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# Research on the auto-removal mechanism of shale aqueous phase trapping using low field nuclear magnetic resonance technique

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## ABSTRACT

Hydraulic fracturing has become a necessary practice in order to obtain economical gas flow rates from gas shale. During and immediately following the creation of a fracture, high injection pressures cause fracturing fluid to leak off into the adjacent matrix, which leads to aqueous phase trapping (APT). However, after the well shut-in for a period of time, the APT can be auto-removed. The experiments of spontaneous imbibition and APT auto-removal combined with the low-field nuclear magnetic resonance (NMR) were performed to investigate the mechanism of the APT auto-removal of shale reservoir. Results show that new pores were not produced in the process of spontaneous imbibition in the tight sandstones and volcanic rocks, and with increasing spontaneous imbibition time, the fluid mass increased uniformly in the whole range of the pores. However, new pores were produced in shale samples in the process of spontaneous imbibition and through observing the process of experiments, the microcracks on the sample surface increased gradually, so we can make sure that new pores are mainly for microcracks. After sandstone had stopped soaking water, the liquid in the pore network wholly remained in the same place, while volcanic rock had certain flowing behavior. However there was obvious movement of fluid in shale sample, due to the tremendous imbibition force caused by ultra-micropores in shale. Therefore, after large-scale hydraulic fracturing of shale reservoir, under the effect of capillary force and clay swelling, on one hand, the retention fluid migrated along the initial microfractures to produce new pores, as can be proved that the new pores is new microcracks, so the gas flowing channel is enlarged by the new microcracks; on the other hand, under the influence of the capillary force, the fluid in the largish pores moved to the ultra-micropores, which apparently increased the area of the main gas flowing channel. The action of the two aspects contributes to the auto-removal mechanism of the shale reservoir APT.

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## 1. Introduction

Hydraulic fracturing has become an indispensable practice in order to attain commercial gas flow rates from low permeability reservoirs. During and immediately following the creation of a fracture, high injection pressures lead to fracturing fluid leaking off into the adjacent matrix. Thus aqueous phase trapping (APT) appears. However, after the well was shut in for a period of time, the APT can be auto-removed, which aroused public attention (Guo et al., 2014; Jia et al., 2013; Zhou et al., 2014). APT was one of the severe damages of formation, and sometimes it posed greatly effect on the productivity reduction. APT occurred during drilling, completion, work over, and stimulation process (Bennion et al., 1996).

In view of the conventional APT, attentions were always focused on the damage caused by APT, so some measures have been taken to reduce the severity of the damage or avoid the occurrence of damage by giving up using water-based fracturing fluid (Bahrami et al., 2012; Elkewidy, 2013; Ahmed Lashari et al., 2013). However, shale reservoir had the characteristic of strong heterogeneity, and pore networks in the shale reservoir were very complex (Clarkson et al., 2013; Bai et al., 2013; Guo et al., 2015; Sakhaee-Pour and Bryant, 2015; Josh et al., 2012; Chalmers et al., 2012; Hao et al., 2013). In view of the research of shale reservoir APT, relevant experts always made use of the spontaneous imbibition experiment to indirect research the shale reservoir APT. Fluid added with wettability altering surfactants can reduce the fluid loss which was caused by spontaneous imbibition, at the same time, due to the multi-porosity, the slope of water content as a function of square root of time presented different value (Roychaudhuri et al., 2013). The residual oil saturation decreased greatly by exposing the rock with oil into the higher PH bine,

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which showed that water-wet system was better for spontaneous imbibition (Takahashi and Kovscek, 2010). Low PH solutions similar to distilled water were helpful to accelerate recovery by spontaneous imbibition in Barnett samples because higher PH solutions were easy to cause alkaline concentration to damage samples and lots of cracks appeared on the surface of the shale samples after soaking liquid (Morsy and Sheng, 2014). Low HCl concentration or NaOH showed poor imbibition in Marcellus shale samples and wettability material had great influence on contact angles (Morsy et al., 2014). Both brine and oil can imbibe spontaneously into the large pores, but only oil can enter into the most of small pores as these pores were oil-wet (Akbarabadi and Piri, 2014). Imbibition rate was higher in the direction parallel to the bedding plane than in the direction perpendicular to the bedding in horn shale, and water in the fracture can imbibe into the matrix (Makhanov et al., 2012). A new anionic surfactant fracturing fluid with easy preparation, good viscosity, low frictional resistance was produced to apply to hydraulic fracturing for reducing liquid loss (Khair et al., 2011). However, there were little researches related to the shale APT auto-removal mechanism. The state-of-the-art of shale APT was that Hayatdavoudi et al. (2015) used distilled water as fracturing fluid to soak the Pierre shale. They measured the change of pH, Eh, and temperature and at the same time, used a video system to record the process of gas bubble flow under microscope. Based on the test, Power Spectrum was constructed by using Fourier Transform of the data for extracting the hidden information in the data which was related to the release of the first bubble of gas from the shale mass. The release of the first bubble was the result of diffusion of water in shale capillaries where it took a certain amount of driving energy by water molecules to displace the sorbed gas from the shale macro, meso, and micro-capillaries. According to these researches, a simple and cost effective methodology was proposed by them to determine the postfrac optimal shut-in time. The state-of-the-art of tight gas reservoirs ATP (except gas shales) remained in the damage caused by ATP, and related formulas were established to forecast the severity of ATP (You and Kang, 2009). Low field nuclear magnetic resonance (NMR) method has been widely used in the field of petroleum, because it had the advantage of fast detection, no damage to the sample and no geometry requirement to the sample for test (Yao and Liu, 2012; Hodgkins and Howard, 1999; Li et al., 2013; Xu et al., 2015; Oduşina and Sigal, 2011; Dada et al., 2015; Al-Mahrooqi et al., 2006). In this paper, in order to investigate the shale reservoir APT auto-removal characteristics, the experiment of researching the shale APT auto-removal mechanism has been carried out based on the low-field NMR method.

