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Simulation Experiment of High-Temperature and High-pressure Supercritical CO2-Saline Water-Rock Reaction in Deep Volcanic Rocks in the Southern Part of Songliao Basin

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ABSTRACT

In order to study the effect of high-temperature and high-pressure CO_2 on the physical properties of deep volcanic rock reservoirs in the southern Songliao Basin, a closed-system temperature and pressure co-controlled water-rock reaction simulation method was used to simulate formation temperatures (120°C, 130°C, 140°C) and formation pressures (20MPa, 60MPa) conditions, 6 groups of supercritical CO_2-water-rock reaction experiments were carried out. The pore characteristics of the reservoir rock samples before and after the reaction were analyzed using QEMSCAN and scanning electron microscopy to determine the modi-fication of reservoir properties due to the water-rock reaction after CO_2 injection into deep volcanic reser-voirs. The experimental results show that the water-rock reaction reaches equilibrium after 96 hours. Super-critical CO_2 has different dissolution effects on rock samples at different temperatures and pressures, with a maximum dissolution rate of 0.2803 and a maximum increase in porosity of 5.18%. Supercritical CO_2 dis-solved in water forms an acidic environment that has a strong acid etching and remolding effect on rock samples, indicating that the high-temperature, high-pressure supercritical CO_2-water-rock reaction is initially dominated by calcite dissolution, followed by dolomite and feldspar minerals dissolve slowly, and quartz minerals are not significantly altered. This experiment shows that selecting representative samples to carry out simulated water-rock reaction experiments under deep formation conditions is an effective and scientific reservoir modification method. In the current context of high cost, high risk, and high investment in deep oil and gas exploration, the results of the high temperature and high-pressure supercritical CO_2-water-rock reac-tion experiment have essential reference value for the next step of deep reservoir exploration and develop-ment.

KEYWORDS

CO_2; Water-rock reaction; Rock microanalysis; Volcanic gas reservoirs; Songliao Basin First Section.

1. INTRODUCTION

The study area is located in the central uplift belt of the southern section of the Songliao Basin, adjacent to the hydrocarbon generation trough. The volcanic structural gas reservoir of the Yingcheng Formation has a good combination of source, reservoir and cap, and the hydrocarbon generation time is well matched with the tectonic formation period, which has good exploration and development potential[1, 2].

The upper part of the Yingcheng Formation is a large section of tuff (local gravel), the middle and lower part is a large section of rhyolite, locally sandwiched with thin-layer tuff, in-situ dissolution breccia and volcanic breccia. The reservoir thickness is between 206~374m, and the gas logging porosity is generally between 5~9%, with an average of 7.3%. It has the characteristics of deep burial, high temperature and high pressure, tight reservoir, low porosity and low permeability, and strong heterogeneity. The reservoir lithology is dominated by silt and fine sandstone, and the maturity of rock minerals is high. Quartz accounts for 28~40%, feldspar accounts for 27~31%, and debris accounts for 29~35%.

The rock debris is mainly composed of volcanic rock particles with a particle size of 0.25~0.5mm, moderate weathering degree, good sorting, and sub-angular-sub-round shape. Quartz increased development, calcite, iron calcite pore cementation, distribution is more uniform, individual debris was completely replaced. The process of reservoir stimulation is very difficult. Therefore, it is very important to study the effect of supercritical CO_2 on reservoir dissolution. [1, 3-7]

Critical CO_2 usually refers to the condition that the temperature is greater than 31.6 °C and the pressure is greater than 7.29MPa (Fig 1). CO_2 presents a supercritical state, with high density (about 0.7g/ml) and low viscosity[31].

The CO_2 injected into the underground is dissolved in water, which reduces the pH value of the formation water and releases H+ ions, resulting in the dissolution of various minerals in the rock, thus affecting the composition of the formation water and changing the porosity of the rock to a certain extent[8-11].

At present, most scholars focus on the geochemical reaction between CO_2, water, sandstone reservoir or carbonate reservoir, and carry out many CO_2-water-rock reaction experiments. For example, Wigand et al. [12-23] carried out CO_2 water-rock reaction experiments for sandstone under different temperature and pressure conditions. Combined with scanning electron microscopy, casting thin sections, whole rock diffraction and other analytical and testing methods, it is confirmed that dissolution is an important factor in reservoir quality improvement, and reveals the effect of CO_2 filling on the formation process of tight sandstone gas reservoirs.

Egermann et al. [24-30] carried out water-rock reaction experiments in carbonate rocks. Most of the experiments reproduced the interaction process between carbonate rocks and CO_2 acid solution dissolved in water under real reservoir temperature and pressure, and enhanced the understanding of reservoir dissolution transformation effect.

