Large-Scale Acid Fracturing Based on a Large-Scale Conductivity Apparatus

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ABSTRACT: The conductivity of an acid-etched fracture is a necessary indicator for the stimulation of dolomite formation, which affects commercial development. The widely accepted test method involves the use of a small-scale conductivity cell for etching and measuring conductivity. However, the field production reflects that the actual conductivity does not match the measured one and is usually lower. Consequently, the existing studies had limitations and hence the stimulation mechanism needed to be explored further. To understand it more realistically, a novel large-scale apparatus was used in this study to test the conductivity of the acid-etched fracture. The use of this apparatus avoided the near-core excessive eroding and weak heterogeneity with continuous etching in a 1000 mm fracture. The results showed that the conductivity was indeed dissimilar to that in small-scale tests. The morphology of etched large-scale cores featured diversity and complexity, including deep and punctate channels, nonuniform pitting grooves with connected channels, and scale-shaped wavy grooves, which exactly demonstrated the multiple morphology under the influence of carbonate heterogeneity in real reservoirs. Moreover, the effect of increasing injection rate led to the unique etching morphology of scale-shaped wavy and pelviform grooves because of scouring flow and turbulence effects. The degree of surface roughness promoted nonuniform etching along the longitudinal and propagation direction, thus enhancing the conductivity of the whole fracture and confirming that the field treatment limited the pressure rather than the injection rate. The conductivity under different acid type, acid concentration, reaction temperature, and injection rate conditions was lower than that reported, confirming the experimental deviation in small-scale conductivity. The proposed large-scale apparatus test represented the acid-etched fracture conductivity more realistically, thus proving beneficial for the development of carbonate reservoirs.

1. INTRODUCTION

Carbonate reservoirs vary with different caves and natural fractures, where mining is usually difficult with unsatisfactory yield. Acid fracturing is the most effective method to improve production.1−4 The key parameter of this treatment is the conductivity of an acid-etched fracture,5,6 which is obtained primarily from experiments and predicted using an empirical formula.7 The measurement method of acid-etched fracture conductivity was introduced in 1968,8 when Nierode and Kruk put forward an empirical equation for estimation based on their experiments.9 Laser scanning technology was used to get the accurate morphology after etching,10,11 revealing various mechanisms of an acid–rock reaction.12,13 Meanwhile, innovative testing techniques and further investigations were used to study the factors influencing the acid-etching conductivity.14−16 Cores of different dimensions were used in experiments on acid-etched fracture conductivity: the columnar cores used in the classical N-K experiment were 25.4 mm in diameter and 50.8−76.2 mm in length;17,18 the core used by Ruffet was 100 mm in diameter and 400 mm in length.19 Antelo and Navarrete in their small-scale core experiment, using a modified API (American Petroleum Institute, which sets some standards for the oil industry) conductivity cell20,21 but with a small size, proposed that representing an accurate heterogeneity of the reservoirs is difficult using small-scale apparatuses. Malagon introduced a 180.59 mm length and a 40.89 mm width as a standard dimension for core experiments.22 Currently, the testing equipment for conductivity has a standard dimension of 178 mm length, 38 mm width, and 25 mm height (with arcs on both ends), similar to that proposed by Navarrete. The inlet erosion effect caused excessive etching on the walls of the acid-etched fractures or even cavities because of the limitation in the dimension of the equipment, leading to a fake increase in the average width of acid-etched fractures because of the short reaction distance. Besides, the results represented the conductivity of near-well-bore areas, instead of the whole fracture, as the influenced inlet area had a portion of the core and was short. Moreover, the test result was an inappropriate representative of the fracture conductivity of the carbonate more than 100 m underground.23,24 Similarly, small-scale rock cores could not fully reflect their heterogeneity and the
heterogeneity affected the morphology of the fracture surfaces, resulting in an inappropriate morphology of the carbonate reservoir where both were related to each other.

This study aimed to investigate the effects of acid type, acid concentration, injection rate, and temperature on acid-etched fracture morphology and conductivity. A novel large-scale acid-fracturing conductivity testing apparatus was proposed based on a core with dimensions of 1000 mm length, 100 mm width, and 20–40 mm height besides a matched conductivity cell and a large 3D laser profilometer. The conductivity obtained from this experiment was compared with that obtained from other small-scale experiments. The findings might provide an important reference for further studying and predicting acid-etched fracture conductivity and optimize the laboratory experiments and design of an acid-fracturing treatment.

