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Study of a new structural emulsion and its application in drilling fluids

Han-Xuan Song, Yun-Jin Wang, Yan Ye *

State Key Laboratory of Petroleum Resource and Prospecting, China University of Petroleum, Beijing, 102249, China

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ABSTRACT

Aiming at the leakage problem in the compact sandstone drilling of the Keziluoyi Formation in Southwest Tarim, Nano-core-emulsion was prepared by coating modified nano-SiO₂ with nano-emulsion, its particle size D_{50} is about 100 nm, with good dispersion stability. When 0.8% Nano-core-emulsion is added to 5% bentonite slurry, the fluid loss can be reduced by 40%, and the filter cake thickness can be reduced by 84%. Using a Nano-core-emulsion to optimize the plugging performance of potassium polysulfonate drilling fluid can reduce the fluid loss of the drilling fluid by 52%, the resulting filter cake is dense and tough, and the thickness is reduced by 40%. Using the pressure conduction method to evaluate the plugging rate, the plugging rate of the drilling fluid of the Nano-core-emulsion on the core of the Keziluoyi Formation is 63.4%, which is 20.9% higher than that of the field drilling fluid. According to microscopic examination and CT scanning analysis, the material has the plugging characteristics of “inner rigid support + outer soft deformation” and has demonstrated good field application results.

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1. Introduction

“The embankment of a thousand miles collapses in an ant nest.” In recent years, during the drilling process, the problem of wellbore instability has frequently occurred due to the leakage of micro-nano pores and the development of micro-nano fractures (Mirabbasi et al., 2022). In response to this phenomenon, scholars have conducted research on micro- and nanoscale plugging (Abdullah et al., 2022; Ghanbari and Naderifar, 2016; Yang et al., 2022), and nanomaterials have also been widely used in drilling fluid optimization (Al-Shargabi et al., 2022; Ibrahim et al., 2022).

The existing nano-plug materials are mainly divided into three types: organic nano-plug materials, inorganic nano-plug materials, and composite nano-plug materials. Among them, the plugging mechanism of nano-organic plugging materials is mainly the flexible deformation of polymers in irregular pores. An et al. (2015) synthesized a kind of nano-terpolymer, and the plugging performance of the terpolymer was evaluated by a specific surface porosity physical adsorption instrument (BET) and membrane efficiency tester. The results show that the terpolymer can effectively plug the nano pores of shale. Huang et al. (2022) used micellar polymerization to synthesize functionalized polystyrene latex (FPL)

as nano-plugging agents (NPA) for drilling fluid. FPL improves the quality of the filter cake and plugs the fine pores of the ceramic filter discs. Lei et al. (2022) used radical graft polymerization to create a series of carboxymethylchitosan-g-polyoligo (ethylene glycol) methyl ether methacrylate-coacrylic acid (GCOA) and developed an intelligent liquid plugging evaluation method for continuous temperature-controlled core flow tests. Evaluation results: When the temperature reaches above the switch point, GCOA can form effective plugs in low-permeability and medium-permeability cores. Ye et al. (2020) introduced nano-emulsion in drilling fluid to improve plugging performance. The plugging performance was evaluated by the pressure transmission method, which confirmed that the nano-emulsion can delay the pressure transmission and filtrate intrusion.

The plugging mechanism of nano-inorganic plugging materials is mainly based on the bridging and filling of nanomaterials in pores (Zhang et al., 2019). Gao et al. (2021) used various nanomaterials to seal shale and conducted pressure transfer experiments for evaluation. The results showed that nano-Al₂O₃ can solve different types of shale hydration and expansion problems. Li et al. (2023) used different types of nano-SiO₂ to seal shale and analyzed the sealing mechanism. The results showed that nano-SiO₂ can improve the hydrophobicity of the shale surface and block the pores in the shale. Ma et al. (2020) developed modified multi-walled carbon nanotubes (MWCNTs) for the plugging of low-permeability reservoirs. Studies have shown that the modified

* Corresponding author.

E-mail address: yeer996@126.com (Y. Ye).

multi-walled carbon nanotubes can be stably dispersed at 170 °C for 24 h. It has good plugging performance and can be used as a nano-plugging agent for water-based drilling fluid. Pourkhalil and Nakhaee (2019) added nano-ZnO particles to the water-based drilling fluid to reduce damage to shale. The plugging performance of nano-ZnO was evaluated by the PPT device, and the results showed that nano-ZnO particles could improve the stability of shale due to their size, hydrophilicity, and positive charge. Wang et al. (2020) used graphene oxide (GO) to plug downhole nano-scale pores to inhibit water intrusion into shale and prevent shale clay mineral expansion. Research has shown that graphene oxide sheets can form a protective film that plugs and fills pores of different shapes in shale.

Nanocomposite materials have the advantages of both organic and inorganic plugging materials (Zhong et al., 2020) and have become a research hotspot in recent years (An et al., 2016; Zhang et al., 2021). Koh et al. (2022) believed that polyacrylamide modified with dopants such as carbon nanotubes, zeolites, silica, and graphene oxide has great application value in drilling fluid. Li et al. (2020) synthesized styrene-butadiene resin/nano-SiO₂ (SBR/SiO₂) composites by continuous emulsion polymerization, and then the plugging ability was evaluated by filtration volume, porosity physical adsorption instrument (BET), and pressure transmission test. The results show that SBR/SiO₂ can enter shale nanopores, significantly reduce fluid invasion, and improve wellbore stability. Huang et al. (2018) synthesized a nano-acrylic resin/nano-SiO₂ composite (AR/SiO₂) with a core-shell structure and evaluated its plugging capacity by using pressure transfer tests and N₂ adsorption tests. The results show that (AR/SiO₂) used in the water-based drilling fluid (WBM) can improve the plugging efficiency of shale pores during shale gas drilling, reduce fluid invasion, and improve wellbore stability. Mao et al. (2015) prepared a novel core-shell structure hydrophobic polymer-based nano-SiO₂ composite material, which has good properties such as thermal stability, rheology, filter loss, and lubricity can effectively plug the formation and significantly improve the pressure-bearing capacity of the formation. Studies by many scholars have shown that nanocomposites have better pressure-bearing capacity and adaptability than water-based drilling fluid additives. It is the trend for the future development of plugging materials.

Tarim Oilfield has become an important area for oil and gas exploration in recent years (Zhu et al., 2018), with abundant oil and gas resources in the ultra-deep layers of this block (Gao et al., 2022; Wang et al., 2022; Zhu et al., 2021). During the drilling of ultra-deep wells in the Yingsha Yulong block, southwest of Tarim, the wellbore instability caused by the micro-nano fracture pore leakage of the tight sandstone of the Keziluoyi Formation (Liu et al., 2021) has seriously affected the engineering progress (Li X. et al., 2022; Qiao et al., 2021; Zhu et al., 2020). Scholars have conducted observational studies on the micro-nano pores contained in tight sandstone in the early days (Li Z. et al., 2022; Qiao et al., 2022; Wang et al., 2022), and the leakage of micro-nano pores can easily cause fractures. The damage between the development of the rock matrix and the rock matrix is a difficult problem that cannot be ignored in the drilling process (Yang et al., 2022).

