

Development and characterization of self-healing gel temporary plugging agent for well killing

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A new type of self-healing gel was developed by using acrylamide (AM) and other materials, and the effects of the content of PEGDA200 and PEGDA400 on the mechanical properties of gel were compared. The results showed that adding PEGDA200 and PEGDA400 to the self-repairing gel significantly improved the performance of the composite gel. When the content of PEGDA200 in the gel was 0.2%, the rheological property after repair was 109.4% of that before repair, the compressive property after repair was 95.2% of that before repair, the adhesive property was increased by 36.8%, and the compressive strength after repair was 92.4% of that before repair. When the content of PEGDA400 in gel was 0.2%, the rheological property of gel after repair was 105.8% of that before repair, the maximum adhesion could be increased by 17.8%.

Keywords: self-healing; gel; temporary plugging; well killing.

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INTRODUCTION

With the increasing difficulty of oil and gas reservoir development, underbalanced drilling technology¹ has emerged. Due to human intervention during the underbalanced drilling process, the formation pressure is greater than the drilling fluid pressure, which can avoid the impact of drilling fluid and other harmful substances on the oil and gas reservoir entering the formation. At the same time, through underbalanced drilling technology, the oil and gas in the formation can automatically flow into the wellbore, so that the drilling process can be monitored by the liquid left at the inlet. In addition, the application of underbalanced drilling technology will not form a filter cake on the wellbore wall, thus reducing the risk of drilling jamming. Although underbalanced drilling technology has many advantages, because the formation pressure is greater than the drilling fluid pressure during the underbalanced completion process, it may cause a rapid influx of formation fluids into the wellbore and even blowout. Therefore, it is necessary to continuously kill the well during the drilling process to balance the formation pressure. At present, the dynamic rubber valve technology²⁻⁴ is commonly used to balance the pressure of the formation and isolate the drilling fluid in the upper part of the wellbore from the oil and gas reservoir in the lower part.

The dynamic rubber valve technology is to inject high-viscosity gel into the wellbore, which can adhere to the wellbore to form a section of dynamic rubber plug. The dynamic rubber plug can seal the drilling fluid on the dynamic rubber plug and the oil and gas reservoir under the dynamic rubber plug through the gravity and adhesion of its own gel, and can also avoid the excessive amount of injected drilling fluid causing overbalance and damaging the reservoir. However, the mechanical strength of the colloid in conventional dynamic rubber valves is low. When the formation pressure is too high, the dynamic rubber valve will rupture due to the pressure of the formation fluid, causing the fluid to quickly flow into the wellbore and cause accidents. In addition,

the gel gel of a conventional dynamic rubber valve will lose its sealing effect if it is difficult to repair itself after being damaged. For example, when the gel is broken by the tubing, the sealing effect in the wellbore will be reduced due to the existence of cracks between the tubing and the pipe wall and the difficulty of self-repair of conventional gel. Therefore, it is necessary to study a gel with high mechanical strength and self-repair to solve the application effect of the dynamic rubber valve technology in the field.

Self-healing gel material is a kind of gel material that can heal itself after damage, and its mechanical properties after healing are close to those before healing. Self-healing gel can achieve automatic healing through physical or chemical action. Physical automatic healing mainly includes hydrogen bonding⁵, metal-ligand interaction⁶, π - π stacking, and host-guest interaction⁷. Chemical self-healing mainly includes Diels Alder bonds⁸, disulfide bonds⁹, acyl bonds¹⁰, and imine bonds¹¹⁻¹². Wang et al.¹³ synthesized a new self-healing gel by dissolving acrylamide in montmorillonite suspension using in-situ polymerization technology. The breaking elongation of the gel is up to 118 times and the tensile strength is 175 KPa. Sun et al.¹⁴⁻¹⁵ prize-winning cationic monomer was homopolymerized and then polymerized with anionic monomer to prepare a new self-healing gel. The gel has excellent mechanical properties and healing properties, with a tensile strength of 3700 KPa, an elongation of 700%, and a tensile strength of 2200 KPa, an elongation of 630% after fracture healing.

