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Experimental Study on Plugging Behavior of Degradable Fibers and Particulates within Acid-etched Fracture

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Abstract: As proven from field practices in North America and Northwest China, temporary plugging and diverting acid fracturing is an indispensable technology to enhance stimulation effect and hydrocarbon production of complex carbonate reservoirs. It's well-known that the key to the success of this technology lies in creating a temporary plugging within the previously created fractures. Consequently, many scholars have conducted laboratory experiments to investigate the plugging behavior of fibers and particulates. However, the current devices nearly have certain limitations in simulating temporary plugging experiments. Aiming at this problem, this paper introduced the fracture temporary plugging evaluation system with large fracture size, and high-pressure resistance, which can meet the requirement of temporary plugging experiment. In addition, 3D printing technology was used to reproduce the roughness of acid-etched fracture surface, improving the experimental accuracy. Based on the device, a series of experiments were performed to study the effect of carrier fluids type, the injection rate, the fracture width, and the fracture morphology on plugging behavior of fibers and particulates. Experimental results show that the plugging effect of HPG fracturing fluid is better than that of slick water. Moreover, the effect of temporary plugging deteriorates with the decrease of injection rate, which can be attributed to the fact that high injection rate increases the probability of bridging and plugging. When it comes to the fracture width, only fibers have favorable plugging effect under the condition of 2 mm fracture width. However, a single type of temporary plugging agent (only fibers or particulates) cannot achieve effective plugging under the condition of 4 mm fracture width. The combination of fibers and particulates can obtain favorable plugging effect. When the fracture width increases to 6 mm, it's difficult to obtain favorable plugging effect if the diameter of particulates is less than 50% of fracture width. Hence, it is recommended to add big particulates whose diameters are at least 50% of the fracture width to improve the plugging effect. Moreover, the fracture surface morphology affects the formation time of temporary plugging, but does not affect whether temporary plugging is formed or not. This study deepens the understanding the plugging behavior of fibers and particulates within acid-etched fracture and provides fundamental for field treatment design.

Key Words: temporary plugging; degradable fibers and particulates; diverting acid fracturing; 3D printing;

1 Introduction

According to the *Annual Energy Outlook 2018* released by the U.S. Energy Information Administration (EIA), more than 60% of the world's oil and 40% of the world's gas reserves are held in carbonate reservoirs (EIA, 2018). Unfortunately, they are becoming more and more expensive and difficult to develop. With the deepening of carbonate reservoirs development and ever-increasing demand for fuels, operators are forced to develop the reservoirs with poor physical properties, such as severe heterogeneity, high temperature and high pressure, and developed natural fractures (Chang, 2017). It is well-known that acid fracturing is an indispensable technology to develop carbonate reservoirs efficiently and economically. However, the conventional acid fracturing technology may not acquire favorable stimulation effect as results of these poor properties (Zhang et al., 2018). In light of this, temporary plugging and diverting acid fracturing technology was proposed to improve the stimulation effect. In general, this technology can be divided into two types, which are mechanical diversion and chemical diversion (Gutierrez et al., 2015). Traditional mechanical diverters mainly include drillable bridge plug, straddle packers and liners while the chemical diverters include temporary plugging agents (particulates and fibers), foam, viscous fluids, etc. Compared with mechanical

diversion, which is time-consuming, high cost and risky, chemical methods have become an effective alternative for safe, efficient and economical advantages.

Among various existing chemical methods, temporary plugging and diverting fracturing by using degradable fibers and particulates is the most promising (Nasr-El-Din et al., 2007; Allison et al., 2011). This technology refers to pumping fibers and particulates during the treatment to create a temporary bridge within the active fracture networks, redirecting the subsequent treatment fluids toward under-stimulated area, thus enhancing the fracture complexity and the stimulated volume (Potapenko et al., 2009). After the treatment, the fibers and particulates which are made from copolymer of lactic acid with glycolic acid, can completely degrade under the effect of formation temperature and flow back without any damage to formation (Zhou et al., 2009; Shi et al., 2015). Moreover, it's noteworthy that the key to the success of this technology lies in the efficient plugging of the previously created fractures. Therefore, a large number of scholars have conducted laboratory experiments to investigate the plugging behavior of fibers and particulates.

