Experimental study on the physical and chemical properties of the deep hard brittle shale

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Summary In the hard brittle shale formation, rock composition, physical and chemical properties, mechanics property before and after interacting with fluid have direct relation with borehole problems, such as borehole wall collapse, mud loss, hole shrinkage. To achieve hard brittle shale micro-structure, physical–chemical properties and mechanics property, energy-dispersive X-ray diffraction (XRD), cation exchange capacity experiment and hardness test are conducted. The result of laboratory experiments indicates that, clay mineral and quartz is dominated in mineral composition. In clay mineral, illite and illite/semectite mixed layers are abundant and there is no sign of montmorillonite. Value of cation exchange capacity (CEC) ranges from 102.5–330 mmol/kg and average value is 199.56 mmol/kg. High value of CEC and content of clay mineral means hard brittle shale has strong ability of hydration. The image of XRD shows well developed micro-cracks and pores, which make rock failure easily, especially when fluid invades rock inside. Shale sample soaked with anti-high temperature KCL drilling fluid on shorter immersing time has stronger strength, whereas shale sample soaked with plugging and film forming KCL drilling fluid on longer immersing time has stronger strength.

Introduction

Wellbore instability has negative effect on drilling operation and cementing quality, pollutes reservoir and triggers many borehole accidents. It dramatically increases cost of drilling and jeopardizes development and exploration of oil and gas.

A wellbore instability problem in shale formation includes borehole wall collapse, plastic shrinkage and fracture leakage (Deng et al., 2008). Wellbore instability is a problem combined with mechanics and chemical factor. When drilling fluid interacts with hard brittle shale, physical–chemical reaction occurs, which causes clay hydration, decreases strength and changes stress balance in formation, eventually leading to wellbore instability. Not like clay shale

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formation, hydration swelling is not only main reason of wellbore instability in hard brittle shale (Wang et al., 2012). Many researches demonstrate, cracks propagation made by hydration and fluid invasion give rise to collapsing in hard brittle shale formation.

Micro-structure, physical and chemical properties of hard brittle shale are the fundamental parts of wellbore instability mechanism. To have deep understanding of those rock properties, a great number of researches have been done. Shi and Xia (2011) presented an analytical technology of digital core from CT image to observe the change of microstructure of hard brittle shale in hydration process. The CT image clearly shows crack propagation phenomenon. Based on diffuse electric double layer theory and Van der Waals theory, Liu et al. (2010) had research of influence of hydration on strength of clay shale, which proves that hydration swelling is the key role of wellbore instability in clay shale formation. Wu and Liu (2007) conducted an experiment to test shale acoustic time before and after soaking fluid. The results show acoustic time increases after soaking fluid, which means fluid have obvious effect on shale structure. By analyzing characteristic of physical and chemical properties of hard brittle shale of Weizhou 2 Member in Weizhou 12-1N oilfield, Yue et al. (2005) suggests film forming drilling fluid with strong sealing ability is most effective measure to prevent borehole collapse. Zhao et al. (2007) performed laboratory studies on hard brittle shale. They conclude that drilling filtrate invasion along micro-crack makes clay mineral hydration and strength reduction, and claim that this invasion is the main factors of causing wellbore instability. Therefore, they propose plugging material matched with pore throat radius should be added into drilling fluid to reduce fluid loss. Wang et al. (2011) analyzed impact of distribution of cracks on shale strength. The different crack distribution leads to different rock strength. Lu et al. (2012) analyzed influence of wettability on crack propagation in hard brittle shale in mechanical aspect. Liu et al. (2014) did similar research. Both of them think wettability have relation with shale strength and wellbore instability due to its effect on crack propagation. Based on that, Liang et al. (2015) take into consideration hydration, wettability, time and size effect to analyze crack propagation mechanism.

In one oilfield, hard brittle shale section is between 3000 m and 5000 m, where serious borehole problems occur. Therefore, we did studies of mineral composition, microstructure, physical and chemical properties of hard brittle shale in this section. besides, mechanics experiment was applied to investigate the mechanics property before and after soaking drilling fluid. From those works, we have deep understanding on mechanism of wellbore instability in hard brittle shale formation in this oilfield.

**Physical—chemical characteristics**

Shale composition and structure means position and geometry of all components, normally are used to describe distribution of particle, crystal and cement in rock (Zhang et al., 1999). Mineral composition, type and content of clay mineral, micro-structure, and physical—chemical property are all able to influence shale mechanics property. So the study of composition and structure is necessary for knowing wellbore instability mechanism.

**Mineralogy compositions and micro-structure**

Energy-dispersive X-ray diffraction was utilized to obtain mineral composition and clay component. Samples come from 5 areas in hard brittle shale of Dongying formation. For each area, 5 samples were analyzed by XRD experiment. Results are presented in Figs. 1 and 2. The samples contain abundant clay mineral and quartz, a little orthoclase, calcite and dolomite. Clay mineral content averaged 40%, are over 50% in area 2 and 4, where sticking of drilling tools happens more frequently.

As shown in Fig. 2, main content of clay mineral composition is illite and illite/semecite mixed layers and illite content averaged 44% and illite/semecite mixed layers content averaged 40%, whereas montmorillonite is not observed.

It is noted that composition in 5 areas is different from each other, which means shale composition is different in transverse distribution. But all of hard brittle shale in Dongying formation is mainly composed of quartz and clay mineral and also its composition of clay mineral are dominantly illite and illite/semecite mixed layers.