Jing et al.¹⁶ prepared a self-healing gel using chitosan and polydopamine as skeletons and cross-linking agents respectively, and graphene oxide as reinforcing nanofiller. The gel has good healing and adhesive properties at room temperature. Shao et al.¹⁷, a new type of furan-modified nanocellulose and polyethylene glycol formed a dynamic chemical bond through Diels Alder reaction, and prepared a new type of self-healing gel. The tensile strength of the gel is 140 KPa, the elongation is 700%, and the tensile strength after fracture healing at room temperature is 120 KPa, the elongation is 400%. Compa-

red with the traditional gel, the cross-linking point of the self-healing gel space network is dynamically reversible and has more excellent mechanical properties. It is widely used in the field of drilling fluid, such as plugging¹⁸⁻²⁰, leak prevention²¹⁻²⁴, etc.

The existing gels have the disadvantages of poor rheological properties and strength and can not meet the requirements. Given the above shortcomings, a new gel system was prepared by introducing peg, which has good rheological properties and high consolidation strength and can meet the temporary plugging requirements under different conditions.

EXPERIMENTS AND TESTING

Gel leakproof materials usually have good adaptive ability, so they can match the crack size well, but usually have low strength, and poor mechanical properties, and gel is easy to break. Because of these shortcomings, composite gel with good mechanical strength and self-healing ability is prepared. The main raw materials of the composite gel are acrylamide (AM) and hydrophobic monomer α -olefin hexadecyl sulfonate. Given the above shortcomings, polyethylene glycol diacrylate (PEGDA) can improve the gel properties. By adding PEGDA 200 and PEGDA 400 for comparative testing, the impact of different contents on the mechanical properties of the self-healing gel was investigated, and the strengthening mechanism of the self-healing gel was explored (Table 1).

Test Materials

Synthesis of Temporary Blocking Agent

The preparation process of test self-healing gel is as follows:

1) Dissolve 0.2% KPS in deionized water and stir with a stirrer for 3 hours (400 r/min).

2) Add 3% SDBS to the solution, and after dissolution, add 6% AM and continue stirring for 3 hours.

3) Add PEGDA200 (or PEGDA400) with different contents (0% -0.5%) to the solution and continue stirring for 1 hour.

4) Put the stirred gel base solution into the oven (70 °C) to observe the effect of the base solution. When the solution in the shaking container is not flowing, it means that the gel has been gelled. The specific preparation process is shown in Figure 1.

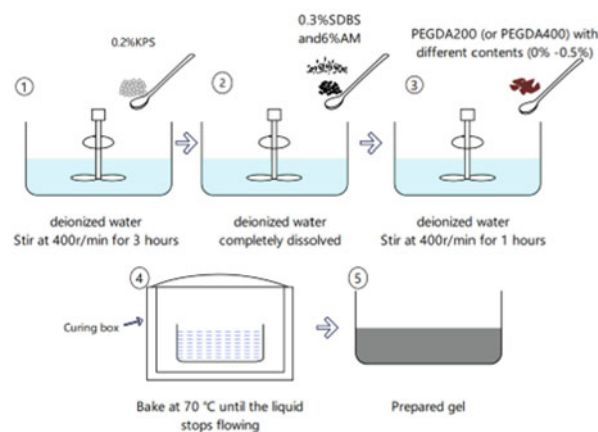


Figure 1. Preparation Process Diagram

Performance Testing

To test the mechanical properties of the self-healing gel, the introduction includes a rheological property test, stress-strain property test, adhesion test, pressure resistance test, etc. have been used (Table 2).

Rheological Performance Testing

The German HAAKE rheometer was used to carry out the rheological property test, and the self-healing gel was made into several samples of $\varphi 10 \times 3$ mm. The stress scanning and frequency scanning were carried out.

Table 1. Materials Required for the Test

Reagent name	Manufacturer	Specifications
Acrylamide (AM)	Beijing Modern Oriental Fine Chemicals Co., Ltd	99%
Olefin hexadecyl sulfonate	Laboratory self-made	—
Potassium persulfate (KPS)	Tianjin Fuchen Chemical Reagent Factory	99.5%
Polyethylene glycol diacrylate (PEGDA200)	Shanghai McLean Biochemical Technology Co., Ltd	98%
Polyethylene glycol diacrylate (PEGDA400)	Shanghai McLean Biochemical Technology Co., Ltd	98%
Sodium dodecylbenzenesulfonate (SDBS)	Tianjin Guangfu Fine Chemical Research Institute	—
Deionized water	Laboratory self-made	—

