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# Monitor the process of shale spontaneous imbibition in co-current and counter-current displacing gas by using low field nuclear magnetic resonance method

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ABSTRACT

Large scale fracturing fluid is injected into the formation to produce fractures for the effective development of shale reservoir. However, the flow back rate of fracturing fluid is often less than the half of the injected liquid, which causes large number of fracturing fluid retaining in the shale reservoir, thus aqueous phase trapping (APT) appears. But after well was shut in for a period of time, the APT can be auto-removed. The experiments that monitored the process of shale spontaneous imbibition in co-current and counter-current displacing gas combined with nuclear magnetic resonance (NMR) were performed. Results show that no matter whether the spontaneous imbibition of sandstone and volcanic rock happened in co-current or counter-current displacing gas, the water content increases in the whole pores range gradually, and no preferential pores for spontaneous imbibition appear. The water content increases with convex curve in the early period of both conditions. Simultaneously, in the process of the experiments no apparent micro cracks appeared on the surfaces of the sandstone and volcanic rock. However, shale has some special characteristics in spontaneous imbibition of both co-current and counter-current displacing gas, which may contribute to the auto-removal mechanism of shale reservoir APT. During the experiments of shale, lots of micro cracks appeared on the surface of the sample gradually. The liquid absorbed into the shale sample fills the micro pores firstly. Subsequently the water takes up the space of mesopores slowly. The liquid in the large pores of shale is too small to be detected, so the water content change in these pores couldn't be distinguished clearly. In the early period, the water content of shale increases with convex curve in the co-current displacing gas, while the water content of shale increases linearly in the counter-current displacing gas. Thus, the counter-current spontaneous imbibition condition is beneficial to protect the reservoir. The results of our study contribute to not only explaining the auto-removal mechanism of shale reservoir APT, but also fixing the optimal flow-back time after hydraulic fracturing.



## 1. Introduction

In order to exploit the shale reservoir efficiently, large scale hydraulic fracturing became an important method to develop shale formations (Roychaudhuri et al., 2013; Cheng et al., 2015; Liu et al., 2015). However, large amount of fracturing fluid retained in the formation after flow-back, which is usually more than 50% of the injected fluid. It severely influenced the formation permeability (Engelder et al., 2014; Liang et al., 2015). Thus, aqueous phase trapping (APT) appeared. ATP was a mechanism of formation damage. It occurred, in most cases, during drilling, completion, work over and stimulation operations. APT damages the formation permeability, especially in low permeable and sub-irreducible (initial water saturation is lower than irreducible water saturation) gas reservoir (Bennion et al., 1996). However, the APT can be auto-removed after well shut-in for a period of time. Subsequently, the gas productivity increased (Makhanov et al., 2014). Aiming at researching this special characteristic, lots of previous work has been done. A microscope has been used to monitor the first bubble released from the soaked shale. Based on this phenomenon, a

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simple and effective methodology was proposed to determine the post frac optimal shut-in time (Hayatdavoudi et al., 2015). In order to research the regained permeability after flow-back with shut-in time, the core flooding experiments were conducted. The tight sand cores did as comparative sample. It is believed that the same shut-in time had more damage to shale cores than tight sand cores permeability. Thus extending shut-in time had more damage to shale permeability (Yan et al., 2015). During exposure to brine, the permeability of Pierre shale increased. This behavior may be caused by mineral dissolution (during imbibition) and crack expansion (induced by clay swelling) (Wang et al., 2011). Extended shut-in time can not only reduce the water rate significantly in shale reservoir, but also increase the initial gas rate without exerting adverse influence to the long-term gas productivity (Cheng, 2012). Water adsorption of brine can induce micro fractures in organic shales. The induced micro fractures can increase the shale permeability. In some samples, the shales were likely to be disintegrated under imbibition without confining pressure (Dehghanpour et al., 2013; King, 2012). Oil recovery conducted by counter-current and co-current spontaneous imbibition was significantly related to sample shape, size (cylindrical cores) and boundary conditions. When assessing the oil recovery capacity on reservoir rock samples by spontaneous imbibition, experiments should be performed under both co-current and counter-current flow conditions as the latter may forecast too low rates and ultimate recoveries under certain conditions (Standnes, 2004). Counter-current imbibition was researched when brine displaced oil spontaneously from a strongly water-wet rock. Piston-like advance of fronts were assumed from the radial outer face and both flat ends. This separated the flow pattern into two types: linear imbibition into cones and radial imbibition into the surrounding toroid ring (Mason et al., 2009). In this paper, we researched the auto-removal mechanism of shale reservoir by spontaneous imbibition experiments combined with nuclear magnetic resonance. The main innovative point of this paper is that through these experiments, we learned how the fluid entered into the rocks in the process of co-current and counter-current displacing gas, in other word, the mall pores or the bigger pores for the fluid firstly entering.

