

Experimental study on interaction between simulated sandstone and acidic fluid

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Abstract: In order to investigate the controlling mechanism of temperature, fluid and other factors on water-rock interaction in the diagenetic process, we performed a series of simulated experiments on the interaction between two kinds of fluids with different salinity and a composite mineral system (simulated sandstone), which contains albite, K-feldspar and other minerals. The experimental results showed that acidity was the most important factor that affected the dissolution of minerals in the composite mineral system. The lower the pH value, the more easily the minerals dissolved. At the same pH value, the dissolution abilities of different acids for various mineral components were also different. Compared to hydrochloric acid (inorganic acid), oxalic acid (organic acid) was more able to dissolve aluminosilicate minerals. However, the dissolution ability of oxalic acid for carbonate minerals was lower than that of hydrochloric acid. In the process of fluid-rock interaction, dissolution of feldspar was relatively complicated. Increase of temperature would accelerate the dissolution of feldspar. Under acidic conditions, albite had a higher dissolution rate than K-feldspar. K-feldspar could dissolve and convert into montmorillonite and kaolinite, while albite could dissolve and convert into kaolinite both at 40°C and 80°C. Presence of organic acid, and decrease of pH value and water salinity were all favorable for the dissolution of feldspar, but weakened the ability to form clay minerals.

Key words: Fluid, simulated sandstone, dissolution, static experiment

1 Introduction

Since the 1970s, people have gradually recognized that the formation of secondary pores in the sandstone reservoirs is one of the key factors affecting the nature of sandstone reservoirs. Over one third of the known sandstone pores at present are secondary pores, which can be regarded as the key reservoir space and permeability in the reservoir beds of many oil and gas fields in the world, and are probably more numerous than primary pores (Xiang et al, 2004). Secondary pores in sandstone reservoirs are controlled by diagenesis (Qiu et al, 1997; Anjos et al, 2000; Dillon et al, 2004). The formation mechanism of secondary pores is closely related to the dissolution of skeleton particles, such as feldspar and carbonate cementation in the burial diagenesis process (Liu and Zhao, 1993; Chen et al, 1999; Shah and Bandyopadhyay, 2005; Li et al, 2005; Ji and Xu, 2007). Therefore, people have made a lot of simulated experiments to study the interaction between fluid and a single mineral, such as feldspar, and to explore the dissolution mechanisms and controlling factors of diagenesis (Wollast and Chou, 1992; Chen et al, 1993; Hellmann, 1994; Shi et al, 1994; Oelkers and Schott, 1995; Xiao et al, 2003; Xiang et al, 2004; Chardon et al, 2006). Research results showed that the dissolution of feldspar is related to its composition and structure, reaction temperature and pressure, and fluid properties. The dissolution of feldspar

is controlled by surface reaction and diffusion. Very few experimental studies on sandstone dissolution have been focused on the dissolution characteristics of feldspar and the formation mechanism of secondary pores (Yang et al, 1995; Ji and Xu, 2007). We performed simulated experiments on the interaction of fluid and a composite mineral system to learn more about the conversion of the mineral components, the effect of reaction temperature and the nature of fluid upon the water-rock interaction in the diagenetic process, as well as to collect data under different experimental conditions for deeper understanding of the fluid-rock interaction mechanism.

2 Experimental principle and approach

2.1 Experimental principle

This research used a static mineral immersion method to study the influence of temperature, pH value, water salinity, and organic acid content on fluid-rock interaction, mainly through the change of fluid composition before and after the reaction. In the simulated experiment, the solid samples are all complex mineral systems containing K-feldspar, albite, quartz, calcite, and other minerals. The compositions of the minerals are similar to those of actual detrital reservoir rocks.

2.2 Experimental samples

2.2.1 Preparation of sandstone samples

Fresh Yangfang granodiorite was used. By crushing,

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screening, and magnetic separation, mixture samples of <0.1 mm, 0.1-0.25mm, 0.25-0.5mm in size that comprise K-feldspar (63%), albite (10%), quartz (25%), and biotite (1%) were obtained. Then the samples were mixed with 1% of Fengshan limestone of the same particle size. Finally the samples were cleaned with ultrasonic wave, and dried for use after wet-screening.

Polished sections were analyzed with an electron microprobe and the results for K-feldspar and albite are shown in Table 1.

Table 1 Chemical compositions of K-feldspar and albite samples

Oxide wt%	K-feldspar			Albite		
	Yfgr-1	Yfgr-2	Yfgr-7	Yfgr-3	Yfgr-4	Yfgr-8
SiO ₂	65.66	66.39	66.1	67.66	66.91	68.3
TiO ₂	0.02	0	0.05	0.02	0.05	0
Al ₂ O ₃	18	17.85	18.51	19.87	19.82	19.93
Cr ₂ O ₃	0	0.09	0	0.12	0.01	0
FeO	0.14	0.05	0.07	0.26	0.12	0.19
MgO	0	0	0	0	0.06	0
MnO	0.07	0.04	0.08	0	0	0.15
CaO	0.12	0.05	0.28	1.01	1.09	0.55
NiO	0	0.18	0.04	0.04	0	0.24
CoO	0	0.13	0	0	0	0.05
Na ₂ O	1.5	0.73	1.34	11.12	10.77	10.94
K ₂ O	14.43	13.47	13.59	0.09	0.43	0.16
P ₂ O ₅	0	0.1	0	0	0	0
Total	99.94	99.08	100.06	100.18	99.25	100.51

2.2.2 Preparation of water samples

Two types of water samples were used in the experiment: distilled water and Dongying simulated water. Dongying simulated water was prepared according to the chemical composition of ancient salt water in the Dongying Depression, Bohai Bay Basin, as shown in Table 2. According to the experiment requirement, a certain amount of distilled water or simulated Dongying water was mixed with oxalic

Table 2 Chemical compositions of experimental water

Water type	Ion content, mg/L					Total salinity, mg/L
	K	Na	Mg	Cl	SO ₄	
Dongying simulated water	81.3	2239.6	245.5	3846.9	537.7	6950.9

acid, and the pH value was adjusted by adding hydrochloric acid or sodium hydroxide.

