



Effects of slick water fracturing fluid on pore structure and adsorption characteristics of shale reservoir rocks



Zepeng Sun^{a,c}, Hailong Zhang^d, Zhifu Wei^{a,*}, Yongli Wang^{a,b,**}, Baoxiang Wu^a, Shengguang Zhuo^e, Zhe Zhao^e, Jing Li^a, Lewei Hao^a, Hui Yang^a

^a Key Laboratory of Petroleum Resources, Gansu Province/Key Laboratory of Petroleum Resources Research, Institute of Geology and Geophysics, Chinese Academy of Sciences, Lanzhou 730000, PR China

^b Key Laboratory of Cenozoic Geology and Environment, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, PR China

^c University of Chinese Academy of Sciences, Beijing 100049, PR China

^d Downhole Operation Branch Daqing Oilfield, Daqing 163000, PR China

^e Department of Environmental Science and Technology, Northeastern University at Qinhuangdao, Qinhuangdao 066004, PR China

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ABSTRACT

The shale-fracturing fluid interaction and its effects on the pore structures and adsorption characteristics of shale are the key factors affecting shale gas exploration. To address this problem, the black shale samples obtained from the Lower Silurian Longmaxi Formation in Sichuan Basin, China were exposed to slick water fracturing fluid at the simulation conditions of 100 °C and 50 MPa for 72 h through a fluid-rock interaction simulation instrument. The slick water fracturing fluid contained 0.2 wt.% friction reducer, 1 wt.% clay control agent, 0.15 wt.% cleanup agent and 0.05 wt.% demulsifier. The mineral composition, pore structure and methane adsorption capacity of shale samples before and after slick water fracturing fluid treatment were measured by X-ray diffraction (XRD), field emission scanning electron microscope (FE-SEM), low-pressure nitrogen adsorption and methane isothermal adsorption experiments using the gravimetric method. The results showed that the carbonate minerals were dissolved during treatment, and as a result, the samples developed many dissolution pores measuring 2–5 μm in diameter, while the other minerals remained relatively undisturbed. The specific surface area and total pore volume of shale sample were reduced after the reaction, and the shale-fracturing fluid interaction exhibited a stronger influence on the mesopores. However, the average pore diameter of nanopore was enlarged after the reaction, increasing from 4.29 nm to 4.78 nm. The changes of fractal dimensions suggested an increase in the roughness of pore surfaces, and the pore structure became more regular. The methane adsorption capacity in shale treated with fracturing fluid was reduced from 1.23 mmol/g to 0.95 mmol/g. The changes in the pore structure and adsorption characteristics of shale could affect the gas flow and gas adsorption capacity. These results indicated that the slick water fracturing fluid may play an important role in shale matrix stimulation.

1. Introduction

Shale gas is an important source of natural-gas that is present in low porosity and low permeability organic-rich shale formations (Chen et al., 2016; Estrada and Bhamidimarri, 2016). To optimize the production, the horizontally drilled shale reservoirs are stimulated by hydraulic fracturing or fracking (Vengosh et al., 2014; Wang et al., 2016a). The fracturing fluid is injected at high pressure into the reservoir to create fractures in the shale. Moreover, the water based

fracturing fluids, especially the slick water are injected at high pressure to create fractures. This method has been widely used for shale reservoirs due to the advantages of high efficiency and low cost (Palisch et al., 2008; Al-Muntasheri, 2014; Du et al., 2014). Based on these advanced technologies, the gas production in the United States has significantly increased (Gale et al., 2007). In addition, extensive exploration and development of shale gas have been conducted in China, and remarkable success has been achieved with marine shale of the Upper Ordovician Wufeng Formation-Lower Silurian Longmaxi

* Corresponding author.

** Corresponding author. Key Laboratory of Petroleum Resources Research, Institute of Geology and Geophysics, Chinese Academy of Sciences, 382 Donggang West Road, Lanzhou 730000, PR China.

E-mail addresses: sunzpeng13@mails.ucas.ac.cn (Z. Sun), jx_zhanghl@petrochina.com.cn (H. Zhang), weizf@lzb.ac.cn (Z. Wei), wyll6800@lzb.ac.cn (Y. Wang), bxwu@foxmail.com (B. Wu), zoe200200@163.com (S. Zhuo), zhaozhe1980@163.com (Z. Zhao), Lj1926@lzb.ac.cn (J. Li), haolewei166@163.com (L. Hao), hyang@lzb.ac.cn (H. Yang).

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Table 1
Geochemical data and mineral composition of the shale sample.

Sample	TOC (wt.%)	Ro (%)	$\delta^{13}\text{C}_{\text{org}}$ (‰)	Mineral composition (%)					
				Quartz	Calcite	Dolomite	Pyrite	Plagioclase	Clay minerals
Longmaxi	3.61	2.3	−29.6	36	36	7	2	4	16

Formation in the Sichuan Basin (Dai et al., 2016).

Due to the increasing demands for unconventional resource exploration, the horizontal well drilling and hydraulic fracturing have increased in recent years. However, certain additives present in the fracturing fluid may enter the fractures and pores and thereby change the pore structures and gas transport in rocks (Lane and Aderibigbe, 2013; Pedlow and Sharma, 2014; Dieterich et al., 2016; Xu et al., 2016). Thus, it is a paramount to understand the chemical and physical reactions that occur between fracturing fluid and the fracture surfaces of the shale reservoirs and to develop an understanding of the changes within the formation and the long-term productivity of the fractured shale (Dieterich et al., 2016; Ali and Hascakir, 2017; Marcon et al., 2017).

