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Study on Plugging Technology in Oil Test

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Abstract

In view of the shortcomings of poor temperature resistance, poor pumpability and poor pressure-bearing capacity of commonly used gel plugging materials, polyacrylamide (HPAM) and N,N-methylene bisacrylamide (BIS) were selected for crosslinking and compounding to make a new type of gel plugging material with high temperature and pressure resistance. The compressive strength and yield stress were measured by inverted observation method to evaluate the gel strength. The anti-temperature, anti-pressure, anti-dilution and gel-breaking properties of the gel were evaluated. Finally, 71 type high temperature and high pressure water loss instrument and high temperature and high pressure filter with slit plate were used to evaluate the plugging capacity of gel plugging agent. The experimental results show that the new gel plugging system between 100˚C - 120˚C, gelation time can be controlled at about 5 h; it has strong temperature resistance, compression resistance, dilution resistance and gel breaking performance. In the face of permeability and fracture leakage simulation experiments, when the ambient temperature below 120˚C, pressure within 5 MPa, the filter loss of gel plugging agent is far less than the market two conventional plugging agent, has excellent plugging performance.

Keywords

Changqing Oilfield, Leakage, New Gel Plugging Agent, Gel Strength, Temperature and Salt Resistance, Plugging Performance

1. Introduction

Petroleum, also known as crude oil, is a mixture of various alkanes, cycloalkanes and aromatic hydrocarbons. It is an important non-recyclable resource and an extremely precious strategic material. It can be used in many industrial countries, industrial systems can also be used for the development of national economy. Moreover, due to the differences in global economic development, the non-sustainable use of oil, and the differences in its energy production methods and consumption regions, it has become an unavoidable thing for countries to develop. China's rapid economic development, the growing demand for oil and gas resources, the development of coal resources is also gradually increasing. Therefore, how to continuously and steadily improve crude oil production and production efficiency has become a top priority [1].

The key to oil and gas development in oilfield development is exploration, drilling, cementing and oil production [2]. The success of oil drilling test not only directly determines whether the oil field can operate safely and stably, but also relates to the production efficiency and service life of the oil field [3]. There are many potential safety problems caused by drilling technology in the current oilfield drilling test. Lost circulation is a common malignant event in drilling operations. It is mainly due to the leakage of various working fluids from pipelines into reservoirs during drilling, resulting in the loss of drilling fluid. With the leakage of drilling fluid, problems such as sticking, well kick and well collapse may occur in drilling. When the drilling fluid leaks into the formation, it will cause great damage to the production reservoir of the oilfield, and it will cause huge losses while plugging the leakage cracks [4]. Therefore, it is urgent to study the oil test plugging technology and materials.

A survey shows that 45% of the oil fields have to carry out lost circulation plugging after drilling to a certain extent [5]. Even so, 18% - 26% of the wells will leak after plugging [5]. Nowadays, with the gradual deepening of drilling depth, more and more lost circulation occurs in drilling. Therefore, targeted treatment of well leakage as soon as possible can reduce the risk and harm of well leakage. In the process of drilling, selecting appropriate plugging materials and adopting corresponding process measures can effectively reduce and deal with the problem of lost circulation in drilling. Compared with conventional plugging materials, the gel type plugging material has better deformation performance and viscous performance, and can form a plugging material with higher strength and better performance. However, the commonly used gel plugging materials have poor temperature resistance, poor pumpability, and poor pressure-bearing capacity, which has brought great trouble to the plugging. Therefore, polyacrylamide and N,N-methylenebisacrylamide were selected for crosslinking and compounding to make high-performance temperature-resistant and pressure-resistant gel plugging materials.

2. Development of New Gel Plugging Agent Formula

2.1. Screening of Crosslinking Agent Monomer

According to the situation at home and abroad, oilfield commonly used synthetic plugging material polymer mainly includes: polyacrylamide, polyacrylonitrile, polyvinyl alcohol, polyethylene amine and AM monomer copolymer, among which polyacrylamide is the most common [6]. However, the common inorganic crosslinking agent has poor temperature resistance and cannot withstand the high temperature and high pressure environment in the formation. Therefore, the organic crosslinking agent is selected for crosslinking to obtain the required gel plugging agent.

In this paper, polyacrylamide and crosslinking agent at 120˚C were selected to evaluate the gelation condition, and the optimal organic crosslinking agent was selected from hexamethylenetetramine (MTA), N,N-methylene bisacrylamide (BIS) and phenol.

