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Performance Evaluation and Field Application of Nano Plugging Agent for Shale Water-Based Drilling Fluid

Minjia Jing [1,2], Zhiping Yuan [1,2], Xiaoyang Li [3], Jinjun Huang [4,*] and Yuexin Tian [4]

1 Drilling and Production Engineering Technology Research Institute, Chuanqing Drilling Engineering Co., Ltd., Guanghan 618300, China
2 Sichuan Key Laboratory of Oil and Gas Field Applied Chemistry, Guanghan 618300, China
3 Chuanqing Drilling Engineering Co., Ltd., Chengdu 610500, China
4 State Key Laboratory of Oil & Gas Reservoir Geology and Exploitation, Southwest Petroleum University, Chengdu 610500, China
* Correspondence: huangjjswpu@163.com; Tel.: +86-158-2819-7882

Abstract: In this paper, nano plugging agent AMPS/AM was prepared, and its plugging performance was evaluated by a microfracture simulation experiment and a shale pressure resistance experiment. The pressure loss decreased by 66.09% compared with the top pressure of 6.9 MPa, and the average core indentation hardness increased 58.12% with 3% AMPS/AM blocking mud. The experiments indicate that AMPS/AM can effectively seal the shale micropore and nanopore structure and greatly improve the stability of fractured shale well wall. The field application results of the YS129H well in the Zhaotong block show that the water-based drilling fluid with nano plugging agent AMPS/AM as the key agent has strong plugging performance. The well mud is of high temperature and pressure water loss < 4 mL, mud cake permeability reduction rate of 85.67%, which indicates that the drilling fluid system has good sealing properties and well wall stability.

Keywords: nano plugging agent; long horizontal section horizontal well; water-based drilling fluid; well wall stabilization

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1. Introduction

During the drilling process of water-based drilling fluids in shale reservoirs, the drilling fluid filtrate will invade into the shale interior along the weak surface structures such as microfractures and laminae under the action of capillary force and pressure difference, leading to the occurrence of a hydration swelling phenomenon, which reduces the rock's own strength and leads to well wall destabilization accidents such as shale spalling and falling blocks [1–5]. In addition, the shale has very low porosity and permeability (10^{-12}~10^{-6} μm^2), and a very small amount of filtrate entering the shale can significantly increase the pore pressure around the well wall, weakening the effect of fluid column pressure on the effective mechanical support of the well wall and leading to the occurrence of well wall instability [6,7]. Therefore, sealing the nanopore fractures can prevent the intrusion of the surrounding fluid phase and achieve the suppression of hydration swelling in the shale and long-term stability of the well wall [8–10]. During the deep development of shale gas formations, water-based drilling fluids are required to have excellent plugging properties for frequent well wall destabilization [11]. Baker Hughes and developed a nano-sized plugging polymer, MAX-SHIELD, with certain deformability [12], which can form a dense protective film on the inner surface of the wellbore wall with good plugging effect when added at 3% in water-based drilling fluids. Del Gaudio L [13] developed a particle size concentration of 30–80 nm by using a homogeneous method to perform nano paraffin emulsion with particle size concentration of 30–80 nm. Spisak B J [14] found that nanoparticle NPS had good sealing effect on Atoka Shale pore throats and fractures. In pressure transfer experiments, 5% nanoparticle NPS added to the foam drilling fluid was able to rapidly reduce the permeability of the shale. Wang Xumei et al. [15] synthesized a polymeric nanotreater PNP, a spherical nanomaterial with a core–shell structure with a particle size of 375 nm. This polymeric nanoparticle treatment agent can reduce the base slurry filtration loss by 60%, the shale swelling rate by 70%, the extreme pressure lubrication coefficient by more than 75%, and the temperature resistance up to 220 °C, while it has no effect on the drilling fluid rheology and has good compatibility with other drilling fluid additives. Wael EL Sherbeny [16] et al. prepared a new polymer nanoparticle and experimentally showed that the best plugging effect was achieved at 2–4% addition in water-based drilling fluid and 0.5–5% addition in oil-based drilling fluid, and the shale permeability was reduced by bridging the pore throat and microfractures, and the well wall stability was improved during drilling. Xie Gang et al. [17] synthesized a water-based nano plugging agent, and the particle size test showed that the size of the synthesized nano plugging agent was less than 100 nm, its size mainly being concentrated between 1.6–99 nm. In addition, thermogravimetric experiments showed that the initial pyrolysis temperature of the material was 270 °C, and that this can be used as a nanopore-plugging material under high-temperature conditions. Zhao Chunhua et al. [18] prepared a nano-emulsified paraffin PF-EPF with a median particle size of 160 nm by a special method. This was tested to have good low-temperature resistance, and the experimental results of plugging performance evaluation showed that PF-EPF has excellent plugging performance, and the API filtration loss was reduced by 10 mL when added at a 3% concentration in water-based drilling fluid.