2.3 Experimental approach

1000mg of solid sample was put into a 250mL conical flask with stopper, and 36mL experimental water was added. The sealed flask was put into a constant temperature incubator first for heating for 15 minutes, and then the reaction duration was recorded.

After the step above, the flask was cooled by flushing with tap water. The solid was filtered from the liquid with a 0.45µm membrane filter and the pH value and the chemical composition of the liquid were measured. The pH value was measured with an ORION720 pH meter with an accuracy of 0.01. Ca, Mg, Fe, Si, Ba in the solution were measured by ICP-AES, with an accuracy of 0.01 µg/mL. The K and Na components were measured by WGH-I flame photometer manufactured in Shanghai with an accuracy of 0.1 µg/mL and a measuring range of 0-35 µg/mL. Samples beyond the above range were diluted appropriately before final measurement. The static experimental conditions of each group are shown in Table 3.

Table 3 Static experimental scheme

Serial number	Numbering	Temperature °C	pH	Oxalic acid content mmol/L	Particle size, mm	Water types
1	RCA-1	40	4	5	0.1-0.25	Distilled water
2	TCA-1	40	8	5	0.25-0.5	Dongying simulated water
3	RCB-1	80	4	5	<0.1	Distilled water
4	TCB-1	80	6	5	0.1-0.25	Dongying simulated water
5	RB-1	80	2	5	<0.1	Dongying simulated water
6	RB-2	80	4	0	<0.1	Distilled water
7	RB-3	80	2	0	<0.1	Dongying simulated water
8	RB-4	80	2	5	<0.1	Distilled water
9	RB-5	80	4	0	<0.1	Dongying simulated water
10	TB-2	80	7	5	0.1-0.25	Dongying simulated water
11	TB-3	80	4	5	0.1-0.25	Dongying simulated water

3 Experimental results and discussions

3.1 Interaction between acidic solution prepared from distilled water and the composite mineral system

Fig. 1 illustrates the change of concentration of Al, Ca, K, Mg, Na, and Si in the solution with the reaction duration in the interaction process between acidic solution prepared from distilled water and composite mineral system. Fig. 1 indicates the following:

1) During the experiment, the oxalic acid solution of pH=2 favored the dissolution of Al in the composite mineral system and the concentration of Al in the solution increased rapidly with the reaction duration, while the other kinds of solutions had relatively poor ability of dissolving Al in the composite mineral system.

2) The oxalic acid solution of pH=2 had the strongest ability of dissolving Ca in the composite mineral system. The concentration of Ca in the solution increased rapidly with the reaction duration. The solution without oxalic acid of pH=4 ranked the second in dissolving Ca, and the dissolved amount of Ca increased little with increasing reaction duration and approached a balance. Oxalic acid solution of pH=4 had relatively poor ability of dissolving Ca both at 80°C and 40°C.

3) The solubility of K in oxalic acid solution of pH=2 gradually increased with increasing reaction duration, and the dissolved amount was the most of all experimental runs. Oxalic acid solution of pH=4 at 80°C and 40°C had a similar dissolution trend for K. It is noticeable that for the reaction duration from 50 hours to 200 hours, the dissolved amount of K changed greatly. From 50 hours to 100 hours, the dissolved amount of K increased rapidly and reached a maximum, whereas from 100 hours to 200 hours, the dissolved amount of K decreased rapidly. Thereafter, the dissolved amount of K increased slowly again with increasing reaction duration.

4) The oxalic acid solution of pH=2 had the strongest ability of dissolving Mg in the composite mineral system. The concentration of Mg in the solution increased rapidly with the reaction duration. As for other solutions, the trends of dissolved amount of Mg were roughly similar. However, the dissolved amounts were different. Oxalic acid solution of pH=4 at 40°C and 80°C also had strong ability of dissolving Mg with a slight difference, while the solution without oxalic acid of pH=4 had the weakest dissolution ability.

5) Eliminating the initial concentration of Na in all solutions before reaction, it was found that the pH=2 oxalic acid solution had the strongest ability of dissolving Na in the composite mineral system. The other solutions had basically similar ability of dissolving Na.

6) The dissolution trend of pH=2 oxalic acid solution for Si in the composite mineral system was almost the same as that for Al. However, the dissolved amount of Si was larger than that of Al, and far larger than that of Si in other three solutions. Throughout the reaction, the change of the dissolved amount of Si was roughly the same among the three solutions. However, it was noticeable that oxalic acid solution of pH=4 dissolved the smallest Si at 40°C, and the dissolved amount was smaller than that in the solution without oxalic

acid of pH=4 at the reaction temperature of 80°C.

The above experimental results showed that:

1) Although the calcite content in the composite mineral system was very low, the dissolution rate of Ca was higher than those of Si, Al, K, and Na. This undoubtedly proved that the solubility of calcite was higher than various aluminosilicate minerals.

