



Original Paper

Research on small molecule wetting agent for drilling fluids applied in Antarctic drilling engineering

Ning Huang^a, Jin-Sheng Sun^{a, b, *}, Jing-Ping Liu^{a, **}, Kai-He Lv^a, Xue-Fei Deng^a, Hai-Jiang Yi^a

^a School of Petroleum Engineering, China University of Petroleum (East China), Qingdao, 266580, Shandong, China

^b CNPC Engineering Technology R & D Company Limited, Beijing, 102206, China

ARTICLE INFO

Article history:

Received 11 December 2024

Received in revised form

1 April 2025

Accepted 1 April 2025

Available online 3 April 2025

Edited by Jia-Jia Fei

Keywords:

Antarctic drilling engineering

Drilling fluid

Wetting agent

Performance evaluation

Mechanism study

ABSTRACT

Antarctica contains numerous scientific mysteries, and the Antarctic ice sheet and its underlying bedrock contain important information about the geological structure of Antarctica and the evolutionary history of the ice sheet. In order to obtain the focus of these scientific explorations, the Antarctic drilling engineering is constantly developing. The drilling fluid performance directly determines the success or failure of drilling engineering. In order to enhance the poor performance for drilling fluids due to poor dispersion stability and easy settling of organoclay at ultra-low temperatures, the small-molecule wetting agent (HSR) for drilling fluid suitable for Antarctica was prepared by oleic acid, diethanolamine and benzoic acid as raw materials. Its chemical structure, properties and action mechanism were investigated by various experimental methods. The experimental results showed that 2% HSR could improve the colloidal rate for drilling fluid from 6.4% to 84.8%, and the increase rate of yield point was up to 167%. Meanwhile, it also made the drilling fluid excellent in shear dilution and thixotropy. In addition, 2% HSR could increase the density from 0.872 to 0.884 g/cm³ at −55 °C. And the drilling fluid with 2% HSR had a good thermal conductivity of 0.1458 W/(m·K) at −55 °C. This study gives a new direction for the research of drilling fluid treatment agents suitable for the Antarctic region, which will provide strong support for the scientific exploration of the Antarctic region.

© 2025 The Authors. Publishing services by Elsevier B.V. on behalf of KeAi Communications Co. Ltd. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

1. Introduction

Antarctica contains countless scientific mysteries and rich resources. It is important to understand the geological structure of Antarctica, to reveal the evolutionary history of the ice sheet and to assess future climate change. Obtaining samples of ice cores and bedrock beneath the ice sheet will be the key to understanding this field. Therefore, Antarctic drilling technology has gradually been developed. However, in the process of Antarctic drilling, the ultra-low temperature and complex geological environment pose great challenges to drilling engineering. Drilling fluid is an irreplaceable and important part of drilling engineering, which directly determines the success or failure of drilling engineering (Rohling

et al., 2009; Turner et al., 2020; Zhang et al., 2014).

Currently, Antarctic drilling fluids mainly suffer from insufficient resistance to ultra-low temperatures, poor rheological properties, poor colloidal stability and low environmental friendliness (Popp et al., 2014; Sheldon et al., 2014a; Sun et al., 2022a). Sheldon et al. conducted ice core drilling in Antarctica Greenland, using a blend of ESTISOL™ 240 and COASOL™, which showed excellent hydrophobicity at ultra-low temperatures and could be well separated from ice debris (Sheldon et al., 2014b). But its viscosity was high, which could have a negative impact on drilling operations. Japan used butyl acetate as drilling fluid for drilling at Dome Fuji in Antarctica and achieved good results. However, it was found that butyl acetate had a pungent odor and poor environmental protection (Motoyama et al., 2021). The Glaciology Group of the Copenhagen University used aqueous ethylene glycol solution with excellent results in warm ice drilling. However, its low-temperature resistance was insufficient and it was prone to freezing under low temperature conditions, which prevented drilling operations from

* Corresponding author.

** Corresponding author.

E-mail addresses: sunjsdri@cnpc.com.cn (J.-S. Sun), liujingping20@126.com (J.-P. Liu).

being carried out (Johnsen et al., 2007).

In the drilling application for high-temperature formations and deepwater (Martyushev and Govindarajan, 2022; El-Masry et al., 2023), organoclay has excellent effect as rheological additive in drilling fluids (Mahmoud et al., 2023; Shi et al., 2019). However, some scholars found that organoclay also had significant effect in improving the rheology of Antarctic drilling fluids (Huang et al., 2023), but its dispersion stability in base oil was poor and easy to settle at ultra-low temperatures, resulting in poor performance. By increasing the lipophilicity and dispersion properties for organoclay in base oil under ultra-low temperature conditions, the rheological performance for drilling fluid can be ensured. Thereby, it is crucial to prepare wetting agent to improve the organoclay dispersibility under ultra-low temperature conditions.

For the study on the wetting agent for conventional drilling fluids, domestic and foreign experts and scholars have done a lot of researches (Skalli et al., 2006; Quintero, 2002; Mahmoud and Dardir, 2011; Wang et al., 2021). Menezes et al. chose EZ-Mul wetting agent to improve the contact angle of quartz surface from 10° to 145° , and the quartz surface became strongly lipophilic (Menezes et al., 1989). Paswan et al. synthesized an anionic wetting agent based on sunflower seed oil as raw material, which could diminish the interfacial tension from 102.5 to 25.35 mN/m, and increase the contact angle from 32.50° to 82.3° , which effectively improved the emulsion stability and oil wettability for drilling fluid (Paswan and Mahto, 2020). Ni et al. synthesized a modified lithium saponite with hydrophobic wettability (Ni et al., 2023). The results showed that 0.1 wt% modified lithium saponite increased the contact angle from 0° to 78.9° and diminished interfacial tension from 32.8 to 13.5 mN/m, as well as raised the emulsion stability. Murtaza et al. addressed the problem of easy settlement for barite by adding wetting agent to the drilling fluids to enhance the lipophilicity of the barite surface and improve the dispersion of barite in the oil (Murtaza et al., 2021). Sui et al. constructed drilling fluid system by selecting the key additives (Sui et al., 2018). When the dosage of wetting agent was 0.4%, the volume of barite was up to 90 mL (standing for 170 min), which improved the lipophilic property of barite. Simultaneously, the drilling fluid rheology was also improved. Lei et al. optimized a wetting agent that could still achieve a volume of 87 mL for barite after standing 420 min, which had good suspension performance for barite (Lei and Li, 2018). In summary, these studies on wetting agents of drilling fluids are all aimed at conventional drilling. They have not been applied to ultra-low temperature drilling fluids suitable for Antarctic drilling. In addition, by adding some conventional wetting agent products to Antarctic drilling fluids, it was found that they mainly suffered from poor resistance to ultra-low temperatures and insignificant improvement of the colloidal rate for organoclay under ultra-low temperature conditions.

Therefore, in response to the problems of insufficient ultra-low temperature resistance and poor wetting performance of existing wetting agents on organoclays, this article described a small molecule wetting agent (HSR) prepared by oleic acid, diethanolamine and benzoic acid. It can withstand temperatures up to -55°C and has good wetting performance for organoclays under ultra-low temperature conditions. It was characterized by infrared spectroscopy scanning and ^1H NMR testing. In addition, the influence of HSR on the lipophilicity and dispersibility of organoclay was evaluated. Meanwhile, its influence on the density and rheological performance was also researched. Eventually, the action mechanism of HSR was analyzed by studying its effect on the particles counts and mean of organoclay, the Zeta potential and microscopic morphology of drilling fluid. It is very significant to further promote the advance of scientific research for Antarctic.

2. Materials and methods

2.1. Materials

Oleic acid (AR) and benzoic acid (AR) were supplied by Shanghai Aotouli Biotechnology Co., Ltd; Diethanolamine (AR) was provided by Shanghai Saihan Technology Co., Ltd; Baker's organoclay was supplied by Qingdao Dingshenghe Technology Co., Ltd; Concentrated sulfuric acid was obtained from Qingdao Hualai Trading Co., Ltd; The rubber ring was purchased from Guangdong Haohuan Sealing Products Co., Ltd; Dynamul-P was purchased from Kaiping Lianji Chemical Co., Ltd; EM-152 was from Tianjin Xiongguan Technology Development Co., Ltd; HCFC-141b was supplied by Shanghai Ruiyi Environmental Protection Technology Co., Ltd. The physical and chemical properties of different materials are shown in Table 1.

2.2. Wetting agent preparation

60 g of oleic acid and 18.5 g of diethanolamine were added into a three-necked flask, introduced nitrogen for 20 min and heated to 170°C in an oil bath; Subsequently, the above reaction system was reacted at 170°C for 4 h. After the reaction was completed, the reaction system was left to be cooled down to $60\text{--}80^\circ\text{C}$; Then, 0.56 g of concentrated sulfuric acid and 32.3 g of benzoic acid were added to the reaction system, and nitrogen gas was introduced for 20 min. The reaction was continued for 3 h at 110°C and cooled to room temperature to obtain the wetting agent (HSR). Fig. 1 showed its preparation process.

2.3. Wetting agent characterization

2.3.1. Infrared spectral analysis

The wetting agent sample was analyzed by 983G Fourier transform infrared spectroscopy scanner (Beijing Bruker Technology Co., Ltd.). Its purpose is to verify whether the functional groups designed in the wetting agent have successfully appeared. And it was studied by the ATR method, and the instrument parameters was set to a resolution of 4 cm^{-1} and a wavenumber range of $400\text{--}4000\text{ cm}^{-1}$.

2.3.2. Nuclear magnetic hydrogen spectroscopy

The HSR was characterized by ^1H -NMR (Shanghai Maikewei Semiconductor Technology Co., Ltd), and the purpose of the experiment is to verify whether the types of hydrogen atoms present in the designed wetting agent have successfully emerged. The solvent was deuterated chloroform.

2.4. Base fluid preparation

Aviation kerosene and white oil were formulated into base oil in the ratio of 7:3 by volume. Subsequently, 3% Baker's organoclay was added to it and stirred for 25 min on homogeniser to make it fully dispersed, resulting in the base fluid preparation.