Many studies have shown that CO_2-water-rock reaction can improve the conditions of oil and gas reservoirs, but there are few studies on the interaction of supercritical CO_2-water-rock in volcanic reservoirs. Liang et al. [32, 33] believed that the primary pores formed after the cooling of volcanic rocks are less affected by destructive diagenesis, and their physical properties do not change with the change of burial depth. Compared with other types of reservoirs, the reservoir capacity of volcanic rocks pays more attention to the formation of secondary pores.

The reservoir environment and fluid properties vary significantly across different regions, leading to inherent limitations in the results of water-rock simulation experiments. Consequently, obtaining a comprehensive understanding of these interactions remains challenging. To address this research gap, this study focuses on the core samples from the Yingcheng Formation of Well C103 in the target area. We design a high-temperature, high-pressure experimental scheme to investigate supercritical CO_2-water-rock interactions. Through this approach, we aim to elucidate the transformation mechanisms of reservoir rocks under varying temperature and pressure conditions in the presence of supercritical CO_2 fluids. The findings of this study are expected to provide a theoretical foundation for the exploration and development of deep reservoirs in subsequent research.

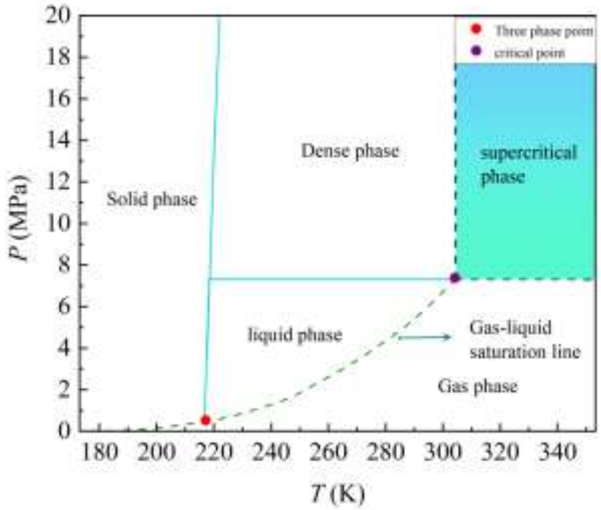


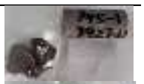
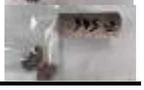
Fig 1. CO_2 phase diagram

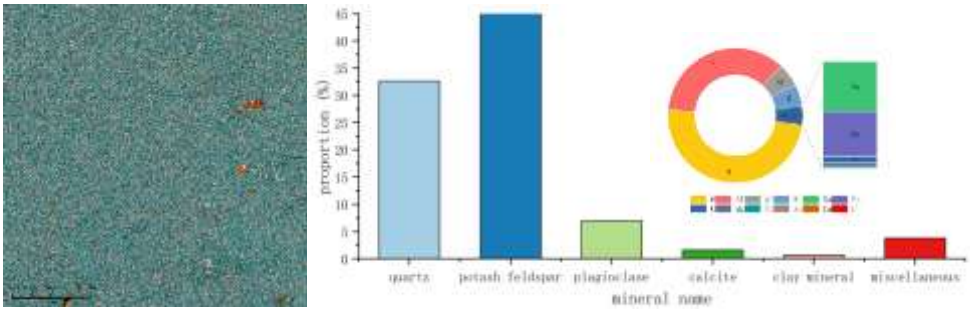
2. EXPERIMENTAL SAMPLES AND EXPERIMENTAL METHODS

2.1. Experimental Samples

The experimental samples were collected from the volcanic reservoirs of the Yingcheng Formation in the southern section of the Songliao Basin. The average length was 5.195 cm, the average diameter was 2.535 cm, and the average porosity was 7.175% (Table 1). The standard small rock column was reprocessed into a rock sample with a diameter of 25 mm and a thickness of 2 mm by a core wire cutter. Some of them were scanned by the QuemScan650 F instrument to scan the element types and mineral distribution of the rock sample. The primary minerals are mainly quartz (32.63%), followed by potassium feldspar (54.23%) and plagioclase (6.99%), with a small amount of calcite (1.65%) and clay minerals (0.7%) (Fig 2).

