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EXPERIMENTAL STUDY ON THE FLOWBACK OF A CARBOXYMETHYL HYDROXYPROPYL GUAR GUM FRACTURING FLUID WITH GOOD TEMPERATURE RESISTANCE

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For unconventional oil and gas reservoirs such as shale oil and gas as well as tight oil and gas, hydraulic fracturing generally enhances oil recovery. However, the flowback rate of the residual fracturing fluid is low. The residual fracturing fluid in the fracture or the rock matrix can reduce relative permeability of oil and gas, and the production rate will decrease. Therefore, it is necessary to study the factors that affect the flowback rate of the fracturing fluid. Most previous studies used the slot model, and viscous and capillary forces explain stable discharge in porous media. The conclusions were only a primarily qualitative analysis. The factors from experimental studies were not comprehensive, and they did not consider the influence of gravity. There are few studies on unstable drainage in porous media under different displacement directions. This paper presents a carboxymethyl hydroxypropyl guar gum fracturing fluid with good temperature resistance, and a fracturing fluid flowback experiment is carried on. The effects of the displacement direction, injection pressure, interfacial tension, fracturing fluid viscosity, and proppant wettability on the flowback rate are analyzed. The research results can provide formulation of the on-site construction scheme.

Keywords: fracturing fluid, backflow rate, injection pressure, displacement direction, proppant wettability

1. Introduction

For shale and tight oil and gas reservoirs, the hydraulic fracturing technology can improve the recovery. Its principle is to inject tens of thousands of cubic meters of a water-based fracturing fluid into the formation to support the layer, and it needs several tons of a fracture proppant at the same time. After the fracturing operation, it is necessary to close the well for a while, and then the well is opened to flowback the fracturing fluid (Fu *et al.*, 2022). The field data show that the current fracturing fluid flowback rate is less than 30 % (Qiu *et al.* 2018). There are two primary purposes for improving the flowback rate of the fracturing fluid. The first is to minimize the formation damage. The fracturing fluid remaining in the fracture or rock matrix can reduce the oil and gas production rate by affecting the relative permeability. The second is to minimize the consumption of fresh water. The future fracturing operations can reuse the backflow fracturing fluid (Zhou *et al.*, 2022a,b; Reis *et al.*, 2022).

The guanidine gum fracturing fluid is the most commonly used water-based fluid. However, the problems caused by the guanidine gum fracturing fluid filtration can damage the reservoir in forn of clay expansion, water lock effect, wettability reversal and poor compatibility of formation fluids (Huang *et al.*, 2021). There are two reasons for the low flowback rate. One is that the fracturing fluid invades the rock matrix due to self-absorption, and the other is that the fracturing fluid stays in the proppant fracture due to the capillary effect, gravity effect and relative permeability effect. The results of the proppant filling by pipes showed that when a surfactant was added to the fracturing fluid, the flowback rate of the fracturing fluid was improved. The experimental study of Shahidzadeh showed that wettability can significantly affect the flowback of the fracturing fluid (Shahidzadeh-Bonn *et al.*, 2003). The fracturing fluid velocity distribution model in the fracture has been established during the flowback process. The calculation results showed that the flowback velocity of the fracturing fluid near the fracture opening is more significant. Wang conducted an experimental study oo the influence of the fracturing fluid flowback speed on the proppant flowback. Based on the fracture simulation of an experimental device, the fracturing flowback process was analyzed. The results showed that the optimal critical flow to control the proppant flowback was 300 mL/min.