Table 2. Information on Instruments Required for the Experiment

Experimental drug	Model	Manufacturer
Electronic balance	BSA224S-CW	Shenzhen Lintao Instrument Co., Ltd
Electric Blender	SciQuip Pro40	Beijing Century Kexin Scientific Instrument Co., Ltd
Electric hot blast drying oven	DHG-101	Shanghai Runtai Automation Equipment Co., Ltd
Rheometer	HAAKE RS600	HAKKE GmbH, Germany
Voltage resistant device	—	Laboratory self-made
Texture analyzer	TA.XT PLUS	Beijing Weixun Chaoji Instrument Technology Co., Ltd

The set stress was 10 Pa from the stress scanning, and the frequency scanning range was 0.01–10 Hz, then the viscoelastic change curve of the gel could be obtained.

Stress-strain performance testing

Using TA XT texture analyzer carried out the compression stress-strain test of the composite gel for PEGDA200 and PEGDA400. Before gelation, the base solution was poured into a $\varphi 15 \times 15$ cylindrical mold, and the mold was placed in an oven to withstand the temperature of 70 °C/24 h to obtain a gel sample, and the gel sample was placed in TA The XT texture analyzer tests the center of the plate and sets the compression speed of the compression plate to 3 mm/s, and sets the compression strain to 95%. The relationship between compressive stress and strain during the compression process is obtained through testing.

Adhesion Test

Using TA XT texture analyzer conducts adhesive force test on PEGDA200 and PEGDA400 composite gel. Before gelation, pour the base solution into a $\varphi 50 \times 100$ cylindrical mold to ensure that the bottom of the rotor can just touch the gel. Place the gel in an oven to withstand 70 °C/24 h until a gel sample is obtained⁸. Take the sample out and fix it in the center of the bottom of the texture analyzer. Pull the rotor up rapidly at a speed of 2 mm/s until the bottom of the rotor separates from the gel. Record the change curve of the force on the rotor and the time during this process as the change curve of the adhesive force.

Voltage resistance performance test

Simulate the pressure resistance of gel in the formation to carry out the pressure test. Pour the gel liquid into the $\varphi 50$ stainless steel pipe and place it in the oven to withstand the temperature of 70 °C/24 h. After taking out the steel pipe, connect the pressure device to the steel pipe and start to press. When the pressure rises to a certain pressure type, the pressure will quickly become 0, which is the breakthrough pressure of gel. Continue to place the steel pipe in the oven to withstand the temperature of 70 °C/24 h. At this time, the broken gel will heal and repeatedly press until the pressure is 0. The pressure at this time is the breakthrough pressure after the gel repair.

RESULTS AND DISCUSSION

Rheological Performance Testing

Add PEGDA200 and PEGDA400 with contents of 0%, 0.1%, 0.2%, 0.3%, 0.4% and 0.5% (Generally, the additive dosage is less than 0.5%) respectively to prepare $\varphi 10 \times 3$ mm self-healing gel samples, and divide them into two groups for testing. One group directly carries out the initial elasticity test, and the other group is cut in the middle and placed in an oven for temperature resistance of 70 °C/24 h. After gel repair, an elasticity test is carried out. The elastic moduli of PEGDA200 and PEGDA400 at different concentrations were measured and shown in Figure 2.

As indicated in Figure 2, the elastic modulus of the composite gel is affected by different concentrations of PEGDA200 and PEGDA400. Whether it is PEGDA200