2) In the composite mineral system after dissolution, the absolute content of K-feldspar was around 63%, and the albite content was about 10%. Nevertheless, in the whole experiment, the dissolved amount of Na was larger than that of K, and the Na/K ratio was larger than the ratio of albite and K-feldspar content in the original samples, which showed that albite dissolved more readily than K-feldspar. This conclusion agrees with the result of the experiment by Huang et al (1995) who used perthite as the dissolution sample; i.e., the dissolution rate of albite is higher than that of K-feldspar.

3) The acidity of solution was the most important factor in dissolution of minerals in the composite mineral system. The lower the pH value, the stronger the dissolution ability. At a strong acidity (pH=2), the solution had the strongest dissolution ability other than Na, and the cations in the solution increased rapidly with the reaction duration. The other solutions had relatively poor dissolution ability for cations in composite mineral system.

4) When the pH value was the same, different types of acids had different dissolution ability for various mineral components. Oxalic acid had stronger dissolution ability for Al and Mg in the composite mineral system. Other acids (mainly hydrochloric acid) had stronger dissolution ability for Ca in the composite mineral system. Except some particular reaction durations, oxalic acid and hydrochloric acid showed similar dissolution ability for K and Si. During the whole reaction duration, oxalic acid and hydrochloric acid showed almost the same dissolution ability for Na. Therefore, oxalic acid was more able to dissolve aluminosilicates than hydrochloride acid, but had weaker dissolution ability for carbonates compared with hydrochloride acid. This was probably because oxalic acid could easily form a calcium oxalate precipitate with Ca^{2+} , which would obstruct further reaction.

5) Except Ca, the oxalic acid solution of pH=4 at 80 °C had higher dissolution ability for Al, K, Mg, Na, and Si in the composite mineral system than that at 40°C. Therefore, a higher reaction temperature was advantageous to aluminosilicate dissolution in oxalic acid solution but disadvantageous to carbonate dissolution, because the ionization ability of oxalic acid was enhanced with the increase of temperature, oxalic acid could provide more H^+ and oxalate for complexing metal elements, which improved the dissolution of aluminosilicate.

3.2 Interaction of Dongying simulated water and composite mineral system

(1) The comparison of reaction results of Dongying simulated water-composite mineral system with an experimental duration of 1,000 hours between the solution containing oxalic acid (5mmol/L) and the solution without

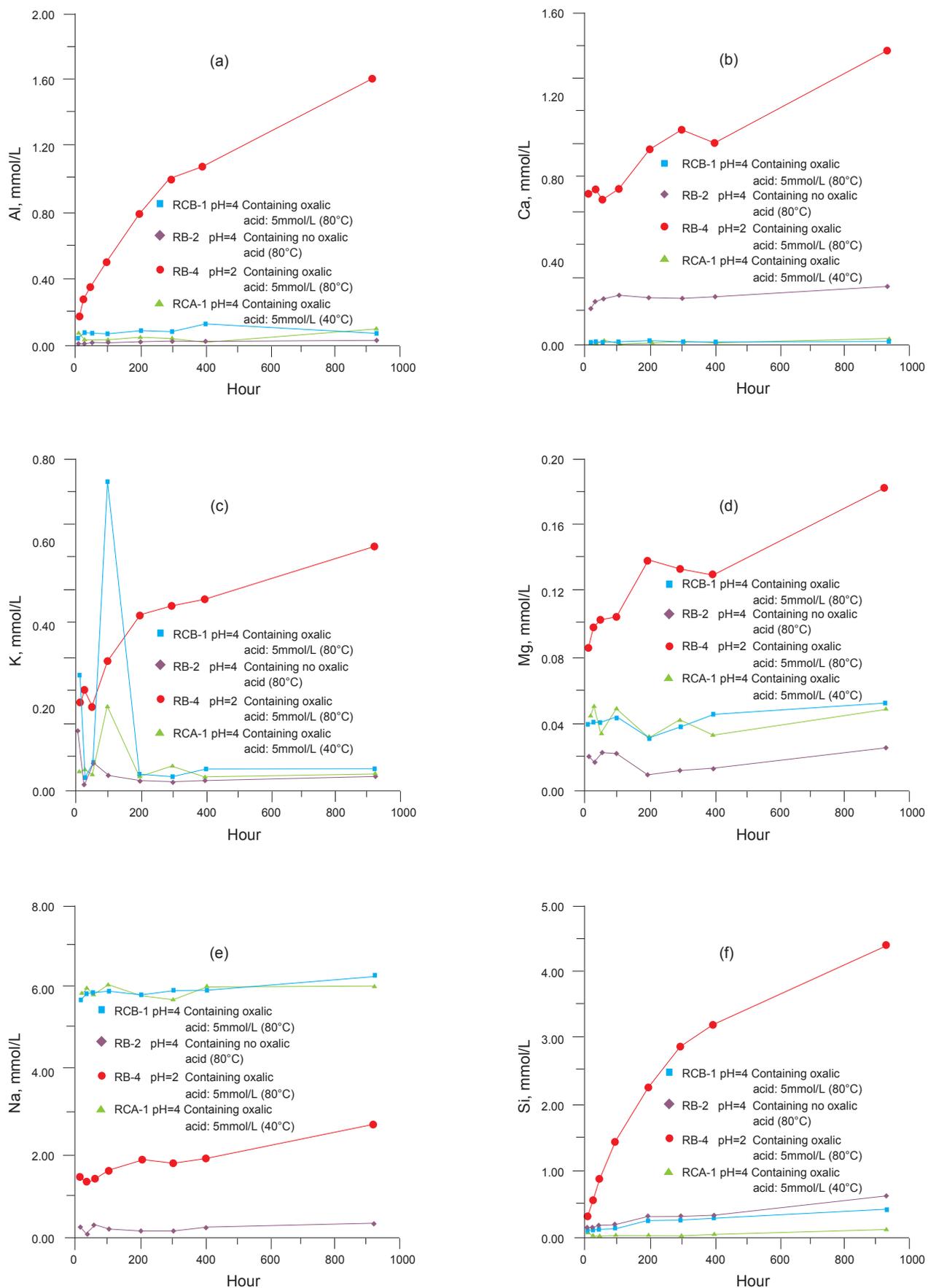


Fig. 1 Concentration change of Al, Ca, K, Mg, Na, and Si in the reaction process between composite mineral system and acidic solution prepared from distilled water

oxalic acid is as follows (Fig. 2(a) and (b)):

