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Original Paper

A novel profile modification HPF-Co gel satisfied with fractured low permeability reservoirs in high temperature and high salinity

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ABSTRACT

Conformance control and water plugging are a widely used EOR method in mature oilfields. However, majority of conformance control and water plugging agents are unavoidable dehydrated situation in high-temperature and high-salinity low permeability reservoirs. Consequently, a novel conformance control system HPF-Co gel, based on high-temperature stabilizer (CoCl₂·H₂O, CCH) is developed. The HPF-Co bulk gel has better performances with high temperature (120 °C) and high salinity (1×10⁵ mg/L). According to Sydansk coding system, the gel strength of HPF-Co with CCH is increased to code G. The dehydration rate of HPF-Co gel is 32.0% after aging for 150 d at 120 °C, showing excellent thermal stability. The rheological properties of HPF gel and HPF-Co gel are also studied. The results show that the storage modulus (G') of HPF-Co gel is always greater than that of HPF gel. The effect of CCH on the microstructure of the gel is studied. The results show that the HPF-Co gel with CCH has a denser gel network, and the diameter of the three-dimensional network skeleton is $1.5-3.5 \mu m$. After 90 d of aging, HPF-Co gel still has a good three-dimensional structure. Infrared spectroscopy results show that CCH forms coordination bonds with N and O atoms in the gel amide group, which can suppress the vibration of cross-linked sites and improve the stability at high temperature. Fractured core plugging test determines the optimized polymer gel injection strategy and injection velocity with HPF-Co bulk gel system, plugging rate exceeding 98%. Moreover, the results of subsequent waterflooding recovery can be improved by 17%.

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1. Introduction

Various chemical oil flooding technologies have been applied to improve oil recovery, such as polymer flooding (Hazarika et al., 2022; Pogaku et al., 2018), surfactant flooding (Liu et al., 2022a; Rezaei et al., 2020), foam flooding (Liu et al., 2022c; Xu et al., 2022), nanofluid flooding (Hemmat Esfe et al., 2020; Liang et al., 2022; Qu

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et al., 2022a; Yakasai et al., 2021), nanofluid imbibition (Nowrouzi et al., 2020; Qu et al., 2022b), etc. However, the increase of water cut gradually become one of the most serious problem with the development of oilfields. When the water cut exceeds 80%, the oilfield enters the mature stage, especially in fractured low-permeability reservoir (Dudek et al., 2020; Liu et al., 2020; Zheng et al., 2019). In the initial stage of oilfield development, oil production is very high due to the fact that fractures have remarkable conductivity. However, with the continuous injection of water phase in fractured low-permeability reservoirs, fractures are gradually flooded to form channels, leading to ineffective circulation of injected water between injection-production wells (Lu et al., 2012; Perez et al., 1994; Wang et al., 2015). Hence, chemical water plugging agents have attracted attention to plug fractures and

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expand the sweep efficiency of injected water (Li et al., 2020).

Polymer gel, as a water plugging agent, has been widely used in many mature fractured low-permeability reservoirs because it has excellent abilities on water conformance control in fractures (Aldhaheri et al., 2016; El-Karsani et al., 2014). When polymer gelant solution is injected into the reservoirs for aging a period of time, the three-dimensional network structures are formed in the polymer bulk gel system. The polymer gel can inhibit the water flow in fractures to expand the sweep efficiency (Bai et al., 2015; El-Karsani et al., 2014; Yang et al., 2019). Majority of polymer gels are mainly composed of polymers, crosslinking agents, and other additives. Crosslinking agents can be divided into inorganic crosslinking agents and organic crosslinking agents.