In this paper, nano-SiO₂ was hydrophobically modified and grafted, and the modified SiO₂ was dispersed and coated into the nano-emulsion to construct a new structure of the plugging system—the Nano-core-emulsion. The Nano-core-emulsion can effectively disperse and coat nano-SiO₂ in the emulsion to form a spherical core-membrane structure. The compact sandstone of the Keziluoyi Formation was tested using the Nano-core-emulsion, and pressure transmission was used to evaluate it. In addition, the plugging of the Nano-core-emulsion was also performed. The mechanism is explored and described, which guides the

development and application of nano-plugging materials.

2. Experiment

2.1. Experimental samples and instruments

2.1.1. Compact sandstone of the Keziluoyi Formation in the southwest block of tarim

During the drilling process, drilling fluid leakage caused well wall instability in this formation, where the dense sandstone of the Keziluoyi Formation is located. Several cores of the dense sandstone of the Keziluoyi Formation were obtained at the site, and the microscopic morphology of the cores was observed by scanning with a HORIBA (7593-H) electron microscope (SEM), and the microscopic characteristics of the cores were obtained as shown in Fig. 1.

It can be seen from Fig. 1 that the compact sandstone of the Keziluoyi Formation contains a large number of irregular micro-nano pores and fractures; the diameter of the pores are between 500 nm and 20 μm, and the opening of the fractures is between 100 nm and 30 μm. Nanopores and cracks are the main causes of leakage.

2.1.2. Experimental samples and instruments

Experimental samples: potassium chloride (AR≥99.5), nano-SiO₂, silane coupling agent KH-550, silane coupling agent KH-570 (AR≥99.5), VTEO (C₈H₁₈O₃Si, AR≥99.5), Tween 80 (AR≥99.5), 1-Butanol (C₄H₁₀O, AR≥99.5), AES (C₁₂H₂₅ NaO₃S, AR≥99.5), *n*-octane (C₈H₁₈, AR≥99.5), bentonite slurry (5%, Tarim Oilfield), water-based drilling fluid (Tarim Oilfield).

Experimental instruments: a heating agitator (TMHB-180C), ultrasonic cleaning machine (KQ3200DE), vacuum oven, six-speed viscometer (ZNN-D6), high-temperature and high-pressure filter loss meter (HDF-1).

Particle size analyzer (Topsizer-JL-1198), contact angle analyzer (XG-CAMA), stability analyzer (Formula-MLS), Fourier transform infrared spectroscopy (FTIR, BRUKER, TENSORII) were used for structural characterization. Morphological observation experiments were performed using field emission environmental scanning electron microscopy (SEM, Gemini SEM 300), computed tomography (CT, Optima CT660), and field emission transmission electron microscopy (TEM, JEM-2100 F).

2.2. Preparation and performance characterization of nano-core-emulsion

2.2.1. Preparation of nano-core-emulsion

10 g of SiO₂ (50 nm) particles were added into 100 mL of absolute ethanol solution and stirred to disperse evenly. Ammonia water was added to the silica suspension to adjust the pH value to be neutral. Then, 5 mL of VTEO was added to the suspension as a modifier. The reaction temperature was controlled at 60 °C and stirred slowly for 20 h. The obtained sample was filtered with a Buchner funnel, repeatedly washed with ethanol, and filtered three times to avoid organic residues. The sample obtained by suction filtration was vacuum dried at 60 °C. As a result, hydrophobic nano-SiO₂ is obtained.

Tween 80 and fatty alcohol polyoxyethylene ether sodium sulfate (AES) was used as composite surfactants (S), *n*-butanol was used as an epistatic agent (A), *n*-octanol containing hydrophobic SiO₂ was used as the oil phase, and Nano-core-emulsion were prepared by the aqueous solution dilution method based on the quasi-three-phase diagram of the nano-emulsion. The dispersion is coated in the nano-emulsion oil equivalent and successfully combined to become a “soft plus hard” plugging emulsion—Nano-core-

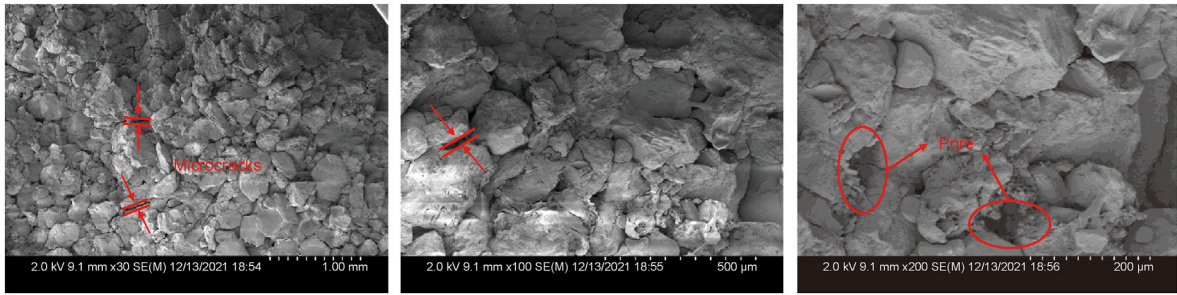


Fig. 1. Microscopic morphology of compact sandstone cores in the Keziluoyi Formation.

emulsion. The synthetic route of the nano-core-emulsion is shown in Fig. 2.

2.2.2. Performance characterization of nano-core-emulsion

Use FTIR spectrometer (Nicolet iS50) to analyze the structure of nano-SiO₂ before and after modification; the particle size of nano-SiO₂ solution and Nano-core-emulsion was measured by particle size analyzer (Topsizer); the micro morphology of Nano-core-emulsion was observed by transmission electron microscope (HT7820); test the stability characteristics of Nano-core-emulsion with temperature change by using a stability analyzer (Formula action MLS); the temperature resistance of Nano-core-emulsion was tested by thermogravimetric analyzer (TG209F1); measure the contact angle of glass sheets before and after coating with Nano-core-emulsion using a contact angle analyzer (OSA200); the

interfacial tension of Nano-core-emulsion with different concentrations was measured by interfacial tensiometer (DCAT25).

2.2.3. Test of influence of bentonite on stability of nano-core-emulsion

Take a certain amount of Nano-core-emulsion and divide it into several parts, add 0.5%, 1%, 1.5% and 2% bentonite, and use the stability analyzer (Formula action MLS) to measure the TSI value of the sample.

