# Controls of Acid-sensitive Minerals on Reservoir Sensitivity Testing: An Example from the Silurian Kepingtage Formation in Tazhong Area, Tarim Basin

HAN Denglin<sup>1, 2</sup>, WANG Qianqian<sup>2</sup>, WANG Chenchen<sup>3, \*</sup>, OUYANG Chuanxiang<sup>4</sup> and YUAN Wenfang<sup>5</sup>

- 1 Key Laboratory of Exploration Technologies for Oil and Gas Resources of the Ministry of Education, Yangtze University, Wuhan 430100, China
- 2 School of Geosciences, Yangtze University, Wuhan 430100, China
- 3 Hubei Cooperative Innovation Center of Unconventional Oil and Gas, Wuhan 430100, China

4 School of Petroleum Engineering, Yangtze University, Wuhan 430100, China

5 Research Institute of Exploration & Development, PetroChina Tarim Oilfield Company, Korla 841000, China

Abstract: The Silurian Kepingtage Formation in Tazhong area is regarded as an acid-sensitive hydrocarbon reservoir. However, formation mechanism of acid-sensitive of the reservoir cannot be interpreted by the existing acid-sensitive evaluation criterion based on damage rate. The contents of acid-sensitive minerals illustrated by bulk-rock XRD, scanning electron microscopy and clay mineral composition analysis exert the dominant control on acid-sensitive flow testing of the reservoir. The iron-bearing minerals (including pyrite cements and chlorite cements) mainly deteriorate reservoir quality, while the iron-free minerals (including calcite cements and dolomite cements) mainly improve permeability. The permeability variation of the tested samples is controlled by the relative content of two acid-sensitive minerals. On the basis of newly established sensitivity mechanism and its influence on permeability, the corresponding ion (Fe<sup>2+</sup>) stabilizer was added to the acidizing fluids during the acidification reconstruction, which inhibited the negative factors of acid-sensitive minerals and improved the target layer quality effectively.

Key words: clay mineral, acidic sensitivity effect, Kepingtage Formation, Tazhong area

# **1** Introduction

With the refinement of exploration and production of oil and gas fields, the sensitive damage of reservoir is evaluated as one of the important factors influencing development effect, thus a lot of studies have been carried on the formation mechanism, distribution out characteristics and protective measures of reservoir sensitivity (Krueger, 1986; Zhang et al., 1996; Menouar et al., 2000; Civan, 2007; Alotaibi et al., 2009; Tian et al., 2015), with main focus on the evaluation and prediction of sensitivity effect and type, and its algorithm (Zhao Xingyuan, 1992; Peng Chunyao et al., 1999; Sun Jianmeng et al., 1999; Kang et al., 2000; Menouar et al., 2000; Zhao Dahua et al., 2002; Li Yonglin et al., 2003; Yin Xin, 2005; Zhao Xingyuan et al., 2005; Wang et al., 2006; Zhang Guanlong et al., 2006; Civan, 2007; Yuan Wenfang et al., 2014; Ma et al., 2016; Zhao Xiaohui et al., 2016). Several qualitative or semi quantitative prediction methods for the sensitivity of acid-sensitive minerals in the reservoir have been established gradually (Min et al., 2004; Zhang et al., 2007; Bahrami et al., 2011; Watheq et al., 2018). However, the current evaluation criterion of reservoir sensitivity is based on the damage rate of permeability in testing samples (absolute value of change rate of permeability) (Moghadasi et al., 2010; Habibi et al., 2012; Dou Honggen et al., 2016; Li et al., 2017), which masks the sensitization mechanism caused by different sensitivity effect, and could not benefit in restraining sensitivity effect in the reservoir (Yu et al., 2014; Dou et al., 2015; Wang et al., 2018).

