significantly enhance the gel strength, as shown in Fig. 2. When nano-TiO$_2$ particles were added to the PEI crosslinked polymer gel solution, nanoparticles aggregated with the polymer molecules to enhance the strength of the grid skeleton structure of

the polymer gel. In addition, the gel strength of 0.002 wt% nano-TiO$_2$ enhanced HPAM/PEI gel system reached F level after aging for 3 d. When the concentration of nano-TiO$_2$ increased to 0.1 wt%, the gelation time of the system shortened to 1 d. The nanoparticles gathered and formed microscopic changes, thereby increasing the skeleton structure strength and apparent viscosity of the polymer gel solution, which shortened the gelation time of the gelants containing nanoparticles.

It can be seen from Fig. 2 that when the concentration of nano-TiO$_2$ was 0.002−0.02 wt%, the gel strength of the enhanced HPAM/PEI gel system was up to G level in 12 d, 0.1 wt% nano-TiO$_2$ enhanced HPAM/PEI gel was reached I level, and 0.05 wt% nano-TiO$_2$ enhanced gel reached H level in 11 d. Moreover, the 0.05 wt% and 0.1 wt% nano-TiO$_2$ enhanced HPAM/PEI gel system formed a high-strength polymer gel hanging on the top of the test tube, which achieved the strength of plugging for high-temperature reservoirs. In addition, the gelation time of the system can meet the effect of injecting it into the target formation before gelation. In summary, the gel strength of polymer gel increased with the increase of nano-TiO$_2$ concentration, but its gelation time was shortened. Therefore, the appropriate nano-TiO$_2$ concentration should be selected according to the actual reservoir conditions to control a particular gelation time for injection into the formation.

3.1.2. The gelation state of nano-TiO$_2$ enhanced HPAM/PEI gel at 150 °C

To further investigate the effect of ultra-high temperature on the gelation time of nano-TiO$_2$ enhanced HPAM/PEI gel system and the gelation strength, we evaluated the system by the Sydansk bottle test method at 150 °C. The experimental results are shown in Table 3. Table 2 shows that 0.1 wt% nano-TiO$_2$ enhanced HPAM/PEI gel formed at 80 °C for 1 d and 0.002 wt% nano-TiO$_2$ formed for 3 d. However, Table 3 depicts that 0.002 wt% nano-TiO$_2$ formed gel at 4 h, and 0.1 wt% nano-TiO$_2$ formed gel at 2 h. Therefore, the gelation time of the system significantly shortened at an ultra-high temperature of 150 °C, which indicates that nanoparticles accelerated crosslinking and aggregated with polymer molecules at ultra-high temperatures. Moreover, the polymer molecules made a high-speed irregular motion at elevated temperatures, which also accelerated the reaction rate and shortened the gelation time. In addition, the gel strength of the HPAM/PEI gel system without the addition of nano-TiO$_2$ reached the maximum F level in 7 h.

In contrast, the gel strength of the system increased significantly with the addition of nano-TiO$_2$ at 150 °C. Among them, the gel strength of 0.005 wt% nano-TiO$_2$ enhanced HPAMPEI gel reached the maximum level of I after aging for 4 d. Moreover, the gel strength of 0.01 wt% and 0.02 wt% nano-TiO$_2$ enhanced HPAM/PEI gel reached H level, and the gel strength was maintained for more than 30 d with excellent high-temperature stability. Therefore, the system can be injected into the target formation to play a strong

Table 2
Gelation time and gel strength of nano-TiO$_2$ enhanced HPAM/PEI gels at 80 °C

Number	Nano-TiO$_2$ concentration, wt%	Gelation time, d											
		1	2	3	4	5	6	7	8	9	10	11	12
Ti80-0	0	A	A	A	B	B	B	C	C	D	D	D	D
Ti80-1	0.002	D	E	F	G	G	G	G	G	G	G	G	G
Ti80-2	0.005	D	E	F	F	F	G	G	G	G	G	G	G
Ti80-3	0.01	D	F	F	F	F	G	G	G	G	G	G	G
Ti80-4	0.02	D	F	F	F	F	G	G	G	G	G	G	G
Ti80-5	0.05	F	F	F	G	G	G	G	G	G	G	H	H
Ti80-6	0.10	F	F	G	H	I	I	I	I	I	I	I	I

Note: In this study, the moment when the gel strength reaches the F level is defined as the gelation time.
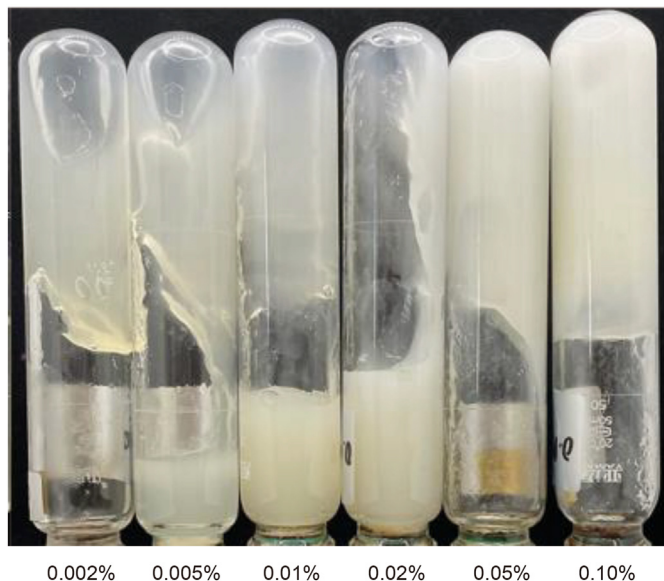
Fig. 2. Gel Strength of nano-TiO$_2$ enhanced HPAM/PEI gels at 80 °C for 12 d (The gels had poor wall-hanging properties, but had high gel strength).

Fig. 3. Gelation state of nano-TiO$_2$ enhanced HPAM/PEI gels at 150 °C for 5 d.
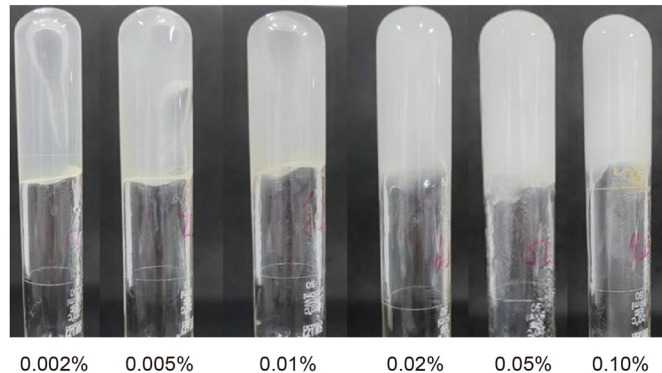
plugging effect. However, it should be noted that the gel strength slightly decreased when the nano-TiO$_2$ concentration exceeded 0.02 wt%, as shown in Fig. 3.