The research on the fracturing fluid flowback has accumulated many achievements. Shao studied salinity of the fracturing fluid flowback from shale gas wells and its damage to reservoirs (Shao *et al.*, 2022). The result showed that salinity of the flowback fracturing fluid is much higher than that of the fracturing fluid. The calculation model of modified proppant sedimentation velocity obtained through experiments, the new calculation model of critical proppant regurgitation velocity, and the wellhead pressure calculation model to form a comprehensive flowback model of the fracturing fluid were integrated (Qu *et al.*, 2021). An optimization model of the fracturing fluid flowback was constructed. A laser macroscope, low-field nuclear magnetic resonance (NMR) instrument, and a permeability device were used to scan fracture surface morphology (Zhang *et al.*, 2021). Fracturing fluid retention and permeability regain experiments were conducted. The experimental results showed that the fracturing fluid present in branch fractures was divided into free movable water (FMW), restricted movable water (RMW) and retained water (RW). A novel nanoparticle-enhanced supramolecular fracturing fluid (NESF) was developed (Xu *et al.*, 2020). A series of experiments was conducted to characterize the molecular structure of HGS and evaluate the heat/shear resistance, rheological property, proppant suspension and transportation, formation damage and imbibition efficiency of NESF. Experiments were designed and conducted to reveal the mechanism of fracturing fluid flowback and water retention under the imbibition effect in tight sandstones (Zhang *et al.*, 2022). The results show that the flowback recovery in tight sandstones is much lower than that in unconsolidated sandstones due to differences in the pore structure. The Lower Silurian Longmaxi shale samples and the backflow fracturing fluid in the Changning Block of the Sichuan Basin were selected to investigate the damage mechanism of the retained fracturing fluid to fractures in shale gas reservoirs (You *et al.*, 2019).

Most of the previous experimental studies used the slot model (Liu *et al.*, 2020; Huang *et al.*, 2019). Based on the viscous and capillary forces, it explained stable discharge in porous media, and the conclusions were only a primarily qualitative analysis (Zhou *et al.*, 2022a,b; Ju *et al.*, 2022; Dong *et al.*, 2019). Experimental studies have not considered the influence of gravity, and there are few studies on the unstable drainage in the porous media under different displacement directions (Guo *et al.*, 2022; Cheng *et al.*, 2022; Yang *et al.*, 2019). Many factors affect the flowback rate of the fracturing fluid, including the displacement direction, injection pressure, interfacial tension, fracturing fluid viscosity and proppant wettability (Fu *et al.*, 2020). The influence law of various factors can provide an experimental and theoretical basis for the fracturing fluid flowback.

2. Optimization of fracturing fluid components

2.1. Thickener optimization

The etherification of guar gum produces carboxymethyl hydroxypropyl guar gum. Thermogravimetric analysis reveals a low weight loss of unmodified guar gum and carboxymethyl hydroxypropyl guar gum in the low-temperature region. When temperature rises above 200°C, both unmodified guar gum and carboxymethyl hydroxypropyl guar gum under go a prominent weight loss process, and the speed is fast. When temperature rises above 400°C, carboxymethyl hydroxypropyl guar gum weight loss tends to be stable, and the weight loss in the high-temperature area is lower than that of unmodified guar gum. It indicates that the etherified guar gum has high thermal stability.

Table 1 shows the comparison data of carboxymethyl hydroxypropyl guar gum and unmodified guar gum. The carboxymethyl hydroxypropyl guar gum has better water solubility and temperature resistance and a smaller water-insoluble content. Due to the surface modification, the number of hydroxyl groups on the surface of guar gum reduced, and the side chain increased. The intermolecular force decreased, so the dissolution rate and ability were enhanced.

Table 1. Parameter comparison of different types of guar gum

Guar gum type	Dissolution time [s]	Stable viscosity [mPa·s]	Water insoluble content [%]
Guar gum	52	60	22
Low molecular weight guar gum	45	65	12
Ultra high temperature guar gum GHPG	40	72	7
Carboxymethyl hydroxypropyl guar gum	25	75	5
Modified crosslinked guar gum MGG	28	70	10
Hydrophobically modified guar gum	30	70	12

2.2. Optimization of the crosslinking agent

An organic boron zirconium composite crosslinker was synthesized by using inorganic zirconium, boric acid, polyol and triethanolamine. The polyol and water evenly were mixed in a flask with three necks. Inorganic zirconium was added to the mixed solution of polyol and water, and stirring was continued until the inorganic zirconium wholly dissolved. Then boric acid and triethanolamine were added. The flask was connected to the condenser and a specific temperature for continuous reaction was maintained. The organic boron zirconium crosslinking agent was obtained.

