Laboratory experiment study on weakly connected crack sealing system

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Abstract. According to the difference of matrix displacement pressure and crack re-tension pressure in fractured reservoirs, it is difficult to develop reservoirs. The gel prepared by using water-soluble phenolic resin as cross-linking agent seals weakly connected cracks. In this paper, the gelation properties, injectability and plugging properties of the gel were studied. The results show that the gel formula was 0.5w% polymer + 0.5w% water-soluble phenolic resin + 0.2w% catalyst NH4Cl, the gelation time was 48h, the elastic modulus after gelation reached 16 Pa, and the long-term stability was good without obvious dehydration. The gelation forming liquid had a viscosity of less than 200 mPa·s in the oil pipe, and the highest flow resistance was 3.77×10^-4 MPa/m; in the reservoir, the pressure gradients of slit widths of 1.0 mm and 0.5 mm (0.1 mL/min) were 0.014 MPa/m and 0.107 MPa/m, respectively. The results of the physical model test show that the breakthrough pressure ladders of the gel in the cracks of different slit widths (1mm, 0.5mm and 0.3mm) were 4.152MPa/m, 4.872MPa/m and 7.160MPa/m, respectively. The 0.5mm slit width could be stabilized for 24h at 1MPa and 2MPa, and the gel had a certain sealing effect after the breakthrough.

1.Introduction

In the fracture-density reservoir, the matrix displacement pressure gradient is large and the crack re-tension pressure is low. Therefore, it is necessary to develop a high-strength plugging agent to seal the crack. Domestic and foreign scholars have extended the application range of gel by studying the traditional HPAM gel formula, and realized the application of gel in various severe reservoir environments such as conventional formation conditions, high temperature and high salt reservoirs[1-8].

In recent years, a large number of mineral applications have been reported for sealing large pores and cracks[9-12]. Wertz Field, Big Horn Basin, Norman Wells, Rangely Weber, Arbuckle, Sacroc and ALASKA oil fields[13-19] used organic chromium gel to seal the cracks and solved problems such as gas and water. R.Seright used pipelines to evaluate the performance of the gel in the cracks, which was consistent with the matrix cracks, and then studied the flow law, fluid loss and stability of the organic chromium gel in the cracks[20-24]. Phenolic resin gel has great difference in cross-linking mechanism with organic chromium gel and high strength[25,26]. The injection performance and plugging performance are quite different from organic chromium gel. There are few studies on blocking cracks. Sun Limei, Qian Xiaolin et al. [27,28] analyzed the synthesis and physicochemical properties of water-soluble phenolic resin. Li Gang, Shi Yun et al.[29,30] studied the gelling factors of water-soluble phenolic resin gel, but the injection performance and plugging performance of water-soluble phenolic resin gel were not evaluated in series. In this paper, water-soluble phenolic resin gel was used as crack plugging agent. The strength of the gel was observed regularly, and the elastic modulus of the gel was measured. The fluid state in the crack was simulated by pipelines with different inner diameters. In the flow viscosity and resistance,
the plugging performance was evaluated by breaking the pressure gradient and the voltage regulation time, so as to study the injection and sealing mechanism of phenolic gel in the crack.

2. Experiment

2.1 Experimental materials

Partially hydrolyzed polyacrylamide, hydrolysis degree 12~14%, molecular weight 14~15 million, Anhui Jucheng Fine Chemical Company; NH₄Cl, analytical grade, Sinopharm Chemical Reagent Company, used as catalyst; water-soluble phenolic resin, solid content 30%, homemade, used as a crosslinking agent.

METTLER AL204 balance, METTLER TOLEDO International Company; HH single hole water bath, Jintan Guosheng Experimental Instrument Factory, Jintan City, Jiangsu Province; electric mixer JJ-1, Jincheng Guosheng Experiment, Jintan City, Jiangsu Province Instrument Factory; Lei magnetic PHS-25 pH meter, Shanghai Yidian Scientific Instrument Company; NDJ-8S rheometer, Anto Paar; thermostat box, intermediate container tank and other displacement devices, Jiangsu Hai’an Petroleum Instrument Company; 2PB00C series Advection pump, Beijing satellite manufacturing plant.

2.2 Experimental method

According to Sydansk's gel strength code standard[31], there were many methods, such as visual observation of gelation time, strength and long-term stability of the gel. The viscosity change rule of the gel (7.34 s⁻¹) and the final gel strength were determined under conditions of shear stress 0.1Pa and frequency 0.2 Hz with Anto Paar rheometer PP25 rotor.