Drilling fluid formulation: 70% aviation kerosene (No. 4) + 30% white oil (QY) + 3% Baker's organoclay + 2% HSR.

2.5. Performance evaluation of wetting agent

2.5.1. Contact angle

The drilling fluid was prepared by adding wetting agent (HSR) to the base fluid. The glass slides were immersed in the drilling fluids and frozen (-55°C) for 24 h in low-temperature test chamber. And

Table 1
Physical and chemical properties of materials.

Materials name	Physical and chemical properties
Oleic acid	Appearance: colorless oily liquid; Density: 0.89 g/cm ³ ; Boiling point: 360 °C; Solubility: soluble in benzene and chloroform.
Benzoic acid	Appearance: white needle shaped; Boiling point: 249.2 °C; Solubility: slightly soluble in water.
Diethanolamine	Density: 1.097 g/cm ³ ; Boiling point: 268.4 °C; Solubility: soluble in water and ethanol, insoluble in ether and benzene.
Baker's organoclay	Appearance: light yellow powder; Dispersibility: easy to disperse in organic solvents such as white oil and aviation kerosene.
Concentrated sulfuric acid	Appearance: colorless and transparent liquid; Concentration: 98%; Boiling point: 337 °C; Density: 1.84 g/cm ³ .
Dynamul-P	Appearance: yellow-brown liquid; Solubility: easily soluble in organic solvents such as aviation paraffin.
EM-152	Appearance: light yellow oily liquid; Solubility: easily soluble in white oil, aviation paraffin and other organic solvents.
HCFC-141b	Appearance: colorless transparent liquid; Density: 1.24 g/cm ³ ; Solubility: miscible with organic solvents such as aviation paraffin.

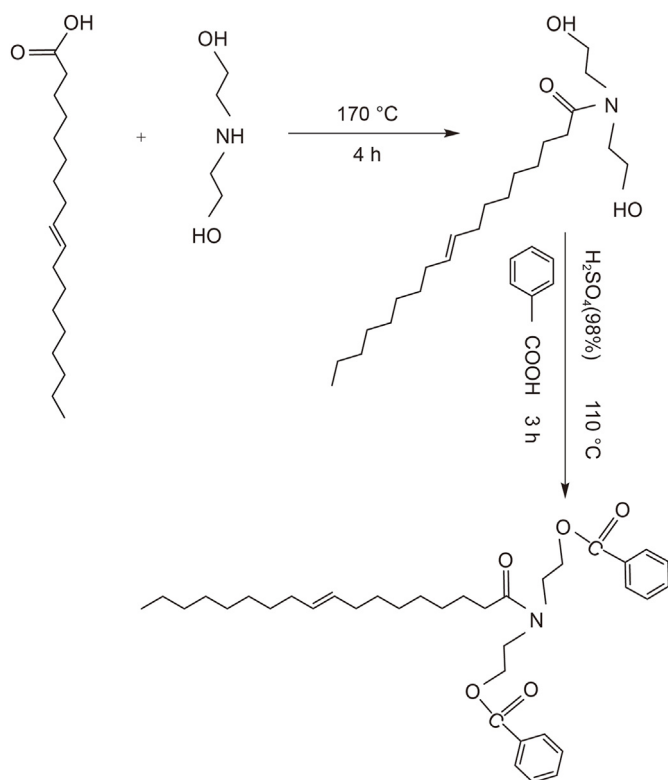


Fig. 1. Preparation process of HSR.

the lipophilicity of the specimens was subsequently characterized by OCA11 contact angle instrument (Dataphysics GmbH, Germany). The purpose of this experiment is to test the lipophilicity of the prepared wetting agent and explore its compatibility with drilling fluids. The measurement range is 0°–180°. The contact angle of the oil droplets was tested by the droplet method on the glass slide. Five points in different areas of each specimen surface were measured and the contact angle was their average value.

2.5.2. Colloidal rate test

Different concentrations of wetting agent (HSR) were added to the base fluid and stirred for 15 min by Hamilton Beach N5000 stirrer. Then the drilling fluid was filled into stoppered cuvette. Lastly, the colloid rate was measured after standing in a low-temperature thermostat for 90 min, 16 h, and 24 h, respectively (Ghavami et al., 2018; da Silva et al., 2023). Its purpose is to test the effect of HSR on the colloid rate of drilling fluid, in order to explore the wetting performance of HSR in drilling fluid. The calculation formula is as follows:

$$Cr = \frac{V}{100.0} \times 100\% \quad (1)$$

In the formula, Cr—colloid rate, percentage;
V—colloidal volume, mL.

2.5.3. Observation of microscopic dispersion morphology

Firstly, the drilling fluid with HSR was stirred for 25 min to make it well dispersed. Secondly, the prepared samples were allowed to stand in a constant temperature bath (−55 °C) for 16 h. Finally, the effect of HSR on the microscopic dispersion morphology for drilling fluid was studied by CX33 optical microscope (Olympus Co., Ltd., Japan). The microscopic morphology of the drilling fluid was observed under microscope to analyze the influence of HSR on the wetting properties of the drilling fluid.

2.5.4. Evaluation of rheological properties

2.5.4.1. The effect of HSR on rheological parameters for drilling fluid. According to the American Petroleum Institute (API) standards (API, 2009), the rheological parameters were tested by ZNN-D6 ultra-low temperature six-speed viscometer (Shanghai Kence Instrument Co., Ltd.) under different temperature conditions, respectively. The rheological parameters were calculated as (Zhao et al., 2017; Leusheva et al., 2021; Meng et al., 2012):

$$AV = 0.5 \times \Phi 600 \quad (2)$$

$$PV = \Phi 600 - \Phi 300 \quad (3)$$

$$YP = 0.5 \times (2 \times \Phi 300 - \Phi 600) \quad (4)$$

where $\Phi 600$ —reading at 600 r·min^{−1}.

$\Phi 300$ —reading at 300 r·min^{−1};
AV—apparent viscosity, mPa·s;
PV—plastic viscosity, mPa·s;
YP—yield point, Pa.

2.5.4.2. The effect of HSR on shear dilution, thixotropy and flow pattern for drilling fluid. The drilling fluid with wetting agent (HSR) were allowed to stand in DW-60W151EU1 Low-Temperature Freezer (Qingdao Haier Special Freezer Co., Ltd., China) for 16 h (−55 °C), and then the following parameters were tested by Haake MARS rheometer (Thermo Fisher Scientific, Inc., USA), respectively. As the shear rate changed, viscosity and shear stress were measured, respectively; Three stage thixotropy curve of drilling fluid: firstly, the drilling fluid samples were subjected to low-speed shear for 60 s (0.01 s^{−1}), followed by high-speed shear for 60 s (200 s^{−1}), and finally low-speed shear for 60 s (0.01 s^{−1}) (He et al., 2023; Meng et al., 2023). The objective of conducting the aforementioned experiments is to analyze the cuttings-carrying capacity

and suspension properties of drilling fluids with the addition of HSR, ensuring safe and efficient drilling. The test temperature was 4 °C.

2.5.4.3. The effect of HSR on drilling fluid modulus. Modulus is an important parameter to characterize the internal structure strength for drilling fluids. The modulus variation was tested by Haake MARS rheometer (Thermo Fisher Scientific, USA) for drilling fluid with wetting agent (HSR). The modulus includes elastic modulus (G') and viscous modulus (G''). By testing the elastic modulus and viscous modulus of drilling fluid containing HSR, the impact of HSR on the internal structural strength of the drilling fluid was analyzed.

2.5.5. Drilling fluid density test

Density is one of the important properties of drilling fluid. Reasonable drilling fluid density will play an important role in wellbore stability. Firstly, the drilling fluid with HSR were stirred on a high-speed stirrer for 25 min. Subsequently, the influence of HSR on the density was evaluated by MDY-2 liquid density meter (Shanghai Fangrui Instrument Co., Ltd.) under different temperature conditions.

2.5.6. Rubber ring corrosion test

Two seals materials (silicone rubber and fluororubber) were put into the drilling fluid with HSR at -55 °C for a specific time to freeze, respectively. The variations of the outer diameter and mass for different rubbers with time were tested to investigate the corrosion of HSR on seals made of different materials. The purpose of the experiment is to investigate the effect of drilling fluid with HSR on the corrosiveness of sealing elements for drilling tools during Antarctic drilling.

2.5.7. Evaluation of the environmental friendliness for HSR

The Antarctic region has high requirements for the environmental friendliness of drilling fluid additives. For this reason, the biodegradability of HSR was tested according to test standards (Chang et al., 2019; Pi et al., 2015). The evaluation indicators are the values of BOD₅ and COD.

2.5.8. Evaluation of thermal conductivity for drilling fluid

The heat transfer of drilling fluid has a significant impact on wellbore stability during drilling operations in the Antarctic ice layer. Therefore, evaluating its thermal conductivity is crucial. The thermal conductivity of drilling fluid with 2% HSR was evaluated by the TC3000E thermal conductivity meter (Xi'an Xiaxi Electronic Technology Co., Ltd., China) under different temperature conditions. The temperature is controlled by a low-temperature circulation pump.

2.6. Mechanism study

2.6.1. The effect of HSR on counts and mean for organoclay particles

Firstly, the prepared drilling fluid was stirred on stirrer for 10 min; Subsequently, the drilling fluid was put into a low-temperature cooling circulating pump; The counts and mean of the organoclay particles were measured by Particle Track G600 Fbrm (Mettler Ltd., Switzerland) at different temperatures. The purpose is to investigate the impact of HSR on the wettability of drilling fluids at ultra-low temperature, and the experimental temperature was controlled by cryogenic cooling circulating pump during the experiment.

2.6.2. The influence of HSR on Zeta potential for drilling fluid

The prepared drilling fluid was stirred on stirrer for 25 min, and

then the samples were allowed to stand in DW-60W151EU1 Low-Temperature Freezer (Qingdao Haier Special Freezer Co., Ltd., China) for 16 h (-55 °C). Finally, the Zeta potential values were tested by SZ901 Zetasizer (Beijing Stawo Technology Co., Ltd., China). The improvement degree of HSR on the wettability of drilling fluid was evaluated by measuring the magnitude of the drilling fluid potential. Each sample was tested three times, and the Zeta potential value was their average value (Liu F. et al., 2021; Liu et al., 2022).

