A laboratory approach to predict the water-based drill-in fluid damage on a shale formation

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Abstract
Shale gas production after drill-in, completion, and hydraulic fracturing is strongly affected by formation damage. In order to determine the damage mechanisms for nonmarine shale reservoir, a series of assessments of sensitivity damage, water block damage, water-based drill-in fluids damage, and water damage to gas diffusion on 20 shale samples obtained from Chang 7 Formation were conducted and analyzed. Results indicate that, in the Chang 7 Formation shale, there is extremely strong stress sensitivity and moderately weak water sensitivity damage. Although the liquid phase invasion depth is shallow and the water block damage is limited, the liquid phase and solid particles would enter the microfractures in the reservoir. The P-1 water-based drill-in fluid is compatible with the Chang 7 Formation shale reservoir which can meet the requirement of Chang 7 Formation shale damage controlling, the effect of water-based drill-in fluid on wellbore stability should be paid more attention. The diffusion coefficient of the shale decreases with the presence of water. A systematic damage evaluation method of working fluid considering the multi-mechanism and multi-scale mass transfer process of shale gas is needed to establish.

Keywords
Shale gas, formation damage, sensitivity damage, water block, water-based drill-in fluid, diffusion

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Shale gas is popular in the exploration and development of unconventional oil and gas in the world due to advances in drilling and fracturing techniques (Charoen suppanimit et al., 2012). In recent years, a significant breakthrough has been made in the southern marine shale gas field represented by the Fluling, Changning-Weiyuan, and Shaotong shale gas in Sichuan Basin, they are the mainly commercial shale gas fields in China, the cumulative developed reserves reached $10,450 \times 10^9$ m$^3$ by the end of 2018, yearly production $110 \times 10^9$ m$^3$ in 2018 (Jiping, 2019). In addition to marine shale gas, nonmarine shale gas is also an important development direction in the future. The Shaanxi Yanchang Petroleum (Group) Corp. Ltd took the lead in theoretical research and technical practice of nonmarine shale gas exploration and development (Xiangzeng, 2018). At present, the Chang 7, Chang 9 in the Mesozoic Yanchang Formation, the Neopaleozoic Shanxi Formation, and Benxi Formation in the Ordos Basin are the main target zones of shale gas; the proven geologic reserves are $1.5 \times 10^{12}$ m$^3$, especially Chang 7 shale in Yanchang Formation. With the exploitation of shale reservoirs, the formation damage and protection which affect the shale gas well productivity has become more and more significant and urgent (Xu et al., 2016).

The natural gas in shale is stored as gas adsorbed onto kerogen and clay-particle surfaces, and as free gas or as dissolved gas (Curtis, 2002). Shales are complex rocks, characterized by heterogeneity in composition and pore volume distribution (Heller and Zoback, 2014). The organic matter and clay minerals are two main compositions, and the content of clay mineral including fine-grained bentonite, illite, kaolinite, etc. in shales is usually larger than that of organic matter (Zhai et al., 2014). The minimum pores in shale reservoir are at nanoscale in diameter (Rui et al., 2015), and the pore size across macro- to meso- and microscales (Heller and Zoback, 2014; Yanyan Chen et al., 2015), so the porosity and permeability are extremely low, and the pore structure is complex. Besides, the shale formation has a low initial water saturation (Shen et al., 2016). These mineral compositions and physical properties combining with the external condition, including long contact time and large contact area between the working fluid and formation in horizontal well, high temperature and pressure in deep shale reservoir, and various working fluid systems, are all challenges to evaluate the formation damage and develop the protection technology in the drilling, completion, and fracturing process in shale gas reservoir.

Although formation damage of conventional natural gas and tight sandstone gas reservoir has been well documented (Dong et al., 2019; Lei et al., 2017; Lufeng et al., 2019; Song et al., 2018; Zhang et al., 2019), few studies which focus on shale formation damage especially for nonmarine shale have been reported, and the details of the mechanism are not well understood. The first aspect involves the shale formation sensitivity damage and water block damage for shale formation. It is generally believed that reservoir sensitivity is the internal cause of reservoir damage and the basis of working fluid damage evaluation (Civan, 2007). Shale reservoir suffers from fluid-sensitive damage and stress-sensitive damage as it is rich in clay minerals. The former is resulted from the hydration and expansion of clay, while the latter is caused by mineral particle migration and fluid lubrication, which leads to reduced rock strength and narrowed seepage channel. Alternatively, because the minimum pores in shale reservoir are at nanoscale in diameter (Javadpour et al., 2012, 2007; Rui et al., 2015),
the capillary force is very strong, and therefore the shale reservoir may be particularly sensitive to various damages, such as water block and pore throat plugging by expanded clay. But water imbibition may benefit fracture generation in shale reservoir and this has attracted the attention of many scholars (Davarpanah, 2018; Davarpanah et al., 2019; Li et al., 2017b; Shen et al., 2016). The second aspect involves the water-based drill-in fluids damage for shale formation. For working fluid damage to shale formation, Xu et al. (2016) preferred the main damage types as chemical damage and physical damage, the former involves incompatibilities between shale and working fluid, and incompatibilities between formation fluid and working fluid, the latter one involves formation fines migration, working fluid particle invasion, phase trapping, and stress damage. Li et al. (2017b) investigated the effect of drill-in fluids on formation damage and wellbore stability for Longmaxi marine shale in Chongqing district of Sichuan Basin in China. Akpan et al. (2019) who studied the effect of pH control agents, thermal stabilizers, and anti-oxidants on the stability of biopolymer in water-based drill-in fluid, and analyzed the reasons caused formation damage for the Marcellus shales. The third aspect relates to the damage of gas diffusion in shale formation. The original concept of formation damage initially refers to the impairment of the permeability of oil and gas reservoir by various adverse processes (Civan, 2007), and conventional assessment methods of formation damage have mainly focused on the decrease of permeability. But it is generally believed that the start of gas transfer in the production is the gas desorption on the nanopore wall of shale matrix, then gas diffusion through primary mesoscale porosity, and flow to wellbore at last (Davarpanah and Mirshekari, 2019; Etminan et al., 2014; Javadpour et al., 2007; King, 1993). Consequently, the formation damage assessment for shale formation must consider the effect of working fluid on the gas desorption and diffusion in the reservoir. Xu et al. (2016) also proposed the formation damage considering the multi-scale mass transfer process including seepage, diffusion and desorption of shale gas in reservoir should be evaluated. Yuan et al., (2014) found that water reduces gas storage capacity and transport rate in shale.