2.3. Experiment of nano-core-emulsion to optimize bentonite slurry filtration loss

Different amounts of Nano-core-emulsion were added to a 5% bentonite slurry to optimize the plugging performance. A six-speed

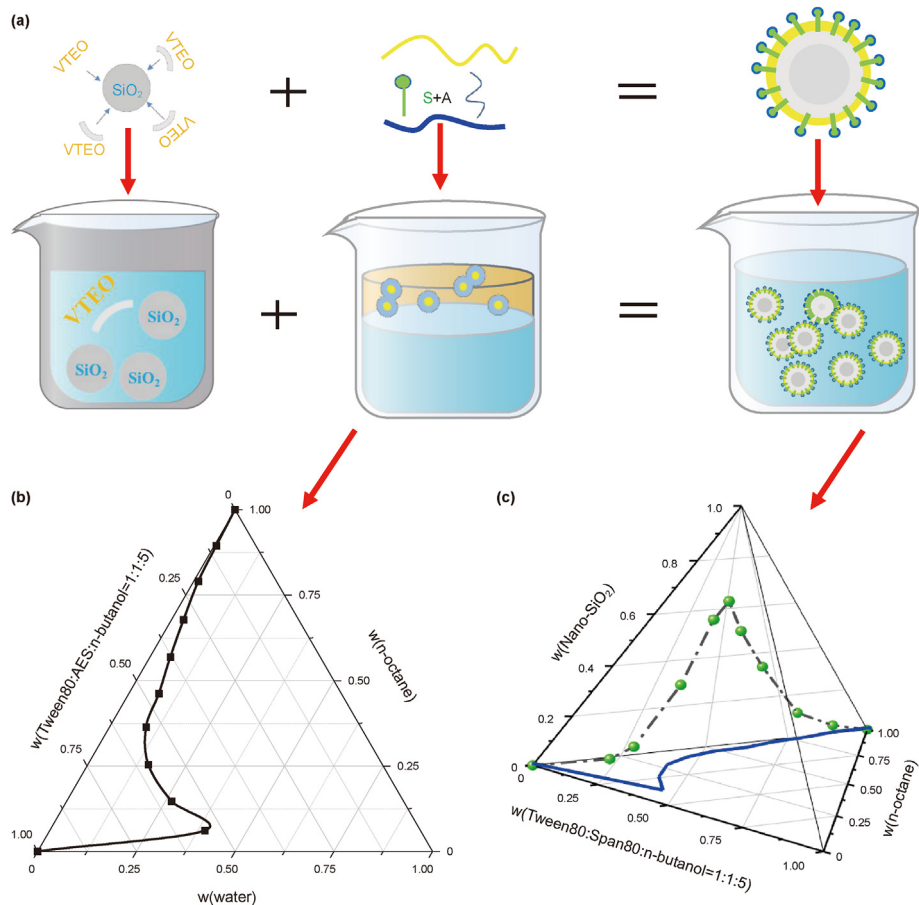


Fig. 2. Flowchart of preparation of Nano-core-emulsion.

rotational viscometer was used to test the viscosity and shear performance of the bentonite slurry, and high-temperature and high-pressure filter testers were used to test the filter loss and cake thickness of the bentonite slurry.

2.4. On-site drilling fluid performance optimization experiment of nano-core-emulsion

Add a certain amount of Nano-core-emulsion to the on-site potassium polysulfonate drilling fluid formula and heat roll at 150 °C for 16 h. A six-speed rotational viscometer was used to test the viscosity and shear performance of the drilling fluid, and high-temperature and high-pressure filter testers were used to test the filter loss and cake thickness of the drilling fluid.

2.5. Evaluation of plugging performance based on pressure conduction

Taking the compact sandstone of the Keziluoyi Formation in the Tarim block as the object of research, the plugging experiment was carried out on the core of the block using the Nano-core-emulsion, and the plugging performance was evaluated using the pressure conductivity instrument developed by China University of Petroleum (Beijing). The basic principle of the pressure transmission method is to establish an initial pressure difference between the upstream and downstream of the core, where the upstream is the fluid to be tested and the downstream is the simulated formation water. The fluid to be measured in the upstream seeps through the core to reach the sealed downstream, and the downstream pressure change is recorded. Fig. 3 shows the plugging performance test mechanism and pressure transmission instrument.

It can be seen from Fig. 3 that the principle of the pressure conductivity meter is to establish a pressure difference between the upper and lower sides of the core, detect the pressure decay curve with time, and compare the pressure transfer efficiency before and after plugging. According to the seepage mechanics equation, the calculation formula for the core permeability (K) was obtained as

follows:

$$K = \frac{\lambda \mu C V L}{A} \quad (1)$$

K — Core permeability, mD;

λ — The slope of the curve of $\ln [(P_m - P(L, t)) / (P_m - P_0)]$ changing with time;

μ — Viscosity of upstream fluid, mPa·s;

C — Downstream fluid compressibility, MPa; The change rate of permeability before and after is used to describe the plugging efficiency of the plugging material:

$$\eta = \frac{K_1}{K_2} \times 100\% \quad (2)$$

2.6. Nano-core-emulsion blocking mechanism study

Electron microscopy (SEM, Gemini SEM 300) was used to microscopically analyze the cores of the Keziluoyi Formation after the plugging experiment and to observe the microscopic characteristics of the plugging body of the Nano-core-emulsion. CT scanning (Optima CT660) was used to scan the plugged core to obtain the plugging state of the Nano-core-emulsion inside the core.

3. Results and discussion

3.1. Discussion on properties of nano-core-emulsion

The performance test and characterization of the prepared Nano-core-emulsion were carried out, and the relevant results are shown in Fig. 4.

Fig. 4(a) is the infrared spectrum of nano-SiO₂ and Nano-core-emulsion, where nano-SiO₂ is a black curve. The absorption band at 1095 cm⁻¹ is the Si—O—Si anti-symmetric stretching vibration peak; the peak at 798 cm⁻¹ is the Si—O bond symmetric stretching

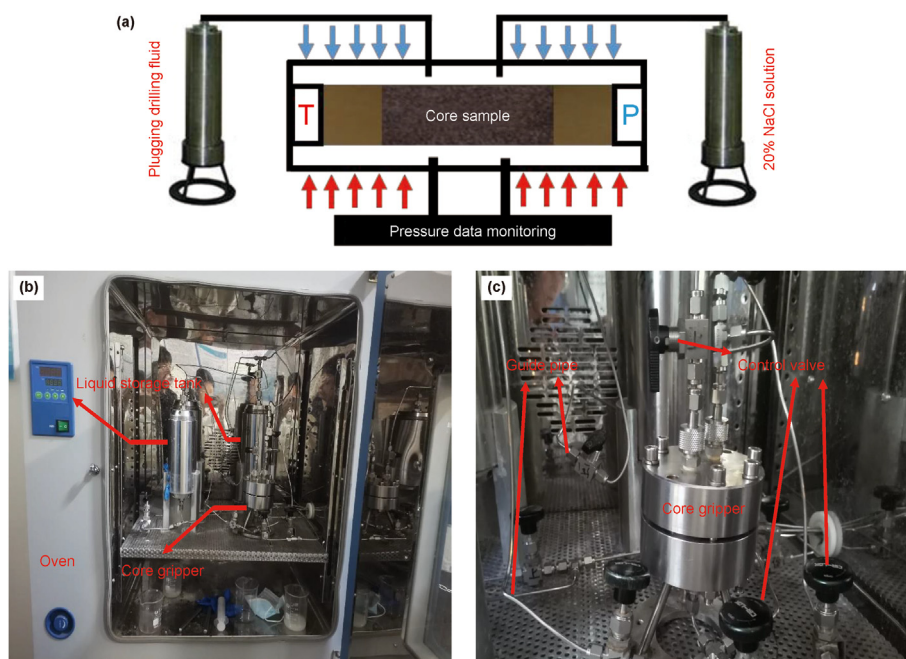


Fig. 3. Pressure conductivity meter: (a) Instrument schematic diagram; (b) Testing equipment; (c) Core gripper.