3.1.3. The gelation state of nano-SiO$_2$ enhanced HPAM/PEI gel at 80 °C

To compare the gelation performance and high-temperature stability of nano-SiO$_2$ and nano-TiO$_2$ enhanced PEI crosslinked polymer gel systems, the nano-SiO$_2$ concentrations were also set to 0.002−0.1 wt%. The rest of the concentrations were consistent with the nano-TiO$_2$-enhanced HPAM/PEI gel formulation in Section 3.1.1. The gelation time and gel strength of the nano-SiO$_2$ enhanced HPAM/PEI gel system was measured at 80 and 150 °C. Table 4 gives the variations in the gel strength of the system at 80 °C for different nano-SiO$_2$ concentrations.

It can be seen from Table 4 that different concentrations of nano-SiO$_2$ enhanced PEI crosslinked polymer gel all gelled at 80 °C for 3 d. Moreover, with the increase of nano-SiO$_2$ concentration, the time for the enhanced HPAM/PEI gel system to reach the G level was shortened. When the nano-SiO$_2$ concentration was less than 0.005 wt%, the G-level strength was reached after aging for 9 d. When the nano-SiO$_2$ concentration was higher than 0.01 wt%, reaching the G level was 1 d earlier. In addition, the addition of nano-SiO$_2$ at a high temperature of 80 °C also enhanced the gel strength of the polymer. The maximum gel strength of nano-SiO$_2$ at six different concentrations was G level within 12 d. Among them, 0.1 wt% nano-SiO$_2$ enhanced crosslinked polymer gel had a better wall hanging property and was hung on the top and wall of the high-temperature test tube, as shown in Fig. 4.

Fig. 4 shows the gelation state of PEI crosslinked polymer gel enhanced with different concentrations of nano-SiO$_2$ at 80 °C after aging for 12 d. Compared with Fig. 3, the wall hanging and strength of nano-SiO$_2$ enhanced HPAM/PEI gel were significantly lower than those of nano-TiO$_2$. Although the strength reached the G level, the

Table 3
Gelation time and gel strength of nano-TiO$_2$ enhanced HPAM/PEI gels at 150 °C

Number	Nano-TiO$_2$ concentration, wt%	Gelation time												
		1 h	2 h	3 h	4 h	5 h	6 h	7 h	8 h	1 d	2 d	3 d	4 d	5 d
Ti150-0	0	C	D	D	E	E	E	F	F	F	F	F	F	F
Ti150-1	0.002	D	D	E	F	G	G	G	G	G	G	G	G	G
Ti150-2	0.005	D	E	F	F	G	G	G	G	H	H	H	I	I
Ti150-3	0.01	D	E	F	G	G	G	G	H	H	H	H	H	H
Ti150-4	0.02	E	E	F	G	G	G	H	H	H	H	H	H	H
Ti150-5	0.05	E	F	G	G	G	G	G	G	G	G	G	G	G
Ti150-6	0.10	E	F	G	G	G	G	G	G	G	G	G	G	G

Table 4
Gelation time and gel strength of nano-SiO$_2$ enhanced HPAM/PEI gels at 80 °C

Number	Nano-SiO$_2$ concentration, wt%	Gelation time, d											
		1	2	3	4	5	6	7	8	9	10	11	12
Si80-0	0	A	A	A	B	B	B	C	C	D	D	D	D
Si80-1	0.002	C	E	F	F	F	F	F	F	G	G	G	G
Si80-2	0.005	C	E	F	F	F	F	F	F	G	G	G	G
Si80-3	0.01	C	E	F	F	F	F	F	G	G	G	G	G
Si80-4	0.02	E	E	F	F	F	F	F	G	G	G	G	G
Si80-5	0.05	D	E	F	F	F	F	F	G	G	G	G	G
Si80-6	0.10	D	E	F	F	F	F	F	G	G	G	G	G
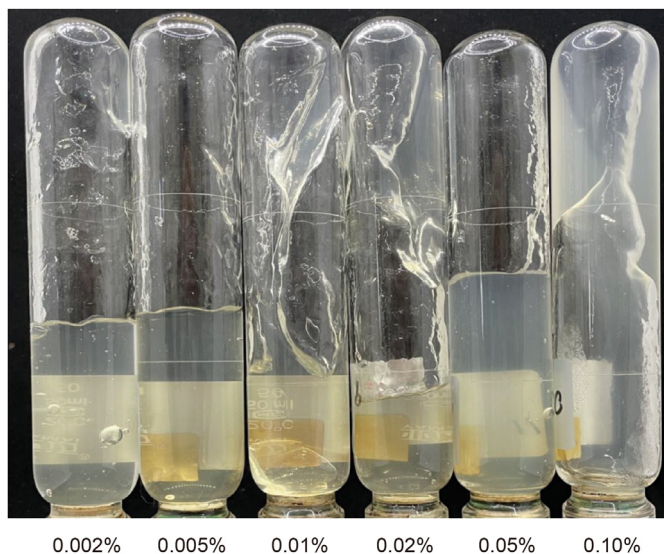
Fig. 4. Gelation state of nano-SiO$_2$ enhanced HPAM/PEI gels at 80 °C for 12 d (The gels had poor wall-hanging properties, but had high gel strength).

