A new drilling fluid system for plugging argillaceous shale reservoirs

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Abstract. Argillaceous shale reservoir development is mainly based on extended reach horizontal wells, and has complex geological characteristics. In the process of long horizontal drilling, wellbore instability is easy to occur. In this paper, the mechanism of argillaceous shale instability is explored by testing the mineral composition of argillaceous shale. The method of plugging argillaceous shale with nano-sio₂ is proposed, and a new nano plugging drilling fluid formulation system is constructed. The system mainly includes specially designed fluid loss additive SFL and EY, flow pattern regulator VBS, lubricant RH, bentonite, nano silica and inorganic salt. Through a series of experimental tests, the rheological property, thermal stability, anti pollution and plugging ability of the system were evaluated. Finally, with the help of SEM, the micro analysis of core profile before and after plugging with nano drilling fluid is carried out, and the plugging mode of nano drilling fluid is further elaborated. The comprehensive results show that nano-sio₂ has good compatibility with water-based drilling fluid, and has good effect on inhibiting argillaceous shale expansion and plugging argillaceous shale fractures. It can play the role of stabilizing shale, effectively meet the needs of target block, and provide reference for the follow-up study of plugging argillaceous shale reservoir.

1. Introduction
With the growth of global energy demand and many problems of existing oil and gas resources, energy problem has become a problem faced by many countries. As an unconventional energy, shale gas has been paid more and more attention by various countries. Vigorously developing shale gas plays an important role in alleviating energy pressure. With the increase of shale gas production depth, shale gas reservoir gradually evolved from shale reservoir to argillaceous shale reservoir [1]-[4]. Argillaceous shale is a transitional form between argillaceous rock and shale. Compared with shale, it has deeper burial depth, higher clay content, more microfractures and more abundant shale gas reserves [5]. At present, a large number of researchers have developed various kinds of drilling fluid systems for shale reservoirs, but there are few reports on drilling fluid systems for argillaceous shale reservoirs. Argillaceous shale formation has obvious bedding, developed microfractures and strong water sensitivity. In the process of drilling in long horizontal section, it is easy to fall off and collapse. On the other hand, the horizontal section of argillaceous shale well is relatively long, which further increases the probability of wellbore instability and formation collapse. Therefore, in the process of drilling argillaceous shale horizontal wells, to solve the problem of wellbore instability has become the key to the selection and design of drilling fluid. Oil based drilling fluid system is often used to drill extended reach horizontal wells in argillaceous shale reservoir, but oil-based drilling fluid usually faces the problems of environmental pollution and high cost, while water-based drilling fluid does not contain base oil, so it has natural advantages in environmental protection [6]. Therefore, it is necessary
to analyze the causes of argillaceous shale wellbore instability and find a solution for drilling argillaceous shale reservoir with water-based drilling fluid.

Compared with the nanometer size of argillaceous shale, the size of plugging materials in drilling fluid, such as bentonite, barite, film-forming plugging agent and calcium carbonate, is generally more than micron [7]. The pore throat diameter of shale is between 30 ~ 5000nm, and the permeability is low. The traditional drilling fluid additives can not effectively block the micro pores of argillaceous shale, so it can not prevent the free water in the drilling fluid from invading the argillaceous shale [8]-[12] Based on the analysis of physical and chemical properties and wellbore instability mechanism of argillaceous shale, this paper proposes a method of plugging micro fractures in argillaceous shale with nano water-based drilling fluid. A set of drilling fluid suitable for argillaceous shale reservoir is constructed, and a lot of experimental analysis is carried out, which provides a scientific basis and theoretical basis for solving the stability of argillaceous shale reservoir, and has high engineering application value.

