

Experimental Investigation of CO₂-Water-Rock Interactions during CO₂ Flooding in Carbonate Reservoir

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Abstract

Injecting CO₂ into underground reservoir to displace oil is a viable means of reducing greenhouse gas emission to the atmosphere and enhancing oil recovery. To evaluate the effect of CO₂-water-rock interactions on the characteristics of carbonate reservoir at high pressure, the mineralogy of calcite, the ion concentration in the reacted solution, the surface texture of calcite, the permeability of calcite after reacted with injected CO₂ and deionized water was investigated by X-ray diffraction (XRD), inductive coupled plasma-atomic emission spectrometry (ICP-AES), scanning electronic microscope (SEM), and sand-packed model at pressure of 5.0 MPa. The results show that the mineral dissolution of calcite would occur when interacting with injected CO₂ and water. The mineral dissolution of calcite caused the change of surface texture of calcite and increase in Ca²⁺, HCO₃⁻ ion concentration in the solution. With the increase of CO₂ pressure, the surface dissolution of calcite appeared more obvious. With the increase of reaction temperature, the surface dissolution of calcite also appeared more obvious and Ca²⁺, HCO₃⁻ ion concentration in the solution increased first, then decreased. The mineral dissolution of calcite caused the improvement in water permeability of calcite/quartzsand-packed model.

Keywords

CO₂ Flooding, CO₂-Water-Rock Interaction, Calcite, Dissolution, Surfacetexture, Permeability

1. Introduction

The emission of carbon dioxide (CO₂) is increasing natural greenhouse gas ef-

fects. It has been achieved a broad consensus to reduce CO₂ emissions on a global scale. One of technological solutions to reduce CO₂ emissions is to inject CO₂ into underground reservoirs to displace oil, which can enhance oil recovery effectively and sequester CO₂ simultaneously, mitigate global warming consequently [1] [2]. CO₂ flooding will yield remarkable social and economic benefits [3] [4].

The CO₂-water-rock interactions caused by CO₂ flooding of reservoirs are complex [5] [6] [7]. These interactions are highly reservoir specific and cannot easily be generalized [8] [9]. Injected CO₂ dissolved in formation water to produce carbonic acid which will react with the reservoir rock [10] [11] [12]. The porosity and permeability of the reservoir [13] [14] [15] [16] [17], the wettability of the rock surface and the characteristics of the crude oil will be changed by the reaction between carbonic acid and the rock [18] [19], which will affect the efficiency of CO₂ flooding and geological sequestration of CO₂ [20] [21] [22]. And the carbonate minerals in the reservoir can especially easily react with CO₂ and water to affect reservoir property. Carbonate reservoir is mainly composed of calcite, dolomite, ankerite and magnesite, and these minerals can easily dissolve in carbonate solution which leads to corrosion of the rock and increase of reservoir permeability. Ross [23] found that after the reaction of CO₂, water and rock with carbonate mineral, carbonate minerals in the rock dissolved, and it formed a large number of secondary dissolution channels. Ross also analysed ion concentration of the displacement solution and found that the Ca²⁺ concentration in the solution increased significantly. Izgec [24] pointed out that a large number of loop hole formed in the process of the dissolution of carbonate mineral, it obviously improved the connectivity of pore, and so caused the permeability improved significantly. Raistrick [25] found that after the reaction of carbonate sample, CO₂ and water, the concentration of Ca²⁺, Mg²⁺ and HCO₃⁻ in effluent solution increased significant which indicating the carbonate mineral dissolution. In addition, some scholars also pointed out that the change of physical property of carbonate reservoir was related to formation water pressure, temperature, and rock mineral composition and CO₂ partial pressure [26].

Therefore, in order to carry further study on the main controlling factors which result in the change of reservoir physical properties in the process of CO₂ flooding in carbonate reservoir, calcite (main ingredient is CaCO₃) was selected to react with CO₂ and water in this paper. Then, the surface texture of calcite, the ion concentration in the reacted solution and the permeability of calcite/quartz packed model were investigated after their reaction. The mechanism of interaction of CO₂-water-calcite was discussed, which provided the valuable reference for CO₂ flooding and geological sequestration of CO₂ in the carbonate reservoir.

2. Experimental

2.1. Materials and Preparation of Sample

Two types of calcite materials were used in the study, the plate and the grained.

The plate samples were made by cutting calcite material into 8 mm sized blocks, washed with deionized water by ultrasonic vibration, dried at 70 °C for 12 h. The calcite material was crushed to grains and selected the grains with average diameter of 0.45 mm. The calcite material was provided by Shijiazhuang Cuanshi mining company, in China. The purity of CO₂ source provided by Beijing Hai-pubeifen gas limited company was >99.95%. The water used in the experiments was deionized.

2.2. Experimental Methods

1) Calcite component analysis

The clay mineralogy of the calcite was identified and quantified by X-ray diffraction (XRD) (D/MAX 2500, Rigaku Industrial Corporation, Japan) which can determine the mineral composition and content in calcite.

2) Determination of the rocks' apparent morphology

SEM measurements were carried out on a Leica Cambridge S-360 (Malvern Instruments Ltd., UK) at 25 °C. The acceleration voltage was 20 kV. The resolution of the instrument was 5 nm. The vacuum of the sample room was 1.33×10^{-3} - 1.33×10^{-4} Pa.

First, the calcite was cut and polished into 20 × 20 mm sheet, and the surface of it was cleaned with ultrasonic waves in deionized water. Then the calcite was dried and stucked on specimen holder by conductive adhesive, and it was coated by means of ion sputtering at last. The morphology of coating calcite slice was observed by scanning electron microscope, and then it was placed in the high pressure reactor. After the reaction, the calcite slice was took out and dried, the newly formed or altered surface texture in the reacted rock sample was investigated by SEM again.

3) The static evaluation method of CO₂-water-rockchemical interaction

The chemical interaction of CO₂-water-rock was identified by exposing the rock samples in a stainless steel reactor of 100 ml (**Figure 1**), equipped with a Teflon (PTFE) internal cup, manometer and automatic temperature controller, connected to an electric heating apparatus. The sheet calcite sample was put into the bottom of the high pressure reactor, and 100 mL deionized water was added into the high pressure reactor, then the CO₂ was injected into it at specific pressure and temperature. After 20 days' reaction, the calcite slice was took out and dried.

4) Aqueous sampling and ion concentration determination

The grained calcite was mixed with deionized water by ratio of 1:20, w:w, and then CO₂ was injected into the reactor to 2.0 MPa. The reacted liquid was sampled every 2 days during reacting for 20 days. The major cation concentrations of the sampled solution were determined by inductive coupled plasma-atomic emission spectrometry, ICP-AES (Profilie, Leeman Labs, USA). The resolution of the instrument is 200 nm. The HCO₃⁻ concentration of the sampled liquid was determined by double-tracer technique.