2. EXPERIMENTAL PROCEDURE AND SCHEME

2.1. Materials. The cores used in the experiments were made of dolomite and sampled from the outcrop of the reservoir of the Qixia Formation in Western Sichuan, China. With an average permeability of less than 4 mD, these samples consisted of dolomite (more than 84.3%), quartz (less than 15.3%), and clay (less than 0.5%). The dimensions of the samples were taken as 1000 mm length, 100 mm width, and 20–40 mm thickness according to the similitude rule of cores and acid fractures to measure the acid etching and conductivity under real reservoir conditions, as shown in Figure 1.

The acids used in the experiments were HCl, gelled acid, and diverting acid. The HCl, gelling agent, diverting agent, corrosion inhibitor, and ferric ion stabilizer were purchased from Chengdu Kelong Chemical Reagent Co., Ltd., and the original samples were used in the experiments. The viscosity of the diverting acid in the experiments was controlled by the concentration of the acids; the specification of the Acids, the concentration of the acids; the specifications are shown in Table 1.

Table 1. Specification of the Acids

| no. | acid            | specification                                      | concentration (HCl) (%) |
|-----|-----------------|----------------------------------------------------|-------------------------|
| 1   | HCl             | HCl + 3% corrosion inhibitor + 1% iron stabilizer | 20                      |
| 2   | gelled acid     | HCl + 2% gelling agent + 3% corrosion inhibitor + 1% iron stabilizer | 5, 10, 15, and 20 |
| 3   | diverting acid  | HCl + 5% diverting agent + 3% corrosion inhibitor + 1% iron stabilizer | 20                      |

2.2. Experimental Apparatus. The experimental equipment is designed and developed on the basis of API (American Petroleum Institute) standard equipment. The interior chamber of the API conductivity cell is 18.4 cm long and 4.5 cm wide, and a slate with a length of 18 cm and a width of 4 cm can be placed to simulate the fracture, while the experimental system can put a slate with a length of 100 cm and a width of 10 cm, which can better predict the acid etching form of the fracture under a large acid pressure and the overall acid etching fracture conductivity.

A large-scale conductivity cell (in Figure 2) was the main equipment used in the experiment, which was designed based on an API fracture conductivity cell and could accommodate the cores. A large rubber tube was placed inside the conductivity cell and used to endure the cell pressure. The core was covered with a 1000 mm-long steel sleeve; it was used to prevent the cores from fracturing when a linearly increased pressure was applied. Metallic shims were also employed to realize the changing pattern of the fracture width.

The main structural components and technical parameters of the experimental equipment are given below.

2.2.1. Conductivity Cell. The conductivity cell can accommodate the size of the rock slab of length × width × thickness = 100 cm × 10 cm × 1.5 ~ 3 cm, the simulated crack width is 1–10 mm (adjustable), the crack length is 100 cm, and the system pressure is 0–12 mPa.

The experimental equipment is designed and developed on the basis of API equipment. The flow of acid in the fracture can be simplified as the flow of acid in a pair of parallel plates.

In the design of the model length, considering that the longer the model seam length is, the less the influence of the plate end on the flow will be, and combining with the technical bottleneck made by the equipment, the model seam length is selected to be 1 m. In order to verify the reliability of similar geometric dimensions between the model and the prototype, a hydraulic radius is introduced to compare the flow capacity between the model and the prototype. For the parallel plate physical model simulated in this paper, the hydraulic radius is

\[ R = \frac{hw_f}{2(h + w_f)} \]  

(1)

where \( R \) is the hydraulic radius; \( h \) is the fracture height (plate height); and \( w_f \) is the fracture width.

The combination of height and width was selected based on the actual fracture height of 40 m, and the relative error of hydraulic radius was calculated, as shown in Table 2.

It can be seen from the calculation results that the smaller the seam width and the larger the seam height, the smaller the
error between the model and the hydraulic radius is. However, considering that the influence of elastic force on the flow increases when the seam width is too small, and considering the conditions of experimental equipment, the selected fracture height and width of the model are 100 and 5 mm, which can ensure that the hydraulic radius error of the model and the prototype is within 5%, satisfying the good geometric similarity with the prototype. When the geometric similarity between the model and the prototype is satisfied, the flow similarity can be satisfied as long as the Reynolds criterion is satisfied, which makes the simulation experiment result similar to the actual construction situation.