For Chang 7 Formation nonmarine shale, one of the reservoir characteristics is that the clay content is higher than that of other marine shales (Curtis, 2002; Xiangzeng, 2018), which may generate serious stress damage and water sensitivity damage resulting from hydration and expansion of clay. Besides, in order to maintain the wellbore stability, oil-based drill-in fluid with good inhibition and lubrication performance was used first in the drilling of nonmarine shale gas well in the Yanchang Formation at early stage, but it has prominent environmental and high cost issues (Akpan et al., 2019). The second choice for drilling shale gas wells, a water-based drill-in fluid, P-1, was developed which has strong characteristics of shale inhibition and sealing, and has been applied in Yanchang oil field recently (Bo et al., 2018). Thus in the drilling process, the corresponding formation damage including particle invasion, water phase trapping damage, and rock–fluid incompatibility (Bennion, 2002; Xu et al., 2016) may occur. So, systemic study about this subject is needed.

In this study, a series of mineral compositions and physical properties test, and assessments of fluid and stress sensitivity damage, water block damage, water-based drill-in fluids damage, and water damage to gas diffusion on Chang 7 Formation shale samples were conducted to analyze the damage characteristic and mechanisms. Then the performance and compatibility of a water-based drill-in fluid were analyzed and approach to protecting the Chang 7 Formation shale was proposed.
Materials and methods

Samples and sample preparation

Shale samples. We used shale samples from Chang 7 of Mesozoic Yanchang formation in this study, which were collected from well FY-2 (burial depth of 1405.13–1440.96 m) belonging to the Shaanxi Yanchang Petroleum (Group) Corp. Ltd, in Yan’an area, southern Ordos in China (Figure 1). Table 1 lists the size for the shale samples. The Chang 7 Formation shales have intermediate to high total organic carbon (1.75–5.88%), and in an immature–mature stage, thermal evolution degree vitrinite reflectance (Ro, 0.72–1.25%) is low, the kerogen is mainly type II, and the clay contents are 42.2–73.7 wt%, in which the dominant clay mineral is illite. There is a wide distribution of the shales with strong gas content (1.87–5.23 ml/g) and the maximum Langmuir adsorption capacity ranges from 1.62 to 5.46 m³/t. The reservoir is tight, and porosity (1.1–4.55%) and permeability (0.8–30.2 × 10⁻⁹ m²) are very low. The details of the reservoir characteristics of Chang 7 shale and comparing to that of other shales are shown in previous studies (Wang et al., 2016; Xiangzeng, 2018).

The shale core samples used in sensitivity damage, water block damage, and drill-in fluid damage assessment were cut, dried by method based on the API Standard RP-40, and then

Figure 1. Structural diagram of Ordos basin with the location of sample well.
preserved in an oven. The shale sample FY34-2 used in the diffusion experiment was crushed to 0.380 mm size particle (ds < 40 mesh), and then was dried for 12 h at 110°C in a temperature-controlled drying oven. Part of the dry sample was placed in to a vacuum dryer loaded with K₂SO₄ supersaturated solution, it was weighed until the change rate of the weight was less than 2% in 24 h, then the water content of the sample was determined.

**Water samples.** The saline water with the salinity of 21,035.92 mg/l was used in the experiment, as the formation water in Chang 7 Formation has this salinity and is CaCl₂ type.

**Water-based drill-in fluids samples.** The water-based drill-in fluids samples used in the experiment, named as P-1, were purchased from the Exploration and Development Research Center, Yanchang Oil Field Co., Ltd, which has strong characteristics of shale inhibition and sealing (Bo et al., 2018). The formula of the drill-in fluids was as follows (wt%): 4.0% amargosite + 1.0% adhesion promoter SM-1 + 0.1% adhesion promoter F3 + 33% formate + 2.0% fluid loss agent + 0.1% thinning agent + 9.0% blocking agent + 2.0% lubricant RH220. The performance parameters of the water-based drill-in fluid are listed in Table 2, pH = 9, the median diameter, \( d_{50} = 11.539 \mu \text{m} \), other diameter parameters, \( d_{10} = 0.661 \mu \text{m} \), \( d_{90} = 109.481 \mu \text{m} \).

**Experimental methods**

*Mineral compositions and physical properties test.* The X-ray diffraction, mercury intrusion porosimetry, and liquid nitrogen adsorption were used to test the mineral compositions and physical properties of the shale samples. They are conventional projects for core analysis, the instruments and methods are presented in the literatures (Dong et al., 2019; Rui et al., 2015), and will not be repeated here.

