Study on the Performance Degradation of Sandstone under Acidification

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ABSTRACT: In most oilfields, acid fracturing is widely used for oil production. Understanding the relationship between the individual factors (i.e., carbonate rock types, acid rock reaction kinetics, and deterioration of rock mechanical properties) can provide practical guidelines that can be used for the design and optimization of acid fracturing operation. This paper takes hydrochloric acid, acetic acid, and citric acid as the main research objects and carries out acidification experiments on sandstone in Changqing Oilfield, China. In addition, the effects of tribasic, dibasic, and monobasic acids on the mechanical properties of sandstone were studied. Results show that in this study area, the most obvious effect was seen with the use of dibasic acids (hydrochloric acid + acetic acid), which effectively reduced the sample quality, uniaxial compressive strength, and elastic modulus. Citric acid and Mg promote the conversion of amorphous calcium carbonate to high-crystallinity calcite, forming a white precipitate. Furthermore, it is found by scanning electron microscopy analysis that experimental results of this study can help to better understand the erosion, failure state, and failure mechanism of different acid types on sandstone, which may provide certain references and guidelines for sandstone acid fracturing oil production.

INTRODUCTION

The focus of the development of low-permeability reservoirs is mainly to maximize the output of a single well while reducing development costs and improving the development benefits of using low-permeability reservoirs. Acid fracturing is currently widely used in oilfield mining to significantly improve recovery. While acidification can be used in both carbonate reservoirs and sandstone reservoirs, the rock mineral structure and mineral chemical composition of sandstone reservoirs are more complicated than carbonate rocks. In 1973 and 1975, Labrind and Lund et al. proposed that the dissolution rate of dolomite at 25 °C will be limited by the surface reaction; however, under the condition of 100 °C, the dissolution rate of dolomite will reach the limit regardless of whether the experiment uses high speed or low speed. Fogler et al. found that the dissolution of terranic acid and feldspar is caused by HCl, which has a catalytic effect on hydrogen fluoride (HF) and can improve the dissolution of minerals.

Fogler and Hekim found that minerals in sandstone have different degrees of solubility in an acidic soil system. In addition, because HF has the dual nature of ionization and association, other forms such as H+, F−, HF, and HF2− can be found in solution. Thus, Fogler et al. proposed that the first step in the melting of HF and siliceous minerals is F− chemistry adsorbed on the siliceous surface. Zhao et al. proposed the exact opposite of the study of Fogler et al. This study reported that when HF reacts with siliceous minerals, it is not the fluoride ion that plays the leading role but the HF molecule. Hartman et al. studied the reaction of various minerals with HCl system and HCl/HF system acid solutions. By detecting the concentration of aluminum, iron, magnesium, sodium, and other metal ions in solution after the reaction of various minerals with an acid solution, the corresponding results were obtained. The dissolution kinetic parameters of minerals and the experimental study of kaolinite were conducted under the same conditions. After comparing the experimental results, the obtained parameters are applied to the sandstone acidification simulation using the geochemical model. Zhao et al. designed a variety of acid solutions to dissolve single minerals and found that 8% HCl + 1.5% HF system acid solution has a higher corrosion rate of chloride at 90 °C,
23.13%; polyhydrogen acid. The corrosion rates of the system to kaolinite, montmorillonite, and chlorite are relatively high, respectively, 27.74, 23.41, and 13.25%. Among them, the corrosion rate of montmorillonite increases obviously with the increase in polyhydrogen acid concentration, with an average that is greater than 25%. Li et al. concluded that high temperature, high pressure, and a strong acidic environment will accelerate sandstone destruction. The research period of this study ranged from 6 h to 12 days. Due to the influence of the Corona Virus Disease 2019 (COVID-19), the research period was extended to 93 days, and it was unexpectedly concluded that the citric acid action promoted the precipitation of regular carbonate in the sandstone, resulting in calcium crystals, and an experimental analysis of the reasons for precipitation.