2.2.2. Three-Plunger Metering Pump. The function of a metering pump is to push acid liquid to inject the fracture with a certain displacement in the acid erosion test and to inject the fracture with a certain pressure and displacement in the conductivity test. The flow range is 0–10 L/min and the pressure range is 5–8 MPa. According to different experimental needs, the liquid can be injected with different flow rates by adjusting the frequency converter.

For the plate flow simulated in this paper, under the condition of geometric similarity between the model and the prototype, the flow similarity can be satisfied as long as the Reynolds criterion is satisfied, that is, the model and the prototype have the same Reynolds number, which makes the simulation experiment result similar to the actual construction situation. Assuming that the fluid viscosity and flow velocity are constant and the compressibility of the fluid is ignored, the Reynolds number is calculated as follows for flat flow with a slit height far greater than the slit width

\[ Re = \frac{W_d P V}{\mu} \]  

(2)

where \( Re \) is the Reynolds number, \( \rho \) is the fluid density, \( v \) is the fluid flow velocity, and \( \mu \) is the fluid viscosity. As the experiment uses the same liquid as the actual construction, in order to meet the Reynolds criterion, the following conditions must be met

\[ Q_d h_1 = 2Q_m h_1 \]  

(3)

where \( Q_d \) is the actual rate, \( Q_m \) is the rate of the model, \( h_1 \) is the actual fracture height, and \( h_1 \) is the fracture height of the model.

Therefore, the length of the experimental slate is 1000 mm, the fracture height is 100 mm, and the width is 5 mm. The experimental displacement is calculated as 3–7.5 L/min according to the actual rates, as shown in Table 3. This experiment can simulate the actual formation condition and the experimental results can be directly used in the design of acid pressure scheme.

2.2.3. Differential Pressure Transducer. The function of a differential pressure transducer is to monitor the pressure difference at the inlet and outlet of the diversion chamber under liquid flow conditions, with a range of 0–500 kPa.

2.2.4. Back-Pressure Valve. As CO₂ is generated after the acid etching test is initiated, in order to ensure that the CO₂ generated during the reaction is dissolved in the acid solution during the test, the pressure can be pressurized to 7 MPa through the return pressure valve to ensure that the test process is above 7 MPa, so as to meet the experimental requirements; the return pressure range is 0–35 MPa.

2.2.5. Heating System. The oil bath heating system consists of imported Hac alloy pipelines coiled into a U-shaped plate and embedded in the middle of the heating aluminum plate. Through electric heating, the experimental process reaches the required temperature to simulate the temperature under the condition of formation. The temperature range is 0–150 °C.

2.2.6. Closed Pressure Pump. The function of the closed pressure pump is to simulate the closed crack pressure in the closed acidification experiment and the acid erosion fracture conductivity experiment. The pressure range is 0–50 MPa.

A large laser profiler (in Figure 3) was designed to facilitate 3D data scanning and analyze the morphology in the core caused by acid etching. It provided a high-accuracy linear laser with a scanning range of 1000 mm length, 100 mm width, and ±48 mm height. The accuracy of the scanning was 0.1 mm. The reflected beam is received by the photoelectric receiver (CCD camera) and the three-dimensional point cloud map of the object under test is obtained. The scanning data are saved in the format of “*.CSV”, which can be processed by third-party software. The greatest advantage of this device is

![Figure 3. 3D laser profilometer.](https://dx.doi.org/10.1021/acsomega.0c03792)

Table 2. Comparison of a Hydraulic Radius of 40 m Seam Height Prototype with Different Sizes of Seam Height and Seam Width Model

| real fracture high (mm) | real fracture width (mm) | fracture high of model (mm) | fracture width of model (mm) | relative error of hydraulic radius (%) |
|------------------------|--------------------------|-----------------------------|-----------------------------|--------------------------------------|
| 40,000                 | 1                        | 100                         | 1                           | 0.99                                 |
| 40,000                 | 3                        | 100                         | 3                           | 2.91                                 |
| 40,000                 | 5                        | 100                         | 5                           | 4.75                                 |
| 40,000                 | 1                        | 40                          | 1                           | 2.44                                 |
| 40,000                 | 3                        | 40                          | 3                           | 6.97                                 |
| 40,000                 | 5                        | 40                          | 5                           | 11.1                                 |
| 40,000                 | 1                        | 10                          | 1                           | 9.09                                 |
| 40,000                 | 3                        | 10                          | 3                           | 23.07                                |
| 40,000                 | 5                        | 10                          | 5                           | 33.33                                |