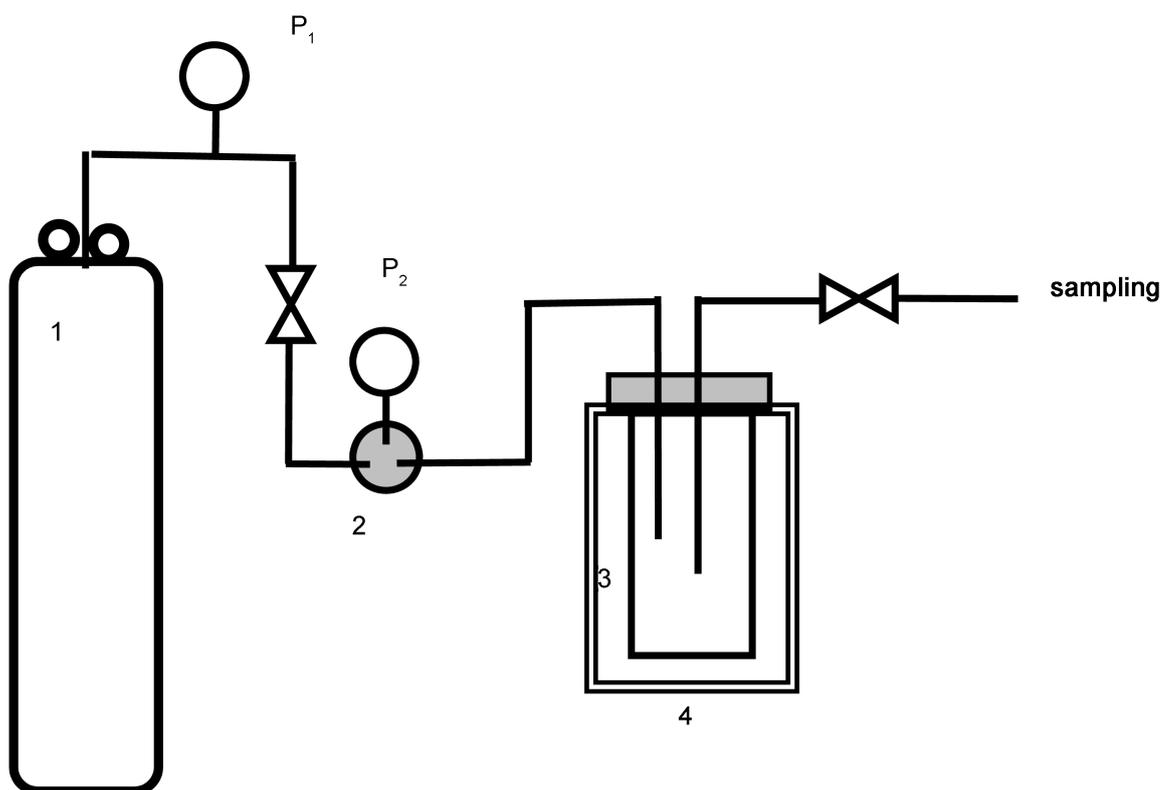


Figure 1. Scheme of equipment used for CO₂-water-rock reactions (1. CO₂ resource; 2. valve; 3. Reactor; 4. electric heating apparatus; P₁, P₂ is pressure gauge).

5) Permeability test

Steady state permeability variation caused by the presence of deionized water and CO₂ was analyzed on a 30 cm sand-packed model which was a sand pipe filled with grained calcite and quartz with the same diameter mixed by a ratio of 1:1. The original permeability of the model was presented by water permeability. Having placed for 3 days, water permeability of the sand-packed model was measured again. Then CO₂ was injected into the model to 5.0 MPa. After that the water permeability of the model was measured every 5 days. The test temperature is 65°C.

3. Results and Discussion

3.1. Composition Analysis of Calcite

The calcite used in the experiments is white. The crystal structure of calcite was shown in **Figure 2**, an obvious rhombus structure. According to the formation of the cubic packing arrangement form, CO₃²⁻ create a diamond surface grid. Most calcite crystal is colorless, transparent. It has smooth plane and straight edge, it aggregates in a variety of forms: flake, fibrous, dense block, membrane and drusy, etc. The result of X-ray diffraction of the calcite was shown in **Figure 3**. Compared with JCPDS (Joint Committee Powder Diffraction Standard), the major chemical component of the calcite was CaCO₃. Based on quantitative calculation of intensity and half band width of the diffraction peak, CaCO₃ accounted for 97.8%, so the calcite crystal had high quality.

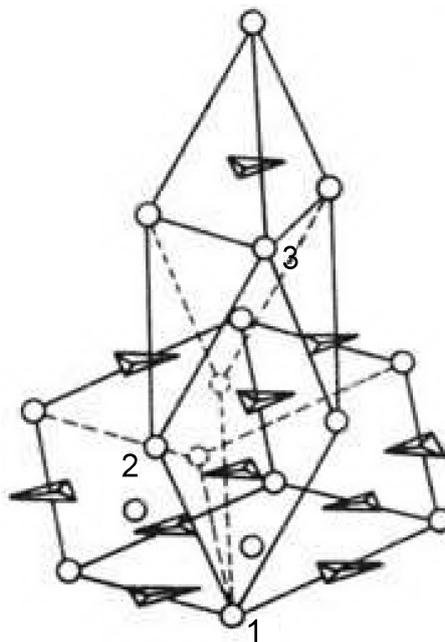


Figure 2. Crystal structure of calcite.