It can be seen from **Table 1** that polyacrylamide can gel in the case of three crosslinking agents. Because phenol has a great impact on people and ecosystems, and the material is flammable [7], It is therefore not selected. Compared with other crosslinking agents, MTA has worse pumpability, which may lead to pump holding in the subsequent practical application process, so it is difficult to enter the formation to be reached and affect the progress of construction. N,N-methylenebisacrylamide (BIS) has both good gelation and good pumpability. Finally, the system of water + polyacrylamide (HPAM) + N,N-methylenebisacrylamide (BIS) was determined.

2.2. Design of Experimental Scheme

The plugging mechanism of the new gel plugging agent is that when the polyacrylamide is fully dissolved in water and the cross-linking agent BIS is added, the active groups on the molecular chain of the polyacrylamide react with the active groups on the BIS molecular chain to form a polymer chain, and the polymer chains will cross-link each other to form a space network structure, which will wrap the free water in it. Finally, the solution loses its fluidity and forms a gel [8], Filling into each pore crack, complete sealing.

Plugging construction, gelation time is an important part, if the gelation time is too short, before the material has been pumped into the formation, has formed a gel, can not complete the plugging work; if the gelation time is too long, it cannot be blocked in time, which will have a serious impact on the field operation. The appropriate gelation period should be 1 hour to 5 hours. So it is very important to accurately determine the gelation time of the gel. After determining the monomer, we will use single factor analysis and quantitative method, by optical micro rheometer to observe the gelation time and gelation state [9]. The formula of new gel plugging agent was determined.

Table 1. Gelation status of polyacrylamide and crosslinking agent.

Crosslinker	Gelation	Pumpability
Phenol	gelatinize	ease
MTA	gelatinize	More difficult
BIS	gelatinize	ease

2.3. Determination of New Gel Plugging Agent Formulation

Based on the characteristics and economy of each chemical composition, the formula concentration of the system was initially determined to be about 2% polyacrylamide and about 0.1% BIS. The experimental conditions were used to simulate the real situation of the downhole, and the experimental temperature was selected to be 100˚C - 120˚C. According to the experimental results, it was judged whether the system could be stably formed in a high temperature environment. Using single factor analysis, a factor for a small range of selection finds out the best situation, and analyzes its causes [10].

2.3.1. Determination of Polyacrylamide Concentration

The mass fraction of polyacrylamide is greatly related to its gelation ability and strength after gelation, so it will have a great influence on the plugging effect. The amount of polyacrylamide was adjusted by fixing the BIS concentration, and the gelation time and state under various reaction conditions were measured to obtain the optimal concentration. The concentration of polyacrylamide was 1.5%, 2%, 2.5%, the concentration of BIS was 0.1%, the experimental temperature was 100˚C, 110˚C and 120˚C, and the gelation was as shown in Table 2 and Figure 1.

As shown in Table 2 and Figure 1, under fixed conditions, with the increase of polyacrylamide content, the gelation time is shortened; when it is at a fixed concentration, its gelation time will also decrease with increasing temperature. This is because the gelation time is related to the formation rate of the network structure. At a higher concentration, due to the presence of a large number of

Table 2. Effect of different concentrations of HPAM on the initial viscosity of gelling.