2.6.3. The influence of HSR on the micromorphology for drilling fluid

The microstructure of the samples surface is usually characterized by cryo-scanning electron microscopy. The Helios G4 UC cryo-scanning electron microscope (Thermo Fisher Scientific, Inc., USA) was used to study the microscopic morphology of base fluid and drilling fluid with HSR, revealing the action mechanism for HSR in the drilling fluid.

3. Experimental results and analysis

3.1. HSR characterization

3.1.1. Infrared spectral analysis

The infrared spectrum for HSR is shown in Fig. 2. The corresponding peaks at the vicinity of 2923 and 2853 cm^{-1} are anti-symmetric and symmetric peaks of C–H, respectively. The peak near 1720 cm^{-1} corresponds to the vibration peak of C=O for carboxyl and ester groups. The vibration peak near 1610 cm^{-1} corresponds to the vibration peak of C=C on oleic acid. The peak near 1268 cm^{-1} is the vibration peak of C–N. The peak near 1172 cm^{-1} is the stretching vibration peak of C–O–C (Zhong et al., 2023). The infrared spectrum confirmed the successful synthesis for HSR.

3.1.2. Nuclear magnetic analysis

The results of ^1H NMR for HSR in CDCl_3 are shown in Fig. 3.

Peak a (0.88 ppm) is the proton peak of CH_3 at the end of oleic acid; Peak b (1.26 ppm) is the proton peak of CH_2 for oleic acid; Peak c (1.58 ppm) is the proton peak of CH_2 in the $-\text{CH}_2\text{CH}_2\text{CO}-$ structure; Peak d (1.95 ppm) is the proton peak of CH_2 in the structure of

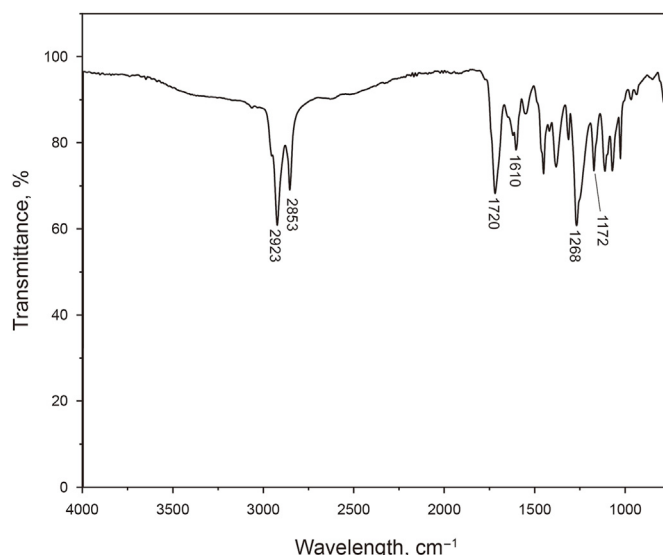


Fig. 2. Infrared spectrum of HSR.

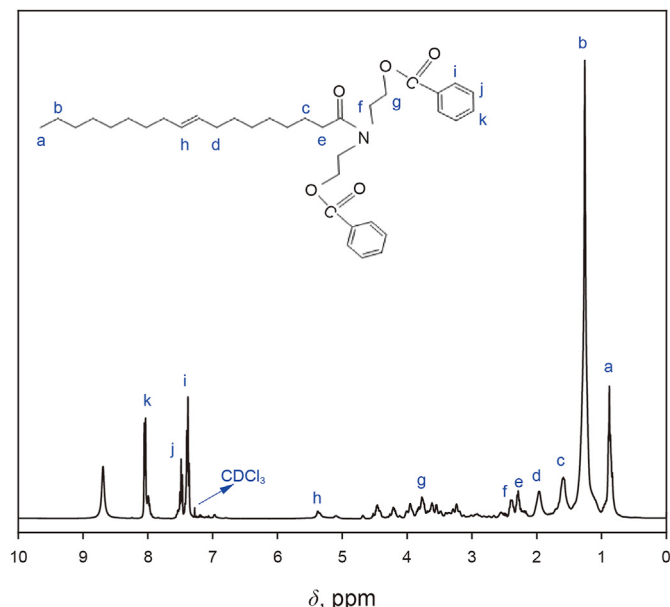


Fig. 3. ^1H NMR diagram of HSR.

$-\text{CH}_2\text{CH}=\text{CHCH}_2-$; Peak e (2.27 ppm) is the proton peak of CH_2 in the $-\text{CH}_2\text{CO}-$ structure; Peak f (2.41 ppm) and peak g (3.74 ppm) are proton peaks of CH_2 for diethanolamine; Peak h (5.36 ppm) is the proton peak of $\text{CH}=\text{CH}$; Peak i (7.36 ppm), peak j (7.49 ppm) and peak k (8.04 ppm) are proton peaks on the benzene ring; The peak (7.26 ppm) is the solvent peak of CDCl_3 . The ^1H NMR results indicate that the synthesis of HSR is successful.

3.2. Performance evaluation of HSR

3.2.1. The effect of HSR on contact angle for organoclay

The organoclay lipophilicity is crucial for improving the drilling fluids properties. The influence of different concentrations for wetting agent (HSR) on the contact angle of organoclay were investigated (Fig. 4).

The study showed that the contact angle for organoclay was 17.2° before adding HSR to the base fluid, while the contact angle of organoclay reached 14.6° , 9.4° and 6.4° after adding 1%, 2% and 3% HSR to the base fluid, respectively. This indicated that HSR improved the organoclay lipophilicity. And as the concentration of HSR increased, the contact angle of organoclay gradually decreased.

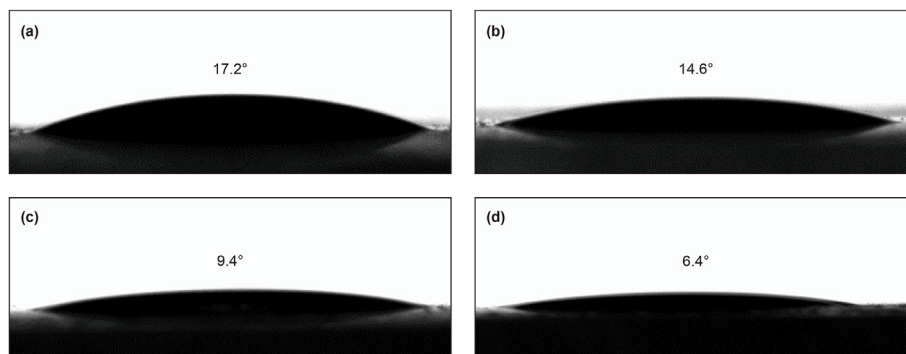


Fig. 4. Effect of HSR concentration on contact angle of organoclay ((a) 0%; (b) 1%; (c) 2%; (d) 3%).

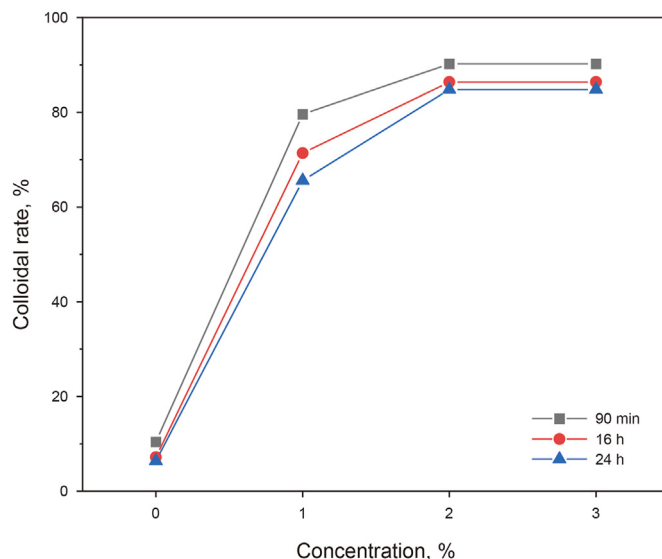


Fig. 5. Variation of colloid rate for drilling fluid with HSR concentration.

3.2.2. The effect of HSR on the dispersibility for drilling fluid

3.2.2.1. The effect of HSR on colloidal rate for drilling fluid. The effect of HSR on the dispersion performance for drilling fluid was evaluated by investigating the colloidal rate at -55°C .

It was found that the colloidal rate was only 6.4% at -55°C when no HSR was added to the drilling fluid. However, 2% HSR could make the colloid rate (standing for 24 h) reach 84.8%, which was a significant effect. Furthermore, as the concentration of HSR increased, the colloid rate gradually increased (Fig. 5).

3.2.2.2. The effect of HSR on the microscopic morphology for drilling fluid. In this section, the microscopic morphology of drilling fluid with different concentrations for HSR was observed separately by optical microscope, and the change of micro dispersion morphology for drilling fluid was investigated (Fig. 6).