*Fluid and stress sensitivity damage assessment.* The instruments of fluid and stress sensitivity damage assessment, water block damage assessment, and water-based drill-in fluids damage assessment are at the Research Center of Development and Management for

**Table 1.** Shale samples in experiment.

| No. | Core sample | Length (cm) | Diameter (cm) | Depth (m) | Fracture | No. | Core sample | Length (cm) | Diameter (cm) | Depth (m) | Fracture |
|-----|-------------|-------------|--------------|-----------|----------|-----|-------------|-------------|--------------|-----------|----------|
| 1   | FY105-1     | 4.744       | 2.511        | 1419      | Y        | 11  | FY45-3      | 4.502       | 2.505        | 1411      | Y        |
| 2   | FY46-1      | 4.637       | 2.518        | 1411      | Y        | 12  | FY92-2      | 4.580       | 2.511        | 1417      | N        |
| 3   | FY43-2      | 5.368       | 2.495        | 1411      | Y        | 13  | FY72-2      | 4.965       | 2.504        | 1414      | N        |
| 4   | FY46-2      | 8.512       | 2.505        | 1411      | Y        | 14  | FY36-1      | 5.235       | 2.496        | 1410      | N        |
| 5   | FY92-1      | 5.350       | 2.498        | 1417      | N        | 15  | FY32-1      | 4.820       | 2.511        | 1409      | Y        |
| 6   | FY34-4      | 5.686       | 2.507        | 1410      | Y        | 16  | FY38-1      | 5.012       | 2.499        | 1410      | Y        |
| 7   | FY49-1      | 5.001       | 2.525        | 1411      | Y        | 17  | FY58-1      | 5.750       | 2.508        | 1412      | N        |
| 8   | FY72-1      | 5.675       | 2.510        | 1414      | N        | 18  | FY24-1      | 5.032       | 2.510        | 1408      | Y        |
| 9   | FY34-1      | 6.095       | 2.505        | 1410      | Y        | 19  | FY35-1      | 4.368       | 2.453        | 1410      | N        |
| 10  | FY34-3      | 6.119       | 2.510        | 1410      | N        | 20  | FY34-2      | 5.686       | 2.507        | 1410      | Crush to |

Note: The N refers to unfractured core, and Y refers to fractured core, which is created fracture by the Brazilian tensile test.
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The apparatus used for fluid and stress sensitivity damage assessment on shale core samples with fracture is a conventional permeameter. For unfractured shale core samples, because they are too tight, the permeability measurement was performed on a pulse decay permeameter, which is attached to a GCTS\textsuperscript{V}R Rapid Triaxial Rock (RTR)-1000 Testing System.

The fluid and stress sensitivity assessment of core with cracks was conducted according to a China Petroleum Standard SY/T 5358-2010; the fluid sensitivity includes velocity sensitivity, water sensitivity, and alkali sensitivity. The details of method were also mentioned in Dong et al.’s (2019) work. For the shale core without fracture, the core was first vacuum pumped, and then saturated with the prepared saline water or alkaline liquor for 12 h. Subsequently, the permeability variation of the core was measured by the pulse decay permeameter to obtain the evaluation results of its water sensitivity and alkali sensitivity damage; the pulse decay method has been clearly described in the API Standard RP-40 (RP40, 1998). The stress sensitivity damage was measured by a similar procedure with altering the confining pressure

\[ D_k = \frac{|K_n - K_i|}{K_i} \]  

where \( D_k \) is the permeability damage rate, \( D_{k-v}, D_{k-w}, D_{k-a}, D_{k-s} \) refer to the velocity-sensitive damage rate, the water-sensitive damage rate, the alkali-sensitive damage rate, and the stress-sensitive damage rate, respectively (%); \( K_n \) is the permeability measured at different condition (10\textsuperscript{-3} \( \mu \text{m}^2 \)); \( K_i \) is the initial permeability (10\textsuperscript{-3} \( \mu \text{m}^2 \)).

**Water block damage measurement.** The water block damage was measured by core imbibition test associated with mobile water saturation test; the experimental devices are the core imbibition apparatus (Figure 2), the HX-BH-I core vacuum and saturation apparatus, and the MesoMR23-060H-I nuclear magnetic resonance (NMR) apparatus.

After weighing the dry shale core, it was put into the core inhibition device to record its weight; the method is similar to the work presented by Shen et al. (2016), so that the amount change of imbibition water over time was obtained, through which the imbibition water volume in pore volume of the core and penetration depth of water into the core were calculated. The core was vacuum pumped by the core vacuum and saturation apparatus and then saturated with the same clean water that was used to prepare the drill-in fluid for 12 h. After that, the core was taken out to wipe the water on its surface. Then the T2 map of

| Temperature (\( ^\circ \text{C} \)) | PV (mPa s) | AV (mPa s) | API (ml) | HTHP (ml) | Initial shearing force (Pa) | Final shearing force (Pa) | Dynamic shearing force (Pa) |
|---------------------------------|-----------|-----------|----------|-----------|-----------------------------|--------------------------|---------------------------|
| 25                              | 40        | 53        | 3.5      | –         | 3.066                        | 4.088                    | 13.286                    |
| 50                              | 22        | 33        | –        | –         | 2.044                        | 2.555                    | 11.242                    |
| 80                              | 15        | 11.5      | –        | 6.5       | 1.022                        | 1.2775                   | 4.088                     |

AV: apparent viscosity; HTHP: filtration rate at high temperature and high pressure; API: filtration rate measured using the API method. PV: plastic viscosity.
the shale core sample was measured by using the NMR spectrometer and the experimental produce has been described by Gao and Li (2015). At last, the mobile water saturation was calculated.

