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Case Study on the Effect of Acidizing on the Rock Properties of the Mahu Conglomerate Reservoir

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Abstract: The development of the Mahu tight reservoir has adopted horizontal wells with staged fracturing. In the fracturing, there is a problem of a high fracturing pressure. Acid treatment is often used to lower the fracturing pressure on site. At present, the impact of this acid treatment on the physical parameters of the rocks of the reservoir in the Mahu region has not been systematically studied. Aiming to solve this problem, this paper conducted an experimental study on how acid dissolution affects the physical properties of the Mahu conglomerate, including its porosity, permeability, triaxial rock mechanical parameters, tensile strength, and mineral composition. First, the experimental scheme was designed. Next, a series of experiments were conducted. Finally, the experiment results were analyzed comparatively before and after acidizing. The acid composition, concentration, and contact time were the main factors for the analysis, based on which the acid system and related parameters were recommended. This study showed that the Mahu conglomerate exhibited brittle plasticity characteristics under stress. The carbonate content in this region was low, while the feldspar content was high, so it was necessary to use mud acid to effectively dissolve feldspar, clay, and other silicates. After acidizing, the porosity was 200% of the original value. The permeability increased by up to 14 times. The tensile strength decreased significantly by up to 84%. The value of Young's modulus of the rock decreased by up to 63.6%. The value of Poisson's ratio was reduced by up to 40.7%. A combination of 6% HF + 15% HCl is recommended, with an effective acid treatment time of over 60 min for the Mahu conglomerate. Acidizing could significantly change the mechanical properties and permeability of the rock of the Mahu conglomerate reservoir, thus effectively reducing the formation fracturing pressure. This research provides technical support for Mahu acid dipping in horizontal well fracturing.

Keywords: experiment; Mahu; acidizing; physical properties; acid system

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1. Introduction

Sandstone and conglomerate reservoirs are widely distributed in the Songliao Basin, the Tarim Basin, the Bohai Bay Basin, and the Junggar Basin in China. The Junggar Basin, with its sizable proven oil reserves and huge development potential [1,2], is located in Xinjiang province in the northwest of China. It is the primary development area of conglomerate reservoirs in Xinjiang and China [3–5]. The Mahu conglomerate oil reservoir in the northwest edge of the Junggar Basin, with an area of nearly 5000 km^2, is an unconventional reservoir with poor physical properties and low permeability [6]. This kind of tight formation generally uses horizontal wells with a staged fracturing stimulation. Horizontal well fracturing in the Mahu area has the problem of that it is hard to break the formations due to the high fracturing pressure required. The main methods of lowering the fracturing pressure include acid pretreatment, hydro jetting, high-energy gas fracturing, and perforation [7–12]. At present, acid pretreatment technology is commonly used to reduce the fracturing pressure in the Mahu area along with perforation.

Li [13] derived an analytical formula of the formation fracture pressure P_f for horizontal well perforation completion based on the formula of formation fracture pressure for vertical well perforation completion.

$$P_f = \frac{9\sigma_h - 3\sigma_V - \sigma_H + 2v(\sigma_V - \sigma_h) - \varphi\frac{1-2v}{1-v}p_0 + \sigma_{\mathrm{f}}}{4 + \varphi_c - \varphi\frac{1-2v}{1-v}} \tag{1}$$

where P_0 is the original formation pore pressure, MPa; φ is the rock porosity, %; φ_c is the rock contact porosity, %; σ_f is the uniaxial tensile strength of rock, MPa; and v is Poisson's ratio of rock, which is dimensionless. It can be seen from Equation (1) that increasing the pore pressure and decreasing the tensile strength can lower the breaking pressure. Acidizing can significantly increase the permeability so as to increase the pore pressure before initiating the fracture. Acidizing also dissolves some minerals so as to decrease the tensile strength. This is why acidizing can decrease the fracturing pressure.

Some researchers have studied the influence of acid pretreatment on the mechanical properties of rock, but this research has mostly been conducted on shale instead of conglomerate. Wang et al. [14] used a triaxial rock mechanics testing system to conduct a comparative study on the compressive strength of reservoir rocks before and after sandstone acidizing, and they found that a high concentration of hydrofluoric acid can greatly reduce the compressive strength of rocks. Wang et al. [15] proposed using a rock mechanics testing system to determine the mechanical properties of rocks before and after acidizing and systematically studied the effects of the acid composition and concentration on the mechanical properties of sandstone rocks. Guo et al. [16] conducted a comparative study on the rock elastic modulus, Poisson's ratio, and rock compressive strength before and after acidizing in ultra-deep reservoirs with a high fracturing pressure by using reservoir rock samples, and the results showed that the acid rock reaction reduced the rock strength, thus reducing the formation fracturing pressure. Deng et al. [17] studied the mechanical properties of sandstone before and after acidizing for high-pressure, high-temperature, and low-permeability reservoirs, and they elaborated the mechanism of acid pretreatment in changing the mechanical properties of rocks and effectively lowering the fracturing pressure from a microscopic perspective. Their results showed that mineral dissolution changed the shape of the intergranular pores and crystal solution pores and improved the connectivity of the pores, thus reducing the rock strength.

Acid pretreatment has been applied to horizontal well fracturing in the Mahu fracturing reservoir for some time, but some treatments take a long time, and some are not effective. Some problems of the acid pretreatment in the Mahu reservoir remain unclear, such as the effective acid composition and concentration, the acid–rock contact time, and the variation in the physical properties of the Mahu conglomerate. Therefore, in this paper, we conducted a systematic experimental study on the interaction between the acid and minerals and the rock properties, and we recommended an acid system and its related parameters and provided guidance for the in-field acidizing pretreatment in the Mahu area.

2. Experimental Apparatus and Approach

2.1. Experimental Apparatus

The acid flooding test was accomplished using apparatus composed of a flooding pump, containers, a thermostat, and Hastelloy acid-resistant holders. The maximum experimental temperature could reach 200 °C, meeting the requirements of simulating the formation temperature. Figure 1 shows the schematic drawing of the acid flooding apparatus.

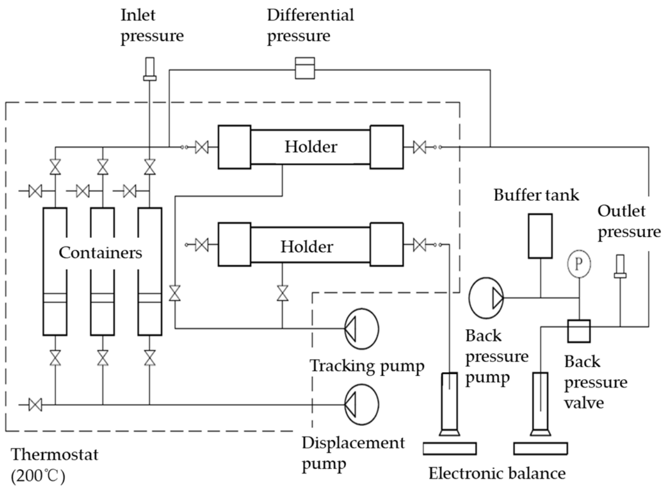


Figure 1. Experimental platform schematic drawing.

2.2. Experimental Samples

This paper took the downhole cores of Well Ma 132, 136, and 139 as the research object. The rock samples are conglomerate with a high gravel content and strong heterogeneity [18]. For triaxial experiment, the cores taken were 25.4 mm in diameter and 50 mm in length. For the tensile strength test, the cores taken were 25.4 mm in diameter and 13 mm in length. The perimeter of the rock sample was ensured to be smooth, keeping the parallelism of the two end faces within 0.02 mm and the perpendicularity of the section to the axis within 0.05 mm. Ensure the cylinder surface is smooth, and avoid uneven or gravel peeling on the cylinder surface.