Fig. 5. Gelation state of nano-SiO$_2$ enhanced HPAM/PEI gels at 150 °C for 5 d.
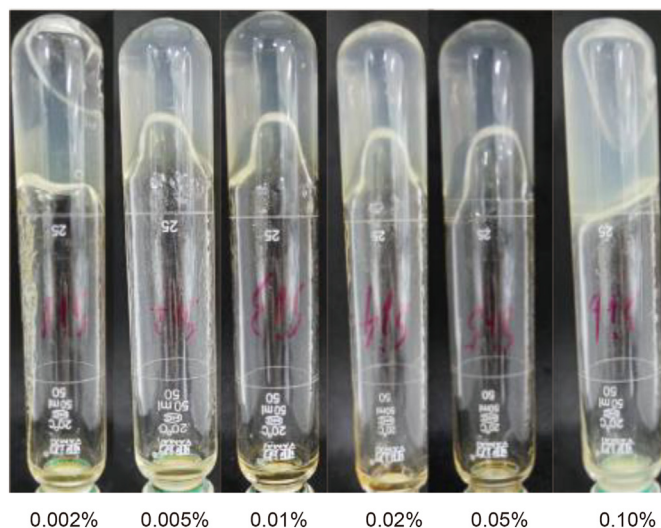
polymer gel fell into the bottle's mouth. The wall hanging was very poor. In addition, 0.01 wt% and 0.02 wt% nano-SiO$_2$ enhanced HPAM/PEI gel parts hung on the top and wall of the high-temperature test tube, but about 80% of the polymer gel fell into the bottle mouth. Therefore, nano-SiO$_2$ had a poor effect on the gel strength of PEI polymer gel compared with nano-TiO$_2$ at 80 °C.

3.1.4. The gelation state of nano-SiO$_2$ enhanced HPAM/PEI gel at 150 °C

To compare the enhancement effect of nano-SiO$_2$ and nano-TiO$_2$ on PEI crosslinked polymer gel at the ultra-high temperature of 150 °C, nano-SiO$_2$ enhanced HPAM/PEI gel systems were also evaluated by Sydansk bottle test at 150 °C. The experimental results are shown in Table 5. All of the nano-SiO$_2$ enhanced HPAM/PEI gels reached the F level in the first hour. Compared with the system

without nano-SiO$_2$, the gelation time of the system was significantly shortened from 7 to 1 h, which remarkably causes the difficulty of injection into the reservoir since it is difficult to inject the system into the target formation in a short period. Therefore, the gelation time of the system needed to be elongated by adding an appropriate amount of retarder; however, it may increase the production cost.

Fig. 5 shows the gel state of PEI crosslinked polymer gel with different concentrations of SiO$_2$ at 150 °C for 5 d. The gel strength of the nano-SiO$_2$ polymer gel system was slightly different, but all the gel strength was G level. The hanging wall of the system was sturdy, with high-strength polymer gel hanging at the top of the high-temperature test tube. It should be noted that the gelation time of the system was very short at 1 h, and the gel strength did not reach the H level, so it was difficult to form a strong plugging in the formation.

The gelation properties of nano-TiO$_2$ and nano-SiO$_2$ enhanced

Table 5
Gelation time and gel strength of nano-SiO$_2$ enhanced HPAM/PEI gel at 150 °C

Number	Nano-SiO$_2$ concentration, wt%	Gelation time												
		1 h	2 h	3 h	4 h	5 h	6 h	7 h	8 h	1 d	2 d	3 d	4 d	5 d
Si150-0	0	C	D	D	E	E	E	F	F	F	F	F	F	F
Si150-1	0.002	F	F	F	F	F	F	F	F	G	G	G	G	G
Si150-2	0.005	F	F	F	F	F	G	G	G	G	G	G	G	G
Si150-3	0.01	F	F	F	F	F	G	G	G	G	G	G	G	G
Si150-4	0.02	F	F	F	F	F	G	G	G	G	G	G	G	G
Si150-5	0.05	F	F	F	F	F	G	G	G	G	G	G	G	G
Si150-6	0.10	F	F	F	F	F	G	G	G	G	G	G	G	G

PEI crosslinked polymer gel were evaluated and analyzed as follows. It can be seen from Tables 2 and 4 that the gel strength of nano-TiO$_2$ enhanced HPAM/PEI gel at a high temperature of 80 °C was more robust than that of nano-SiO$_2$. The mechanism differences in these two different nanomaterials might be related to their physical and chemical interactions with the polymer gel. In Section 3.3, the mechanism was analyzed at the molecular level using instrumental analysis methods such as infrared spectroscopy and scanning electron microscopy.

3.2. Gel strength of different nanomaterials enhanced HPAM/PEI gel

3.2.1. Gel strength of nano-TiO$_2$ enhanced HPAM/PEI gel at high temperature

To further evaluate the effect of nano-TiO$_2$ on the enhanced PEI crosslinked polymer gel strength, we tested the rheological properties of the nano-TiO$_2$ polymer gel system with different concentrations using a rheometer. Fig. 6 shows the viscoelastic change curves of the nano-TiO$_2$ enhanced HPAM/PEI gel system after aging at 80 °C for 5 d.

It can be seen from Fig. 6 that the storage modulus G' of the nano-TiO$_2$ enhanced HPAM/PEI gel system with different concentrations was significantly higher than the loss modulus G'' at low shear rates. In addition, the storage modulus G' of the nano-TiO$_2$ enhanced HPAM/PEI gel system was higher than 5 Pa. The storage modulus G' of 0.02 wt% nano-TiO$_2$ enhanced HPAM/PEI gel reaches a maximum value of about 8 Pa, indicating that the enhanced HPAM/PEI gel exhibits excellent elastic properties, which was consistent with the high gel strength of the system observed macroscopically, as shown in Fig. 3. When the enhanced HPAM/PEI gel system is injected into the target formation, it will play a better plugging effect. However, with the increase of nano-TiO$_2$ concentration, the viscoelasticity of the PEI crosslinked polymer gel system did not change significantly. When the concentration of nano-TiO$_2$ was higher than 0.02 wt%, the storage modulus G' and loss modulus G'' of the system decreased. In summary, higher strength polymer gel could be obtained by changing the concentration of nano-TiO$_2$, but the increase of the concentration of nano-TiO$_2$ decreased the gelation time.

Fig. 7 shows the viscosity changes of the nano-TiO$_2$ enhanced PEI crosslinked polymer gel system after aging at 80 °C for 5 d. The viscosity of the PEI crosslinked polymer gel system did not change significantly with the increase in nano-TiO$_2$ concentration. The viscosity of PEI crosslinked polymer gel enhanced by 0.02 wt% nano-TiO$_2$ reached the maximum value of 1250 mPa·s. When the concentration of nano-TiO$_2$ exceeded 0.02 wt%, the viscosity of the system decreased, and the viscosity of the gel with 0.1 wt% nano-TiO$_2$ reached the minimum value of 830 mPa·s.