2. Experiment

2.1. Materials

Four core samples are taken from Fuling argillaceous shale reservoir, with reservoir depth of 2564m ~ 3046m; chemical substances such as bentonite, NaOH, Na₂CO₃, HCOONa and KCl are purchased from Wuhan Tianli Biochemical Technology Co., Ltd.; drilling fluid additive VBS is a flow pattern regulator, and its main component is a polymer obtained by chemical modification, which is used to increase the viscosity and shear of drilling fluid; SFL is a fluid loss reducer, which is mainly composed of a new type of modified starch. It can form dense mud cake on the surface of wellbore and prevent free water in drilling fluid from invading deep formation; EY is also a fluid loss reducer, but it is a modified cellulose, which can be used together with SFL to minimize the filtration of drilling fluid; RH is a lubricant, and its main component is polyol, which forms particulate emulsion in drilling fluid through cloud point effect They are all provided by Jiahua Technology Co., Ltd. The nano materials used are composed of three different sizes of nano silica, and their mass ratio is 1:1:1. The parameters of three kinds of nano silica are shown in Table 1.

| Material type | Average particle size (nm) | Apparent density (g/cm³) |
|---------------|---------------------------|-------------------------|
| NP-SiO₂-A     | 30~60                     | 0.15~0.18               |
| NP-SiO₂-B     | 100~200                   | 0.18~0.2                |
| NP-SiO₂-C     | 300~500                   | 1.8~2.2                 |

2.2. Experiment method and formula

The preparation methods of drilling fluid in this study are in accordance with "GB / T 16783.1-2006 / ISO 10414-12001 and SY / T 7377-2017". The basic drilling fluid formula is as follows: Water+3%Bentonite+0.2%NaOH+0.2%Na₂CO₃+40%HCOONa+5%KCl+1%VBS+2%SFL+0.5%EY +3%RH+10%Nano plugging agent (1.3g/cm³)

3. Results and discussion

3.1. Core mineral analysis

Argillaceous shale is a kind of laminated rock with transition form from argillaceous rock to shale. In the process of deposition, it is easy to form imperfect texture and foliation. In the process of geological tectonic movement, argillaceous shale swells with the increase of burial depth, which leads to certain cracks in its structure. Therefore, argillaceous shale generally has the characteristics of large specific surface area, small pores, complex structure, developed microfractures and easy to absorb water and expand. The argillaceous core of shale gas well in Fuling block is analyzed by XRD, and the composition and content of clay minerals and non clay minerals are analyzed, so as to provide valuable information for us to explore the causes of shale instability. The analysis results are shown in Figure 1.
From the geological point of view, it is generally believed that the higher the content of clay minerals in rock, the stronger its hydration expansion; the higher the content of quartz, the stronger its hard brittleness. According to the analysis of argillaceous shale mineral composition, the argillaceous shale minerals in Fuling block are mainly clay and quartz, followed by plagioclase and dolomite, and a small amount of potash feldspar, calcite and pyrite. The content of quartz ranges from 34% to 51%. The high content of hard brittle quartz makes argillaceous shale hard brittle, which adds many factors to argillaceous shale instability. The distribution of clay minerals is between 22% and 47%. The clay minerals are mainly mixed layer and illite, without montmorillonite. The content of hydration dispersion and expansive clay minerals is low. It can be concluded that the hydration dispersion and expansion ability of argillaceous shale are weak, and the main way of wellbore instability is spalling. Therefore, it is necessary to slow down the cracking and shedding of argillaceous shale, so as to improve the wellbore stability.

3.2. Nano silica plugging argillaceous shale

The pore throat and microfracture size of argillaceous shale are generally between tens of nanometers and hundreds of nanometers. Most of the hard brittle argillaceous shale which is easy to cause wellbore instability contains closed or open bedding and microfracture, and the capillary force is strong. Under the action of downhole positive pressure difference, the free water in drilling fluid is easy to invade the formation, leading to the expansion and cracking of argillaceous shale along the primary fractures and bedding. With the continuous invasion of free water, the fractures continue to develop and expand, eventually leading to wellbore collapse. The particle size of nano materials is generally between 1 ~ 100nm, which is matched with the size of micro pores of argillaceous shale, and can enter into the micro cracks of argillaceous shale for better plugging. Nano silica can be divided into surface modification and no surface modification. The surface modified nano-SiO$_2$ can disperse well in solution, but it is expensive and not suitable for oilfield chemistry. The unmodified nano-SiO$_2$ is cheaper, but its dispersibility is poor. On the other hand, a single size of nano materials is difficult to meet the needs of different sizes of cracks, so we use ultrasonic vibration instrument to evenly disperse three different sizes of nano silica in aqueous solution to make nano solution. The inhibition effect of different concentrations of nano solution on argillaceous shale expansion is different. The linear expansion rate experiment is adopted to investigate the inhibition effect of different concentrations of nano solution. The experimental results are shown in Figure 2.