concentration (%)	1.5	2.0	2.5
initial segment viscosity (MPa·s)	3000	5700	8800

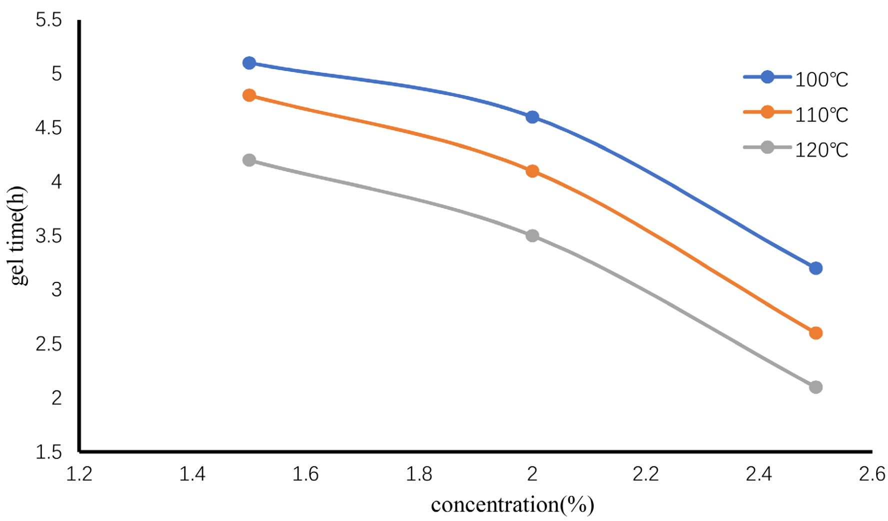

Figure 1. Effect of different reaction temperature and different concentration of HPAM on gelation.

active groups in the solution, cross-linking polymerization is prone to occur, thereby gelation, so the gelation time is reduced. As the temperature increases, the network structure forms faster, resulting in faster gelation [11].

The gelation time and gelation state were observed by optical microrheometer. The elastic factors (EI) of 1.5%, 2%, 2.5% polyacrylamide at 120˚C are 0.0016 nm^{-2}, 0.007 nm^{-2}, 0.03 nm^{-2}. Elasticity index (EI) is an important index to reflect the bonding strength between the grids in the gel. With the increase of the strength of the grid structure in the gel, the EI value increases and the elasticity of the gel becomes stronger. Based on the test results, the concentration of polyacrylamide was set at 2% from the factors of pumpability, gelation time and elastic strength after gelation.

2.3.2. Determination of N,N-methylene Bisacrylamide (BIS) Concentration

After determining the concentration of polyacrylamide, it is necessary to observe the effect of crosslinking agent BIS concentration on the gelation of the system, so as to determine the optimal concentration of BIS. Therefore, the concentration of polyacrylamide was fixed, the concentration of BIS was adjusted, and the gelation time and state at different reaction temperatures were observed to obtain the optimal concentration. The concentration of BIS was 0.05%, 0.1%, 0.15%, the concentration of polyacrylamide was 2%, the experimental temperature was 100˚C, 110˚C and 120˚C, and the gelation was as shown in Table 3 and Figure 2.

It can be seen from Table 3 and Figure 2 that the concentration of BIS has no

Table 3. Effects of different reaction temperatures and different concentrations of BIS on gelation.

concentration (%)	0.05	0.1	0.15
initial segment viscosity (MPa·s)	5880	5600	5650

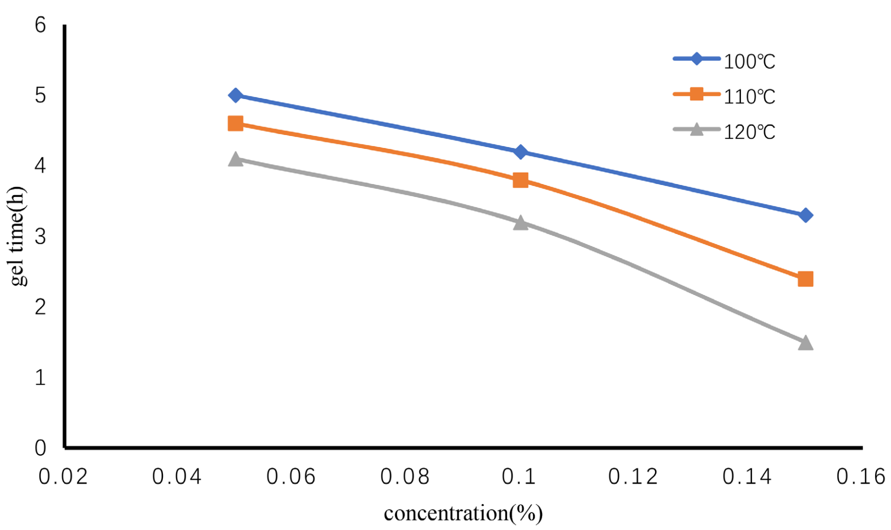

Figure 2. Effects of different reaction temperatures and different concentrations of BIS on gelation.

obvious change on the initial viscosity of the solution. With the increase of BIS concentration, the gelation time is shortened accordingly, which is in line with the general gelation law. This is due to the higher the concentration of BIS, the more BIS reacts with polyacrylamide, the greater the possibility of crosslinking, thereby shortening the gelation time.