Based on the theory of collapse prevention and plugging, this paper researches collapse prevention drilling fluid technology for hard and brittle shale formations in the Longmaxi Formation of Changning, Weiyuan, Zhaotong block etr, and develops a new nano plugging agent. The field application results of YS129H well show that the water-based drilling fluid system with AMPS/AM as the key agent has outstanding effects. This can enrich and improve the drilling fluid collapse prevention technology to meet practical needs.

2. Materials and Methods

2.1. Main Drugs and Instruments

Acrylamide (99%, A.R. grade) and 2-acrylamido-2-methylpropanesulfonic acid (99%, A.R. grade) were purchased from Hubei Xinrude Chemical Co, Wuhan, China. WSD (median particle size D_{50} = 354.26 μm) andnano pluggingagent NR-1 (median particle size D_{50} = 287.91 μm) were purchased from Xi'an Qiyue Biotechnology Co., Xi'an, China. All chemicals were of analytical grade and no further purification was required.

The simulated microfracture sealing performance evaluation experiment was carried out with the data measurement of core sealing pressure transfer experiment instrument (SUP, Hangzhou Futong Technology Co., Ltd., Hangzhou, China), and the data of press-in hardness performance evaluation experiment was measured by the hardness tester (HRS-150, Zhengzhou Yipin Instrument Co., Ltd., Zhengzhou, China).

2.2. Preparation of Nano Plugging Agent AMPS/AM

In the three-neck flask first add 100 mL of deionized water, set the stirring revolution to 1200 r/min; add 16 g 2-acrylamido-2-methylpropanesulfonic acid and 23 g acrylamide respectively, stirring at room temperature for 30 min to dissolve; turn on the cooling water circulation, slowly add 16 g 30% sodium hydroxide solution for neutralization reaction, control the reaction below 40 °C, and react for 30 min; then add 50 g liquid paraffin at a rate of Add 50 g of liquid paraffin wax dropwise at a rate of 30 g/h, and then self-emulsify for more than 2 h after the dropwise addition; add 3 g of initiator azobisisheptanenitrile and slowly increase the temperature to 60 °C for 5 h. Through the above preparation steps, the viscous white liquid nano plugging material AMPS/AM solution can be obtained.

2.3. Performance Evaluation of Nano Plugging Agent AMPS/AM

2.3.1. Simulation of Microcrack Sealing Evaluation

The instrument used is the core-blocking pressure transfer experiment instrument, which fixes the core and provides the surrounding pressure through the core holder system. The fluid to be tested circulates through the fluid circulation system, the surrounding pressure and temperature control system. The data acquisition system regulates the experimental conditions and collects the recorded data. The instrument can simulate the downhole operating environment with the following technical specifications: (i) the diameter of the tested core is 25.4/38.1 mm; (ii) the pressure-bearing capacity of the kettle is 0–20 MPa; (iii) the surrounding pressure is 0–30 MPa; (iv) the temperature control range of the kettle is 0–150 °C; (v) the filling capacity of the kettle is 1.0–1.5 L; (vi) the simulated fracture width is 0–1500 μm; (vii) the flow measurement accuracy is 0.1 mL.

Experimental methods: (1) Firstly, load the prepared shale core into the core holder with the pressure-bearing capacity experimental evaluation device, and load the core-surrounding pressure to 15 MPa; (2) open the liquid-filled kettle of the experimental device, pour the prepared liquid to be tested into it, and screw the kettle tightly; (3) open the regulator connecting the gas cylinder and the kettle, set the pressure of the kettle to P_0 and keep it stable, then open the kettle stirring device, and P is the top pressure of the core; open the valve at the outlet end of the kettle, and collect the end pressure of the core P; (4) close and clean the experimental apparatus, and save the experimental data. P_0 is the pressure at the top of the core; open the valve at the outlet end of the core holder and collect the pressure at the end of the core P_t; (4) shut down and clean the experimental apparatus and save the experimental data. By measuring the transfer pressure P_t at the end of the core and comparing the pressure drop ΔP in the shale core after blocking, it is used to evaluate the blocking ability of the blocking agent on the shale core; (5) the calculation process is expressed by $\Delta P = P_0 - P_t$.