Simple hydraulic fracturing has limited damage to the rock, resulting in a relatively single fracture initiation and a fixed direction. The use of acid for fracturing will destroy the internal structure of the rock through chemical reactions, thereby creating more cracks, and the cracks are complex and not fixed in direction so that more oil can be extracted (Figure 1). For rock samples in different blocks, different acid types are needed. In this study, seven experimental groups and a control group were set up to analyze the chemical reaction between acid and rock. Finally, it is concluded that the mixed use of hydrochloric acid and acetic acid is most suitable for this block. When compared to the previous work, this work, at first, studied the influence of monobasic acid, dibasic acid, and tribasic acid on the acidification of sandstone and concluded that dibasic acid (hydrochloric acid + acetic acid) has provided the best effect. Further, ICP-OES analysis of the ion composition and concentration in the solution after the reaction solidly revealed the trend of the acid rock reaction. However, this analysis method is not generally used. Finally, it was found that the coexistence of citric acid and magnesium will promote the formation of amorphous calcium carbonate (ACC). Further, it is transformed into calcite with high crystallinity and forms a white precipitate, which is irreversible due to underground environmental pollution factors. Thus, this method is not proposed for the acidification in oilfields.

First, the sandstone used in this study is taken from a depth of 2154 m in China Changqing Oilfield, Yuan 284 block. The acid solution used in the experiment consists of hydrochloric acid, acetic acid, and citric acid. Further, seven experimental groups and one control group are set (see Table 2). Corrosion of a core with a diameter of 25 mm and a length of 50 mm at room temperature for 93 days.

## RESULTS AND DISCUSSION

### Change in the Mass of Sandstone Samples Subjected to Acid Treatment

The calculation equation based on the mass loss rate was used and is as follows

\[
m = \frac{m_1 - m_0}{m_0} \times 100\%
\]

where \( m \) is the mass loss rate, \( m_1 \) is the mass before acid etching, and \( m_0 \) is the mass after acid etching.

The calculated mass loss for all samples is shown in Figure 2a. Acid etching can be clearly seen from the appearance of the cores before and after etching. There is no obvious change in groups 1–3, 5, and 6, but the cores in groups 4 and 7 have been obviously damaged and white substances have been precipitated. From the appearance analysis, the greatest degree of damage to the core can be seen in the treatment of acetic acid + citric acid and in the presence of citric acid alone. As shown in Figure 2b, the two groups with the most mass loss were 3 and 5 followed by 1, 4, 6, and 7. Group 2 resulted in a reddish brown color. This is because H+ in the acid liquid reacted with illite and chlorite to form Fe3+, which further reacted with oxygen to form Fe3O4, and finally reacted with acetic acid to form a colloid with Fe3+, resulting in a reddish brown color. The acid etching reaction was fully confirmed by the absence of a precipitate in the presence of strong acid, and only Ca3(PO4)2 will be consumed quickly. In the presence of citric acid, calcium carbonate crystals (aragonite) will be precipitated, causing the core to burst.

### Analysis of Mechanical Properties, Specific Surface Area, and Porosity

The uniaxial compressive strength of the samples after acid etching was tested, and the stress–strain curves of sandstone samples exhibited three typical stages: compaction, elastic, and yielding (Figure 3a). The strength and stiffness of the samples after acid etching are significantly smaller than those of the untreated samples. It can be found that the stress–strain curves of the samples decreased after different acid etching. Compared with the untreated samples, the compaction phase becomes longer, the elastic phase becomes shorter, and the strain value at the peak load point is also not the same. Sample 7 was damaged by complete acid etching, and thus, the uniaxial compressive strength was not measured. In addition, the bottom of sample 4 had been damaged by acid etching and the compressive strength was low. The results indicate that group 5 (hydrochloric acid + acetic acid) showed the most obvious decrease in mechanical properties.