The gelation time and gelation state were observed by optical microrheometer. The elastic factors (EI) of 0.05%, 0.1%, 0.15% BIS gel at 120˚C were 0.0017 nm^{-2}, 0.006 nm^{-2}, 0.003 nm^{-2}. According to the change of elastic coefficient (EI) of the three components, with the increase of BIS concentration, the elastic coefficient (EI) showed a trend of first increasing and then decreasing, and when the concentration of BIS was 0.1%, the gel strength was the highest. Based on the comprehensive test results, the concentration of BIS was set to 0.1% from the factors of gel stability, gel time and strength after gel formation.

3. Performance Evaluation of New Gel Plugging Agent

3.1. Evaluation of Conventional Performance

3.1.1. Anti-Dilution Performance

Determination of Antidilution Performance of New Gel Plugging Agent by Sieve Residue Method [12].

$$\Delta m = \frac{m_2 - m_1}{m_1} \quad (1)$$

Under the change of glue ratio, the anti-dilution ability of the colloid is quite different, but the glue ratio cannot be too low or too high, so the glue ratio is set to 1:5.

1) Effect of temperature

Under certain conditions, temperature can promote the thermodynamic behavior of the gel, thereby accelerating the absorption rate of water by the gel [13]. Therefore, according to the experimental steps, the anti-dilution performance of the gel was evaluated after stirring at different temperatures for 5 minutes.

From **Table 4**, under certain conditions, the dilution degree of the gel increases with the increase of temperature, but the difference is not large, indicating that it has a strong anti-dilution performance.

2) Effect of time

Using the same method, the relationship between anti-dilution property and dilution time was studied at room temperature.

Table 4. Anti-dilution properties of gel at different temperatures

Temperature (˚C)	m_1 (g)	m_2 (g)	Δm (%)
25	9.85	13.27	34.72
100	10.24	14.55	42.09
110	10.12	14.63	44.57
120	10.11	14.87	47.08

Table 5. Anti-dilution properties of gel at different time.

Time (min)	m_1 (g)	m_2 (g)	Δm (%)
3	9.85	13.27	34.72
5	10.12	14.19	40.23
10	9.65	15.71	62.75
15	9.96	17.50	75.69
20	10.35	19.08	84.31
30	10.67	21.47	101.24

From **Table 5**, it can be seen that the degree of dilution of the gel increases with time, and the value of Δm in each period is positive, which shows that the anti-dilution performance of the gel is excellent. From the experimental process, we can know that the gel not only has good anti-dilution performance, but also has slight water absorption and expansion, which can make it swell in the leakage channel, thus improving its own plugging ability.

By analyzing the experimental results of the anti-dilution performance of the gel under the above different conditions, it can be found that the gel has good anti-dilution performance.

3.1.2. Strength

The gel strength was evaluated by visual evaluation method. Refer to the GSC visual code table in Sydansk [14]. By observing that the gel we obtained is placed in the beaker, when the beaker is vertically inverted, only the surface of the gel will undergo slight deformation. It can be seen that the gel strength we obtained is E-F grade.

Compressive strength is also one of the indicators of the performance of the reaction material. Due to the increase of formation depth and formation pressure, the plugging material needs to plug the leakage channel under high pressure conditions. If the pressure bearing capacity is low, it is difficult to play a plugging role [15]. Therefore, it is necessary to test the compressive strength of the solidified product to meet the requirements of high pressure formation.

It can be seen from **Figure 3** that the compressive strength of all solidified 5h gel samples is above 15 MPa. The gel has high compressive strength. In summary, the gel has good strength.

3.1.3. Temperature Resistance

Evaluation of the Temperature Resistance of a New Gel Plugging Agent by Measuring the Changes of Water Locking Ability and Strength of Gel after a Period of High Temperature Heating [16].

$$S = \frac{m_1 - m_2}{m_1} \tag{2}$$

A polyacrylamide solution of 2% concentration and a BIS of 0.1% concentration are completely cross-linked to form a gel plugging agent. Four groups of gel plugging agents of certain quality were weighed and their mass m_1 was recorded. Three groups of samples were placed in three aging tanks and heated at 120˚C for 8 h, 16 h and 24 h, while the other group was not heated as a blank control group; the heated gel was taken out and the mass of the remaining gel was weighed m_2. Calculate the water loss rate S of the gel; by inversion method, the gel tongue length was observed to intuitively evaluate the gel strength.