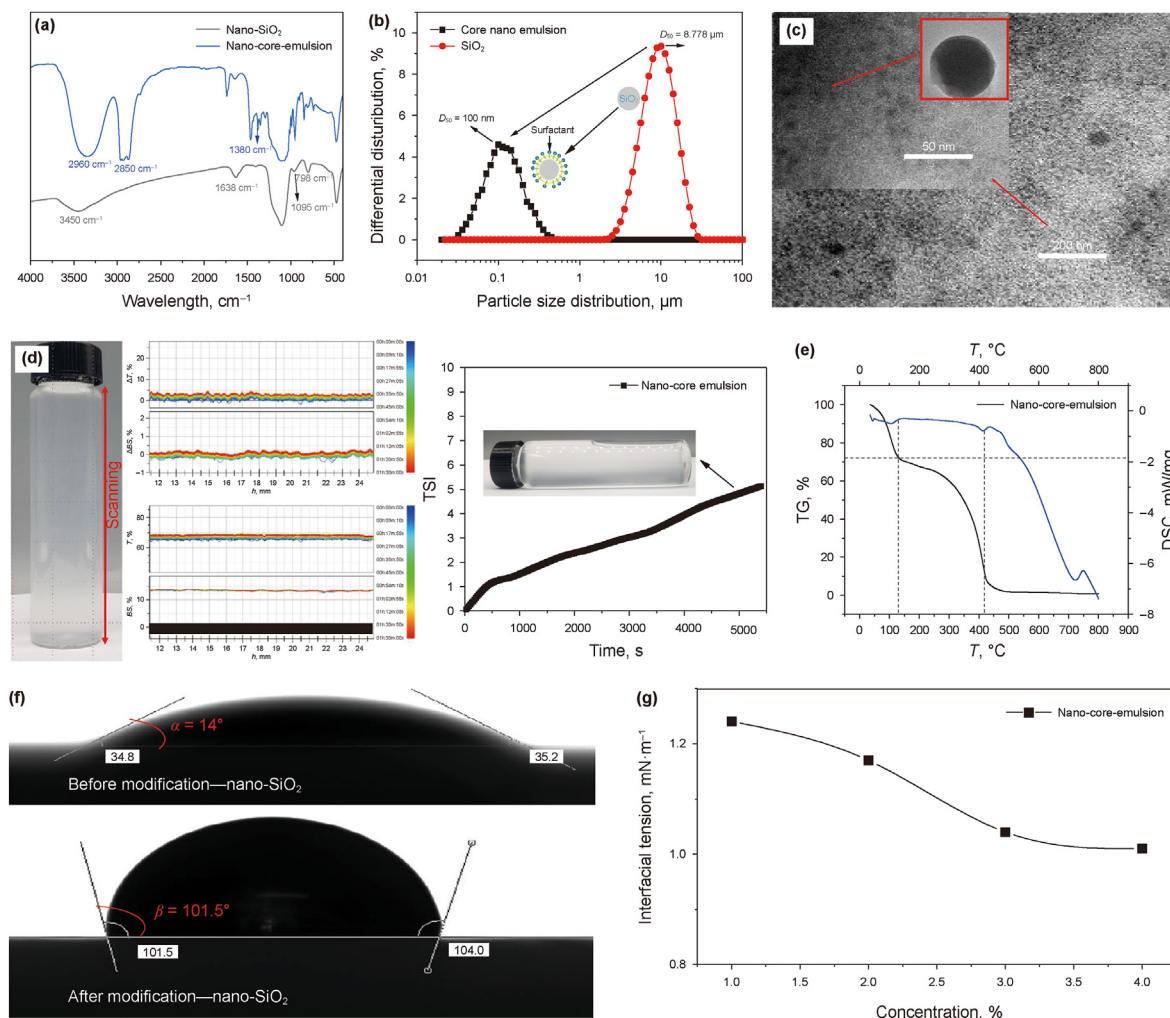


Fig. 4. (a) Infrared spectrum of Nano-core-emulsion; (b) Particle size analysis test of Nano-core-emulsion; (c) TEM of Nano-core-emulsion; (d) Stability analysis of Nano-core-emulsion; (e) TG-DSC analysis of Nano-core-emulsion; (f) Measurement of nano-SiO₂ contact angle after modification; (g) Measurement of interfacial tension of Nano-core-emulsion.

vibration peak; the broad peak at 3450 cm^{-1} is structural water and —OH anti-symmetric stretching vibration peaks; the peak near 1638 cm^{-1} is the H—O—H bending vibration peak of water; and the peak at 955 cm^{-1} belongs to the bending vibration absorption peak of Si—OH . In the Nano-core-emulsion (blue curve), 1380 cm^{-1} is the C—H vibration absorption peak of *n*-octane, and 2960 cm^{-1} is the C—H stretching vibration absorption peak of *n*-octane. Among them, 2873, 1111, 950 and 425 cm^{-1} are the characteristic spectra of Tween-80. Based on the above analysis, the Nano-core-emulsion contains surfactant and oil phases, which confirms the structure of the emulsion-coated nano-SiO₂.

Fig. 4(b) is a particle size distribution diagram of the Nano-core-emulsion. Nano-emulsion is used to coat and disperse nano-SiO₂. The particle size of nano-silica D_{50} is $8.7\text{ }\mu\text{m}$. The D_{50} particle size is around 100 nm , reaching the nanometer level.

Fig. 4(c) is the microscopic morphology of the Nano-core-emulsion. It can be seen that the particle size of the Nano-core-emulsion is about $20\text{--}50\text{ nm}$, and it is monodispersed, and it bears a layer of oil film structure on its surface, which is consistent with the design structure and confirms that the prepared product is a Nano-core-emulsion.

Fig. 4(d) is the stability test result of the Nano-core-emulsion. According to the sedimentation stability of the Nano-core-

emulsion, it can be seen that the Nano-core-emulsion still has good stability at 180 min and can be used as a good plugging additive.