A large amount of water is required during hydraulic fracturing, and several studies have focused on the influence of water on the physical properties of shale. Yuan et al. (2014a) studied the shale's wettability and water adsorption capacity and observed that swelling strain had a linear relationship with the water content. Zhang et al. (2016) reported that the clay content in shale rocks is the fundamental cause for the significant reduction of shale fracture conductivity after water flow. However, the chemical interactions between shale and fracturing fluid have not been thoroughly elucidated (Lane and Aderibigbe, 2013).

Black shales constitute many components: in addition to organic matter, it also contains clay minerals, carbonate and brittle minerals (Jarvie et al., 2007). The interactions between these minerals and fracturing fluid would affect the pore structure and permeability of the shale. Sanaei et al. (2016) reported that the composition and content of clay minerals determined the clay swelling and the extent of double diffusive layer expansion, and they investigated the production loss owing to clay swelling in a realistic complex hydraulic fracture network. Wu and Sharma (2017) observed that the acid fracturing could change the structures of micropores in the carbonate rich shales. Chen et al. (2017b) also indicated that the impact of oxidative reagents on composition and pore structure could improve the matrix diffusivity of shale. Previous studies have shown that organic matter and clay minerals could also affect the gas adsorption capacity of shale (Ross and Marc Bustin, 2009; Weniger et al., 2010; Ji et al., 2012; Zhang et al., 2012; Yuan et al., 2014b; Chen et al., 2017a). Furthermore, the effect of moisture on the methane adsorption capacity of shale has been studied extensively (Gensterblum et al., 2014; Li et al., 2016b; Liang et al., 2016; Wang and Yu, 2016; Zhao et al., 2017), and the methane adsorption capacity may be reduced by capillary water blocking the pore space (Li et al., 2016b). However, the effects of fracturing fluid on the methane adsorption and diffusion in shale are not clear. Knowledge of the influence of fracturing fluids on pore structures and adsorption characteristics of shale gas reservoirs could improve the performance of hydraulic fracturing.

This work used the slick water fracturing fluid that is commonly used in commercial shale gas development, and high temperature and high pressure experiments were conducted on the marine shale samples of China. Applying a series of analytical methods, the mineral composition, pore structure and the methane adsorption characteristics of shale were characterized for shale samples before and after slick water fracturing fluid treatment. The primary objective of this study is to investigate the influence of slick water fracturing fluid on the pore structure and adsorption characteristics of shale in order to gain a greater understanding of shale-fracturing fluid interactions. The results of this study may improve the understanding of the mechanisms of

shale formation damage and optimize the use of slick water fracturing fluid for hydraulic fracturing in shale reservoirs.

2. Materials and methods

2.1. Sample preparation

The shale samples were obtained from the outcrops of the Lower Silurian Longmaxi Formation in Changning County located in the Sichuan Basin of China. Before the sampling, the disintegrated and weathered rocks on the shale surface were removed, and the unweathered shales were collected by digging 3 m into fresh exposure of the Longmaxi Formation. The Longmaxi Shale in this field is the most important target zone for shale resources exploration and development (Dai et al., 2014). The geochemical data and mineral composition of the shale sample used in this experiment are shown in Table 1. The values of TOC and R_o in the research sample are 3.61 wt.% and 2.13%, respectively, which indicates that the formation is an organic-rich and over-mature shale. The $\delta^{13}\text{C}_{\text{org}}$ value of the shale sample is -29.6‰ , which is consistent with the results of Wang et al. (2015), and the unusually low $\delta^{13}\text{C}_{\text{org}}$ values of Longmaxi Formation shale can be explained by extremely anoxic oceanic environment probably caused by rising sea levels (Wang et al., 2015). The primary mineral constituents are quartz (36%), calcite (36%) and clay minerals (16%). Clay minerals of the shale mainly consist of a mixed layer of 97% illite/smectite and a small amount of chlorite.

After sample acquisition, the block sample was drilled into cylindrical samples with a diameter of 2.5 cm and small cubes with a volume of 1 cm^3 to 2 cm^3 for the purpose of high pressure and temperature fracturing fluid-rock interaction experiments. The small shale cubes were used for comparing the argon ion milled-field emission scanning electron microscope (FE-SEM) images of shale before and after reaction. After capturing the “before” images, the surface of the specimens were cleaned using clean water and kept in an oven at 50 °C for 12 h.

2.2. Fracturing fluid

In this study, a type of slick water fracturing fluid was used with pH of 5.37, wetting contact angle of 60.11° and surface tension of 29.31 mN/m. The fluid contained 0.2 wt.% friction reducer, 1 wt.% clay control agent, 0.15 wt.% cleanup agent (fluorocarbon surfactants) and 0.05 wt.% demulsifier (SP). The friction reducer is a water-soluble terpolymer polymerized of acrylamide (AM)/acrylic acid (AA)/2-acrylamido-2-methyl-propane sulfonic acid (AMPS). The infrared spectrum of the friction reducer is shown in Fig. 1. The broad band at around 3120 cm^{-1} can be ascribed to the stretching vibrations of N-H bands of AM. There are three bands at 1660, 1600 and 1400 cm^{-1} originated from the $-\text{COO}^-$, $-\text{C}=\text{C}-$ and $-\text{CH}_2$ stretching vibration of AA, respectively. The peaks for $\text{S}=\text{O}$ at 1190 cm^{-1} and for $-\text{S}-\text{O}$ at 1110 cm^{-1} are characteristic adsorption peaks of AMPS. These characteristic peaks indicated that the friction reducer contains the units of AM, AA and AMPS.

2.3. Apparatus and procedure

The experiments were conducted in a high-temperature and high-pressure fluid-rock interaction simulation instrument (Fig. 2). The main

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