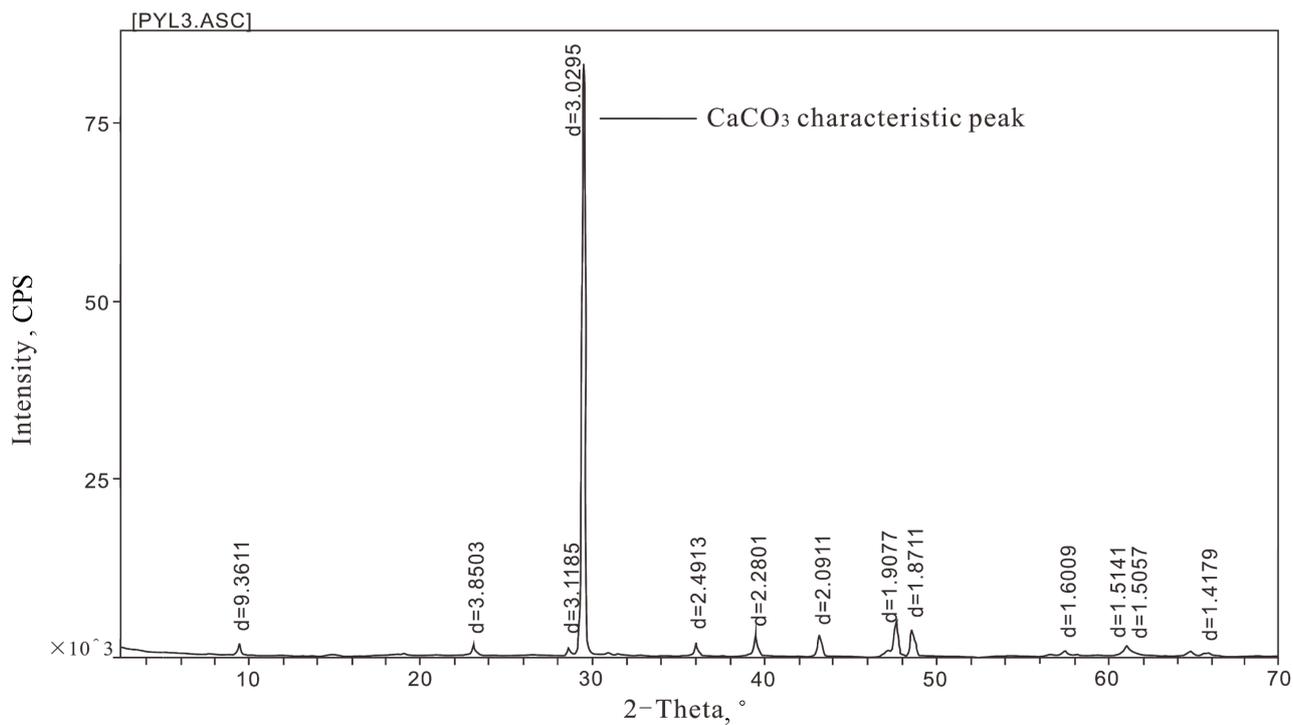


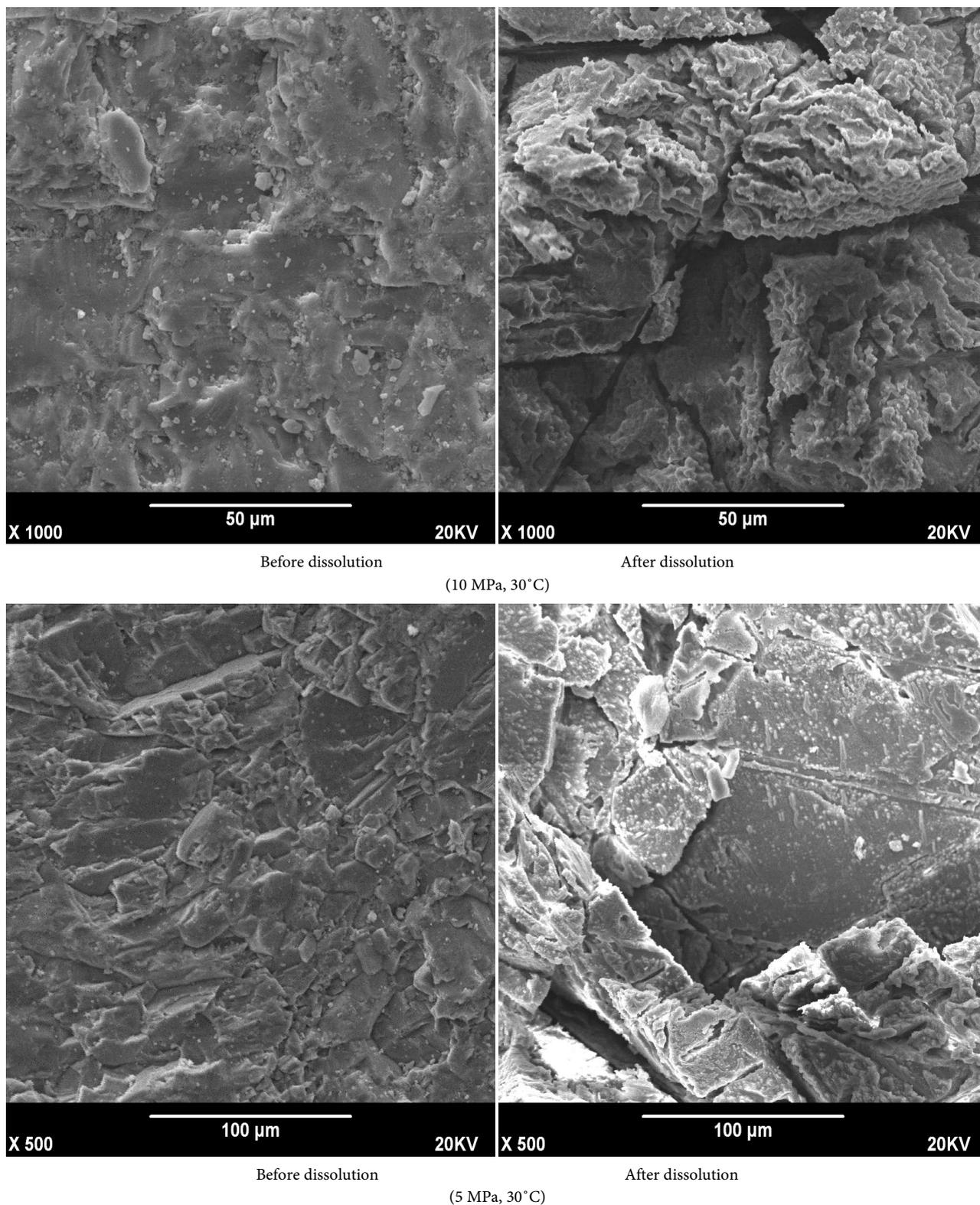
Figure 3. X-ray diffraction of calcite.

3.2. Surface Texture of Calcite after Reacted with CO₂

In order to investigate the dissolution of rock caused by CO₂-water-rock interactions, the surface of calcite slice dissolved by CO₂ was observed by scanning electron microscope after the calcite slice reacted with CO₂ under different pressure and temperature. The experiment results were shown in Figure 4 and Figure 5.

1) Surface texture of calcite before and after the interactions of CO₂-water-calcite at different pressure

The change of the calcite morphology in different pressure and 30°C was shown in **Figure 4**. As shown in **Figure 4**, the surface morphology of calcite