It can be seen from Table 6 that the water loss rate of the gel increased with the increase of heating time, but only about 10% of the water was lost when the gel was heated for 24 h, indicating that the gel had good temperature resistance and water locking ability. The gel heated at 120˚C for 24 h is not easy to be degraded, and can maintain similar strength with the original, with good temperature resistance.

3.1.4. Salt Resistance

In liquids with different salt concentrations, the absorption performance of the gel is different. Therefore, the effects of different salt concentrations on gel strength were studied by experiments. We added 3%, 6%, 9% concentrations of NaCl solution to the prepared polymer base liquid, and placed it in a 120˚C environment to make it completely cross-linked and condensed into a gel, and then observed the gel tongue length by inversion method to intuitively evaluate the gel strength.

It was found that the gel in the three sample bottles flowed to the cup mouth

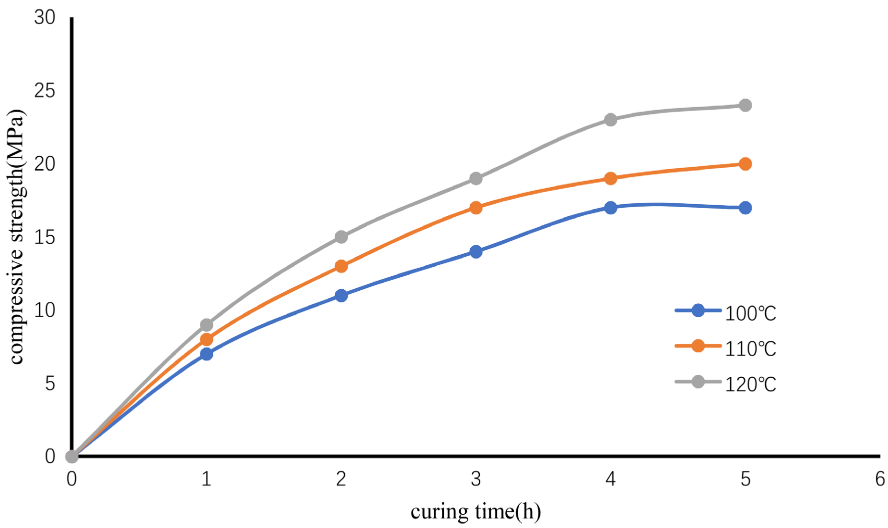

Figure 3. Changes of compressive strength of gel with time at different temperatures.

Table 6. The degree of dehydration of gel under different heating time.

heating time (h)	m_1 (g)	m_2 (g)	S (%)
8	246	234	5
16	251	230	8
24	238	210	12

at an extremely slow speed, which was grade E gel. The gel strength generated under the condition of 6% NaCl solution was the strongest, and the gel strength generated under the condition of 3% and 9% NaCl solution was slightly weaker, but the difference was not significant. It is proved that the gel has good salt resistance.

3.1.5. Gel Breaking Performance

In order to achieve automatic gel breaking, it is necessary to encapsulate the oxidant as the gel breaker of the system to achieve automatic gel breaking at 90˚C. With the increase of gel breaking time, the gel mass decreases and the aqueous solution increases gradually, so the gel breaking rate can be calculated by the mass of the rubber block before and after gel breaking [17], rubber breaking rate calculation formula is:

$$C = \frac{m_1 - m_2}{m_1} \quad (3)$$

Prepare 6500 mL beakers, and place each concentration of gel breaker in them; the gel mass m_1 that had been gelled for 3 days was weighed, and the gel breaker of each concentration was added to the gel respectively. The amount of gel breaker added was 1/8 volume ratio of the previously prepared polymer base solution. Six groups of materials were poured into the aging tank and placed in a roller heating furnace at 120˚C. The materials were heated without turning over, and removed every 12 hours. The solid was filtered out with a sieve, and the gel mass m_2 was weighed. Until the final gel solid mass does not change, calculate the gel breaking rate.