Table 1 shows the summary of previous works on plugging behavior of fibers and particulates. Petapenko et al. (2009) conducted the pioneering experiments to study the plugging behavior of fibers and particulates, and found that the plugging effect was greatly affected by rheology of the base fluid, concentration of fibers and particulates, fracture size and geometry, and injection rate. The fly in the ointment is that the pressure bearing capacity of the slot bridging apparatus is too low (Petapenko et al., 2009). Based on a slot with the length of 46 mm and the width of 1-5 mm, Kefi et al. (2010) evaluated the plugging effect of fibers and particulates. The experimental results show the lower concentration limit of fibers and particulates to form plugging (Kefi et al., 2010). Cohen et al. (2010) used epoxy resin groove to simulate the fracture and conducted the fiber temporary plugging experiments. Results showed that the permeability of fiber filter cake and the spurt loss were two key parameters to control acid diversion. However, the surface of the epoxy resin groove was smooth without considering the effect of fracture surface morphology on temporary plugging (Cohen et al., 2010). Zhou et al. (2014) investigated the filtration characteristic of fibers and the length of filter cake under different fracture widths, where split core sample was used to simulate the fracture. However, it's difficult to ensure the consistency of fracture morphology due to the randomness of artificial splitting (Zhou et al., 2014). Kang et al. (2015) simulated the roughness of fracture by grooving on the surface of steel core sample and studied the effect of different temporary plugging materials, and different concentration and proportion of them on temporary plugging. However, their device simplifies the fracture too much to reflect the roughness of the real fracture surface. In addition, the device is also not easy to adjust the fracture width (Kang et al., 2015). Cortez et al. (2015) developed a dynamic flow diverter testing apparatus, and evaluated the effects of injection rate, temperature, diversion agent concentration, and pressure on temporary plugging. This research provided an insight into understanding factors that affect diversion and data that can help optimize future diversion treatments to some degree. Similarly, the roughness of fracture surface was still not considered (Cortez et al., 2015). Baker Hughes proposed the concept of far-field diversion in acid fracturing, which refers to pumping fibers and particulates to create a temporary bridge at the fracture tip. To support and promote this concept, a series of studies including bridge plugging test, diversion efficiency, carrier fluid properties, and concentration of diversion agents have been conducted. In their experiments, a steel disk with a slot was used to simulate fracture, which is too simple to reflect the real fracture morphology (Williams et al., 2016; Gomaa et al., 2016). Ray et al. (2017) developed a visual experimental device to simulate the fracture surface by 3D printing technology and investigated the role of particulate transport, bridging, settling, and resuspension within fracture. Compared with previous experimental setups, this device made progress to some degree by reproducing the real fracture surface. However, due to the poor property of high-pressure resistance and low injection rate, the device still cannot stimulate temporary plugging diversion very well (Ray et al., 2017). Recently, Yang et al. (2019) developed a visualized slot-flow system with high speed camera to observe the dynamic plugging performance of fibers and particulates in a mimicked fracture. Since the fracture morphology and high-pressure resistance were not taken into consideration, this setup is also not suitable for simulating temporary

88 plugging experiments (Yang et al., 2019).

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Table 1 The summary of previous work on plugging behavior of fibers and particulates

Reference	Fracture model (material & size)	Fracture roughness	Plugging pressure	Setup structure
Petapenko et al. 2009	Several slot geometries; Fracture width: 2-6 mm	Smooth	200 psi
Kefi et al. 2010	Metal slot Fracture width: 1-5 mm	Smooth	500 psi
Cohen et al. 2010	Epoxy or plastic groove Fracture width: 2 mm	Smooth	65 psi
Zhou et al. 2014	Split core sample Fracture width: 0.5-2 mm	Real fracture surface	1500 psi
Kang et al. 2015	Steel core samples Fracture width: 2 mm	Grooving on steel core sample surface	2000 psi
Cortez et al. 2015	stainless steel cells	/	1000 psi

Reference	Fracture model (material & size)	Fracture roughness	Plugging pressure	Setup structure
Williams et al. 2016 & Gomaa et al. 2016	Steel disk with a slot Fracture width: 1-5 mm	Smooth	1000 psi
Ray et al. 2017	Channel flow device Fracture width: 4 mm	Channels are printed with one wall only	Cannot bear pressure
Yang et al. 2019	visualized slot-flow experimental system Fracture width: 3 or 5 mm	Transparent plexiglass plates/smooth	15 psi

According to the above literature review, it can be concluded that the current devices have certain limitations in investigating plugging behavior of fibers and particulates. Therefore, this paper introduced the fracture temporary plugging evaluation system, which utilized the data acquisition system and the press of fracture conductivity evaluation system. It also manufactured the matching conductivity cell to investigate the temporary plugging behavior of fibers and particulates. This modified device with large fracture size, and high-pressure resistance can meet the requirement of temporary plugging experiment. In addition, 3D printing technology was used to reproduce the roughness of fracture surface, improving the experimental accuracy. Based on the device, a series of experiments were performed to study the effect of carrier fluids type, the injection rate, the fracture width, and the fracture morphology on temporary plugging of fibers and particulates. This study deepens the understanding on the plugging behavior of fibers and particulates within acid-etched fracture and provides fundamental for field treatment design.