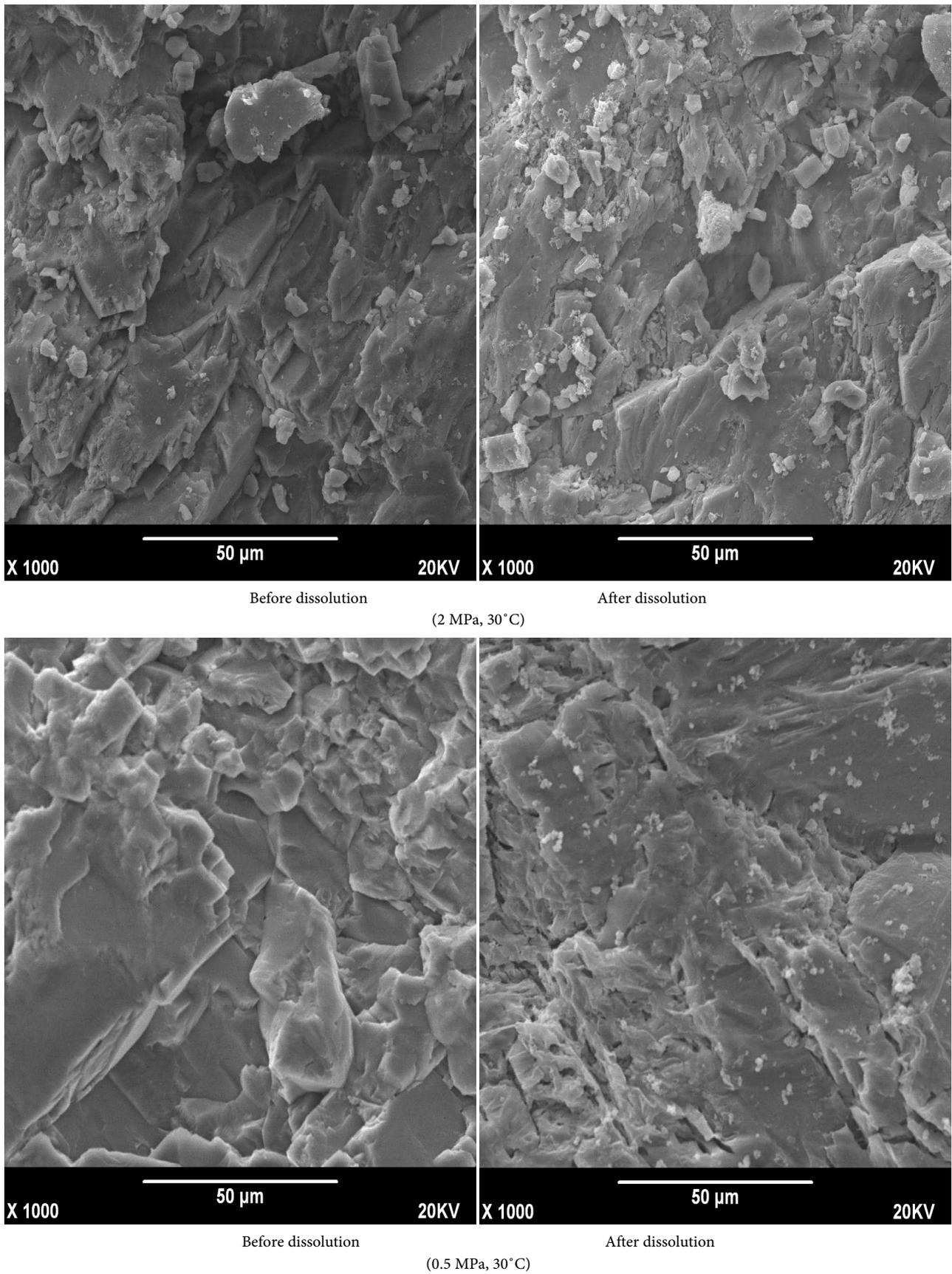
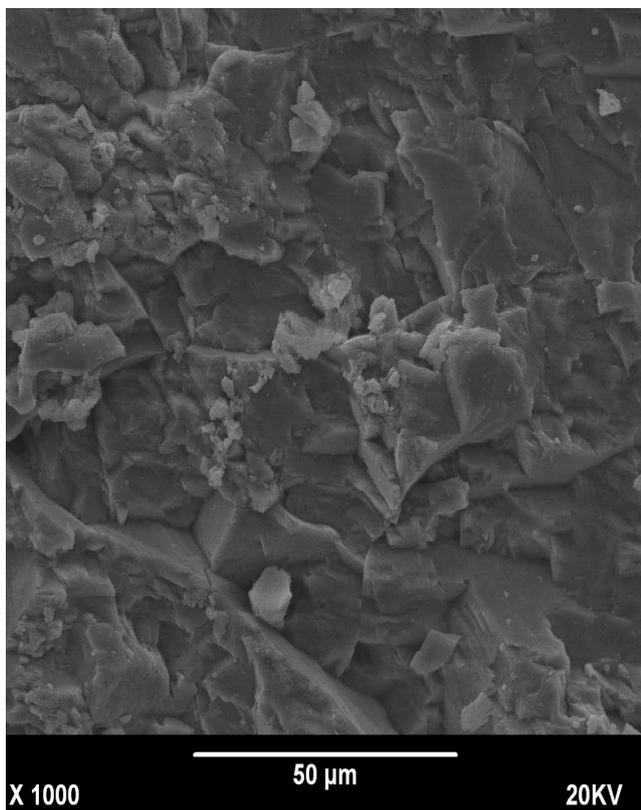
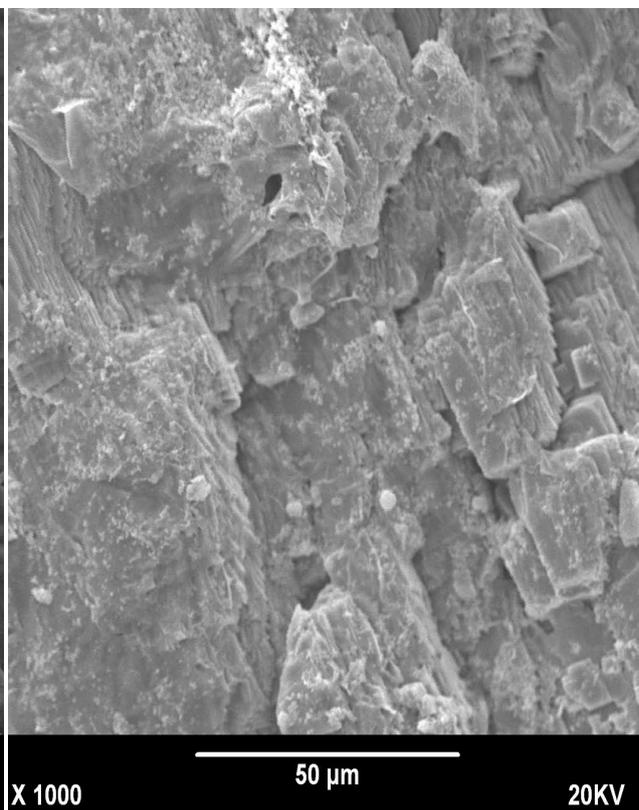


Figure 4. SEM micrographs of calcite samples that pre-and post-reacted with CO₂ under different pressures.

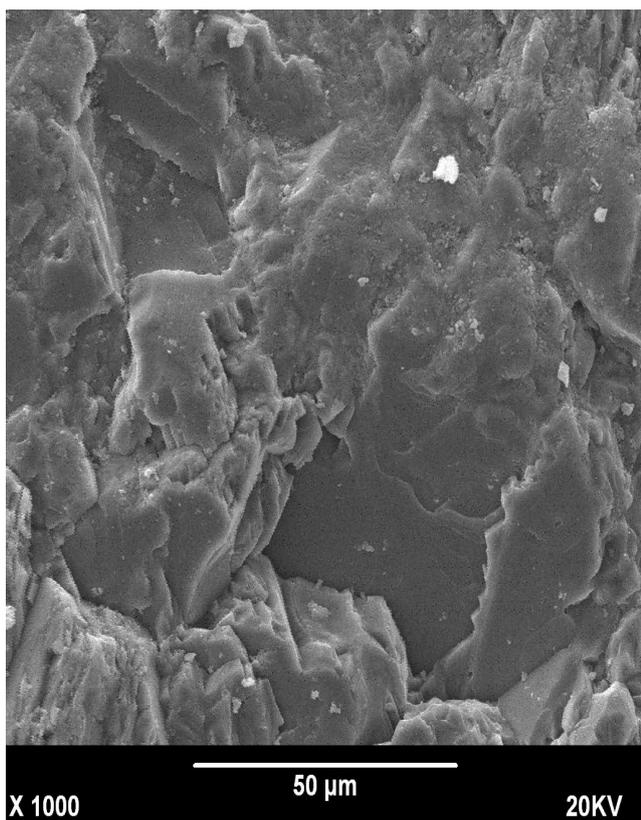


Before dissolution

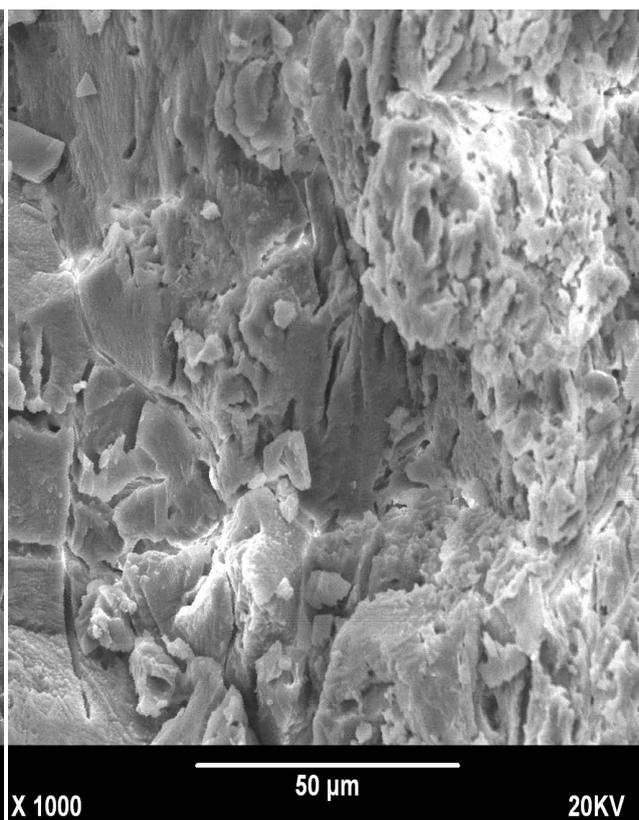


After dissolution

(2 MPa, 90°C)