As shown in Figure 3b, the BET test results show that group 5 has the highest specific surface area and porosity after (hydrochloric acid + acetic acid) treatment, which proves that the acid etching effect is the most pronounced. The second most pronounced effect was found in group 3, which proves that the erosion effect of acetic acid alone on the core is also ideal. Groups 4 and 7, which contained citric acid, show no

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**Figure 1.** (a−d) Simplified diagram of oil reservoir production: (a) hydraulic fracturing, (b) acid fracturing, (c) hydraulic fracturing fracturing-oil production, and (d) acid fracturing-factoring-oil production.
obvious changes in the specific surface area and porosity. The results indicate that in acidic solutions, protons promote the displacement reaction of intergranular cations, thereby inducing the formation of larger pores in the mineral and loosening the structure. Overall, the main process of water−rock chemical interactions is the gradual dissolution of clay minerals, which increases the porosity of the rock mass. It can be seen from Figure 4 that the physical adsorption and desorption curves of group 5 are the most obvious, indicating that the porosity is the largest.

**SEM Analysis.** An obvious Imon mixed layer can be seen for the control group from the SEM results (Figure 5a). As shown in Figure 5b, the surface is covered with more citric acid, which hinders the acid rock reaction to proceed further. Calcium carbonate whiskers that have been generated can be clearly seen in Figure 5e, and after Figure 5h was analyzed, it
was clear that acetic acid + citric acid had little effect on the acid rock reaction. It can be concluded from the energy spectrum that the contents of C and O are extremely high compared with other treatments (Figure 5i). It is a calcium carbonate whisker generated without whiskers, and the length is about 20−80 micrometers. The sample liquid after the number reaction showed pH = 7 by pH measurement.

The calcium carbonate whisker reaction equation is as follows:

$$\text{CaO} + \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2$$

$$\text{Ca(OH)}_2 + \text{Mg}^{2+} \rightarrow \text{Mg(OH)}_2 + \text{Ca}^{2+}$$

$$\text{Mg(OH)}_2 + \text{Ca}^{2+} + \text{CO}_2 \rightarrow \text{Mg}^{2+} + \text{CaCO}_3 + \text{H}_2\text{O}$$

The experiments of Rodriguez-Blanco et al. showed that pH and Mg have an important influence on the pathway and mechanism of the conversion of amorphous calcium carbonate (ACC) to crystalline CaCO$_3$. The neutral initial pH or the

Figure 5. (a−i) SEM-EDS analysis diagram after acid etching: (a) unprocessed, (b) tribasic acid, (c) HCl, (d) acetic acid, (e) citric acid, (f) HCl + acetic acid, (g) HCl + citric acid, (h) acetic acid + citric acid, and (i) precipitate.
The presence of Mg in the solution will prompt the system to develop in the direction of ACC directly crystallizing into calcite. Conversely, a higher initial pH value promotes the formation of metastable intermediate spherulites, which drives the system toward calcite through the second dissolution-recrystallization step. Mg can improve the stability of ACC and inhibit the crystallization of ball stone, which is beneficial to the direct conversion of ACC to calcite.

**Analysis of ICP-OES.** The ICP-OES elemental composition and content of the acid solution after the core erosion were measured. As shown in Figure 6a, the newly added group 9 was the white material precipitated from core samples 4 and 7. Concentrations of Mg, Al, Fe, and K all show that group 1 has the least content, the order is 2 > 3 > 4 and 5 > 6 > 7, and the Si element content is basically the same. The related reaction of feldspar mineral and acidic solution is as follows:

\[
\text{Na}_2\text{K}_2\text{AlSi}_3\text{O}_8 + 4\text{H}^+ + 4\text{H}_2\text{O} \rightarrow 3\text{H}_4\text{SiO}_4 + \text{Na}^+ + \text{Al}_3^+ \\
\]

\[
\text{CaAl}_2\text{Si}_2\text{O}_8 + 8\text{H}^+ \rightarrow 2\text{H}_2\text{SiO}_4 + \text{Ca}^{2+} + 2\text{Al}_3^+ \\
\]