As for inorganic crosslinking system, polyvalent metal ions can form ionic bond or complexation bond with the carboxyl group (-COOH) on the main chains of hydrolyzed polyacrylamide (Jia and Chen, 2018; Karimi et al., 2016; Liu et al., 2022b; Zhang et al., 2015; Zhu et al., 2017a). Sun et al. (2018) discussed the polymer-Cr (III) crosslinking system and investigated its plugging behaviors. After gelation, the pressure gradient during the subsequent water flooding is 8.543 MPa/m at 65 °C, which greatly exceeds the pressure gradient during polymer solution injection (0.011 MPa/m). Karimi et al. (2016) studied the effect of temperature on the dehydration of HPAM-Cr (III) gel. The dehydration rate of HPAM-Cr (III) gel can reach 80% after aging for 10 d at 100 °C. Zhang et al. (2020) developed a polyacrylamide gel whose strength can still remain at the code H (The strength code is shown in Fig. 1) after aging 120 d at 140 °C. In this polyacrylamide gel, both chromium (III) and phenolic resin are regarded as crosslinking agents. However, the salt tolerance of polyacrylamide gel has not been studied and investigated. For organic crosslinking system, the amide group (-CONH₂) on the main chains of the polymer can be crosslinked with the phenolic resin or furfural resin agents by forming covalent bonds (Jia and Chen, 2018; Zhu et al., 2017a, 2018, 2017b, 2017c). Adewunmi et al. (2018) synthesized PAM/PEI-CFA hydrogels with coal fly ash (CFA) by using the crosslinking agent of polyethyleneimine (PEI). It is found that the introduction of CFA can reinforce the elasticity and resistance of thermal decomposition of hydrogels. PAM/PEI-CFA hydrogels have long-term stable plugging effects at 90 °C by forming a dense 3D network structure. Xie et al. (2021) prepared a modified polyacrylamide gel (HMP-Gel) by crosslinking hydrophobically modified polyacrylamide (HMP) with polyethyleneimine (PEI). Compared with the strength retention percentage (38.1%) of HPAM-Gel, the strength retention percentage of HMP-Gel aged at 140 °C for 48 h is 64.3%. The HMP-Gel shows good stability at high temperature because it contains many 2acrylamido-2-methylpropane sulfonic acids (AMPS) side groups and long carbon chains which can protect the amide segment of polymer molecules. Researches demonstrate that organic crosslinked gels have poor plugging performances in hightemperature and high-salinity conditions due to the break of main carbon chains and the hydrolysis of crosslinked bonds and amide groups (Liu et al., 2021, 2022b; Zhu et al., 2017a). Prada et al. (2000) synthesized a terpolymer P (AA/AM/ATBS) to form gel which can be applied in 60-120 °C reservoirs. Zhu et al. (2017c) synthesized an in-situ ternary copolymer gel system. When the concentration of NaCl is 0.05 mol/L (2922 mg/L), the gelation time of the system is 6 h, it can remain stable for 5 months at 150 °C. With the salinity increasing, performance of ternary copolymer gel will deteriorate. Thus it is suitable for salinity conditions below 1×10^5 mg/L. The research on conformance control does not address both high-temperature (\geq 120 °C) and high-salinity (\geq 1 \times 10⁵ mg/L) conditions. In order to implement deep profile control between injection well and production well, gelation time of conformance control agents should be prolonged.

In this work, an organic crosslinking system of polymer gel (HPF-Co gel) is developed. During the experiments, gelation time is evaluated by visual inspection based on gel strength code method. Shear resistance, viscoelastic properties are characterized by using rheometer. The microstructure and morphologies of HPF-Co gel are investigated by using Scanning Electron Microscope (SEM). Analysis of the structural functional groups is also conducted by using Fourier Transform Infrared Spectroscopy (FTIR) for HPF-Co gel. Moreover, optimizations of injection strategy and injection rate of HPF-Co gel are carried out by core plugging experiments. Results show that, HPF-Co gel has low initial viscosity and slow gel formation time, so it has good flow ability and migration ability in the formation. Besides, the addition of CCH to HPF-Co gel improves the thermal stability and environmental adaptability of the gel structure. Therefore, HPF-Co gel can be applied to low permeability fractured reservoirs with high temperature of 120 °C and high salinity of 1×10^5 mg/L.

2. Experimental part

2.1. Materials and instruments

Anionic low molecular weight polyacrylamide AN 934 BPM (China SNF Co., Ltd.), furfural crosslinking agent (brown-red solution, 1:1 molar ratio of phenol to furfural, sodium hydroxide, ethanol), gel stabilizer (thiourea, urea), gel accelerator (benzoic

Table 1 Formation water ion ratio, $mg \cdot L^{-1}$.

$Na^+ + K^+$	Ca^{2+}	${\rm Mg}^{2+}$	Cl-	SO_4^{2-}	$HCO_{\overline{3}}$	CO_{3}^{2-}	Total salinity
44580.2	925.9	210.5	51657.9	38.8	2404.5	18.6	99836.4



Fig. 1. Sydansk gel-strength code, A: No detectable gel formed, B: Highly flowing gel, C: Flowing gel, D: Moderately flowing gel, E: Barely flowing gel, F: Highly deformable nonflowing gel, G: Moderately deformable nonflowing gel, H: Slightly deformable nonflowing gel, and I: Rigid gel (Liu et al., 2022b).

acid, resorcinol, oxalic acid), high-temperature stabilizer CCH (cobalt chloride hexahydrate) and deionized water are used in this work. The ion ratio of formation water is provided by Shengli Oilfield, as shown in Table 1.