It can be seen from the above figure that before 4h, the concentration of nano solution has little effect on the linear expansion rate of argillaceous shale. After 10h, the argillaceous shale expansion rate of clean water is significantly higher than that of nano solution, which indicates that nano solution can significantly inhibit the hydration expansion of argillaceous shale. The linear expansion rate of argillaceous shale has a positive correlation with the immersion time of argillaceous shale, and a negative correlation with the concentration of nano solution. After 48 hours, the expansion rate of
argillaceous shale in each group basically approaches the fixed value. The higher the concentration of nano solution, the stronger the inhibition and the lower the argillaceous shale expansion rate. However, when the concentration of nano particles reaches 60%, the inhibition tends to the maximum, which has little relationship with the concentration. This may be because the volume of microfracture space in argillaceous shale is limited, and excessive nano particles will not enter the microfracture. Therefore, the linear expansion rate of argillaceous shale with 80% and 60% concentration of nano solution has little difference. Considering the preparation cost of nano solution, 60% concentration of nano solution is selected as the plugging agent of shale in the follow-up test of this study.

![Figure 2](image)

*Figure 2. Inhibition of nano solution with different concentrations*

3.3. Performance evaluation of nano drilling fluid

4. Rheological properties

The dosage of any kind of drilling fluid treatment agent in drilling fluid is limited to a certain extent. It is not that the more the dosage is, the better the performance is. However, if the dosage is too small, the performance of drilling fluid can not be improved correspondingly. Generally, the dosage of additives should not only meet the requirements of drilling fluid performance, but also ensure the principle of minimum cost. Therefore, in order to determine the optimal dosage of nano solution in drilling fluid, the effects of different dosage of nano solution from 0% to 30% on the basic properties of drilling fluid were investigated. The experimental results are shown in Table 2.

| Dosage(%) | Status | AV(mPa·s) | PV(mPa·s) | YP(Pa) | Φ6/Φ3 | FL_API(ml) | pH |
|----------|--------|----------|----------|--------|-------|-----------|----|
| 0        | B-R    | 23       | 14       | 9      | 5/3.5 | 6.2       | 9.6|
|          | A-R    | 22       | 14       | 8      | 7/6   |           |    |
| 5        | B-R    | 22       | 14       | 8      | 4.5/3.5 | 6.0     | 9.8|
|          | A-R    | 24       | 15       | 9      | 10.5/8.5 |       |    |
| 10       | B-R    | 23.5     | 15       | 8.5    | 6/5   |           |    |
|          | A-R    | 27       | 17       | 10     | 10/8.5 | 5.6      | 9.8|
| 15       | B-R    | 25       | 16       | 9      | 7/5.5  | 5.8      | 10.1|
|          | A-R    | 29       | 18       | 11     | 11/9   |           |    |
| 20       | B-R    | 28       | 17       | 11     | 8/6    | 5.6      | 10.2|
|          | A-R    | 33       | 21       | 12     | 11/10  |           |    |
| 25       | B-R    | 35       | 13       | 12     | 10/9   | 5.2      | 10.2|
|          | A-R    | 40       | 25       | 15     | 14/12.5 |         |    |
| 30       | B-R    | 37       | 22       | 15     | 15/13.5 | 4.8     | 10.4|
|          | A-R    | 41       | 23       | 18     | 16/15.5 |         |    |

*B-R—Before rolling; A-R—After rolling
It can be seen that the viscosity and water loss of nano drilling fluid increase with the increase of nano API content. It shows that the nano solution has a certain thickening and filtration reducing effect, and the nano material itself is neutral, does not react with water-based drilling fluid, does not cause pH change, and has good compatibility with water-based drilling fluid. However, when the dosage of nano solution exceeds 20%, the viscosity and shear force of drilling fluid increase sharply. In the process of tripping, the drilling fluid with too high viscosity and shear force is easy to produce greater exciting pressure and suction pressure, which leads to wellbore instability. Considering the performance of drilling fluid and the preparation cost of nano solution, the recommended dosage of nano solution is 10%.