Similarly, the rheological properties of the enhanced HPAM/PEI gel system formed by different concentrations of nano-TiO$_2$ at 150 °C were tested using a rheometer. Fig. 8 shows the viscoelastic modulus curves of the nano-TiO$_2$ enhanced HPAM/PEI gel system at an ultra-high temperature of 150 °C for 5 d. The storage modulus G' and loss modulus G'' of the PEI crosslinked polymer gel system first increased and then decreased with the increase of nano-TiO$_2$ concentration. The storage modulus and loss modulus of nano-TiO$_2$ enhanced HPAM/PEI gel system with the concentration of 0.02 wt% were the maximum value, which was 13 and 2.5 Pa respectively. It can meet the need for conventional plugging strength (it is generally believed that the storage modulus over 10 Pa can meet the need for plugging strength). However, when the concentration of nano-TiO$_2$ exceeded 0.02 wt%, the viscoelastic modulus of the enhanced HPAM/PEI gel system decreased slightly, which indicates that the crosslinking between nanoparticles and polymer molecules is not sufficient when the nano-TiO$_2$ concentration exceeds a specific value, thus affecting the gel strength. Therefore, the nano-TiO$_2$ concentration should be controlled below 0.02 wt% to enhance the strength of the polymer gel. In addition, it is worth noting that the storage modulus of the system was stable at 10–13 Pa at the low shear rate, and the loss modulus was stable at 1.2–2.5 Pa at the beginning. Therefore, nano-TiO$_2$ concentration should not exceed 0.02 wt%. Otherwise, the gel strength of the system should reduce, and the expected plugging effect will not be achieved.

Fig. 9 shows the viscosity changes of the nano-TiO$_2$ enhanced PEI crosslinked polymer gel system after aging at 150 °C for 5 d. With the increase of nano-TiO$_2$ concentration, the viscosity of the PEI crosslinked polymer gel system first increased and then decreased. When the concentration of nano-TiO$_2$ was 0.02 wt%, the system's viscosity reached the maximum value of 3000 mPa·s. However, when the concentration of nano-TiO$_2$ exceeded 0.02 wt%, the viscosity decreased slightly. The viscosity of 0.05 wt% and 0.1 wt% nano-TiO$_2$ enhanced HPAM/PEI gel system decreased to about 2000 mPa·s. It was worth noting that the viscosity changed
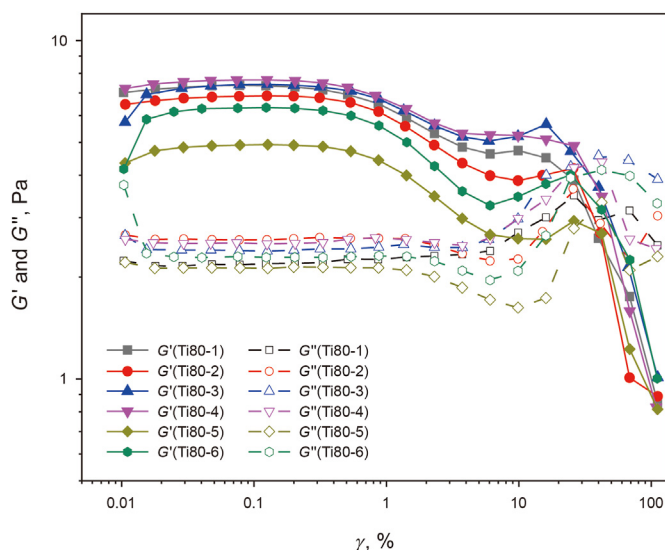
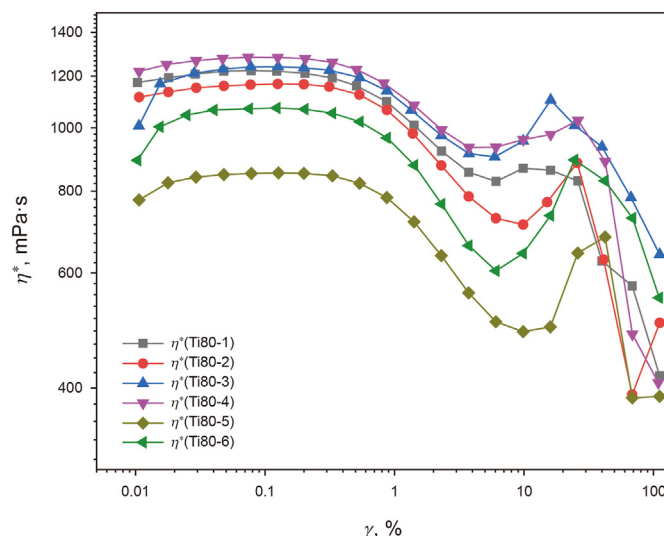**Fig. 6.** Storage modulus G' and loss modulus G'' of nano-TiO$_2$ enhanced HPAM/PEI gels at 80 °C.

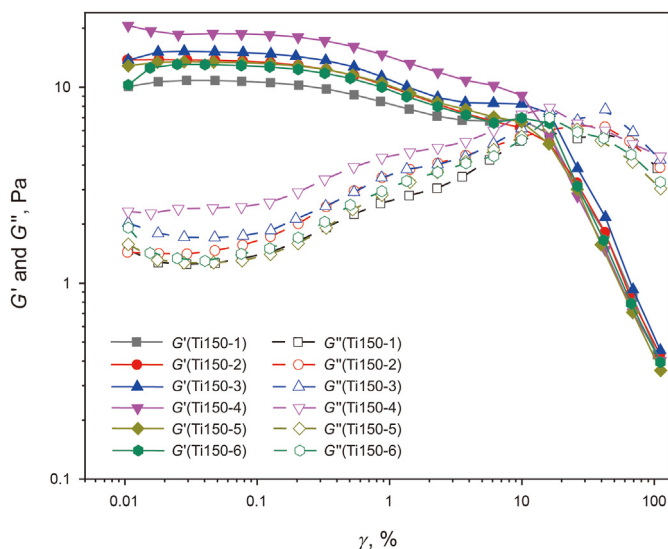**Fig. 7.** Viscosities of nano-TiO$_2$ enhanced HPAM/PEI gels at 80 °C.

Fig. 8. Storage modulus G' and loss modulus G'' of nano-TiO$_2$ enhanced HPAM/PEI gel at 150 °C.

smoothly at a low shear rate. When the shear rate exceeded 20 rad/s, the viscosity decreased rapidly, consistent with the viscoelastic changes in Fig. 4.