The addition amount of various materials and reaction conditions were optimized. Finally, the optimal reaction conditions were determined as follows: the proportion of inorganic zirconium and boric acid was 1:15, the proportion of polyol and water was 1:2.5, and the total amount of inorganic zirconium and boric acid accounted for 25% of the solvent, and triethanolamine accounted for 4.5% of the total solvent.

The fracturing fluid prepared was a 0.5% carboxymethyl hydroxypropyl guanidine gel. It was added different cross-linking agents to evaluate rheological properties. The results showed that the residual viscosity of the organic boron zirconium fracturing fluid after shearing was more significant, and it was more resistant to high temperature than the ordinary organic zirconium crosslinking agent.

Table 2 shows a comparison between the high temperature resistant organic boron zirconium crosslinking agent and the conventional crosslinking agent. The addition amount of the organic boron zirconium crosslinking agent was smaller than the conventional crosslinking agent. With the same addition, the delayed crosslinking performance was better. The residue content of the gel breaker of the organic boron zirconium crosslinker was the smallest, so the damage to the reservoir was the smallest as well. With an increase in the amount of the crosslinking agent, the crosslinking time decreased, and the temperature resistance increased. When the dosage of the crosslinking agent exceeded 0.5%, the temperature resistance of the fracturing fluid gel was not improved, but the delayed crosslinking performance significantly decreased. Therefore, the best dosage of the organic boron zirconium crosslinking agent was 0.5%.

Table 2. Parameters comparison of common crosslinking agents

Type of crosslinking agent	Temperature resistance [°C]	Viscosity after 2h of shearing [mPa·s]	Reservoir damage
Boron chelate crosslinker	160	185	large
Organic zirconium crosslinker	180	176	small
Organic boron titanium composite crosslinker	150	65	small
Amine containing nano crosslinking agent	130	150	small

2.3. Optimization of the temperature stabilizer

In order to improve the temperature resistance of the guar gum fracturing fluid, a temperature stabilizer HTR-1 was developed. The base solution was prepared with a 0.5% carboxymethyl hydroxypropyl guar gum. A 0.5% organic boron zirconium crosslinking agent was added. Then different concentrations of temperature stabilizers were added to test the rheological properties.

Table 3 shows different temperature stabilizers effects on the fracturing fluids performance. Temperature stabilizers can increase the temperature resistance of the guar gum fracturing fluid, such as triethanolamine and HTR-1. When the dosage of temperature stabilizer changed from 0.6% to 0.8%, the temperature resistance performance was improved. When the dosage of temperature stabilizer increased to 1.0%, the improvement of temperature resistance was not noticeable. Therefore, the preferred addition amount of temperature stabilizer was 1%.

Table 3. Temperature resistance of different temperature stabilizers

Type of temperature stabilizers	Temperature resistance [°C]	Concentrations [%]
JPG	135	0.8
JPG	138	0.9
JPG	143	1.0
JPG	145	1.1
HTR-1	142	0.8
HTR-1	146	0.9
HTR-1	151	1.0
HTR-1	152	1.1

2.4. Formula of guar gum fracturing fluid

Based on the optimization of fracturing fluid formula, a high temperature resistant fracturing fluid system formula was determined: 0.5% carboxymethyl hydroxypropyl guar gum + 0.5% organic boron zirconium crosslinker + 1% HTR-1 temperature stabilizer + 0.1% organic compound bactericide + 0.2% cationic quaternary ammonium salt clay stabilizer + 0.05% potassium permanganate breaker. Laboratory experiments showed that the additives had good compatibility and no flocculation or sedimentation.