When Dongying simulated water contained oxalic acid, various chemical components were transferred from solid phase (minerals) to liquid phase (Dongying simulated water), which meant that dissolution was the major reaction. Only when pH=7, a very small amount of Mg, Fe, and Al were transferred from liquid phase to solid phase; that is to say, precipitation took place. When there was no oxalic acid and pH=4, Mg, Fe, and Al were transferred from liquid phase to solid phase, and the precipitation amount of Mg was the largest.

Dongying simulated water of pH=2 containing oxalic acid had the strongest ability of dissolving various mineral components. Dongying simulated water of pH=6 ranked the second. When there was no oxalic acid, Dongying simulated water of pH=2 had the strongest dissolution ability.

(2) Comparing the effect of oxalic acid on the dissolution of the composite mineral system between pH=2 and pH=4, we found that:

When pH=2, Dongying simulated water with oxalic

acid had stronger dissolution ability for various mineral components than that without oxalic acid. However, the change of the dissolved amount of various chemical components was almost the same. This indicated that when pH=2, the differences in the type of acidic medium only affected the dissolved amount and had no obvious effect on the trend of dissolution/precipitation of various mineral components (Fig. 2(c)).

When pH=4, the dissolution ability and reaction results of Dongying simulated water with and without oxalic acid differed greatly. Dongying simulated water with oxalic acid mainly caused the dissolution of various mineral components that were transferred from solid phase to liquid phase. However, Dongying simulated water without oxalic acid caused precipitation. At this time, the dissolved amounts of Ca and Si were much higher than those in Dongying simulated water with oxalic acid (Fig. 2(d)). Therefore, when pH=4, the acidic medium not only affected the dissolved amount of mineral components, but also had a major influence on the tendency of dissolution/precipitation.