Therefore, the effect of nano-TiO$_2$ on the gelation performance of HPAM/PEI gels at the above two different temperatures differed. At a high temperature of 80 °C, the gel strength of polymer gel was enhanced with the increase of nano-TiO$_2$ concentration, but its gelation time was shortened. Moreover, the gelation time of the system was significantly shortened under an ultra-high temperature of 150 °C, and all of them were formed within 4 h. It should be noted that the gel strength of the system was slightly reduced when the nano-TiO$_2$ concentration exceeded 0.02 wt%.

3.2.2. Gel strength of nano-SiO$_2$ enhanced HPAM/PEI gel at high temperature

The rheological properties of the nano-SiO$_2$ polymer gel system with different concentrations were tested using a rheometer. Fig. 10 shows the viscoelastic changes of the PEI crosslinked polymer gel
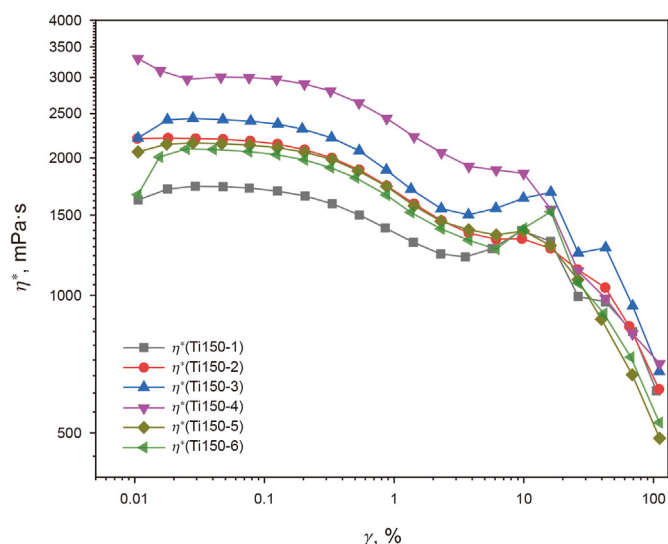
system enhanced with different nano-SiO$_2$ concentrations after aging for 5 d. The storage modulus G' of nano-SiO$_2$ enhanced HPAM/PEI gel at six concentrations maintained at 3.3–4.6 Pa. The storage modulus G' was all lower than 5 Pa, which means the plugging effect of the system in the target formation was not very excellent. In addition, the viscoelastic modulus of the PEI crosslinked polymer gel system did not change significantly with the increase of nano-SiO$_2$ concentration. It decreased slightly when the nano-SiO$_2$ concentration exceeded 0.02 wt%. When the nano-SiO$_2$ concentrations were 0.002 wt% and 0.02 wt%, the storage modulus G' and loss modulus G'' of polymer gel reached the maximum values, which were 4.6 and 2.1 Pa, respectively.

Fig. 11 shows the viscosity changes of the HPAM/PEI gel system with different nano-SiO$_2$ concentrations after being aged at 80 °C for 5 d. It could be seen from the figure that 0.002 wt% nano-SiO$_2$ enhanced HPAM/PEI gel could reach a maximum value of 800 mPa·s. In addition, with the increase of nano-SiO$_2$ concentration, the viscosity of the enhanced PEI crosslinked polymer gel system decreased slightly. The viscosity of nano-SiO$_2$ enhanced PEI crosslinked polymer gel with concentrations of 0.01 wt%, 0.05 wt%, and 0.1 wt% decreased more obviously. Therefore, the concentration of nano-SiO$_2$ should not be too high, otherwise, it was difficult to form a high-viscosity polymer gel to plug the target formation.

Fig. 12 depicts the viscoelastic change curve of the nano-SiO$_2$ enhanced PEI crosslinked polymer gel system after aging at 150 °C for 5 d. The viscoelastic modulus of the HPAM/PEI gels first increased and then decreased with the increase of the concentration of nano-SiO$_2$. The storage and loss modulus of the nano-SiO$_2$ enhanced HPAM/PEI gel system with a concentration of 0.01 wt% reached the maximum, which were 5 and 1 Pa, respectively. In addition, the storage modulus G' of the system was stable at a low shear rate of 3.5–5 Pa, and the loss modulus G'' was stable at 0.2–1 Pa. It could be seen that the nano-SiO$_2$ enhanced system had low gel strength under ultra-high temperature conditions, which means the plugging strength was weak.

Fig. 13 shows the viscosity changes of the nano-SiO$_2$ enhanced polymer gel systems aged at 150 °C for 5 d. The viscosity of the PEI crosslinked polymer gel system gradually increased slightly with the increase of nano-SiO$_2$ concentration, and the viscosity of the nano-SiO$_2$ enhanced PEI crosslinked polymer gel with a concentration of 0.01 wt% reached a maximum of 780 mPa·s. It could be
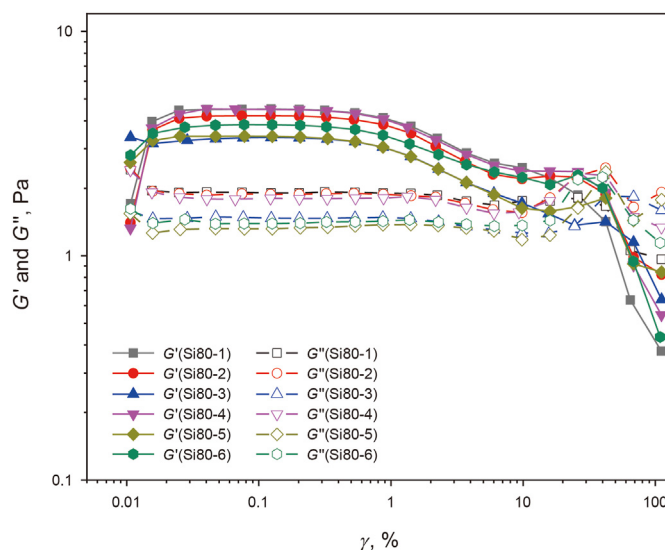

Fig. 9. Viscosities of nano-TiO$_2$ enhanced HPAM/PEI gel at 150 °C.