3. The flowback experiment of the guanidine gel fracturing fluid

3.1. The experimental device

Figure 1 shows the experimental device for simulating the fracturing fluid flowback in fractures. The experimental container was composed of two transparent plates made of plexiglass. The gap between the two plates was filled with a proppant. Three injection ports and one flow outlet were set. The size of the vessel $L \times H \times W$ was $500\,\mathrm{mm}\times500\,\mathrm{mm}\times10\,\mathrm{mm}$.

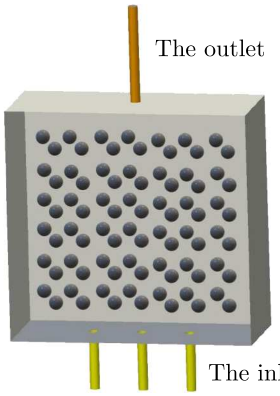

Fig. 1. Experimental container

Before the experiment, the container injected the fluid with a plunger pump, and the fluid was evenly distributed into the proppant. The nitrogen gas source was used as the driving force of displacement, and the gas source pressure simulated the injection pressure. The displacement direction indicated the influence of gravity on the flowback rate, and it was divided into three cases: the same as the gravity direction, the opposite to the gravity direction, and no gravity effect. During the experiment, it collected the discharged fluid at the outlet. An electronic balance to the computer was connected, and the flowback rate was calculated.

The experiment used two types of proppants: hydrophilic ceramsite with a particle size of 20/40 and oleophilic ceramsite with a particle size of 20/40. Three fluids were used in the experiment: distilled water, sodium dodecylbenzene sulfonate solution and the guar gum fracturing fluid. It used a sodium dodecylbenzene sulfonate solution to study the effect of surface tension on the flowback rate. The guar gum fracturing fluid was implemented to study the effect of fluid viscosity on the flowback rate. DVNext Well-Brookfield rheometer measured the fluid viscosity at different shear rates ($0.01\,\mathrm{s}^{-1}$ to $200\,\mathrm{s}^{-1}$).

3.2. Experimental steps

Taking into account the influence of displacement direction as an example, the specific experimental steps are as follows.

(1) Fill the space between two glass plates with ceramsite. A plunger pump injects the fluid from the bottom of the container. The container is fixed in a vertical position with a bracket.
(2) Adjust the nitrogen supply pressure to control the flowrate of the liquid. Inject nitrogen with a specific pressure through three injection ports after stabilization. Meantime, collect the fluid at the outlet. The computer automatically records the data and calculates the flowback rate.
(3) Nitrogen is injected from the top of the container to simulate displacement in the same direction as gravity. Nitrogen is injected from the bottom of the container to simulate

displacement in the direction opposite to gravity. The container is horizontal, and the injected nitrogen is to simulate the non-gravity effect.

(4) Turn on the fluorescent lamp at the back of the container, and observe the backflow of the fluid.

4. Analysis of influencing factors of the fracturing fluid flowback rate

Experiments were carried on by controlling a single variable to study the effects of displacement direction, injection pressure, interfacial tension, viscosity and proppant wettability on the fracturing fluid flowback rate.

4.1. The influence of the displacement direction

The pressure of nitrogen injection was maintained at 0.5 MPa. The container was filled hydrophilic ceramsite with a particle size of 20/40 and the distilled water. The influence law for displacement direction was analyzed. Figure 2 shows the results.

