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# The Development of the New High Temperature Resistance Profile Control Agent Which is Compound With Inorganic Particles and Gel

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## Abstract
At present, high temperature profile control technology has become the key technology to improving recovery efficiency, management of steam channeling in thermal recovery. The particle of regular application is dosage big, poor injection and easy to cause the rigid block, Gel and foam profile control agent is poor stability, low intensity and short validity period. For the above problems, Developed a new type of high temperature resistance particle-gel complex profile control system through the theoretical analysis and the ratio optimization of indoor experiment and evaluate its performance. The formula of the system: 0.03%Coagulant + 2.2% cross-linking agent I + 1.8%cross-linking agent II + 6% modified high temperature resistance main agent + 0.7% new type inorganic particles + 0.5% suspending agent. At least 280 ℃ of heat-resistant, two-fluid process injection, injection performance is good, the plugging rate is more than 99.05%, scouring resistance and it has good thermal stability. This study provides a new direction for the thermal profile and theoretical basis for profile control construction.

**Key words:** Thermal recovery; Steam channeling; Compound; Profile control agent; Two-fluid process

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## INTRODUCTION
High temperature profile control medium at home and abroad are mainly for the high temperature resistant foam, gel, inorganic particles. Under the condition of meeting the requirements of heat resistance, foam profile control agent is poor stability, the validity of gel profile control agent is short, the particle profile control agent is easy to cause the rigid block and poor injection, and then damage the formation[1,2]. The profile control agent is inefficiency when used alone. This project adopts the modification technology to improve the heat-resistant ability of the traditional high temperature resistant main agent, with coagulant and two types of cross-linking agent to form a space mesh quadripolymer gel and with new type of high temperature resistance inorganic particle. The temperature resistance and erosion resistance of the profile control agent are further improved. Gels and the particle which is fell off has the effect of deep migration, it is an ideal profile control agent. This study provides reference for other high-temperature profile control technology research and has a guiding significance for site construction applications.

## 1.THE MECHANISM OF THE CONPLEX SYSTEM
The system is compound with gel and particles, using the modified high temperature resistant main agent. The main agent with coagulant and two types of cross-linking agent makes a quadripolymer reaction, generated the gel system with dense interpenetrating space network structure. The new type of high temperature resistant inorganic particles suspend in the gel solution uniformly and support each other with gel for improving the temperature resistance, erosion resistance of the profile control agent. Inorganic particles are the tiny particles, which have the effect of deep migration with the gel ball which is cut into small pieces.

## 2. THE DEVELOPMENT OF HIGH TEMPERATRUE RESISTANT GEL MAIN AGENT

65% concentrated nitric acid is used in experiments by a certain percentage which makes modification reaction with the traditional high-temperature main agent, on the basis of the original molecules, increasing nitrocellulose to improve heat resistance performance. By contrasting gelling colloidal temperature resistance, screening and optimizing formulation, the best of the main agent are made[3].

High temperature resistant test results show that when the solid-liquid ratio is 1:6, it has the least amount of dehydration at high temperatures after the Generated main agent gelled, and still keep high strength and toughness.

The PH of the preparation of modified high temperature resistant main agent product is between 2 and 3 in the end, the PH of aqueous solution value is neutral, when the mass fraction is greater than or equal to 14%, the solubility is saturated and is dark red, the generated gel meet the requirements of high temperature resistant performance under the high temperature of 280 ℃[4].

## 3. THE EXPERIMENTS OF RATIO OPTIMIZATION

Experimental drug: Coagulant, Modified high temperature resistant main agent, cross-linking agent I, cross-linking II, PH adjusting agent, formation water.

Experiment instrument: Hydrothermal synthesis reactor, electric balance, Brookfield rotary viscometer, incubator, electric blender, PH meter, measuring cylinder, beaker and so on.

### 3.1 The Influence of Coagulant Aid HPAM on the Gel System

Main agent content is selected 6 wt. %, the content of cross-linking agent I is 2.2 wt. %, the content of cross-linking agent II 1.5 wt. %, Investigation into the effects of the content of HPAM on the gelling properties of the gel system.

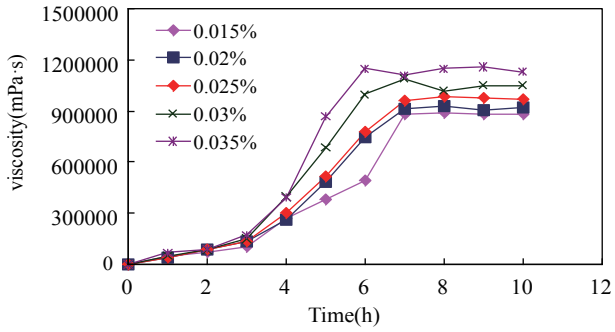


**Figure 1**
**The Influence of HPAM on the Gel System**

As shown in Figure 1, the content of HPAM is proportional to the initial viscosity of the solution, the viscosity increases with the content of HPAM increases, but the gelling time reduces. So, the content of HPAM is 0.03 wt. %.