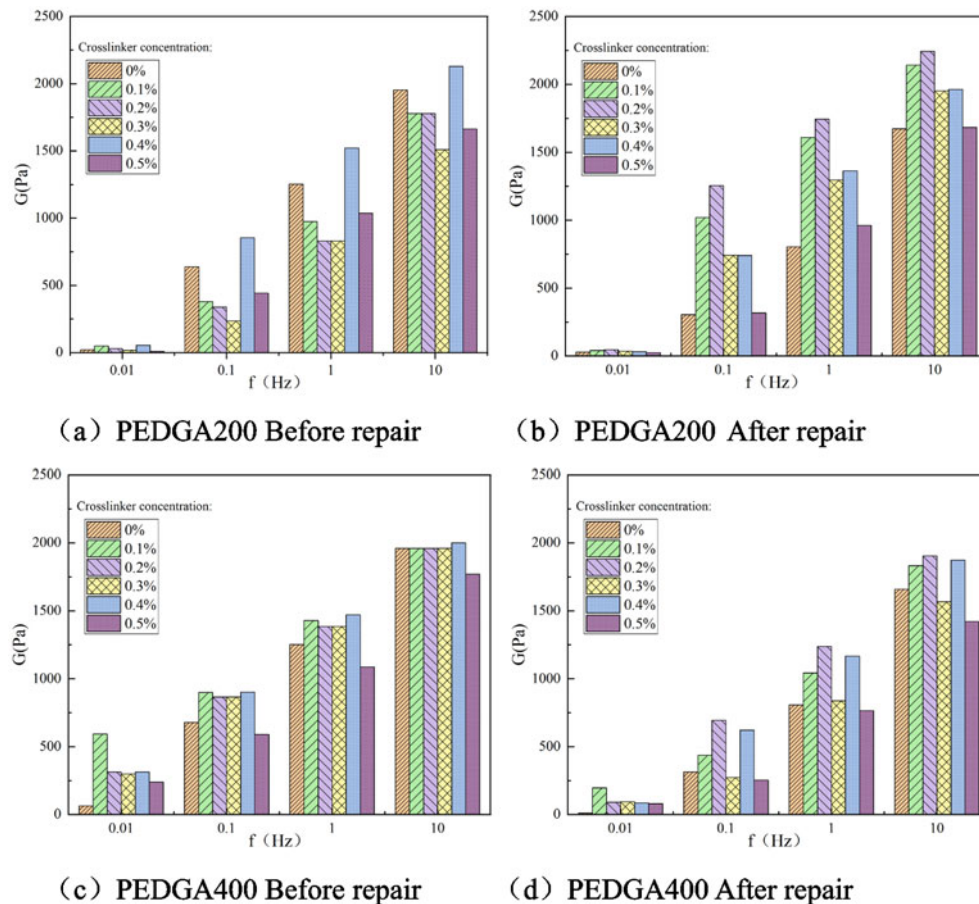


Figure 2. Comparison of elastic modulus changes before and after PEGDA200 and PEGDA400 repair

or PEGAD400, the elastic modulus of the composite gel changes with the concentration of the crosslinking agent in a similar manner.

Before the repair of the composite gel, the elastic modulus first decreases, then increases, and subsequently decreases again with the increase of the concentration of the crosslinking agent. In contrast, after the repair of the composite gel, the elastic modulus initially increases and then decreases as the concentration of the crosslinking agent rises.

Notably, when the concentration of the crosslinking agent before repair is 0.4%, the elastic modulus reaches its maximum. Similarly, when the concentration of the crosslinking agent after repair is 0.2%, the elastic modulus also peaks.

Specifically, before repair, the elastic modulus of PEGDA200 and PEGAD400 composite gels was 2128 Pa and 2000 Pa, respectively, at a concentration of 0.4%. After the repair, the elastic modulus for both types of gels was reduced to 1848 Pa and 1710 Pa, respectively, at the same concentration. Following repair, the elastic modulus of PEGDA200 and PEGAD400 composite gels achieved 86.8% and 85.5% of their values before repair.

Furthermore, when the crosslinking agent content is 0.2%, the elastic modulus of PEGDA200 and PEGAD400 composite gels before repair is 2050 Pa and 1800 Pa, respectively. After repair, the elastic modulus for PEGDA200 and PEGAD400 composite gels increased to 2243 Pa and 1905 Pa, which corresponds to 109.4% and 105.8% of the values before repair.

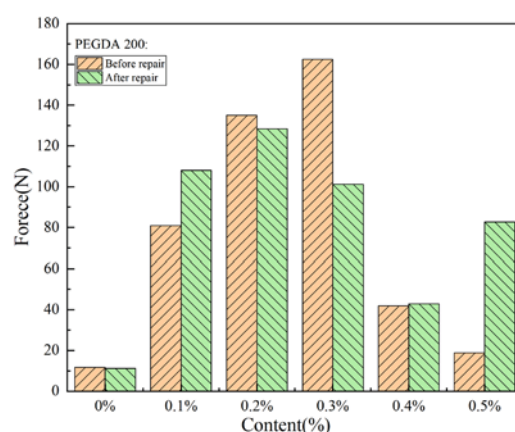
Compression Performance

Different concentrations of PEGDA200 and PEGAD400 gel base solutions were made into a number of $\varphi 15 \times 15$ gel samples and were divided into two groups for testing. One group directly carried out the initial elasticity test, and the other group was cut horizontally and vertically in the mold and placed in the oven for 70 °C/24 h. After the gel was repaired, the elasticity test was carried out. The elastic moduli of PEGDA200 and PEGAD400 at different concentrations were measured and shown in Figure 3.