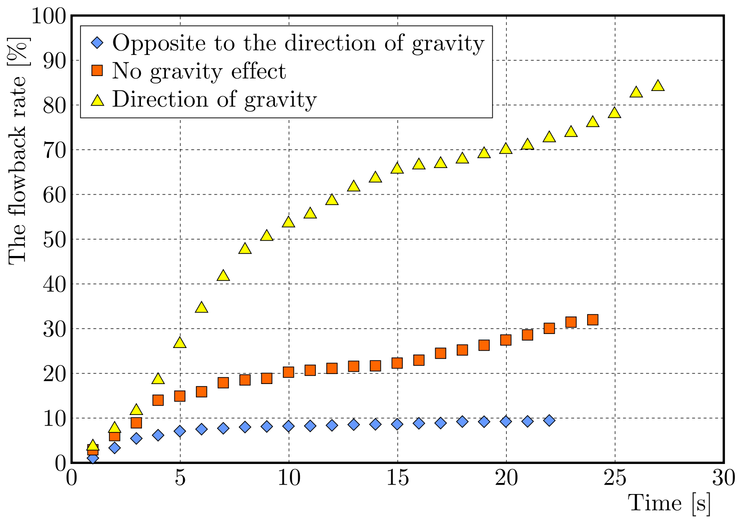

Fig. 2. Influence of the displacement direction on the backflow rate of fluid

When the displacement direction was opposite to the gravity direction, the final flowback rate of the fracturing fluid was very small (only 9.4%). When the displacement direction was the same as the gravity direction, the final flowback rate of the fracturing fluid was much more significant (84.6%). When there was no gravity effect, the final flowback rate of was 32.1%. It was larger than that in the opposite direction of gravity but far smaller than in the experimental results in the same direction of gravity.

Fingering occurred when the displacement direction was opposite to the gravity direction. With an extension of displacement time, the fingering phenomenon became more apparent, gradually forming a smooth flow path. Most gases flowed through the pointed preferred path and gathered at the container top without displacing the fracturing fluid in the proppant. Fingering was highly detrimental to the flowback of the fracturing fluid. The gas swept only a tiny part of the porous medium, and the area sweep coefficient was small. Even after a long time, a large amount of the fracturing fluid was still left in the formation. Therefore, fingering can be avoided by adjusting the performance of the fracturing fluid.

4.2. The influence of injection pressure

The container was filled with hydrophilic ceramsite with a particle size of 20/40 and distilled water. The nitrogen was injected in the opposite direction of gravity, and the control pressure was 0.1 MPa, 0.3 MPa, 0.5 MPa, 0.7 MPa, and 0.9 MPa. The law of the influence of injection pressure was studied (Fig. 3). Nitrogen was injected in the same direction as gravity without controlling the injection pressure. The influence of pressure stability on the flowback rate was studied (Fig. 4).