The organoclay showed the phenomenon of particle aggregation, indicating that its lipophilicity was poor under the ultra-low temperature condition, and it was not able to be effectively dispersed in the base oil (Fig. 6(a)). After adding 1% HSR to the base fluid, the aggregation phenomenon was obviously reduced, but there was still the aggregation phenomenon of small particles into large particles (Fig. 6(b)). However, after the HSR concentration reached 2%, the large particles were significantly reduced and the organoclay dispersion became more and more homogeneous. The analysis may be that HSR can adsorb onto the organoclay particles

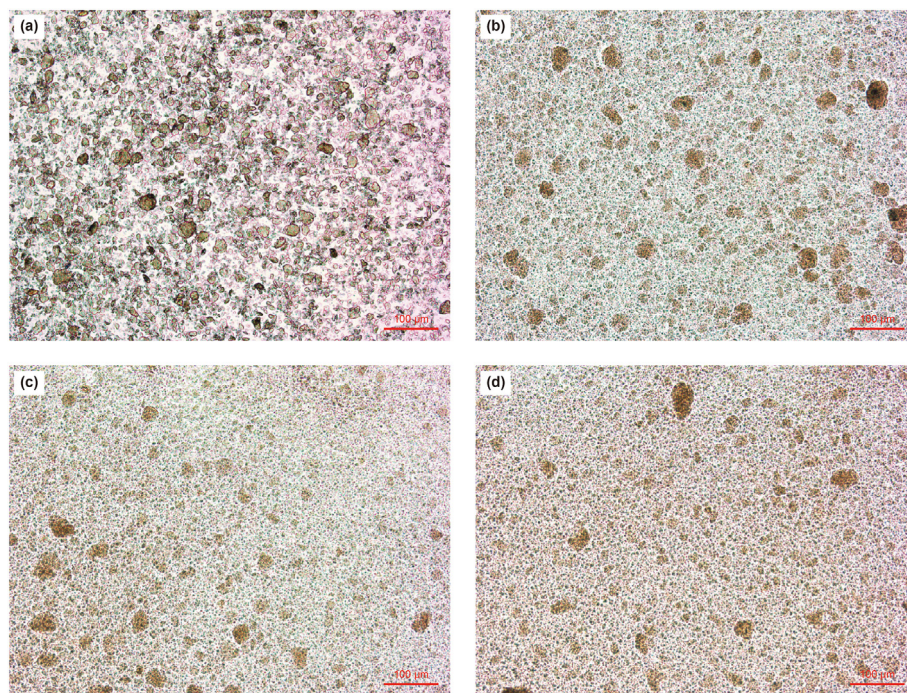


Fig. 6. Effect of HSR concentration on the drilling fluid microstructure ((a) 0%; (b) 1%; (c) 2%; (d) 3%).

through polar groups to form intermolecular forces, which prevents the organoclay aggregation. And as the dosage of HSR improved, the force became stronger, which made the organoclay particles more dispersed in the drilling fluid.

3.2.3. The effect of HSR on the drilling fluid rheology

3.2.3.1. The effect of HSR concentration on viscosity, yield point and gel strength. As shown in Fig. 7, HSR was able to increase the yield point at ultra-low temperatures. Additionally, 2% HSR could improve the yield point to 2.0 Pa at -55°C . At the same time, the gel strength of drilling fluid increased from 0 to 1.0 Pa. These changes will significantly improve the efficiency of drilling fluid in carrying and suspending ice and rock debris.

3.2.3.2. The effect of HSR concentration on shear dilution, thixotropy and flow pattern for drilling fluid. Shear dilution and thixotropy are important properties for drilling fluids, which can reflect the suspension and cuttings-carrying for drilling fluids. The better the shear dilution and thixotropy, the better the suspension and cuttings-carrying for the drilling fluids. In this section, the effects of HSR on the shear dilution characteristics, “three-stage” thixotropy and flow pattern for drilling fluids were researched.

From Fig. 8, as the shear rate improved, the viscosity basically did not change when HSR concentration was 0. Meanwhile, as the shear rate enhanced, the shear stress increased linearly, indicating that the drilling fluid belonged to the Newtonian fluid. In addition, when the HSR concentration is 0, the viscosity did not vary significantly under different shear rate conditions, and the thixotropic property was poor. However, with the shear rate improved, the viscosity of drilling fluid containing HSR decreased rapidly, and the relationship was nonlinear. This indicated that HSR enabled drilling fluid to have the non-Newtonian properties. It was worth mentioning that HSR made the drilling fluid have high viscosity at low shear rate, while the viscosity decreased rapidly at high shear rate. However, when the low shear rate was given again, the viscosity of drilling fluid quickly recovered. This showed that HSR also

made the drilling fluid have excellent thixotropy.

3.2.3.3. The impact of HSR dosage on drilling fluid modulus. Modulus is an important indicator for characterizing the internal structural strength for drilling fluids (Kim et al., 2003). The modulus (G' , G'') variations with the increase of shear stress were explored respectively under the action of HSR.

For the base fluid, when the shear stress was higher than about 7×10^{-4} Pa, G'' started to be larger than G' , indicating that its internal structure started to be gradually destroyed (Fig. 9(a)). For the drilling fluid with 2% and 4% HSR, G'' started to be larger than G' when the shear stress was higher than about 9.5×10^{-4} and 10.7×10^{-4} Pa, respectively (Fig. 9(b) and (c)). It showed that HSR could obviously improve the internal structural strength for drilling fluid, enhancing its rheological properties.

3.2.4. The influence of HSR on drilling fluid density

Reasonable drilling fluid density is one of the prerequisites for ensuring safe and efficient drilling (Ahmadi et al., 2018; Liu N. et al., 2021), while improper density will lead to drilling accidents. In this section, the impact of HSR on the density was investigated under different temperature conditions.

Fig. 10 showed that as the temperature decreased, the density gradually increased. In addition, 2% HSR could increase the density from 0.872 to 0.884 g/cm³ at -55°C . This indicated that HSR was beneficial for increasing the density of drilling fluid, and thus it can be considered to have the effect of weighting drilling fluid. It also indicated that its presence could reduce the use of weighting agents and diminish drilling costs.

3.2.5. Effect of HSR on the corrosiveness of rubber rings

During polar drilling, rubber materials are usually used as sealing components for drilling tools (Liu et al., 2016). However, rubber materials are inevitably corroded by drilling fluids, which in turn may affect the drilling tools performance. For this reason, the effect of HSR on the two rubber materials was studied (Fig. 11).

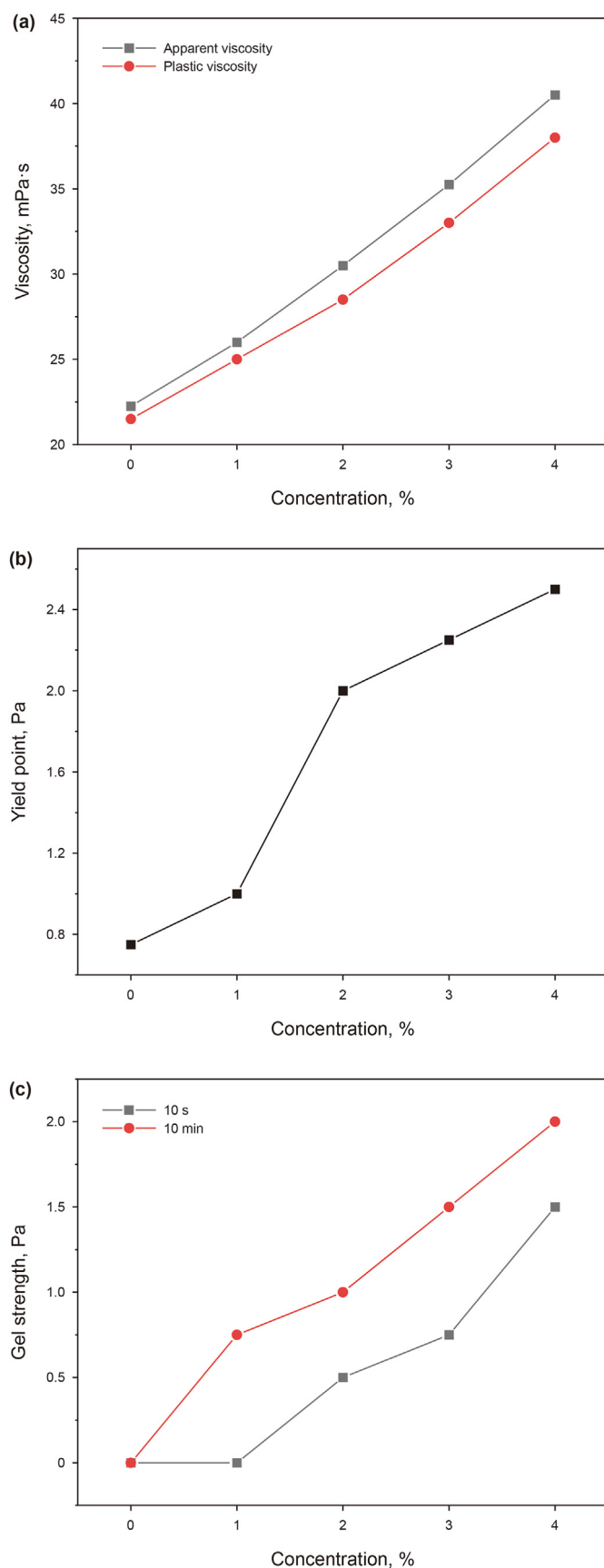


Fig. 7. Variation of rheological parameters for drilling fluid with HSR concentration at -55 °C ((a) viscosity; (b) yield point; (c) gel strength).