Table 3. Comparison of On-Site and Indoor Rates under the Reynolds Number Criterion

| condition | rate, m³/min | width, m | height, m | density, kg/m³ | velocity, Pa·s | Reynolds number |
|-----------|--------------|----------|-----------|----------------|----------------|----------------|
| on-site   | 6            | 0.005    | 40        | 1000           | 0.1            | 12.5           |
| indoor 1  | 0.0075       | 0.005    | 0.1       | 1000           | 0.1            | 12.5           |
| indoor 2  | 0.0006       | 0.005    | 0.1       | 1000           | 0.1            | 1              |
that it can quickly obtain the three-dimensional point cloud map of the measured surface without damaging the slate surface and minimize the human error to the greatest extent. A schematic diagram of a 3D laser scanning system is shown in Figure 4.

2.3. Experimental Procedure. The experimental procedure was divided into five steps: core sample preparation, surface profile characterization prior to the reaction, acid etching, surface profile characterization after the reaction, and fracture conductivity measurement. The core samples were cut into designated shapes, which could fit the steel sleeve, as shown in Figure 5. Then, the core with a steel plate was scanned with the profilometer to get its surface profile. Two cores were put into the chamber according to the designated width of the fractures. Subsequently, to maintain the wettability of the core, saturated brine was injected into the diversion chamber before initiating acid fracturing.

2.3.1. Acid Etching. The basic width of the core simulated by metallic shims was 4 mm when the core was put into the steel sleeve. According to the similarity law, the basic blank reaction test included an injection rate of 3 L/min and a gelled acid with a concentration of 20% and a volume of 40 L. The acid was placed in the container before closing the channels of the conductivity cell. The brine was saturated before acid etching, and the temperature of the heater was set as 120 °C after the saturation. The schematic of the acid-etching apparatus is shown in Figure 6.

2.3.2. Characterization of the Fracture Surface. The core before and after the reaction was scanned using a 3D laser profilometer to investigate the effects of acid-fracturing techniques on the acid-fracturing efficiency. The morphology of the etched surface was analyzed to evaluate the acid-etching effect using a special spraying system to spray all the cores before scanning.

2.3.3. Fracture Conductivity. The core after acid fracturing was put into the diversion chamber horizontally and perpendicularly (without steel sticks to fix the width of the fracture), ensuring that the liquid could flow along the vertical fractures. Then, the brine procedure was switched to test the conductivity. The injection rate was measured with a mass flow meter, and the confining stress was increased gradually with a step of 5 MPa until it reached 40 MPa, and 10–20 values were recorded at every step. The conductivity was calculated using eq 4.

\[ k_f w_f = 1.67 \times 10^{-3} \frac{Q \mu L}{w \Delta p} \]  

where \( k_f w_f \) refers to the acid-etching fracture conductivity, D·cm; \( Q \) refers to the acid injection rate, mL/min; \( \mu \) refers to the acid viscosity, mPa·s; \( L \) refers to the core length, cm; \( w \) refers to the rock plate width, cm; and \( \Delta p \) refers to the pressure difference between the ends of the core.

Figure 4. Schematic diagram of a 3D laser scanning system.

Figure 5. Large-scale core after assembly.

Figure 6. Schematic of the acid-etching apparatus.
Ten experiments categorized into four different groups were conducted to study the effects of different acid types, injection rates, reaction temperatures, and acid concentrations on the morphology and acid-etched fracture conductivity. The acids mainly included HCl, gelled acid, and diverting acid. Acid types are commonly used in the field, except for HCl which was used as a control group. The injection rates were selected as 3, 5, and 7 L/min. The concentration of the acids varied from 5 to 20%, with a 5% increment. The basic blank experiment was the one with a gelled acid concentration of 20% and an injection rate of 3 L/min at 120 °C. Most of the experiments were conducted at 120 °C except for two experiments at 140 °C and used for comparison with both gelled acid and diverting acid. In all experiments, the initial fracture width was 4 mm, the total volume of acid was 40 L, the back-pressure was controlled at 7 MPa to maintain the dissolved CO2 in the spent acid, and all the cores were made of dolomite. The experimental scheme and conductivity results are shown in Table 4.