The gelation liquid had shear thinning characteristics in the pipeline migration, and the shear stress and viscosity of the gelation liquid at different shear rates were determined, which had shear thinning characteristics. According to the power law formula equation (1), the consistency coefficient $K$ (Pa·s) and the power law index $n$ of the gelation are calculated by fitting the curve. According to the different injection speed and the inner diameter of the tubing, combined with the Rabinowich-Mooney formula[28], the shear rate calculation of the power law fluid is shown in Equation (2)[32], and the shear rate of the gel forming solution during the injection process is calculated, and then the rheometer measures the viscosity of the gum at different shear rates by using the calculation results.

$$\tau = K \gamma^n \quad (1)$$

$$\gamma = (3n+1) Q / (n \pi r^3) \quad (2)$$

Where: $\gamma$ is the power law fluid shear rate, s⁻¹; $Q$ is the fluid flow rate, m³/s; $r$ is the radius inside the pipe, m.

When the gelation was flowing in the oil pipe, the Reynolds number was up to 100,000, which was turbulent flow. According to the relationship between the friction coefficient of the power law fluid derived by Dodge and Metzer, Equation (4) [33], using Newton iteration method to find the friction The resistance coefficient $f$ is then calculated using the turbulence formula, Equation 3 [34]. According to different pipe diameters and flow rates, the injection pressure under each injection condition is calculated and expressed by the injection pressure gradient (MPa/m) (regardless of gravity).

$$P_f = f \rho v^2 / (2d) , Re>2000 \quad (3)$$

$$1 / (f)^n = 4 \log[f^{(1-0.5n)} Re] / n^{0.75}, 0.395 / n^{1.2} \quad (4)$$

When the gelation was transported in the reservoir (70 ℃), the injection pressure at different flow rates in different inner diameter lines was recorded.

The gelation was injected into a 0.5 m long pipeline of different inner diameters (1 mm, 0.5 mm, and 0.3 mm), sealed, and placed in a 70 ℃ incubator for 10 d. When the gel was driven in the pipeline under 0.1ml/min, the linear velocity in 1mm was 183.35m/d, the simulated reservoir waterline advancing speed was similar, recorded the displacement pressure change with time, and the first droplet appears at the outlet end. The pressure of time was to break through the pressure gradient. After stopping the pump for 120 minutes, it continued to drive for 60 minutes. After stopping the pump for 120 minutes again, it was driven for 60 minutes to observe the sealing performance of the gel after water swelling. Based on the breakthrough pressure gradient, the pressure is selected for 24 hours to evaluate the performance of the gel.
3. Results and discussion

3.1 Gelation performance evaluation

The phenolic system used in the experiment was 0.5 w% + 0.5 w% water-soluble phenolic resin + 0.2 w% catalyst NH₄Cl, and the crosslinking mechanism is shown in Figure 1. The gelation was placed in a 70°C water bath, sampled every 2 hours, and the viscosity of the gelation was measured with time using the rheometer, which is shown in Figure 2 to determine the gelation time of the system. The viscoelastic modulus is shown in Figure 3.

![Figure 1. Water-soluble phenolic resin gel cross-linking reaction.](image1)

![Figure 2. Water-soluble phenolic resin gelation curve](image2)

As can be seen from Figure 2, the gelation process is roughly divided into three stages.[35-36]: (1) Slow induction phase, 0 h ~ 24 h, on the one hand, the amide group in the polymer and the hydroxymethyl group in the phenolic resin were cross-linked, and the intramolecular cross-linking did not form a network structure, which increased the viscosity to some extent; NH₄Cl is continuously hydrolyzed, the gelatinous solution was weakly acidic, the polymer molecules were distorted, and the viscosity was reduced. Therefore, the viscosity of the gelling solution fluctuated significantly at this stage. The previous NH₄Cl hydrolysis released a large amount of H⁺, which was mainly reduced in viscosity and later in acidity. Under the catalytic conditions, the crosslinking reaction was mainly carried out, and the viscosity was slightly increased; in general, the viscosity of the gel-forming liquid was stable at 250 mPa·s, and the strength of the gel was B. (2) In the rapid growth phase, 24 h ~ 36 h, the cross-linking reaction was mainly intermolecular cross-linking. Due to the presence of a large number of benzene rings in the phenolic resin, a dense spatial network structure could be formed, the viscosity of the system rapidly increased to 3088 mPa·s. The strength of the gelation gradually changed from C to E, at which time the gelation became fluid. (3) In the perfect stage of gelation, 36 h ~ 48 h, under the conditions of high temperature and acidic catalysis, the uncrosslinked amide group and methylol group in the system were gradually reduced, the intramolecular and intermolecular
crosslinks were basically completed, and the viscosity of the gelling solution rises slowly to 5338 mPa·s, indicating that the system has been gelatinized.