5. Thermal stability
With the increase of formation depth, the formation temperature of argillaceous shale formation increases gradually. Therefore, in drilling operation, with the increase of well depth, the temperature resistance of nano drilling fluid is also increasing. The depth of argillaceous shale reservoir is generally between 2000 ~ 3600m, and the reservoir temperature is between 80 ℃ and 130 ℃. The dosage of nano material is controlled at 10% in laboratory. The influence of temperature on nano water-based drilling fluid is investigated by setting different aging temperatures. The results are shown in Table 3.

| Temperature(℃) | Status | AV(mPa·s) | PV(mPa·s) | YP(Pa) | Φ6/Φ3 |
|---------------|--------|-----------|-----------|--------|-------|
| 80            | B-R    | 22        | 14        | 8      | 4.5/3.5 |
|               | A-R    | 24        | 15        | 9      | 10.5/8.5 |
| 100           | B-R    | 23        | 15        | 8      | 6/5    |
|               | A-R    | 26        | 15        | 11     | 10/9   |
| 120           | B-R    | 24        | 15        | 9      | 7/5.5  |
|               | A-R    | 28        | 16        | 12     | 11/9.5 |
| 140           | B-R    | 26        | 16        | 10     | 8/5.5  |
|               | A-R    | 25        | 16        | 9      | 9/8    |

A complete statistics of drilling fluid performance is made when the temperature changes between 80 ℃ and 140 ℃. Through comparative analysis, we can find that. At the same temperature, the performance of drilling fluid before and after aging has little change. With the increase of temperature, the rheological value fluctuates in a small range, and the maximum variation of single property is less than 5%. It shows that nano materials basically do not affect the temperature resistance of nano water-based drilling fluid, which is also in line with the properties of nano materials.

6. Anti pollution
In the process of argillaceous shale drilling, the drilling fluid contacts with the rock wellbore for a long time. It is inevitable that part of rock debris will dissolve in drilling fluid, and this part of foreign matter belongs to pollutants relative to drilling fluid. Therefore, when drilling argillaceous shale formation, the requirement of anti pollution of drilling fluid is relatively high. By adding 5% of different types of pollutants into nano drilling fluid, the pollution process of drilling fluid was simulated, and the anti pollution ability of nano drilling fluid was investigated. The experimental results are shown in Table 4.

Compared with the blank group, the addition of pollutants will pollute the basic properties of drilling fluid, and will not affect the pH of drilling fluid. The addition of pollutants results in the increase of viscosity and water loss of drilling fluid. The pollution ability of different kinds of pollutants is different. The pollution effect of bentonite is the strongest and that of inorganic salt is the weakest. However, even in the case of the most serious pollution, the drilling fluid shear can still be maintained between 8 ~ 12Pa, which can fully meet the requirements of drilling fluid suspended cuttings. In general, nano drilling fluid has strong anti pollution ability and great application potential.
Table 4. Anti pollution performance of nano drilling fluid

| Contaminants | Status | AV(mPa·s) | PV(mPa·s) | YP(Pa) | φ6/φ3 | FLAPI(ml) | pH  |
|--------------|--------|-----------|-----------|--------|-------|-----------|-----|
| Nothing      | B-R    | 23        | 14        | 9      | 5/3.5 | 6.2       | 9.6 |
|              | A-R    | 22        | 14        | 8      | 7/6   |           |     |
| Debris       | B-R    | 28        | 17        | 12     | 8/6.5 | 6.8       | 9.5 |
|              | A-R    | 24        | 15        | 10     | 10/8  |           |     |
| Bentonite    | B-R    | 30        | 18        | 12     | 9/7.5 | 7.4       | 9.6 |
|              | A-R    | 27        | 17        | 10     | 10/9  |           |     |
| NaCl         | B-R    | 23        | 16        | 7      | 5/4   | 6.4       | 9.6 |
|              | A-R    | 25        | 16        | 9      | 8/7   |           |     |
| MgCl₂        | B-R    | 23        | 15        | 8      | 5/3.5 | 6.0       | 9.4 |
|              | A-R    | 24        | 16        | 8      | 8/7   |           |     |
| CaSO₄        | B-R    | 24        | 17        | 7      | 6/4.5 | 6.2       | 9.5 |
|              | A-R    | 25        | 15        | 10     | 10/9  |           |     |

7. Plugging property

Argillaceous shale is the rock type with the smallest pore size in sedimentary rocks, and nano pores make plugging extremely difficult. The particle size of conventional plugging materials is generally in the range of micron size, which can only plug micron pores, but can not solve the problem of nano pores. Micron scale materials are often accumulated outside the argillaceous shale fractures, but not deep into the fractures, which can not achieve the plugging effect. Nano materials can only be used to plug micro pores in argillaceous shale. By measuring the pressure changes at the front and back ends of argillaceous shale, the plugging effect of nano drilling fluid on argillaceous shale is evaluated. This study has a good guiding role in evaluating the plugging performance of argillaceous shale, and can help solve the problem of wellbore instability in the process of argillaceous shale drilling. The argillaceous shale inlet pressure is set at 3.5Mpa, and the experimental results are shown in Figure 3.