2 Laboratory experiments

Based on the reproduced acid-etched fracture model, a series of experiments were conducted to study the temporary plugging behavior of fibers and particulates. However, in order to obtain the 3D printing model of acid-etched fracture, there are some preparatory works to be done. **Fig. 1** shows the flow diagram of the preparatory work. First, the conventional hydraulic fracturing experiment was conducted to create hydraulic fractures, in which natural carbonate outcrop cubic block with the dimension of 300 mm × 300 mm × 300 mm was used. Second, wire-electrode cutting instrument was used to produce core slabs with these fracture surface. Then, based on the acid fracturing conductivity evaluation system, the acid-etched experiment was performed on core slabs to obtain

acid-etched fracture surface. The acid type, optimal acid-rock contact time, and acid injection rate herein were gelled acid, 60 min, and 60 ml/min, respectively (Zhang et al., 2018). Next, micro-nano laser scanner was applied to scan the acid-etched fracture surface. Finally, based on the above scanning data, the 3D printing model was printed by a 3D printer.

Fig.1 The flow diagram of preparatory work for making 3D printing model

2.1 Experimental materials

2.1.1 3D printing model

Based on digital model files, 3D printing is a technique for constructing objects by layer-by-layer printing using adhesive materials such as powder-like metal or plastic. It has been widely used in many industries such as automatic, aerospace, construction, medical, geographic information systems (Han et al., 2016). Generally, the precision of 3D printing can reach as high as 0.006 mm. In this paper, the printing precision of the acid-etched fracture is 0.05 mm so that the real acid-etched fracture morphology can be reproduced. **Fig. 2** shows the 3D printing model with dimensions of 180 mm × 45 mm used for temporary plugging experiments. The material of this model is photosensitive resin. Its hardness (shoreD), tensile strength, and bending strength is 86, 40 MPa, and 70 MPa, respectively. Hence, the model will not deform during the temporary plugging experiments to ensure the consistency of the experiments. In order to avoid bridge temporary plugging of diversion agents at the fracture entrance as results of the variation of flow radius, the wedge-shaped entrance was introduced to the temporary plugging experiments. In addition, the acid-etched fracture with different width can be simulated by adding steel gasket.

A: wedge-shaped entrance; B: steel gasket

Fig.2 3D printing model of acid-etched fracture in the plugging experiments

2.1.2 Experimental fluid

1) Gelled acid

This acid was used in the acid-etched experiments, which refer to in the part of 2 (preparatory work for making 3D printing model). It was applied to etch core slabs with fracture surface to provide acid-etched fracture for scanning and 3D printing. In this experiment, the acid-rock contact time, acid volume, and acid injection rate were 60 min, 3600 ml, and 60 ml/min, respectively. Furthermore, the formula of acid is based on the field application, and it was composed of 1 wt% BZJ thickener, 0.5 wt% MJZ cleanup additive, 0.5 wt% BF529 clay stabilizer, 1 wt% BZH high temperature corrosion inhibitor, and 1 wt% BZJ ferric ion stabilizer. Its physicochemical properties, such as rheological property, reaction rate, have been reported in our previous study (Zhang et al., 2018).

2) Carrier fluids

In this study, hydroxypropyl guar gum (HPG) fracturing fluid and slickwater were selected as carrier fluids for temporary plugging experiments. Similarly, the formula of fracturing fluid is also based on the field application, and it was consisted of 0.5 wt.% hydroxypropyl guar, 0.06 wt.% citric acid, 1 wt.% flowback surfactant, 0.1 wt.% bactericide, and traces of other additives. Slick water composed of 0.1 wt% DR800 as the friction reducer (an anionic polyacrylamide), 0.5 wt% COP-2 as the clay stabilizer, and 0.5 wt% TGF-1 as the flowback surfactant (Liang et al., 2017; Zhang et al., 2019). The apparent viscosity of HPG fracturing fluid and slickwater are about 130 mPa s and 6 mPa s, measured by a rotational viscometer.

2.1.3 Diversion agent

Fig. 3 presents fibers and particulates used in the experiments. The length and diameter of fibers are approximately 6 mm and 10 μm , respectively. There are three kinds of particulates with diameters of 1 mm, 2 mm, and 3 mm, respectively. In addition, the fiber and particulate are both made of copolymer of lactic acid with glycolic acid (Liang et al., 2018). The density of fiber and particulate is 1.24 g/cm³ and 1.15 g/cm³, respectively.

