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New Method to Differentiate between Matrix and Micro-fractures Using Diffusion Data in Gas Shale

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ABSTRACT: The fact that salt ions in shale pores diffuse into fracturing fluids is key factor to lead to recovered water with high salinity. In this paper, the authors conduct the test of mineral composition and SEM to understand the reservoir characteristics. The diffusion experiments are conducted on crushed samples, and a new method is proposed to differentiate between matrix and micro-fractures by using diffusion data. A large amount of salt ions exit in shale pores and can diffuse into fracturing fluids after fracturing operations. To a great extent, ion diffusion rate is determined by the development of microfractures. The crushed samples with smaller grain diameter contain have lower diffusion rate due to the low probability of microfractures development. When the grain diameter is lower than critical value, the crushed samples cannot contain microfractures. As for Longmaxi formation sample, the fracture-matrix boundary is about 80mesh.The research contributes to understanding the reservoir characteristics and salinity profiles of gas shale.

1. INTRODUCTION

The field observations show that the salinity of recovered water is generally high. What's more, the salinity increases continuously over time and even exceeds 10%. It should be noted that the salinity of slick water is about 0.1%. The researchers tend to attribute this observation to the salt ions diffusion into fracturing fluids (Wang et al., 2016).

The salt ions concentration and type in recovered water can act as the indicator to evaluate the development of fracture network. Unlike primary fractures, the secondary fractures are induced fractures that are covered by connate water film. The connate water film can mixes easily with fracturing fluids to increase the salinity of fracturing fluids (Woodroof et al., 2003). The secondary fractures with smaller aperture size tend to forms high exposure area that can enhance the ion diffusion capacity. In addition, the ion type in secondary fractures is different from that in primary fractures (Gdanski et al., 2007). The study found that Ba^{2+} exits in secondary fractures and the development of micro-fractures are evaluated based on the concentration of Ba^{2+} (Agrawal and Sharma, 2013).

The ions transport from shale to fracturing fluids includes two dominant mechanisms (i.e. convection and diffusion) (Haluszczak et al., 2013). At the beginning stage of flowback, the fracturing fluids flow on the fracture surface, intensifying the ion convection to enhance the salinity of fracturing fluids. Nevertheless, the ion convection is not obvious in shale matrix pores, and ion diffusion is the dominant mechanism (Ballard et al., 1994). At present, the researcher use laboratory experiment and field test comprehensively to study the ion transport mechanism. As for laboratory experiments, the shale samples are placed in the solution and conductivity meter is used to measure the solution conductivity to explore the ions transport characteristics and influencing factors (Ghanbari etal., 2013). As for the field tests, the ions concentration and type in recovered water is analyzed to obtain the salinity profiles (Yang et al., 2015).

The salinity profiles of recovered water can provide a great deal of information from fracture network development (Fakcharoenphol et al., 2014). Similarly, the ion diffusion characteristics are also used to study physical properties of shale. In this paper, the diffusion experiments are conducted on crushed samples with different grain diameters, and a new method is proposed to differentiate between matrix and micro-fractures by using diffusion data.

2. EXPERIMENTS AND METHODS

2.1. *Materials*

Longmaxi formation is the most potential for shale gas production, and small-scale commercial exploitations have been realized in Sichuan Basin of China. Longmaxi shale is the most representative marine shale. The shale reservoir thickness is about 9~23m. The total organic carbon (TOC) is about is 2.3%. The maturity Ro is about 3.1%. The content of quartz and clay mineral is 44% and 30% respectively (Fig. 1). The helium porosity is about 1.2%, and nitrogen permeability is about 0.0035 mD.

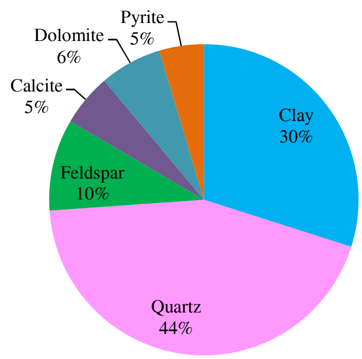


Fig. 1 The mineral composition of Longmaxi formation

Scanning electron microscope (SEM) can help understand the microstructure of shale. The pores in Loangmxi shale include mainly intergranular and intercrystalline pores, and the pore diameter is about 100nm~50um. In addition, the microfractures are extensively developed and fracture width is about 2~6 μm (Fig. 2).