According to the data in **Figure 4** and **Table 7**, under the action of 10% and

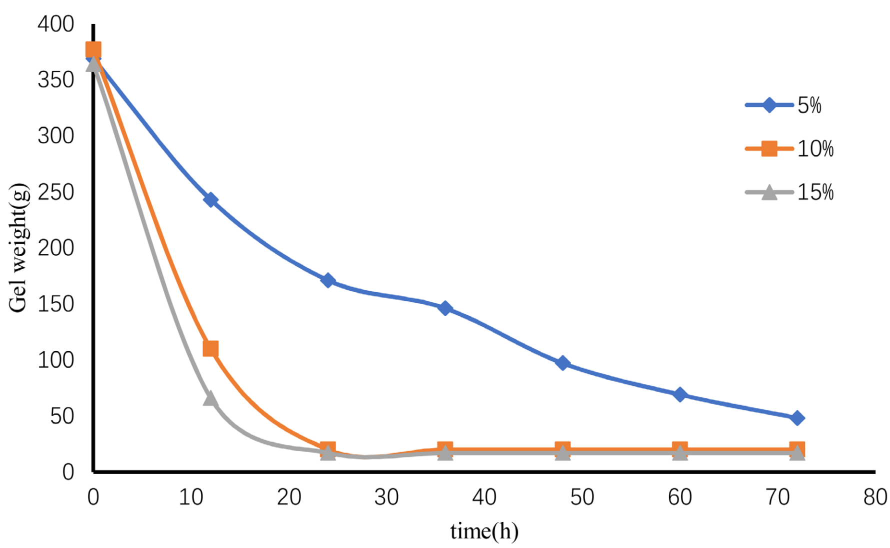

Figure 4. Gel quality under different dosage of breaker at 120˚C.

Table 7. Breakage rate at 120˚C with different amount of gel breaker.

Breaker concentration/%	5	10	15
Degrees of gel breaking/%	87.0	94.7	95.3

15% capsule oxidation gel breaker gel breaking rate reached more than 90%, are completed within 24 h gel breaking. Therefore, the new gel plugging agent has good gel-breaking performance by using the capsule oxidation gel-breaking agent under the corresponding temperature conditions.

3.2. Evaluation of Sealing Performance

3.2.1. Plugging Effect of Permeable Leakage

The sand bed with heterogeneous porosity can be used to simulate the state of underground permeable formation, and the pore size of different formations can be simulated by adjusting the particle size of quartz sand [18].

Prepare sufficient base slurry for use; the plugging system is prepared according to the formula and is formed at an appropriate temperature for use; weigh 200 g quartz sand of different mesh numbers into the high temperature and high pressure filter press, add the formed plugging system, and then inject a certain amount of base slurry; the instrument is connected with the pressure device by sealing the cover, and the heating switch is opened to heat to the target temperature. Put a measuring cylinder in the corresponding place to measure the leakage of the sand bed; when the target temperature is reached, the initial pressure of 0.5 MPa is injected, and the pressure is gradually increased from 0.5 MPa to 5 MPa, and each time is stabilized at different pressures for 30 min. Measure and record the filter loss in the graduated cylinder.

According to **Figure 5**, under the same pressure, the filter loss of base slurry increases with the increase of sand bed pore. Under the same pore size, the filtration loss of the base slurry increases first and then stabilizes with the increase of pressure. In the whole simulation experiment, the leakage is small when facing the simulated sand bed with different pore sizes, and no colloid is pressed out. It can be seen that the new gel plugging system can plug 10 - 60 mesh sand beds with different simulated permeability.

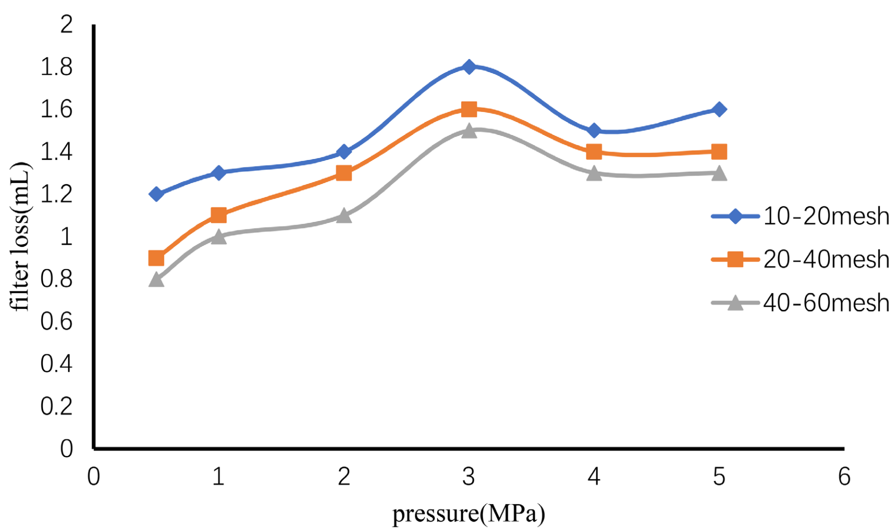

Figure 5. Filtration loss of quartz sand with different particle sizes under different pressures at room temperature.