Before dissolution



After dissolution

(2 MPa, 60°C)

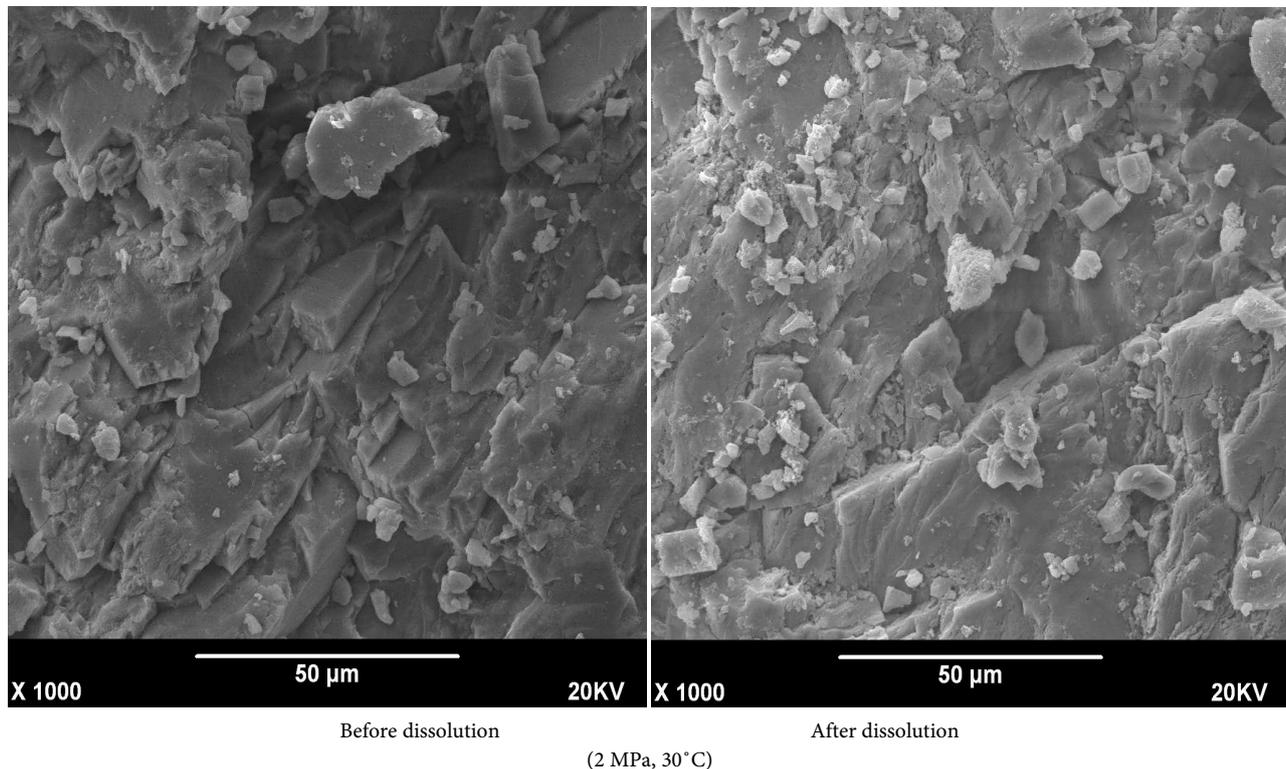


Figure 5. SEM micrographs of calcite samples that pre- and post-reacted with CO₂ in different temperature.

changed obviously after reaction with CO₂ and water. At 30°C, with the increase of CO₂ pressure, honeycomb cave phenomenon of the calcite is more obvious caused by the reaction of CO₂. At the pressure of 0.5 MPa, the surface of calcite appeared only a little corrosion phenomenon caused by the reaction of CO₂. As the pressure rose to 10 MPa, the surface of calcite appeared obvious corrosion phenomenon, namely the morphology of calcite changed more obvious. This is mainly because the reaction of CO₂ and calcite is greater with the increase of CO₂ pressure, the surface corrosion of calcite appeared more obviously. This result is consistent with the study by Gilfillan and Tang *et al.* [27] [28] [29].

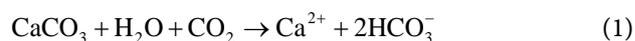
2) Surface texture of calcite before and after the interactions of CO₂-water-calcite at different temperature

The change of the calcite morphology in different temperature at 2 MPa was shown in **Figure 5**. As shown in **Figure 5**, at the temperature of 60°C and 90°C, the reaction of CO₂ and calcite is greater than which was at the temperature of 30°C, and the surface corrosion of calcite appeared more obviously. After the reaction of CO₂ and calcite at 60°C, many large pores appeared in the calcite surface, namely the calcite was dissolved more.

3.3. Ion Concentration in the Solution after Reacted with CO₂

The main reason for the changes of the surface morphology of calcite is carbonic acid which is produced when CO₂ dissolved in the water. Carbonic acid dissociated hydrogen ion which can react with calcium carbonate in calcite and pro-

duced calcium soluble bicarbonate. That is CO_2 -water-dolomite interactions lead to calcite corrosion. The chemical reaction formula of CO_2 -water-calcite interactions is shown in formula (1).



There was Ca^{2+} ion in the solution after calcite dissolution and the dissolution trace was shown in the surface of the calcite. For proving that, the major Ca^{2+} and HCO_3^- concentration of the solution after having reacted with calcite and CO_2 at different pressure and temperature were investigated. The results were shown in **Figures 6-9**. The type of calcite was grained in this experiment [30] [31] [32].

1) Ion concentration in the reacted solution after the interactions of CO_2 -water-calcite at different pressure

Figure 6 and **Figure 7** shows that with the increase in reaction time, Ca^{2+} and HCO_3^- ion concentration in the solution are also increased; with the increase of reaction pressure, Ca^{2+} and HCO_3^- ion concentration in the solution are increased as well. Ca^{2+} and HCO_3^- ion concentration in the solution are increased