Table 1. Physical parameters of water-rock reaction core

sample number	well number	depth of embedment(m)	diameter(cm)	Length(cm)	porosity(%)	dry weight(g)	lithologic characters	picture
345-1	C103	3729.12	2.53	4.72	7.51	59.22	Purple breccia-bearing rhyolite	
345-2			2.54	5.67	6.84	71.39		



(a) 345 rock samples

Fig 2. Mineral scanning imaging result map

2.2. Laboratory Apparatus

The water-rock reaction simulation experiment (experimental temperature range 120~140°C, pressure 20~40MPa) was carried out in a high temperature and high pressure reactor (Fig 3). The surface of mineral samples was observed by JSM-6700F scanning electron microscope, and the reaction solution was analyzed by intelligent ion chromatograph. The pH value of the solution after reaction was measured by PHS-3C pH tester.

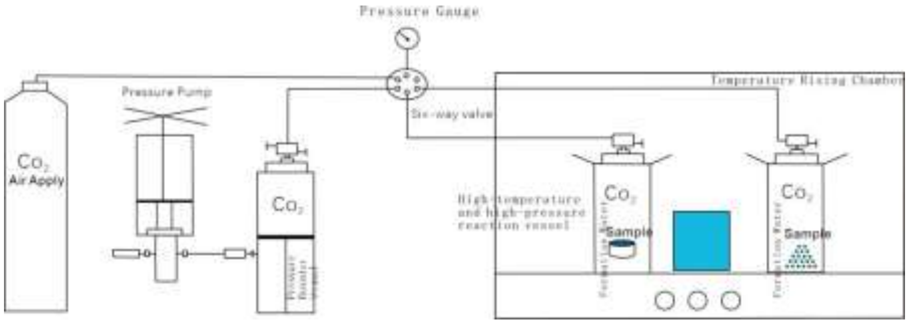


Fig 3. Schematic diagram of experimental setup of high temperature and high pressure reactor

2.3. Experimentation

2.3.1. Experimental reaction solution configuration

According to the actual formation water data of the Yingcheng Formation in the study area, the simulated formation water solution was configured (Table 2). The average salinity (DTS) of experimental formation water is 33418.47mg/L.

Table 2. Analysis results of Yingcheng Formation and simulated formation water in well C103

	Na^+	K^+	Mg^{2+}	Ca^{2+}	Total cation value	pH	Cl^-	SO_4^{2-}	HCO^{3-}	CO_3^{2-}	Total anion value	water classifying
C103	6836.09	1973.89	7.65	14.54	8832.17	7.4	2999.76	575.9	17579.07	654.36	21758.06	$NaHCO_3$
simulation formation water	6925.25	2028.27	21.3319	14.9311	8989.783	7.34	2407.23	398.307	21044.78	735.12	23850.32	$NaHCO_3$

2.3.2. Experimental design of water-rock reaction

Comparing the changes of rock mass, porosity and ion concentration of simulated formation water solution before and after water-rock reaction, the same water-rock ratio was used to add simulated formation water solution into the high-temperature and high-pressure reactor and inject CO_2. The supercritical CO_2-water-rock reaction simulation experiments at different temperatures of 120°C, 130°C, 140°C and different experimental pressures of 20MPa, 40MPa, 60MPa were carried out in turn (Table 3).

Table 3. Porosity of rock samples before reaction and experimental arrangement

Rock specimen	reaction temperature(°C)	reaction pressure(MPa)	Dry weight of rock sample before reaction(g)	Porosity (Anhydrous ethanol)(%)	reaction time(h)
345-1-1	130	20	12.23	7.20	96
345-1-2	130	40	13.11	7.09	96
345-1-3	130	60	12.30	6.56	96
345-2-1	120	40	12.38	6.65	96
345-2-2	130	40	12.50	6.42	96
345-2-3	140	40	12.46	6.49	96

The specific steps are as follows;

1) Cleaning and drying rock samples, each natural core is made into 2~5mm thick, 25mm diameter core slices;

2) Crushing one of the rock samples to test the reaction equilibrium time;

3) The experimental reaction solution is configured according to the actual formation water analysis test data;

4) After weighing the experimental rock samples, using the same water-rock ratio (2; 1), put the experimental rock samples into the high temperature and high pressure reactor, tighten the lid of the reactor, and use the vacuum pump to exclude the gas in the reactor;

5) The reactor was placed on the heating box, and the temperature was adjusted to the experimental temperature.

6) CO2 is injected into the reactor through the gas compression pump, and the heating box is swayed to ensure that the three-phase material of CO_2, saline water and rock sample in the reactor is fully contacted, and the start time of water-rock reaction is recorded.

7) Change the experimental temperature to 120°C, 130°C, 140°C, repeat step 6;

8) Change the experimental pressure to 20MPa, 40MPa, 60MPa, repeat step 6;

9) After the reaction reaches the predetermined time, the heating switch is closed. When the temperature in the reaction kettle returns to room temperature, the pressure control valve is opened to release the pressure. After the pressure is vented, the reaction kettle is opened, the solution is filtered and collected, and the rock sample is taken out.