The experimental instruments include mechanical stirrer (IKA EUROSTAR 60 control), advection pump (2 PB series, Shenzhou Weico, China), oven, intermediate container, precision pressure sensor module, several pipelines, and other instruments. The main characterized experimental instruments are rheometer (HAAKE RS600, Germany), brookfield viscometer (DV2T, America), scanning electron microscope (Gemini SEM 300, ZEISS Company, Germany), fourier transform infrared spectrometer (Brookfield TENSOR II, Germany).

2.2. Experimental method

2.2.1. Preparation of gel

The 0.5 wt% polymer is slowly added to a beaker with 100 mL deionized water and stirred continuously for 1 h to ensure complete dissolution. Then, 0.3 wt% furfural crosslinking agent, 0.1 wt% stabilizer, 0.3 wt% accelerator and a desired amount of high-temperature stabilizer are introduced into the beaker and stirred continuously for 10 min until the crosslinking agent and other additives are completely dissolved.

In order to determine the optimal concentration of the hightemperature stabilizer (CCH), HPF-Co gelant solutions with different concentrations of CCH (0, 0.02, 0.04, 0.06, 0.08, 0.10, 0.12 wt%) are prepared and poured into glass pressure-resistant bottles. The sealed glass pressure-resistant bottles are placed in a constant temperature oven at 120 °C for aging, and the gelation time is recorded. The optimal concentration of high-temperature stabilizer (CCH) is determined by summarizing the dehydration percentage and evaluating the long-term thermal stability of different gels. Meanwhile, the strength of HPF-Co bulk gel system is evaluated through Sydansk inverted bottle test method according to Fig. 1. Gelation time is defined as the time interval required between the start of aging of the gel solution and the stabilization of the gel strength.

When the gelant solutions with different CCH concentrations are completely gelation, the levels of syneresis of HFP-Co gel after aging for 5, 15, 30, 60, 90 and 150 d at 120 °C are recorded, respectively. By recording the volume of drainage liquid (V_t) at different aging times, the dehydration percentage (S) at different aging times is calculated, as shown in Eq. (1).

$$S = \frac{V_t}{V} \times 100\% \tag{1}$$

2.2.2. Characterization of HPF-Co gel

(1) The shearing tests are performed on various gelant solutions after all agents are completely dissolved. The shearing rate is 300 rpm and the shearing time lasts 1 h. Then viscosity of HPF gel and HPF-Co gel are measured by HAAKE RS600 rheometer at a constant temperature of 60 °C, shearing rate is 7.34 s⁻¹, shear frequency is 0.1 Hz and shear stress is 1 Pa. The viscosity retention percentage (Φ) of gelant is calculated by the viscosity (η) before and after shearing, as shown in Eq. (2). Besides, the gelation time is monitored and recorded

after HPF-Co bulk gel system is completely gelated and the viscosity of the gel is measured.

$$\Phi = \frac{\eta_1}{\eta_0} \times 100\% \tag{2}$$

- (2) Viscoelasticity can reflect the elastic solid property and viscous liquid property of gel systems (Seright, 2001; Seright et al., 2003). The elastic performance of gel is characterized by the storage modulus G' while the viscous performance is characterized by the loss modulus G". Storage modulus represents the properties of solids, which refers to the elastic (reversible) deformation capacity of materials. Loss modulus represents liquid properties and refers to material viscosity. The storage modulus (G') and loss modulus (G'') are measured using the lamina system of the HAAKE RS600 rheometer with fixed shear stress (the scan frequency varies from 0.1 to 10 Hz) under the constant shear (CR) mode. Since the measuring system is not closed and the water in the gel is extremely volatile under high temperature environment, the test temperature of rheology measurement in this paper is constant at 60 °C.
- (3) The microstructure of HPF-Co gel is characterized by SEM. After the optimal concentration of the high-temperature stabilizer (CCH) is determined, micrographs of HPF bulk gel system and HPF-Co bulk gel system are taken respectively to observe the microstructures of gels. Briefly, gel samples are frozen with liquid nitrogen, then the gel samples are completely dehydrated by using a freeze-dryer. Finally, bulk gel samples are sprayed with gold for observations.
- (4) FT-IR is used to characterize the structures and chemical compositions of different HPF-Co gels with various CCH concentrations. The experiment steps are as follows:

The freeze-dried sample of HPF-Co gel are fully ground in agate grinding bowl. Then, KBr and HPF-Co gel at a ratio of 100:1 are mixed by using potassium bromide tablet method. Put the mixture in a special mold and sample can be prepared when the pressure is 10 MPa for 1 min. Bruker TENSOR II FT-IR is continuously used to detection, the spectra range from 8000 to 350 cm⁻¹, the spectral resolution is 0.4 cm⁻¹, the wavenumber accuracy is better than 0.005 cm⁻¹, the absorption accuracy is better than 0.07%T.