<sup>\*</sup> Corresponding author. E-mail: wcc1220@163.com

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The Silurian Kepingtage Formation in Tazhong area, Tarim Basin, is regarded as a typical marine clastic reservoir and widely concerned as main exploration target (Lv Xiuxiang et al, 2008; Zhang et al, 2008; Pang et al, 2012; Li et al, 2015). Previous studies suggested that it has an obvious acid-sensitive effect. The main minerals caused acid-sensitive effect include carbonate cements, pyrite cements and chlorite cements, but any acidification measurement is not effective enough (Zhang et al, 2008). Based on the characteristics, contents and sensitization mechanism of acid-sensitive minerals, we carried out the acid-sensitive flow testing of the corresponding samples (change of permeability), and discussed the impact of mineral type and content on sensitivity testing to understand mechanism of acid-sensitive reservoir and its protection.

# **2** Geological Settings

Tazhong area is located in the middle part of central uplift belt in the Tarim Basin with oil and gas mainly distributing in the northern slope of Tazhong area, including the Tazhong 11 block, Tazhong 12 block, Tazhong 16 and Tazhong 35 block (Fig. 1).

The lithology of the Kepingtage Formation is composed of sandstone and mudstone which deposited in the offshore shelf and tidal flat. The Kepingtage Formation can be divided into two members, i.e., the Upper member and the Lower member. The Upper member can be further divided into the third sub-member, the second submember and the first sub-member from bottom to top. Current study focuses on the third sub-member, which is characterized by siltstone and fine sandstone and is the main oil producing interval in Tazhong area.

The framework grains in the third sub-member are litharenite to feldspathic litharenite quartzose sandstone with predominance of fine grains (Fig. 2a). The sandstones are moderately sorted with sub-round to round grain shapes. The grain contacts are dominantly point to line shaped. The main detrital compositions are quartz (mainly monocrystalline vs polycrystalline), feldspar and rock fragment with minor amount of clays and mica (0–17.8%). Volcanic and metamorphic fragments are generally ubiquitous in the sandstones, which are more abundant than sedimentary rock fragments (Fig. 2b).

### **3** Samples and Methods

Samples from well Tazhong 161, well Tazhong 31, well Tazhong 111 and well Tazhong 122 are distributed in different blocks (Fig. 1). Representative samples from the third sub-member of the Upper member are composed of fine sandstones with different petrological composition and no obvious microfracture to avoid influence of physical properties during acid-sensitive flow testing. During the sampling process, core plug with diameter of 2.54 cm were drilled along diameter direction. The middle of the core plug was cut out in 4 cm long for acid-sensitive flow testing. The remaining parts were used for scanning electron microscope observation (SEM) and X-ray diffraction test (XRD, the National Industry Standard is SY/T 5163-2010) for the composition and content of bulkrock and clay minerals respectively (Fig. 3). Eight well sorted fine sandstone samples were chosen for XRD



Fig. 1. Tectonic units of the Tazhong uplift.



Fig. 2. Detrital composition in the Silurian Kepingtage Formation in Tazhong area.

Q, Monocrystalline and polycrystalline quartz; F, plagioclase and K-feldspar; Ls, sedimentary lithic fragments; L $\nu$ , volcanic lithic fragments; Lm, metamorphic lithic fragments; L, including Ls, L $\nu$  and Lm.



Fig. 3. The diagram of sample separation for analysis.

analysis. The composition and content of bulk-rock minerals and clay minerals were measured by D/Max-RC diffractometer (Table 1). The result of XRD analysis

shows that the content of acid-sensitive minerals differs apparently (Table 2). Four representative samples were selected for scanning electron microscope observation with plat gold on sample surfaces (the National Standard is GB/T 18295-2001) and the acid-sensitive flow testing (the National Industry Standard is 5358-2010 SY/T).

### 4 Characteristics of Acid-sensitive Minerals

In acidic fluid medium, acid-sensitive minerals in the reservoir would react with fluids (fluid-rock interaction). Some materials would be dissolved while other materials would be precipitated, resulting in change of reservoir permeability, which is called acid sensitivity effect (Zhang Shaohuai et al., 1996; Chang et al., 1997; Civan, 2007). The dissolution of feldspar and volcanic rock fragment was not strong in 15% hydrochloric acid and during 48-60 hours, according to the existing acid-sensitive evaluation criterion. And no obvious dissolution in the feldspar and volcanic rock fragments were found under the microscope (Figs. 4a-4b). The acid-sensitive minerals are not the grains, but the cements, such as carbonate cements. According to the criterion, the test result is the absolute value of permeability change, which could be used to characterize the strength of acid sensitivity of sample, regardless of permeability changes (increase and decrease). But the mechanism of acid-sensitive effect reflected by these two changes is substantially different.