Fig. 10. Storage modulus G' and loss modulus G'' of nano-SiO$_2$ enhanced HPAM/PEI gel at 80 °C.
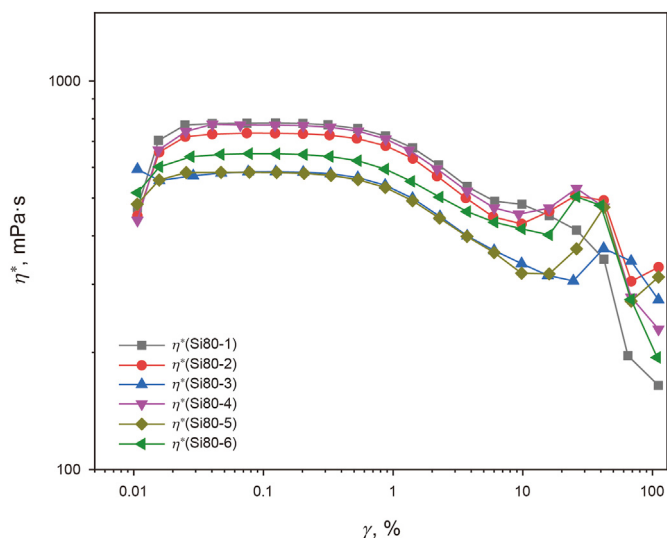
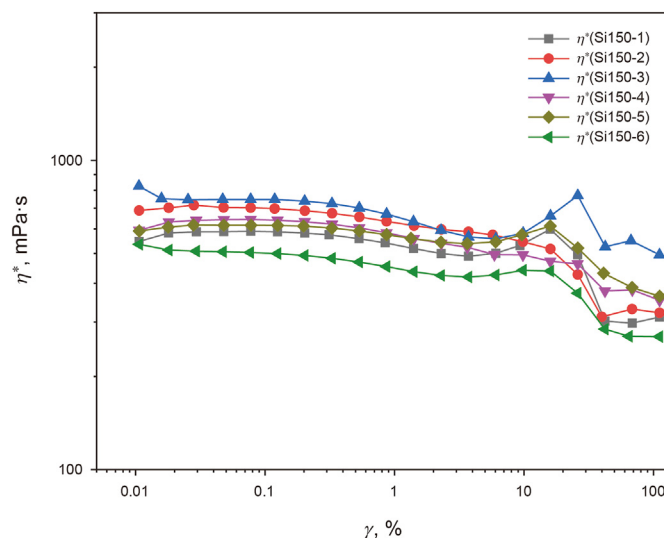**Fig. 11.** Viscosities of nano-SiO$_2$ enhanced HPAM/PEI gel at 80 °C.

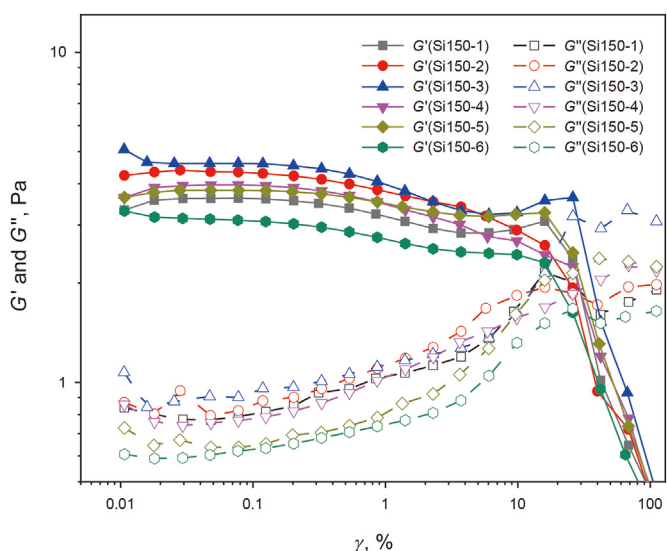**Fig. 13.** Viscosities of nano-SiO$_2$ enhanced HPAM/PEI gel at 150 °C.

Fig. 12. Storage modulus G′ and loss modulus G″ of nano-SiO$_2$ enhanced HPAM/PEI gel at 150 °C.

seen that the viscosity of the system was very low under ultra-high temperature conditions, and it was unable to meet the plugging requirements in ultra-high temperature reservoirs. It is worth noting that when the concentration of nano-SiO$_2$ exceeded 0.01 wt %, the viscosity of the polymer gel system decreased slightly.

Therefore, the effect of nano-SiO$_2$ on the gel strength of HPAM/ PEI gel was not significant at the two different temperatures mentioned above. At a high temperature of 80 °C, the PEI cross-linked polymer gel enhanced with different concentrations of nano-SiO$_2$ cross-linked after aging for 3 d. The gelation time of the enhanced HPAM/PEI gel system to reach the G level was shortened with the increase of the nano-SiO$_2$ concentration. However, the wall-hanging property of the system was inferior, and most of the polymer gel fell off the bottle mouth. Therefore, the nano-SiO$_2$ did not significantly enhance the gel strength of PEI crosslinked polymer gel under ultra-high temperature conditions, and the gelation time was also shortened to 1 h. The mechanism of the effect of nanomaterials on the enhanced HPAM/PEI gel was analyzed later

by IR spectroscopy and SEM.

It could be seen from Figs. 6 and 10 that the storage modulus of polymer gel formed by nano-TiO$_2$ could reach up to 7 Pa at 80 °C. In addition, the storage modulus (G′) of 0.002 wt%, 0.01 wt%, and 0.02 wt% nano-TiO$_2$ enhanced HPAM/PEI gel was the largest. It indicated that the enhanced HPAM/PEI gel of it had excellent elasticity so that the system could play a specific plugging effect after being injected into the target formation. However, it should be noted that when the concentration of nano-TiO$_2$ exceeded 0.02 wt%, the storage modulus of the system would decrease slightly. The storage modulus of nano-SiO$_2$ enhanced HPAM/PEI gels was lower than 5 Pa, so they showed poor plugging ability, and the temperature application range of the reservoir was also limited. Hence, the viscoelasticity of nano-TiO$_2$ enhanced HPAM/PEI gels was stronger than those with nano-SiO$_2$ at 80 °C, which could better meet the plugging requirements in high-temperature formations.