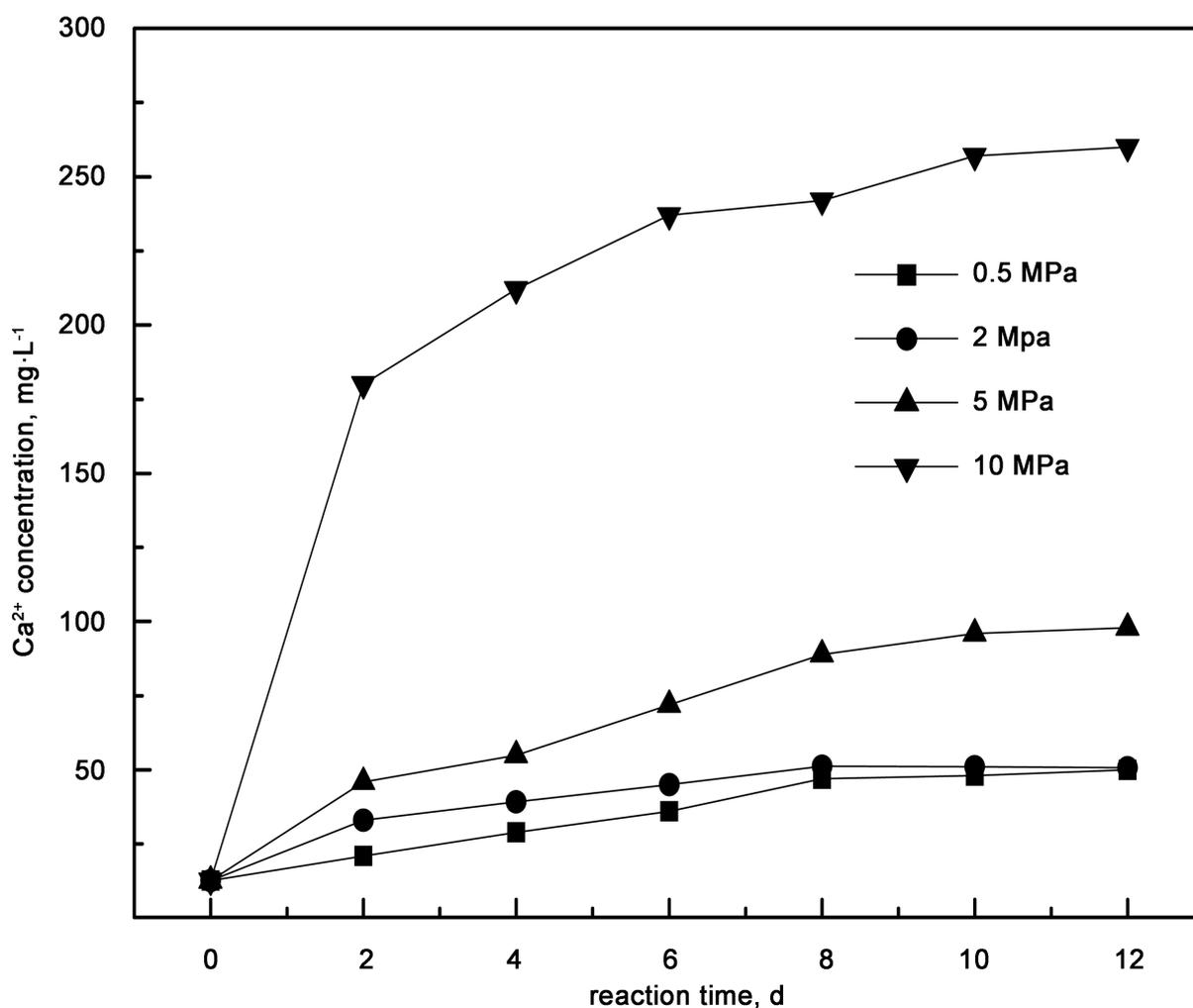


Figure 6. Ca^{2+} ion concentration vs. reaction time at different reaction pressure.

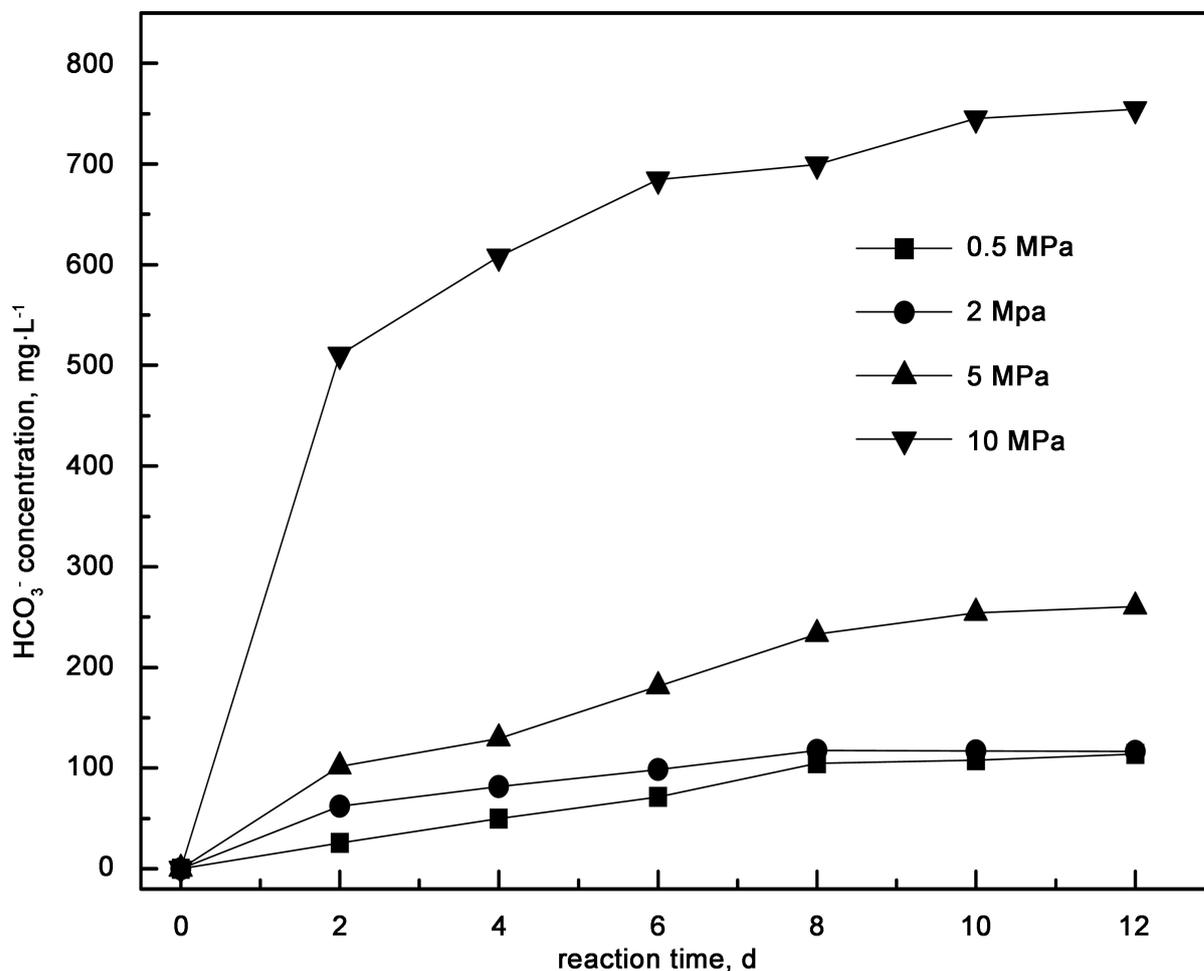


Figure 7. HCO₃⁻ ion concentration vs. reaction time at different reaction pressure.

at the pressure of 0.5 MPa, 2 MPa and 5 MPa, but the increment is small. Ca²⁺ and HCO₃⁻ ion concentration in the solution are increased obviously at the pressure of 10 MPa. This is mainly due to the reaction pressure of 10 MPa is higher than the critical pressure of CO₂, so the reaction condition is close to supercritical condition, the diffusivity and permeability CO₂ are stronger, which speed up the reaction, and lead to the significant increase of Ca²⁺ and HCO₃⁻ concentration in the solution.

The major chemical component of calcite is CaCO₃ which reacted with carbonic acid to cause the dissolution of calcite. Consequently, Ca²⁺ and HCO₃⁻ ion concentration increased. The variation of Ca²⁺ and HCO₃⁻ ion concentration also proved the dissolution of calcite after the interaction of CO₂-water-rock which led to forming the new surface texture of the calcite.

2) ion concentration in the reacted solution after the interactions of CO₂-water-calcite at different temperature

Figure 8 and **Figure 9** shows the changes of Ca²⁺ and HCO₃⁻ ion concentration in the solution with the reaction temperature at 30°C, 60°C, 90°C respectively.