Test of water-based drill-in fluids damage. The test of water-based drill-in fluids damage utilized a JHMD-II drill-in fluids damage assessment system (Figure 3) and an Olympus DSX-500 optical digital microscope.

The experimental method was based on China Petroleum Standard SY/T 6540-2002 and other literature (Li et al., 2017b; Lufeng et al., 2019). Both dynamic damage and static damage simulation of drill-in fluids were carried out in the drill-in fluids damage assessment.
system. The only difference is that the drilling liquid did not circulate during static damage simulation. The experiment time was 2 h, the temperature was 50°C, the confining pressure was 6 MPa, and the pressure difference was 3.5 MPa. After the damage simulation, the core was taken out and displaced with nitrogen, and the value of stable gas-measured permeability was recorded. The permeability values at the contaminated end surface and at the cutting surface 0.7 cm from the end were measured to evaluate the penetration depth of drilling fluid. Finally, the core surfaces before and after the contamination as well as after the removal of the contaminated section were observed by the optical digital microscope to capture plane view image and three-dimensional (3D) image using a visual image processing method (Bai et al., 2018), and compared to further determine the degree of damage.

Evaluation of water damage on gas diffusion. The instrument used in the sorption and diffusion experiment is the HX-I volumetric sorption apparatus, which is an improvement of the device used in our previous study (Rui et al., 2015).

The details of measurement have been reported in the previous studies (Chen et al., 2018; Li et al., 2017a; Zhong et al., 2019), which involves three steps: the methane adsorption of shale sample was carried out, and measuring gas desorption amounts over time, then the diffusion coefficient was calculated based on the desorption data. The method of the adsorption is as same as the method of the isotherm adsorption (Rui et al., 2015). In the experiment, the adsorption equilibrium pressure is set to 8 MPa and the temperature is set to 30°C.

After the measurements, the obtained desorption capacities with the time were fitted to the unipore diffusion model (Crank, 1975), which is based on the Fick’s second diffusion law. The solving process was shown in the literature (Davarpanah and Mirshekari, 2019; Yang Qiluan, 1986), and the formula to calculate the diffusion coefficient is expressed as

\[
D_e = \frac{\pi}{t} \left( \frac{V_t}{6V_\infty} \right)^2
\]

where \(D_e\) is the effective diffusion coefficient \(D_e = D / r_p^2\), \(m^2/s\); \(r_p\) is the diffusion path, \(m\); \(V_t\) is the cumulative desorption amount with time \(t\), \(10^{-3} m^3/kg\); \(V_\infty\) is the maximum desorption amount, \(10^{-3} m^3/kg\) (S.C.). Thus, the relationship between the ratio of the desorption amount to the maximum desorption amount and the square root of time is

\[
\frac{V_t}{V_\infty} = 12 \left( \frac{D}{\pi} \right)^{0.5} d^{-1} t_{0.5}
\]

It can be linear fitted, and the slope of the straight line is \(12 (D/\pi)^{0.5} d^{-1}\), from which the diffusion coefficient \(D\) was calculated.

Results

Mineral compositions and physical properties

The mineral compositions, relative contents of clay mineral, and the physical properties of the shale samples are shown in Tables 3 and 4, and comparison of solid particle size in drill-in fluid and pore size distribution are shown in Figure 4.
As can be seen from Table 3, the Chang 7 Formation shale has a clay content of up to 3.31%, so it is likely to be highly stress-sensitive damage. Among the clay minerals, 88.78% are velocity-sensitive minerals (illite, kaolin, and chlorite) and 11.22% are water-sensitive minerals (bentonite, andreattite), which indicates that there may be some damages caused by velocity sensitivity or water sensitivity in the reservoir. Table 4 reveals that the Chang 7 Formation porosity ranges from 0.43 to 3.80% and permeability is mainly less than $0.05 \times 10^{-2} \text{ m}^3/\text{C0}$ since the minimum diameter of the pores in the reservoir is at nanoscale (< 0.1 μm) (Figure 4), which is far below the size of solid particle ($d_{50} = 11.539 \mu m$) in the

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**Table 3.** Mineral compositions and relative contents of clay mineral of the shale samples.

| Mineral compositions (wt%) | Relative contents of clay mineral (wt%) |
|---------------------------|---------------------------------------|
| Clay | Quartz | feldspar | Plagioclase | Calcite | Dolomite | Siderite | Pyrite | Illite | Bentonite | Andreattite | Kaolin | Chlorite |
| Clay | 34.31 | 43.81 | 4.95 | 9.17 | 3.05 | 1.61 | 0.95 | 62.53 | 0.00 | 13.26 | 10.46 | 13.72 |

**Table 4.** Physical properties of the shale samples.

| Porosity (%) | Permeability ($10^{-3} \mu m^2$) | Mercury intrusion | Liquid nitrogen adsorption | Surface to volume ratio (m$^2$/g) |
|--------------|---------------------------------|-------------------|---------------------------|-------------------------------|
| Range        | Average | Range | Distribution | Range | Average | Range | Main peak | Range | Average |
| 0.43–3.80    | 1.38    | 0.00034–7.6544 | <0.05 (52.94%) | 0–100 | 50–70 | 1–55 | 2–5 | 0.397–0.861 | 1.931 |

**Figure 4.** Comparison of solid particle size in drill-in fluid and pore size distribution.
drilling fluid, the invasion of external solid will cause small damage, and the fine pore throats, however, may cause serious water block damage under strong capillary force. But if microfractures in formation may develop, solid particles will enter the microfractures and together with the drilling liquid damage the reservoir. Therefore, it is necessary to improve the sealing characteristics of drilling liquid and mud cake quality.