Fig.3 Diversion agents in the experiments

2.2 Experimental setup and procedure

As shown in **Fig. 4**, the fracture temporary plugging evaluation system was used to investigate the plugging mechanism of diversion agents. This system consists of a displacement pump, a confining pressure pump, a pressure gauge, a modified conductivity cell, a press, a data acquisition system, and so on. The displacement and confining pressure pump both have a maximum capacity of 266 ml, a flow accuracy of 0.5% of the set point and a maximum flux of 107 ml/min. The pressure gauge with a calibration range of 0 to 35 MPa and a measurement accuracy of 0.06% of the range was used to monitor the pressure in the temporary plugging experiment. In order to avoid the diversion agent blocking the pipeline, the inner diameter of pipeline in the experiment was increased to 12.7 mm.

The experimental procedure is as follows:

1) Adjust the thickness of steel plate gasket and set the fracture width; then place the 3D printing model into the conductivity cell;

- 2) Place the conductivity cell on the press;
- 3) Prepare 4000 ml mixture of fracturing fluids and diversion agents and put it into an intermediate container; connect the pipeline and pump;
- 4) Load the confining pressure according to a set value (20 MPa) onto the model by the confining pressure pump;
- 5) Start the displacement pump to inject the mixture of fracturing fluids and diversion agents, and record the inlet pressure of the conductivity cell;
- 6) When the inlet pressure reaches a certain value (10 MPa) or the injection volume reaches a design value, stop pumping;
- 7) Unload the confining pressure and conductivity cell;
- 8) Open the 3D printing model, observe and take photos of the plugged zone.

1: displacement pump; 2: confining pressure pump; 3: fracture conductivity cell; 4-5: intermediate containers; 6-10: control valve; 11: press; 12: collector and balance; 13: pressure gauge; 14: data acquisition system

Fig.4 Schematic diagram and physical picture of temporary plugging evaluation system

2.3 Experimental schemes

In this study, 17 groups of experiments were performed to investigate the plugging behavior of combination of fibers and particulates. **Table 2** lists all experimental schemes to study the effect of carrier fluid type, injection rate, fracture width, and acid-etched fracture morphology on temporary plugging rules. In the field, slick water and HPG fracturing fluid are the most common carrier fluids. Hence, experimental group (1, 2) were performed to study the temporary plugging rules of slick water and HPG fracturing fluid, respectively. Furthermore, as injection rate is also an important factor affecting the temporary plugging effect, experimental groups (2-4) were conducted to investigate the influence of injection rate on temporary plugging effect. Experimental groups (2, 5-15) were conducted to investigate the influence of fracture width on temporary plugging effect. Experimental groups (2, 16, 17) were conducted to investigate the plugging behavior under different acid-etched patterns.

Tab.2 The scheme of temporary plugging experiments

No.	Carrier fluid	Fracture width mm	Injection rate ml/min	Fiber concentration wt%	1mm Particle concentration wt%	2mm Particle concentration wt%	3mm Particle concentration wt%	Note
1	Slick water	4	60	1.0	/	1.0	/	Carrier fluid type
2	Guar gel	4	60	1.0	/	1.0	/	
3	Guar gel	4	40	1.0	/	1.0	/	Injection rate
4	Guar gel	4	20	1.0	/	1.0	/	
5	Guar gel	2	60	1.0	/	/	/	Fracture

No.	Carrier fluid	Fracture width mm	Injection rate ml/min	Fiber concentration wt%	1mm Particle concentration wt%	2mm Particle concentration wt%	3mm Particle concentration wt%	Note
6	Guar gel	2	60	1.0	0.8	/	/	width
7	Guar gel	4	60	1.0	/	/	/	
8	Guar gel	4	60	1.2	/	/	/	
9	Guar gel	4	60	/	/	1.0	/	
10	Guar gel	4	60	1.0	/	0.8	/	
11	Guar gel	4	60	1.0	/	/	0.8	
12	Guar gel	6	60	1.0	/	1.2	/	
13	Guar gel	6	60	1.0	/	/	0.8	
14	Guar gel	6	60	1.0	/	/	1.0	
15	Guar gel	6	60	1.0	/	/	1.2	
16	Guar gel	4	60	1.0	/	1.0	/	3D model-2
17	Guar gel	4	60	1.0	/	1.0	/	3D model-3

Note: Experimental groups (1-15) used the same 3D printing model (Model-1). The remaining two groups of experiments (16, 17) used different 3D printing fracture models (Model-2 and Model-3).