10) Put the rock sample into the drying oven at 105°C for 6h, and weigh it again;

11) The chemical composition of the collected filtrate was determined and compared with the prepared formation solution to analyze the change of the concentration of anions and cations in the solution.

3. EXPERIMENTAL RESULT ANALYSIS

3.1. Determination of experimental reaction equilibrium time

In order to reduce the experimental error, four groups of parallel experiments were set up. The experimental temperature was 130°C, the pressure was 40MPa, and the powder quality of the rock sample was weighed every 24 hours. Many experiments showed that the volcanic rock powder was basically balanced after 96 hours of dissolution.

It can be seen from Fig 4 that with the increase of water-rock reaction time, the dissolution amount of volcanic rock powder also increases, but the amplitude slows down and tends to be stable.

Before 72h, the dissolution rate was fast, and then the dissolution rate slowed down. The dissolution amount was basically unchanged at 96h and 120h, and the reaction reached equilibrium.

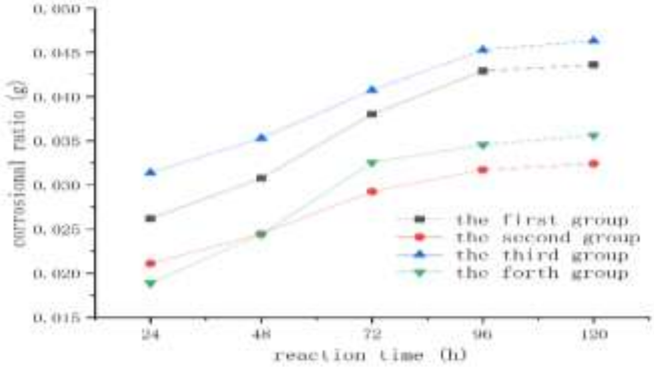


Fig 4. Volume-temporal relationship of volcanic powder dissolution

Based on the temporal evolution of ionic concentrations in the solution (Fig 5), a clear increasing trend is observed for both Mg^{2+} and Ca^{2+} concentrations over time. The Mg^{2+} concentration approaches equilibrium at approximately 48 hours, while Ca^{2+} requires a longer duration, reaching equilibrium at about 96 hours. In contrast, the K^+ concentration exhibits a continuous upward trend without showing signs of stabilization within the experimental timeframe. Mineralogical analysis of the rock sample reveals that quartz (SiO_2) remains chemically inert throughout the reaction process. Among the feldspar group minerals, potassium feldspar ($KAlSi_3O_8$) predominates, followed by albite ($NaAlSi_3O_8$). Carbonate minerals are primarily represented by calcite ($CaCO_3$), with dolomite ($CaMg(CO_3)_2$) present in minor quantities. Quantitative evaluation of minerals by scanning electron microscopy (QuemScan) further confirms this mineralogical distribution, indicating potassium feldspar as the most abundant phase, followed by calcite and dolomite in decreasing order of abundance. This mineralogical composition provides a plausible explanation for the observed dissolution kinetics, where the differential release rates of cations reflect the relative abundance and dissolution characteristics of their host minerals.

The experimental results demonstrate distinct equilibration times for carbonate mineral dissolution: dolomite reaches near-equilibrium conditions at 48h, while calcite requires approximately 96h. Notably, both Mg^{2+} and Ca^{2+} concentrations exhibit a decline after reaching peak values, suggesting secondary precipitation processes or competitive reactions with feldspar minerals. This phenomenon coincides with a gradual shift in the reaction environment toward weakly acidic conditions and reduced ionic strength effects. Previous studies[14, 34, 35] have established that feldspar mineral reactions proceed slowly, often requiring months to years for completion. This kinetic constraint informed our experimental design, justifying the selection of a 96h reaction period as sufficient to capture the dominant carbonate dissolution processes while remaining practical for laboratory investigation. The observed temporal patterns highlight the complex interplay between carbonate dissolution kinetics and subsequent silicate mineral reactions in water-rock systems.