The gelation time of the system was 48h, which indicates that the gelation could be fully transported to the reservoir crack before gelation, which could meet the requirements of deep flood control.

![Gel modulus](image1)

**Figure 3. Gel modulus (0.2Hz)**

Figure 3 shows the elastic modulus of the gel that was aged at 70 °C for 10 d. It can be seen from Figure 3 that the elastic modulus and storage of the water-soluble phenolic resin gel system in the low shear stress range (0.01 Pa ~ 10 Pa) remained basically unchanged, the linear viscoelastic region was relatively wide, and intramolecular and intermolecular crosslinks between the phenolic resin and the polymer form a dense and stable spatial network structure with certain shear resistance. When the shear stress was more than 30Pa, the elastic modulus of the gel decreased and the storage modulus increased, indicating that the structure of the gel is shear damaged.

![Gel modulus](image2)

**Figure 4. Gel modulus (0.2Hz)**

Figure 4 shows the visual state of the water-soluble phenolic resin gel at different aging times. As can be seen from Figure 4, the gel still maintained high strength with no prolonged dehydration over time. The gel maintained stability in the formation and met the requirements of subsequent profile control and water shutoff.

### 3.2 Injection performance evaluation

There were two stages during the gelation injection process: the flow in the oil pipe and the migration in the reservoir fracture. Firstly, the viscosity and flow resistance of the gelation forming liquid in the oil pipe were calculated, and the displacement pressure gradient in the crack was measured to evaluate the injection property of the gelation.

#### 3.2.1 Injection performance of gelation into the tubing

The viscosity and shear stress of the gelation at different shear rates were determined, as shown in Table 4.

| Shear rate / s⁻¹ | Shear stress / Pa | Viscosity / (mPa·s) |
|------------------|------------------|---------------------|
| 3                | 1.6585           | 552.82              |
According to Equation 1, the curve was fitted and $K = 1.0192$, $n = 0.41$ was calculated. Assume that the injection rate of the in-situ gelation is $2 \sim 4 \text{ m}^3/\text{h}$, and the inner diameter of the simulated tubing was 62 mm and 76 mm.

According to Equation 2 and Equation 3, combined with different pipe diameters and flow rates, the injection pressure under each injection condition was calculated and expressed by the injection pressure gradient (MPa/m) (regardless of gravity, gravity was the power during the injection process), and the calculation result is in Table 2.

### Table 2. Injection pressure gradient under different injection conditions ($\times 10^{-4}$ MPa/m).

| Flow rate / (m$^3$/h) | 2   | 3   | 4   |
|------------------------|-----|-----|-----|
| Pipe diameter / mm     |     |     |     |
| 61                     | 2.83| 3.35| 3.77|
| 76                     | 1.74| 2.05| 2.31|

Taking the distance from the tank to the reservoir in the field construction, taking 1km as an example, the above flow resistance was 0.377MPa, indicating that the injection performance was good.

3.2.2 Injection performance of gelation into the reservoir
The gelation was injected into the 0.5 mm and 1.0 mm inner diameter lines at a flow rate of 0.1, 0.3, 0.5, 1.0, 2.0, 3.0, 5.0, 8.0, and 10.0 mL/min to simulate the migration of the gelation into the crack. After the pressure was stabilized at a flow rate, the pressure change during the injection process was recorded.

![Figure 6. Water-soluble phenolic resin into a gelation flow resistance](image)

It can be seen from Figure 6 that the displacement pressure increased with the increase of the flow rate. Compared with the slit width of 1.0mm, the migration resistance of the gelation in the 0.5mm in the slit was more obvious with the flow rate change. After the flow rate was greater than 1.0 ml/min, the pressure rose linearly. When the slit width was 1.0 mm, 0.3 ml/min, the corresponding linear velocity was 550.05 m/d, and the pressure gradient was 0.02 MPa/m; When the slit width was 0.5 mm and the injection flow rate was 0.1 ml/min, the corresponding linear velocity was 733.39 m/d and the pressure gradient was 0.045 MPa/m. The oilfield test results show that the waterline advancing speed was up to 473 m/d, and the highest injection pressure gradient was 0.045 MPa/m, indicating that it had better migration ability in the crack before gelation.