Calcite easily reacts with the acidic solutions as follows:

\[
\text{CaCO}_3 + 2\text{H}^+ \rightarrow \text{Ca}^{2+} + \text{H}_2\text{O} + \text{CO}_2 \uparrow \\
\]

The Si element content was measured in all samples and showed that quartz exhibits a slight hydrolysis effect:

\[
\text{SiO}_2 + 2\text{H}_2\text{O} \rightarrow \text{H}_4\text{SiO}_4 \\
\]

A small amount of biotite and muscovite may react with acidic solution as follows:

\[
\text{KAl}_3\text{Si}_3\text{O}_{10}(\text{OH})_2 + 10\text{H}^+ \rightarrow 3\text{H}_2\text{SiO}_4 + \text{K}^+ + 3\text{Al}_3^+ \\
\]

Elemental analysis showed that feldspar, mica, and calcite in the sandstone reacted with H⁺ in acidic solution, indicating that the sandstone mineral composition used in this article reacted with H⁺ in acidic solution in chlorite than the chemical reaction in quartz and acidic solution more obvious. As shown in Figure 6b, the Ca²⁺ content of group 1 is relatively low because the citric acid is limitedly attached to the rock surface, preventing further the reaction of H⁺. Group 5 shows a relatively high ion content, indicating that hydrochloric acid and acetic acid both react with minerals in the sandstone, although the cores used in groups 4 and 7 are the most damaged due to the formation of precipitated substances by weak acids. Cracks and pipeline blockages are caused by the construction of a site, and thus, it is not recommended to use them separately in construction. The results show that the sandstone damage is causally related to the acidity of the infiltrating fluid, and similar results have been obtained under various acidic conditions.

**Figure 6.** (a) Composition and content of ions in the solution after acid etching. (b) Content of the Ca²⁺ ion in the solution.

**Figure 7.** (a, b) XRD analysis of the core after acid etching: (a) experimental groups 1−4 and control group 8 and (b) experimental groups 5−7, control group 8, and white precipitation (group 9).
chemical corrosion in water–rock interactions.\textsuperscript{18} In terms of sandstone composition, it contains a high proportion of clay minerals (montmorillonite, illite, chloride, etc.), which expand and contract with hydration and water loss and are easy to develop in combination with structural and weathered cracks. In addition, the three dominant clay minerals in the sandstone are silicate minerals; the acidification of sandstone in this block is suitable for the joint use of hydrochloric acid and acetic acid.

**XRD Analysis.** XRD analysis resulted in groups 2–8 all showing a significant decrease in the peak intensity corresponding to the chlorite, which proved that the acid had effectively eroded the core (Figure 7). Group 1 had the smallest decrease in sensitive minerals. It has also been verified that in the monobasic acid, group 4 has the least remaining sensitive minerals, but at the same time, it also generates new white precipitated materials. The dibasic acid performs best in group 7 followed by groups 6 and 5. The same XRD analysis was performed on white precipitated materials (group 9 in Figure 7), and a more obvious aragonite crystal peak appeared at 68°, proving that the precipitated crystal was a calcium carbonate crystal.\textsuperscript{19}

### SUMMARY AND CONCLUSIONS

In this study, a series of uniaxial compression, BET, SEM-EDS, ICP-OES, and XRD tests were performed on tight sandstone samples corroded by different types of acids. Destruction characteristics including mass, porosity, specific surface area, uniaxial compressive strength, ionic components in acid solution, and acid rock reaction failure mechanisms were investigated. The microstructure and damage mechanism of sandstone after multiacid erosion were systematically studied. The damage to morphology and the mechanism depend on the type of acid and the rock mineral composition. According to the experimental results, the following three major conclusions can be drawn.

1. The sandstone samples treated with different acid solutions showed a decrease in quality and mechanical properties. The hydrochloric acid and acetic acid test group having the highest porosity, specific surface area, and mechanical properties was significantly weaker than the untreated samples.