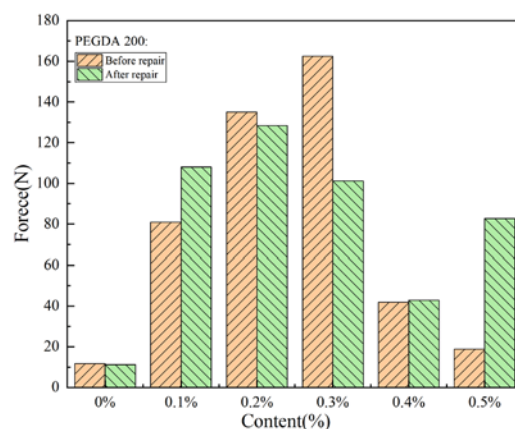
As shown in Figure 3, it can be seen that without the addition of crosslinking agents PEGDA200 and PEGAD400, the compression performance of the composite gel before and after repair is the lowest. This indicates that the crosslinking agent can improve the compression performance of the composite gel.

The influence of the content of crosslinking agent at different concentrations on the compression performance of the composite gel is slightly different. For the composite gel with the addition of PEGDA200, the compression stress is the highest when the content of the crosslinking agent before repair is 0.3%, which measures 162.6 N at that moment. The compressive stress after repair is 101.2N, representing 62.2% of the performance before repair.

When the crosslinking agent content is 0.2% after repair, the compression performance is optimal, with a compression stress of 128.5 N. Before repair, the same crosslinking agent content resulted in a compression stress of 135 N, corresponding to 95.2% of the pre-re-



(a) PEDGA200 Before repair



(a) PEDGA200 Before repair

Figure 3. Comparison of Compression Performance Changes Before and After PEGDA200 and PEGDA400 Repair

pair value. Therefore, when the PEGDA200 crosslinking agent content is 0.2%, the self-healing effect is better.

For PEGAD400, when the pre-repair concentration is 0.2%, the maximum compressive stress is 124.4 N. After repair, the maximum compressive stress at the same concentration is 80.7 N, which is 64.9% of the pre-repair value. Conversely, when the pre-repair concentration is 0.5%, the compressive stress is 84.1 N, and after repair, the compressive stress increases to 160.8 N, which is an impressive 191.2% of the pre-repair value. Therefore, when the PEGAD400 crosslinking agent content is 0.5%, the self-healing effect is better.

Adhesive performance

Make several $\varphi 50 \times 100$ gel samples from PEGDA200 and PEGAD400 gel base solutions of different concentrations, and test the adhesion of gel samples of different concentrations with a texture analyzer. The maximum adhesion of PEGDA200 and PEGAD400 of different concentrations is measured as shown in Figure 4.

As shown in Figure 4, the adhesive force of the composite gel before and after repair is the lowest without the addition of a crosslinking agent, 10.8 N and 12.9 N respectively. The adhesive force will increase when the crosslinking agents PEGDA200 and PEGAD400 are added, so the crosslinking agent can improve the adhesive force of the composite gel. For PEGDA200, the maximum adhesion forces are 17.9 N and 17.1 N at concentrations of 0.1% and 0.2%, respectively. For PEGDA400, the

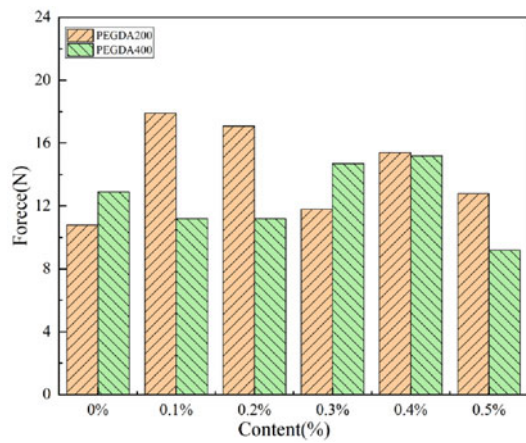


Figure 4. Comparison of adhesion performance changes between PEGDA200 and PEGDA400 at different concentrations

maximum adhesion forces are 14.7 N and 15.2 N at concentrations of 0.3% and 0.4%, respectively.