### 4.1 Iron-free acid-sensitive minerals

The iron-free acid-sensitive minerals mainly refer to nonferrous carbonate minerals, including calcite cements

Table 1 The content of bulk rock of different samples in the Kepingtage Formation before acid sensitivity testing

Sample number	Well number	Depth	Stratum	Clay (%)	Quartz (%)	K-Feldspar (%)	Plagioclase (%)	Calcite (%)	Dolomite (%)	Pyrite (%)	Anhydrite (%)	Illite/Smectite Mixed-Layer (%)	Illite (%)	Kaolinite (%)	Chlorite (%)
2-1	Tazhong 161	4094.35	$S_1k_u^{-1}$	17.8	57.1	0.9	9.5	4.0	0.0	8.6	2.1	7.0	73.0	20.0	0.0
2-2	Tazhong 161	4224.60	$S_1 k_u^{3}$	19.5	58.2	0.0	10.0	5.6	0.0	5.0	1.7	0.0	22.0	78.0	0.0
2-3	Tazhong 31	4603.44	$S_1 k_u^{3}$	6.1	75.4	3.1	6.7	5.3	0.7	2.7	0.0	22.0	31.0	35.0	12.0
2-4	Tazhong 31	4600.49	$S_1 k_u^{3}$	4.6	78.9	0.5	6.4	7.3	1.6	0.7	0.0	15.0	47.0	22.0	16.0
2-5	Tazhong 111	4470.00	$S_1 k_u^{3}$	7.0	83.0	4.0	5.0	0.0	0.0	1.0	0.0	17.0	7.0	69.0	7.0
2-6	Tazhong 111	4474.30	$S_1 k_u^3$	4.0	84.0	2.0	4.0	0.0	2.0	4.0	0.0	15.0	12.0	65.0	8.0
2-7	Tazhong 122	4345.80	$S_1 k_u^{3}$	4.0	88.0	2.0	4.0	1.0	0.0	1.0	0.0	13.0	16.0	66.0	5.0
2-8	Tazhong 122	4342.32	$S_1 k_u^{3}$	12.0	72.0	6.0	10.0	0.0	0.0	0.0	0.0	3.0	44.0	44.0	9.0

Note:  $S_1 k_u^1$  is the first sub-member of upper Kepingtage member,  $S_1 k_u^3$  is the third sub-member of upper Kepingtage member.

Table 2 The content of acid-sensitive minerals and damage rate of acidic sensitivity of different samples in the Kepingtage Formation

Sample	Wall number	Denth	Stratum	Initial Permeability	Content of aci	Damage rate caused by acid			
number		Depui	Stratum	(mD)	Carbonate minerals	Pyrite minerals	Chlorite minerals	sensitivity (%)	
2-1	Tazhong 161	4094.35	$S_1k_u^{-1}$	0.210	4.0	8.6	0.0	22.6	
2-2	Tazhong 161	4224.60	$S_1k_u^3$	—	5.6	5.0	0.0	—	
2-3	Tazhong 31	4603.44	$S_1k_u^3$	0.765	6.0	2.7	0.7	41.0	
2-4	Tazhong 31	4600.49	$S_1k_u^3$	—	8.9	0.7	0.7	—	
2-5	Tazhong 111	4470.00	$S_1 k_u^3$	0.175	0.0	1.0	0.5	61.5	
2-6	Tazhong 111	4474.30	$S_1 k_u^3$	—	2.0	4.0	0.3	—	
2-7	Tazhong 122	4345.80	$S_1 k_u^{3}$	0.868	1.0	1.0	0.2	34.0	
2-8	Tazhong 122	4342.32	$S_1 k_u^3$	_	0.0	0.0	1.1	—	