Rock micro-structure includes orientation arrangement of clay mineral, development and distribution of bonding structure, micro cracks and pore. Those structures play a key role in drilling fluid optimizing.

**Micro-structures**

In this paper, SEM was conducted to observe micro structure of shale sample. From Figs. 3 and 4, micro cracks and micro pore are well-developed in Dongying shale formation and clay mineral is schistose in orientation arrangement.
The existence of well-developed micro-cracks damages rock integrity and reduces strength. Besides, in the drilling operation, crack can be a space for fluid invasion. When fluid invade into shale formation along with cracks, cohesion between cracks will decrease and hydraulic wedge effect occurs, leading to wellbore instability and formation fracturing. According to logging datum and well history, during drilling operation in Dongying formation, collapsing and breaking situation occur frequently. Field engineer tried to increase fluid density to stabilize borehole, but it didn’t work out. Because increasing fluid density enhances fluid invasion along cracks and improves crack propagation, which exacerbates wellbore instability. Based on that, for this kind of formation, increasing fluid density is not first option but improving sealing ability of fluid is priority for wellbore stability.

Physical-chemical properties

Cation exchange capacity is an effective reflect of shale hydration ability. There are so many ways to obtain shale cation exchange capacity, like ammonium acetate test, methylene blue test and barium chloride-sulfuric acid test.

In this paper, cation exchange capacity is achieved by methylene blue test. The formula is shown as the following:

$$\text{CEC} = \frac{a}{b} \times 10$$  \hspace{1cm} (1)

where the CEC is cation exchange capacity, $a$ is the volume of methylene blue, $b$ is the weight of shale.

The result of methylene blue test is shown as Fig. 5. Through formula (1), value of cation exchange capacity can be obtained, as presented in Fig. 6. The value of CEC ranges from 102.5–330 mmol/kg and averaged 199.56 mmol/kg. The value is high, indicating strong hydration corresponding to high content of illite/smectite. Value of CEC is different in transverse distribution, which is consistence with composition difference in transverse distribution.

After soaking water, physical-chemical change of shale sample can reflect hydration capacity. Therefore, immersion test was applied to investigate influence of water on shale. The picture of shale sample before and after soaking water is shown in Fig. 7. In the beginning, shale samples have no change in the surface. After soaking 1 h, fracture appears.
in the surface. After 4 h, lots of fracture occur and connect with each one, leading sample to break along those fractures. Meanwhile, even soaked in water, shale sample have no sign of softening or deformation.

Based on above experiments, we conclude that there are 2 reasons of shale failure after soaking water. First reason is its micro-structure. Existence of numerous cracks and uneven bedding plane make water easily invade into shale formation. In addition, when water invades inside, capillarity effect can cause stress concentration at crack tip, leading to crack propagation. Secondly, High value of CEC and clay content demonstrate shale sample has strong hydration capacity. Therefore, when it contacts with water, hydrate swelling cause to rock failure.

**Mechanics property**

To investigate the influence of different drilling fluid on shale mechanics, hardness test was applied. First of all, without soaking fluid, we test shale samples in Dongying formation from 5 areas. After that, shale samples soaked with different drilling fluid were tested.

Two types of drilling fluid system were used in this test, anti-high temperature resistant KCL drilling system, plugging and film forming KCL drilling system. The condition for soaking experiment is that pressure is 3 MPa, temperature is 100 °C and soaking time is 48 h and 72 h.

The hardness of shale sample in 5 areas before soaking is presented in Fig. 8. Value of hardness in area 5 is highest, followed by area 1. The values of areas 4 and 3 are lowest. So in different areas, shale hardness varies, which indicates its strong anisotropy in transverse distribution.

It can be seen from Fig. 9 that shale sample soaked by the fluid becomes weak with increase of soaking time, and shale sample soaked by anti-high temperature drilling fluid on shorter immersing time has stronger strength than that soaked by plugging and film forming drilling fluid. In addition, with the increase of immersing time, the decreasing amount of the strength of shale sample soaked by anti-high temperature drilling fluid is bigger. However, the decreasing
amount of the strength of shale sample soaked by plugging and film forming drilling fluid is smaller. The results show that shale sample soaked with anti-high temperature KCL drilling fluid on shorter immersing time has stronger strength, whereas shale sample soaked with plugging and film forming KCL drilling fluid on longer immersing time has stronger strength.

Conclusion

1) Hard brittle shale in Dongying formation is mainly composed of quartz and clay mineral. In clay mineral, illite and illite/seemctite are predominant and montmorillonite is not seen. Clay mineral is schistose in orientation arrangement.
2) In this formation, bedding plane, micro-cracks and micro-pore are rich, which are good path for fluid invasion. Therefore, capacity of sealing and controlling water loss is the first consideration for drilling fluid. Keeping fluid from invading along with crack and pore is key point for wellbore stability.
3) From analyzing and comparing strength of shale sample soaked 2 types of drilling fluid, we find that shale sample soaked with anti-high temperature KCL drilling fluid on shorter immersing time has stronger strength, whereas shale sample soaked with plugging and film forming KCL drilling fluid on longer immersing time has stronger strength.

Conflict of interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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References

Yue, Q., Li, Y., He, B., et al., 2005. Research on the hard brittle shale and drilling fluid type in WZ-12-1N oilfield. China Offshore Oil Gas 17 (1), 44—47.