Fig. 4(e) is the TG-DSC of the Nano-core-emulsion. From $130\text{ }^{\circ}\text{C}$, the weight of the Nano-core-emulsion has decreased significantly, which is the result of the desorption of some bound water. From $420\text{ }^{\circ}\text{C}$, the weight of the system gradually began to stabilize, which is because the rest of the system is mainly nano-SiO₂. Nano-SiO₂ has excellent temperature resistance, which can maintain the overall temperature resistance level of the system.

Fig. 4(f) shows that the contact angle with the water changed from 35° to 101.5° before and after the modification, and VTEO was successfully modified to obtain hydrophobic, lipophilic nano-SiO₂.

Fig. 4(g) shows the interfacial tension of the Nano-core-emulsion. It can be seen that the interfacial tension of the Nano-core-emulsion is between 1.0 and 1.3 mN/m , with ultra-low interfacial tension, which can be better dispersed in the drilling fluid, and can easily enter the micro nano pores to achieve the sealing of nano pores.

Based on the above analysis results, the Nano-core-emulsion is based on the rigid nano-silicon as the core, the outer deformable nano-emulsion is covered with deformable nano-emulsion “inner rigid support + external soft deformation” (soft and hard merger),

and the broad-spectrum nano-blocking mechanism integrating bridging and deformation is prepared. Its high dispersion and stability can ensure the effective plugging of micro-nano pores in the formation.

3.2. Study on the influence of bentonite on the stability of nano-core-emulsion

Test the influence of different dosage of bentonite on the TSI of Nano-core-emulsion, and the results are shown in Fig. 5.

As shown in Fig. 5, with the increase of bentonite dosage, the TSI value of the solution gradually increases. When the amount of bentonite added is 0.5%–2%, the TSI value of the solution is within the range of 24–56, and the relationship between the amount of bentonite added and the TSI value shows a linear relationship. It can be seen that in the Nano-core-emulsion, the concentration of bentonite particles has a certain impact on the stability.

3.3. Optimization of plugging performance of nano-core-emulsion

3.3.1. Effects of different nano-core-emulsion additions on bentonite slurry properties

Prepare 5% bentonite slurry from drilling fluid raw materials obtained on site and let it stand for 24 h. The basic properties of the bentonite slurry were measured with a six-speed viscometer and a high-temperature, high-pressure fluid loss tester. The results are shown in Table 1.

The 5% bentonite slurry was optimized by using Nano-core-emulsion with different concentrations and dosages, and the performance of the optimized formula is shown in Table 2.

It can be seen from Table 1 that the amount of Nano-core-emulsion has little effect on the plastic viscosity of the bentonite slurry. As the amount increases, the apparent viscosity and dynamic shear force of the bentonite slurry will be slightly reduced. Compared with No.0 bentonite slurry, it is found that the Nano-core-emulsion can enhance the structural strength of the system and contribute to the stability of the drilling fluid. High-temperature and high-pressure filter loss testers were used to evaluate the filter loss and wall-building properties of the optimized bentonite slurry, and the results are shown in Fig. 6.

It can be seen from Fig. 6(a) that under the addition of different

Nano-core-emulsion, the filtration loss of the bentonite slurry is different, and under the addition of a 0.8% Nano-core-emulsion, the filtration loss of the bentonite slurry is the smallest, reaching 20.8 mL within 30 min. The filtration loss is reduced by 40% compared with the original bentonite slurry.

Fig. 6(b) shows the thickness of the filter cake after the addition of different Nano-core-emulsion. It can be seen from the data that the thickness of the filter cake is 1–2 mm, the filter cake is thinner. When 0.8% Nano-core-emulsion is added, the thickness of filter cake decreases by 74%, and the thickness is 1.1 mm.

Compared with the filter cake photo in Fig. 6(c), the surface of the filter cake with an added amount of 0.8% is smooth and flat, showing strong toughness. Combining the loss and filter cake quality, the optimal addition of Nano-core-emulsion in the 5% matrix was determined to be 0.8%. The Nano-core-emulsion participates in the formation of the filter cake, which reduces the loss of drilling fluid filtration and reduces the damage to the formation by forming a dense filter cake.

3.3.2. Nano-core-emulsion improves drilling fluid performance on site

By adding the optimal amount of Nano-core-emulsion, 0.8% is obtained as the optimal amount, 0.8% Nano-core-emulsion is added to the on-site potassium polysulfonate drilling fluid formulation, and the properties of potassium polysulfonate drilling fluid before and after addition are shown in Table 3.

It shows from Table 3 that the potassium polysulfonate drilling fluid after adding the Nano-core-emulsion has good rheology, and the fluid loss is only 3.6 mL, which is reduced to 48% of the original drilling fluid. The filter cake thickness is only 1.2 mm, which is 60% of the thickness of the original drilling fluid filter cake, which greatly improves the performance of the drilling fluid itself.

It can be seen from the SEM micromorphology of the filter cake that the surface of the unoptimized drilling fluid No.0# filter cake is uneven, contains a large number of microcracks, and the toughness is insufficient. After adding the Nano-core-emulsion, the surface of the filter cake produced by the drilling fluid is dense and flat, without significant micro-nano pores.

3.4. Evaluation of compact sandstone cores plugged by nano-core-emulsion

The leakage of compact sandstone strata of the Keziluoyi Formation in the southwest region of Tara was analyzed, and it was concluded that the instability of the wellbore in this section is caused by a large amount of micro nano pore leakage and the development of micro cracks, which affects the overall drilling progress (Feng et al., 2021; Mirramezani et al., 2013).

The drilling fluid was optimized by using the Nano-core-emulsion, and the core of the block was plugged by the optimized drilling fluid filtrate. This was compared with the nano-SiO₂ plugging reagent purchased in the market, and the plugging efficiency of the Nano-core-emulsion calculated was fitted, and the results are shown in Fig. 7.

It can be seen from Fig. 7(a)–(1), the pressure conduction data before plugging of on-site drilling fluid is attributed to 0.5 MPa within 790 min, and the balance after plugging is within 990 min. Furthermore, the difference between before and after plugging is small, indicating that drilling fluid No. 0's ability to plug micro-nano pore cracks is poor. Fig. 7(a)–(2) is the permeability curve calculated based on the pressure conduction data, and according to Table 4, the permeability of the core before plugging is calculated to be 1.441×10^{-7} mD and the permeability of the core after plugging is 8.82×10^{-8} mD. According to Eq. (2), the occlusion rate of the tight sandstone core of the Keziluoyi Formation by the on-site

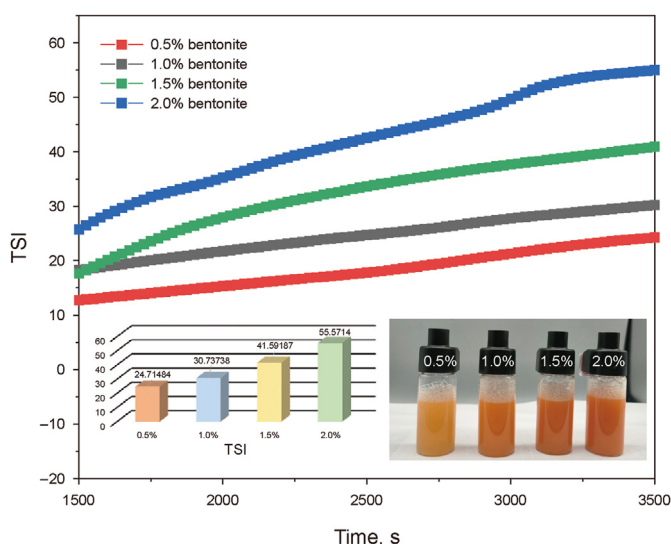


Fig. 5. Effect of different concentrations of bentonite on TSI value of inner Nano-core-emulsion.