2.3.2. Press-In Hardness Test

The rock hardness experimental test procedure was based on DZ/T 0276.6-2015 "Rock Physical and Mechanical Properties Test Procedure Part 6: Rock Hardness Test". A cylindrical flat-bottomed indenter was used for the experiment, and the diameter of the indenter was 2.32 mm, and the equation of rock indentation hardness was

$$P_k = \frac{P}{S} \times 1000$$

where: P_k—indentation hardness of the rock, Mpa; P—load when complete crushing occurs, kN; S–bottom area of the compression mold, mm^2.

2.3.3. Biotoxicity Evaluation Experiments

At present, the common biological toxicity testing method for drilling fluids is the luminescent bacteria method, and the biotoxicity of drilling fluid was reflected by the number of toxicants of luminescent bacteria at 50% luminescence. This was performed according to the biotoxicity grading standard of oilfield chemicals and drilling fluids stipulated in Q/SY111-2007 "Grading and testing method of oilfield chemicals and drilling fluids biotoxicity by luminescent bacteria method". When EC_{50} value > 25,000 mg/L is non-toxic state.

3. Results

3.1. Nano Plugging Material AMPS/AM Particle Size Analysis

The particle size distribution of the nano blocker AMPS/AM was measured using a laser particle size meter. The results are shown in Figure 1.

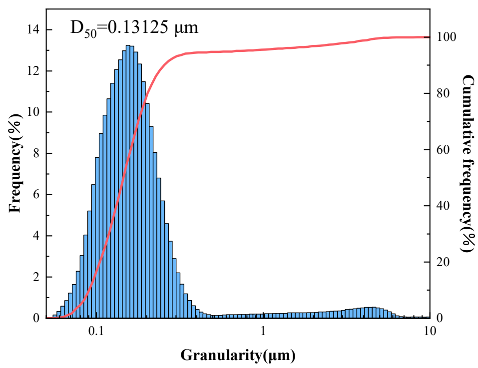

Figure 1. Particle size distribution of nano blocker AMPS/AM.

According to Figure 1, the median particle size of the nano blocker AMPS/AM reached 131.25 nm before the use of dispersion means, which has reached the nanometer level.

3.2. Simulated Microcrack Sealing Performance Evaluation

Cores for the core-blocking evaluation experiments were prepared using outcrop rock samples from the Longmaxi Formation shale in Changning, with a core size of 25.4 mm × 50 mm (diameter × length) (gas-side permeability of 3.24 × 10^{-4} μm^2). The experimental test temperature was 120 °C, and the test pressure at the top of the core was P_0 = 6.9 MPa. The original core, original core + base mud (4% bentonite, the same below), rock core + base mud + 3% AMPS/AM sealer, rock core + base mud + 3% NR-1 sealer, and rock core + base mud + 3% WSD sealer after repetition were tested respectively. The results are shown in Figure 2 for the variation of core end transfer pressure P_t with time.

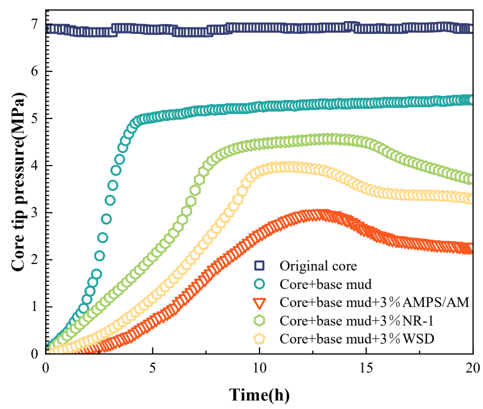

Figure 2. Pressure variation curve at the end of the core after the action of the fluid to be tested.

As can be seen from Figure 2, the end pressure of the core after the core+base mud test was 5.06 MPa after 5 h under the effect of 6.9 MPa differential pressure at the top of the core, and the differential pressure Δp loss was about 1.87 MPa. When the fluid to be tested for the experiment was the base mud containing 3% AMPS/AM, the end pressure of the core did not increase significantly in the first 3 h. After 13 h, the end pressure of the core rose to 3.06 MPa; with the test after 13 h, the pressure at the end of the core increased to 3.06 MPa; with the extension of the test time, the pressure at the end of the core decreased continuously to 2.34 MPa and stabilized. Compared with the top pressure of core 6.9 MPa, the pressure loss decreased by 66.09%, while the end pressure of core tended to 3.71 MPa and 3.28 MPa after 13 h when the fluid to be tested was 3% NR-1 and WSD-based mud, and the pressure loss decreased by 46.23% and 52.46% compared with the top pressure of core 6.9 MPa, indicating that the pressure loss of the nano sealer was reduced to 3.71 MPa and 3.28 MPa after 13 h, respectively. After the contact with shale, it enters the seepage channel inside the core under the effect of pressure difference, seals the micro-nanopores, retards the liquid phase intrusion into the core, and prolongs the time of pressure transfer. The performance comparison with nano plugging agent WSD and NR-1 shows that the plugging agent AMPS/AM has good pressure-bearing and plugging ability.