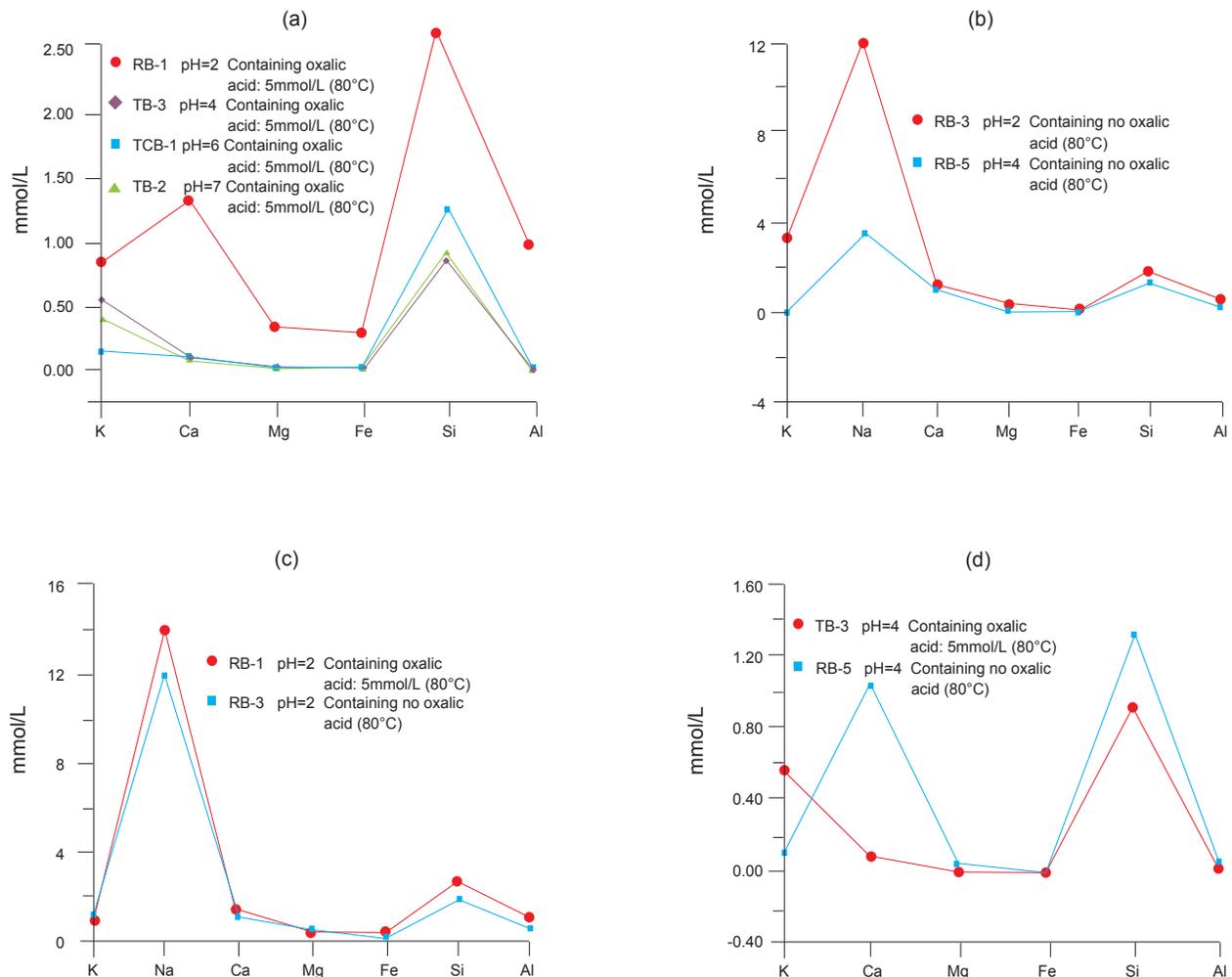


Fig. 2 Dissolved amount of various components of composite mineral system in Dongying simulated water containing different acidic medium

Through simulated experiments of the interaction between distilled water or Dongying simulated water and composite mineral system, we can conclude that the influence of water salinity on the solubility of various mineral components was different at different pH values. When pH=2, Ca, Si, and Al were more easily dissolved in distilled water than in Dongying simulated water, but Dongying simulated water

was more able to dissolve Na in the composite mineral system than distilled water (Fig. 3(a)). Dongying simulated water of pH=4 had higher dissolution ability for K and Si in the composite mineral system than distilled water (Fig. 3(b)). In conclusion, the dissolution ability of the fluid for the composite mineral system would become weaker with increasing water salinity.

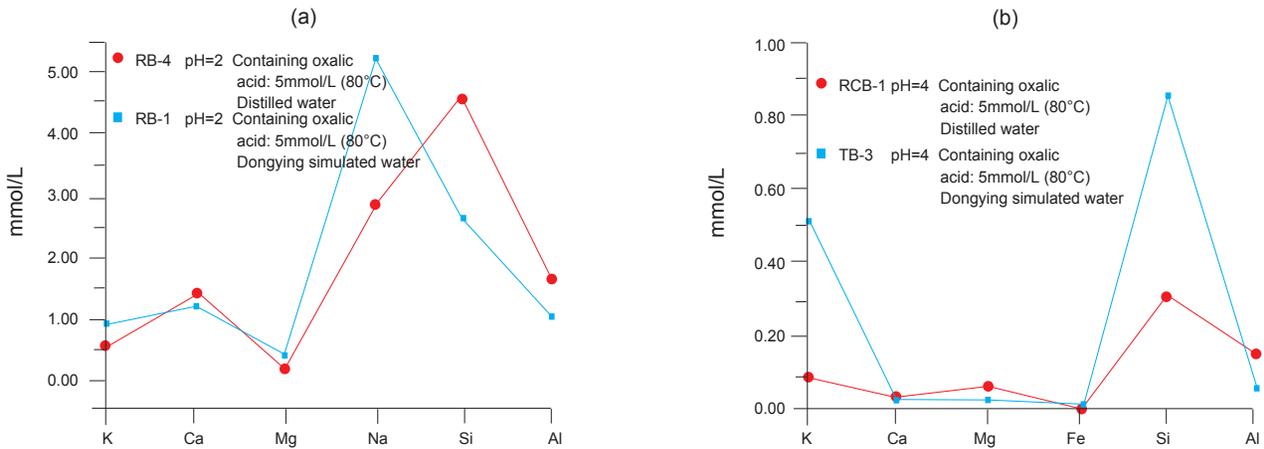


Fig. 3 Influence of salinity on dissolved amount of various components in composite mineral system at different pH values

3.3 Controlling factors of feldspar dissolution

In the process of fluid-rock interaction, the dissolution of feldspar is relatively complicated. There are many factors affecting the dissolution and conversion of feldspar, including its composition and structure, temperature and pressure of reaction, fluid pH value, and types and contents of organic acids. Research and discussions on various factors of feldspar dissolution and conversion under experimental conditions are of great significance for the study of dissolution mechanism of feldspar and development of secondary pores in the

process of diagenesis.

3.3.1 Influence of temperature on feldspar dissolution

The result of thermodynamic calculation shows that the stable area of intermediates for feldspar dissolution varies at different temperatures. With respect to K-feldspar, the stable area of montmorillonite enlarges significantly at 40°C compared to that at 80°C (Fig. 4(a) and (b)). With respect to albite, the stable area of montmorillonite enlarges slightly at 40°C compared to that at 80°C. By comparison of various minerals, the stable area of secondary montmorillonite from albite dissolution is much smaller than that of K-feldspar.