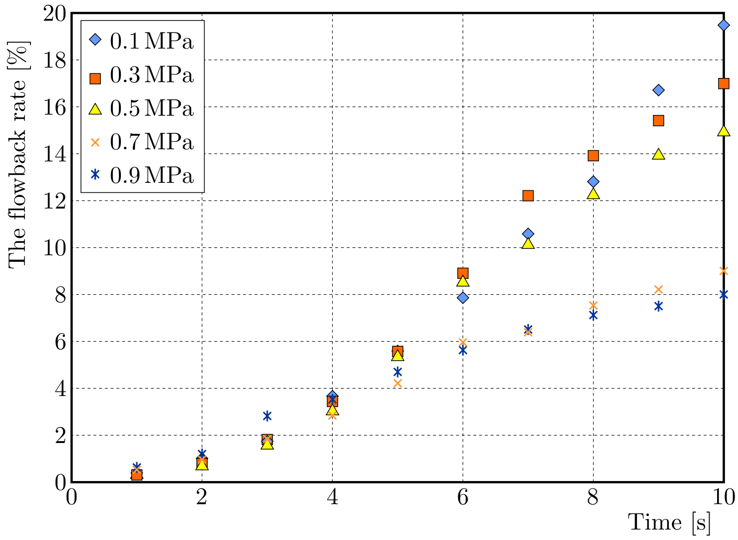

Fig. 3. Influence of injection pressure on the backflow rate

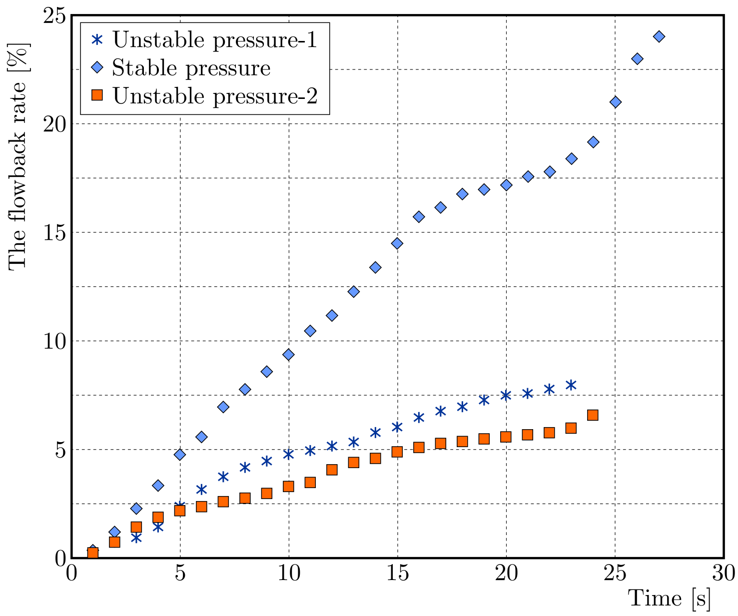

Fig. 4. The influence of pressure stability on the backflow rate

The slope of the flowback curve indicated the flowback rate. At the initial stage of flowback, the higher was the injection pressure, the higher was the flowback rate. The flowback rate corresponding to high injection pressure decreased with time. The flowback rate decreased. The final flowback rate decreased with an increase of injection pressure. Because of high injection pressure, the displacement power was strong. The flowback rate was fast at the initial stage, but it was easy to form a fingering phenomenon. It led to a decline of the flowback rate at a later stage. When the pressure was 0.5 MPa, only one fingering phenomenon breaking through the outlet end was observed. When the pressure was 0.7 MPa and 0.9 MPa, one could observe many fingering phenomena, and prominent branches formed at the bottom of the container. The stable injection pressure was conducive to the backflow of the fracturing fluid. Because the fluctuation of injection pressure would lead to a change in the flowback system, the fracturing fluid was unstable. The flowback rate was low. It would lead to the formation of sand spitting during the actual flowback process.

After fracturing construction, it should control the blowout by the nozzle. One method is to control the injection pressure by changing nozzles with different diameters, which makes the initial flowback rate reduce to prevent fingering. The second method is to control the stability of injection pressure, which shall prevent rapid depletion of formation energy. It will ensure the smooth flowback, so that the more fracturing fluid can flow back.

4.3. The influence of interfacial tension

The nitrogen was injected in the opposite direction of gravity. The pressure of nitrogen injection was maintained at 0.5 MPa. The container was filled with hydrophilic ceramsite with a particle size of 20/40 and distilled water, 0.5%, 1.0%, 1.5% and 2.0% sodium dodecylbenzene sulfonate solution, respectively. The effect of interfacial tension was studied (Fig. 5).