3.3. Press-In Hardness Performance Evaluation

The indentation hardness of the shale dry core of Longmaxi Formation, the rock sample after the repulsion of 4% bentonite-based mud and the core after the repulsion of blocking mud containing 3% AMPS/AM were tested respectively at three test points. The axial load at the time of core destruction was experimentally derived, and the indentation hardness values of the shale core after mass transfer with different media could be derived according to the indentation hardness calculation formula. The results of these procedures are shown in Table 1.

Table 1. Hardness test results of cores before and after replacement.

Recipe	Press-in Hardness/MPa			
	H1	**H2**	**H3**	**Have**
Original Rock	1185.68	959.54	953.48	1031.54
Base mud	360.48	266.34	228.31	285.05
Base mud + 3% AMPS/AM	542.30	431.55	380.56	450.88

From the calculation results in Table 1, it can be seen that the average compressive hardness of the dry rock sample of the Longmaxi Formation shale outcrop is 1031.54 MPa after testing; after blocking by base mud, the average compressive hardness of the shale core is 285.05 MPa, which is sharply reduced compared with the original rock hardness. After blocking with 3% AMPS/AM blocking mud, the average compressive hardness of cores was 450.88 MPa, which increased by 58.12% compared with that of cores after the action of base mud. Combined with the core-plugging evaluation results, it can be seen that the plugging agent AMPS/AM can, to a certain extent, seal the shale micro-nanopores, retard the liquid phase intrusion and pressure transfer, reduce the destructiveness of water and pressure on the rock, and thus the core indentation hardness exhibits a certain degree of improvement.

3.4. Performance Comparison of AMPS/AM and Other Nano-Sealers

The nano blocker AMPS/AM, nano blocker WSD and nano blocker NR-1 synthesized in this paper were added into the water-based drilling fluid base mud, and the filtration loss before and after aging was measured by high-temperature and high-pressure water loss meter, and the experimental conditions were: 150 °C and 16 h of aging, and their experimental results before and after aging are shown in Figure 3.

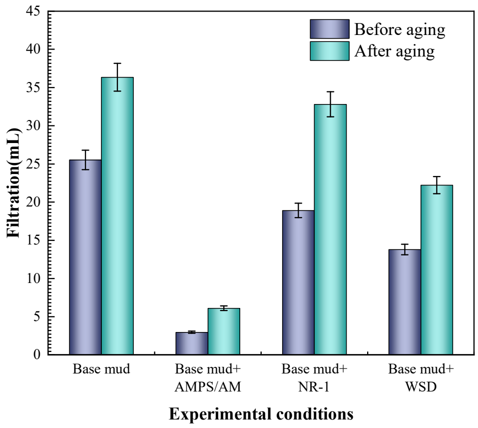

Figure 3. Comparison of the filter loss reduction performance of three blocking agents in base mud.

It can be seen from Figure 3 that the reduction of filtration loss of water-based drilling fluid base mud and the threenano pluggingagents differed significantly, the filtration loss of base mud was larger before the addition ofnano pluggingagents, and the filtration loss of base mud before and after aging was reduced when the threenano pluggingagents were added, and the reduction of filtration loss of base mud was 32.2 mL and 24.4 mL after the addition ofnano pluggingagent NR-1 andnano pluggingagent WSD, respectively. The filtration loss of the mud was significantly reduced by the nano blocker AMPS/AM compared with the above two nano blockers, especially in the high-temperature environment. The filtration loss was only 6.1 mL, indicating that AMPS/AM can effectively seal the micro and nanopores of the mud and reduce the permeability, which can significantly reduce the filtration loss of the mud.

3.5. Biotoxicity Evaluation

The results of the biological toxicity test of nano blocker AMPS/AM are shown in Table 2.

Table 2. Biotoxicity test results of nano blocker AMPS/AM.