3 Results and discussion

In this section, the effect of carrier fluids type, fracture width, injection rate, and fracture morphology on plugging behavior were systemically analyzed. The inlet pressure was monitored in real-time by the data acquire system during the experiment, which was taken as an important parameter to evaluate whether effective plugging was formed. In this paper, when the inlet pressure reaches 10 MPa, it's indicated that the temporary plugging zone can bear this pressure and avoid breakthrough. Therefore, it's considered that the fibers and particulates plug the fracture and the effective temporary plugging has been formed.

3.1 The effect of carrier fluids type

Slick water and HPG fracturing fluids, two of the most common carrier fluids, were used in the temporary plugging and diverting fracturing treatments to stimulate the conventional/unconventional reservoirs (Josifovic et al., 2016; Hammond et al., 2017). **Fig. 5** shows the relationship between inlet pressure with time and the picture of plugging zone during the temporary plugging experiments of slick water (Fig. 5a) and HPG fracturing fluid (Fig. 5b), respectively. As can be seen from Fig. 5(a), the inlet pressure presented a slow rising trend from the beginning to the end of the experiment. During the whole experiment, the maximum pressure was approximately 0.4 MPa. This phenomenon indicates that the diversion agents entering the fracture model increase the flow resistance. However, there is no effective plugging in the fracture model. As shown in the picture of plugging zone, it is obvious that only a few diversion agents with loosen structure distributed in the fracture model, which further proves this point. In addition, if the mixture of diversion agents and slick water is continuously displaced, the effective plugging zone may eventually be formed as the diversion agents accumulate in the fracture model.

As shown in Fig. 5(b), the variation of inlet pressure can be divided into three stages. In the first stage, the inlet pressure nearly kept stable with a small fluctuation from the beginning to 20 min. At the beginning of experiment, the mixture of diversion agents and slick water needs to be filled with the whole pipeline and fracture model. There are only a few diversion agents adhere to fracture surface. In the second stage (from 20 min to 40 min), the inlet pressure experienced a process of ascending and descending repeatedly. The reasons for this phenomenon are as follows. During this period, the diversion agents tried to form bridging plugging in the fracture model with the accumulation of diversion agents. However, the plugging zone was always broken. In the third stage (from 40 min to 58 min), the inlet

pressure gradually increased to 10 MPa, indicating that an effective plugging zone has been formed in the fracture. It should be noted that the pressure fluctuation during the rising stage indicates that the formation of plugging zone is a process of repeated plugging-breakthrough- plugging. Moreover, a mass of diversion agents was distributed in the fracture and a tight plugging zone can be founded near the entrance of the fracture, which further verifies the formation of the effective plugging zone.

Fig.5 The relationship between inlet pressure and time and the picture of plugging zone (No. 1 and No. 2)

Under the condition of laboratory experiments, the plugging effect of HPG fracturing fluid is better than that of slick water. The main reason for the different phenomenon can be contributed that the viscosity of fracturing fluid is higher than that of slick water. On the one hand, the diversion agents were difficult to settle in the intermediate containers as a result of high viscosity. On the other hand, the diversion agents may be easier to adhere to the fracture surface under the condition of high viscosity (Wang, 2017). However, in fact, the slick water also has been widely used in the field due to its characteristics of simple preparation, low cost, and low damage to formation (McClure, M., 2018; Thomas, L. et al., 2019; Mahta Vishkai et al., 2019). Hence, if slick water is used as a carrier fluid in the field, it's recommended to add proper thickener to improve its viscosity.

3.2 The effect of injection rate

Injection rate also has an important influence on temporary plugging effect. Three groups of experiments (No.2-No.4) were conducted to study the effect of injection rate. Based on the above study, the expected plugging effect can be obtained when the injection rate is 60 ml/min. **Fig. 6(a)** shows the variation curve of inlet pressure and the morphology of plugging zone at the injection rate of 40 ml/min. From the beginning to 20 min, the inlet pressure nearly kept stable with a small fluctuation. After that, it began to experience drastic fluctuations over and over again until the end of the experiment, demonstrating that the diversion agents (fibers and particulates) repeated the process of temporary plugging and breakthrough in the fracture. The maximum inlet pressure was approximately 1.8 MPa during the experiment, showing relatively poor plugging effect. In addition, although there were some diversion

agents evenly distributed within the fracture, no tight plugging zones similar to those in **Fig. 5(b)** were formed. Hence, the absence of an effective temporary plugging zone has been further proven. **Fig. 6(b)** shows the relationship between inlet pressure with time at the injection rate of 20 ml/min. It can be seen that the inlet pressure showed a tendency to rise slowly from the beginning to 30 min, and then experienced the processes of ascending and descending for many times. However, the frequency of bridging and plugging is much lower than the above case. Moreover, the maximum inlet pressure was about 1.3 MPa, indicating that there is no effective temporary plugging zone in the fracture. The picture of plugging zone morphology shows that only a few diversion agents with loosen structure distributed at the fracture model inlet and outlet, which further proves this point.