The characteristics of shale formation had strong heterogeneity (Josh et al., 2012; Sakhaee-Pour and Bryant, 2015; Wang et al., 2015; Liang et al., 2014; Hao et al., 2013; Chalmers et al., 2012), so the properties of shale formations changed greatly from different reservoirs. Due to the different properties of shale samples in different regions and the fracturing fluid consisting of different constituents, these factors may significantly influence the interaction between the fluid and shale rocks. However, the fluid used in this paper is distilled water. So the results of this paper are adaptable to the rocks and liquid used in this study. A lot of work concerning the different fracturing fluids and different shale rocks from various regions will be performed in the future research.

## 2. Samples and experimental methods

### *2.1. Samples*

Shale samples are selected from the Lower Silurian Longmaxi formation in southeast of Chongqing, China; volcanic rock sample is taken from the Lower Cretaceous Yingcheng formation in Yingtai of Jilin, China; sandstone sample is chosen from the Lower Cretaceous Denglouku formation in Changhuan of Daqing, China. Sandstone and volcanic rock do as control samples. The samples were dried under the 65 °C before the experiment was performed, until the mass remains unchanged. The characteristic parameters of the Samples can be found in Table 1. Samples porosity were measured by helium pycometer (KXD - III type). Samples pulse-decay permeability was determined by the ultra-low permeability measurement instrument (YRD - CP200 type). Confining pressure was exerted by water, and pore pressure was exerted by helium. Testing condition: temperature was 25 °C; confining pressure was 8 MPa; pore pressure was 5 MPa. The porosity of sandstone and volcanic rock are close, but the permeability of sandstone is far larger than that of volcanic rock, which may be due to the good connectivity of sandstone pores. The porosity of shale Y1 is less than that of shale Y2. However, the permeability of shale Y1 is much larger than that of shale Y2, which may be account for shale Y1 full of connected micro fractures.

Bulk mineralogical compositions and total organic carbon (TOC) of two shale samples are listed in Table 2. Bulk mineralogical compositions were derived from the X-ray diffraction patterns which were measured by D/max 2500 diffractometer. TOC analysis on powdered samples was performed by Leco CS-230 carbon analyzer. Both these two experiments were carried out at State Key Laboratory of Petroleum Resources and Prospecting in China University of Petroleum, Beijing. From Table 2, we can find that the total clays content and TOC content of the two shale samples are similar, which may be due to the near location of these two samples. These two samples were made by crushing the two cylindrical samples which were used in the spontaneous imbibition experiments.

### *2.2. Nuclear magnetic resonance technique*

Low field nuclear magnetic resonance (NMR) is a non-destructive technique and has been widely used in the exploration and research of oil/gas field. NMR method involves the motion of the protons (Hydrogen $^1H$) occurring in water and hydrocarbon fluids relative to the porous rock. In shale formation, the difference in magnetic interaction between the fluids saturating the pore space, the solid matrix and also the whole sample was measured through relaxation time determinations of proton (Coates et al., 1999). The NMR technique was an efficient method of acquiring information such as total porosity, clay bound water, capillary and free porosity, pore size distribution and derived permeability (Rezaee et al., 2012). Washburn and Birdwell (2014) provided a model to distinguish the different constituents of shale by application of binomial-edited CPMG. Xu et al. (2015) applied NMR to research the porosity of shale, and found that echo time had a great influence on the accurate measurement of shale porosity.

When the pore space of a sample is saturated with 100% fluid, total porosity includes the pore space occupied by clay bound fluid, capillary bound fluid and movable fluid. If the times between echoes, TE (the time between two sequential 180-degree in the CPMG sequence) larger than 0.3 ms, the information from small pores will be partially lost, so the total porosity is not accurate (Mirotchnik et al., 2001; Wang et al., 2007). Fluid in large pores has longer T2 value because more nuclei are available to exhibit the NMR effect, and fluid in small pores has short T2 value. T2 relaxation time is in inverse proportion to specific surface of samples (Appel, 2004).

In this paper, the low-field NMR apparatus (Fig. 1) was produced by Shanghai Niumag Corporation. The main magnetic field were 23 MHZ frequency; signal superposition times NS were 64; waiting time were 3000 ms; echo spacing TE were 0.116 ms.

### *2.3. Experiment*

#### *2.3.1. Spontaneous imbibition in co-current displacing gas*

Monitor the process of shale spontaneous imbibition combined with the low-field NMR method and analytic balance. The purpose of this experiment focused on simulating the liquid displacing shale