Fig. 4. Microscopic characteristic of the acid-sensitive minerals in the Kepingtage Formation. (a), calcite cements (C, Tazhong122, 4345.80m,  $S_1k_u^3$ ); (b), dolomite cements (D, Tazhong31, 4603.44 m,  $S_1k_u^3$ ); (c), pyrite cements (Py, Tazhong161, 4094.35m,  $S_1k_u^3$ ), (d), chlorite cements and pyrite cements (Ch and Py, Tazhong111, 4470.00m,  $S_1k_u^3$ ), (e), chlorite cements (Ch, Tazhong31, 4603.44m,  $S_1k_u^3$ ), (f), chlorite cements (Ch, Tazhong31, 4603.44m,  $S_1k_u^3$ ), (f), chlorite cements (Ch, Tazhong31, 4603.44m,  $S_1k_u^3$ ). Kao: kaolinite; Kf: K-feldspar; Q: quartz.

(CaCO<sub>3</sub>) and dolomite cements [CaMg(CO<sub>3</sub>)<sub>2</sub>] filling in pores (Figs. 4a–4b). Different content of nonferrous carbonate minerals in rocks from different wells varies significant (Table 2). The higher values are distributed in samples from well Tazhong 161 and well Tazhong 31. The carbonate minerals are unstable and therefore easy to be dissolved during the acid sensitivity testing process (Aguilera, 2004; Wang Jian et al., 2017; Guo Biao et al., 2018). During the dissolution, new pores and throats in the test sample would be generated, resulting in permeability increment (Zhu Dongya et al., 2017).

#### 4.2 Iron-bearing acid-sensitive minerals

Iron-bearing acid-sensitive minerals mainly refer to minerals consisting  $Fe^{2+}$  ions (Cao Tingli et al., 2017; Dai Chaocheng et al., 2017). The common iron-bearing minerals in the samples include chlorite cements coated other clastic grains, and pyrite cements filled in pores (Figs. 4c–4f). The contents of chlorite cement in the studied samples are less 1% generally (Table 1). Content of the pyrite cement from different wells differs obviously, and higher values only distribute in the sample from well Tazhong 161.

With continous influxion of external acidic and oxydic fluid,  $Fe^{2+}$  ions of the chlorite and pyrite cement in the samples generated flocculent precipitate  $Fe(OH)_3$  during the acid sensitivity testing (Fig. 5). These precipitates would block throat easily, causing permeability deterioration.

According to the content of acid-sensitive minerals in the samples (Table 1, 2), the studied samples can be roughly divided into four types: (1) the content of the ironbearing minerals is greater than that of the iron-free minerals (Sample 2-1), (2) the content of the iron-free minerals is less than that of the iron-bearing minerals (Sample 2-3 and Sample 2-4), (3) the content of the ironbearing minerals is much higher than that of the iron-free minerals (Sample 2-5 and Sample 2-6), (4) the content of the iron-free minerals is roughly equal to that of the ironbearing minerals (Sample 2-2, Sample 2-7 and Sample 2-8).

## **5 Acid Sensitive Flow Test**

According to the content of two different acid-sensitive mineral types, four samples (Sample 2-1, Sample 2-3, Sample 2-5 and Sample 2-7) were selected for the acidsensitive flow testing. With continuous increase of volume ratio of acidic fluid injected into the samples, the acidsensitive minerals in the samples kept reacting with acidic fluid, and permeability of the sample was changed gradually (Fig. 6).

The difference between initial permeability of four samples is negligible (Table 2). The results of acid-sensitive test show that the difference of acid sensitivity damage rate among four samples is not remarkable (Table 2). The acid sensitive effect of Sample 2-5 is medium to strong, and Sample 2-1 is weak, Sample 2-3 and 2-7 is



Fig. 5. Microscopic change of pores in the Kepingtage Formation before and after acid sensitivity simulation testing (26 hours immersion in 15% hydrochloric acid).