2.3. Experimental Approach and Procedures

The temperature of acid flooding test was set as 90 °C based on the formation temperature in the Mahu region; the test pressure was 0.101325 MPa.

The specific experimental steps are as follows: ① carry out experimental tests on the original rock samples, including an XRD mineral composition test, porosity and permeability test, rock mechanics triaxial test, and tensile strength test. Obtain the fundamental physical properties of rock before acidizing. ② Conduct an acid flooding experiment with a fixed rate of 0.2 mL/min. Set up different acidizing parameters, including acid composition, acid concentration, and acid–rock contact time to conduct the acid flooding test. ③ Clean the acidized rock samples, place them for drying, and conduct mineral composition and mechanical parameters tests again for acidized rock samples. A complete study workflow is exhibited in Figure 2 below.

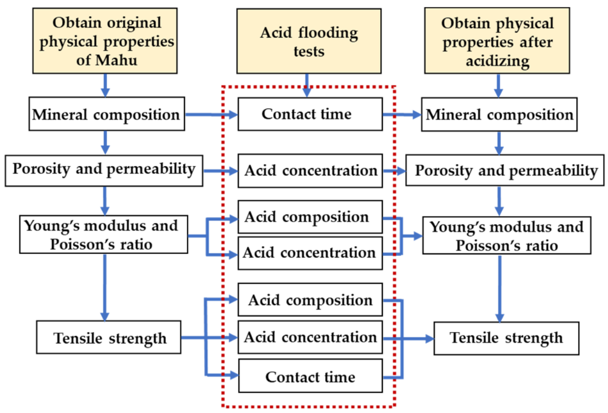


Figure 2. Workflow process chart of acidizing factors study.

The acidizing parameters were designed as follows: ① the mineral composition experiment was conducted in 13 groups of rock samples, including nine unacidified rock samples used to obtain the original mineral content. The acid composition was 6% HF + 15% HCL, and the acid treatment time was set as 30 min, 60 min, 90 min, and 120 min, respectively. ② The porosity and permeability comparative experiments were conducted in 18 groups of rock samples, including 9 groups of unacidified rock samples used to obtain the original porosity and permeability. Others were treated with mud acid, with concentrations of 1.5% HF + 15% HCL, 3% HF + 15% HCL, and 6% HF + 15% HCL. ③ In the triaxial test comparison experiment, 15 groups of rock samples were tested, including 3 groups of unacidified rock samples. Hydrochloric acid and mud acid were selected for treatment. Acid concentrations were set as 15% HCL, 20% HCL, 1.5% HF + 15% HCL, and 6% HF + 15% HCL. In total, 22 tensile strength tests were performed, including 11 groups of unacidified rock samples. First, the effects of hydrochloric acid and mud acid were compared. Next, concentrations of 10% HCL, 15% HCL, 20% HCL, 1.5% HF + 15% HCL, 3% HF + 15% HCL, and 6% HF + 15% HCL were considered. Finally, different contact times of 10 min, 20 min, 30 min, 60 min, and 120 min were considered. Table 1 represents the overall experiment scheme.

Table 1. Experiment scheme.

Experiment	Acid Composition	Acid Concentration	Contact Time	Experiment Groups	Control Groups
Mineral content test	HF + HCL	6% HF + 15% HCL	30 min	1	9
	HF + HCL	6% HF + 15% HCL	60 min	1	
	HF + HCL	6% HF + 15% HCL	90 min	1	
	HF + HCL	6% HF + 15% HCL	120 min	1	
Porosity and permeability test	HF + HCL	1.5% HF + 15% HCL	-	3	9
	HF + HCL	3% HF + 15% HCL	-	3	
	HF + HCL	6% HF + 15% HCL	-	3	
Triaxial rock mechanics test	HCL	15% HCL	-	3	3
	HCL	20% HCL	-	3	
	HF + HCL	1.5% HF + 15% HCL	-	3	
	HF + HCL	6% HF + 15% HCL	-	3	
Tensile strength test	HCL	10% HCL	60 min	1	11
	HCL	15% HCL	60 min	1	
	HCL	20% HCL	60 min	1	
	HF + HCL	1.5% HF + 15% HCL	60 min	1	
	HF + HCL	3% HF + 15% HCL	10 min	1	
	HF + HCL	3% HF + 15% HCL	20 min	1	
	HF + HCL	3% HF + 15% HCL	30 min	1	
	HF + HCL	3% HF + 15% HCL	60 min	2	
	HF + HCL	3% HF + 15% HCL	120 min	1	
	HF + HCL	6% HF + 15% HCL	60 min	1	

3. Mineral Content Variation by Acidizing

The acid composition selection and acid rock reaction rate depend on the mineral composition. Therefore, it is necessary to test and obtain the mineral composition of the target reservoir. Meanwhile, the carbonate rock content determines how to recommend the acid composition. The MiniFlex II benchtop X-ray diffractometer was used for mineral composition testing. Figure 3 illustrates how the apparatus tests.

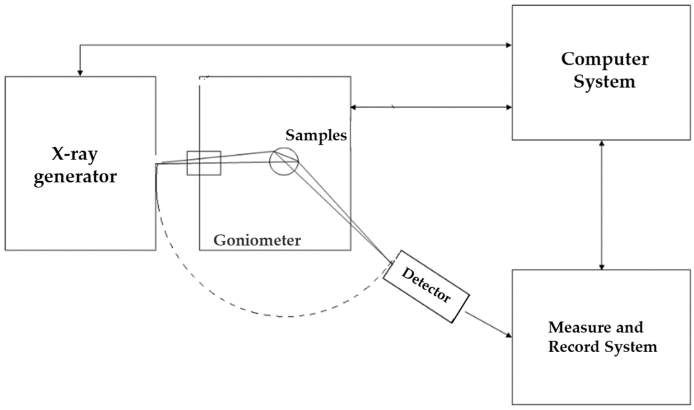


Figure 3. X-ray diffraction structure diagram.

The mineral composition of Mahu was obtained by the test; the results are revealed in Table 2, which contains the original mineral component of Mahu sag, and mineral composition after 60 min acidizing. The original value statistics are shown in Figure 4. As can be seen from the results, the mineral composition of Mahu sag is mainly dominated by quartz with an average content of 46.54%, carbonate rock with an average content of 6.08%, clay with an average content of 8.02%, potassium feldspar with an average content of 13.84%, and plagioclase with an average content of 20.82%. The clay and carbonate rock content are low, and the feldspar content is high at more than 30%. The rock samples were treated with 6% HF + 15% HCL for 1 h; calcite was dissolved completely. Hydrochloric acid cannot dissolve mud or quartz, while hydrofluoric acid reacts not only with carbonate, but also with silicate. Clay and feldspar were dissolved; they were reduced by almost 34%.

Table 2. Mineral composition variation after 60 min acidizing.