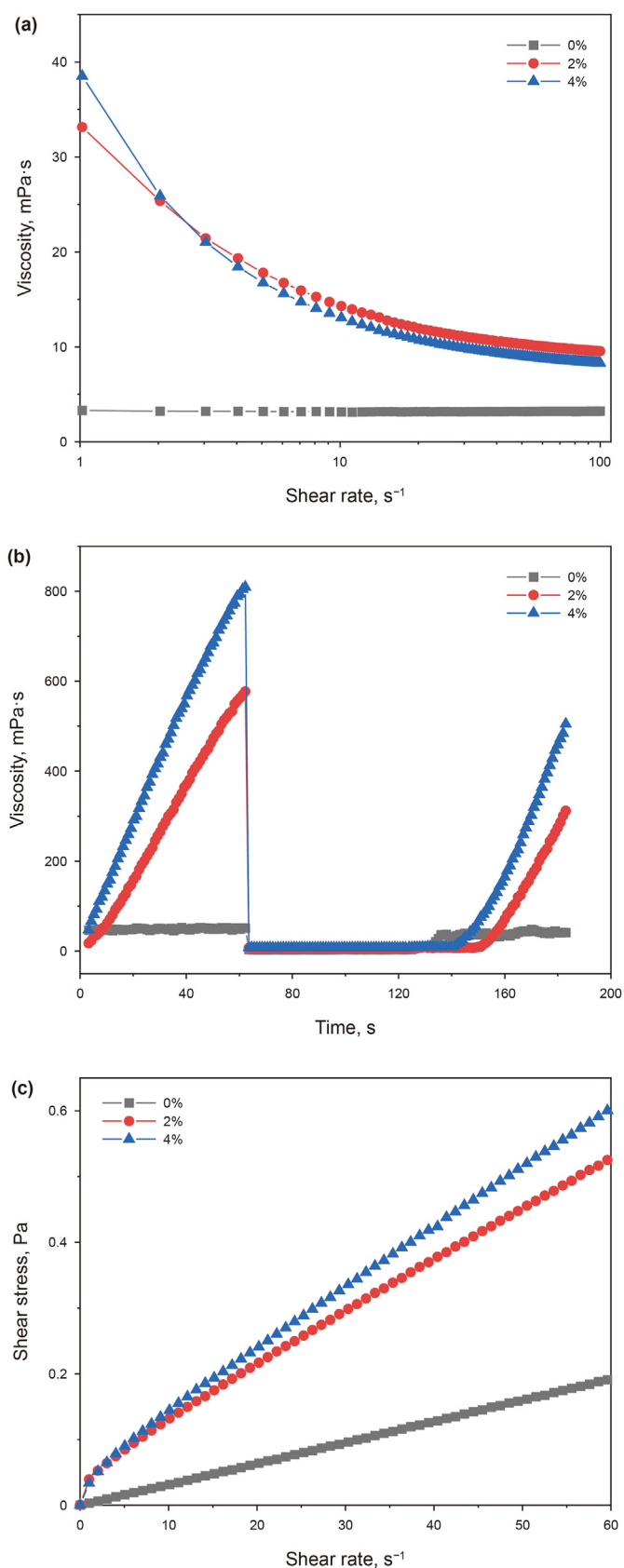


Fig. 8. Effect of HSR concentration on shear dilution, thixotropy and flow pattern for drilling fluid ((a) shear dilution; (b) thixotropy; (c) flow pattern).

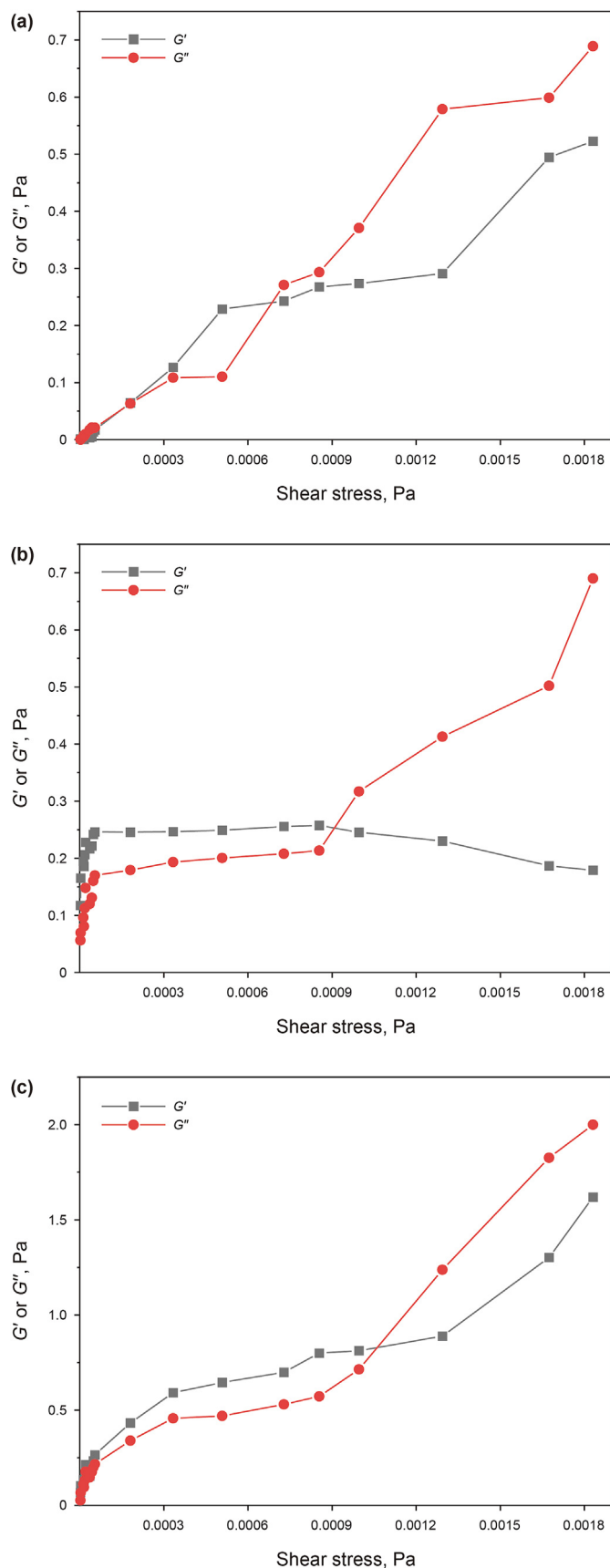


Fig. 9. Effect of HSR concentration on drilling fluid modulus ((a) 0%; (b) 2%; (c) 4%).

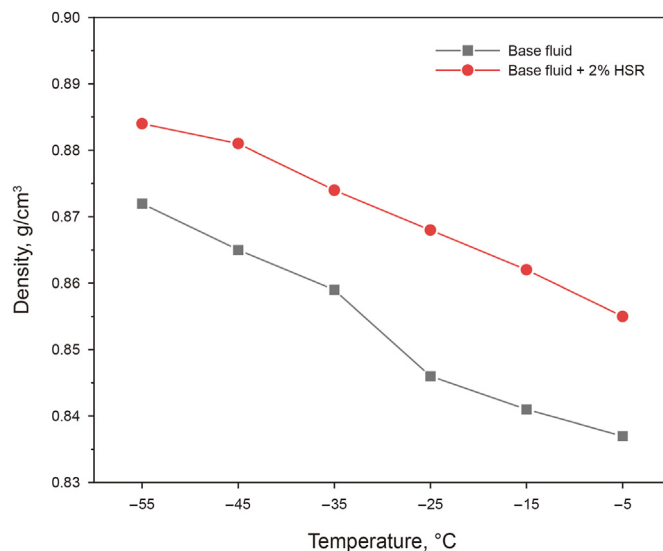


Fig. 10. Effect of HSR on drilling fluid density.

Compared to the base fluid, after soaking silicone rubber in drilling fluid with 1% HSR for 24 h, its mass and outer diameter increased by 0.005 g and 0.018 cm, respectively. Meanwhile, the quality of fluororubber did not change, and the outer diameter increased by 0.001 cm. This indicated that HSR had less corrosiveness to different rubbers.

3.2.6. Compatibility analysis of HSR with conventional additives

The compatibility of HSR with other additives is also one of the important methods to evaluate its performance. The compatibility of HSR with weighting agent (HCFC) and rheology modifier (DA) in drilling fluids was explored.

As can be seen in Fig. 12, the colloid rate of drilling fluid with HSR was improved by adding HCFC and DA, which indicated that HSR had good compatibility with other additives.

3.2.7. Performance comparison of HSR with commercial wetting agents

On the basis of evaluating the performance for HSR, the performance of two commercial wetting agents (EM-152 and Dynamul-P) was also evaluated at $-55\text{ }^{\circ}\text{C}$. The experimental results are shown in Fig. 13.

The study showed that EM-152 and Dynamul-P respectively achieve the colloid rate of 69.6% and 16% for drilling fluid at $-55\text{ }^{\circ}\text{C}$ (standing for 24 h). This indicated that compared to HSR, their performance was relatively low. In addition, by evaluating the prices of HSR and commercial products, it was found that the cost of HSR was lower than that of traditional commercial products.

3.2.8. Evaluation of the environmental friendliness for HSR

Research suggested that when the value of BOD_5/COD was greater than 25%, the material was considered environmentally friendly. For HSR, the biodegradability test revealed that the value of BOD_5 was 9751.4 mg/L, and the value of COD was 13465.6 mg/L. Therefore, the value of BOD_5/COD was 72.4%, which was in accordance with the environmental protection standard. It is an environmentally friendly new material.

3.2.9. The analysis of long-term stability for drilling fluid

According to the study in Section 3.2.2, under the condition of $-55\text{ }^{\circ}\text{C}$, 2% HSR was able to achieve the colloid rate (standing for