Figure 8 shows that with the increase in reaction time, Ca²⁺ ion concentration

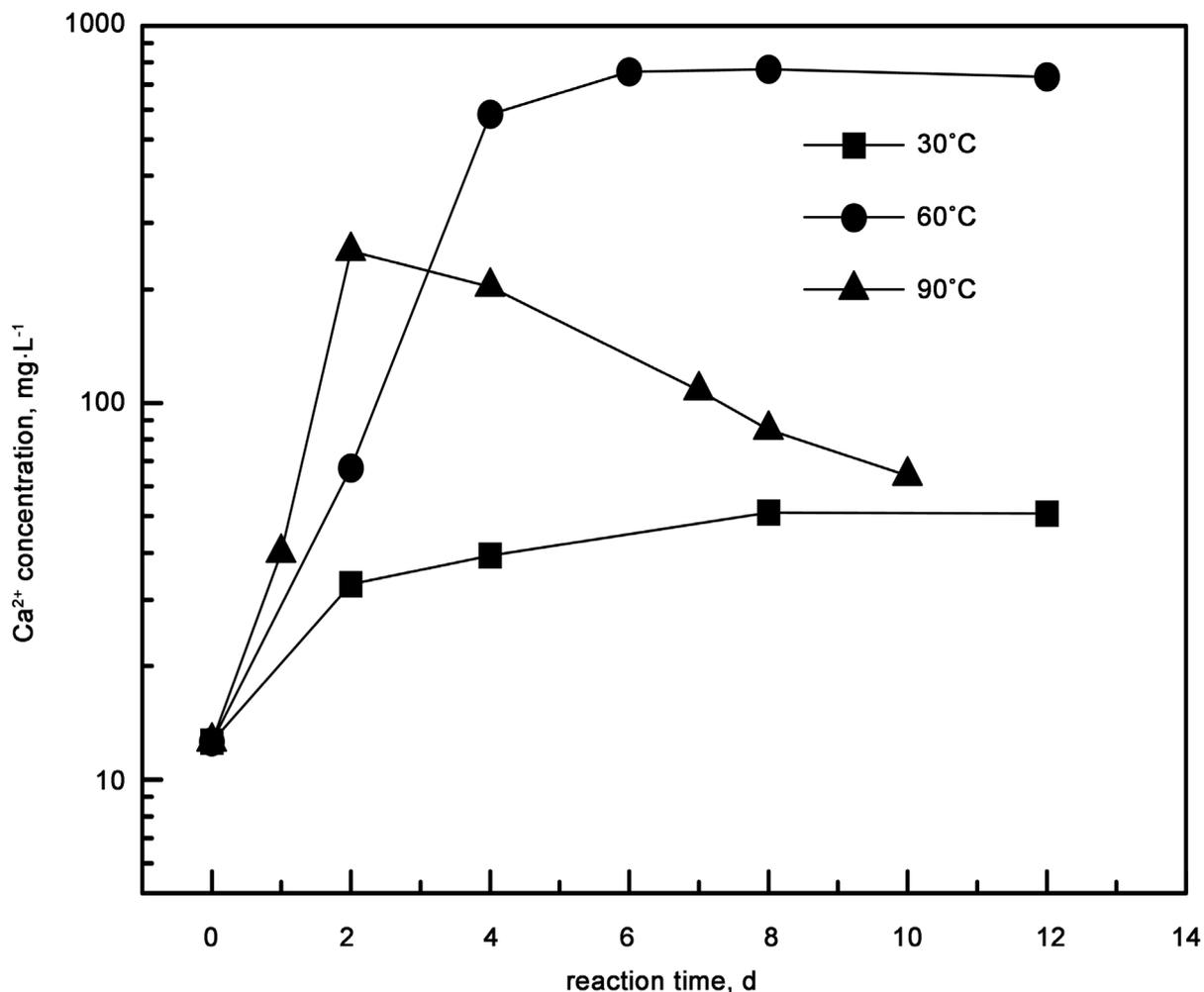


Figure 8. Ca²⁺ ion concentration vs. reaction time at different reaction temperature.

in the solution was improved at 30°C and 60°C. But the extent of the change of the concentration at different reaction temperature was different. Ca²⁺ ion concentration in the solution remained unchanged after reacted for 8 days at 30°C. It took 6 days to reach a plateau of Ca²⁺ ion concentration in the solution at 60°C. Ca²⁺ ion concentration in the reacted solution was higher at 60°C than the ion concentration in the reacted solution at 30°C at the same reaction time.

Figure 9 shows that the change trend of HCO₃⁻ ion concentration in the solution was the same as Ca²⁺ ion concentration. With the increase of reaction time, HCO₃⁻ ion concentration in the solution was improved at 30°C and 60°C. HCO₃⁻ ion concentration in the solution remained unchanged after reacted for 8 days at 30°C. It was 6 days at 60°C. HCO₃⁻ ion concentration in the reacted solution was higher at 60°C than the ion concentration in the solution at 30°C at the same reaction time. The results show that the reaction rate was increased and the reaction reached the equilibrium much faster at higher reaction temperature.