(a), microscopic characteristics of initial pore before the testing (Tazhong471D, 5018.00m,  $S_1k_u^3$ ); (b), microscopic characteristics of pore after the testing (Tazhong471D, 5018.00m,  $S_1k_u^3$ ).



Fig. 6. Curves of acidic sensitivity of different samples in the Silurian Kepingtage Formation in Tazhong area.

medium to weak.

#### **6** Discussions

Although the difference of damage rate among four samples is not remarkable, the permeability variation curve during the testing is meaningful.

(1) Sample 2-1. The permeability value decreases

steadily during the process of testing (Fig. 6a). The reason is that the content of the iron-bearing minerals is significantly higher than that of the iron-free minerals. Consequently, the iron-bearing minerals which cause decrease of permeability, determined completely changing trend of permeability during the testing process.

(2) Sample 2-3. The permeability value increases quickly during the process of testing (Fig. 6b). This is

because that the content of the iron-free minerals is significantly higher than that of the iron-bearing minerals. Then the iron-free minerals which result in increase of permeability, dominated completely the changing trend of permeability in the testing process.

(3) Sample 2-5. The permeability value of the sample decreases substantially during the process of testing (Fig. 6c). Only the iron-bearing minerals are included in the sample, and no iron-free minerals are detected. The iron-bearing minerals causing permeability decrease determine completely the change trend of permeability during testing.

(4) Sample 2-7. The permeability value decreases slightly in the process of testing (Fig. 6d). But there is a clear fluctuation in the changing curve of permeability during the test. The content of the iron-bearing minerals is almost equivalent to that of the iron-free minerals. Two types of acid-sensitive minerals cause the opposite change of permeability, which is restricted each other during the testing, and this restriction is reflected in the changing curve of permeability.

By comparing the content of acid-sensitive minerals in different samples, the results show that permeability increment is caused by iron-free minerals, while permeability deterioration is induced by restriction of the iron-containing minerals, which can be reflected in the change of permeability test curve during acid-sensitive flow testing.

Although the difference of acid sensitivity damage rate among samples is not obvious, the sensitization mechanism is significantly different. The iron-free minerals are responsible for the acid-sensitive damage rate, and cause permeability increase to improve reservoir properties. So if the acid-sensitive mechanism of the reservoir is clear, some specific and workable proposals can be carried out to inhibit acid-sensitive effect of ironbearing minerals in the reservoir. For example, some Fe<sup>2+</sup> stabilizer can be added in acidizing fluids during acidizing treatment, which inhibits precipitation of iron hydroxide {Fe(OH)<sub>3</sub>} by reducing Fe<sup>3+</sup> to Fe<sup>2+</sup> (Zhang Shaohuai et al., 1996; Civan, 2007). This method can optimize acidizing effect and improve reservoir permeability to a certain extent.

# 7 Conclusions

(1) The Kepingtage Formation has an obvious acidsensitive effect. The minerals causing acidic sensitivity in the reservoir can be divided into two categories: the ironbearing minerals including pyrite cements and chlorite cements, and the iron-free minerals including calcite cements and dolomite cements. (2) Different acid-sensitive minerals can have opposite sensitization mechanisms. During acid-sensitive flow testing, the iron-bearing minerals cause the decrease of the permeability, and the iron-free minerals lead to the increase of reservoir permeability. Permeability variation trend is the result of mutual restriction of different sensitization mechanisms.

(3) If sensitization mechanisms and their restriction effect on changing trend of reservoir permeability is clear, then some effective acidizing treatment, for example, adding corresponding  $Fe^{2+}$  ion stabilizer into acidizing fluids, can restrain unfavorable factors of the acid-sensitive mechanism and improve reservoir properties.

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#### About the first author

HAN Denglin, male; Born in 1979 in Nanyang City, Henan Province; Doctor; Graduated from Institute of Geology and Geophysics, Chinese Academy of Sciences; Professor of School of Geosciences, Yangtze University. He is now interested in the study on reservoir diagenesis, and prediction of reservoir quality. Email: handl@yangtzeu.edu.cn; Phone: 18163308273.