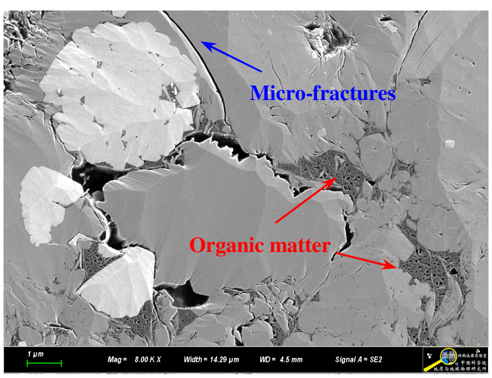


Fig. 2 SEM of shale sample

2.2. *Experimental setup*

The solution conductivity is determined by electrolyte concentration, and can act as an indicator to reflect the ions content and type. In general, the larger ions concentration, the higher solution conductivity is. In this experiment, the shale samples are crushed into powder and placed in the water. The conductivity meter is used to measure the solution conductivity to explore the ion diffusion characteristics. The experimental setup is conductivity meter of Mettler Toledo SevenExcellence (Fig. 3). The precision is 0.1uS/cm.

Fig. 3 The pictures of conductivity meter

2.3. *Experimental procedure*

The experimental procedure includes:

- The shale samples are crushed into power (i.e. 6 mesh, 8 mesh, 10 mesh, 20 mesh, 40 mesh, 60 mesh, 80 mesh, 100 mesh, 400 mesh), and are dried at 105℃.
- Take 10g of crushed samples into the 200ml deionized water, stir evenly and test the initial conductivity G_0.
- Keep fluid still, and test the conductivity G with time (Fig.4).

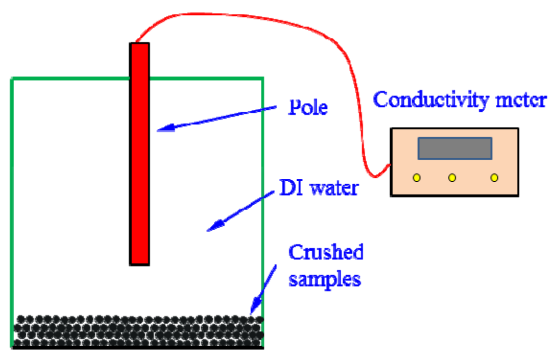


Fig. 4 The diagram of experimental methods

3. RESULTS

3.1. *Typical diffusion curves*

In Fig.5, the fluid conductivity increases with time, illustrating that the saline ions diffuse into fluid. In the initial stage, the slope of curve is relatively high and then tends to be gentle, which illustrates that ion diffusion process tends to be in equilibrium state.

According to Yang et al. (2017), ion diffusion and imbibition obey the same rule, which is in linear relation with the square root of time. The research approach on spontaneous imbibition can be used to analyze ion diffusion. The variation curve of fluid conductivity over the square root of time is drawn, as shown in Fig. 6. It can be seen that most of the conductivity curves are linear. The initial conductivity G_0 increases with the square root of time. While not all the curves are linear, the slope of curve is the ion diffusion rate D. As the diameter of particle gets larger, the correlation tends to present curve behaviors. The samples in 6 mesh, 8 mesh and 10 mesh have obvious multi-stage characteristics, and the ion diffusion rate goes from high to low, suggesting that LM formation develops micro-fractures. The higher ion diffusion rate at early stage is mainly caused by micro-fractures. Besides, it is worth noting that the multi-stage phenomenon gradually disappears as the meshes increase (the particle diameter decreases). When it reaches to 80 mesh, the multi-stage phenomenon completely disappears, and the curve tends to be straight. This shows that the micro-fracture size are continuously variable. As the particles diameter gets smaller, the micro-fracture that can exist gets smaller. The samples that is larger than 80 meshes does not contain micro-fractures, so the conductivity curve mainly reflect the characteristics of matrix ion diffusion.