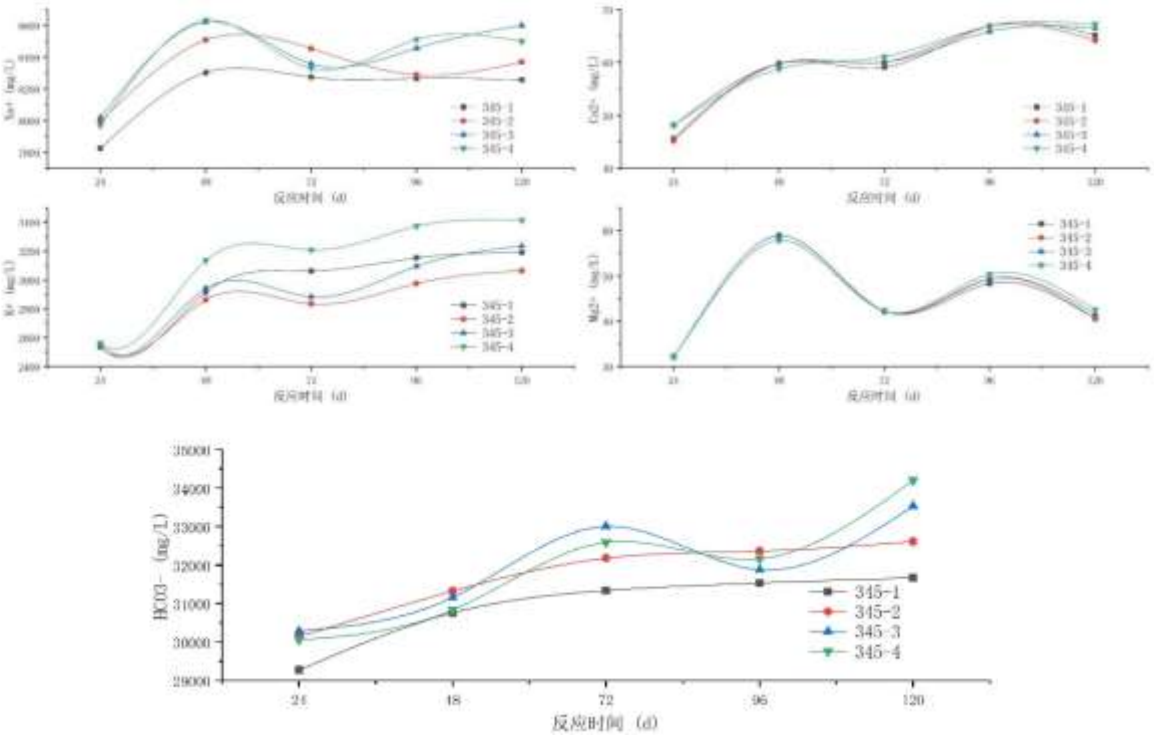


Fig 5. Ion mass concentration-time relationship in 130°C, 40 MPa system

3.2. The change of ion mass concentration and pH value of formation water

3.2.1. The influence of different experimental pressures on water-rock reaction

Under constant temperature conditions (130°C) and a reaction duration of 96h, experimental results demonstrate a positive correlation between reactor pressure and both mineral dissolution extent and porosity development in volcanic rock samples (4). Elevated reactor pressures promote greater CO_2 dissolution in the formation water, resulting in the formation of an acidic CO_2-rich aqueous solution with pH values ranging from 3.07 to 3.26. This acidic environment facilitates the preferential dissolution of chemically unstable mineral phases within the volcanic rocks. Quantitative analysis reveals that the reacted samples exhibit an average mass loss of 0.2460g, corresponding to an average porosity increase of 4.73%. The observed pressure-dependence suggests that CO_2 solubility-and consequently, solution acidity-serves as the primary control on mineral dissolution rates and associated porosity modification.

Table 4. Dissolution volume-pressure relationship for 96h reaction volcanic rock samples

Rock specimen	reaction pressure(MPa)	Quality of rock sample before reaction(g)	Quality of rock sample after reaction(g)	corrosional ratio(g)	Increased porosity(%)	Before reaction (pH)	after reaction (pH)
345-1	20	12.23	12.01	0.2254	4.43	3.26	7.33
	40	13.11	12.86	0.2489	4.57	3.14	7.35
	60	12.30	12.04	0.2638	5.18	3.07	7.53

There are some differences in the concentration of each ion, which is positively correlated with the pressure (Fig 6). Mg^{2+}, Ca^{2+} and K^+ come from dolomite, calcite and K-feldspar, respectively. It can be seen from Figure 6 that the dissolution intensity increases with the increase of reaction pressure. For every 20 MPa increase in reaction pressure, the growth rate of K^+ does not change significantly. The average mass concentration of $Mg2^+$ is 26.76mg/L at P=20MPa, 33.06mg/L at P=40MPa, and 51.53mg/L at P = 60MPa. The main growth range is from 40MPa to 60MPa, and the growth range is

2.93 times from 20MPa to 40MPa. Mg^{2+}.Similarly, when P=20MPa, the average mass concentration of Ca^{2+} is 46.95mg/L, when P=40MPa, the average mass concentration is 93.11mg/L, when P=60MPa, the average mass concentration is 122.28mg/L. The main growth range is between 20MPa and 40MPa, and the growth rate is 1.58 times that of 40MPa to 60MPa. It shows that in the same acidic environment, calcite is more likely to react with acid than dolomite. With the reaction, in the weak acid environment, calcite and dolomite react to reach equilibrium. The feldspar minerals are unsaturated during the whole reaction process, and the reaction continues, so the growth of K^+ does not stop.