### 3.2 The Influence of the Modified High Temperature Resistant Main Agent on the Gel System

The content of HPAM is selected 0.03 wt. %. The content of cross-linking agent I is 2.2 wt. %. The content of cross-linking agent II is 1.5 wt. %. Investigate into the effects of the content of high temperature resistant main agent on the gelling properties of the gel system.

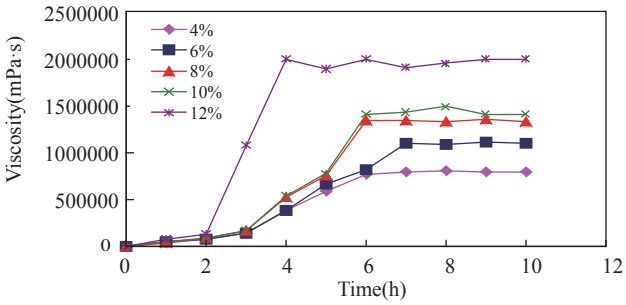


**Figure 2**
**The Influence of the Content of Main Agent on the Gelling Time and the Gelling Viscosity**

As shown in Figure 2, the content of the main agent is proportional to the gelling viscosity, but inversely proportional to the gelling time, when the content of the main agent is 6%, gelling time is the longest, the content of the main agent is between 6% and 10%, gelling viscosity change little. So, the content of the main agent is 6%.

### 3.3 The Influence of the Cross-Linking Agent I on the Gel System

Main agent content is selected 6 wt. %, the content of HPAM is 0.03 wt. %, the content of cross-linking agent II 1.5 wt. %. Investigate into the effects of the content of cross-linking agent I on the gelling properties of the gel system.

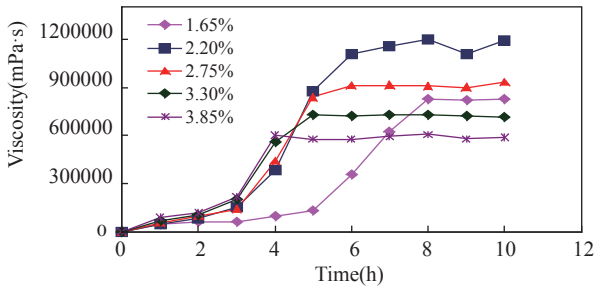


**Figure 3**
**The Influence of the Content of Cross-Linking Agent I on the Gelling Time and the Gelling Viscosity**

The content cross-linking agent I is 2.2%, the mole ratio of cross-linking agent and cross-linking agent I II is 2:5, the gelling viscosity of the System is the largest, when the content of cross-linking agent increase, the viscosity decrease; The gelling time of the cross-linking agent I content within 1.65%-3.85% reduce with the increase of the content of cross-linking agent. So, the content of cross-linking agent I is 2.2%.

### 3.4 The Influence of the Cross-Linking Agent II on the Gel System

Main agent content is selected 6 wt. %, the content of HPAM is 0.03 wt. %, the content of cross-linking agent I 2.2 wt. %. Investigate into the effects of the content of cross-linking agent II on the gelling properties of the gel system.

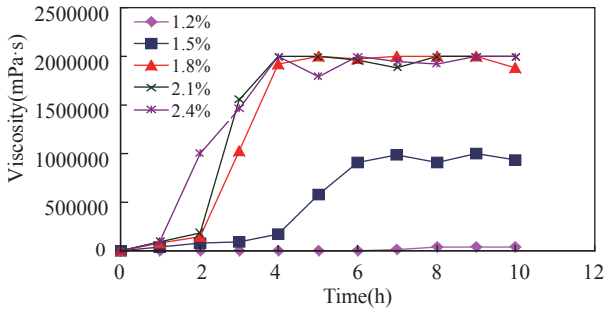


**Figure 4**
**The Influence of the Content of Cross-Linking Agent II on the Gelling Time and the Gelling Viscosity**

The content of cross-linking agent II is proportional to the gelling viscosity, is inversely proportional to the gelling time, When the content of cross-linking agent II is more than 1.8%, the final gel is semi-solid state, the gel is strength and the gel time is less than 4 hours, gelling time is short.

At last, through the experiment of ratio optimization, the formula is 0.03% coagulant aid 2.2%cross-linking agent I + 1.8% cross-linking II + 6% high efficient main agent.