The experimental results illustrated that the effect of temporary plugging deteriorates with the decrease of injection rate. The main reason can be attributed to the fact that high injection rate increases the probability of bridging and plugging. On the one hand, there are more fibers and particulates flowing into the fracture model at the same time under the condition of high injection rate, which is conducive to the formation of bridging and plugging. On the other hand, the fibers are more prone to twine around each other at high injection rate, thus capturing the particulates to form tight plugging zones (Wang et al., 2015a, 2015b). Therefore, it is recommended to use high injection rate for construction on the premise of on-site conditions.

Fig.6 The inlet pressure curve and the morphology of plugging zone (No. 3 and No. 4)

3.3 The effect of fracture width

Generally, the designed fracture widths are not always the same in the different fracturing treatment. Therefore, it is necessary to investigate the effect of fracture width on plugging behavior and optimize the optimal combination of fibers and particulates under different fracture width. A total of twelve groups of plugging experiments were conducted and the corresponding pressure curves were shown in **Fig.7**. It can be seen from **Fig.7 (a)** that the only fibers and combination of fibers and particulates both have favorable plugging effect under the condition of 2 mm fracture width. For the case of fibers with the concentration of 1 wt%, the inlet pressure nearly kept stable from the

beginning to 17 min and then increased linearly to 10 MPa in a short time. For the case of the combination of fibers and particulates, the inlet pressure kept stable at first and then increases rapidly to 10 MPa in about 2 minutes. Hence, it can be concluded that the addition of particulates was conducive to speeding up the formation of bridging and plugging. In addition, the rate of pressure rise has also increased considerably.

Based on above studies, the fibers with the concentration of 1 wt% have the favorable temporary plugging effect for the case with fracture width of 2mm. Hence, its temporary plugging effect was also studied under the condition of fracture with the width of 4mm. **Fig. 7(b)** shows the relationship between inlet pressure and time for fibers with different concentration and particulates with the concentration of 1 wt% and diameter of 2 mm. For the fibers with the concentration of 1 wt%, there has been a tendency to form temporary plugging zone which is always broken. During the whole experimental process, the maximum inlet pressure is 0.7 MPa, indicating that the 1 wt% fibers cannot effectively plug 4 mm fracture. Therefore, the similar experiment was performed again by increasing the fibers concentration to 1.2 wt%. Compared with the above case, the frequency of bridge temporary plugging and the plugging pressure is slightly higher, demonstrating that the plugging effect increased with the rise of fibers concentration. However, the effective temporary plugging zone is still not formed as the maximum inlet pressure is approximately 1.2 MPa. Hence, it's difficult to obtain the expected plugging effect without greatly increasing the concentration of fibers. The particulate with the concentration of 1 wt% and diameter of 2 mm was also introduced into the temporary plugging experiments. It can be seen that the variation of inlet pressure is similar to the case of fibers. It should be noted that the bridging plugging frequency of particulates is higher than that of fibers with the concentration of 1 wt%, but lower than that of fibers with the concentration of 1.2 wt%. During the experimental process, the maximum inlet pressure only reached approximately 0.9 MPa, indicating that no effective plugging zone has been formed.

Based on above experimental results, a single type of temporary plugging agent (only fibers or particulates) cannot achieve effective plugging. Consequently, the composite temporary plugging (combination of fibers and particulates) was introduced to improve the plugging effect. As shown in **Fig. 7(c)**, the relationship between inlet pressure with time of the different combinations were presented. For the combination of 1 wt% fibers and 0.8 wt% particulates, the inlet pressure experienced a process of ascending and descending within 40 min, indicating the diversion agents repeated the process of temporary plugging and breakthrough in the fracture. Then, the bridge temporary plugging starts to be formed and the inlet pressure rises to 2 MPa. Unfortunately, the temporary plugging zone has been still broken and no effective plugging has been formed in the fracture. When the concentration of particulates increased to 1 wt%, the variation of inlet pressure from the beginning to 40 minutes was similar to that of the above case. However, the difference is that the inlet pressure eventually rises to 10 MPa, indicating that the tight temporary plugging zone has been created. When the concentration of the particulates remains unchanged and the diameter of the particulate increases to 3 mm, the variation of inlet pressure was completely different from the above two case. It can be seen that the inlet pressure remained stable within 5 min and underwent a more gradual ascent from 5 min to 24 min followed by a rapid ascent. The main reason is that the particulates with large diameter firstly bridging and plugging in the area with a smaller fracture width, then the fibers can further accumulate on the particulates which increases the flow resistances and narrows down the flow path. Finally, the temporary plugging zone with tight structure was formed as results of sustained aggregation of diversion agents. The experimental results demonstrated that the temporary plugging effect can be significantly improved by increasing the size or the concentration of particulates under the condition of 4 mm fracture width.