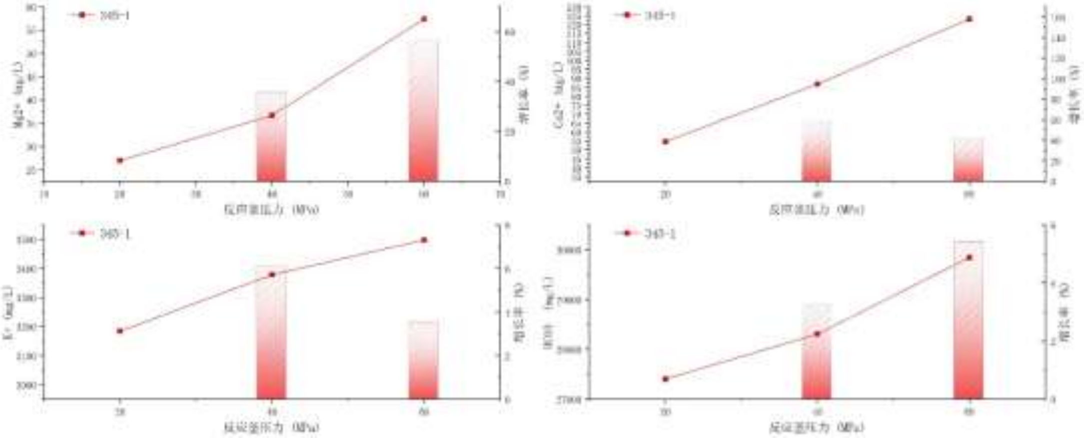


Fig 6. Relationship between mass concentration of ions in solution after reaction and change in reactor pressure

3.2.2. The effect of different experimental temperatures on water-rock reaction

When the pressure of the reaction kettle is constant at 40MPa and the reaction time is 96h, the dissolution amount and porosity change of the rock sample are negatively correlated with the reaction temperature (Table 5). The increase of the reaction temperature will reduce the dissolution degree of CO_2 in the solution on the one hand, and on the other hand, it will increase the ion exchange rate in the reaction solution and reduce the ion effect. It can be seen from Table 7 that with the increase of reaction temperature, the dissolution amount of rock samples decreases gradually. When T=120°C, the average dissolution amount of rock samples is 0.2803g. When T=140°C, the average dissolution amount of rock samples is 0.2242g, which is reduced by 20.01%.

Table 5. Dissolution volume-temperature relationship for 96h reaction volcanic rock samples

Rock specimen	reaction temperature(°C)	Quality of rock sample before reaction(g)	Quality of rock sample after reaction(g)	corrosional ratio(g)	Increased porosity(%)	Before reaction (pH)	after reaction (pH)
345-2	120	12.38	12.09	0.2803	4.37%	3.11	7.79
	130	12.50	12.24	0.2645	4.21%	3.14	7.69
	140	12.46	12.23	0.2242	3.04%	3.18	7.62

The mass concentrations of Mg^{2+}, Ca^{2+} and K^+ increase with the increase of reaction temperature, but the change of ion concentration is negatively correlated with the reaction temperature. It can be seen from figure 7 that the increase of reaction temperature will not affect the reaction process of water-rock reaction. When the temperature increases from 130°C to 140°C, the growth rate of K^+ mass concentration decreases from 2.71% to 1.78%, the growth rate of Mg^{2+} mass concentration decreases from 10.71% to 9.18%, and the growth rate of Ca^{2+} mass concentration decreases from 25.92% to 5.45%. It shows that the increase of reaction temperature has obvious inhibition effect on calcite dissolution, and the inhibition effect on dolomite and feldspar minerals is relatively weak.