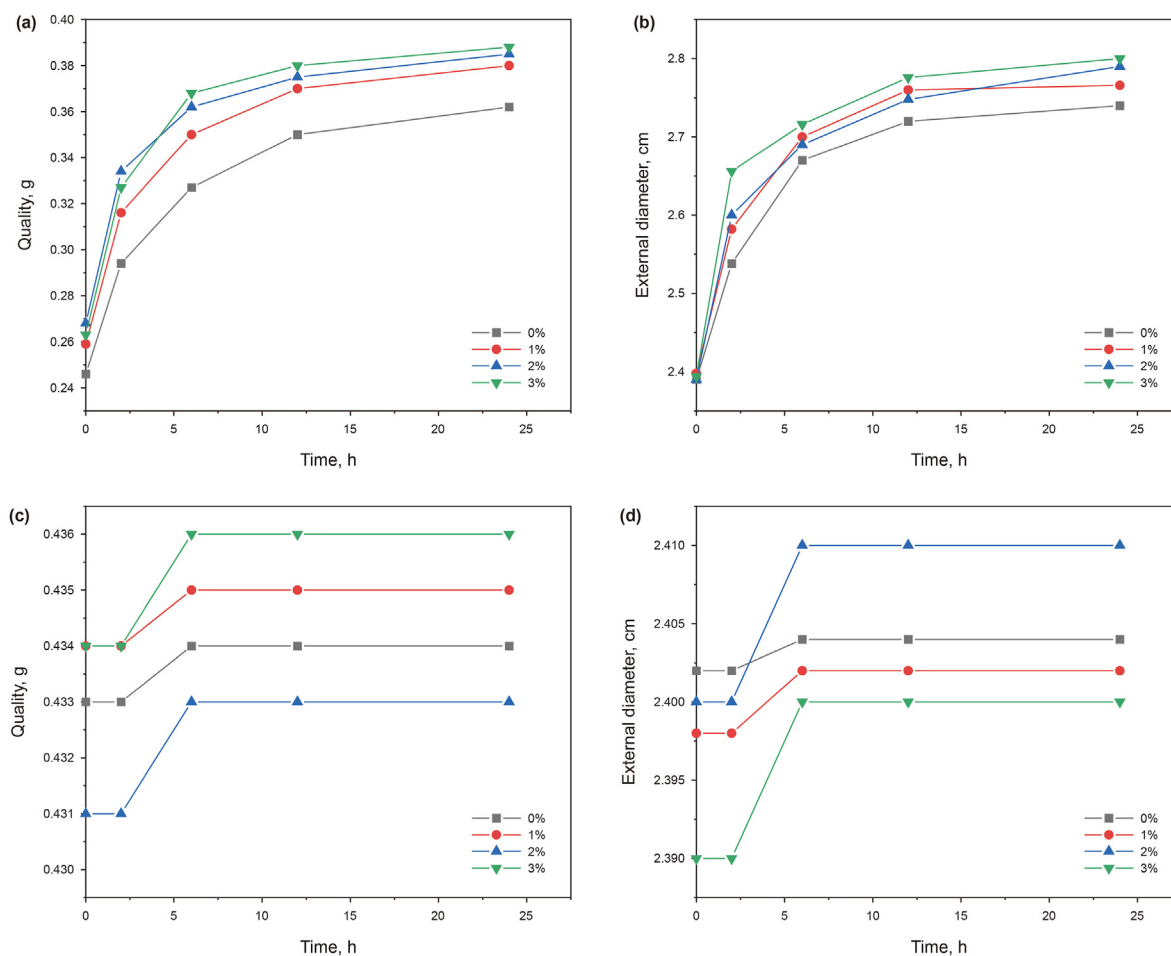


Fig. 11. The effect of HSR concentration on the corrosiveness of different rubbers ((a) quality of silicone rubber; (b) outer diameter of silicone rubber; (c) fluoroelastomer quality; (d) outer diameter of fluoroelastomer).

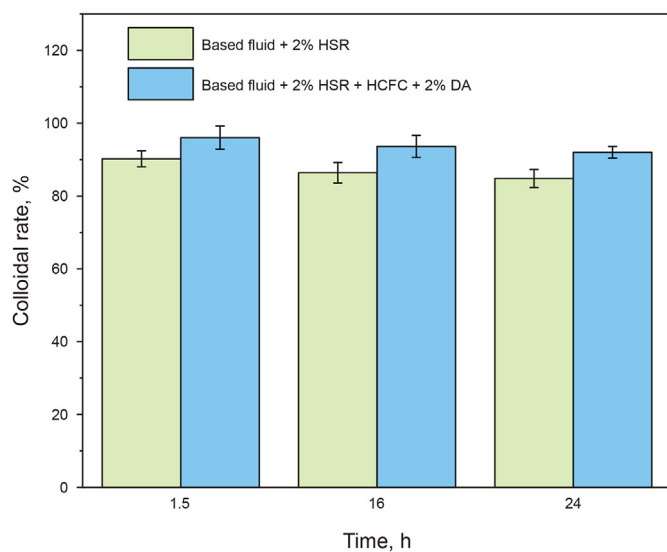


Fig. 12. Compatibility analysis of HSR with conventional additives.

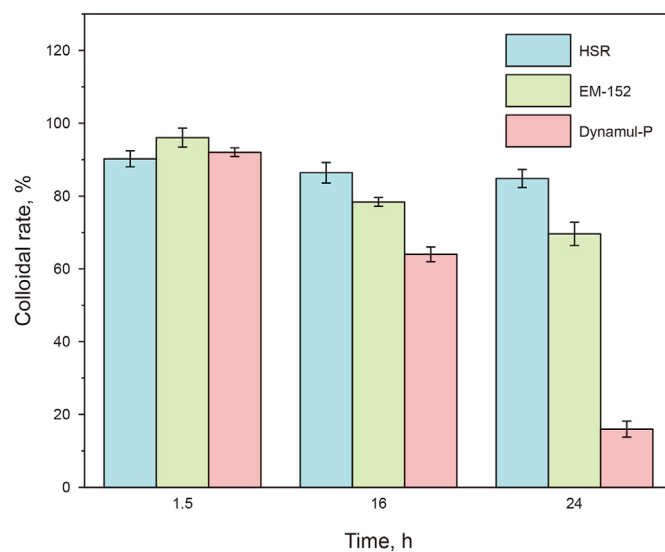


Fig. 13. Performance comparison of different wetting agents.

24 h) of 84.8% for organoclay. However, when HSR was not added to the drilling fluid, the colloid rate is only 6.4%. This indicates that HSR can make drilling fluid have long-term thermal stability. This

analysis is attributed to the ability of HSR to adsorb on the surface of organoclay particles through polar groups and form a spatial network structure with the organoclay particles, thereby

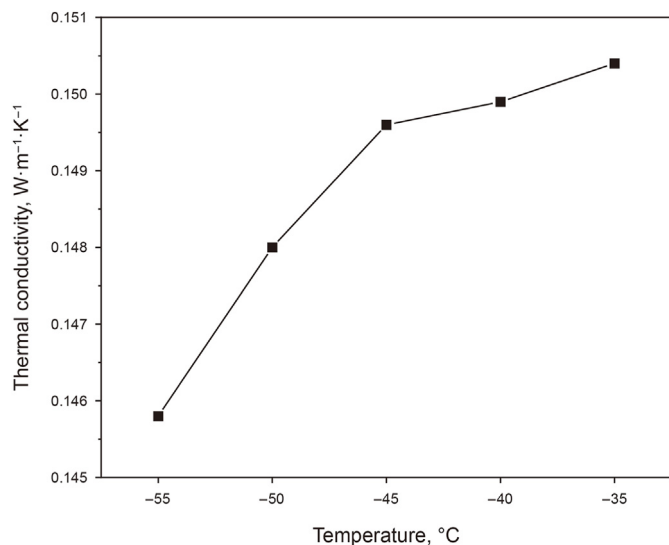


Fig. 14. Thermal conductivity of drilling fluid at different temperatures.

preventing the aggregation of organoclay and significantly improving the colloidal stability of drilling fluid.

3.2.10. Evaluation of thermal conductivity for drilling fluid

The thermal conductivity is one of the important properties of drilling fluid. Especially for ice coring drilling in Antarctic, good thermal conductivity of drilling fluid will be the key to ensuring wellbore stability. This section studied the thermal conductivity of drilling fluid with 2% HSR at different temperatures (Fig. 14).

The thermal conductivity decreased with the decrease of temperature. And when the temperature was below -35°C , the thermal conductivity was always less than $0.1504\text{ W}/(\text{m}\cdot\text{K})$. This indicated that the drilling fluid with HSR also had good thermal conductivity.

3.3. Mechanism studies of HSR

3.3.1. The effect of HSR on particle counts and mean for organoclay

By exploring the effect of HSR on the particles mean and counts for organoclay at different temperatures, the action mechanism of HSR was revealed. In this section, the effect of HSR on the particles

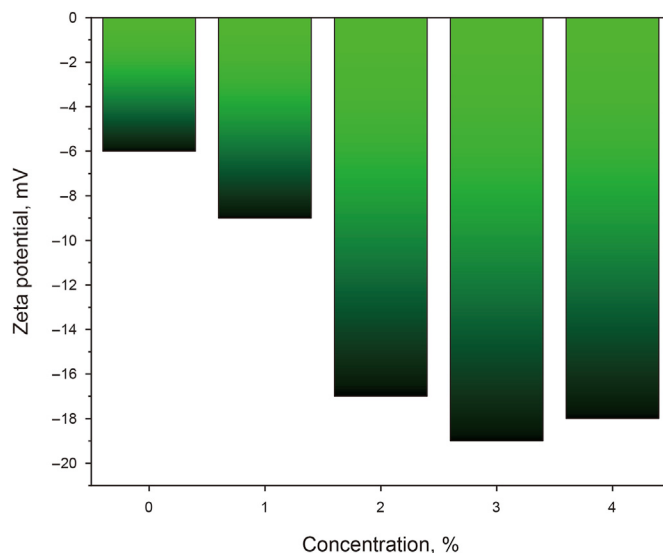


Fig. 16. Effect of HSR concentration on Zeta potential of drilling fluid.

mean and counts for organoclay was explored at different temperatures.

From Fig. 15, HSR was able to significantly increase the particles counts and decrease the particles mean for organoclay under different temperature conditions. In addition, 2% HSR was able to increase the particles counts of organoclay from 3264 to 4216 and reduce the particles mean from 46.90 to $42.54\text{ }\mu\text{m}$ under the condition of -55°C . This indicated that HSR effectively improved the dispersion stability of organoclay for drilling fluids at ultra-low temperatures. It was analyzed that the formation of hydrogen bonds between the polar groups of HSR molecules and the organoclay particles, as well as the entanglement between the long carbon chains formed network structure, which effectively inhibited the aggregation between the organoclay particles.

3.3.2. The effect of HSR on Zeta potential for drilling fluid

Antarctic drilling fluid is a colloidal dispersion system consisting of base oil, organoclay and other additives. The Zeta potential value can indicate its colloidal stability, which in turn can be used to analyze the wetting action mechanism of HSR (Li et al., 2023; Sun et al., 2022b). The influence of HSR on the Zeta potential was

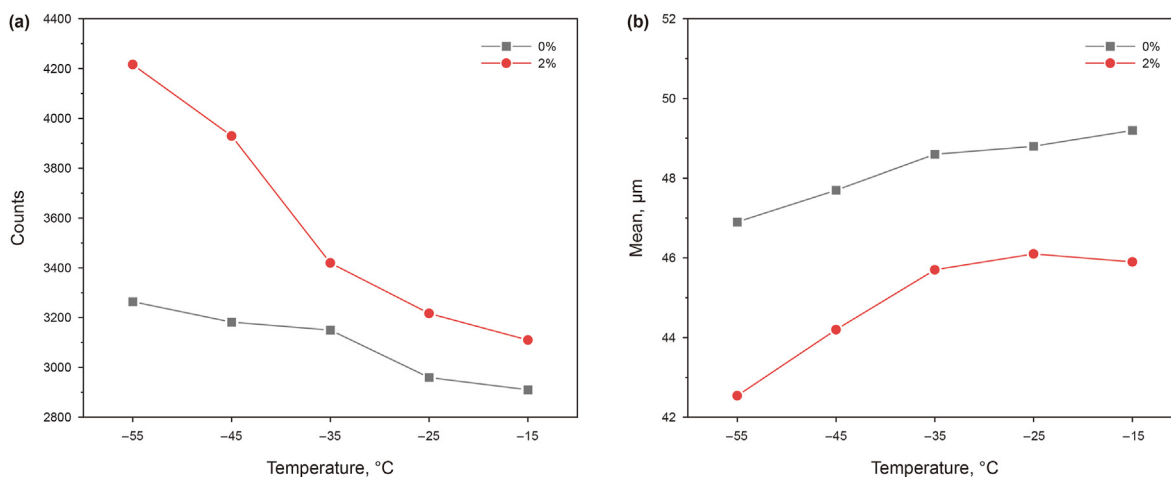


Fig. 15. Effect of HSR on the particles counts and mean for organoclay ((a) particles counts; (b) particles mean).