Table 1
In-situ bentonite slurry stick-shear properties.

Number	(Nano-core-emulsion) Plugging agent	AV, mPa·s	PV, mPa·s	YP, P	G'/G''	Filtration, mL	Filter cake thickness, mm
0	/	28	10	18	6/14	34.2	4.2

Table 2
Nano-core-emulsion optimized viscosity-shear properties of bentonite slurry.

Number	(Nano-core-emulsion) Plugging agent	AV, mPa·s	PV, mPa·s	YP, Pa	G'/G''
1	0.3%	28.5	10	18.5	13/21
2	0.5%	25	10	15	10/18
3	0.8%	22	11	11	9/15
4	1%	25	10	15	11/19

(Note: 1: 5% bentonite slurry + 0.3% Nano-core-emulsion; 2: 5% bentonite slurry + 0.5% Nano-core-emulsion; 3: 5% bentonite slurry + 0.8% Nano-core-emulsion; 4: 5% bentonite slurry + 1% Nano-core-emulsion).

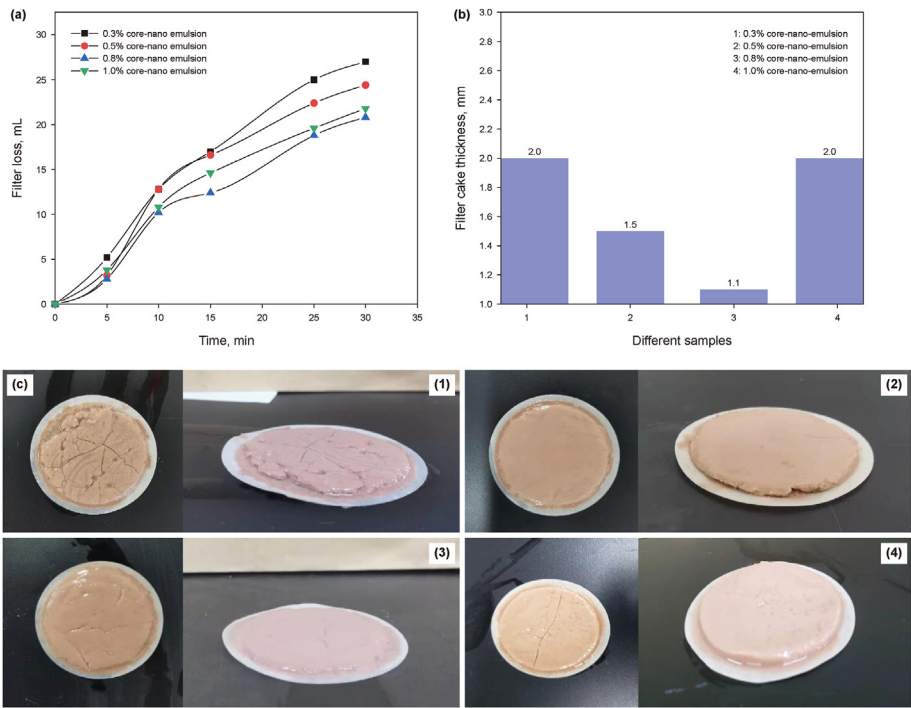


Fig. 6. (a) Variation of bentonite slurry filtration loss with different Nano-core-emulsion additions; (b) Filter cake thickness with different Nano-core-emulsion additions; (c) Filter cake morphology with different Nano-core-emulsion additions.

drilling fluid is 42.5%.

It can be seen from Fig. 7(b)-(1), the pressure conduction data of the drilling fluid before plugging with nano-SiO₂ was attributed to 0.5 MPa within 46 min and balanced within 107 min after plugging, and the test results were brought into Eq. (1) to calculate the core permeability (*K*). According to Table 4, the core permeability before plugging was 1.54×10^{-4} mD, and the core permeability after plugging was 0.772×10^{-5} mD. From Eq. (2), it can be calculated that the plugging rate of drilling fluid after adding nano-SiO₂ is 49.8%. Compared with the on-site drilling fluid, the plugging capacity has improved, but it still cannot meet the plugging requirements of the Keziluoyi formation, which contains a large number of micro-nano formations.

It can be seen from Fig. 7(c)-(1) and Table 4 that according to the pressure conduction data before plugging of drilling fluid with Nano-core-emulsion, it is attributed to 0.5 MPa within 445 min, and equilibrium within 2684 min after plugging, and the equilibrium curve is nonlinear, indicating that there is a more obvious tight

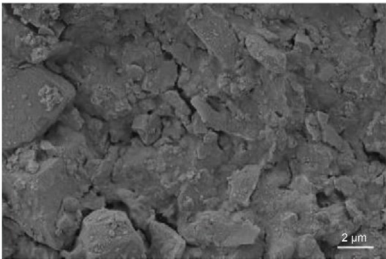
accumulation effect in the plugging process, which is also the reason why the permeability change curve is gradually flattened and the principle of multi-stage filling with low particle size is adopted so that the drilling fluid has higher plugging ability. Taking the test results into the core permeability *K* calculated by Eq. (1), the core permeability before plugging is 6.67×10^{-6} mD and the core permeability after plugging is 2.44×10^{-6} mD. From Eq. (2), it can be calculated that the plugging rate of drilling fluid with Nano-core-emulsion is 63.4%, which is 20.9% higher than that of on-site drilling fluid.

By comparing the plugging efficiency of drilling fluid of different formulas, it can be seen that for compact sandstone cores of the Keziluoyi Formation, the plugging efficiency of the Nano-core-emulsion is the highest; that is, the Nano-core-emulsion can expand the particle size gradation of drilling fluid, improve the plugging of micro-nano pores in the formation by drilling fluid, form a broad-spectrum plugging system, and realize efficient plugging of micro-nano pores (Chen et al., 2020; Martin et al.,

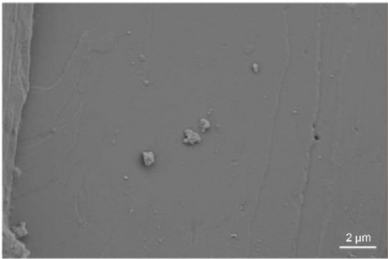
Table 3
Nano-core-emulsion optimizes performance change of potassium polysulfonate drilling fluid.