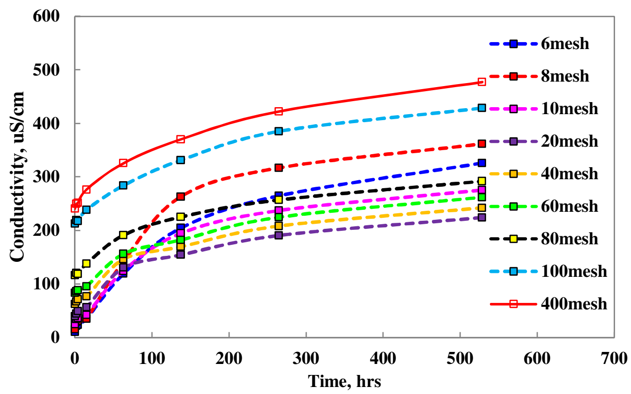


Fig. 5 The curves of conductivity vs. time

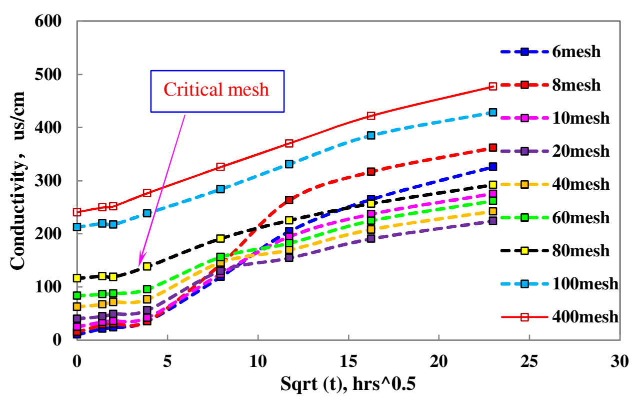


Fig. 6 The curves of conductivity vs. $t^{0.5}$

3.2. The pore connectivity exponent

The characters of micro-fracture and matrix is reflected in Fig.7. The curve can be divided into two phases: micro-fracture region Ⅰ and matrix region Ⅱ. As the mesh increases, the time index of Ⅰ gradually decreases from 0.5 to 0.3, while the time index of Ⅱ mainly keep stable at 0.26, as shown in Table 1. According to Hu et al. (2012), the time index of shale matrix is 0.26, which represents the relative low pore connectivity. The connectivity of micro-fracture is better than matrix. As the micro-fracture size gets smaller, the connectivity gets worse.

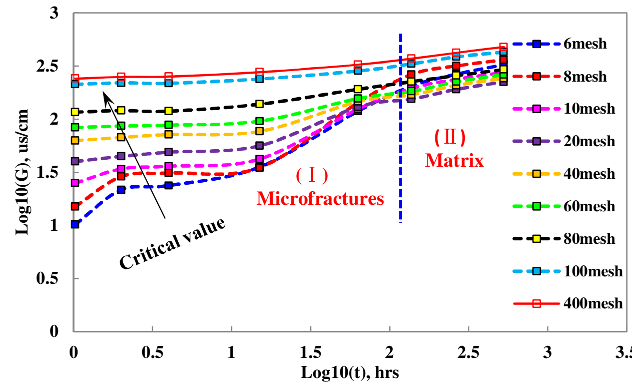


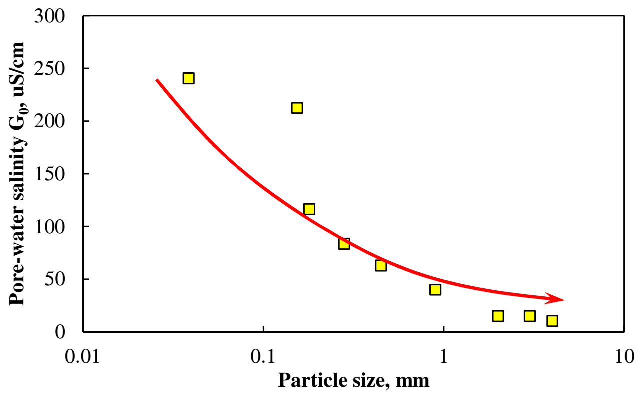
Fig. 7 The log-log plots of G vs. t

Table 1. The values of pore connectivity exponent

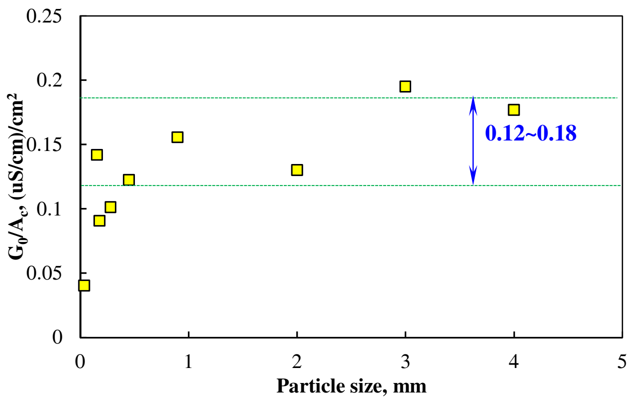
mesh	Pore-water salinity G_0,uS/cm	Diffusion rate D, us/(cm*$t^{0.5}$)	Ⅰ exponent	Ⅱ exponent
6	10.2	15.1	0.5	0.26
8	15	17.2	0.46	0.26
10	15	12.3	0.42	0.26
20	39.9	8.8	0.4	0.26
40	62.7	8.5	0.35	0.24
60	83.5	8.5	0.3	0.25
80	116.3	8.3	—	0.25
100	212.5	10.1	—	0.24
400	240.5	10.7	—	0.24

3.3. The separation between matrix and micro-fractures

The correlation between initial conductivity G_0 and contacting area is shown in Fig.8a, which can be seen that the initial conductivity increases as the contacting area increases. The initial conductivity is tested when the crushed sample and fluid are mixed, which reflects the surface ion adhesion amount of the sample. The correlation between G_0/Ac and contacting area is shown in Fig. 8b. It can be seen that the ion adhesion amount in per unit is very steady without the influence of particle size, which indicates that ion adhesion amount in per unit is a characteristic parameter.