### 3. EXPERIMENTAL RESULTS AND INTERPRETATION

Ten experiments categorized into four different groups were conducted to study the effects of different acid types, injection rates, reaction temperatures, and acid concentrations on the morphology and acid-etched fracture conductivity. The acids mainly included HCl, gelled acid, and diverting acid. Acid types are commonly used in the field, except for HCl which was used as a control group. The injection rates were selected as 3, 5, and 7 L/min. The concentration of the acids varied from 5 to 20%, with a 5% increment. The basic blank experiment was the one with a gelled acid concentration of 20% and an injection rate of 3 L/min at 120 °C. Most of the experiments were conducted at 120 °C except for two experiments at 140 °C and used for comparison with both gelled acid and diverting acid. In all experiments, the initial fracture width was 4 mm, the total volume of acid was 40 L, the back-pressure was controlled at 7 MPa to maintain the dissolved CO2 in the spent acid, and all the cores were made of dolomite. The experimental scheme and conductivity results are shown in Table 4.

### 3.1. Acid Type

The 3D morphologies of cores after etching with three different acids are shown in Figures 7–9. The core etched using HCl was characterized by a relatively uniform morphology with slightly shallow and punctate channels; the acid–rock reaction concentrated in the lower parts of the core (Figure 7) with smooth channel grooves. The morphologies of the cores etched with gelled acid and diverting acid (Figures 8 and 9) featured nonuniform pitting corrosion, continuously etched grooves, and clear columnar
nail supporting points, indicating the strong nonuniform acid-etching effects of these two acids. A seepage channel was formed by connected pitting near the axis of the gelled acid-etched core, while continuous nonuniform etching occurred in the diverting acid-fractured core with a decrease in the acid concentration. The volumes and concentrations of the spent acid in gelled acid and diverting acid etching (Table 5) were about 70 cm$^3$ and 17%, illustrating a similar dissolution capacity of gelled acid and diverting acid as well as dominant nonuniform etching. However, HCl mainly etched a uniform surface on the core, with a dissolution volume of 154.28 cm$^3$, which was about two times larger than those of gelled acid and diverting acid.

The conductivities of the cores etched using different acids at different confining stresses are shown in Figure 10. The core etched using HCl showed the largest conductivity (about 60 D·cm$^{-1}$), but it decreased rapidly with its ascent, as the smooth etching form of the core had low capacity to maintain the fracture. The conductivity was only 1/15 of its initial value when the confining stress was 15 MPa, and it was 0.4 D·cm$^{-1}$ when the confining stress increased to 40 MPa. The roughness of the surfaces was better than that of HCl due to the diversity and complexity of the morphologies caused by nonuniform acid etching, leading to larger chemical self-supporting capacities. The conductivity of the core etched using diverting acid was larger than that using gelled acid at a lower confining stress, but the values were close to each other (7 D·cm$^{-1}$) when the confining stress was larger than 30 MPa.

### 3.2. Acid Concentration.
Set B was arranged for comparing the effect of acid concentration on acid etching. The morphologies of the cores etched using different concentrations of gelled acid are shown in Figures 11–13. Different acid concentrations caused different morphologies, and the morphologies changed their patterns from being single to mixed and diverse. To be specific, the morphologies featured from smooth flattening surfaces to partially point-etched shallow grooves and then nail-shaped connected deep trenches. The core etched with 5% gelled acid was generally smooth and flattened with some small pits, presenting a single etching pattern (Figure 11). With an increase in concentration to 10%, the core showed columnar points and emerging asperities in certain parts (Figure 12), besides relatively smooth and shallow grooves. Figure 13 shows that the front core reflected multiple lines converging to a clear and deep groove channel. In the middle part, the channel was eliminated with a heterogeneous change in lithology; continuous grooves appeared again in the end area, which was typical of the mixed form. A strong nonuniform acid–rock reaction and a diverse surface morphology were observed when the acid concentration was 20%. As indicated in Table 6, the dissolution volumes varied from 28.44 to 70.13 cm$^3$ with an increase in acid concentration from 5 to 20%, indicating that more H$^+$ was involved in the acid–rock reaction. However, the common-ion effect due to a higher acid concentration, which led to a reduction in the dissolution volume, should not be neglected.