It could be seen from Figs. 7 and 11 that the viscosity of nano-TiO$_2$ enhanced HPAM/PEI gel was higher than that of nano-SiO$_2$. When the concentration of nano-TiO$_2$ was 0.02 wt%, the viscosity of the system could reach the maximum value of 1228 mPa·s, while the maximum viscosity of nano-SiO$_2$ enhanced HPAM/PEI gel was 790 mPa·s. It was worth mentioning that except for 0.05 wt% nano-TiO$_2$ enhanced HPAM/PEI gel, the viscosity of the other nano-TiO$_2$ enhanced HPAM/PEI gel was higher than 1000 mPa·s. It indicates that nano-TiO$_2$ would significantly enhance the viscosity of polymer gel. It might be because the long chains of the polymer would be intertwined, thereby increasing the viscosity of the base fluid, and further enhancing the viscosity of the enhanced HPAM/PEI gel.

Figs. 8 and 12 show that the storage modulus of the nano-TiO$_2$ enhanced HPAM/PEI gel system at different concentrations were higher than 10 Pa at an ultra-high temperature of 150 °C. The storage modulus of the 0.002 wt% nano-TiO$_2$ enhanced HPAM/PEI gel system even reached 15 Pa, and the system showed excellent elasticity. In contrast, nano-SiO$_2$ had little effect on improving the storage modulus of polymer gel at ultra-high temperatures, and the storage modulus of the system was still lower than 5 Pa. In summary, under ultra-high temperature conditions, the viscoelasticity of the nano-TiO$_2$ enhanced HPAM/PEI gel system was better than that of nano-SiO$_2$, which can plug porous media under ultra-high temperature reservoirs, and the gel strength of the system can maintain for a long time. In addition, it can be seen from Table 3 that the increase in nano-TiO$_2$ concentration can shorten the

gelation time of the system. Therefore, it was necessary to reasonably select the concentration of nano-TiO_2 and regulate the gelation time to meet the ground configuration and the time required for safe pumping, thus forming a plugging of porous media.

It could be seen from Figs. 9 and 13 that the rheological properties of nano-TiO_2 and nano-SiO_2 enhanced HPAM/PEI gel were quite different at the ultra-high temperature of 150 °C. The viscosity of the nano-TiO_2 enhanced HPAM/PEI gel system was higher than 1500 mPa·s at different concentrations. Moreover, the viscosity of 0.02 wt% nano-TiO_2 enhanced HPAM/PEI gel system reached the maximum 3000 mPa·s. It could be seen that the nano-TiO_2 enhanced HPAM/PEI gel system could meet the requirements of plugging the target formation under ultra-high temperature reservoir conditions. However, the viscosities of the nano-SiO_2 enhanced HPAM/PEI gels at different concentrations were all lower than 1000 mPa·s, which indicated that the nano-SiO_2 enhanced HPAM/PEI gel system had weak plugging ability under ultra-high temperature conditions.

3.3. Mechanism analysis of the effect of different nanomaterials on the high-temperature gelation properties of polymer gel

3.3.1. Comparison of infrared spectra of different nanomaterials enhanced HPAM/PEI gel

The functional groups of nanomaterial enhanced PEI crosslinked polymer gel system were analyzed by infrared spectroscopy. Fig. 14 shows the comparison of IR spectra of the control group and 0.002 wt% nano-TiO_2 and nano-SiO_2 enhanced HPAM/PEI gel. The spectrum Ti150-0 represented the infrared spectrum of PEI crosslinked polymer gel without nanomaterials, spectrum Ti150-1 represented that with 0.002 wt% nano-TiO_2, and Si represented nano-SiO_2. By comparing infrared spectra, it could be more intuitive to judge whether adding nanomaterials would produce new functional groups in PEI crosslinked polymer gel to analyze its gelation mechanism further. For the control sample, 3314 cm^{-1} was the characteristic peak of O—H inside the PEI structure, and 2860 cm^{-1} was the double characteristic peak of —C—H inside the PEI structure. It could be seen from the infrared spectra of different nanomaterials with a concentration of 0.002 wt% that the infrared characteristic peaks of different nanomaterials were consistent with those of PEI crosslinked polymer gel. That was, the addition of nanomaterials did not produce new characteristic peaks, and no new functional groups appeared. Therefore, the nanomaterial did not react with PEI, which inflects that the nanomaterial enhances the gel strength of the PEI polymer gel system mainly by embedding nanoparticles into the polymer.

To further investigate the effect of nanoparticle concentration on the chemical structure of polymer gel, Fig. 15 compares the infrared spectra of the control sample and 0.01 wt% nano-TiO_2 and nano-SiO_2. In the spectra of the control sample, 3345 cm^{-1} was the characteristic peak of O—H inside the PEI structure, and 2862 and 2830 cm^{-1} were the C—H double characteristic peaks inside the PEI structure. It could be seen from the infrared spectra of different nanomaterials with a concentration of 0.01 wt% that increasing the concentration of nanomaterials would broaden the infrared characteristic peaks of nanomaterials. However, the increase in nanomaterial concentration did not make PEI crosslinked polymer gel produce new characteristic peaks, so no new functional groups were produced. In summary, when the nanomaterial was added less, it would not react with PEI crosslinked polymer gel, mainly by embedding the polymer network structure to enhance the strength of the skeleton structure. In addition, the peak area of the —OH increased significantly when 0.01 wt% TiO_2 was added, mainly because the polarity of the Ti—O bond in nano-TiO_2 was very large (greater than the Si—O bond). Therefore, the water adsorbed on the surface of nano-TiO_2 particles in the gelant dissociates due to polarization, which was easier to form hydroxyl groups. The formation of these hydroxyl groups could further sharpen the strength of the polymer gel system. The HPAM/PEI gels with nano-TiO_2 particles had higher gel strength and better thermal stability at 150 °C.