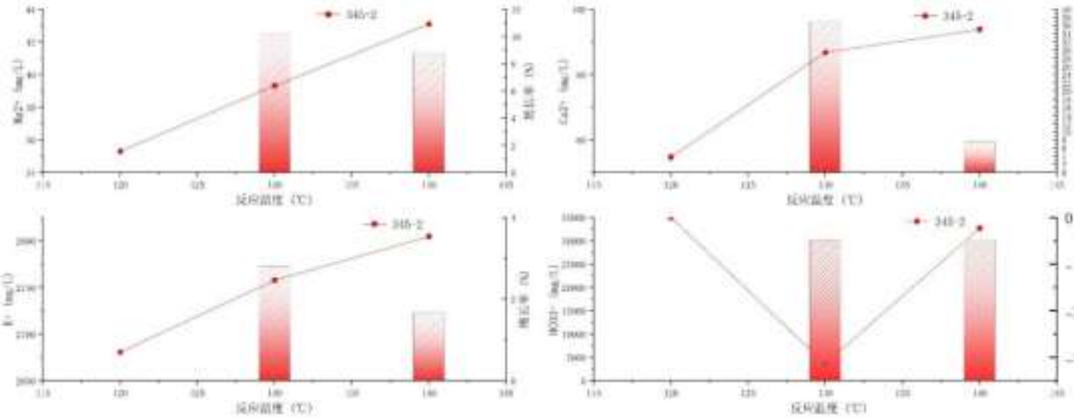


Fig 7. Plot of ion mass concentration in solution after reaction-reaction temperature variation

3.3. The change of surface microstructure characteristics of rock samples

Combined with the observation results of scanning electron microscopy (SEM), under the experimental environment of high temperature and high pressure, different degrees of dissolution occurred on the surface of rock samples. The dissolution pores increased significantly and secondary pores were produced. The main rock components that reacted were unstable minerals (potassium feldspar, albite, calcite, dolomite and clay minerals). After the water-rock reaction, the main manifestations are as follows: calcite, dolomite and feldspar mineral particles are dissolved, dissolution pores (pits) are formed on the surface or concentrated dissolution occurs along the direction of joint joints, and clay minerals are selectively dissolved according to their mineral components. As shown in figure 8, in the rock sample before the experiment, the surface of the calcite crystal is flat and the edges and corners are clear (figure 8 a). After the reaction, the surface of the calcite crystal appears dissolution holes (pits), and the edge has obvious dissolution (figure 8 b); after the reaction of potassium feldspar water-rock, a large number of dissolution pits are formed, and the edge of the particles is concave-convex or bay-like (Fig 8 c).

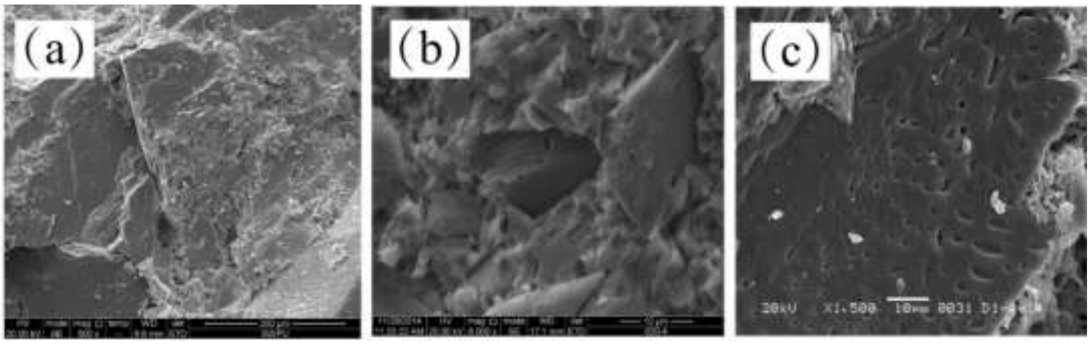


Fig. 8 Scanning electron microscope photographs before and after water-rock reaction of rock samples in the southern part of Songliao Basin

4. DISCUSSION

4.1. Analysis of the dissolution mechanism

In the high temperature and high pressure environment, the supercritical CO_2 solution is formed by injecting carbon dioxide gas into the simulated formation water, and the solution shows significant dissolution and transformation ability. Compared with the previous experimental results, this experiment fills the gap of water-rock reaction of supercritical CO_2-salt water-volcanic rock samples under high temperature and high pressure. The dissolution minerals of water-rock reaction are unstable mineral components (calcite, dolomite, potassium feldspar and clay minerals) in rock samples. In the high temperature and high pressure experimental environment, CO_2 gas is mixed with the simulated formation water solution in a supercritical form to dissociate H^+ (Table 6), which changes the original acid-base equilibrium environment. Lead to a decrease in the pH value of the reaction system [36].