### 3.3 Plugging performance evaluation

#### 3.3.1 Gel breakthrough breakthrough pressure gradient

Sections 2.1 and 2.2 indicate that the system had high gel strength and good injection properties, and then its plugging performance needs to be evaluated.

![Figure 7. Gel breakthrough pressure gradient](image)

(Inner diameter 1mm, length 0.5m, flow rate 0.1mL/min)
Figure 8. Gel breakthrough pressure gradient 
(Inner diameter 0.5mm, length 0.5m, flow rate 0.1mL/min)

Figure 9. Gel breakthrough pressure gradient 
(inner diameter 0.3mm, length 0.5m, flow rate 0.1mL/min)

For the aged 10d gel, the pressure changes were recorded during the displacement and pump stop of different pipe diameters (1mm, 0.5mm and 0.3mm), as shown in Figure 7~9. The pipe diameter, the first peak, the second peak, the subsequent water drive stability value and the curve integral of the above three figures are plotted into a table, as shown in Table 6. The integral of the curve is the integral of the abscissa (injection time) and the ordinate (displacement pressure), and the integral formula is shown in Equation 5.

\[ \text{Curve integral} = \int P(t) \, dt \quad (5) \]

| Pipe diameter / mm | 1   | 0.5 | 0.3 |
|--------------------|-----|-----|-----|
| First peak / MPa   | 2.076 | 2.436 | 3.824 |
| Second peak / MPa  | 0.904 | 2.264 | 2.6 |
| Subsequent water drive stability / MPa | 0.14 | 1.992 | 1.75 |
| Curve integral      | 16.396 | 91.754 | 175.543 |

In Table 3, the first peak indicates the first breakthrough of water-flooding gel, and the breakthrough pressure gradient of the gel can be calculated, which was 4.154 MPa/m (1 mm), 4.872 MPa/m (0.5 mm), and 7.16 MPa/m (0.3mm) with high strength plugging performance. After the pump was stopped for 120 minutes, the water flooding will continue to show a second peak, indicating
that the gel was swollen and swelled when the pump was stopped. The hole formed by the previous water flooding healed, the water drove again, and the gel was formed at the outlet end, forming a breakthrough again. Subsequent water flooding, the stable pressure in the pipe diameter of 0.5mm reached 1.992MPa, indicating that the gel had good erosion resistance. The curve integral reflects the sealing performance and erosion resistance of the gel in the crack. The integral was 16.396 in the 1mm crack, the pressure gradient was 4.154MPa/m, and the erosion resistance was in the later stage.

3.3.2 Gel pressure regulation performance

The gel was formed a gel in the reservoir. In the subsequent water flooding, the gel needs to bear the displacement pressure without breakthrough, thereby achieving the purpose of sealing the crack. Therefore, with a 0.5 mm inner diameter line, the simulation slit width was 0.5 mm crack. According to Figure 10, the breakthrough pressure gradient in the 0.5 mm crack was 4.872 MPa/m and the oil phase starting pressure in the reservoir was 1 MPa/m, and the displacement pressure was selected to be 1 MPa and 2 MPa for voltage regulation. Observe whether there was a droplet at the outlet end, that was, whether the gel was broken.

4.Conclusions

(1) The gelation process of water-soluble phenolic resin gel was divided into three stages, slow induction stage (0 h ~ 24 h), rapid growth stage (24 h ~ 36 h) and stable stage (36 h ~ 48 h). The gelation time is 48h. It had high strength after gel formation, elastic modulus 16Pa, good long-term stability and no obvious dehydration.

(2) The injection process of the gelation formation was simulated by calculating the shear rate under different tube flow conditions and determining the viscosity of the corresponding gelation. The results showed that the viscosity of the gelation was controlled at 200 mPa·s under various injection conditions. The highest flow resistance in the tubing was $3.77 \times 10^{-4}$ MPa/m; the pressure gradients of the reservoir crack width of 1.0 mm and 0.5 mm (0.1 mL/min) were 0.014 MPa/m and 0.107 MPa/m, respectively.

(3) Through the physical model experiment, the sealing, breakthrough and re-blocking process of the gel in the crack were simulated, and the plugging effect under different conditions was quantitatively evaluated. The results showed that the measured gel was 1 mm in different slit widths. The breakthrough pressure ladders in the 0.5mm and 0.3mm cracks were 4.152MPa/m, 4.872MPa/m
and 7.160MPa/m, respectively. The gel was stabilized at 1MPa and 2MPa in the 0.5mm slit width. At 24h, the gel still had a certain sealing effect after the breakthrough, indicating that the gel had good sealing performance and long-lasting erosion resistance.

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