2.2.3. Fractured core plugging

To manufacture AFC (artificial fractured core), the homogeneous low permeability cuboid cores are split through the middle, and the copper sheets are filled to support the fractures. AFC is composed of 200 mesh fine sand. The sizes of AFC are 30 cm in length, 4.5 cm in height and 4.5 cm in width. The water permeability of matrix is approximately $20 \times 10^{-3} \ \mu m^2$ and the aperture is 0.15 mm. The schematic of fractured core plugging experiments is shown in Fig. 2. The experimental steps of HPF-Co gel on water conformance control are as follows:

- Saturate the fractured core with formation water after vacuuming 6 h, calculate the porosity based on saturated water volume;
- (2) Saturate the fractured core with silicone oil for wettability alteration, and then the core is aged for 7 d at 120 °C;



Fig. 2. Schematic of core flooding setup.

- (3) Saturate the fractured core with crude oil and age the core for 7 d;
- (4) Carry out the first waterflooding until the water cut reaches 98%;
- (5) After the HPF-Co gelant solution injection, few drops of water is filled to displace HPF-Co solution from pipeline to AFC completely. Then, put the core holder into the oven at 120 °C for aging 3 d at least;
- (6) Subsequent waterflooding is conducted until the water cut reaches 98%;
- (7) During the flooding experiments, the oil recovery for each injection stage and the changes in pressure are monitored and recorded over time;
- (8) The confining pressure of the above steps is maintained at 10 MPa.

3. Results and discussions

3.1. The properties of HPF-Co gel

3.1.1. Screening of high-temperature stabilizer CCH concentration

Compared with HPF-Co gels without CCH (the gel system without CCH addition is denoted by HPF in all the subsequent manuscript), the different concentrations of CCH are added to HPF-Co gels to improve thermal stability. And then, the properties of aging dehydration period and gel strength of the gel system are analyzed. The concentrations of CCH are 0.00, 0.02, 0.04, 0.06, 0.08, 0.10, and 0.12 wt%, respectively. The strength level of HPF-Co gel with various CCH concentrations is shown in Table 2. Results indicate that HPF after galated is level F based on Sydansk standard code. HPF-Co is bigger than level F which means CCH can increase gel strength of HPF-Co gel significantly with the same test conditions. Therefore, in order to improve the strength of HPF-Co to obtain better plugging effect, CCH is indispensable.

Subsequently, gel strength code of HPF-Co gel with concentrations from 0.02 to 0.08 wt% approaches level F-G. When the mass fraction of CCH in the HPF-Co gel exceeds 0.1 wt%, the gel strength code of HPF-Co gel approaches level G.

Table 2

The gel strength and gelation time of gels with different concentrations of CCH.

Concentration, wt%	Gel strength	Gelation time, h
0.00	F	22
0.02	F-G	24
0.04	F-G	24
0.06	F-G	24
0.08	F-G	24
0.10	G	24
0.12	G	24



Fig. 3. Dynamic dehydration percentages of gels with different concentrations of CCH.

The relationship between dehydration percentages of gels and aging time are shown in Fig. 3. With the increase of aging time, the dehydration percentage of gels with and without CCH increases. The dehydration percentage of HPF gel is 100% after the gel is aged for 150 d. However, the dehydration percentages are 73%, 59%, 54%, 42% after aging 150 d when the 0.02, 0.04, 0.06 and 0.08 wt% CCH are added into HPF-Co gels. When the concentration of CCH exceeds 0.10 wt%, the dehydration percentages of HPF-Co gel are 13% after aging 90 d and 32% after aging 150 d. In addition, the dehydration percentages are 0.10 and 0.12 wt% respectively. Therefore, it can be concluded that 0.10 wt% of CCH is the optimal concentration for stabilizing HPF-Co gel. In the following description, gel without CCH addition is called HPF gel.