3.3.2. Comparison of the microstructure of different nanomaterials enhanced HPAM/PEI gel

Fig. 16 shows the scanning electron micrographs (SEM) of the blank control sample and the enhanced HPAM/PEI gels formed by adding different nanomaterials. Fig. 16(a) shows that they exhibited a three-dimensional network structure at the micro level. The mesh size was about 30—50 μm, which could bind water under high-temperature conditions. However, under ultra-high temperature conditions, the thermal vibration of the grid would intensify so that
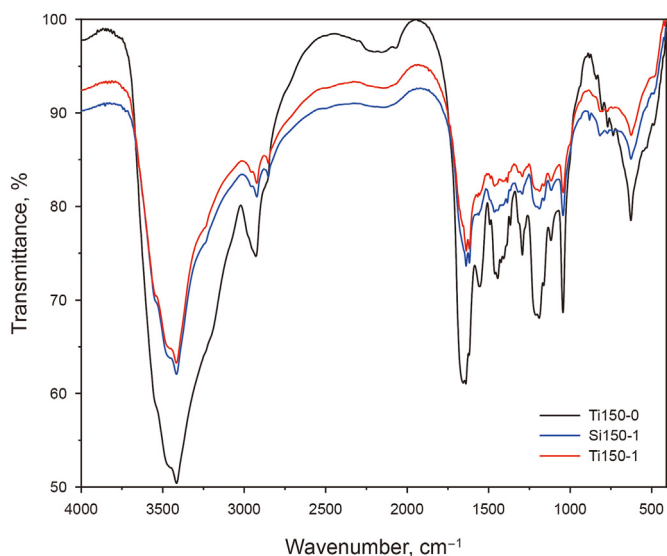

Fig. 14. Comparison of infrared spectra of the control sample and 0.002 wt% different nanomaterials enhanced HPAM/PEI gel.
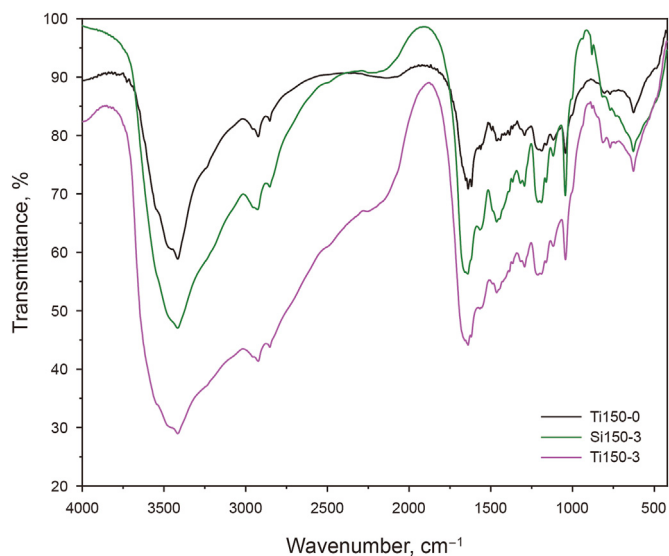

Fig. 15. Comparison of infrared spectra of the control sample and 0.01 wt% different nanomaterials enhanced HPAM/PEI gel.
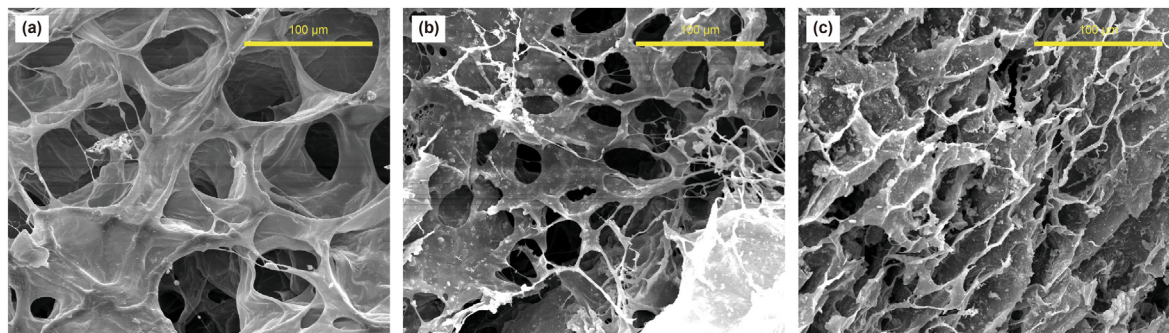
Fig. 16. SEM photograph of the control sample and different nanomaterials enhanced HPAM/PEI gel. (**a**) blank control sample, (**b**) with SiO_2, and (**c**) with TiO_2.

some water molecules would be thrown out. Therefore, PEI cross-linked polymer gel could only be stable for about one month at 150 °C. It could be seen from Fig. 16(b) and (c) that nanoparticles adhered to the surface of the mesh skeleton structure after adding nanoparticles. The Si–O and Ti–O bonds of the nano-SiO_2 and TiO_2 attached to the surface of the grid skeleton had a particular polarity in the gelant, which would cause the water adsorbed on the surface of the particles to be dissociated due to the polarization effect, forming hydroxyl groups. Then, they formed hydrogen bonds with water, which would increase the strength of the nanoparticle-enhanced PEI crosslinked polymer gel system. In addition, the polarization of the Ti–O bond was more substantial than the Si–O bond, so it was more attractive to H_2O. Macroscopically, nano-TiO_2 enhanced HPAM/PEI gels had better thermal stability at 150 °C. In addition, the addition of nanoparticles would also reduce the size of the microgrid to form a denser crosslinked network, making it more capable of binding water under thermal vibration conditions. Macroscopically showed stronger viscoelasticity and thermal stability.

4. Conclusion

(1) The nano-TiO_2 and nano-SiO_2 enhanced HPAM/PEI gel systems could be prepared from nanomaterials as reinforcing agents. Compared with the conventional HPAM/PEI gel system, the nanomaterial-enhanced HPAM/PEI gel system had better gel strength and long-term stability.

(2) The gel strength of HPAM/PEI gels could be significantly enhanced by nano-TiO_2 at a high temperature of 80 °C. The highest gel strength (grade I) could be achieved with 0.1 wt% nano-TiO_2 enhanced HPAM/PEI gel. With the increase of nano-TiO_2 concentration, the gel strength of polymer gel was also enhanced, but its gelation time was shortened.

(3) At a high temperature of 80 °C, the nano-SiO_2 enhanced HPAM/PEI gel formed a three-dimensional network structure. The gelation time shortened with the increase of nano-SiO_2 concentration, but the gel strength was slightly lower than that of nano-TiO_2.

(4) At an ultra-high temperature of 150 °C, nano-TiO_2 enhanced HPAM/PEI system had better high-temperature stability.

(5) Infrared spectroscopy showed that nanomaterial-enhanced HPAM/PEI gel did not produce new functional groups. Nano-TiO_2 enhanced the thermal stability of polymer gel by forming titanium hydroxyl groups on its surface, which made the micro network structure thicker.

(6) The difference in plugging performance of different nano-materials in reservoir cores needs further study.

(7) The cost of nano-TiO_2 is higher than that of SiO_2, and the gel performance strengthening effect of their combination needs to be further analyzed.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

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