Number	ρ , g/cm ³	AV, mPa·s	PV, mPa·s	YP, Pa	G'/G''	Filtration, mL	Filter cake thickness, mm
0#	1.95	47	40	7	2.5/16	7.4	2.0
0# + 0.8% Nano-core-emulsion	1.95	38	32	6	2.5/17.5	3.6	1.2

SEM of Filter cake
Filter cake (0#)



Filter cake (0# + 0.8% Nano-core-emulsion)



(Note: (0#): 3% bentonite slurry + 0.4% NaOH + 0.3% PAC-LV + 0.1% AP-220 + 4% SMP-3 + 4% SPNH + 2.5% DYFT-2 + 2% Ft-1 + 3% KCl + barite; (0# + 0.8% Nano-core-emulsion): 3% bentonite slurry + 0.4% NaOH + 0.3% PAC-LV + 0.1% AP-220 + 4% SMP-3 + 4% SPNH + 2.5% DYFT-2 + 2% Ft-1 + 3% KCl + barite + 0.8% Nano-core-emulsion).

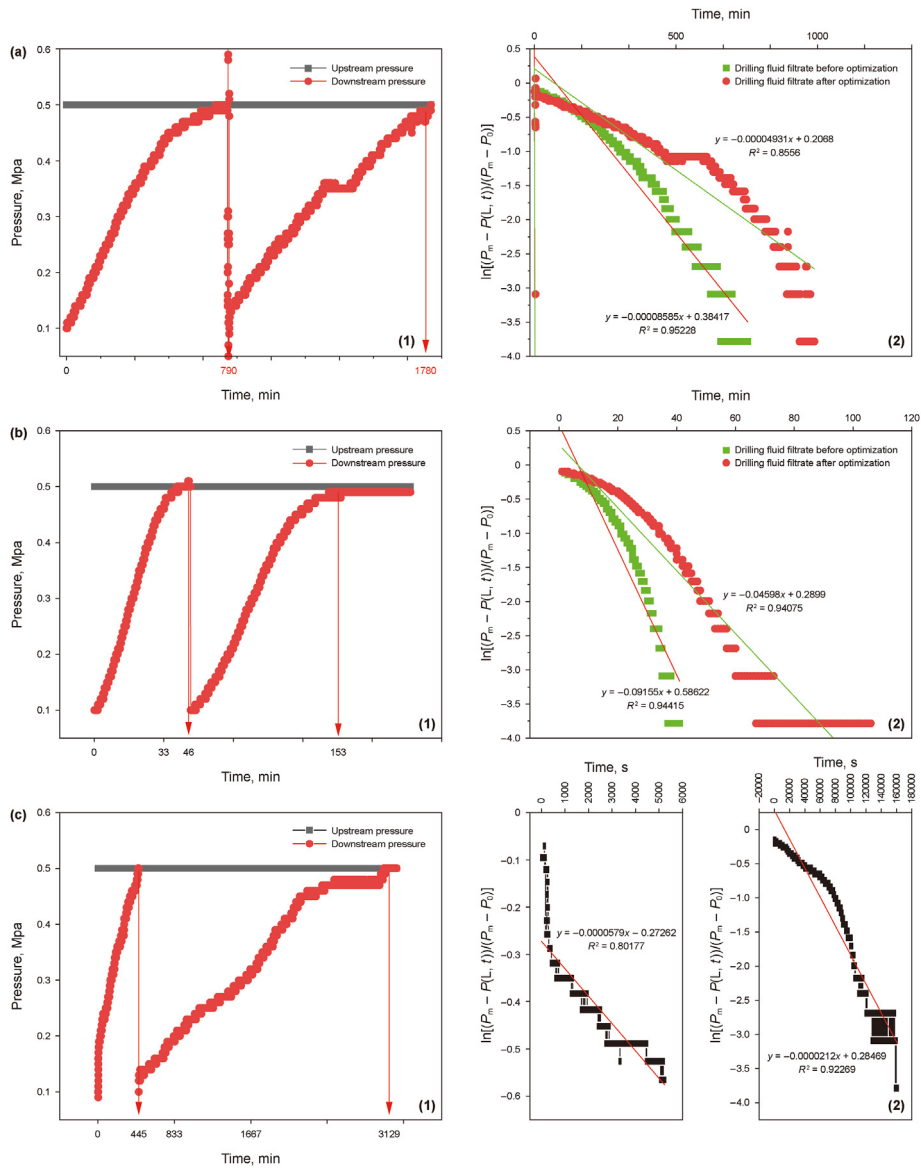


Fig. 7. (a) On-site drilling fluid filtrate pressure conduction test; (b) Adding 0.8% nano-SiO₂ drilling fluid filtrate pressure conduction test; (c) Adding 0.8% Nano-core-emulsion drilling fluid filtrate pressure conduction test.

Table 4
Permeability of Nano-core-emulsion before and after plugging.

	On-site drilling fluid	Drilling fluid after adding nano-SiO ₂	Drilling fluid after adding Nano-core-emulsion
Permeability of the core before plugging, mD	1.441×10^{-7}	1.54×10^{-4}	6.67×10^{-6}
Permeability of the core after plugging, mD	8.82×10^{-8}	0.772×10^{-5}	2.44×10^{-6}
Occlusion rate	42.5%	49.8%	63.4%

2023).

3.5. Plugging mechanism of nano-core-emulsion

The microscopic morphology of the filter cake was obtained by microscopic observation, and a CT scan was used to scan the core before and after the pressure conduction experiment (Tang et al., 2018; Yang et al., 2020). The blocking mechanism of the obtained Nano-core-emulsion is shown in Fig. 8.

It can be seen from Fig. 8 that after the optimization of the Nano-core-emulsion, the drilling fluid can produce a relatively dense filter cake with nano-SiO₂ particles attached to the surface, which confirms that the Nano-core-emulsion participates in the formation of the filter cake and fills the micropores formed during the accumulation of clay particles, making the filter cake itself have low permeability and preventing further leakage of water. Using CT scanning to observe the core slices before and after plugging, it is found that after the Nano-core-emulsion is plugged, the pores of the core are largely filled, and the pore volume gradually decreases with the invasion depth, which confirms that nano-plugging can

effectively prevent the further development of micro-fractures.

3.6. Field application of nano-core-emulsion

Well MT-1 is a risky exploration well deployed in the southwest of the Tarim Basin. The research results of this paper guide the design of the drilling fluid for this well. The specific application is shown in Fig. 9. Well MT-1 is still being drilled, and the performance of the used drilling fluid is good, and no downhole complications have occurred in the drilled section.