**Table 5. Results of velocity sensitivity damage test.**

| Shale core sample | Fracture | Q (ml/min) | 0.030 | 0.050 | 0.075 | 0.100 | Degree of damage |
|-------------------|----------|------------|-------|-------|-------|-------|-----------------|
| FY105-1           | Y        | $K \left(10^{-3} \ \mu m^2\right)$ | 0.2400 | 0.2010 | 0.1740 | 0.1670 | – Moderately weak |
|                   |          | $D_{k-v}$ (%) | 0.00 | 16.25 | 27.63 | 30.42 | – |
| FY46-1            | Y        | $K \left(10^{-2} \ \mu m^2\right)$ | 0.0835 | 0.0715 | 0.0539 | 0.0449 | – Moderately weak |
|                   |          | $D_{k-v}$ (%) | 0.00 | 14.33 | 35.70 | 46.16 | – |

**Table 6. Results of water sensitivity damage test.**

| Shale core sample | Fracture | Salinity | $S_{fw}$ | 0.75$S_{fw}$ | 0.5$S_{fw}$ | 0.25$S_{fw}$ | 0 $S_{fw}$ | Extent of damage |
|-------------------|----------|----------|----------|--------------|-------------|--------------|-------------|-----------------|
| FY43-2            | Y        | $K \left(10^{-3} \ \mu m^2\right)$ | 0.2542 | 0.2478 | 0.2230 | 0.1856 | 0.1588 | Moderately weak |
|                   |          | $D_{k-w}$ (%) | 0.00 | 2.18 | 7.31 | 26.99 | 34.01 | |
| FY46-2            | Y        | $K \left(10^{-3} \ \mu m^2\right)$ | 10.1164 | 9.6225 | 9.1477 | 6.8350 | 5.1820 | Moderately weak |
|                   |          | $D_{k-w}$ (%) | 0.00 | 4.88 | 9.58 | 32.436 | 48.78 | |
| FY92-1            | N        | $K \left(10^{-6} \ \mu m^2\right)$ | 1.3032 | 1.1423 | 1.0641 | 0.9072 | 0.8305 | Moderately weak |
|                   |          | $D_{k-w}$ (%) | 0.00 | 12.35 | 18.35 | 30.39 | 36.27 | |

**Table 7. Results of alkali sensitivity damage test.**

| Shale core sample | Fracture | pH | 7 | 8.5 | 10 | 11.5 | 13 | Extent of damage |
|-------------------|----------|----|---|-----|----|------|----|-----------------|
| FY34-4            | Y        |    | 4.5812 | 3.7811 | 3.0049 | 2.4585 | 2.0839 | Moderately strong |
|                   |          | $D_{k-a}$ (%) | 0.00 | 17.46 | 34.41 | 46.34 | 54.51 | |
| FY49-1            | Y        |    | 2.2078 | 1.6265 | 1.2732 | 1.0079 | 0.8055 | Moderately strong |
|                   |          | $D_{k-a}$ (%) | 0.00 | 26.33 | 42.33 | 54.35 | 63.52 | |
| FY72-1            | N        |    | 16.8380 | 14.6200 | 12.7800 | 10.9500 | 10.2660 | Moderately weak |
|                   |          | $D_{k-a}$ (%) | 0.00 | 13.17 | 24.10 | 34.97 | 39.03 | |

Note: Q is flow rate (ml/min).

Fluid and stress sensitivity damage

**Velocity sensitivity, water sensitivity and salinity sensitivity damage.** The measured results of velocity sensitivity, water and salinity sensitivity damage test are listed in Tables 5 to 7.

The velocity-sensitive critical flow rates of FY105-1 and FY46-1 (core samples with fracture) were 0.075 ml/min, and the corresponding velocity-sensitive damage rates ($D_{k-v}$) were 30.42 and 46.16%, respectively, and the extent of formation velocity-sensitive damage was moderately weak, $30\%<D_{k-v}<50\%$. FY43-2 and FY46-2 (core samples with fracture) were sensitive to the formation water with low salinity and the maximum water sensitivity
damage rate was 48.78%, which was higher than the water-sensitive damage rate of core samples without fracture \( D_{k-w} = 36.27\% \), thus the extent of water-sensitive damage was moderately weak, \( 30\% < D_{k-w} < 50\% \), and the critical salinity was 1/2 of the formation water salinity. The maximum alkali-sensitive damage rate of FY34-4 and FY49-1 (core samples with fracture) was 63.52%, and the alkali-sensitive damage rate of FY72-1 (core sample without fracture) was 39.03%, the extent of alkali-sensitive damage of the former two was moderately strong, \( 50\% < D_{k-a} < 70\% \), while that of the latter one was moderately weak, and their critical pH values were all 8.5.

**Stress sensitivity damage.** The measured results of stress sensitivity damage test are presented in Figure 5 and Table 8.

Figure 5 and Table 8 show that when the net stress of FY34-1 (core sample with fracture) rose from 2.5 to 20 MPa, the damage rate was over 90% and then declined to 72.43% after the net stress dropped from 20 to 2.5 MPa. By contrast, when the net pressure of FY34-3 (core sample without fracture) increased from 2.5 to 20 MPa, the damage rate reached 95.74%, then it declined to 83.67% as the net stress fell back to 2.5 MPa. It is therefore concluded that the core samples with and without fracture both have severe stress-sensitive damages, extents of damage are all extremely strong \( (D_{k-s} > 70\%) \), but the stress-sensitive damage of the latter is greater.