When the fracture width increases to 6 mm, the temporary plugging effect of different combinations were also investigated. **Fig. 7(d)** shows the relationship between inlet pressure with time for different combinations of diversion agents. For the case of particulates with the concentration of 1.2 wt% and the diameter of 2 mm, the inlet pressure nearly kept stable with a small fluctuation. During the whole experimental process, the maximal pressure is up to approximately 0.3 MPa. In addition, the pressure fluctuations between 20 and 30 minutes indicated that temporary

plugging agents were trying to bridge and plug. However, no effective temporary plugging zone has been formed in the fracture. In order to get favorable plugging effect, the temporary plugging ball with the diameter of 3 mm was introduced into the experiments. For the case of particulates with the concentration of 0.8 wt% and the diameter of 3 mm, the inlet pressure increased to 0.5 MPa in 20 minutes and then fluctuates around this pressure. During the experiment, the maximum inlet pressure only reached approximately 0.8 MPa, indicating that no effective plugging zone has been formed. When the concentration of temporary plugging ball increased to 1.0 wt%, the frequency of bridge plugging and maximum pressure were both enhanced, indicating that the plugging effect was improved. Hence, the concentration of particulate was further increased to 1.2 wt%. It can be seen that the inlet pressure finally reached 10 MPa after repeated the process of temporary plugging and breakthrough. Therefore, it can be concluded that the concentration and size of particulates should be increased at the same time, which can achieve expected temporary plugging effect under the condition of 6 mm fracture width.

Fig.7 The effect of fracture width (No. 2, No. 5 - No. 15)

3.4 The effect of fracture morphology

All of the above experiments were carried out under the same fracture morphology (3D printing Model-1). However, the surface morphology of acid-etched fracture which is determined by the reservoir heterogeneity, mineral distribution, acid type, and injection rate, also has a significant impact on the temporary plugging effect. Therefore, three 3D printing models with different surface morphologies of fractures were used to conducted temporary plugging

experiments (No. 2, No. 16, and No. 17). It should be noted that all experimental conditions except surface morphology are consistent. **Fig. 8** shows the relationship between inlet pressure with time for these three groups of experiments. It can be seen that three groups of experiments can eventually form an effective plugging, but the time of forming the plugging was different. Therefore, it can be concluded that the surface morphology affects the formation time of temporary plugging, but does not affect whether temporary plugging is formed or not. The internal reason of formation time difference will be discussed next.

Fig.8 The relationship curve of inlet pressure changes with time for different fracture morphology (No. 2, No. 16, No. 17)

In order to further investigate the effect of surface morphology on temporary plugging, this paper introduced the definition of the minimum fracture width (FW_m). Due to the rough surface of acid-etched fracture, the vertical distance of fracture width (FW_v) is usually not the minimum distance. **Fig. 9** shows the schematic diagram of the FW_m . The minimum fracture width is defined as the minimum distance between a point on the upper fracture surface and all points in a square area ($L \times L$; L is equal to forty step sizes, and each step size is 0.05 mm.) on the lower fracture surface, and it can be obtained by processing the treated scanning data.