Figure 3. Plugging performance of nano drilling fluid

It can be seen from the curve in the figure above that the plugging ability of clean water to argillaceous shale is poor, and the outlet pressure is close to 3.5Mpa after about 20h. It shows that under the condition of clean water plugging, the pressure of the front section of clean water passes through the argillaceous shale about 20 hours, and the clean water loses its plugging effect. The water-based drilling fluid itself contains a large number of plugging substances. When the water-based drilling fluid is used to plug argillaceous shale, the final outlet pressure of shale is only 2.3Mpa; when the nano water-based drilling fluid is used to plug argillaceous shale, the final outlet pressure of
argillaceous shale is only 1.5MPa, compared with the water-based drilling fluid, the plugging performance is improved by 34.8%. After the nano materials enter the drilling fluid, the micro fractures of argillaceous shale are effectively blocked, and the transmission of argillaceous shale inlet pressure to outlet pressure is retarded, which indicates that the nano water-based drilling fluid can improve the stability of argillaceous shale.

8. **Analysis of core micro morphology**

In order to further describe the plugging mode of nano materials, we selected the core profile before and after plugging with nano drilling fluid for SEM analysis, and the plugging time of nano drilling fluid was 48 h. The experimental results are shown in Figure 4.

![Figure 4](image_url)

**Figure 4.** Micro morphology of argillaceous shale

Figure 4 (a) shows the micro morphology of argillaceous shale before plugging with nano drilling fluid. It can be seen from Figure 4 (a) that the mineral bedding of argillaceous shale is obvious, and the micro fractures between different mineral particles are developed, which is not completely dense cementation. It is because of abundant fractures in argillaceous shale that natural gas can be stored in argillaceous shale. However, in the process of shale reservoir exploitation, this kind of microfracture is easy to combine with drilling fluid, resulting in further expansion and amplification of fractures. Finally, it will lead to wellbore instability, and even worse, it will cause the whole well to be scrapped. Therefore, in the process of drilling, it is necessary to add special materials in the drilling fluid to plug these fractures, so as to ensure the safety of the drilling process. Figure 4 (b) shows the micro morphology of argillaceous shale after plugging by nano drilling fluid. It can be seen from Figure 4 (b)
that other solid materials in nano drilling fluid are plugged on the fracture surface, but it is difficult to plug nano fractures because their particle diameter is generally larger than 1 μm. However, nano-SiO$_2$ with different sizes basically gathered at the crack edge. Some nanoparticles even enter into the fracture to repair part of the fracture, shorten the width and length of the fracture, and improve the wellbore stability of argillaceous shale.

9. Conclusions

1. The argillaceous shale reservoir is rich in microfractures, and the clay mineral content is between 22% and 47%. There is no montmorillonite, the hydration expansion ability is weak, and the main way of wellbore instability is spalling. Through a large number of evaluation experiments, the optimal concentration of nano solution is 60%, and the optimal dosage of nano solution in drilling fluid is 10%.

2. The nano drilling fluid system has good rheological properties between 80 °C and 130 °C. And it can resist the pollution of debris, bentonite and inorganic salt. When the inlet pressure is 3.5MPa, the outlet pressure of argillaceous shale sealed with ordinary drilling fluid is 2.3MPa, and the outlet pressure of argillaceous shale sealed with nano drilling fluid is only 1.5MPa. Nano drilling fluid can effectively seal micro fractures in argillaceous shale.

3. The solid particles in common drilling fluid are micron level, most of which are attached to the fracture surface, so it is difficult to plug nano level fractures. Nano silica in nano drilling fluid can enter nano fractures, and improve the stability of argillaceous shale by shortening the width and length of fractures.

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