3.1.2. Rheology of the gel

(1) Viscosity retention

It can be seen from Table 3, viscosities of both HPF gelant solution and HPF-Co gelant solution are decreased after shearing. The viscosity retention percentage of HPF-Co gel after shearing is 90.4%, while the viscosity retention percentage of HPF gel after shearing is 71.2%. Hence, compared with HPF gel system, HPF-Co gel system possesses higher viscosity retention percentage. Subsequently,

Table	3
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Effect of shearing	on viscosity	v of HPF gel	and HPF-Co gel.

8	, ,	8				
Category	Viscosity of system before gelation, mPa·s		Viscosity retention percentage, %	Gelation time after shearing, h	Viscosity after gelation, mPa·s	
	Before shearing	After shearing				
HPF gel HPF-Co gel	211 161.1	150.2 145.7	71.2 90.4	30 28	3264 16,440	

both of HPF gel and HPF-Co gelant solutions are placed in an oven at 120 °C for gelation. Results show that the gelation time of HPF gel and HPF-Co gel after shearing is delayed 8–10 h. Due to the competing effects of breaking aggregate structures and accelerating crosslinking reaction at high shear rates, gelation time can be prolonged (Yu et al., 2021). The viscosities of HPF gel and HPF-Co gel after gelation are 3264 and 16,440 mPa ·s respectively. Therefore, compared with HPF gel system, HPF-Co gel system possesses better shear resistance and better plugging potential. The experiment indicates that CCH can improve the shear resistance of gels.

(2) Viscoelasticity

In order to analyze the effect of CCH on the viscoelasticity of gels, scanning frequency range is setting from 0.01 to 10 Hz, as shown in Figs. 4 and 5. In this section, Fig. 4 shows the viscoelastic results of HPF gel and HPF-Co gel before gelation. According to Fig. 4, the storage modulus G' and loss modulus G'' of HPF gelant solution and HPF-Co gelant solution increase with the increase of shear frequency. Meanwhile, the storage modulus G' is higher than loss modulus G'', indicating that both of HPF gel and HPF-Co gel exhibit elastic properties.

Fig. 5 shows the viscoelastic results of HPF gel and HPF-Co gel after gelation. When the frequency is less than 1 Hz, the storage modulus (G') and loss modulus (G'') of HPF and HPF-Co bulk gel systems do not change significantly with the increase of frequency. When the frequency is greater than 1 Hz, the storage modulus of HPF bulk gel system and HPF-Co bulk gel system obviously increases. Especially, the viscoelasticity of HPF-Co bulk gel system increases more. Experimental results indicate that the high-temperature stabilizer (CCH) is helpful to improve the elastic properties and reduce the viscous properties of bulk gel system.







Fig. 5. Viscoelasticity of HPF gel and HPF-Co gel after gelation.

Meanwhile, the storage modulus (G') of HPF-Co gel is always greater than the loss modulus (G'') under different shear frequencies. Therefore, the HPF-Co gel exhibits better elasticity and has certain deformability and restorability. In addition, the core breakthrough pressure experiments of the HPF gel and HPF-Co gel show that the breakthrough pressure gradient of HPF-Co gel is 41.17 MPa/m, which was higher than that of HPF gel, as shown in Table 4. Therefore, HPF-Co gel has strong impact resistance.

3.1.3. Microstructure of gel

The microstructures of hydrogels determine their macroscopic properties (i.e., stability, strength, etc.) (Bonhome-Espinosa et al., 2017). Fig. 6 shows the SEM images of HPF gel and HPF-Co gel. Both of HPF and HPF-Co gels possess three-dimensional network structures. The 3D structure of HPF bulk gel is densely covered with meshes. The network skeletons of HPF bulk gel are slender relatively and most of them are connected by filaments. The mesh size of HPF bulk gel ranges from 2 to 5.3 µm. The network structure of HPF bulk gel is easy to be broken at 120 °C, which can reduce the strength of HPF bulk gel and cause poor thermal stability. However, the 3D network skeleton of HPF-Co bulk gel system is bulkier at the same conditions and the mesh size decreases to 1.5-3.5 µm. Fig. 7 shows the SEM images of HPF gel and HPF-Co gel after aging 90 d. The microstructure of the HPF gel after 90 d of aging shows a large number of pores and the skeleton diameter of the threedimensional structure of the gel becomes very small and almost disconnected, as shown in Fig. 7(a). The microstructure of the HPF-Co gel after 90 d of aging shows that the diameter of the pores in its three-dimensional structure becomes larger, reaching 9.4 µm, but the skeleton of the three-dimensional structure still exists, as shown in Fig. 7(b). It shows that the larger the gel skeleton diameter, the smaller the pore diameter in the three-dimensional

Table 4

The breakthrough pressure gradient of HPF and HPF-Co gel in homogeneous core (Water permeability is 20 mD).