**Water block damage**

Figure 6 shows the results of core imbibition test and mobile water saturation test using NMR; the value of the relevant parameters is listed in Table 9.

As can be seen from Figure 6(a), the imbibition rate of core was relatively fast within the first 5 min and then gradually decreased 10 h later; the imbibition amount reached saturation and did not increase any more. Specifically, the imbibition rate of FY45-3 (core with fracture) was higher than that of FY92-2 (core without fracture), so FY45-3 had a larger amount of imbibition water. The calculation results listed in Table 10 show that the imbibition water accounts for less than 20% of the pore volume of core samples. In addition, the T2 maps measured by NMR display a bimodal peak (Figure 6(b)), in which the area beneath
Table 8. Results of stress sensitivity damage test.

| Shale core sample | Fracture | Net confining pressure (MPa) | 2.5 | 3.5 | 5  | 7  | 9  | 11 | 13 | 15 | 20 |
|-------------------|----------|-----------------------------|-----|-----|----|----|----|----|----|----|----|
| FY34-1 Y Stress up| $K \left(10^{-3} \mu m^2\right)$ | 0.0504 | 0.0437 | 0.0346 | 0.0253 | 0.0186 | 0.0130 | 0.0107 | 0.0082 | 0.0043 | Extremely strong |
| Stress down       | $D_{k,s} (%)$            | 0.00  | 13.15 | 31.25 | 49.69 | 63.05 | 72.94 | 78.73 | 83.77 | 91.37 |                 |
| FY34-3 N Stress up| $K \left(10^{-6} \mu m^2\right)$ | 2.8445 | 2.6011 | 2.1215 | 1.3218 | 0.8456 | 0.3337 | 0.2484 | 0.1489 | 0.1211 | Extremely strong |
| Stress down       | $D_{k,s} (%)$            | 0.00  | 8.55  | 25.41 | 53.53 | 70.27 | 88.26 | 91.26 | 94.76 | 95.74 |                 |

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the left peak represents the bound fluid content, yet that under the right peak represents the movable fluid content, so the bound water saturation is over 90% (Table 9). From the above, it can be summarized that the pore throats in the Chang 7 Formation shales are tiny, and the capillary force is quite strong, once the water phase enters the throats, it will become bound water, leading to water block which will effectively prevent the follow-up fluid phase from entering, and thus the water lock penetration depth is shallow.

Water-based drill-in fluids damage

The permeabilities of shale core samples before and after contamination, after washing end face, and cutting surface 0.7 cm from the end in dynamic and static damage simulation are listed in Table 10.

The damage rates of cores FY32-1, FY38-1, and FY58-1 were up to 70–80% after dynamic contamination, and the higher the permeability, the greater the damage rate. The gas-measured permeability rebounded slightly after washing the end surface, and the recovery rate of gas surveying permeability after removal of 0.7 cm contaminated section was 50–80%. Fractures had large impact on damage and recovery rate, so the cores with cracks had a high permeability and damage rate, and the recovery rate after cutting the contaminated section was small. For instance, the damage rate of FY32-1 (core with fracture) was 87.42% and its recovery rate after removal of contaminated end was 55.80%; in contrast, the damage rate of FY58-1 (core without fracture) was 71.04%, and its recovery rate after removing

Table 9. Results of core imbibition test and mobile water saturation test using NMR.

| Core imbibition test | NMR |
|---------------------|-----|
| Shale core sample | \(\phi\) (%) | \(\phi_{sw}\) (%) | Shale core sample | \(\phi_{NMR}\) (%) | SWB (%) | SMW (%) |
| FY45-3 | 1.84 | 19.18 | FY72-2 | 1.13 | 97.35 | 2.65 |
| FY92-2 | 1.21 | 11.87 | FY36-1 | 1.31 | 95.11 | 4.89 |

NMR: nuclear magnetic resonance.
Note: \(\phi\) is porosity (%); \(\phi_{sw}\) is rate of imbibition water to pore volume (%); \(\phi_{NMR}\) is porosity measured by NMR (%); SWB is bound water saturation (%); SMW is mobile water saturation (%).
contaminated end was 85.02%. Relative to dynamic contamination, the static damage rates of FY24-1 and FY35-1 were both over 90%, which were pretty high. And their recovery rates measured at washed end surface and the surface after removal of 0.7 cm contaminated end were not significantly different from those under dynamic state. However, owing to the cracks in FY24-1, its original permeability was high; hence, the recovery rates of FY24-1, whether measured at washed end surface or the surface after removal of 0.7 cm contaminated end, were low in comparison with FY35-1, the core without cracks.

The photos of end surface for cores FY 32-1 after dynamic contamination and FY 24-1 after static contamination are shown in Figure 7, and the plane view images and 3D black-white and colored images for core FY32-1 and FY 24-1 are shown in Figure 8.