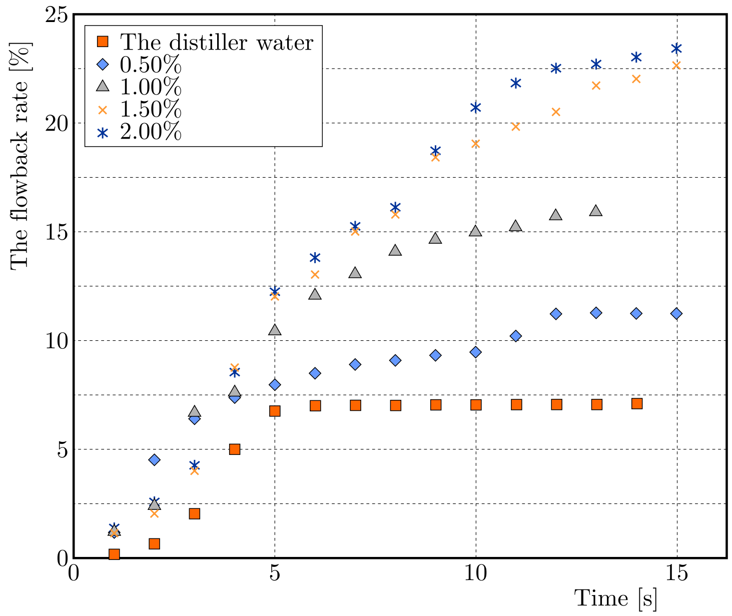

Fig. 5. The effect of interfacial tension on the backflow rate of the fluid

The final flowback rate of sodium dodecylbenzene sulfonate solution was significantly more extensive than that of distilled water. The surfactants such as sodium dodecylbenzene sulfonate can reduce the interfacial tension of the liquid. The liquid was evenly dispersed in the pores of the proppant, and it could form an integral displacement effect during the displacement process. The area displacement efficiency was significant, so the flowback rate was more significant. With the increased mass fraction of sodium dodecylbenzene sulfonate, the liquid interfacial tension decreased gradually. The flowback rate increased gradually, but the increasing trend decreased gradually. The difference between the final flowback rate of sodium dodecylbenzene sulfonate solution with a mass fraction of 2.0% and that with a mass fraction of 1.5% was slight. Therefore, there was an optimal surfactant concentration for the fracturing fluid system, so the final flowback rate reached the optimal value. This study revealed that the optimal mass fraction of sodium dodecylbenzene sulfonate was 1.5%.

4.4. The influence of viscosity

The nitrogen was injected in the opposite direction of gravity. The pressure of nitrogen injection was at 0.5 MPa. The container was filled with hydrophilic ceramsite with a particle size of 20/40 and distilled water, carboxymethyl hydroxypropyl guar gum with mass fractions of 0.01%, 0.02%, 0.03%, and 0.04%, respectively. The effect of viscosity was studied (Fig. 6).

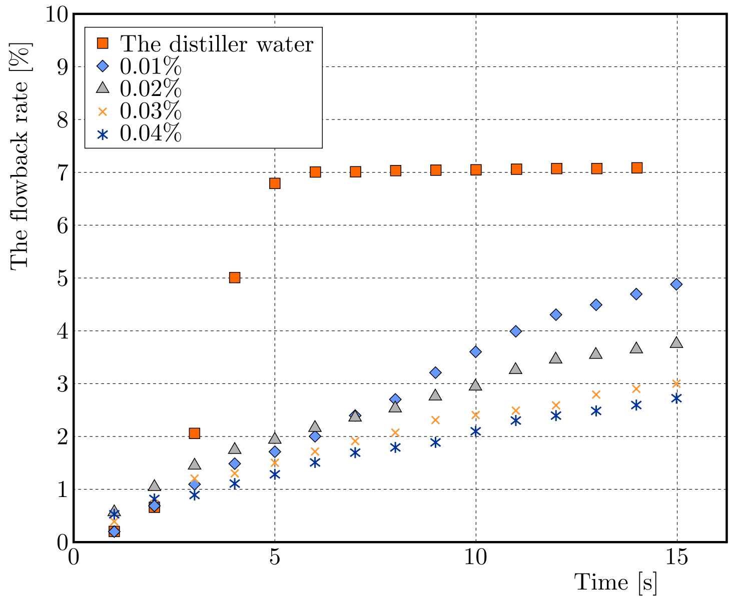

Fig. 6. The effect of viscosity on the backflow rate of the fluid

Compared with the distilled water and carboxymethyl hydroxypropyl guar gum, the final flowback rate of the carboxymethyl hydroxypropyl guar gum was small. With an increase of the guar gum mass fraction, the final flowback rate gradually decreased. Because viscosity of the carboxymethyl hydroxypropyl guar gum increased with the increase of mass fraction, pressure of 1.0 MPa was not enough to overcome viscosity. Therefore, the flowback rate of the carboxymethyl hydroxypropyl guar gum significantly decreased. For the fracturing fluid system, as long as the fracturing fluid can meet sand carrying requirements, viscosity of the fracturing fluid should be reduced as far as possible.