The most innovation of our paper is that we found that the microcracks produced in the process of spontaneous imbibition and ultra-micropores absorbing liquid after well was shut in are two important factors for shale APT auto-removal.

## 2. Samples and experimental methods

### 2.1. Samples

Shale samples were selected from the Lower Silurian Longmaxi formation in southeast of Chongqing, China; Volcanic rock samples were taken from the Lower Cretaceous Yingcheng formation in Yingtai of Jilin, China; Sandstone samples were chosen from the Lower Cretaceous Dengloulou formation in Changhuan of Daqing, China. The samples were dried under the 65 °C before the experiment, until the mass remained unchanged. The physical parameters of the samples can be found in Table 1. Samples porosity was measured by helium porosimeter (KXD-III type) presented in Fig. 1 which was made by Jiangsu Hua'an Scientific

**Table 1**  
Basic property of rocks.

Sample	Diameter (cm)	Length (cm)	Porosity (%)	Pulse-decay permeability (mD)
Sandstone S1	2.52	2.85	7.72	0.0048
Sandstone S2	2.51	1.91	12.26	0.063
Sandstone S3	2.51	2.32	13.41	0.031
Volcanic rock L1	2.49	2.02	6.58	0.0022
Volcanic rock L2	2.48	2.38	6.37	0.00099
Volcanic rock L3	2.49	2.03	7.45	0.0029
Shale Y1	2.46	1.00	6.47	0.00065
Shale Y2	2.47	1.28	4.28	0.045
Shale Y3	2.47	1.10	4.35	0.024
Shale Y4	2.47	1.21	5.01	0.0015

Annotate: Pulse-decay permeability for dry rock pulse-decay permeability.

Instrument Co., Ltd. in China. The experiment temperature and pressure were 25 °C degree and 200 psi, respectively. The porosity of volcanic rock is close to the shale, but porosity of sandstone is larger than the two types. Samples pulse-decay permeability was determined by the ultra-low permeability measurement instrument (YRD-CP200 type) shown in Fig. 1, which was made by Beijing Yongruidai Technology Co., Ltd. in China, confining pressure exerted by water, pore pressure exerted by helium. Its testing condition: temperature was 25 °C, confining pressure was 8 MPa and pore pressure was 5 MPa. Shale rocks porosity is close, but pulse-decay permeability is enormously different. This may be due to the effect of microcracks in shale, when the microfractures connect to each other, high permeability appearing. Samples used in the spontaneous imbibition experiment were sandstone S1, volcanic rock L1, shale Y1 and shale Y2; samples used in APT auto-removal experiment were sandstone S2, sandstone S3, volcanic S2, volcanic S3, shale Y3 and shale Y4.

The mineralogy content and TOC content of two shale samples are listed in the Table 2. The mineralogy content was measured by D/MAX 2500X X-ray diffractometer, at 20 °C and 40% relative humidity, according to the testing standard of SY/T5163-2010 (Sedimentary clay minerals and common non clay minerals X-ray diffraction analysis method). The TOC was measured by LECO CS230 carbon sulfur analyzer made in America, at analysis service of 17 °C and 35% relative humidity, according to the testing standard of GB/T19145-2003 (the determination of total organic carbon in sedimentary rocks), and the testing condition were carrier gas of 0.27 MPa, oxygen purity of 99.5%, combustion gas velocity of 2 L/min and analysis gas flow velocity of 0.5 L/min. In Table 2, the four shale samples were made by crushing the four cylindrical samples which were used in the spontaneous imbibition experiment. The minerals of the shale samples are characterized by a high proportion of illite (22–28%) and quartz (36–55%). The total clays range from 1.7 wt% to 4.9 wt%.

### 2.2. Nuclear magnetic resonance (NMR) method

Nuclear magnetic resonance (NMR) namely is the interaction between the nucleus and the magnetic field. Nuclear magnetic resonance (NMR) method has been widely used in oil/gas field. The principle applied in the research of oil/gas is based on that the fluid in the macropore has longer NMR T2 transverse relaxation time, and NMR T2 relaxation time of small pore is short, and the higher the signal amplitude, the more the fluid. According to this principle, the pore size of the sample can be differentiated, and the fluid content in a certain pore can be reflected in the NMR T2 spectrum. From the NMR T2 spectrum, a lot of parameters of the

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