Figure 7 shows that after the drilling fluid dynamically polluted the core FY 32-1, the mud cake formed at the end surface of the core was thinner than that formed due to static contamination of core FY 24-1. Through optical microscope observation at the same magnification (Figure 8), \( M = 250 \), it was found that the end surface of the core FY32-1 was covered with drilling fluid, the 3D black-white and colored images show that the end surface of the core was highly uneven after dynamic contamination, indicating that the solid particles in drilling fluid accumulated, leading to block off on the end surface of the core (Figure 8(a)). Through the microscopic images of the end surface of core FY 24-1 after static contamination, it was found that the end surface was even more uneven, compared with dynamic contamination, the drilling fluid did not wash the end surface during static contamination and thus the solid particles were only affected by the vertical force, so the mud cake formed was thicker (Figure 8(b)). The end surfaces after dynamic and static contamination differ significantly from those before pollution (Figure 8(c)) and after

| Damage simulation | Shale core sample | Fracture | \( K \times 10^{-3} \mu m^2 \) | \( D_k \) (%) | \( R_k \) (%) |
|-------------------|------------------|---------|-----------------|------------|----------|
| Dynamic           | FY 32-1 Y        | Before contamination | 2.1999 | – | – |
|                   |                  | After contamination | 0.2768 | 87.42 | – |
| FY 38-1 Y         |                  | Before contamination | 0.0712 | – | – |
| FY 58-1 N         |                  | Before contamination | \( 0.7183 \times 10^{-3} \) | – | – |
| Static            | FY 24-1 Y        | Before contamination | 13.118 | – | – |
| FY 35-1 N         |                  | Before contamination | 0.05104 | – | – |

Note: \( R_k \) is permeability recovery rate, \( R = 1 - D_k \).
removal of ≤1 cm end (Figure 8(d)) for core FY24-1. The end surfaces of the latter two were cleaner, which suggests that the penetration depths of solid phase and liquid phase drilling fluid into the core were below 1 cm.

**Water damage on gas diffusion**

The relationship between the methane desorption capacity for the Chang 7 shale samples and desorption time, the relationship between the ratio of desorption amount to maximum desorption amount and the square root of time, and the linear fitting curve are shown in Figure 9.

As can be seen in Figure 9(a), methane desorption capacity and the time when the desorption amount reached the total desorption amount for moist shale sample FY34-2 (water content is equal to 0.55 wt%) are greater than those for dry shale sample. The methane adsorption isotherm for moist shale sample is no longer as type I, and show negative adsorption, thus moisture content of shale has enormous influence on the adsorption, water reduces the adsorption amount, accordingly, it increases the desorption amount (Figure 9(b)). Figure 9(c) shows the unipore diffusion model (equation (1)) provides satisfactory fit for desorption data measured at different states and the correlation coefficient is R²>0.98. The diffusion coefficient of the dry shale sample is 0.228 × 10⁻¹² m²/s, but that of the moist one is 0.082 × 10⁻¹² m²/s, the damage rate of diffusion coefficient is 64.04%, thus, this indicates the Chang 7 Formation shale shows a medium to strong diffusion damage, the effect of water on diffusion is great and cannot be neglected.

**Discussion**

*Damage of Chang 7 Formation shale sensitivity and water block*

In the experiment of this paper, there are moderately weak velocity sensitivity, water sensitivity, and alkali sensitivity, and extremely strong stress sensitivity in the Chang 7
Formation shale, and the damage rates of cores with fracture are slightly higher than those of unfractured cores. With a high content of clay, the Chang 7 Formation shale suffers from serious stress sensitivity damage, and the irreversible stress-sensitive damage rate is over 70%. Therefore, during the fracturing stimulation treatment process, the impact of irreversible damage to fracture faces on base rock should be considered. This performance of stress sensitivity is stronger than that of the tight sandstone gas reservoir in the Sulige gas field, China (Dong et al., 2019), and similar to the observation in Longmaxi Formation shale in the Sichuan Basin, South China (Wentong et al., 2018). Alternatively, the damage rates of unfractured cores are slightly lower than those of cores with fracture. The microfractures in the Chang 7 Formation are relatively developed, and the opening width is 5 μm under natural state (Pingquan et al., 2018), so solid particles are likely to enter the microfractures and damage the reservoir.

**Figure 8.** Plane view image, 3D black-white image and colored image for cores. (a) FY 32-1, plane view image, 3D black-white image and colored image after dynamic contamination; (b) FY 24-1, plane view image, 3D black-white image and colored image after static contamination; (c) FY 24-1, plane view image, 3D black-white image and colored image before dynamic contamination; and (d) FY 24-1, plane view image, 3D black-white image and colored image after removal of 0.7 cm contaminated end.
The pore throats in the Chang 7 Formation shale are extremely small, so despite that the capillary force is particularly strong, once the water phase enters the throat, it will turn into bound water (immobile water) to effectively prevent the follow-up liquid phase from entering. The penetration depth of liquid phase in the imbibition can be calculated by the

![Figure 9. Results of water damage on gas diffusion for shale sample FY34-2. (a) Methane desorption capacity with time, (b) methane adsorption isotherm, and (c) desorption ratio with square root of time.](image)

The pore throats in the Chang 7 Formation shale are extremely small, so despite that the capillary force is particularly strong, once the water phase enters the throat, it will turn into bound water (immobile water) to effectively prevent the follow-up liquid phase from entering. The penetration depth of liquid phase in the imbibition can be calculated by the
Assuming the water uniformly imbibed into the core, the penetration depth is equal to the thickness of the cylindrical shell of the core, the shell is porous and filled with imbibed water (Figure 10(a)). Thus, the penetration depths of the liquid phase imbibition for core FY45-3 and core FY92-2 are 0.1003 and 0.0566 cm, respectively. Therefore, the penetration depth of the liquid phase imbibition is low. Since the content of water-sensitive clay minerals in the Chang 7 Formation shale is low, the water-sensitive damage is moderately weak. In addition, due to the effect of water block, the liquid phase penetration depth is limited. For the core with fractures, the results are entirely different; if we assume the fracture is a tube (Figure 10(b)) with a diameter of 5 μm (Pingquan et al., 2018), then the invasion depth is greater than 1000 m. So, the filtrate and solid particles in drill-in fluid are likely to enter the microfractures and damage the reservoir and result in an unstable wellbore. Therefore, water-based drill-in fluids can be used to replace oil-based drill-in fluids on condition that the quality of mud cake which seal fractures is ensued and request of wellbore stability is satisfied during drilling. Alternatively, in fracturing, liquid phase imbibition benefits fracture generation, which may increase the production of shale gas well (Li et al., 2017b; Shen et al., 2016). Under this condition, the effect of water imbibition on shale formation transfer from damage to stimulation.