4.5. The influence of proppant wettability

The nitrogen was injected in the opposite direction of gravity. The pressure of nitrogen injection was at 0.5 MPa. The container was filled with hydrophilic ceramsite and oleophilic ceramsite with a particle size of 20/40 and distilled water. The influence of proppant wettability was studied (Fig. 7).

The final flowback rate of the pressure fluid filled with lipophilic ceramsite was nearly twice higher than that filled with hydrophilic ceramsite. It showed that the lipophilic proppant was conducive to the flowback of the fracturing fluid. When the container was filled with lipophilic ceramsite, continuous gas pushed the fracturing fluid as a whole, and a fingered gas channel formed in the hydrophilic ceramsite. Field application usually incorporates a water-based fracturing fluid system. The lipophilic proppant was hydrophobic, and the fracturing fluid was easier to be displaced. Its sweep coefficient was more significant than that of porous media filled with

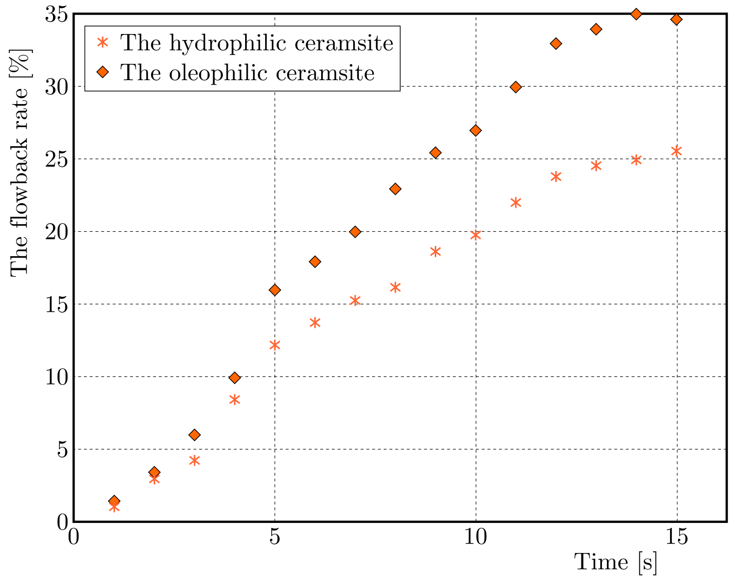

Fig. 7. The influence of wettability on the backflow rate

the hydrophilic proppant. For oil production after fracturing, the lipophilic proppant was adverse to production of crude oil. More works related to hydraulic fracturing should be done as suggested by several studies (Al-Shalabi and Ghosh, 2018; Al-Shalabi *et al.*, 2022; Khurshid *et al.*, 2022; Adegbite and Al-Shalabi, 2020). Therefore, it will be an essential research direction to study the proppant with wetting self-reversal ability. The proppant showed lipophilicity during the fracturing fluid flowback, and the proppant automatically reversed to hydrophilicity during oil production.

5. Conclusion

- When the displacement direction was the same as the gravity direction, the final flowback rate of the fracturing fluid was significant. Fingering occurred when the displacement direction was opposite to the gravity direction. Therefore, fingering can be avoided by adjusting performance of the fracturing fluid.
- The final flowback rate of the fracturing fluid decreased with an increase of injection pressure. Stable injection pressure is more conducive to the backflow of the fracturing fluid. Therefore, it is necessary to use a nozzle with an appropriate diameter to control the backflow of the fracturing fluid.
- With a decrease of interfacial tension, the final flowback rate of fracturing fluid increased, but the upward trend gradually slowed down. For oil production after fracturing, the lipophilic proppant was adverse to production of crude oil. Therefore, it will be an essential research direction to study the proppant with wetting self-reversal ability.

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