Category	Breakthrough pressure, MPa	Breakthrough pressure gradient, MPa/m
HPF gel	7.12	23.73
HPF-Co gel	12.35	41.17



Fig. 6. SEM micrographs of gel samples: (a) HPF gel, (b) HPF-Co gel.



Fig. 7. SEM micrographs of gel samples after aging 90 d: (a) HPF gel, (b) HPF-Co gel.



Fig. 8. EDS results: (a) HPF gel, (b) HPF-Co gel.

structure, and the more stable the gel at high temperature. Therefore, it can be concluded that the structural stability of HPF-Co bulk gel system is stronger and its thermal stability is better at 120 °C.

Fig. 8 shows the EDS results of HPF and HPF-Co bulk gel systems. It can be seen that no cobalt element is found in the HPF gel spectrum image while the cobalt element and more chlorine element are found in the HPF-Co gel spectrum image. The results indicate that $CoCl_2 \cdot 6H_2O$ is involved in the reaction.

3.1.4. Fourier transform infrared spectroscopy results

In the infrared spectrum, when the chromophore is affected by other parts of the molecule connected to it, the absorption peak position will move. If the absorption peak moves to the low frequency direction, it is called 'red shift', and when it moves to the high frequency direction, it is called 'blue shift'. When Co forms coordination bonds with N and O atoms in the amide group, the absorption peaks of C–N and C=O bonds in the amide group move to low frequency, resulting in red shift phenomenon (Jin et al., 2016; Kondratenko et al., 2020).

Fig. 9 shows that the FT-IR spectra curves of HPF gel and HPF-Co gel. It can be seen that the positions of absorption peaks are basically similar, except for the marked absorption peak. A relatively wide characteristic absorption peak ranging $3400-3200 \text{ cm}^{-1}$ is shown in HPF gel spectrum curve, which represents the compounded peak ascribed to multimolecular association absorption peak of hydroxyl group (–OH) and the secondary amine group adsorption peak. The characteristic absorption peaks at 1566 cm⁻¹ of curve (a) and 1559 cm⁻¹ of curve (b) are the amide II band absorption peaks of HPF gel and HPF-Co gel, respectively. Compared



Fig. 9. The infrared spectra of bulk gel systems: (a) HPF gel, (b) HPF-Co gel.

with HPF gel, the absorption peak of amide II band in HPF-Co gel moves to low frequency, which is called red shift (Karpfen, 2019; Wood et al., 2021). In addition, the absorption peak at 523 cm^{-1} of curve (b) represents the coordination bond formed by cobalt atom and nitrogen atom. The coordination between atom Co and atom N restricts the stretching vibration of v (N–H) bond, resulting in the absorption peak of N-H bond moving to the left. This is also the reason why curve (b) has a wider wave peak in the band of $3500-3000 \text{ cm}^{-1}$. Moreover, in the band of $3500-3000 \text{ cm}^{-1}$, the multimolecular association absorption peak of hydroxyl (-OH) and the adsorption peak of secondary amine have the same coincidence peak. Moreover, both the absorption peak of 1669 cm^{-1} in curve (a) and the absorption peak of 1665 cm^{-1} in curve (b) are C=0 bond stretching vibration absorption peaks in amide groups. The wavenumber of absorption peak in curve (b) is smaller than that in the curve (a), indicating that Co is coordinated with the O of amide groups. This coordination can restrict the stretching vibration of v (C=O) bond, resulting in the absorption peak moving to low frequency.

According to the above analysis, the mainly gelation reaction of the HPF-Co bulk gel system occurring between polyacrylamide and furfural crosslinking agent is proposed, as shown in Fig. 10. $CoCl_2 \cdot 6H_2O$ can coordinate with the amide group in the bulk gel system, so that the nitrogen and oxygen atoms in the amide group of the HPF-Co gel are connected by ion Co^{2+} to form the coordination bond, as shown in Fig. 11. The presence of coordination bonds can enhance the strength and stability of gel by constraining the vibration of C==O, N-H and C-N bonds, reducing the vibration of chemical bonds at high temperature and inhibiting the hydrolysis of amide group. However, the content of Ca^{2+} and Mg^{2+} in formation water is high, and excessive Ca^{2+} and Mg^{2+} will destroy the crosslinking point between polymer and crosslinking agent,



Fig. 11. Structure of the reaction product between gel molecules and CCH.

 Table 5

 Different polymer gel injection strategies of HPF-Co bulk gel system in fractured core.