## 4. THE OPTIMIZATION EXPERIMENT PF COMPOUNDED SYSTEM

Drugs: Sodium carboxymethylcellulose (CMC), graphite particle, Coagulant aid, high temperature resistant main agent, cross-linking agent I, cross-linking agent II.

Instrument: tranquil flow pump, blender, PH meter, the sand filling tube, incubator, some pipelines, some the valves and four-ways, beaker, glass rod.

(i) The content of suspending agent

Experiments are aim to explore the suspension ability of the suspending agent to graphite particles, By changing the content of suspending agent for suspension rate as the evaluation standard of the suspension ability[5,6], the computation formula of the rate of suspension is as follows:

$$Q = (h_1\text{-}h_2)/h_1 \quad (1)$$

Where: Q is the rate of suspension, ml/ml; $h_1$ is the original particle volume of the suspension,ml; $h_2$ is the water which is separated out of the suspension after sit48 hours, ml.

The results of the experimental show that when the content of CMC is 0.5%, the suspension performance is good, After 48 hours it almost won't appear the settlement, when it is 25 ℃,the viscosity is the 61.4 mPa·s, When the content of the graphite particles is 2%, the liquidity and dispersion performance are good, it has not the phenomena of water separating. So, the content of CMC is 0.5% at last.

(ii) The content of the new inorganic particles

By measuring the plugging rate of sand filling tube of compounded system under different content of inorganic particle and the plugging rate which is scoured by 30 PV steam as the standard to screen and optimize the content of inorganic particles in experiment.

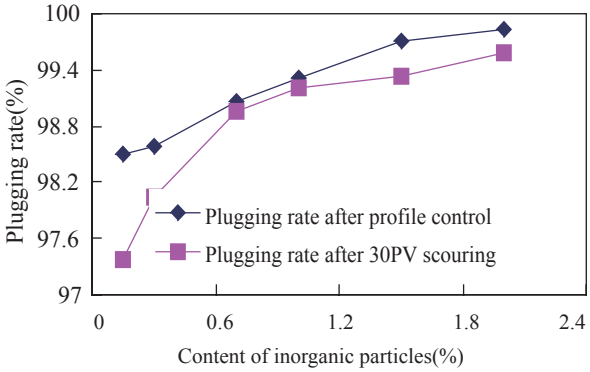


**Figure 5**
**The Content of New Inorganic Particle**

As shown in Figure 5, with the content of the particle increasing, plugging rate increases, plugging rate is more than 98.4%, when the content of the graphite is more than 2%, it is difficult to injection. The experiments of scouring resistance show:After scouring, with the increasing of the content of particles, the plugging rate reduces until leveled off. When the content of the particle is 0.7%, the curve appears inflection point. So the choice of the content of particles of compounded systems is 0.7%.

## 5. STATIC PERFORMANCE EVALUTI-N OF PLUGGING AGENTS SYSTEM

### 5.1 The Influence of Temperature on the Gel Properties

Set the temperature 160 ℃, 180 ℃, 200 ℃, 240 ℃, 280 ℃, examine the impact of temperature on the gel properties (Table 1).

Experiment results show that the gelling viscosity of the profile control agent system is more than 43.2 $\times 10^4$ mPa·s in the range of the simulated formation temperature, it can meet the needs of the plugging, the system has the advantage of high efficiency and the wide range of gelling temperature[7].

**Table 1**
**The Influence of Temperature on Gelling Time and Viscosity**

| T/℃ | 160 | 180 | 200 | 240 | 280 |
|---|---|---|---|---|---|
| Gelling viscosity /mPa·s×$10^4$ | 43.2 | 125 | 200 | 135 | 200 |

### 5.2 The Influence of PH Value on Gel Properties

Set reaction temperature 200 ℃, studying the effect of PH on gel properties (Table 2).

Experiments show that when PH is between 6 and 8, it has a little influence on gelling viscosity and gelling time. When the PH > 9 or PH < 5, the change of the gelling viscosity is irregular over time, and it can't be gelled. Salinity has little influence on the performance of gel. Therefore it is no longer here.

**Table 2**
**The Influence of PH Value on Gelling Time and Viscosity**

| No. | PH | Gelling time /h | Gelling viscosity / mPa·s |
|---|---|---|---|
| 1 | 6 | 6 | $2\times10^6$ |
| 2 | 7 | 5 | $2\times10^6$ |
| 3 | 8 | 7 | $1.57\times10^6$ |
| 4 | 9 | - | No gelling |

## 6. DYNAMIC EVALUATION TEST

### 6.1 Plugging Ratio and Residual Resistance Factor

Turn the sand filling tube into vacuum, water-saturated. Then, measure the pore volume and the permeability before plugging, displace 3PV profile control agent into sand filling tube at a constant speed of 1 ml/min, sealed, and put it into thermostat at the temperature of 200℃ for 12 hours until gelatinized, compare the plugging ratio and residual resistance factor of gel system and composite system at different temperature (Table 3).