Well	Clay (%)		Quartz (%)		Potash Feldspar (%)		Plagioclase (%)		Calcite (%)		Dolomite (%)		Augite (%)	
	Without Acidizing	60 min Acidizing	Without Acidizing	60 min Acidizing	Without Acidizing	60 min Acidizing	Without Acidizing	60 min Acidizing	Without Acidizing	60 min Acidizing	Without Acidizing	60 min Acidizing	Without Acidizing	60 min Acidizing
MA132	8.87	4.44	43	65.76	11.53	5.12	24.13	18.03	4.47	0.00	2.73	0.95	5.27	5.7
MA136	6.23	3.32	47.17	62.45	15.33	10.1	20.65	16.05	-	-	4.95	1.72	5.67	6.36
MA139	8.97	4.55	49.43	67.2	14.67	10.55	17.67	11.68	3.63	0.00	2.47	1.05	3.17	4.97
Average	8.02	4.11	46.54	65.13	13.84	8.59	20.82	15.25	2.7	0.00	3.38	1.24	4.70	5.68

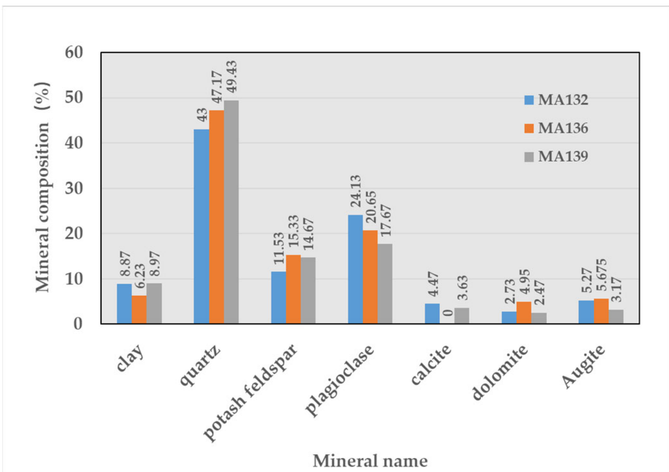


Figure 4. Mineral component distribution in Mahu reservoir.

Figure 5 is a graph which presents the image of rocks before and after acidizing. The rocks were dissolved partially, which can be seen from the edge of the rocks clearly. The micro-cracks and corrosion holes were irregularly distributed over the whole surface of the rocks.

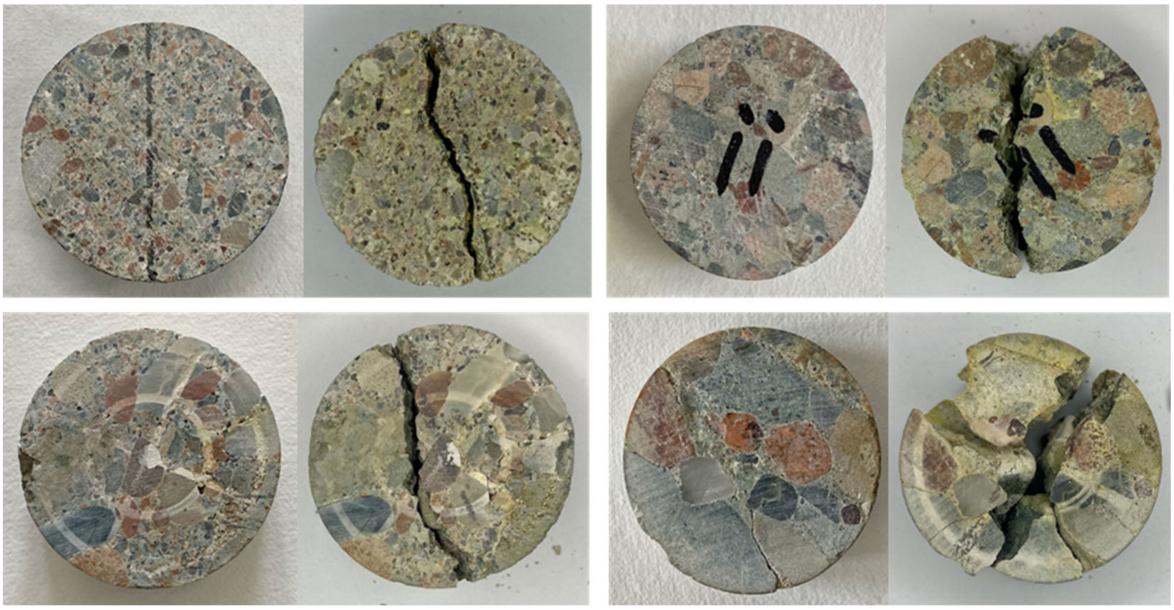


Figure 5. Mineral dissolution by the acid.

Figure 6 shows a mineralogy change section through a rock sample. The acid flowed through and left distinct marks. There are several manifest cracks which can be observed inside the rock after acidizing.

Figure 6. Acid marks and obvious fractures after acidizing.

The rock samples were treated with 6% HF + 15% HCL. We tested and obtained the mineral composition of the rock samples after acidizing at different treatment times to analyze the variation in the carbonate minerals and clay and feldspar minerals.

Figures 7 and 8 reflect variations in the clay, feldspar minerals, and carbonate minerals when the acid treatment time was increased. Under the condition of the same acid concentration, with the increase in the acid treatment time, the content of calcite, dolomite, and other carbonate minerals in Mahu conglomerate decline continuously. After 30 min acidizing, the average content of carbonate rock reduces by 37%. In contrast, the content of quartz sees an opposite trend. The content of clay and feldspar also drop significantly; they drop by 19.7% for 30 min, 34.4% for 1 h, 54.3% for 90 min, and 61.7% for 120 min. Furthermore, the clay minerals grow down more than feldspar. Mud acid can effectively dissolve feldspar and clay. Therefore, the mixture of hydrofluoric acid and hydrochloric acid is chosen to have a better acidizing effect. Mud acid was mainly used in this study, and some experiments considered hydrochloric acid for comparative experiments.

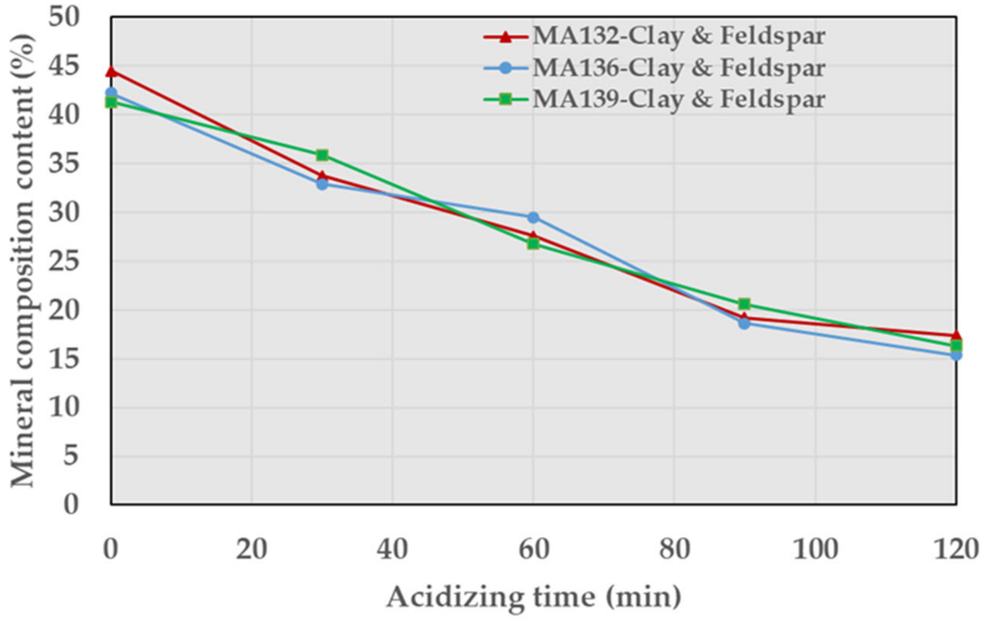


Figure 7. Changes in clay and feldspar minerals of Mahu conglomerate under different acid treatment times.