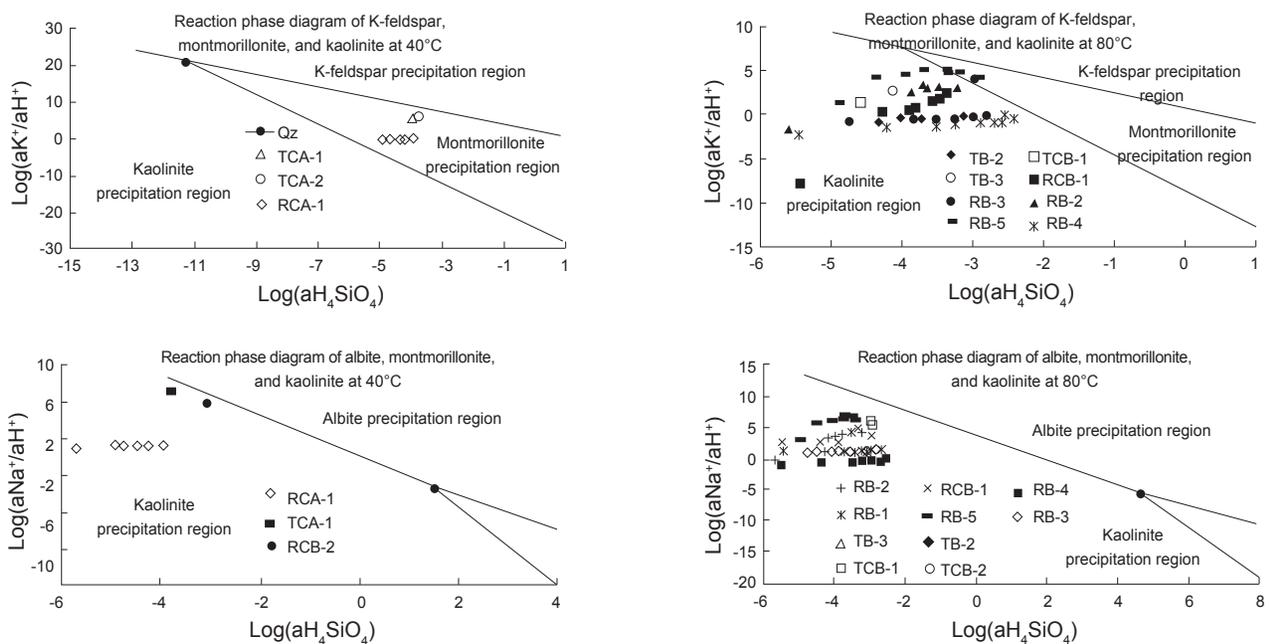


Fig. 4 Reaction phase diagram of K-feldspar (albite)-montmorillonite-kaolinite at 40°C and 80°C (Brantley, 1992)

That is to say, K-feldspar dissolves more easily and converts into montmorillonite at 40°C, and K-feldspar dissolves more easily and converts into kaolinite at 80°C. However, it is difficult for albite to be decomposed and convert into montmorillonite whether the temperature is high or low (Fig. 4(c) and (d)).

The simulated experimental results show that the decomposition intermediates of K-feldspar were all distributed in montmorillonite region at 40°C, and were distributed in kaolinite and montmorillonite regions, mostly in kaolinite region at 80°C. The decomposition intermediates of albite were distributed in kaolinite region whether the temperature was high or low (Fig. 4).

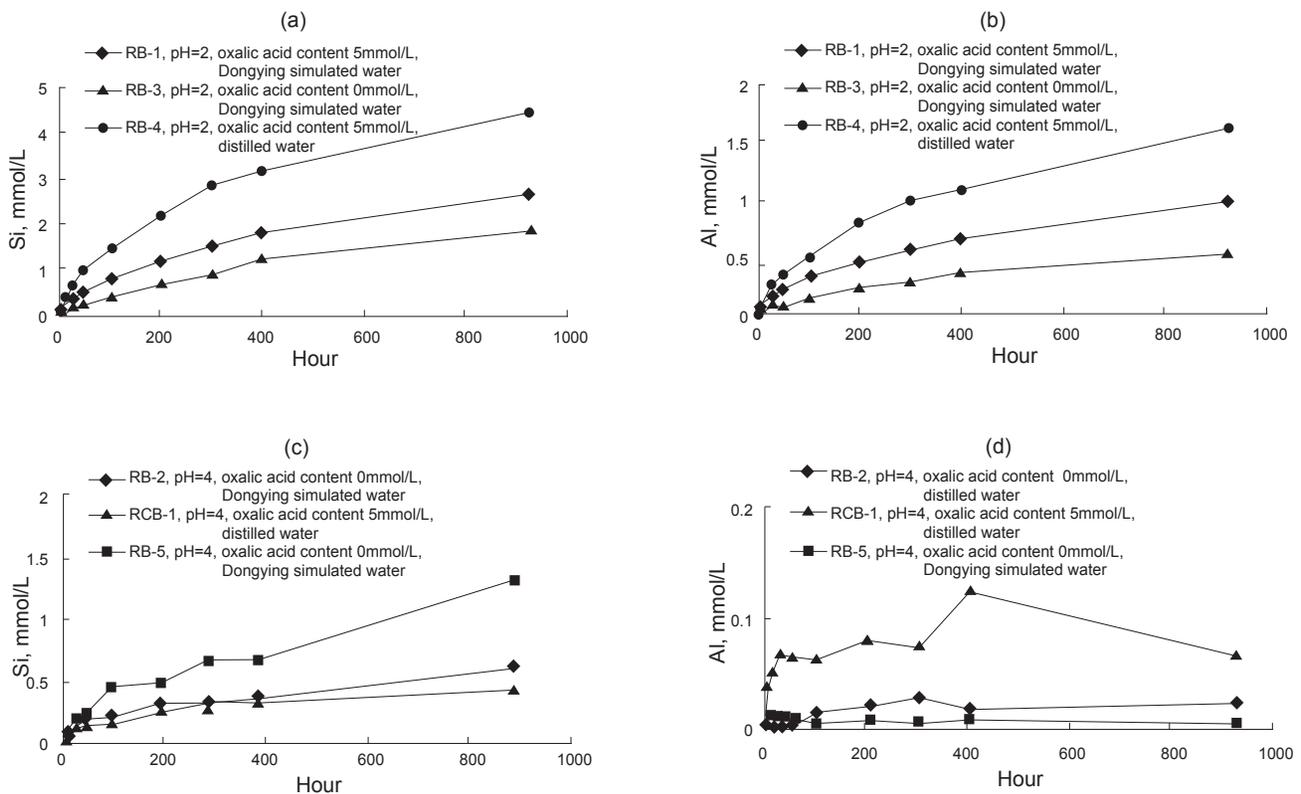


Fig. 5 Concentration change of Si and Al in solutions containing organic acid or not