Number	Drilling Fluid Category	EC_{50}/mgL-1	Toxicity Level
1	Nano plugging agent AMPS/AM	27,500	Non-toxic

As shown in Table 2, the EC_{50} of AMPS/AM = 27,500 mg/L, which is greater than the critical value of 25,000 mg/L, indicating that the indoor synthesized nano-sealing agent AMPS/AM is non-toxic, harmless to the environment and meets the environmental requirements.

3.6. Field Applications

3.6.1. Overview of the YS129H Well in the Zhaotong Block

The Longmaxi shale in the Zhaotong block is a hard and brittle shale with particularly developed fracture laminae and high fragmentation, which poses a serious challenge to the plugging performance of the drilling fluid. When drilling in the horizontal section, the collapse of the well wall is often caused by the insufficient sealing performance of the system, which is mainly manifested by the serious falling of blocks from the well wall, in addition to the technical difficulties such as high frictional resistance and difficulty in controlling the rheology of drilling fluid.

In response to the characteristics of the shale formation in this block, a high-performance shale horizontal well water-based drilling fluid system was constructed with nano plugging agent AMPS/AM as the core treatment agent, other plugging agents, surface hydration inhibitor polyamine-3 and highly efficient environmental protection lubricants.

3.6.2. Drilling Fluid Formulation for the Test Section of Well YS129H

The formulation of the strong plugging field water-based drilling fluid system formed by compounding and optimization is as follows:

2~3% bentonite + 0.25% NaOH + 0.3% FA367 (amphoteric polymeric encapsulant) + 1% Redu1 (filter loss reduction agent) + 0.3% QWA (high-temperature filter loss reduction agent) + 2% RSTF (high-temperature anti-salt filter loss reduction agent) + 1% SOLTEX (drilling fluid anti-collapse agent) + 0.125% CaO + 2% WN2-1 (drilling fluid sealer) + 1% WN2-2 (drilling fluid sealer) + 1% surface hydration inhibitor polyamine-3 + 1~1.5% nano blocker AMPS/AM + 1% double sparse inhibitor + 1~1.5% bonding lubricant + 1~3% YH-150 (filter loss reduction agent) + 5% JD-6 (filter loss reduction agent) + 7% KCl + 5~10% Weigh-2 (water-soluble weighting agent) + barite (as needed by density).

YS129H well test formations: Hanjiadian~Longmaxi. Test well depth of straight section: 1308~1766 m (458 m); test well depth of slanting section: 1766~2420 m (654 m); test well depth of horizontal section: 2420~3920 m, horizontal section length 1500 m.

The basic properties of the field-configured water-based drilling fluid system are shown in Table 3.

Table 3. Field water-based drilling fluid performance.

Density (g/cm^3)	Funnel Viscosity (s)	Contains Sand (%)	Static Shear Force (Pa)		Filtration Loss/mL		Extrusion Lubrication Coefficient	pH
			First Cut	Final Cut	API Medium Pressure Filter Loss	High-Temperature and -Pressure Filter Loss		
2.0~2.2	45~50	<0.3	2~6	9~12	≤1.5	≤5.0	0.060~0.064	7~10

3.6.3. Plugging Performance of Water-Based Drilling Fluids

The drilling fluid system sealing performance was evaluated by taking 3500 m well mud through high-temperature and -pressure water loss and mud cake permeability reduction rate, where the measurement of mud cake permeability reduction rate was mainly calculated by measuring the change of mud cake permeability before and after sealing. The experimental results are shown in Table 4.

Table 4. Field evaluation of plugging performance of water-based drilling fluid system.

System Blockability Evaluation Method	High-Temperature and -Pressure Water Loss/mL	Mud Cake Permeability Reduction Rate/%
Evaluation results	<4 mL	85.67

As can be seen from Table 4, the shale gas water-based drilling fluid high temperature and pressure tested in this well lost <4 mL of water, the mud cake thickness was 1~2 mm (experimental conditions: 90 °C, 3.5 MPa), and the mud cake formed was of thin quality and good toughness. The mud cake permeability reduction rate was 85.67%. In summary, it shows that the field water-based drilling fluid system has a good sealing effect.

The well mud at 2820 m was also taken and added with 3% nano plugging agent AMPS/AM to investigate its effect on well mud performance, and the results are shown in Table 5.

Table 5. Field evaluation of plugging performance of water-based drilling fluid systems.