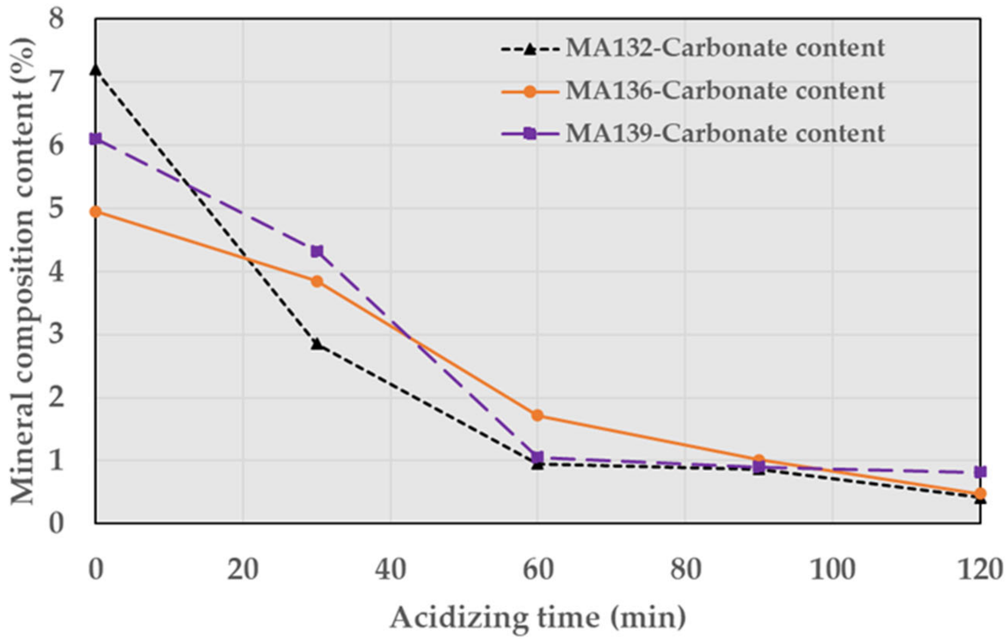


Figure 8. Changes in carbonate minerals of Mahu conglomerate under different acid treatment times.

4. Porosity and Permeability Increase after Acidizing

The volume in which a rock can store fluid and the ability of fluid transport in it are usually characterized by porosity and permeability. The ratio of the pore volume to the total rock volume is defined as porosity [19,20]. Based on the connecting status of pores in rocks, the porosity can be divided into effective and total porosity. Effective porosity is used to measure the volume of interconnected pores in rocks, which was measured in this paper. The quantity, indicating the ease of fluid flow in the medium, is called permeability. The permeability test was conducted by a 38 mm core holder and micro gas flow meter, which is the gas testing method. It is obtained by reading the micro gas flow meter, and the permeability can be calculated by combining the formula. The weight difference calculates the effective porosity before and after the core is saturated. The equations for calculating the permeability and porosity are given below.

$$K = \frac{2P_2Q_0\mu L}{A(P_1^2 - P_2^2)} \times 1000 \tag{2}$$

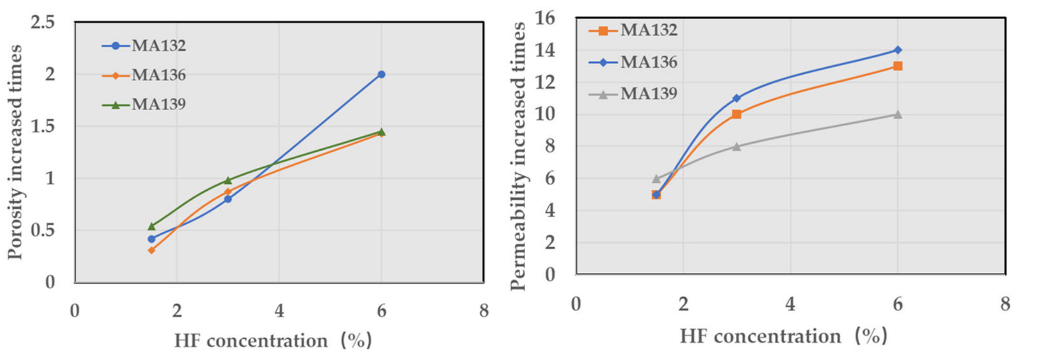
where K is the permeability, 10^{-3} um^2; A is the sectional area of the core, cm^2; L is the rock length, cm; P_1 is the absolute pressure at the inlet of the rock sample, 0.1 MPa; P_2 is the absolute pressure at the outlet of the rock sample, 0.1 MPa; μ is the gas viscosity, mPa.s; and Q_0 is the flow rate at the atmospheric pressure condition, cm^3/s.

$$\phi = \frac{V_p}{V_f} = \frac{V_f - V_g}{V_f} \tag{3}$$

where ϕ is the porosity, %; V_f is the gross rock volume, cm^3; V_p is the rock pore volume, cm^3; and V_g is the aggregate particles volume, cm^3.

The average porosity and permeability of unacidified rock samples were obtained by the tests. The average porosity of the Mahu region is 8.74%, and the average permeability is 4.18×10^{-4} μm^2. At the same time, the control groups were treated with different concentrations of mud acid. After acidizing, the rock samples were tested again to obtain the porosity and permeability.

As can be seen from Figure 9, the curves reflect the change in the trends of rock porosity and permeability after acid treatment. Different acidizing parameters on rock porosity and permeability arouse different results. Figure 9a shows the effect of acid concentrations on the porosity. The porosity is 200% of the original value. Figure 9b represents the effect of acid concentrations on the permeability, which increases by 5 times to 14 times. In general, the porosity and permeability see an upward trend with the increase in the acid concentration, but there is no obvious linear rule.



(**a**) Porosity (**b**) Permeability

Figure 9. Growth multiples under different acid concentrations. (**a**) Growth multiples of porosity. (**b**) Growth multiples of permeability.

5. Triaxial Rock Mechanics Parameter Variation by Acidizing

The triaxial rock mechanics test is carried out with the GCTS RTR-1500 test system. The axial stress is 2000 KN, the maximum allowable confining pressure is 120 MPa, and the maximum pore pressure is 70 MPa. The sample produces longitudinal displacement and transverse displacement under the action of external force. The deformation of the sample can be calculated by the displacement value and the geometric size of the sample. Young's modulus and Poisson's ratio can be calculated by measuring the longitudinal and transverse deformation of the rock sample with a regular shape under axial and transverse pressure. The calculation formula is as follows:

$$E = \sigma_{(50)} / \varepsilon_{h(50)} \tag{4}$$

where E is Young's modulus, MPa; $\sigma_{(50)}$ is 50% of the maximum principal stress difference, MPa; and $\varepsilon_{h(50)}$ is the axial compression strain at $\sigma_{(50)}$, dimensionless.

$$v = \left| \frac{\varepsilon_{\mathrm{d}(50)}}{\varepsilon_{\mathrm{h}(50)}} \right| \tag{5}$$

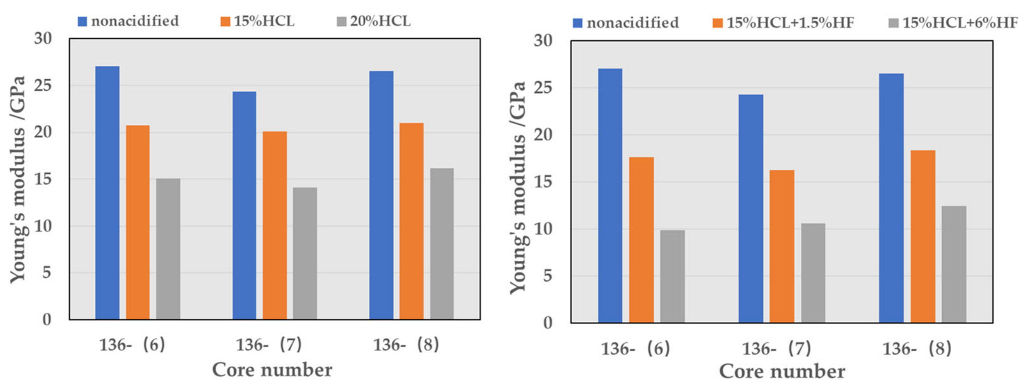
where ν is Poisson's ratio, dimensionless; $\varepsilon_{h(50)}$ is the axial compression strain at $\sigma_{(50)}$, dimensionless; and $\varepsilon_{d(50)}$ is the radial compression strain at $\sigma_{(50)}$, dimensionless.