In the whole range of the confining stresses shown in Figure 14, the higher the acid concentration, the higher the conductivity of the cores. Figures 11–13 and Table 6 show that a lower acid concentration limited the acid–rock reaction, resulting in a monotonous and uniform acid-etching surface, which might not maintain the etched roughness facture to provide enough conductivity. The diversity and heterogeneity were affected with an increase in acid concentration, leading to a mixed morphology characterized by columnar supporting points, connected grooves, and punctate channels, which in turn improved and increased the conductivity. Subsequently,
the conductivity increment was gradually stable because of the common ion effect, which limited the nonuniform etching capacity at the bottom. The total increase in conductivity due to an increasing acid concentration was limited as the concentration reached 20% (Figure 14).

3.3. Injection Rate. This study thoroughly analyzed the relationship between injection rate and morphology or conductivity. The core in Figures 15 and 16 had very complicated and different surface morphologies compared with that in Figure 7. Scale-shaped and wavy channels (Figure 15), which were connected by relatively shallow grooves at the end, had quite unique morphologies. The morphology (Figure 16) characterized the complexity of the nonuniformly etched core with wavy patterns and deeper peaks and troughs. In the middle of the core, the morphology featured etched grooves and stacked scale-shaped surfaces, with nail-shaped and bulging points; the grooves deepened into pelviform trenches at the end of the waves, just like a vortex.

Figures 15 and 16 show that the rapid acid flush accelerated the acid—rock reaction, and the turbulent effect was enhanced.
by the heterogeneity of the rock, increasing the nonuniform etching width. This was the reason why the compound morphology featured continuous, scale-shaped, wavy, pelvis-form grooves. As shown in Table 7, the etching volume increased with an increase in injection rate (from 80.55 to 130.45 cm\(^3\)), and the concentration of the spent acid reduced. This was different from the phenomenon where the nonuniform morphology did not change much, but the etching volume increased with the increase in acid concentration.

Figure 17 shows that a larger injection rate resulted in a larger conductivity, and the conductivity at a flow of 7 L/min was the largest among all the conductivities at all confining stresses. The largest conductivity at lower confining stresses was 66.1 D·cm, and the conductivity at a flow of 3 L/min was the smallest but 6.6 D·cm at a high confining stress. At the same confining stress, a larger flow contributed more to the conductivity: the difference in conductivity between 3 and 5 L/min and between 5 and 7 L/min was 4.9 and 6.6 D·cm, respectively. The same trend also existed in other confining stress levels. The conductivity had an increasing trend with the increase in injection rate, leading to an increment in the conductivity. This was because a large flow resulted in a combined, multiple, and diverse rather than a monotonic morphology. The turbulent effect in cores would cause different kinds of nonuniform etching; the larger the injection rate, the more evident the turbulent effect. Fundamentally, the occurrence of the turbulent effect was representative of heterogeneity of the lithology, which could only be reflected in large-scale experiments instead of small-scale experiments. This finding was also consistent with the construction treatment in oil fields where engineers usually increased the injection rate to improve the reforming efficiency at an allowable construction stress. Gelled acid enveloped H\(^+\) due to its viscosity, which slowed down the reaction rate between hydrogen ions and rock minerals. Meanwhile, the product also attached to the surface after the reaction, which reduced the mass transfer coefficient of hydrogen ions in the acid and weakened the convective reaction. In the process of liquid etching, the nonuniform etching ability is gradually enlarged due to the influence of construction discharge and rock properties. The hydrochloric acid reaction rate is fast, the convection after hydrogen ion reaction is not affected, and can be quickly etched in the leading edge without a difference.

### Table 7. Experimental Program and Results with Different Injection Rates

| Conditions | Results |
|------------|---------|
| Acid       | Concentration (%) | Temperature (°C) | Injection Rate (L/min) | Concentration of Spent Acid (%) | Etching Volume (cm\(^3\)) |
| gelled acid| 20      | 120          | 3                    | 17.45   | 70.13  |
|            | 5       | 15.92        | 98.54                |
|            | 7       | 13.19        | 130.45               |

Figure 16. 3D scanned surface morphology after acid etching with an injection rate of 7 L/min.

Figure 17. Fracture conductivity for different injection rates at different confining stresses (set C).