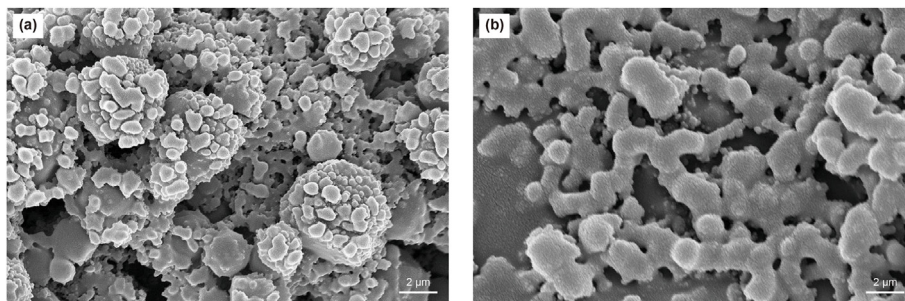


Fig. 17. Effect of HSR on surface micromorphology for drilling fluid ((a) 0%; (b) 2%).

investigated in Fig. 16.

Before adding HSR, the Zeta potential value was about -6 mV. However, as the HSR dosage increased, it showed a gradual decrease. When the HSR concentration improved from 0% to 2%, it decreased from -6 to -17 mV. It indicated that the HSR molecules adsorbed onto the organoclay particles, which effectively inhibited the aggregation between organoclay particles, and thus making the drilling fluid exhibit good colloidal stability.

3.3.3. Microscopic morphology analysis

To further investigate the wetting mechanism for HSR, the drilling fluid morphology was investigated by cryo-scanning electron microscopy.

From Fig. 17(a), before adding HSR, the organoclay was present in the form of large particles, and there is aggregation between particles, resulting in poor dispersibility. The analysis was due to the poor lipophilicity for organoclay, which formed "face to face" connection structure, resulting in poor dispersibility. However, HSR

made the organoclay particles evenly dispersed without the phenomenon of large particle aggregation (Fig. 17(b)). It was analyzed that the HSR adsorbed onto the organoclay particles and interacted with them, which in turn altered the organoclay arrangement in the drilling fluid.

3.3.4. Mechanism analysis

To illustrate thoroughly the effect for wetting agent (HSR) on improving the organoclay dispersibility, the schematic diagram of action mechanism was drawn (Fig. 18).

The HSR molecular structure is mainly composed of long carbon chains, benzene rings and polar groups. The benzene ring and long carbon chain ensure that the HSR can dissolve well with the drilling fluid, and the polar groups may form hydrogen bonds with the hydroxyl groups for organoclay and adsorb on organoclay layer. Before the addition of HSR, the organoclay mainly aggregated in the drilling fluid by the form of "face-to-face" connection. When HSR is added, the organoclay dispersion is significantly improved, and the

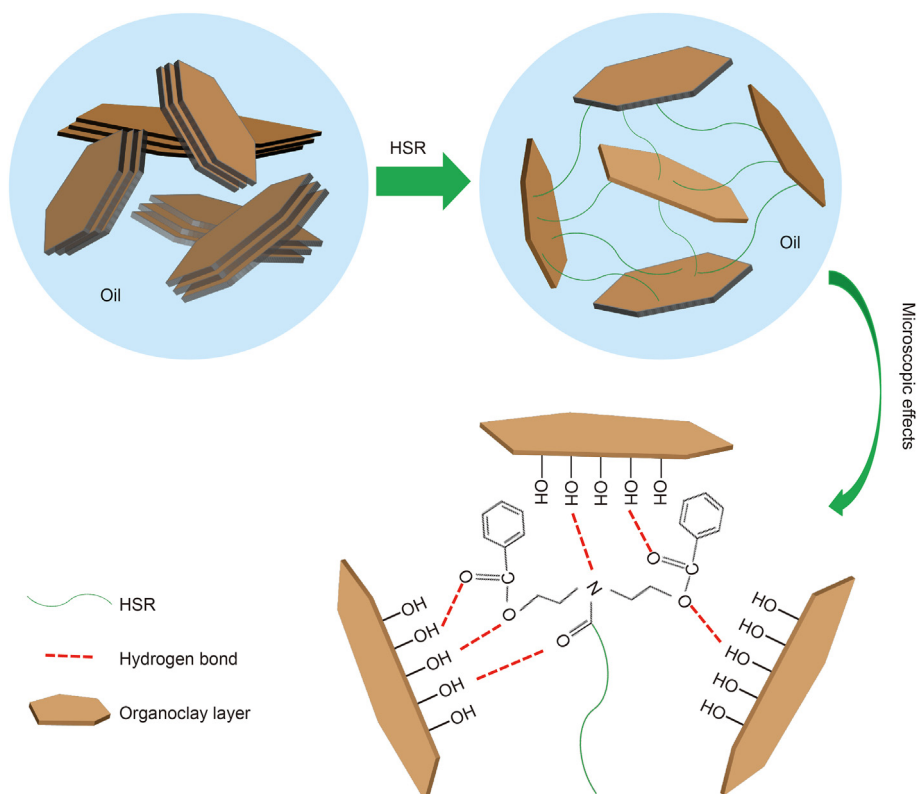


Fig. 18. Schematic diagram of action mechanism for HSR.

rheology performance for the drilling fluid are also effectively enhanced. This is mainly because HSR can adsorb on the organoclay particles through polar groups, forming a spatial network structure with organoclay particles, thereby preventing the aggregation of organoclay particles. In addition, this spatial grid structure also enhances the internal structural forces of drilling fluid, improving its yield point, shear dilution and thixotropy.

In future research, there is a need to further develop new materials that can more effectively improve the wettability properties of drilling fluids at ultra-low temperatures. Meanwhile, due to the high environmental requirements of materials in Antarctica, the new prepared materials also need to be more environmentally friendly.

4. Conclusions

In this study, a new wetting agent (HSR) of drilling fluid suitable for Antarctic region was prepared by oleic acid, diethanolamine and benzoic acid. Infrared spectroscopy and ^1H NMR studies showed that the synthesis of HSR was successful. The performance research indicated that 2% HSR diminished the contact angle of organoclay from 17.2° to 9.4° and increase the colloid rate from 6.4% to 84.8%. Meanwhile, for the drilling fluid, the yield point can be increased to 2.0 Pa at -55°C . And the shear dilution and thixotropy were also significantly enhanced. In addition, HSR had little effect on the two rubber rings, which had almost no corrosiveness. Mechanism studies showed that HSR adsorbed on the organoclay particles and interacted with them, which in turn changed the organoclay arrangement in the drilling fluid. This significantly improved the colloid performance and rheology for drilling fluid. The research promotes the development of Antarctica drilling fluid technology, which has a good application prospect in ice core drilling. However, the main objective of this study focuses on ice drilling, with less consideration given to drilling in rock formations. In the future, efforts can be made to study drilling technology for rock formation.

CRediT authorship contribution statement

Ning Huang: Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Jin-Sheng Sun:** Writing – review & editing, Validation, Supervision, Funding acquisition, Conceptualization. **Jing-Ping Liu:** Writing – review & editing, Supervision, Project administration, Funding acquisition. **Kai-He Lv:** Visualization, Supervision, Resources, Project administration. **Xue-Fei Deng:** Visualization, Software, Investigation. **Hai-Jiang Yi:** Investigation.

Declaration of interest statement

We declare that we have no financial and personal relationships with other people or organizations that can inappropriately influence our work, there is no professional or other personal interest of any nature or kind in any product, service and company that could be construed as influencing the position presented in, or the review of, the manuscript entitled.

Acknowledgments

This research was financially supported by the National Natural Science Foundation of China (No. 52274021) and the National Key Research and Development Program of China (No. 2021YFA0719102).

Abbreviations

AR	Analytical purity
Cr	Colloid rate
HSR	Name of wetting agent
Fbrm	Focused Beam Reflectometer
HCFC	Name of commercial density regulator
DA	Dimer acid