Under the conditions of reservoir temperature and stress, the confining pressure was set at 25 MPa; the measured average Young's modulus of unacidified cores was 26 GPa. The average Poisson's ratio was 0.29. The triaxial test was conducted again on the rock samples after acidizing with different experiment parameters. The results are shown in Table 3.

Table 3. Young's modulus and Poisson's ratio at different acid concentrations.

	Core Number	Unacidified	15% HCL	20% HCL	15% HCL + 1.5% HF	15% HCL + 6% HF
Young's modulus (GPa)	136-(6)	27.09	20.74	15.06	17.66	9.85
	136-(7)	24.33	20.11	14.12	16.24	10.57
	136-(8)	26.57	21.02	16.17	18.39	12.42
Poisson's ratio	136-(6)	0.28	0.24	0.19	0.21	0.17
	136-(7)	0.32	0.29	0.22	0.25	0.21
	136-(8)	0.27	0.23	0.18	0.21	0.16

The above table provides information about the value changes in Young's modulus and Poisson's ratio. Young's modulus has a similar tendency to Poisson's ratio after the acid treatment. The effects of the acid concentration on Young's modulus and Poisson's ratio are revealed in Figure 10.



(**a**) HCL (**b**) HF + HCL

Figure 10. Influence of acid concentration on Young's modulus. (**a**) Hydrochloric acid treatment. (**b**) Mud acid treatment.

It is apparent from the diagram that Young's modulus witnesses a downward trend after being treated with both hydrochloric acid and mud acid, which declines more significantly after mud acid treatment on the condition of the same acid concentration and contact time.

Figure 11 illustrates how the acid concentration affects Poisson's ratio. There is a slight decrease after acidizing. The decrease in Poisson's ratio after hydrochloric acid treatment is less than that after mud acid treatment.

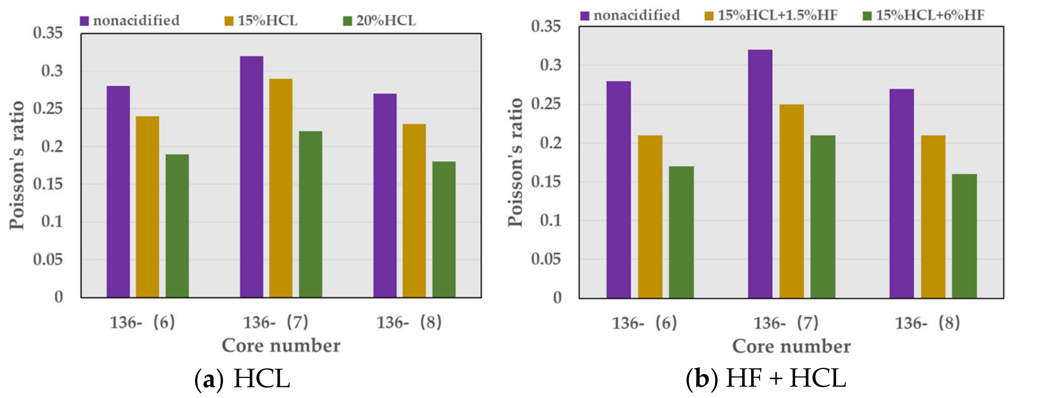


Figure 11. Influence of acid concentration on Poisson's ratio. (**a**) Hydrochloric acid treatment. (**b**) Mud acid treatment.

Figure 12 depicts the impact on Young's modulus and Poisson's ratio. Acid composition has prominent effects on Young's modulus, which shows a sharp drop, whereas Poisson's ratio only has a slight decrease.

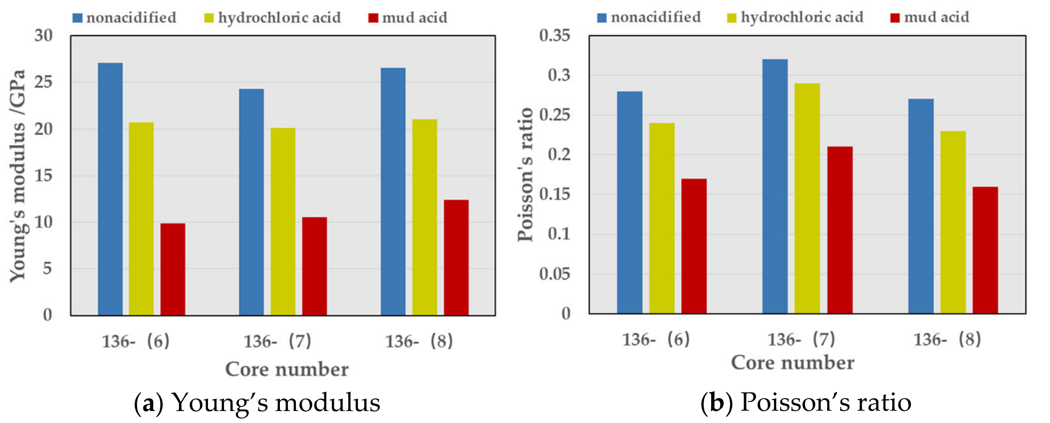


Figure 12. Effect of acid composition on Young's modulus and Poisson's ratio. (**a**) Effect of acid composition on Young's modulus. (**b**) Effect of acid type on Poisson's ratio.

The fracture modes of rocks obtained from the triaxial test are displayed in Figure 13. It shows the fracture morphology of unacidified rocks and acidized rocks under 25 MPa confining pressure. The failure modes of Mahu conglomerate are mainly tensile failure. It can be clearly seen from the graph that the fractures of acidized rocks become more obvious and the number of fractures increases after acidizing. More micro-cracks occurred around the main cracks after acidizing, and the cracks apparently increased compared with the unacidified rocks. The reasons accounting for this phenomenon can be explained that acid dissolves minerals and destroys cement, making the rock easier to crack. It was also found that the fractures propagation of unacidified rocks is mainly around the gravel, and some are wear gravel as well as acidized rocks. Moreover, acidized rocks have more wear gravel than unacidified rocks. The reason lies in that acidizing changed the rock strength.