No.	Polymer gel injection strategies
1	0.3 PV primary slug (gelant solution)
2	0.2 PV primary slug (gelant solution)
3	0.1 PV front slug (polymer solution) +0.2 PV primary slug (gelant solution)
4	0.05 PV front slug (polymer solution) +0.15 PV primary slug (gelant solution)

resulting in the decrease of polyacrylamide gel performance. The coordination reaction between CCH and gel molecules increases the stability of the charge distribution of N and O atoms at the crosslinking points, thereby increasing the resistance of HPF-Co gel to Ca^{2+} and Mg^{2+} .

The polymer gel at high temperature is prone to polymer main chain fracture and crosslinking bond fracture, resulting in gel dehydration degradation. The problem of polymer backbone breakage can be solved by grafting branched chains containing heat-resistant functional groups. CCH can inhibit the thermal vibration of the cross-linking bond by forming a coordination bond with the N and O atoms of the amide group, thereby inhibiting the fracture of the cross-linking bond.

3.2. Plugging performance of HPF-Co gel in fractured core

3.2.1. Fractured core plugging effect

(1) Influence of polymer gel injection strategies on plugging effect



Fig. 10. Reaction formula of polyacrylamide and furfural crosslinking agent.

Table 6

Plugging rates	under	different	polymer	gel	injection	strategies.
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No.	Pore volume, mL	Porosity, %	Aperture, mm	Water permeability of matrix, mD	Pressure difference, kPa		Plugging rate, %
					Before plugging	After plugging	
1	111.3	18.3	0.15	24.1	1.70	169.94	98.93
2	99.7	16.4	0.15	20.0	2.13	140.24	98.48
3	104.7	17.2	0.15	21.7	2.04	173.17	98.82
4	95.4	15.7	0.15	19.2	2.26	131.95	98.29

Fractures are regarded as flowing channels of water in fractured core plugging test. The initial injection rate is 0.5 mL/min. Different injection strategies of HPF-Co bulk gel system are shown in Table 5. The plugging rate (*PR*) is calculated by recording the pressure difference (Δp) at both ends of the core before and after plugging, as shown in Eq. (3).

$$PR = \frac{\Delta p_1 - \Delta p_0}{\Delta p_1} \times 100\%$$
(3)

As shown in Tables 5 and 6, the total injection volume of No.1 and No.3 is more than No.2 and No.4, and the pressure difference is higher after plugging, the average pressure difference of No.1 and No.3 is 171.56 kPa. No.2 and No.4 with a total injection of 0.2 PV have a lower pressure difference, the average pressure difference of Nos.2 and No.4 is 136.10 kPa. The greater of the pressure difference indicates that more gelant solution is injected into the matrix pores during the injection process. Consequently, No.2 and No.4 have low damage for matrix pores since the plugging rate is similar. Besides, No.4 uses 0.05 PV polymer solution as the front slug, which can reduce the dosage of crosslinking agent, stabilizer, and other auxiliary agents when the plugging rate of these combinations are similar. At the same time, the front slug can displace water and reduce the dilution effect of water on the primary slug. Considering

the plugging effect and dosage of agent comprehensively, No.4 is chosen. Pressure difference data diagrams under different polymer gel injection strategies are shown in Fig. 12.

(2) Influence of injection rates on plugging effect

In order to confirm the optimal injection rate of No.4 in Table 5, the injection rates are set as 0.1, 0.3 and 0.5 mL/min, respectively. The corresponding parameter values are shown in Table 7 and Fig. 13. Although the injection rates are different, the plugging rates of HPF-Co bulk gel system is basically the same. However, the higher injection rates contribute to a larger pressure difference. Therefore, the optimal injection rate is 0.1 mL/min.

3.2.2. Water-controlling and oil-increasing effect of HPF-Co gel

The experiments are conducted at the temperature of 120 °C. Reference values of injection parameter can be found in section 3.2.1 (injection rate: 0.1 mL/min, polymer gel injection strategy: 0.05 PV polymer solution+0.15 PV HPF-Co gelant solution). Corresponding parameter values of fractured core are shown in Table 8.

When the injection volume of premiere water is 0.6 PV, the water cut at the outlet can exceed more than 98% and oil recovery reach 14.2% at that time. After 0.05 PV polymer solution and 0.15 PV HPF-Co gelant solution are injected, the core is aged for 3 d until



Fig. 12. Pressure difference of different polymer gel injection strategies (The injection rate is 0.5 mL/min. Aging time is 3 d): (a) 0.3 PV HPF-Co gel, (b) 0.2 PV HPF-Co gel, (c) 0.1 PV polymer solution+0.2 PV HPF-Co gel, (d) 0.05 PV polymer solution+0.15 PV HPF-Co gel.