3.3.3 Influence of pH on feldspar dissolution

Provided that the pH values were different while other conditions remained the same, the change of Si and Al contents in the water-rock interaction system demonstrated that Si and Al contents in the solution increased significantly with decreasing pH value, and the Si/Al ratio greatly decreased (Fig. 6). Decrease of pH value would enhance the complete decomposition of feldspar, while the increase of pH value would make feldspar convert into clay minerals.

The hydrocarbon generation process of organic matter is accompanied with the release of a large amount of organic acids into the water phase. When the water enters into the reservoir rocks, the contained organic acids could accelerate the complete decomposition of feldspar. Besides, it also helps

3.3.2 Influence of organic acids on feldspar dissolution

The experimental results of the solution with 5 mmol/L oxalic acid and without oxalic acid showed changes in Si and Al contents over time. Si and Al contents increased considerably over time when the solution contained oxalic acid. Si content increased to a specific degree over time when the solution contained no oxalic acid, but the Al content remained at a relatively low level (Fig. 5). This phenomenon proved that the presence of organic acids was favorable to dissolution of Si, Al, K, Na, Ca, and other components into the solution. The feldspar dissolution in the solution without organic acid occurred along the reaction pathway of converting to new clay minerals and the undissolved Al crystallized into new minerals.

to reduce the pH value of pore water in original reservoir rocks, which becomes an important factor for further dissolution of feldspar and the formation of secondary pores.

3.3.4 Influence of water salinity on feldspar dissolution

The impact of water salinity on feldspar dissolution is complex. In the condition of low pH value (pH=2), the Si and Al contents in the solution increased with the decrease of water salinity (Fig. 7(a) and (b)). At a high pH value (pH=4), Al content in the solution increased and Si content decreased with the decrease of salinity (Fig. 7(c) and (d)). This demonstrated that the increase of water salinity was disadvantageous to the complete decomposition of feldspar, but advantageous to the formation of clay minerals.

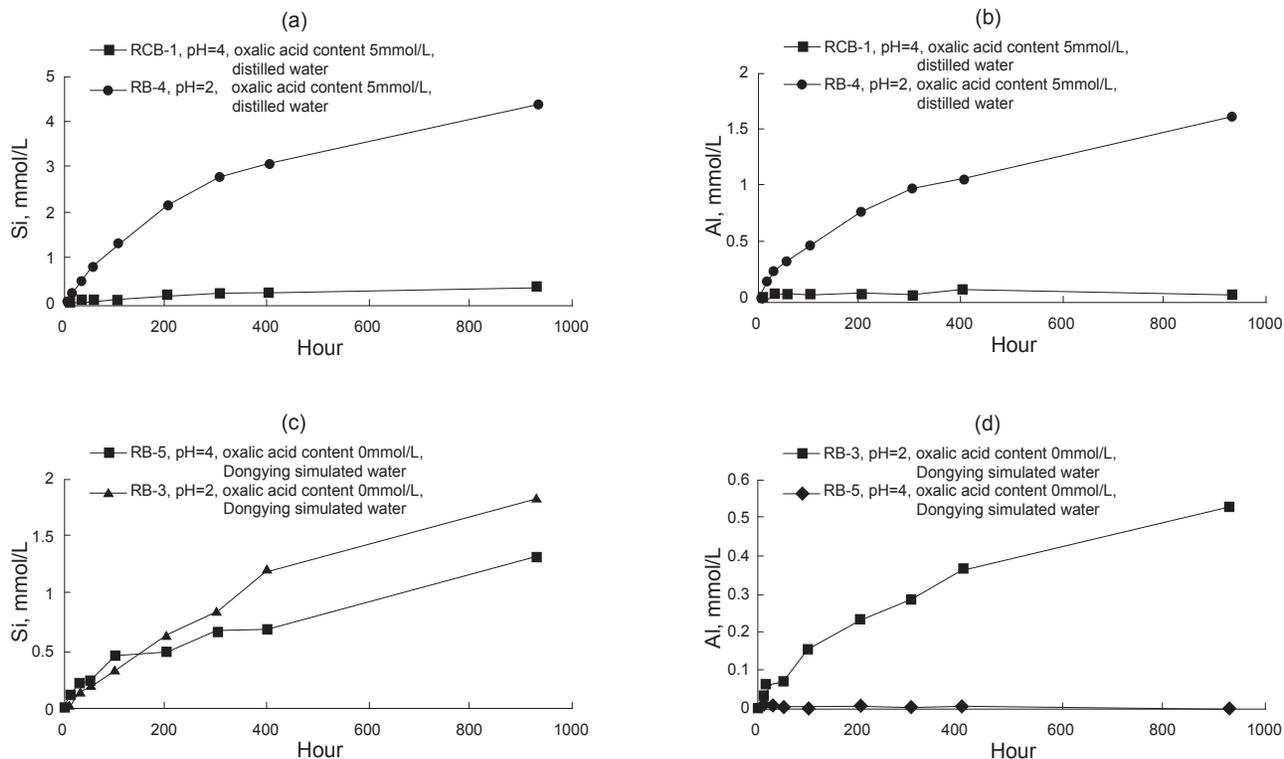


Fig. 6 Concentration change of Si and Al in distilled water and Dongying simulated water with different pH values