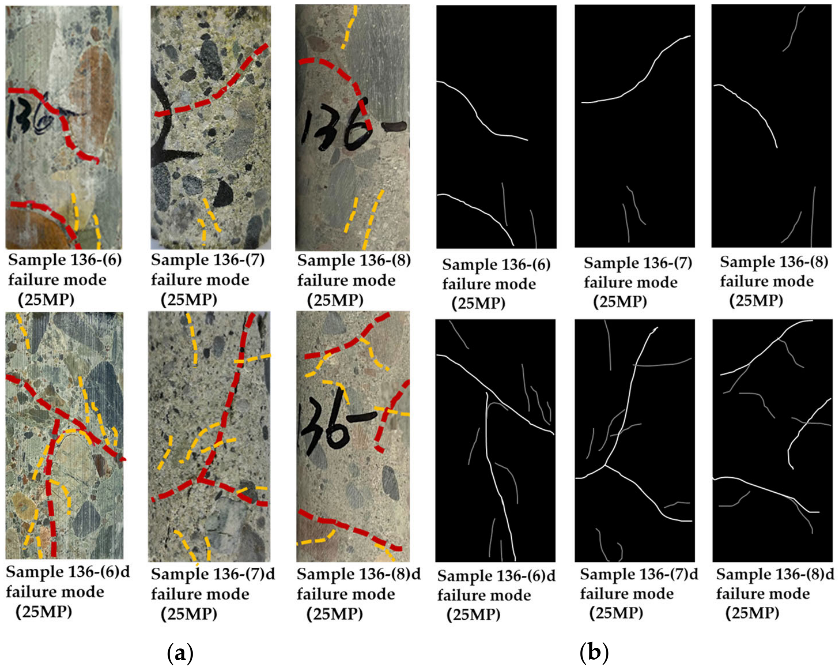


Figure 13. Failure modes of Mahu conglomerate under 25 MPa confining pressure. (**a**) Rock samples, (**b**) failure modes.

The stress–strain curve can reflect the brittle plastic characteristics of the rock. Figure 14 shows the stress–strain curves of the rock samples obtained from the triaxial experiment, and Figure 15 shows three typical stress–strain curves. Brittle rock has a good linear relationship between the stress and strain, which suddenly breaks after stress loading to the peak value. For brittle plastic rock, the initial stress–strain correlation is linear and nonlinear in the later stage. The fracture occurs after the stress reaches the peak value. Plastic rock exhibits nonlinear characteristics under low stress. The stress increases slowly with strain, without an obvious breaking point. The stress–strain curves of the Mahu conglomerate reservoir show that the stress and strain have a good linear correlation in the early stage, showing brittle characteristics. After the stress reaches the peak, there is no sudden fracture and there are nonlinear characteristics before breaking, revealing the plastic characteristics. To conclude, the Mahu conglomerate belongs to brittle plastic rock.

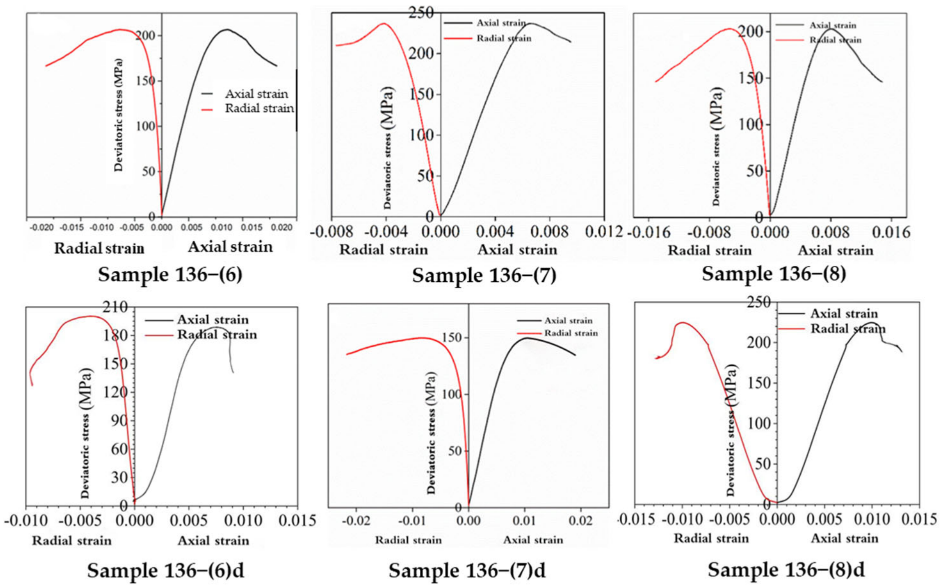


Figure 14. Stress–strain curve to determine the lithological characteristics of Mahu reservoir.

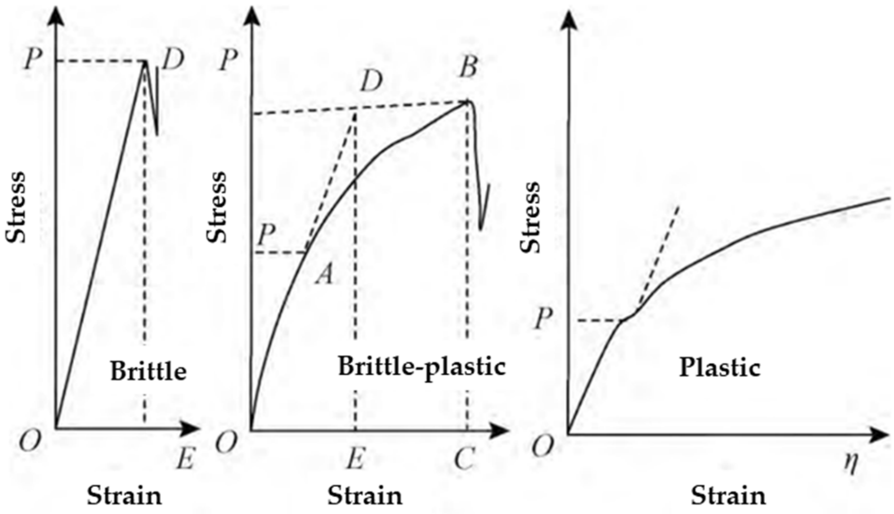


Figure 15. Typical stress–strain curve. (Reprinted with permission from Ref. [21]. 2022, Liu, G.)

6. Tensile Strength Decrease after Acidizing

The Brazilian splitting test is the most common method to obtain the rock tensile strength [22], which is determined by measuring the failure load in the diameter direction and calculating the size of the cylinder sample. The experimental diagram is shown in Figure 16.

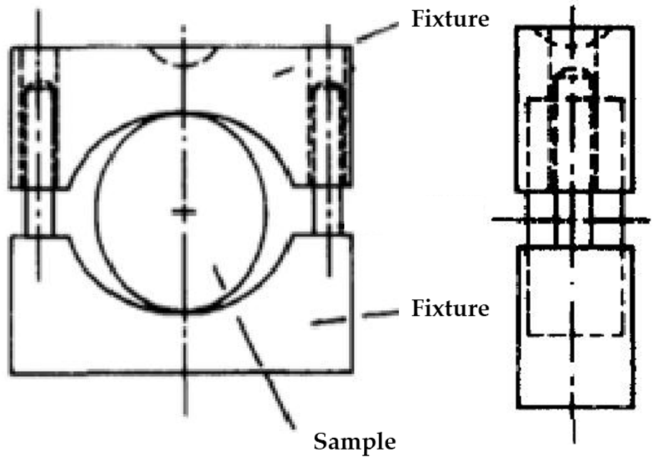


Figure 16. Schematic diagram of splitting experiment.

The tensile strength of the rock sample can be determined by Equation (6).

$$S_t = \frac{2P}{\pi DL} \tag{6}$$

where S_t is the tensile strength of rock, MPa. P is the failure load, N. D is the diameter of the specimen, cm. L is the thickness of the specimen, cm.

First, 11 rock samples as control groups were tested to obtain the original value. Second, the left rock samples were treated with different acids. The rock tensile strength test was conducted again. The influence law of acid composition, acid concentration, and acid treatment time on the tensile strength were obtained. The results are shown in Table 4. The left side is the original tensile strength value, and the right side is the tensile strength after acidizing.