System Blockability Evaluation Method	FV/s	High-Temperature and -Pressure Water Loss/mL	Kf
Before joining AMPS/AM	66	3.8 mL	0.0887
After joining AMPS/AM	68	2.0 mL	0.0891

From Table 5, it can be seen that the addition of nano-AMPS/AM can increase the sealing capacity of well mud, with excellent effects of filtration loss reduction, and also improve the quality of mud cake, with no significant effect on the viscosity and lubricity of well mud.

3.6.4. Borehole Stability

The shale gas water-based drilling fluid test in the YS129H well had no block drop during the completion of the diameter section to the horizontal section, and the well wall was stable. The well diameter expansion rate and start-up and downhole friction are shown in Table 6.

Table 6. Well diameter expansion rate and start-up and downhole friction in YS129H.

Well Number/Well Section	Well Diameter Expansion Rate (%)	Drill Start Friction (ton)	Down-Drilling Friction (tons)
YS129H straight well section: 1308~1766 m	5.56	5~10	5~10
YS129H inclined section: 1766~2420 m	7.98	10~15	5~15
YS129H horizontal section: 2420~3920 m	13.20	10~30	5~35
The average well diameter enlargement by electrical measurement was 11.11%			

As can be seen from Table 6, the starting and downhole friction in the straight section is 5–10 tons, the starting and downhole friction in the sloping section is 10–15 tons and downhole friction is 5–15 tons, the downhole friction in the horizontal section is 10–30 tons and downhole friction is 5–35 tons. During the drilling process with water-based drilling fluid, the starting and downhole friction are in the normal range, indicating that the drilling fluid system has good stability. The average borehole expansion rate was 5.56% in the straight section (1308~1766 m), 7.98% in the inclined section (1766~2420 m) and 13.2% in the horizontal section (2420~3920 m), and the average borehole expansion rate was 11.11% by electrical measurement, which was within the normal range. Meanwhile, it can be seen from Table 7 that the value of water-based drilling fluid recovery and reuse occupies 50% of the cost of recovered drilling fluid, accounting for a relatively large proportion. The drilling fluid recovery and reuse technology can substantially reduce the cost of drilling fluid, and it can realize less discharge of waste drilling fluid and reduce the pollution of the environment by waste drilling fluid, which has high economic value and environmental protection value.

Table 7. Shale gas water-based drilling fluid recycling and reuse.

Amount of Mud Used for Drilling/m^3	Drilling Consumption/m^3	Recovery of Solid Wells/m^3
461	234	227
Cost of recovered drilling fluid: 227 m^3 × 5447 yuan = 1,236,500 yuan		
Recycling value: 1,236,500 yuan × 50% = 618,300 yuan		

In summary, the system has good sealing and inhibiting properties, which can improve the stability of the well wall during the drilling process and reduce the incidence of complex situations. The well diameter expansion rates are all within the normal range. During the drilling process with water-based drilling fluid, the start-up and downhole friction were all within the normal range, indicating that the drilling fluid system has good stability.

4. Conclusions

In this paper, AMPS/AM was prepared by using acrylate, propylenesulfonate and liquid paraffin as the main raw materials. Compared with other plugging agents, the synthetic raw material of nanometer plugging agent in this paper is easy to obtain and cheap, the synthesis process is simple, while the low-dosage treatment agent can meet the field plugging performance effect, can greatly save the drilling cost, and has broad application prospects. The median particle size of AMPS/AM reached 131.25 nm before the use of dispersion means. The results of compression hardness test show that the average core compression hardness is 450.88 MPa after sealing with 3% AMPS/AM plugging agent, which is 58.12% higher than the core compression hardness after base slurry action. Combined with the core-plugging evaluation results, it can be seen that the plugging agent AMPS/AM can, to a certain extent, seal the shale micro-nanopores, retard the liquid phase intrusion and pressure transfer, reduce the destructive effect of water and pressure on the rock, and thus the core indentation hardness exhibits a certain degree of improvement. The field test results show that the strong plugging system formed by adding the plugging agent AMPS/AM has better inhibition and solidification plugging, which can improve the stability of the well wall during drilling and reduce the incidence of complex situations.

The following limitations exist in this paper. Only the plugging performance of AMPS/AM is evaluated, but the microstructure of AMPS/AM is not characterized, which makes it impossible to understand the plugging mechanism of AMPS/AM intuitively. In addition, the plugging effect of the drilling fluid system with the addition of plugging agent AMPS/AM alone should be considered for field application to better reflect the plugging performance of plugging agent AMPS/AM.

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Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

Data Availability Statement: Data is contained within the article.

Conflicts of Interest: The authors declare no conflict of interest.

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