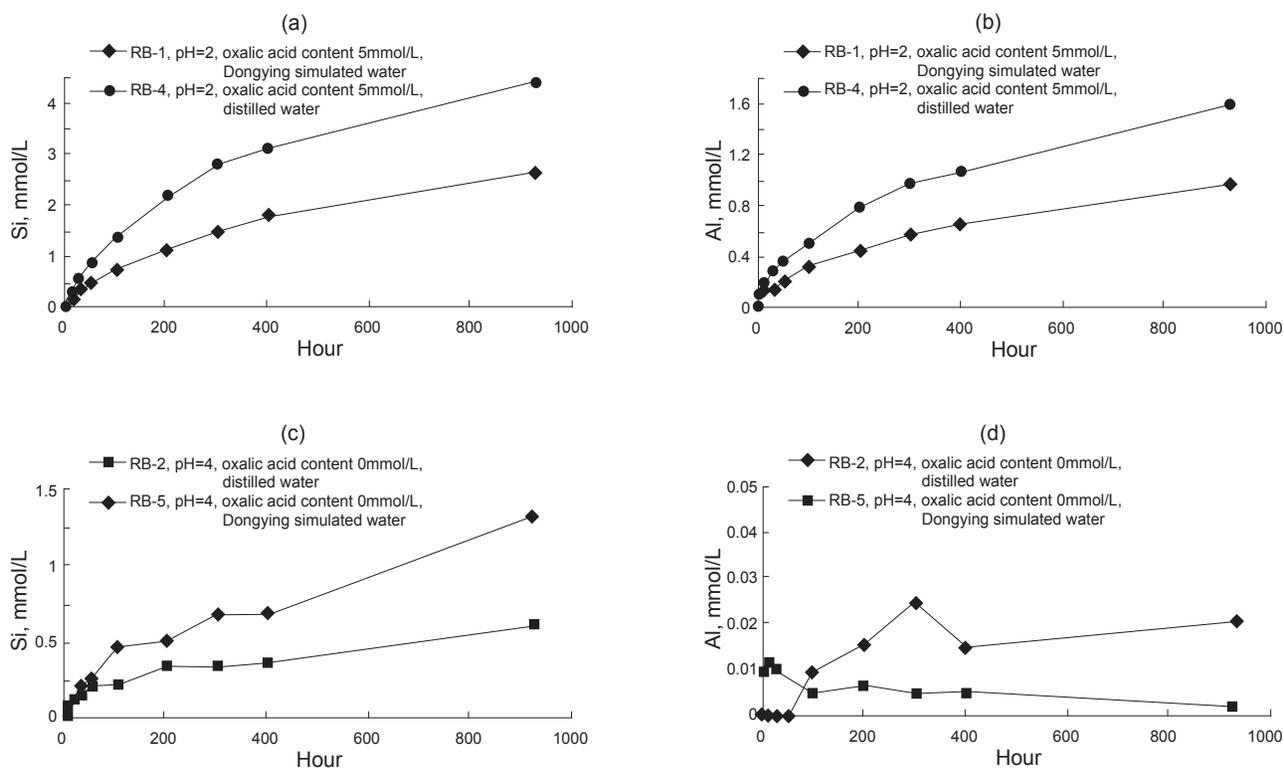


Fig. 7 Concentration change of Si and Al in solutions with different salinity at pH=2 and pH=4

The research of Megahan and Clayton (1986) showed that the lower the water salinity, the more easily the feldspar would dissolve. Blake and Walter (1996) held the opinion that the dissolution rate of orthoclase decreased with increasing

salinity. The results of Blake and Walter (1999) supported the above opinion. Organic acid in the fluid, and decrease of NaCl enhanced dissolution of feldspar. Alkali cations such as Na⁺ decreased the dissolution rate of feldspar in dilute acidic

solutions by competing with protons for reactive exchange and adsorption sites on the feldspar surface, which can decrease the proton-promoted effect.

4 Conclusions

1) In the process of fluid-rock interaction, the solubility of carbonate minerals was higher than aluminosilicate minerals. The dissolution rate of albite was higher than that of K-feldspar under acidic conditions, which is consistent with the simulated experimental results of feldspar single mineral.

2) The pH was the most important factor affecting the dissolution of mineral components. The lower the pH value of the fluid, the stronger the solubility of the mineral components. Under strongly acidic conditions, the difference in types of acidic medium only affected the dissolved amounts of various mineral components, and it did not have an obvious influence on the dissolution/precipitation trend. When the pH value was the same, oxalic acid had stronger dissolution ability for aluminosilicate minerals compared with hydrochloride acid. Hydrochloric acid had stronger dissolution ability for carbonate minerals. A higher temperature favored dissolution ability of oxalic acid for aluminosilicates but not for carbonates.

3) Experimental studies showed that an increase in temperature was favorable to feldspar dissolution. K-feldspar can form clay minerals, such as montmorillonite and kaolinite under acidic conditions. The decomposition intermediates of albite were distributed in kaolinite region, whether the temperature was high or low. The presence of organic acids was favorable to the dissolution of Si, Al, K, and Na components of feldspar into the solution as ions, and the dissolution of feldspar without organic acids occurred along the reaction pathway of the formation of new clay minerals. The influence of water salinity on feldspar dissolution was rather complex. Generally speaking, increase of water salinity was favorable to the formation of clay minerals but unfavorable to the complete decomposition of feldspar.

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