Table 4. Rock tensile strength test results.

Core Number	Tensile Strength (MPa) (before)	Core Number	Tensile Strength (MPa) (after)	Acidizing Time (min)	Reduction Rate (%)	Acidizing Fluid System
136-(1)a	2.57	136-(1)b	0.41	60	84	6% HF + 15% HCL
136-(2)a	4.19	136-(2)b	1.17	60	72	3% HF + 15% HCL
136-(3)a	6.19	136-(3)b	2.02	60	67	1.5% HF + 15% HCL
136-(4)a	3.16	136-(4)b	1.07	60	66	15% HCL
136-(6)a	5.27	136-(6)b	2.69	60	49	12% HCL
136-(5)a	8.32	136-(5)b	4.74	60	43	10% HCL
136-(7)a	4.72	136-(7)b	4.53	10	4	3% HF + 15% HCL
136-(8)a	4.33	136-(8)b	3.59	20	17	3% HF + 15% HCL
136-(9)a	6.83	136-(9)b	4.64	30	32	3% HF + 15% HCL
136-(10)a	5.39	136-(10)b	3.13	60	71	3% HF + 15% HCL
136-(11)a	6.42	136-(11)b	3.08	120	83	3% HF + 15% HCL

As is demonstrated in Figure 17, the reduction rate of tensile strength soared to 84%. The red trendline represents mud acid treatment, and the blue trendline expresses hydrochloric acid treatment. The effect of mud acid acidizing is better and more obvious. The explanation is that the carbonate minerals of the Mahu area is not high, while the feldspar minerals are high. The mud acid can effectively dissolve feldspar, clay, and other minerals, so the damage to the mechanical properties of the rock is more evident.

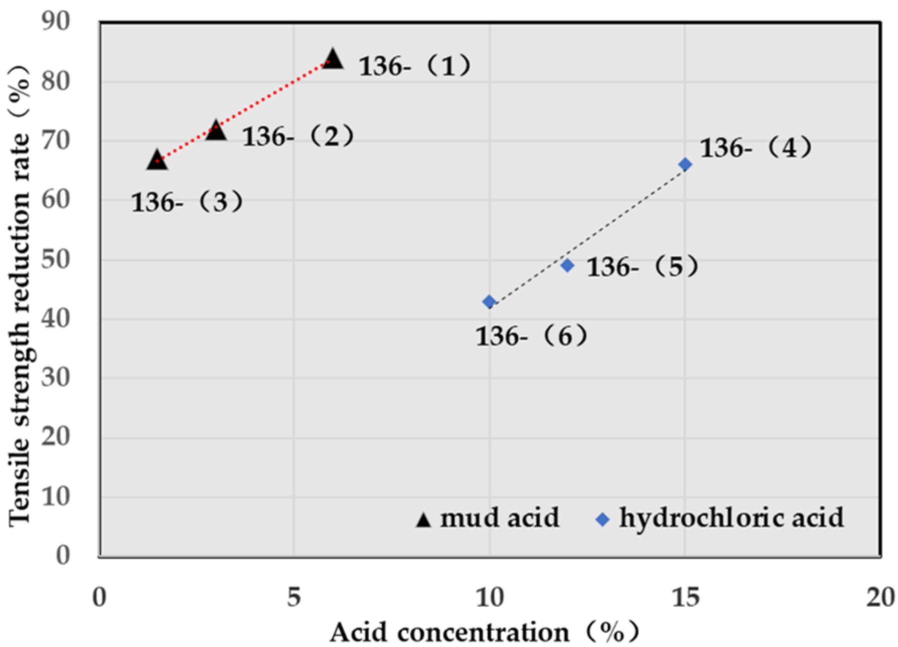


Figure 17. Effect of acid concentration and acid composition on the tensile strength.

Figure 18 represents how the acid treatment time impacts the tensile strength. The longer the acid treatment time is, the more sufficient the dissolution is, and the greater the decline proportion is. Within 60 min of acidizing, the tensile strength decreases sharply, and above 60 min of acidizing, the rate of decrease slows down, indicating that the dissolution is gradually sufficient. Therefore, it is necessary to ensure a certain effective acid–rock contact time to allow the acid to fully dissolve the rock minerals.

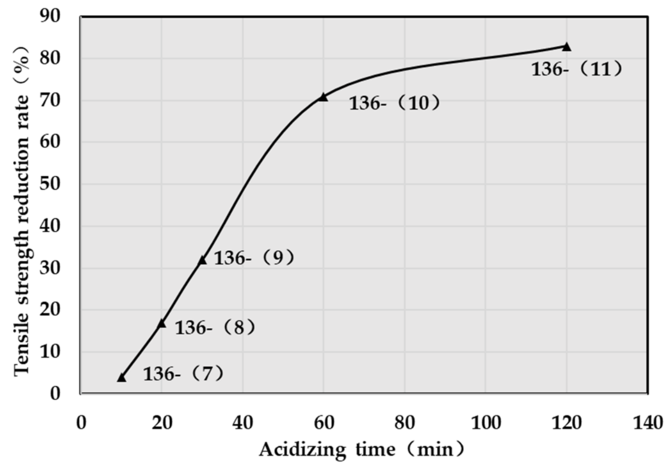
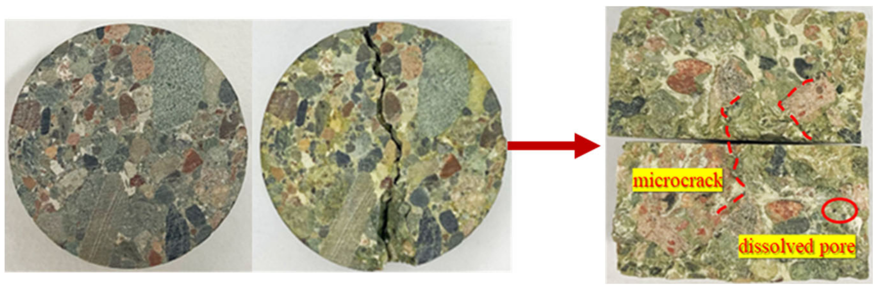


Figure 18. Effect of acid treatment time on the tensile strength.

Figure 19 provides a comparison photograph of a rock sample before and after testing. Figure 19a is the top view of the rock, which contains an unacidified rock sample image on the left and the same rock after acid treatment on the right. It is obvious that the fractures mainly propagate around the gravel. Figure 19b represents a cross section of the same rock sample after acidizing. The whole rock has been treated thoroughly, which can be seen from the color changed inside. The acidizing was sufficient, and micro-cracks and corrosion holes are also clearly observed inside.



(**a**) Top view (**b**) Section plane

Figure 19. Micro-cracks and dissolved pores after acidizing. (**a**) Top view of rock sample before and after acidizing. (**b**) Rock section plane after acidizing.