Compounded system has better sealing ability than gel system. Plugging rate achieves above 99%. Plugging effect is little changed with temperature.

**Table 3**
**Result of Plugging Ratio and Residual Resistance Factor**

| Type | T(℃) | Perm-plug method(mD) | Water permeability measurement(mD) | | Plugging ratio(%) | RRF |
|---|---|---|---|---|---|---|
| | | | Before the seal | After the seal | | |
| Gel | 200 | 3435 | 680 | 14 | 97.94 | 48.57 |
| | 240 | 2830 | 670 | 12 | 98.21 | 55.83 |
| | 280 | 3611 | 801 | 26 | 96.75 | 30.81 |
| Compound | 200 | 3015 | 988 | 9 | 99.08 | 109.8 |
| | 240 | 3244 | 912 | 8 | 99.12 | 114 |
| | 280 | 3158 | 945 | 9 | 99.05 | 105 |

### 6.2 Scouring Resistance and Thermal Stability

#### 6.2.1 Scouring Resistance

Injecting 30 PV steam into the sand filling tube at the rate of 3 ml/min after sealed, Simulation environment temperature is 280 ℃, back pressure is 2.4 MPa. Measure water phase permeability after scoured, and compare the plugging ratio before and after scoured (Table 4). Compare with gel system, after 30 PV high speed and high temperature steam flushing. Plugging ratio of compounded system only drops in 0.35%. It means that compounded system has excellent perform in terms of scouring resistance and it can extend the period of validity.

**Table 4**
**The Compared Result of Scouring Test**

| Injection medium | Perm-plug method (mD) | Water permeability measurement(mD) | | | | Plugging ratio(%) | | |
|---|---|---|---|---|---|---|---|---|
| | | Before the seal | After the seal | 15 PV | 30 PV | After the seal | 15 PV | 30 PV |
| Gel | 3435 | 680 | 14 | 71 | 336 | 97.94 | 89.56 | 50.59 |
| Composite | 3224 | 912 | 8 | 9 | 11 | 99.12 | 99.01 | 98.78 |

### 6.2.2 Thermal Stability

Under the condition of high temperature, most polymers are prone to degrade to hydration. In the steam flooding, retention ability of profile control agent can be measured by thermal stability through the curve of time-plugging ratio.

The formation temperature of simulated is 280℃. By plugging rate change with time to study thermal stability[8].

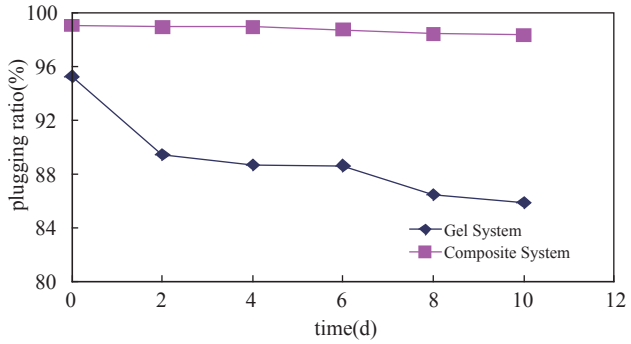


**Figure 6**
**Result of Gel and Compounded Thermal Stability**

At the temperature of 280 ℃, plugging ratio is still above 98.4% after 10 days and gradually to be steady. Particles and gel support each other to approve excellent thermal stability. The compounded profile control agent still has good performance after gel system degrade.

## CONCLUSION

a. Compounded with the new high-temperature -resistant inorganic materials and the modified high-temperature-resistant gel system, New thermal recovery high-temperature-resistant composite profile control agent is provided through simple preparation process, excellent performance and widely sources of materials;

b. The gel system contained in the compounded system has a wide reaction temperature. It withstands at least 280℃. And suitable pH is range from 6 to 8. The compounded control agent has good performance in injection, scouring resistance and thermal stability. Plugging rate is more than 99%;

c. Compounded system has high plugging rate with the less amount of particle, Gel can be degraded by the steam washing, the lubricity of particles make it harmless to pump during the injection and production;

d. Using the double-fluid method to inject under the condition of low pressure and low emissions. The new thermal recovery high-temperature-resistant compounded profile control agent owes a high practical value and a wide application prospect.

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