Figure 18. 3D scanned surface morphology using gelled acid at 140 °C.
3.4. Temperature. The morphologies caused by gelled acid and diverting acid at 120 and 140 °C were analyzed to study the effects of temperature on acid etching. The higher the temperature, the faster the H+ mass transfer rate and the faster the reaction rate. A high temperature can help the acid etching but accelerate the consumption of acid, which leads to the decrease of effective etching length. Figures 18 and 19 show that the morphologies at 140 °C caused by gelled acid and diverting acid were rougher and more nonuniformly etched; the morphological abundance was more than that at 120 °C. The morphologies caused by gelled acid at 140 °C were mainly deep pits and short channels, with parts of channels connected together. The morphologies caused by diverting acid at 140 °C were mainly circuitously connected grooves with pitting nail and pelviform grooves connected together, showing an abundance of etching patterns. Besides, as indicated in Table 8, the volumes of acids at 140 °C were more than those at 120 °C. Thus, diverting acid was better than gelled acid in terms of morphology and etching volume.

With the same confining stress and acid, the conductivity of the core (Figure 20) acidized by acids at 140 °C was larger than that at 120 °C, proving that the temperature of 140 °C still had a positive effect on the conductivity. The etching volume, roughness, and etching gradation increased with the increase in conductivity. However, a higher temperature required more acid volume, leading to limited effective etching length. A significant difference was seen in 100 m hydraulic fractures, although it was not obvious in 1 m cores.

3.5. Comparison with the Published Findings. To deeply investigate the relationship between acid etching and conductivity in large-scale experiments, the results of this study were compared with the findings of other small-scale experiments.11,22–24 The physical and scanned morphologies in small-scale experiments had deeper channels in the fracture where an initial acid–rock reaction occurred compared with the other locations. The etched grooves were more obvious in the vicinity of the inlet. The inlet effect was introduced,24 but no solution was proposed. From the inlet, the deepened etched rock comprised more than 20% of the whole length of the core along the flow direction, and this should not be neglected due to the small dimension of the core. Figure 21 shows that the inlet of the core was not excessively etched but had the real morphology, and the large dimension could offset the errors caused by a small core.

Table 8. Experimental Program and Results at Different Temperatures

| Conditions | Gelled Acid | Diverting Acid |
|------------|------------|---------------|
| Temperature (°C) | Concentration (%) | Injection Rate (L/min) | Concentration of Spent Acid (%) | Etching Volume (cm³) |
| 120 | 17.45 | 70.13 |
| 140 | 16.92 | 78.64 |
| 120 | 17.15 | 72.55 |
| 140 | 16.38 | 81.79 |

Figure 19. 3D scanned surface morphology using diverting acid at 140 °C.

Figure 20. Effect of temperature on fracture conductivity for two different acid types (set D).

Figure 21. C7 and B6 cores after etching in the present study.

Different conductivity results in previous studies were selected for comparison: #1 comprised SET2 4 and SET3 7 cores used by Malagon,23 #2 comprised the samples by Pournik,11 and #3 comprised the Indiana limestone data from Hill.22 All the data were from the experiments on gelled acid etching under similar experimental conditions. Figure 22 shows that the conductivities in the aforementioned three studies were larger than those obtained from the large-scale experiments, and the difference was near 1 order of magnitude. The acid-etched fracture conductivity was estimated to be larger than the real value, with an error of 1 order of magnitude.
because the small cores were not representative of the true conditions in the reservoir and the error caused by the inlet effect.

The difference and heterogeneity of cores were considered in another group of small cores (set E), which were subjected to acid etching and conductivity experiments for a comparison. The experiments were conducted at 120 °C in 20% gelled acid, and the outflow parameter and reaction time were the same as mentioned in Malagon.23 The morphologies and conductivity are shown in Figures 23 and 24. The acid etching in small cores caused obvious jet-flow outlines. Also, the main etched groove was formed along the extension line, accounting for 50% of the whole core, characterized by bulging nonetched minerals on the surface, large etching depth, and single morphology. As shown in Figure 24, the conductivity in set E reduced from 80.4 to 10.2 D·cm, which was close to the range 86.1–3.17 D·cm stated in Malagon23 and larger than those in set A2. The groove formed in the vicinity of the inlet by the jet flow was the main factor resulting in high conductivity. The diversity in morphology due to the inlet effect and heterogeneity was not likely to occur in the cores with a dimension of 178 mm. Hence, the results from cores with a dimension of 1000 mm were more reliable..