7. Conclusions

In this paper, through conducting a series of experiments before and after acid treatment, we analyzed how acid dissolution affects rock properties and the effect on fracturing pressure in the Mahu conglomerate reservoir, which provided a theoretical basis for the acid pretreatment of hydraulic fracturing in the Mahu conglomerate reservoir. The following conclusions were obtained:

1. The Mahu conglomerate has a mineral composition of 46.54% quarts, 6.08% carbonate, 34.66% feldspar, 8.02% clay, and 4.70% others. The carbonate content is relatively low, and the clay and feldspar content are relatively high. Contacted by 6% HF + 15% HCl for 1 h, carbonate was dissolved almost, and more than 40% of clay and feldspar were dissolved; other minerals were consumed only a little. Mineral dissolution by the acid was obvious in acid dipping, resulting in a significant property change in the rock, which beneficially promotes breaking the formation in hydraulic fracturing in the Mahu conglomerate reservoir.
2. Acid treatment can increase the porosity and permeability significantly, as the porosity increased by up to 2 times and the permeability increased up to 14 times.
3. After acid treatment, the tensile strength decreased by up to 84%, Young's modulus decreased by up to 63.6%, and Poisson's ratio decreased up to 40.7%

4. Mud acid has a stronger dissolution to the Mahu conglomerate than HCl. A 6% HF + 15% HCl acid composition is recommended for 60 min plus the acid–rock contact time.
5. The stress–strain curves of the Mahu conglomerate showed that the rock is a brittle-plastic type. The mechanism that acid treatment lowers the fracture pressure is that it reduces the tensile strength and increases the permeability. The increased permeability let the fluid pressure propagate into the formation to increase the pore pressure. The raised pore pressure can reduce the fracturing pressure.

In this study, the experiments were conducted with small-size standard core samples. Whether the law can be generalized to the field scale should be validated by field applications. Further study is recommended, such as acid pretreatment modeling and how the acidizing lowers the formation breaking pressure in hydraulic fracturing.

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Data Availability Statement: The raw data required to reproduce these findings cannot be shared at this time as the data also form part of an ongoing study.

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Conflicts of Interest: The authors declare no conflict of interest.

References

1. Wang, S.; Deng, H.; Yu, H.; Zhu, J.; Tian, G.; Lin, Y.; Tang, W.; Guo, C. Rock damage and fracturing pump pressure reduction mechanism of conglomerate reservoirs in Baikouquan Formation of Mahu Sag after acidizing treatment. *Sci. Technol. Eng.* **2021**, *21*, 8841–8850.
2. Yang, Z.; He, R.; Chen, J.; Li, X.; Xie, B.; Meng, J.; Li, W. A Hybrid Model for Selecting Horizontal Candidate Wells for Re-Fracturing of Tight Oil Reservoirs—A Case Study in the Baikouquan Formation, North Mahu Oil Field, Western China. In Proceedings of the SPE Russian Petroleum Technology Conference, Moscow, Russia, 22–24 October 2019.
3. Lu, C.; Ma, L.; Guo, J.; Xiao, S.; Zheng, Y.; Yin, C. A new hydraulic fracturing method for large-thickness conglomerate reservoirs. *Nat. Gas Explor. Dev.* **2020**, *43*, 64–70.
4. Yang, L.; Gao, J.; Chang, S.; Wan, Q.; Liu, C. Study on Fracture Conductivity of Mahu Sandy Conglomerate Tight Oil Reservoir. In Proceedings of the SPE/IATMI Asia Pacific Oil & Gas Conference and Exhibition, Virtual, 12–14 October 2021.
5. Xu, J.; Li, J.; Wu, Y.; Ding, K.; Jiang, H. Exploration and practice of volume fracturing technology in horizontal well of Mahu tight conglomerate reservoirs. *China Pet. Explor.* **2019**, *24*, 241–249.
6. Yun, J.; Qin, G.; Xu, F.; Li, X.; Zhong, N.; Wu, W. Development and utilization prospects of unconventional natural gas in China from a low-carbon perspective. *Acta Pet. Sin.* **2012**, *33*, 526–532.
7. Tan, P.; Jin, Y.; Han, L.; Shan, Q.; Zhang, Y.; Chen, G.; Zhou, Y. Influencing mechanism of acidification pretreatment on hydraulic fracture for deep fractured shale reservoirs. *Chin. J. Geotech. Eng.* **2018**, *40*, 384–390.
8. Wu, Y.; Li, M.; Zhang, J.; Xu, C.; Tian, X. Research and application for fracturing technology of volcanic reservoir in Chagan Depression. *Drill. Prod. Technol.* **2016**, *39*, 60–63.
9. Zou, C.; Yang, Z.; Zhu, R.; Zhang, G.; Hou, L.; Wu, S.; Tao, S.; Yuan, X.; Dong, D.; Wang, Y.; et al. Progress in China's unconventional oil & gas exploration and development and theoretical technologies. *Acta Geol. Sinca* **2015**, *89*, 979–1007.
10. Morsy, S.S.; Hetherington, C.J.; Sheng, J.J. Effect of low-concentration HCl on the mineralogical, mechanical, and physical properties of shale rocks. In Proceedings of the SPE Eastern Regional Meeting, Pittsburgh, PA, USA, 20–22 August 2013.
11. Morsy, S.S.; Sheng, J.J.; Hetherington, C.J.; Soliman, M.Y.; Ezewu, R.O. Impact of matrix acidizing on shale formation. In Proceedings of the Nigeria Annual International Conference and Exhibition, Lagos, Nigeria, 5–7 August 2013.
12. Morsy, S.S.; Sheng, J.J.; Soliman, M.Y. Improving hydraulic fracturing of shale formations by acidizing. In Proceedings of the SPE Eastern Regional Meeting, Pittsburgh, PA, USA, 20–22 August 2013.
13. Li, P. Analytical formula of formation breakdown pressure for horizontal well. *J. Shanghai Univ. Eng. Sci.* **2011**, *25*, 41–45.

14. Wang, B.; Wang, X.; Feng, P. Determination of rock mechanical parameters of reservoir before and after sandstone matrix acidizing. *J. Xinjiang Pet. Inst.* **1999**, 11–14.
15. Wang, X.; Zou, H.; Cheng, X.; Jiang, X.; Zhang, G. Study on Rock Mechanics Properties Before and After Acidizing of Sandstone Matrix. In Proceedings of the Chinese Society for Rock Mechanics & Engineering, Wuhan, China, October 2000.
16. Guo, J.; Xin, J.; Zhao, J.; Li, G. The calculation analysis of decreasing formation fracturing pressure by acidizing pretreatment. *J. Southwest Pet. Univ. (Sci. Technol. Ed.)* **2008**, *30*, 83.
17. Deng, Y.; Xue, R.; Guo, J. The mechanism of high-pressure high-temperature and low permeability acid pretreatment to reduce fracturing pressure. *J. Southwest Pet. Univ. (Sci. Technol. Ed.)* **2011**, *33*, 125–129.
18. Yang, Y.; Li, X.; Wang, Z.; Ju, Y. Influence of Perforation Parameters on Propagation Law of Hydraulic Fracture in Heterogeneous Sandstones. *China Civ. Eng. J.* **2022**, *55*, 1–9.
19. Zhang, L.; Pan, B.; Shan, G. Progress in Experimental Research on Porosity and Permeability of Core Samples. *Prog. Geophys.* **2018**, *33*, 777–782.
20. Zhang, L.; Pan, B.; Shan, G. Experimental Study on Rock Porosity and Permeability under Overburden Pressure. In Proceedings of the Annual Meeting of Chinese Geoscience Union, Beijing, China, 20 October 2014.
21. Liu, G.; Xiao, F.K.; Guo, Z.B.; Chi, X.H.; Jiang, Y.N.; Yu, H.; Hou, Z.Y.; Zhao, R.X. Plastic characteristics of rock under point load. *Sci. Technol. Eng.* **2018**, *18*, 217–222.
22. Deng, H.; Li, J.; Zhu, M.; Wang, R.; Yuan, X.; Luo, Q. Research on Effect of Disc Thickness-To-Diameter Ratio on Splitting Tensile Strength of Rock. *Chin. J. Rock Mech. Eng.* **2012**, *31*, 792–798.