3.2.2. Fractured Leakage Plugging Effect

Plugging experiment of simulating leakage in fractured formation by fracture plate simulating fractures in formation [19]. Different crack widths are achieved by replacing different slit plates. preparing sufficient base slurry for use; a sufficient amount of plugging system is prepared according to the formula and completely formed at a suitable temperature for standby; preparation of 1 mm, 2 mm, 3 mm, 4 mm slit plate, respectively, in the four groups before the experiment installed at the bottom of the high temperature and high pressure filter, and then to form the plugging system to join, and finally poured into a certain amount of prepared base slurry; sealed with a cover, the instrument is connected with the pressurizing device, the heating switch is turned on and heated to the target temperature; put a measuring cylinder in the corresponding place to measure the leakage of the sand bed; when the target temperature is reached, the initial pressure of 0.5 MPa is injected, and the pressure is gradually increased from 0.5 MPa to 5 MPa, and each time is stabilized at different pressures for 30 min. Measure and record the filter loss in the graduated cylinder.

According to **Figure 6**, with the increase of fracture width, the filtration loss of drilling fluid also increases. Under the same gap size, the filter loss of base slurry increases first and then becomes stable with the increase of pressure. In the whole simulation experiment, the leakage is small when facing cracks of different sizes, and no colloid is pressed out. It can be seen that the new gel plugging system can plug different simulated fracture leakage of 1 - 4 mm.

4. Conclusions

According to the experimental results, the basic formula of the new gel plugging agent was determined as water + 2% polyacrylamide + 0.1% BIS from the aspects of initial pumping, gelation time, gel strength and gel stability.

By using various experimental methods, it can be seen that the new gel plugging

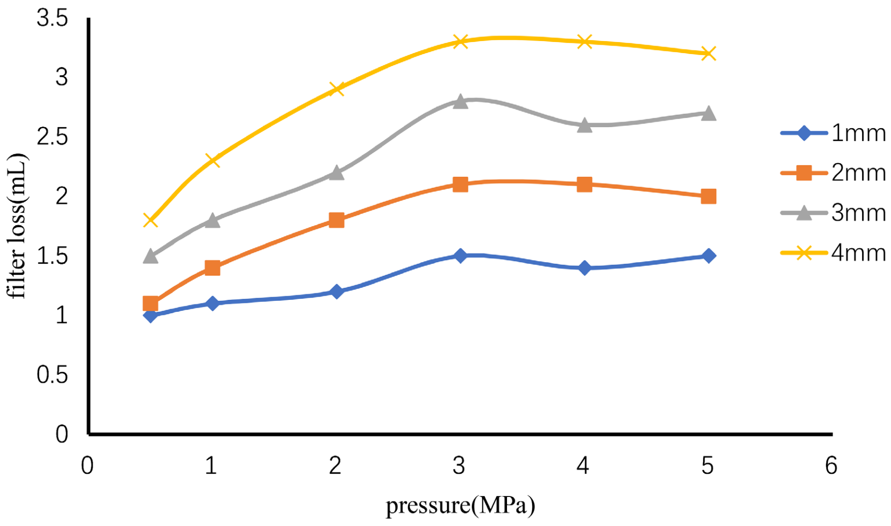

Figure 6. Filtration loss of different width cracks under different pressures at room temperature.

system has good dilution resistance, temperature resistance and salt resistance, as well as high strength, and can maintain the stability of the structure for a long time.

Under the action of 10% and 15% capsule oxidation breaker JPC, the gel breaking rate of the gel reached 90%, and reached 94% and 95% at 120˚C, respectively, both within 24 h. Therefore, the new gel plugging agent has good gel breaking performance by using the capsule oxidation gel breaker under the corresponding temperature conditions.

The new gel plugging agent can achieve plugging in the face of pores of different sizes and cracks of different widths in an environment below 120˚C and within 5 MPa. Compared with the two conventional plugging agents on the market, the leakage of the new gel plugging agent is smaller, so the new gel plugging agent has good plugging performance for both permeable leakage and fractured leakage.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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