Compressive strength

Pour PEGDA200 and PEGAD400 gel base solutions of different concentrations into the steel pipes to make gel samples, press them with the pressure device, place the broken gel steel pipes in the oven after the gel breaks through and withstand the temperature of 70 °C/24 h, and conduct the compressive strength test after the gel is repaired. The compressive strength of PEGDA200 and PEGAD400 at different concentrations was measured and shown in Figures 5 and 6.

As shown in Figure 5, it can be seen that the self-repairing gel has good pressure resistance without the addition of crosslinking agents PEGDA200 and PEGAD400,

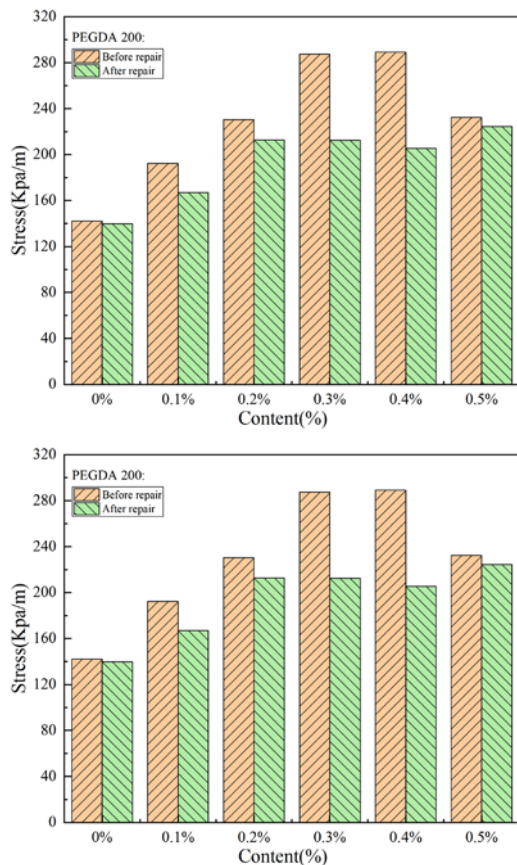


Figure 5. Test of compressive strength of PEGDA400 at different concentrations

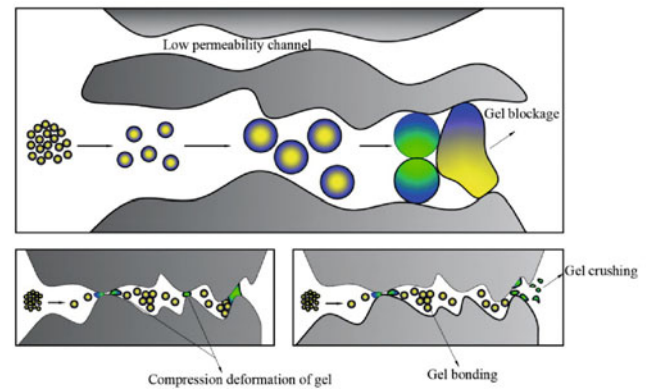


Figure 6. Deformation and Crushing Process of Self-healing gel Temporary Plugging Agent

and the pressure resistance after repair is about 98.3% and 94.5% of that before repair, respectively. When the content of self-repairing gel added with PEGDA200 is 0.2% and 0.5%, the self-repairing gel has good pressure resistance, 92.4% and 96.8% of that before repair, respectively. When the content of the self-repairing gel added with PEGDA400 was 0.5%, the self-repairing gel had the best repair effect, which was 106% of that before repair. As shown in Figure 6, the action process of self-healing gel temporary plugging agent includes compression deformation, bonding, crushing and other processes.

CONCLUSION

1) The addition of PEGDA200 and PEGDA400 to self-repairing gel can significantly improve the performance of composite gel, especially for the compression and adhesion properties of gel.

2) When the content of PEGDA200 and PEGDA400 in the gel is 0.2% or 0.5%, the rheological properties, compression properties, adhesion properties, and compressive strength of the self-healing composite gel are the best.

3) The action process of a self-healing gel temporary plugging agent includes compression deformation, bonding, crushing, and other processes.

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