Figure 8 also shows that Ca²⁺ ion concentration in the solution reached a

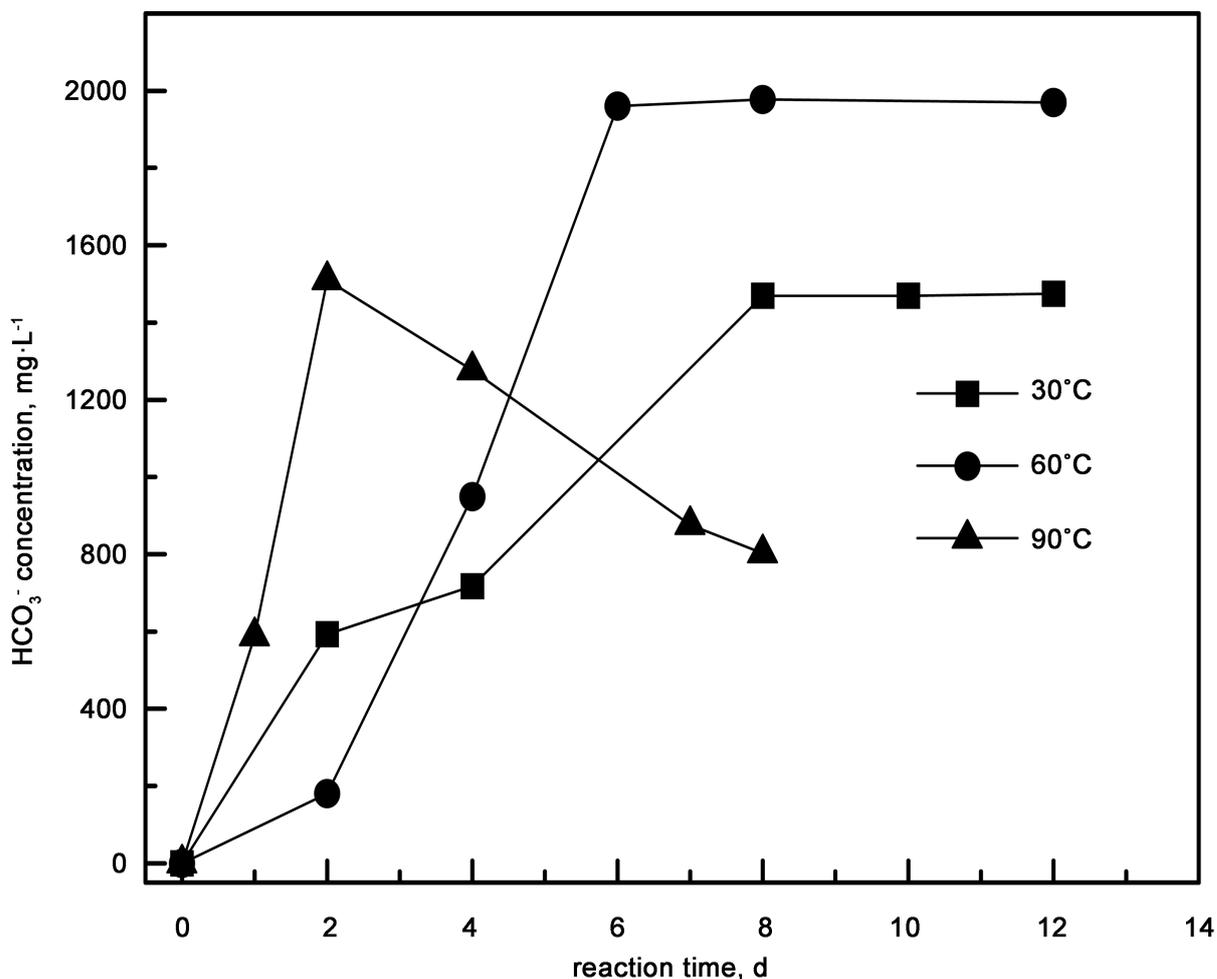


Figure 9. HCO₃⁻ ion concentration vs. reaction time at different reaction temperature.

maximum after reacted with CO₂ for 2 days at 90°C, and then decreased with the increase in the reaction time. HCO₃⁻ ion concentration in the solution showed the similar trend. The final Ca²⁺ ion concentration and HCO₃⁻ ion concentration at 90°C were all lower than those at 60°C. This is because when the temperature is higher than 50°C, the dissociation constant of carbonic acid decreases with the increase of temperature [33]. So the H⁺ ion concentration in the solution is reduced, the effective H⁺ ion concentration which can react with CaCO₃ is also reduced, which lead to the less corrosion of CaCO₃, lower concentration of Ca²⁺ ion and HCO₃⁻ ion in the solution.

3.4. Water Permeability of Sand-Packed Model after Reacted with CO₂

The dissolution of calcite to Ca(HCO₃)₂ in the solution under the interaction of CO₂-water-rock would affect the permeability of reservoir rock. **Figure 10** shows that the change of water permeability of sand-packed model filled with mixed grained calcite and quartz under the presence of CO₂ and water at 65°C. Before injecting gas, the water permeability of the sand-packed model remained relatively stable during placing for 3 days. After injecting CO₂, the water permeability of

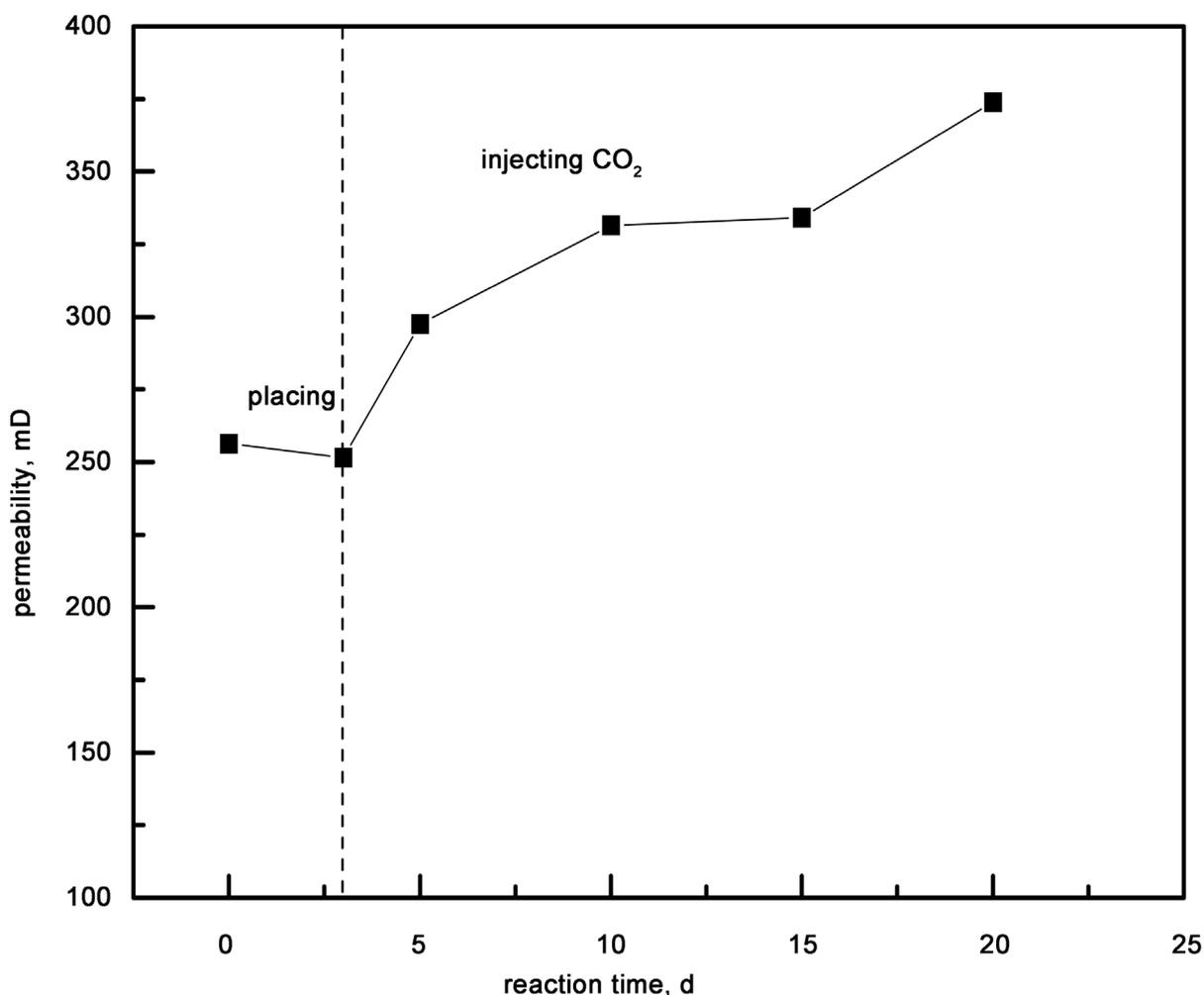


Figure 10. Water permeability of sand-packed model vs. reaction time, 65°C.

the model increased with the increasing of the reaction time. The reason caused the results above was that the grained calcite (CaCO_3) in the sand pipe reacted with injected CO_2 and water, and partial dissolution of the calcite occurred, which led to the appearance of the larger pores and the increase in the permeability of the model. Therefore, the porosity and permeability of reservoir containing the chemical component of CaCO_3 will be likely increased due to the mineral dissolution of CaCO_3 during CO_2 flooding, which will affect the efficiency of CO_2 flooding.

4. Conclusions

- 1) The mineral dissolution of calcite would occur when interacting with injected CO_2 and water, and the dissolution caused the change of surface texture of calcite.
- 2) The surface dissolution of calcite appeared more obvious with the increase of pressure when reacting with injected CO_2 and water.
- 3) The dissolution of calcite caused the increase in Ca^{2+} , HCO_3^- ion concentration in the solution. The dissolution equilibrium would be brought into much

faster at higher reaction temperature.

4) When the reaction temperature is lower, the dissolution of calcite increases with the increase of temperature. When the reaction temperature reaches up to a certain value, the dissociation constant of carbonic acid decreases which leads to less dissolution of calcite.

5) The dissolution of calcite caused improvement in water permeability of sand-packed model filled with mixed grained calcite and quartz.

Acknowledgements

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