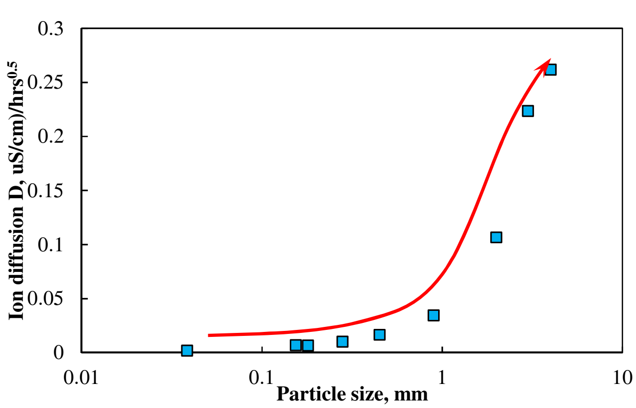
(a)



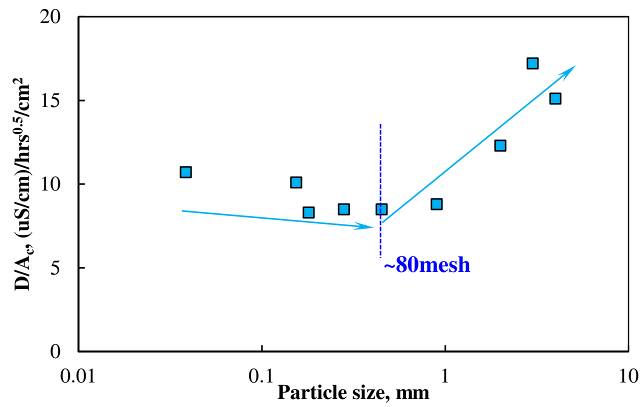
(b)

Fig. 8 The curves of G_0 and G_0/Ac vs. particle size: (a) G_0, (b) G_0/Ac

As can be seen from Fig. 9a, as the exposure area gets larger, the ion diffusion rate get higher. It conforms with conventional view. The correlation between D/Ac and contact area is shown in Fig. 9b. Different from initial conductivity G_0, D/Ac is not a constant. The diffusion rate gets lower as the particle diameter increases. When the diameter exceeds the critical value (80mesh), the ion diffusion rate increases rapidly. This indicates that micro-fracture can obviously improve ion diffusion rate. Moreover, the micro-fractures have a much higher impact on diffusion rate than the exposure area (Cui et al., 2009).



(a)



(b)

Fig. 9 The curves of D and D/Ac vs. particle size: (a) D, (b) D/Ac

Shale has complex multi-porosity feature that includes both matrix and micro-fractures (Fig.10). As the grain diameter of crushed samples gets larger, the possibility of micro-fracture existence gets higher. When the particle diameter is small enough, the particle only contains matrix pores. The boundary of Longmaxi shale is 80mesh.

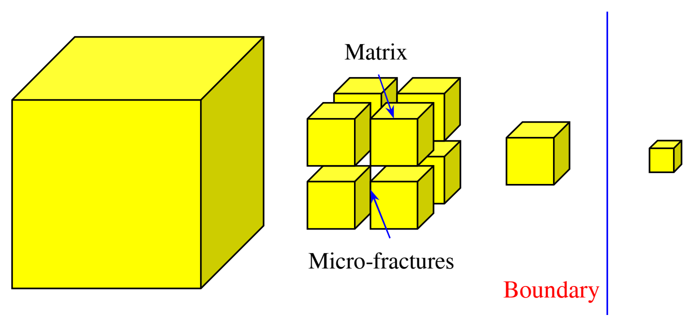


Fig. 10 The diagram of crushed samples

4. CONCLUSIONS

- Shale formation contains large amounts of saline ions. When contacting with water, a mass of saline ion dissolves into water, which is the main reason for high-salinity recovered water in shale gas well.
- A new method to distinguish micro-fracture and matrix based on conductivity experiments is proposed. The experiments showed that as the diameter of crushed sample gets smaller, the possibility of micro-fracture existence gets smaller. The fracture-matrix boundary of Longmaxi shale is 80 mesh.

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