Fig.9 Schematic diagram of the minimum fracture width

Based on the definition of FW_m , the FW_m distribution of the whole fracture surface can be calculated as shown in **Fig. 10** left column. **Fig. 10** right column shows the morphologies of temporary plugging zones for three different 3D printing models in these experiments. By comparing the morphology of temporary plugging zone with the FW_m distribution, it can be found that temporary plugging zone was always formed at the location with a smaller FW_m and

extends to the inlet end. For the FW_m distribution of the 3D printing model-1, there are three obvious areas (A, B, C) with smaller FW_m in the fracture. Area 'A' is perpendicular to the flow direction and area 'B' is along the flow direction while area 'C' is at the angle to flow direction. Amounts of fibers are accumulated in these three areas as shown in morphology of temporary plugging zone. Fibers are preferentially adhering to these areas and then capture the subsequently injected fibers and particulates. Since the area A is near to the inlet, one side of fibers forming the bridge in the area A extend to the inlet and the other side penetrates to area B due to its smaller FW_m . Therefore, a transverse "7" shape of the temporary plugging zone has been created near the inlet. In addition, there are still a small number of fibers and particulates propagate to area 'C' with smaller FW_m which is relatively far from the inlet. But the diversion agents are not enough to form tight temporary plugging zone in area 'C'. In addition, for the FW_m distribution of the 3D printing model-2, the area with smaller FW_m was mainly located in the first half of the model, and there is an obvious area with smaller FW_m perpendicular to the flow direction in the middle portion. Undoubtedly, the location of the temporary plugging zone is basically consistent with the distribution of the smaller FW_m in the fracture. By the way, the similar phenomenon also can be found in the No. 17 experiment which the 3D printing model-3 was used.

According to the description of the acid-etched fracture surface morphology, the reason for the difference of formation time can be further analyzed. As shown in **Fig. 8**, it can be seen that the formation time of temporary plugging from short to long is the experimental group 17, group 16, and group 2. And the corresponding 3D printing models are model-3, model-2, and model-1, respectively. In the model-3, there is an obvious region with the smaller FW_m near the inlet, which is perpendicular to the flow direction. Hence, there is no doubt that it takes the shortest time to form an effective plugging zone. Compared with the experimental group 17 (model-3), group 16 (model-2) takes a slightly longer time to form temporary plugging as a result of the area with smaller FW_m (easy to form bridging plugging) located in the middle of the model. Based on the experimental result of group 2 (model-1), it takes the longest time to form an effective temporary plugging zone in the fracture. Unlike the two models mentioned above, there is an area (Area "B") with smaller FW_m in the parallel flow direction for the model-1, thus leading to an increase of the formation time of temporary plugging. Therefore, it can be concluded that the area with smaller FW_m in the vertical flow direction is favorable for the formation of temporary plugging, while the area with smaller FW_m in the parallel flow direction is not conducive to the formation of temporary plugging.

Fig.10 The morphology of temporary plugging zone and the minimum fracture width distribution within fracture (From top to bottom: No. 2, No. 16, No. 17)

4 Conclusion

Based on the 3D printed model and fracture temporary plugging evaluation system, the effects of different factors on plugging behavior of fibers and particulates were systemically investigated. The analysis of the experimental results reaches the following conclusion:

(1) Under the experimental condition, the temporary plugging effect of HPG fracturing fluid is better than that of slick water. The intrinsic reason is that the viscosity of HPG fracturing fluid is higher than that of slick water. If slick water is applied in field, it's recommended to add proper thickener or use a large injection rate, making up for its poor capacity of carrying fibers and particulates.

(2) The effect of temporary plugging deteriorates with the decrease of injection rate. The main reason can be attributed to the fact that high injection rate increases the probability of bridging and plugging. Therefore, it is recommended to use high injection rate for construction on the premise of on-site conditions.

(3) For the case of 2 mm fracture width, fibers and the combination of fibers and particulates both have favorable plugging effect. However, the addition of particulates was conducive to speeding up the formation of bridging and plugging.

(4) For the case of 4 mm fracture width, a single type of temporary plugging agent (only fibers or particulates) cannot achieve effective plugging. The combination of fibers and particulates can obtain favorable effect of temporary plugging. Furthermore, the temporary plugging effect can be significantly improved by increasing the size or the concentration of particulates.

(5) For the case of 6 mm fracture width, it's difficult to obtain favorable plugging effect even if the concentration of particulates increases when the diameter of particulates is relatively small (less than 50% of fracture width). Consequently, it's a wise choice to add big particulates whose diameters are at least 50% of the fracture width, which can improve the plugging effect.

(6) The fracture surface morphology affects the formation time of temporary plugging, but does not affect

whether temporary plugging is formed or not. Moreover, the location of the temporary plugging zone is basically consistent with the distribution of the smaller FW_m in the fracture. The area with smaller FW_m in the vertical flow direction is favorable for the formation of temporary plugging, while that in the parallel flow direction is not conducive to the formation of temporary plugging.

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Highlights:

1. 3D printing technology was introduced to reproduced the acid-etched fracture surface.
2. Temporary plugging behavior of fibers and particulates within acid-etched fracture was revealed.
3. A new parameter – the minimum fracture width was defined to describe the fracture morphology.