References

- Ahmadi, M.A., Shadizadeh, S.R., Shah, K., et al., 2018. An accurate model to predict drilling fluid density at wellbore conditions. *Egypt. J. Pet.* 27 (1), 1–10. <https://doi.org/10.1016/j.ejpe.2016.12.002>.
- API, 2009. Recommended Practice for Laboratory Testing of Drilling Fluids.
- Chang, X., Sun, J., Xu, Z., et al., 2019. Synthesis of a novel environment-friendly filtration reducer and its application in water-based drilling fluids. *Colloids Surf.* 568, 284–293. <https://doi.org/10.1016/j.colsurfa.2019.01.055>.
- da Silva, R.P., de Castro, T.N., Barillas, J.L.M., et al., 2023. The use of organo-palygorskite as rheological additive in non-aqueous drilling fluids: colloidal stability, contact angle, and cutting's transport ratio. *Geoenery Sci. Eng.* 223, 211499. <https://doi.org/10.1016/j.geoen.2023.211499>.
- El-Masry, J.F., Bou-Hamdan, K.F., Abbas, A.H., et al., 2023. A comprehensive review on utilizing nanomaterials in enhanced oil recovery applications. *Energies* 16 (2), 691. <https://doi.org/10.3390/en16020691>.
- Ghavami, M., Hasanzadeh, B., Zhao, Q., et al., 2018. Experimental study on micro-structure and rheological behavior of organobentonite/oil-based drilling fluid. *J. Mol. Liq.* 263, 147–157. <https://doi.org/10.1016/j.molliq.2018.04.137>.
- He, Y., Du, M., He, J., et al., 2023. An amphiphilic multiblock polymer as a high-temperature gelling agent for oil-based drilling fluids and its mechanism of action. *Gels* 9 (12), 966. <https://doi.org/10.3390/gels9120966>.
- Huang, N., Lv, K., Sun, J., et al., 2023. Study on the low-temperature rheology of polar drilling fluid and its regulation method. *Gels* 9 (2), 168. <https://doi.org/10.3390/gels9020168>.
- Johnsen, S.J., Hansen, S.B., Sheldon, S.G., et al., 2007. The Hans Tausen drill: design, performance, further developments and some lessons learned. *Ann. Glaciol.* 47, 89–98. <https://doi.org/10.3189/172756407786857686>.
- Kim, J.Y., Song, J.Y., Lee, E.J., et al., 2003. Rheological properties and microstructures of Carbopol gel network system. *Colloid Polym. Sci.* 281 (7), 614–623. <https://doi.org/10.1007/s00396-002-0808-7>.
- Leusheva, E., Brovkina, N., Morenov, V., 2021. Investigation of non-linear rheological characteristics of barite-free drilling fluids. *Fluids* 6 (9), 327. <https://doi.org/10.3390/fluids6090327>.
- Lei, S., Li, H., 2018. Research and evaluation of high-temperature and high-pressure drilling fluid measuring system. *IOP Conf. Ser. Earth Environ. Sci.* 170 (2), 022046. <https://doi.org/10.1088/1755-1315/170/2/022046>.
- Li, J., Sun, J., Lv, K., et al., 2023. A zwitterionic copolymer as fluid loss reducer for water-based drilling fluids in high temperature and high salinity conditions. *Geoenery Sci. Eng.* 222, 111200. <https://doi.org/10.1016/j.petro.2022.111200>.
- Liu, F., Yao, H., Liu, Q., et al., 2021. Nano-silica/polymer composite as filtrate reducer in water-based drilling fluids. *Colloids Surf., A* 627, 127168. <https://doi.org/10.1016/j.colsurfa.2021.127168>.
- Liu, L., Sun, J., Wang, R., et al., 2022. Synthesis of a new high temperature and salt resistant zwitterionic filtrate reducer and its application in water-based drilling fluid. *Colloids Surf., A* 651, 129730. <https://doi.org/10.1016/j.colsurfa.2022.129730>.
- Liu, N., Zhang, D., Gao, H., et al., 2021. Real-time measurement of drilling fluid rheological properties: a review. *Sensors* 21 (11), 3592. <https://doi.org/10.3390/s21113592>.
- Liu, N., Xu, H., Yang, Y., et al., 2016. Physicochemical properties of potential low-temperature drilling fluids for deep ice core drilling. *Cold Reg. Sci. Technol.* 129, 45–50. <https://doi.org/10.1016/j.coldregions.2016.06.004>.
- Mahmoud, A., Gajbiye, R., Elkatatny, S., 2023. Application of organoclays in oil-based drilling fluids: a review. *ACS Omega* 8 (33), 29847–29858. <https://doi.org/10.1021/acsomega.2c07679>.
- Mahmoud, S.A., Dardir, M.M., 2011. Synthesis and evaluation of a new cationic surfactant for oil-well drilling fluid. *J. Surfactants Deterg.* 14, 123–130. <https://doi.org/10.1007/s11743-010-1214-7>.
- Martyushev, D.A., Govindarajan, S.K., 2022. Development and study of a visco-elastic gel with controlled destruction times for killing oil wells. *J. King Saud Univ. Eng. Sci.* 34 (7), 408–415. <https://doi.org/10.1016/j.jksues.2021.06.007>.
- Meng, X., Zhang, Y., Zhou, F., et al., 2012. Effects of carbon ash on rheological properties of water-based drilling fluids. *J. Petrol. Sci. Eng.* 100, 1–8. <https://doi.org/10.1016/j.petro.2012.11.011>.
- Meng, X., Huang, X., Lv, K., et al., 2023. Core-shell structured polystyrene microspheres for improving plugging performance of oil-based drilling fluids. *Colloids Surf., A* 674, 131841. <https://doi.org/10.1016/j.colsurfa.2023.131841>.
- Menezes, J.L., Yan, J., Sharma, M.M., 1989. The mechanism of wettability alteration due to surfactants in oil-based muds. *SPE International Conference on Oilfield Chemistry? Houston, Texas, SPE-18460-MS*.
- Motoyama, H., Takahashi, A., Tanaka, Y., et al., 2021. Deep ice core drilling to a depth

- of 3035.22 m at Dome Fuji, Antarctica in 2001–07. *Ann. Glaciol.* 62 (85–86), 212–222. <https://doi.org/10.1017/aog.2020.84>.
- Murtaza, M., Alarifi, S.A., Kamal, M.S., et al., 2021. Experimental investigation of the rheological behavior of an oil-based drilling fluid with rheology modifier and oil wetter additives. *Molecules* 26 (16), 4877. <https://doi.org/10.3390/molecules26164877>.
- Ni, X., Shi, H., Zhang, J., et al., 2023. Modified Laponite synthesized with special wettability as a multifunctional additive in oil-based drilling fluids. *J. Petrol. Sci. Eng.* 220, 111211. <https://doi.org/10.1016/j.petrol.2022.111211>.
- Paswan, B.K., Mahto, V., 2020. Development of environment-friendly oil-in-water emulsion based drilling fluid for shale gas formation using sunflower oil. *J. Petrol. Sci. Eng.* 191, 107129. <https://doi.org/10.1016/j.petrol.2020.107129>.
- Pi, Y., Zheng, Z., Bao, M., et al., 2015. Treatment of partially hydrolyzed polyacrylamide wastewater by combined Fenton oxidation and anaerobic biological processes. *Chem. Eng. J.* 273, 1–6. <https://doi.org/10.1016/j.cej.2015.01.034>.
- Popp, T.J., Hansen, S.B., Sheldon, S.G., et al., 2014. Deep ice-core drilling performance and experience at NEEM, Greenland. *Ann. Glaciol.* 55 (68), 53–64. <https://doi.org/10.3189/2014AoG68A042>.
- Quintero, L., 2002. An overview of surfactant applications in drilling fluids for the petroleum industry. *J. Dispersion Sci. Technol.* 23 (1–3), 393–404. <https://doi.org/10.1080/01932690208984212>.
- Rohling, E.J., Grant, K., Bolshaw, M., et al., 2009. Antarctic temperature and global sea level closely coupled over the past five glacial cycles. *Nat. Geosci.* 2 (7), 500–504. <https://doi.org/10.1038/ngeo557>.
- Sheldon, S.G., Popp, T.J., Hansen, S.B., et al., 2014a. Promising new borehole liquids for ice-core drilling on the East Antarctic high plateau. *Ann. Glaciol.* 55 (68), 260–270. <https://doi.org/10.3189/2014AoG68A043>.
- Sheldon, S.G., Steffensen, J.P., Hansen, S.B., et al., 2014b. The investigation and experience of using ESTISOL™ 240 and COASOL™ for ice-core drilling. *Ann. Glaciol.* 55 (68), 219–232. <https://doi.org/10.3189/2014AoG68A036>.
- Shi, H., Jiang, G., Wang, K., et al., 2019. Synthesis of a dendrimer as rheological modifier for deep-water drilling fluids and study of its interaction with organo-clay at different temperatures. *Key Eng. Mater.* 792, 111–118. <https://doi.org/10.4028/www.scientific.net/KEM.792.111>.
- Skalli, L., Buckley, J.S., Zhang, Y., 2006. Morrow NR. Surface and core wetting effects of surfactants in oil-based drilling fluids. *J. Petrol. Sci. Eng.* 52 (1–4), 253–260. <https://doi.org/10.1016/j.petrol.2006.03.012>.
- Sui, D., Sun, Y., Zhao, J., et al., 2018. Research on a new oil based drilling fluid system. *IOP Conf. Ser. Earth Environ. Sci.* 170 (2), 022044. <https://doi.org/10.1088/1755-1315/170/2/022044>.
- Sun, J., Wang, Z., Liu, J., et al., 2022a. Research progress and development direction of low-temperature drilling fluid for Antarctic region. *Petrol. Explor. Dev.* 49 (5), 1161–1168. [https://doi.org/10.1016/S1876-3804\(22\)60340-9](https://doi.org/10.1016/S1876-3804(22)60340-9).
- Sun, J., Zhang, X., Lv, K., et al., 2022b. Synthesis of hydrophobic associative polymers to improve the rheological and filtration performance of drilling fluids under high temperature and high salinity conditions. *J. Petrol. Sci. Eng.* 209, 109808. <https://doi.org/10.1016/j.petrol.2021.109808>.
- Turner, J., Marshall, G.J., Clem, K., et al., 2020. Antarctic temperature variability and change from station data. *Int. J. Climatol.* 40 (6), 2986–3007. <https://doi.org/10.1002/joc.6378>.
- Wang, T., Sun, M., Pan, Y., et al., 2021. Development a new type of oil based drilling fluid with good temperature resistant. *Energy Sources Part A.* 12, 1–15. <https://doi.org/10.1080/15567036.2021.1970291>.
- Zhao, X., Qiu, Z., Huang, W., et al., 2017. Mechanism and method for controlling low-temperature rheology of water-based drilling fluids in deepwater drilling. *J. Petrol. Sci. Eng.* 154, 405–416. <https://doi.org/10.1016/j.petrol.2017.04.036>.
- Zhang, N., An, C., Fan, X., et al., 2014. Chinese first deep ice-core drilling project DK-1 at Dome A, Antarctica (2011–2013): progress and performance. *Ann. Glaciol.* 55 (68), 88–98. <https://doi.org/10.3189/2014AoG68A006>.
- Zhong, H., Guan, Y., Qiu, Z., et al., 2023. Application of carbon coated bentonite composite as an ultra-high temperature filtration reducer in water-based drilling fluid. *J. Mol. Liq.* 375, 121360. <https://doi.org/10.1016/j.molliq.2023.121360>.