4. RESULTS AND DISCUSSION

(1) Acid fracturing offered an effective and necessary means to stimulate limestone reservoirs, with dissolution of the rocks, weakening of the rock strength, and a nonuniform surface profile, finally leading to high flow conductivity. The study had two important observations. First, the previous conductivity tests created the forward error because of the core and conductivity cell scale. The limitation of the heterogeneity of the core sample was eliminated by choosing a length of 1000 mm instead of 178 mm, considering the obvious heterogeneity of limestone. Meanwhile, the 1000 mm acid-etching fracture reflected the underground situation accurately. In contrast, the small fracture formed a magnified channel near the well bore with a scouring effect, which probably represented the whole fracture surface, resulting in overoptimistic estimates. As the large-scale model stimulated the underground conditions clearly and precisely, it was meaningful to further explore acid etching and conductivity. Second, the conductivity influenced by multiple factors, such as acid types, acid concentration, injection rate, and temperature, was the same as examined in previous studies. A high pump rate is more beneficial for forming the deep notch, trench, and wave-shaped morphology, indicating that the more nonuniform the profile etched, the more the conductivity.

(2) The difference in the results of large-scale and small-scale experiments was mainly due to the heterogeneity of carbonate rocks, and the heterogeneity-induced morphology was seen in samples with a large dimension. The morphology in small-scale experiments was single and isolated, characterized by being monotonic, point-shaped, nail-shaped, and groove-like. The morphology in the large-scale experiments in this study featured a mixed and diverse appearance, including deep grooves, nonuniform pitting corrosion, and connected grooves in B4. The combined morphology was characterized by wavy grooves connected with tails and scale-shaped grooves in C8. These continuous and mixed morphologies could not be obtained from small-scale experiments, accounting for the errors in describing the morphology and measuring conductivity.

(3) Unfortunately, the present study did not consider the acid leak-off factor without fracture breakthrough or wormhole development observed in the experiments. Despite numerous contributions made by researchers in the leak-off of acid-fracturing fluids,23–28 some issues still need to be resolved. During the acid-fracturing process, the matric porosity changed with the dissolution; the number, length, and size of the formed wormhole were hard to determine, which made the leak-off of acids more complicated than hydraulic fracturing.
and using a constant injection rate as a substitute for the leak-off of acids was not appropriate. Although most of the acid-fracturing experiments mentioned considering the leak-off of acids, their results did not verify the wormhole development and breakthrough into the cores. Based on the previous findings, the leak-off of acids was not explored in this study; however, it should be investigated in future studies.

5. CONCLUSIONS

Four different sets of large-scale conductivity experiments were conducted on outcrops to study the mechanisms of acid-etching morphology and conductivity. Furthermore, the effects of the acid fluid types, acid concentration, injection rate, and temperature on the etching mechanism were investigated. A large-scale 3D scanner was employed to obtain and exhibit the temperature on the etching mechanism were investigated. A large-scale 3D scanner was employed to obtain and exhibit the temperature on the etching mechanism were investigated. The main conclusions of this study were listed as follows.

(1) The results of large-scale experiments reflected the heterogeneity of the cores and more complicated acid-etched fractures with layered, multiple, and diverse patterns. The experiments proved that the real acid-etching morphology was not single and monotonous but a combination of different morphologies. The conductivity of the core etched using diverting acid was larger than that using gelled acid at a lower confining stress.

(2) The conductivity increment was gradually stable because of the common ion effect, which limited the nonuniform etching capacity at the bottom. The total increase in conductivity due to an increasing acid concentration was limited as the concentration reached 20%. The turbulent effect in cores would cause different kinds of nonuniform etching; the larger the injection rate, the more evident the turbulent effect. Fundamentally, the occurrence of turbulent effect was representative of the heterogeneity of the lithology, which could only be reflected in large-scale experiments instead of small-scale experiments. This finding was also consistent with the construction treatment in oil fields where engineers usually increased the injection rate to improve the reforming efficiency at an allowable construction stress. The diverting acid was more sensitive to temperature because the conductivity in the diverting acid experiment was significantly larger than that in the gelled acid experiment. The groove formed in the vicinity of the inlet by the jet flow was the main factor resulting in high conductivity. The diversity in morphology due to inlet effect and heterogeneity was not likely to occur in the cores with a dimension of 178 mm. Hence, the results from cores with a dimension of 1000 mm were more reliable.

(3) The conductivity in previous small-scale experiments were usually smaller than those obtained from the large-scale experiments in this study. The heterogeneity of the large samples reduced the errors in the measurement of conductivity, and the acquired conductivity was representative of the conductivity in real carbonate rocks where the sample size was enlarged. The results of the large-scale experiments could provide a better reference for decision-making in the development of carbonates at larger depths.
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