It can be seen from Fig. 9 that the current drilling depth of Well MT-1 has reached 5329 m, and there is no wellbore instability problem in the tight sandstone formation section (5078–5329 m). It is detected that after adding Nano-core-emulsion, the drilling fluid has good rheology and filtration wall building performance. In the 5000 m well section, the drilling fluid filtration rate remains stable at around 6.2 mL, the mud cake thickness is small at around 1.5 mm, and the high temperature stability is good. Field experiments show that the Nano-core-emulsion can effectively seal micro and nano fractures in the formation, and has great potential for

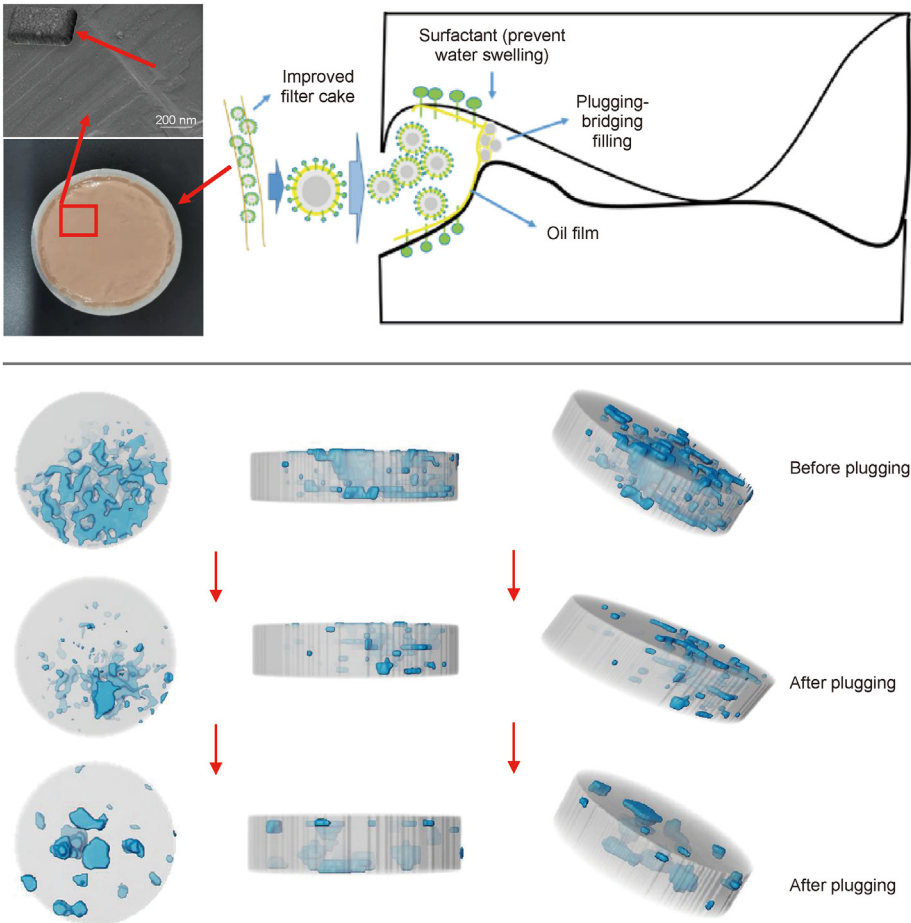


Fig. 8. The blocking mechanism of the Nano-core-emulsion.

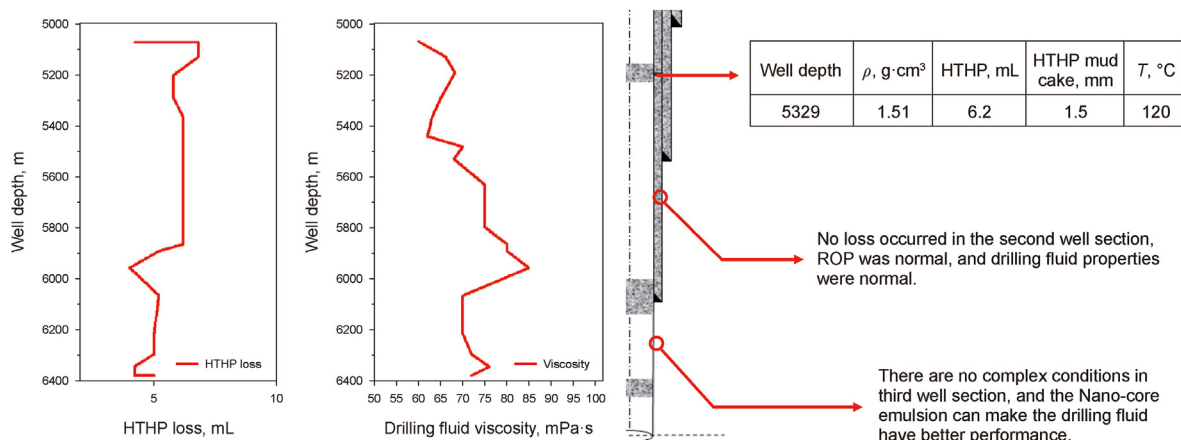


Fig. 9. Field application of Nano-core-emulsion in well MT-1.

field application.

4. Conclusions

We synthesized a novel nano-blocking reagent (Nano-core emulsion) for water-based drilling fluid, and characterized the properties of the Nano-core-emulsion itself. The influence of Nano-core-emulsion on the basic properties of bentonite slurry and drilling fluid was analyzed, and the plugging performance of Nano-core-emulsion was evaluated by the pressure conduction method. The results were as follows.

- (1) The Nano-core-emulsion is prepared as a core membrane structure of rigid nano-SiO₂ coated with an oil film, and the D_{50} particle size distribution is about 100 nm, which has good dispersion stability.
- (2) The 0.8% Nano-core-emulsion can reduce the bentonite slurry fluid loss by 40%, and the filter cake thickness by 74%. After the Nano-core-emulsion was added to the potassium polysulfonate drilling fluid, the fluid loss decreased by 52%, the formed filter cake was dense and tough, and the thickness decreased by 40%.
- (3) With a sealing rate of 63.4%, the Nano-core-emulsion can effectively seal the micro-nano pores of the compact sandstone of the Keziluoyi Formation in the southwestern Tarim Basin. During the plugging process, the nano-cores are bridged and accumulated, and the oil film adheres to the surface of the cores to effectively block water and build a “soft + hard” plugging body.
- (4) The Nano-core-emulsion has a good application prospect in the field and can effectively improve the plugging performance of drilling fluid.

The Nano-core-emulsion prepared in this paper effectively solves the problem of spontaneous agglomeration of nano-materials in an aqueous solution, realizes the whole process of plugging in the drilling fluid system, and solves the problem of wellbore instability in the southwestern block of the tower. The application of materials in ultra-deep wells provides a strong guide.

CRedit authorship contribution statement

Han-Xuan Song: Writing – original draft, Funding acquisition, Conceptualization. **Yun-Jin Wang:** Validation, Data curation. **Yan Ye:** Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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