**Performance of water-based drill-in fluids and its damage on Chang 7 Formation**

The filter loss of the P-1 water-based drill-in fluid is 3.5 ml at room temperature and is 6.5 ml at a high temperature of 85°C, which meets the requirement of the industrial standard, and the formed mud cake is thin and tough, and has good plugging performance. The rheological characteristics of water-based drill-in fluid were measured at 25°C, 50°C, and 80°C, the results show that its plastic viscosity is low and it has a good shearing force. Also, its pH value is 9, which can effectively reduce the occurrence of alkali-sensitive damage.
Furthermore, the median diameter of solid phase particles, $d_{0.5} = 11.539\, \mu m$, which is far greater than the radius of pore throat in matrix core. In conclusion, the diameters of solid phase particles in the P-1 water-based drill-in fluid are bigger than 0.1 $\mu m$, which are far above those of the pore throats in the Chang 7 Formation shale, so the solid phase particles in the drill-in fluid will not enter the pore throat and cause blocking damage. Also, because of the water block effect, the liquid phase penetration depth of the water-based drill-in fluid into the reservoir is relatively shallow, causing limited water-sensitive and water-blocking damage.

Furthermore, the water-based drill-in fluid does slightly greater damage to the Chang 7 Formation shale in static conditions than in dynamic conditions. The permeability can be restored to more than 80% after 1 cm of the contaminated core end by the drill-in fluid is cut off, indicating that the overall damage of water-based drill-in fluid to the Chang 7 Formation shale is limited and the damage depth is less than 1 cm. In addition, as the existence of mud cake can greatly reduce the damage of solid phase and liquid phase in the drill-in fluid to the reservoir, the damage caused by drill-in fluid can be further reduced by improving the quality of mud cake and controlling fluid loss performance. Therefore, the water-based drill-in fluid and the Chang 7 Formation shale reservoir are quite compatible, so the P-1 water-based drill-in fluid can meet the requirement of Chang 7 Formation shale damage controlling in drilling; furthermore, the effect of water-based drill-in fluid on wellbore stability should be paid more attention. Besides, in this study only one drill-in fluid sample was used, other drill-in fluid with different formula was not involved, for example, the effects of formate content and salts concentration on the damage, as done by Davarpanah et al. (2019), which will be investigated in the future.

**Evaluation of water damage on gas diffusion**

In this work, the diffusion coefficient is much less for moist shale sample than the dry one; the damage rate is 64.04%. The diffusion coefficient of the rock sample decreases with the presence of water; similar results have been found in studies about coalbed methane. For instance, Pan et al. (2010) observed that moisture in coal matrix would cause coal swelling/shrinkage, and the impact of moisture content on the diffusion is strong, which changed in pores with different size; Xu et al. (2015) noted the diffusion coefficients of dry coal samples are higher than those of the samples saturated with water; the moisture reduced the gas adsorption amount and effect its concentration gradient in coal. For shales, Yuan et al. (2014) found that macropore and micropore diffusion coefficients of shale samples were all significantly reduced with the presence of water; Lyu et al. (2015) investigated the influence factors of shale swelling, and shown that water adsorption creates higher swelling volume in shale matrix. It seems that the pore radius is reduced by the adsorbed water molecule layer, and hydration and expansion of clay can account for this result. But all of those works did not investigate the damage of working fluids and its additives on the diffusion, and the relationship between the diffusion damage and the permeability damage was not clear. Thus, as done in this work, the evaluation criteria of diffusion damage can refer to that of formation sensitivity formation, and the diffusion coefficient considering the presence of water can be used to calculate the apparent permeability, which was a concept proposed first by Javadpour (2009) and was modified by introducing the adsorption and diffusion and slippage effect in our previous study (Rui Wang, 2013).
As mentioned above, the effect or damage of working fluid on shale gas production has multi-mechanism and spatial and time multi-scale, which is also a result caused by combining the damages of the formation sensitivity, the water block, working fluid, and gas desorption and diffusion. Therefore, further research is needed to establish the damage evaluation system of working fluid considering the multi-mechanism and multi-scale mass transfer process of shale gas.

Conclusions
In this study, a series of mineral compositions and physical properties test, and assessments of fluid and stress sensitivity damage, water block damage, water-based drill-in fluids damage, and water damage to gas diffusion on 20 shale samples obtained from Chang 7 of Mesozoic Yanchang formation were conducted to analyze the damage characteristic and mechanisms. The work reveals some special law and finds. The most significant conclusions are listed as below: First, in the nonmarine Chang 7 Formation shale, there is extremely strong stress sensitivity and moderately weak water sensitivity damage. Although the liquid phase invasion depth is shallow and the water block damage is limited, the liquid phase and solid particles would enter the microfractures in the reservoir. Second, the P-1 water-based drill-in fluid is compatible with the Chang 7 Formation shale reservoir, which can meet the requirement of Chang 7 Formation shale damage controlling, the effect of water-based drill-in fluid on wellbore stability should be paid more attention. Third, the diffusion coefficient of the shale decreases with the presence of water. A systematic damage evaluation method of working fluid considering the multi-mechanism and multi-scale mass transfer process of shale gas is needed to establish.

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