Table 7

Results of plugging fractured cores with different	injection rates.
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No.	Injection rate, mL/min	Pore volume, mL	Porosity, %	Aperture, mm	Water permeability of matrix, mD	Pressure difference, kPa		Plugging rate, %
						Before blocking	After blocking	
1	0.1	138.6	22.8	0.15	21	1.09	62.14	98.25
2	0.3	148.3	24.4	0.15	23	1.53	70.32	97.82
3	0.5	142.3	23.4	0.15	19	2.04	82.41	97.52



Fig. 13. Pressure difference of different injection rates (aging time is 3 d).

HPF-Co system gelation completely. Subsequent water flooding continues until the outlet has no oil appearance, then oil recovery increases an additional to 17.4%. As illustrated in Fig. 14, the ultimate oil recovery rate is 31.6%, which indicates that suitable polymer gel injection strategies and injection rate of HPF-Co bulk gel system has outstanding potential to be applied in low-permeability fractured reservoirs.

Experimental results indicate that the optimized injection strategy of HPF-Co bulk gel system is 0.15 PV primary slug followed by 0.05 PV front slug, which has a better plugging performance in low-permeability fractured core. The injection strategies of HPF-Co bulk gel system can plug the fractures and prevent injection water from flowing into the fracture effectively, which has a good plugging effect. Subsequent water flooding can spread into more areas in the low permeability rock matrix after HPF-Co gel plugging, improving sweep volume of water and promoting the increase of ultimate recovery significantly.

4. Summary and conclusion

(1) HPF-Co bulk gel system is synthesized by 0.5 wt% HPAM, 0.3 wt% furfural crosslinking agent, 0.1 wt% stabilizer, 0.3 wt% gel accelerator and 0.1 wt% high-temperature stabilizer cobalt chloride hexahydrate (CCH). It can be applied in high temperature (120 °C) and high salinity (1×10^5 mg/L) conditions.



Fig. 14. The curve of water control and oil enhancement effect of the first group of fractured cores (aging time is 3 d).

- (2) Due to the addition of high-temperature stabilizer CCH in HPF-Co gel with 0.1 wt%, complexation reaction increases the intermolecular forces of the gel. As a result, HPF-Co bulk gel system spends 24 h to gel completely, gel strength achieves level G. The dehydration percentage decreases from 100% (HPF gel) to 40% (HPF-Co gel) after aging for 150 d.
- (3) HPF-Co gel displaces the elastic fluid characteristics and has certain deformability and restorability according to rheology test. SEM images show the average mesh size is 1.5–3.5 μm in 3D network skeleton of the HPF-Co gel. FT-IR indicates the presence of coordination bonds can bind the vibration of C= O, N-H and C-N bonds to a certain extent and reduce the vibration of chemical bonds at high temperature, inhibit the hydrolysis of amide group so that enhancing the strength and stability of gel.
- (4) The conclusion of dynamic experiments in fractured core indicates that optimized injection strategy of HPF-Co bulk gel system is 0.15 PV primary slug followed by 0.05 PV front slug, injection velocity is 0.1 mL/min. According to these parameters, the average plugging rate with HPF-Co bulk gel system exceeds 98%. Meanwhile, when HPF-Co system gelated completely in fractured core with oil wet, subsequent water flooding can improve 17.4% oil recovery after premiere water flooding. Optimal polymer gel injection strategy and injection rate of HPF-Co bulk gel system possesses great potential to be used in low-permeability fractured reservoirs.

Table	8
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Parameters of	fractured	cores.
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Aperture, mm	Water permeability of matrix, mD	Pore volume, mL	Porosity, %	Saturated oil volume, mL	Oil saturation, %
0.15	19	111.2	18.3	68.1	61.2

(5) The prolonged gelation time indicates that it allows enough time for the gel to enter the target layer before gelation. Therefore, the HPF-Co gel has the potential for deep migration distance in-situ.

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.

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Nomenclature

- *S* dehydration percentage, %
- *V* initial volume of the gel after gelation, mL
- *V*_t volume of drainage liquid after gel aging, mL
- Φ viscosity retention percentage, %
- η_0 initial viscosity of the gelant solution, mPa · s
- η_1 viscosity of the gelant solution after shearing, mPa s
- *PR* plugging rate, %
- Δp_0 pressure difference at both ends of core before plugging, kPa
- Δp_1 pressure difference at both ends of core after plugging, kPa

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