Table 6. CO_2-formation water chemical reaction equation

reaction equation	
$CO_2(g) + H_2O(l) \Leftrightarrow H_2CO_3(aq)$	(1)
$H_2CO_3(aq) \Leftrightarrow H^+(aq) + HCO_3^-(aq)$	(2)
$HCO_3^- \Leftrightarrow H^+(aq) + CO_3^{2-}(aq)$	(3)

The change of dry weight before and after the reaction of rock samples in the study area shows that the mineral components such as potassium feldspar, calcite and dolomite in the rock samples are consumed in the water-rock reaction, which improves the porosity of the rock samples. On the one hand, the high pressure environment in the reaction increases the solubility of CO_2 in the simulated formation water solution, on the other hand, it inhibits the transformation of clay minerals and increases the contact time and dissolution strength of soluble minerals[15]. The high temperature and salt water environment in the reaction are not conducive to the dissolution of CO_2. The solubility of CO_2 decreases with the increase of temperature and salinity of simulated formation water[13, 34, 37] (Fig 9). The temperature of this experiment is 120~140°C and the pressure is 20~60MPa, which represents the temperature and pressure range of the reservoir buried depth of 3200~4000m in the study area. From Figure 6 and Figure 7, it can be seen that when the pressure is constant, the increase of temperature is not conducive to the dissolution of volcanic rock samples. When the temperature is constant, the pressure has a significant increase in the water-rock reaction. In the volcanic rock samples, calcite has the strongest dissolution in all unstable minerals, followed by dolomite.

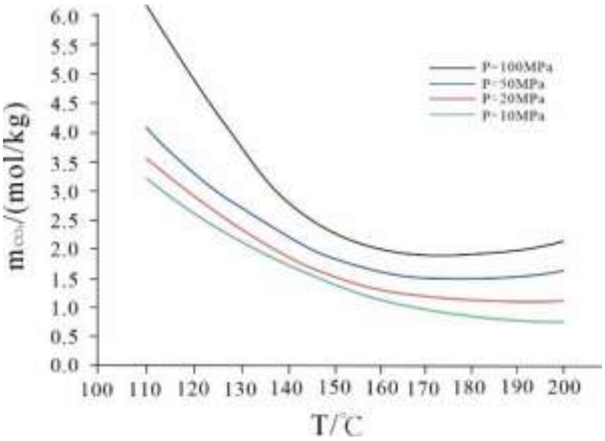


Fig 9. Solubility of CO_2 versus temperature and pressure[34, 37]

4.2. Experimental geological significance

The essence of the high temperature and high pressure supercritical CO_2-salt water-rock reaction process is the equilibrium process of the physical and chemical action of the mineral-fluid in the system[15]. The simulation experiment shows that the CO_2 solution formed by injecting CO_2 into the deep volcanic reservoir and the formation fluid can dissolve the silicate and carbonate minerals in the reservoir rock, and the deep reservoir has the ability to develop secondary pores.

The reservoir genesis and geological background of deep volcanic gas reservoirs in the southern section of Songliao Basin are unique. High temperature and high pressure environment and high salinity salt water in reservoirs are significant features that distinguish other oil and gas basins.

Therefore, under the background of high investment, high cost and high risk of deep oil and gas exploration, it is of great practical significance to carry out water-rock simulation experiments for deep reservoir reconstruction of volcanic reservoirs.

5. CONCLUSION

(1)High temperature and high pressure supercritical CO_2-salt water-rock sample reaction equilibrium experiments show that the dissolution rate of unstable minerals in rock samples is faster in the first 72h, then slows down, and the water-rock reaction basically reaches equilibrium in the 96h. The difference between the amount of dissolution and the amount of dissolution at 120h is 2.33%.

(2)Quantitative analysis showed that the dissolution amount increased by 17.04% and the average porosity increased by 4.72% in the range of 20~60MPa when the reaction temperature was constant. Under the same reaction pressure, in the range of 120~140°C, the dissolution amount decreased by 20.01%, and the average porosity increased by 3.87%.

(3)The increase of Ca^{2+} mass concentration in simulated formation water comes from the dissolution of calcite minerals. The increase of Mg^{2+} mass concentration comes from the dissolution of dolomite minerals. The increase of K^+ mass concentration comes from the dissolution of feldspar minerals. Comparing before and after the water-rock reaction, it can be seen from the scanning electron microscope images that a large number of dissolution pores (pits) appear on the surface of the mineral crystals caused by dissolution, and the edges of feldspar minerals are concave-convex or harbor-shaped.

(4)Selecting representative rock samples to carry out high temperature and high pressure supercritical CO_2 water-rock reaction simulation experiments can scientifically and effectively provide evidence of dissolution, which has important practical significance for reservoir reconstruction of deep volcanic gas reservoirs.

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