2. As the strength of the acid solution changes from strong to weak, the leaching amount of alkaline cations in the sandstone increases. This is mainly because H\textsuperscript{+} not only acts as a reactant but also provides energy for physical and chemical weathering.\textsuperscript{16} Compared with monovalent cations, the leaching of divalent cations (Ca\textsuperscript{2+} and Mg\textsuperscript{2+}) in sandstones is largely limited by the environmental pH. The calcium and magnesium contents in group 5 are 9.05 and 15.66 times those of the control group, respectively, and the aluminum and iron contents are 11.71 and 12.53 times those of the control group, respectively. However, the K\textsuperscript{+} in the sandstone does not change much, mainly because the potassium feldspar is difficult to be acidified and destroyed.\textsuperscript{20–25}

3. SEM-EDS results show that citric acid promotes the precipitation of regular aragonite crystals of calcium carbonate with a length of 20–80 micrometers. It is recommended not to add citric acid in acid pressure treatment. It is recommended to use hydrochloric acid and acetic acid in the acid pressure of this test block.

The main advantage of this study is that the most commonly used acid is compounded and optimized, and the acid solution suitable for the acid fracturing system used in the Yuan 284 block of Changqing Oilfield is obtained. Further, it is aimed at the ion exchange of different acid solutions and rock reactions. Performed ICP-OES, XRD, and SEM-EDS characterization and analyses revealed that the best option is the mixture of hydrochloric acid and acetic acid. Moreover, the presence of citric acid will promote the formation of white precipitates, pollute the underground environment extensively, and block the crude oil pipelines. The limitations of this study are as follows: (1) The obtained sandstone samples did not reflect the entire area as they are not taken from all the areas due to the difficulty of coring in the entire Changqing Oilfield. (2) The developed (indoor) experimental pressure does not correspond (not able to attain) fully to the underground confining pressure. Thus, the number of samples is increased and tests are carried out under high temperature and high pressure. The test is repeated.

The acid rock reaction mainly produces precipitation materials such as iron precipitation, calcium precipitation, and Na salt and K salt precipitation. When the reservoir contains minerals such as chlorite, the acid will chemically react with the minerals to form a precipitate. For example, in experimental group 3, calcareous precipitation is formed mainly from carbonate minerals such as dolomite and calcite, but it will be dissolved with the further addition of strong acid (hydrochloric acid), and because of this, precipitation will not affect the entire acid fracturing process. The white precipitates obtained in experimental groups 4 and 7 are amorphous calcium carbonate (ACC) precipitates, which will seriously threaten the underground environment and block the wellbore. Therefore, citric acid should not be used for acid fracturing during field application.

When hydrochloric acid, acetic acid, and citric acid are compared, citric acid has the strongest calcium ion chelating ability. The reaction temperature in this study is room temperature, which is highly favorable for the chelation of calcium ions. It is found that the white precipitates generated in experimental groups 4 and 7 will cause great damage to the formation after acid pressure and will severely affect the future work. Therefore, citric acid should not be used in field applications. Acetic acid also has the same complexation effect on iron ions, so experimental group 5 (hydrochloric acid + acetic acid) has the best effect without precipitation and will not cause any damage to the optimal group.

### MATERIALS AND METHODS

**Materials Used.** Rock Samples. The core samples were taken from the Yuan 284 block of Changqing Oilfield, China. The lithology is mainly fine-feldspar sandstone. The detrital components are mainly feldspar (average, 34.82%) and quartz (average, 29.33%). The cement type was made mainly from carbonate minerals such as dolomite and calcite, with the main components being iron calcite (average 2.7%) and iron carbonate (1.5%). The core sample was taken from the Yuan 284 block of Changqing Oilfield of the crude oil pipelines. The limitations of this study are as follows:

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**Acidizing Fluids.** In this paper, the impact of polycarboxylic acids on the performance of sandstone was studied. Triacids (hydrochloric acid, acetic acid, and citric acid correspond to experimental group 1), dibasic acids (hydrochloric acid + acetic acid, hydrochloric acid + citric acid, and acetic acid +
Table 1. Sandstone Rock Physical Properties, Main Mineral Composition, and Burial Depth

| sample number | major mineral composition | sampling depth |
|---------------|---------------------------|----------------|
| 1             | illite (K) + Al(Fe3+)Mg([Si-Al]-O)n + H2O; clorite | 2154.94 |
| 2             | (Mg, Fe) Al [(Si-Al)]2(OH)2; montmorillonite | 2154.87 |
| 3             | Al[(Si-Al)]2(OH)2 feldspar xAl2O3, x = Na and K | 2154.53 |
| 4             |                                       | 2154.89 |
| 5             |                                       | 2154.90 |
| 6             |                                       | 2154.54 |
| 7             |                                       | 2154.60 |
| 8             |                                       | 2154.50 |

citric acid correspond to groups 5–7, respectively), and monobasic acids (hydrochloric acid, acetic acid, and citric acid correspond to the experimental groups 2–4, respectively) and the control group 8 for a total of eight experimental groups. The specific acid ratio and concentration are shown in Table 2. Table 3 lists the main mineral content of the samples used in the main experiments.

Table 2. Acid Ratio and Concentration

| group | acid type and concentration ratio |
|-------|----------------------------------|
| 1     | 2.5% HCl (30%) (83.25 mL) + 6% acetic acid (24 mL) + 1% AC (4 mL) + 138.75 mL of H2O |
| 2     | 2.5% HCl (30%) (83.25 mL) + 166.75 mL of H2O |
| 3     | 6% acetic acid (24 mL) + 226 mL of H2O |
| 4     | 1% AC (4 mL) + 222 mL of H2O |
| 5     | 2.5% HCl (30%) (83.25 mL) + 6% acetic acid (24 mL) + 213.75 mL of H2O |
| 6     | 2.5% HCl (30%) (83.25 mL) + 1% AC (4 mL) + 218.75 mL of H2O |
| 7     | 6% acetic acid (24 mL) + 1% AC (4 mL) + 222 mL of H2O |
| 8     |                                       |

Testing Equipment and Test Procedures. Acid Erosion. The seven cores were dried in an oven at 120 °C for 24 h, and the mass was weighed and recorded. The samples were then added them to wide-mouth jars with the corresponding acid concentration, capped with stoppers, and placed in a dark place at room temperature. In the liquid erosion experiment, because of the Corona Virus Disease 2019 (COVID-19), the research period was extended to 93 days. After 93 days, the core was taken out and placed in an oven to dry at 120 °C for 24 h. The weight was weighed again and recorded.

Mechanical Property Tests. The mechanical properties of the carbonate rocks were measured by using the RTR-1000 rock mechanics servo testing system manufactured by GCTS Company. Uniaxial compression tests were run at room temperature, and the deterioration degree of the mechanical properties of carbonate rocks was determined under acidified conditions.

To analyze the degradation mechanism of sandstone mechanical properties under acidizing conditions, scanning electron microscopy (SEM-EDS) images were used to identify the composition and type of sandstone microcrack fillers and cement. A surface scan analysis was conducted, as well as the composition of precipitated materials. After the core erosion was diluted with water, the acid solution had a solvent ratio of 1:300, and the elemental composition and content determined by inductively coupled plasma emission spectrometry (ICP-OES) were used to further analyze the impact of different acids on the composition of sandstone. A BET analysis of the change in the specific surface area and porosity after acid etching was conducted, and finally, an X-ray diffraction (XRD) analysis of the crystalline form of the core after acid etching was conducted to further corroborate the degradation mechanism.

Table 3. Main Compositions of the Experimental Core

| ingredient | quartz | feldspar | interstitial |
|------------|--------|----------|-------------|
| content (mass score, %) | 35.9  | 24.3    | 10.54       |
| clay minerals (relative content, %) | ilite | kaolinite | chlorite | illite mixed layer |
| content | 27.5 | 14.8 | 31.7 | 17.6 |

Table 3. Main Compositions of the Experimental Core
selection for matrix-stimulation treatments. SPE Prod. Oper. 2006, 21, 194–204.

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